National Measurement System Programme
for
Materials, Mathematics and Modelling Metrology

Programme Document 2013 - 2014
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National Measurement System Programme

for

Materials, Mathematics and Modelling Metrology

Programme Document 2013 - 2014

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NPL

Authorised by:
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NMO
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Annex 1 Amendments to Materials Programme Contracts .............................................. Error! Bookmark not defined.
This document outlines the projects that are contracted from the Department for Business, Innovation and Skills (BIS) National Measurement System Knowledge Base programme for Materials, Mathematics and Modelling to be carried out at the National Physical Laboratory (NPL).

NMS Knowledge Base programmes contain projects that are predominantly geared towards the essential economic and quality of life requirements for definitive measurement standards and techniques. Knowledge Base projects maintain and build on capability already established in the NMS infrastructure; they provide core support for the development and maintenance of SI system over the long-term; they provide traceability to SI through measurement services and the development of measurement standards; and they undertake research to address national challenges centred on particular science disciplines (not addressing horizontal technology challenges).

The Materials, Mathematics and Modelling Metrology programme specifically focuses on the advanced engineering materials, electrochemistry, functional materials, composites and polymers, mathematics, and modelling metrology areas. It currently benefits from projects in the Strategic Capabilities programme that develop a set of capacities, resources, and skills that create a long-term reputational and competitive advantage for the NMS, and supports related projects in the IRD programme aimed at developing new measurement capabilities in areas of strategic importance to addresses national challenges – as identified by government and captured in the NMS strategy documents.
<table>
<thead>
<tr>
<th>Proposal #</th>
<th>LS001P05</th>
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<tr>
<td>Price to DIUS</td>
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| Co-funding Target | £200k |
| Start Date | Oct 2009 |
| End Date | Mar 2013 Ext Sept 2013 |

<table>
<thead>
<tr>
<th>Proposal Title</th>
<th>Multiple AXis Indentation Measurement and Indentation Size effect Exploitation for Surface Engineering, “MAXIMISE”</th>
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<tbody>
<tr>
<td>Lead Scientist</td>
<td>Nigel Jennett, W G Champion, Ben Beake</td>
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**Vision**

Through development and use of novel multi-axis Tribo-Mechanical Spectroscopy, UK industry will generate new advanced materials, engineered surfaces, and tougher interfaces with step change property enhancements obtained from length scale manipulation of structure or composition.

**Impact & Benefits**

High resolution test methods will generate valid data (at all length scales, nano to macro) to enable reliable virtual prototyping and Quality Control (QC); reducing the cost of innovation and increasing the speed to market. Novel surface engineering products worth over £200M p.a. will be enabled (Metals Finishing Institute foresight estimate).

**Support for Programme Challenge**

**Healthcare** – Improved Artificial joints and implants

**Sustainability** – Increased efficiency and lifetime of products (reduced material use/wastage)

**Energy** – Materials for renewable energy generation, Fuel cells and batteries; Fuel-efficient transport

**Advanced Manufacturing** – development of high value added components

**Support for Government Strategies**

**TSB:** – High Value manufacturing; Energy generation (renewables), Advanced materials and nanotechnology (nanosctructured materials and coatings); Environmental sustainability (fuel efficiency); Medicine & Healthcare.

**The Need**

Length scale effects offer step changes in product performance; “Smaller is harder and tougher.” This is true, however, both for small contact sizes and for small volumes of material. It is therefore essential to know which effects are solely the result of a small contact size due to a high-resolution test method, and which effects can be used to gain genuine performance enhancement. A clear understanding of the mechanisms generating length scale effects is needed for input into “right-first-time” design codes. This would speed up innovation and reduce cost but only if there is valid input data available. Some model parameters are currently unavailable. Length scale engineering strategies require higher resolution material property characterisation that provides all the required parameters for FEA analysis of nanostructures and nano-structured materials.

Design rules are needed to harness size effects driving contact performance both in-plane and perpendicular to a surface. Existing high-resolution methods cannot be made quantitative. Vertical indentation methods do not directly evaluate the length scale effects or performance of materials in lateral motion contact. Many key industrial applications e.g. cutting tools, anti-wear coatings in bearings, fuel injectors, cam shafts, hip joints etc. undergo primarily lateral motion contact.

**Current State of the Art (NMI and elsewhere)**

Atomic Force Microscopes (AFM) in general have high-resolution and some multi-axis capability but no tip shape certification route.

Scratch testers generally measure vertical and horizontal force but not displacement. Some capabilities exist to perform scratch testing in instrumented indenters but there is no attempt at multiple axis stiffness measurement and few instruments are stiff enough to take lateral forces.

**The Solution**

Indentation and tribology are well established but operate in only one dimension/plane. NPL will develop a radically new hybrid technology capable of multi-axis Tribo-Mechanical Spectroscopy; using this to extend understanding of plasticity length scale effects suitable for exploitation in surface engineering. New instrumentation combined with advances in contact mechanics algorithms/theory will enable the following:

- Multiple axis contact stiffness measurement at macro, micro and nano scales.
- An understanding of size effects in both indentation and shear (lateral force) contact.
- In situ derivation of contact size; overcoming the need for direct tip shape calibration in SPM.

The project team have extensive experience in instrument design and in contact mechanics theory and propose to develop prototype software and add-on instrumentation to: an existing macro-scratch tester; the NPL MkII Microtribometer; the NPL AFM-based contact resonance spectrometer. This will provide a suite of new capability covering macro to nano length scales.

**Metrology Capability to be Delivered**

A new generation of indentation devices capable of multiple axis tribo-mechanical spectroscopy will be developed and used to measure new properties (e.g. Poisson ratio and surface stress states) and extend understanding of plasticity length scale effects and their exploitation in surface engineering.

**Project Description**

MAXIMISE comprises three development strands: Multi-length scale, multi-axis contact spectroscopy hardware; Multi-axis understanding of plasticity length scale effects; Robust, validated measurement and analysis methodologies.

The aim is to provide both the design rules for step change performance via length scale engineering and the high-resolution measurement technology necessary for product development.

**Exploitation/Spin Offs**

The technology will be licensed for direct exploitation and instrument sales. The market share for next generation
products should eventually reach 10% of the current £7M p.a. (120 indentation systems, 100 tribo-scratch testers p.a.)

**Summary of Technical Work**

New hardware will be designed and built to modify an existing macro-scratch tester (or a macro instrumented indentation tester), the NPL MkII Micro-tribometer and the NPL AFM-based contact resonance spectrometer to obtain contact stiffness measurements in multiple axes and degrees of freedom. Measurements will be made using this equipment to evaluate new contact mechanics models. These models offer a step improvement in surface tribo-mechanical property characterisation and simulation. They use ratios of the different axis stiffnesses to obtain values for properties previously unobtainable by indentation (such as Poisson ratio and the intrinsic stresses in a surface). Alternatively specific stiffness ratios may be used to determine the area of contact without the need for direct measurement of the stylus geometry. This is of particular sought after in the analysis of nano-scale contacts in AFM based instruments, where the ability to determine the contact area would make direct quantitative elasticity measurements on the nano-scale possible; a step improvement in resolution of property mapping.

Recent research has demonstrated the length scale dependence of plastic deformation in nanocompression and nanoindentation. This project will directly investigate the applicability of indentation derived plasticity size effects to tribological contacts, where the yield is the result of shear stresses, by comparing the scratch hardness of highly characterised, differently-sized contacts. Output from this project will contribute to international efforts to improve the definition of the geometry of scratch test indenters and update current ISO standards.

**Synergy with Other Projects**

The project follows on from the size effect work in SE03 “Nanostructured coatings” and complements UP09 “Next generation Contact Mechanics” (which has links to CU03 “modelling”) and A20 – “Residual stress mapping.” It will provide proof of concept evidence and will complement an EU FP7 “Research for SMEs” proposal, call expected before mid 2009.

**Risk**

Preliminary work has had success in measuring surface stress states in micro ranges but elements of this project are extremely challenging. It is currently not known how easy it will be to exclude cross talk between compliances in the different degrees of freedom, nor how sensitive the theory will be to such effects. Mitigation will be to develop instrumentation that covers macro, micro and nano ranges, assisted by adapting existing well-tried instruments, e.g. scratch testers. Size effects may also cause problems for the new contact analysis methods but these will be studied independently and can be identified by comparison with results in the macro range, for example.

**Knowledge Transfer Plan**

To maximise the potential to improve the competitiveness of both the partners and UK industry, access to the new measurement capability at NPL will be promoted to all sectors of industry immediately upon its availability. Satellite projects co-funded by industry will be sought to demonstrate the methodology to potential end users of the technology. Intellectual property (IP) developed will be protected and directly transferred to instrumentation companies via licensing. The anticipated cutting edge scientific and technological discoveries, for example design rules for exploitation of plasticity size effects, will be published in science journals and the Trade Press. The project outputs will also be made available as background IP for an EU project which will develop the technology further and expedite wider direct commercial exploitation by the partners. Improvements to various measurement methods and stylus geometry definitions are anticipated and will be fed directly into ISO standards.

**Project Partners**

Details available on request.

**Co-funding**

Partners will provide substantial in-kind support. An allied project will be submitted to the EU FP7 “Research for SMEs” call, expected before mid 2009.

**Deliverables**

<table>
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<tr>
<th>1</th>
<th>Start: 01/10/09</th>
<th>End: 30/04/11</th>
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<tbody>
<tr>
<td>Proof of concept Multi-axis instrumented tribo-indentation facilities for macro, micro and nano scale contacts.</td>
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**Evidence:**

Validated data set

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<th>Start: 01/04/11</th>
<th>End: 31/03/12</th>
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<tbody>
<tr>
<td>Feasibility study of the use of multiple axis stiffness ratios to eliminate the need to know contact area in the determination of elastic modulus and for the determination of additional properties such as Poisson ratio and surface stress state.</td>
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**Evidence:**

Draft scientific paper for submission to Applied Physics Letters

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<th>Start: 01/10/09</th>
<th>End: 31/03/12</th>
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<tr>
<td>Design Rules for application of Indentation Size Effects to enhance the performance of engineered surfaces in tribological contact.</td>
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</table>

**Evidence:**

Draft scientific paper for submission to J. Phys D
Vision
This project will allow UK companies to predict and improve reliability of electronics, especially in areas of safety critical applications. In these areas industry will achieve improvements by understanding materials properties as higher temperatures are encountered. The project forms part of a series to improve material characterisation in interconnect properties.

Impact & Benefits
Significant reduction in field failures, and hence an improvement in product quality, leading to cost savings.

The UK electronics industry contributes around 10% of GDP (£40billion) and 15% of UK trade (Intellect President report, May 08, http://www.intellectuk.org/component/option,com_docman/task,doc_download/gid,2459/itemid,102/). It is estimated that in the high end high reliability harsh environment market sector that the defect rate is 1 in 10^6. This project will significantly reduce this failure rate.

Support for Programme Challenge
Developments in interconnection technologies support and underpin all the programme areas, which are dependent on reliable electronics. This work enables the development of systems that yield uninterrupted lifetime service in safety critical applications.

Support for Government Strategies
TSB strategy has identified as a key technology area “electronics, photonics and electrical systems”. This work supports many of the identified areas there, for example power electronics, where substrate technology is an intrinsic part of system performance.

The Need
A number of drivers are converging, impacting on the technology window for successful circuit assembly manufacture. Changes are occurring in materials, increasing processing temperatures, harsher use environments, finer scale geometries, and extended supply chains.

Manufacturers need to better understand the electrochemical breakdown within the bulk of their laminates and hence adopt preventative strategies and better work practices. Ideally there should be a rapid test that quantitatively predicts susceptibility to failure. Industry does not have the necessary skills and resources to tackle this problem. Hence, the expertise at NPL will be used to develop a suite of tools to evaluate susceptibility for the formation of sub-surface electrochemical conductive paths, commonly known as conductive anodic filamentation (CAF).

Metrology Capability to be Delivered
An impedance capability for predicting subsurface defects that lead to electrochemical defects. This requires techniques that are sensitive to debonding along potential failure pathways, such as at the glass fibre interface, or ground plane. The development of electrochemical detection techniques, sensitive to various PCB design...
features will also be developed.

**Project Description**

Project comprises four stages: 1) Review impedance techniques to identify both the best sensitivity, and the electrical structures needed within the laminate material. 2) Develop the electrochemical test for quantifying CAF susceptibility, with a focus to identify the incipient causes, including electrode design, materials, and manufacturing parameters. An analytical model will be developed, that includes experimental parameters and predicts time to failure. 3) Optimisation of the impedance technique and electrical structure, in conjunction with a comparison with the electrochemical test method will be made to form an overall test methodology. 4) Establishing material, manufacturing and processing factors that trigger CAF susceptibility, that include mechanical and thermal history, as well as residues from the process using the above techniques.

The aim of the project is to improve laminate integrity and robustness to environmental factors and lead to enhanced quality products.

**Exploitation/Spin Offs**

Improved electronics products, better quality at lower cost. Improved laminate materials and better laminating processes. Development of a test methodology will help NPL generate 3rd party income and secure NPL longer term development of interconnect characterisation tools that assist industry in meeting the miniaturisation goals in microelectronics.

**Summary of Technical Work**

**Electrochemical test methods for CAF**

CAF grows under the combined effect of electric field and humidity. The optimal accelerating stress screening environments will be found. The failure path is critically dependent on electrode design and glass reinforcement structure in the composite, and test vehicle design will be explored. An analytical model will be built taking into account material, design and test parameters to characterise CAF failure.

**Impedance techniques for CAF**

Inherent susceptibility to CAF will be related to intrinsic latent defects in the PCB. This work will explore the sensitivity of electrical based techniques, such as capacitance, scanning electric potential microscopy, and time domain reflectometry. To optimise these approaches the test feature sizes will be modified. Further, for these measurements to be successful it will probably need to be combined with environmental stress, specifically humidity testing, where the moisture can significantly affect the dielectric properties.

**Materials factors effecting CAF**

There are a number of material and process factors affecting CAF, and it is necessary to understand the impact on reliability and electrical properties. The conducting species originating from the process residues will be characterised. The failure path will be characterised which is critically dependent on mechanical processing and PCB architecture, and thermal history during PCA assembly.

**Synergy with Other Projects**

This project will be complimentary to a project running looking at the performance of interconnects. Together they will assist the industry in developing future competitive products with demonstrable reliability. Electrical expertise in TDR and capacitance measurements from other areas in NPL will be drawn upon.

**Risk**

Project risk lies in achieving the application of the impedance techniques to detect the small areas of debonding. Mitigation is achieved by expanding alternative identified methods.

**Knowledge Transfer Plan**

Peer reviewed papers, trade articles and conference papers will be produced. Papers will be submitted to Journal of Materials Research, and trade articles to EPP Europe and Electronics Manufacture & Test.

**Project Partners**

Details available on request.

**Co-funding**

Cofunding will be sought from the partners.

**Deliverables**

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<tr>
<td>Evidence:</td>
<td>The work will be reported in a peer reviewed scientific journal, where the response to the CAF failure with various test conditions is discussed and an analytical model will describe the impact of the various parameters</td>
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<tbody>
<tr>
<td>Evidence:</td>
<td>The work will be reported in a peer reviewed scientific journal, where the usefulness of the above electrical techniques in predicting CAF susceptibility will be presented</td>
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<th>Start: 01/10/11</th>
<th>End: 31/03/12</th>
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<tbody>
<tr>
<td>Evidence:</td>
<td>The work will be reported in a NPL report and will be phenomenological description of CAF, encompassing the known failure modes, and the material and process variables</td>
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</table>
Vision
To enable full field monitoring of large infrastructure assets (power plant, bridges and tunnels) by giving end users confidence in the data produced and by overcoming the problems experienced on-site.

Impact & Benefits
Full-field optical monitoring of strain and displacement using digital image correlation (DIC) allows low cost and frequent examination of large structures where access may be difficult. Full-field techniques can also be used to highlight critical positions where continuous point monitoring sensors should be placed.

This project will benefit the power generation and transport industries by providing a new capability/technique for plant and structural inspection, at reduced cost, whilst providing earlier detection of any defects that may cause an unacceptable safety or serviceability risk or a serious and costly maintenance requirement. The project partners have the necessary skills and contacts to ensure direct and immediate uptake of the project outputs and communication/transfer to other potential users within the UK to enable them to respond to the environmental and social challenges faced within their industries.

Support for Programme Challenge
Supports the NMS Materials Energy and Sustainability Challenges.

The Need
As one of the ways for the UK to meet its emissions targets and not be overly dependent on foreign sources of energy then nuclear energy generation must be increased. As each new nuclear power plant (NPP) costs approximately £3 billion, then life extension of existing plant is a priority. As each NPP produces £1M electricity a day reducing outages and repair time is of high priority.

Monitoring is part of the overall operations and maintenance costs of a nuclear power plant (estimated at £90 million per annum) and can be a significant economic burden to the asset holder as typical site inspections cost up to £20k per day.

The focus on sustainability and the low carbon agenda is also driving the need for increased efficiency in conventional power plants. The target of extended lifetimes, reduced emissions and more extreme operating conditions are placing severe demands on existing plant. Critical components are being worked harder and for continued safe operation, must be monitored more reliably.

Similarly in the transport infrastructure, transport corridors exist where loss of capacity can have wide scale economic and social impact. There are currently over 300 km of tunnels and many thousands of bridges and other structures that need regular, costly inspection and repairs. New optical inspection techniques based on DIC will make this process more cost-effective.

Current State of the Art (NMI and elsewhere)
DIC is a full-field optical technique for making low-cost remote surface displacement measurements. Aspects of the technique have been developed in recent NMS projects and industry funded research projects, positioning NPL as one of the leading exponents and developer of the method. NPL has already shown the potential of the technique to several power generation and transport infrastructural asset owners leading to a series of industrial feasibility case studies for:

- Nuclear industry
  - Waste storage, power generation plant
- Transport infrastructure
  - Network Rail: Tunnels, bridges
  - Highways Agency/URS: Bridges, road side structures

Work has also been presented at relevant conferences such as BINDT2009 and through published papers, in industry journals such as Concrete magazine and Civil Engineering, describing the state of the art application of these techniques for civil engineering structures. There is considerable research in optical measurement techniques by French academia, but these are mainly aimed at laboratory measurements.

To date most applications for DIC have been laboratory-based. There is a significant challenge and opportunity in this project for NPL to become the first to develop a robust DIC system that can be used on-site, and cope with the challenges of changing environment and system repositioning, where the target is to resolve damage or changes in the structure of the order of 1mm from a stand off of several metres. The non-ideal conditions experienced during on-site operation reduce the reliability of the data especially over long periods of time. Anything that affects the appearance of the object being imaged or the image itself degrades the quality of the data that can be derived.

The Solution
Confidence in decision making is essential for infrastructural asset owners to prevent costly unnecessary repairs and shutdowns whilst maintaining safety standards. The work highlighted here focuses on overcoming the issues experienced in the demonstration phase not present under ideal laboratory conditions.
Currently, making in-situ DIC measurements in the field is compromised by the lack of methodologies to cope with the effect of disturbance, accurate repositioning of the imaging equipment and the effect of changes in appearance of the structure due to the weather.

Additionally for high definition full field monitoring it is essential to minimise the data processing requirement to maximise speed without affecting accuracy in a scenario where camera resolution is still increasing.

**Metrology Capability to be Delivered**
- Reliable protocols for calculating the uncertainty associated with each step in a full-field measurement to underpin structural assessment and statement of uncertainty
- Methods that use the point-to-point image uncertainties to provide uncertainty estimates of end-user relevant measurands
- Methodologies for using cameras that need to be repositioned
- Image treatments to enable data to be taken from structures that change appearance (such as weathering, wet and streeky concrete, oxide scales)
- Techniques to optimise data capture and processing requirements for given uncertainties

**Exploitation/Spin Offs**
Doosan Babcock are interested in developing DIC for nuclear structure monitoring. URS Corp. are interested in DIC for highways applications. Network Rail are interested in DIC and optical techniques for tunnel inspection and continuous monitoring for repairs within structures.

**Project Partners**
Details available on request.

**Co-funding**
TSB project IMPACT in Energy Generation and Supply Carbon Abatement technologies (€115k) requires the application of DIC for high temperature non-contact in situ monitoring of creep strain in power plant components (E.ON, RWE, Npower, Doosan Babcock, Alstom). TSB project SUMS to be resubmitted with Doosan Babcock in a future nuclear power call within the lifetime of the project.

**Synergy with Other Projects**
Current IRD projects on wireless sensors for NPL bridge specimen, full field strain measurement in miniaturised testing, and proposed project MAT 2010 33 on monitoring subsurface conditions in civil engineering structures.

**Summary of Technical Work**
Within this project in-situ image comparison techniques will be developed that provide practical solutions for measuring cracking and critical deflections in concrete, steel and masonry structures and components such as those found in power plant and civil engineering structures. The measurement of creep strain in high temperature power plant components like pipes is another application of in-situ DIC measurement.

The following steps are needed to provide robust in-situ measurements:
- Develop techniques to mitigate the effects of the environment, such as rain, fog, haze, extreme temperature, and lighting changes
- Develop procedures for accurate repositioning of cameras between measurements and an understanding how the above factors affect the measurement accuracy
- Benchmark the system against conventional techniques (LVDTs, strain gauges etc) and DIC software via the “DIC Challenge”, to be organised by NPL
- Develop computing resources and algorithms to speed up the calculation and calculate the uncertainties
- Undertake industrial case studies to test and demonstrate the outcomes of the project

**Risk**
Feasibility studies have identified that DIC and optical techniques can be used for Civil Engineering applications. The risk of not doing this project would allow French, Japanese and German competitors to establish a base of non-UK expertise.

**Risk 1:** DIC computation is too complicated or time-consuming. **Mitigation 1:** Initial developments in parallel computing have shown a development route that is easily scalable

**Risk 2:** Access to infrastructure for trials needed to develop and validate the scientific solutions developed in this project **Mitigation 2:** The NPL bridge is a very well instrumented, low capital investment suited to this project. Members of the SHM IAG and initial contacts with the Nuclear industry and Network Rail have also identified suitable large scale demonstrators to help promote these techniques.

**Knowledge Transfer Plan**
This will be performed through the use of the sensors KTN and MBE KTN network of contacts and workshops organised by the Forum on Engineering Structural Integrity and the British Society of Strain Measurement. The project partners have the commercial skills and appropriate supply chain representation to develop methods and ensure their deployment and exploitation in the UK and the rest of the world.

The Civil Engineering Sector is very layered and based on recent discussion we believe all layers would benefit from this project; the project partners have contacts in sensor manufacturers, engineering consultants, asset owners and regulators and so can widely disseminate the outputs of this project.

**Deliverables**

<table>
<thead>
<tr>
<th></th>
<th>Start: Apr 2010</th>
<th>End: Mar 2013</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Co-funding TSB IMPACT</td>
<td></td>
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<tr>
<td></td>
<td>Evidence: Technique Trials</td>
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</table>
Vision

To place NPL at the forefront of thermal analysis of advanced materials at extreme temperatures, enabling the UK to take the lead in application of high temperature materials.

This project delivers a validated measurement system, equipment, reference materials, and algorithms to measure the properties of multi layered components.

Part cofunding has been secured from the European Metrology Research Programme (EMRP)

Impact & Benefits

Materials that can reliably operate at higher temperatures will yield increased energy efficiency of power plants and result directly in lower CO₂ emissions. Fossil fuel power plants contribute ~34 % of the CO₂ emissions and 40 % of electricity globally. A 2% efficiency gain will save at least 2% of the CO₂ emissions and this is a key and ambitious goal of European policy in support of the Kyoto Protocol.

Support for Programme Challenge

This project supports the “Energy Generation & Transmission, Emissions Reduction” challenge on the Materials Programme top-level roadmap.

The Need

The improvement of the thermal efficiency of power plants requires the development of high temperature components utilizing refractory alloys and ceramic coatings. The accurate determination of thermophysical properties of coated materials is the key to their rapid uptake, as end users need to have confidence that the components will perform.

The need is to support the development and application of advanced materials and coatings operating at higher temperatures in future boilers and turbines, by characterising oxide layers and coatings on internal and external surfaces.

- Steam temperatures of modern power plants are in the region of 600 °C. By increasing this to 650°C - 700 °C an improvement in the efficiency of between 36% to 50% could be achieved.

- For gas turbines the increase of gas inlet temperatures from 1300 °C to 1500 °C would result in efficiencies of about 60 %. This increase in efficiency not only lowers emissions but also makes Carbon Capture technology viable.

- Coating materials are only available as coatings not in a bulk form that would permit thermal property measurement in existing equipment.

Current State of the Art (NMI and elsewhere)

- Direct measurement of thermal conductivity is limited to below 800°C.
- Thermal diffusivity measurements are possible above 1600°C but are unvalidated. Work on oxides and coatings at these temperatures is limited, existing methods do not analyse the test data to give the properties of multi layered components
- Reference metrological setups and methods for the measurement of thermal properties (notably thermal diffusivity, specific heat and thermal expansion) of advanced materials (e.g. refractory metals, ceramic, coated materials) at high temperature (> 1000°C) and very high temperature (up to 1600 °C).

The solution

The solution provided by this project is to combine modeling with measurement and validate this approach using calibrants and well characterized layered materials. The data can then be used to optimize the design of multi layered coated systems and to improve the lifetime prediction of materials that develop oxides during their use.

Metrology Capability to be developed

This project will develop:

- Reference metrological setups and methods for the measurement of thermal properties (notably thermal diffusivity, specific heat and thermal expansion) of coated materials (to at least 1600 °C).
- A measurement system to ascertain the thermal properties of oxide films formed in service to provide fundamental data vital for accurate modelling and prediction of oxidation and lifetimes.
- Models of the metrology set up to understand the response of these materials systems during measurement to enable thermal properties of their component parts (substrate and coating) to be measured.
- Reference materials and methodologies to transfer calibrations to third parties and enable validation.

Exploitation/Spin Offs

The outputs that can generate supporting revenue include:

- Sales of certified reference materials for use in high temperature DSC instruments.
A measurement service offering extended temperature range of specific heat capacity, thermal expansion and thermal diffusivity capability, allowing calculation of thermal conductivity to at least 1600°C.

Licence for algorithms to be embedded in manufactures software

World leading measurement capability for the characterisation of high temperature, innovative structural materials and coatings.

Project Partners
Details available on request.

Co-funding
This project will be cofunded by a collaborative EU project entitled: “POWERPLANT: Metrology for improved power plant efficiency” which will provide £150k of support.

Synergy with Other Projects
- EMRP Metrology for New Generation Nuclear Power Plants
- Proposed project for ITCC WG on thermal diffusivity round robin
- IMPRESS project on reactive materials, EU follow up project.

Summary of Technical Work
Calorimetry
- Produce high temperature calibrants for DSC measurements, e.g. NiPd, Co-C or iron, with particular care being taken to control atmosphere up to at least 1600°C.

- Use levitated drop calorimetry for characterisation and evaluation of potential calibrant materials with sharp metal-liquid transitions, for comparison with other methods

- Calculation of thermal conductivity values of nickel base alloys and TBCs obtained using thermal expansion measurements and specific heat data validated by new high temperature calibrants.

- Study effect of measurement atmosphere on specific heat measurements, and the relationship between thermal expansion and microstructure for nickel base alloys

Thermal diffusivity and conductivity
- Validated measurement of thermal diffusivity of nickel alloys and industrially relevant alloy/TBC systems to at least 1600°C for calculation of thermal conductivity

- Modelling of the thermal properties of coated materials using existing NPL Thermol package

- Modelling of the metrology of coated materials to support analysis of raw experimental data and conversion to materials properties using finite element approach

- Design and manufacture of specimens with layered structures and predictable properties to validate methodology.

- Coding and embedding of algorithms into equipment software to enable data analysis

Risk
Production of suitable reference materials in the quantities required will be a considerable challenge, creating reproducible material for distribution, could apply to EU to create certified reference materials.

Knowledge Transfer Plan
- Outputs from the project will be disseminated via the UK Special Metals Forum and the Thermal Methods Group of the Royal Society of Chemistry.

- Presentations will be made at the North American Thermal Analysis Society Conference, and the International Thermal Conductivity Conference

- Presentations will be made at NPLs existing IAGs and at a new IAG formed to focus on materials metrology at high temperatures

Deliverables

<table>
<thead>
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<td>Co-funding EMRP POWERPLANT - A validated capability for obtaining thermal conductivity values for coated materials to at least 1600°C.</td>
<td>Evidence: A peer reviewed paper on the science of this measurement technique</td>
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<td>2</td>
<td>Validated models for measurement of high temperature properties of layered materials systems</td>
<td>Evidence: Peer reviewed paper and workshop</td>
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<tr>
<td>3</td>
<td>Reference materials for validation of high temperature systems up to 1600°C</td>
<td>Evidence: Peer reviewed paper with round robin results and statement of uncertainty</td>
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<tr>
<td>Proposal #</td>
<td>MAT/2010/CF2 TSB SUSCOAT</td>
<td>Price to NMO</td>
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<td>Proposal Title</td>
<td>High Velocity Erosion of Coatings and Wear Resistant Materials</td>
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<td>End Date</td>
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<tr>
<td>Lead Scientist</td>
<td>Mark Gee / Andrew Gant</td>
<td>Project Champion</td>
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</table>

**Vision**
This project will enable UK designers and engineers to test and predict the durability of materials subjected to high velocity impact erosion from fluids through the provision of new world leading technical capability.

This work is cofunded by the TSB collaborative project SUSCOAT

**Impact & Benefits**
The provision of a leading edge facility for high velocity erosion testing, metrology and advanced engineering materials characterisation will enable UK designers and engineers to develop new products that have extended durability and capability by comparison with the competition. The better scientific understanding of high velocity erosion will give a step-change improvement in the performance of materials in exacting sectors as diverse as aero engine structural components, power plant manufacturers and the oil extraction industry. An example of the benefit that will be achieved is an additional market of over £3M for the coating of turbine blades for industrial gas turbine applications.

**Support for Programme Challenge**
This project supports the materials programme challenge of environmental sustainability with respect to resource efficiency, the TSB Advanced Materials and High Value Manufacturing strategies, aligns with the strategic goals of NPL and forms a key element of the performance of engineered surfaces group’s road map.

**The Need**
Resistance to water droplet erosion is becoming a crucial constraint to further improved performance of steam turbines for power plant. Pushing thermal efficiencies (by ~10%) in the Rankine cycle through the use of supercritical steam is constrained, amongst other factors, by materials performance limits. Specifically in LP turbines, the use of increased main-steam and reheat pressures will increase the moisture content at the LP turbine exhaust. This would increase the erosion rate on the last-stage blades and may necessitate additional blade protection. There is thus a pressing requirement to establish a national facility for the testing of new and improved materials that will allow the operation of these turbines under more extreme conditions.

Methods to assess the damage of structural components for supersonic aircraft from rain are also needed. This is driven by the increasing use of polymeric composites in aeroplane construction. These are sensitive to water droplet erosion, leading to a need to evaluate candidate damage reduction technologies.

**Current State of the Art (NMI and elsewhere)**
There are only a few water droplet erosion tests systems worldwide. In the US there is a supersonic water droplet erosion rig at EPRI. In Europe there are facilities in Paris, in Alstom, Switzerland and EADS in Munich. These test systems are often large test facilities with relatively simple systems for the delivery of either a spray of droplets or a jet of water. The main technical difficulty is the need to produce a supersonic water stream; this is accomplished either by moving samples at high velocity through a spray of droplets or by accelerating a jet by using high pressure. In most of the current systems there is little or no provision for measuring the interaction of the water droplets or jet with the sample, so that understanding of the mechanisms that underlies the degradation of materials can only be obtained through post-test analysis. Normally materials damage is monitored by the simple measure of materials loss; this do not normally allow for thorough studies of the relationship between materials structure, the mechanisms of damage, and the volume of damage that occurs. Many of the systems also have poor control of test parameters.

NPL is a world leader in developing the metrology of methods to evaluate the degradation of materials from wear. Previous work has included the development of a novel stepwise method for the evaluation of the mechanisms of erosive wear, and the development of a new test system for fluid jet particulate erosion testing.

**The Solution**
The solution is to provide new capability for the UK for supersonic water jet erosion testing. The aim would be to develop a test system with full control of test parameters that is uniquely instrumented so that an understanding of the damage mechanisms that underly the wear of materials subject to these modes of degradation can be developed. This would not only enable guidance for UK industry on the testing and selection of materials to be developed, but would also form the foundation for a predictive capability that could be used in design. The new facility will also be essential to support the Suscoat TSB project.

**Metrology Capability to be Delivered**
- A new test facility for supersonic water jet erosion testing will be designed and built.
The facility will be fully instrumented so that real time information on degradation of samples and the interaction of the erodent with the surface can be obtained to give a scientific understanding of the mechanisms of wear that are occurring.

The establishment of the new facility will be coupled with the development of other characterisation techniques to give further information on wear processes.

Exploitation/Spin Offs
The intention is to establish a national centre for high velocity erosion testing. There is significant demand for testing in these areas from the power generation and oil sectors. It is also expected that the new facility will be further exploited through other projects funded by the EU and TSB.

Project Partners
Details available on request.

Co-funding
This project is supported by the TSB SUSCOAT project that is concerned with the sustainable development of new coating systems. Other co-funding is also expected from a collaborative project with industry in the fluid jet particulate erosion area.

Synergy with Other Projects
This project follows on from the fluid jet particulate erosion testing in project SM05 under the characterisation programme, but extends the work to higher velocities more relevant to industrial applications. There has also been previous work on gas blast solid particle erosion testing in previous NMS projects that underpins the development of the new facility.

The project will make use of and extend microstructural characterisation techniques that will be developed in project LS01: “Traceable 3D measurements to underpin Optical and Electron Microscopy”. It also has aspects that are complementary to the IRD project T11 on monitoring the degradation of materials from wear. The project will apply stepwise techniques to determine the mechanistic evolution of damage to surface that have been developed in previous NMS projects.

Summary of Technical Work
Deliverable 1 is concerned with the design and manufacture of a test facility, and test method development for supersonic water jet erosion. The first stage is to decide on the best design from a number of competing potential routes balancing the need for a compact and reliable test system with the requirements for instrumentation and access to give a versatile well characterised test system. Manufacture of the facility will be carried out, probably in house, but making use of the latest technology available for key components such as the high pressure pump.

The facility will be designed and constructed with instrumentation such as sensitive load cell technology that can be used to determine the impact of individual droplets on the test sample with high speed digital imaging of the impact event. This will enable a scientific understanding of the degradation mechanisms for water jet erosion to be determined. This will be complimented by stepwise erosion experiments and advanced microscopy of the degraded surface.

The aim is to develop unique world leading scientific understanding of the mechanisms of wear from water droplet erosion that will put NPL on the map as a key centre for erosive wear testing.

Risk
The main risk in the project is that the new water jet erosion test facility will not perform in the required way. This risk will be mitigated by careful examination of the alternative methods to achieve the required function. There is a risk that the instrumentation necessary to determine the mechanisms of degradation will not perform as required, but the experience of NPL in this type of task should provide success in this regard.

Knowledge Transfer Plan
Primary knowledge transfer for this project will take place through the collaborative partners in the co-funding projects.

The UK national facilities developed in this project will be promoted as a measurement service for the oil and power generation industries on a UK, European and worldwide basis.

A major aim of the project is to make step change progress in developing a scientific understanding of the mechanisms that underlie degradation of materials from water jet and fluid jet particulate erosion. This new science will be published in high impact materials science and physics journals.

The project results will also be communicated to the surface technology Industrial Advisory Group.

Deliverables

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<tbody>
<tr>
<td>1</td>
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<td></td>
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<tr>
<td>New UK facility for supersonic water droplet erosion</td>
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<tr>
<td>The new facility will be instrumented to give the ability to develop understanding of mechanisms of water droplet erosion leading to improved prediction of materials performance for design.</td>
<td></td>
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<tr>
<td>Evidence: Submission of peer reviewed paper</td>
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Vision

This project will develop the metrology required to accelerate the development of a new generation of high energy density capacitors and capacitive boost technology for mass-market hybrid vehicles in the medium term and mass energy storage in the low carbon economy of the future.

A TSB cofunded project on this theme has already been won and this project will provide the key new metrology to the partners.

Recent research work has reported ferroelectric perovskite materials with a permittivity as high as 450,000. Our calculations show that a material with these properties could provide energy densities of 0.5MJ/kg (1µm films at 42V) - comparable with Li-ion batteries.

Impact & Benefits

Transport accounts for 24% of all UK CO₂ emissions, the majority of which is produced by cars. Hybrid vehicles are a key part of the UK strategy for reducing vehicle emissions and capacitor storage is a key enabling element in making this possible and in stabilising the supply of energy for power from renewables such as wind and solar.

The development of two new core capabilities will deliver key support for industry wishing to develop new high permittivity materials for energy storage and energy conversion applications. This could include hybrid vehicles, and storage for all-electric vehicles and renewable energy supplies. Timely development in this field would provide a wide range of UK companies with a commercial lead in low carbon vehicle technology and more widely in the emerging low carbon economy.

Support for Programme Challenge

Energy and transport challenges - this project will directly support the challenge of reducing carbon emissions from transport and energy storage for renewables.

The Need

While the UK has achieved reductions in CO₂ emissions overall, those from domestic transport have increased 9% from 1990 to 2006. The majority of these emissions are from cars. Without new technologies, the emissions from this sector are set to continue to increase with improvements in efficiency of conventional technologies being offset by increasing demand for private transport. Hybridised vehicles can meet this challenge by providing reduced CO₂ emissions and enhanced efficiency whilst retaining acceptable levels of performance only if step changes in the capacity and power delivery per kg of storage technologies can be achieved.

Development of new high permittivity materials based on novel compositions and nano-structured ceramics would therefore represent a materials technology platform to design such high energy storage applications.

This development requires a new approach to materials design specifically for energy applications. A key part of the design cycle is new metrology to assess the energy capacity and reliability of materials under high energy and high power conditions.

Current State of the Art (NMI and elsewhere)

Current metrological approaches to dielectric characterisation of materials properties focus on low electric field coefficients such as permittivity. Ferroelectric materials are highly non-linear and saturate at high electric fields. This can be assessed by “PE loop” (charge voltage) measurement but current technology uses simple waveforms that don’t adequately replicate real life conditions, and does not address traceable measurement of energy density or power capacity of these non-linear ferroelectric materials. NPL has been at the forefront of development of the linear methods and models [NPL Best Practice Guide no. 34 and refs therein] and is in a unique position to extend UK capability with this project. Electrode and space charge effects can significantly affect the high field dielectric properties of thin ferroelectric films. Techniques for probing the dielectric properties through the depth of a material in the thin (10-100nm) layer near the electrode are not currently available, a subject of strong interest [Nature Materials 8, 366 - 368 (2009)].

The Solution

The capacity and reliability of energy storage requires a new integrated metrological approach. This must address the capability of a material to store large amounts of energy for long periods of time whilst being able to deliver high power when required. This integrated measurement capability must address energy capacity at high electric fields, the effects of saturation, leakage current, breakdown, and fatigue at very high electric fields. New metrology is also required to measure the effects of electrodes and space charge at electrode interfaces, which is seen to radically affect the performance of all ferroelectric thin films and multilayered structures. Because energy scales with the square of voltage ($0.5C V^2$) increasing the working voltage would significantly increase energy density. Consistent metrology of leakage current and breakdown processes would permit designs to push
the limit and provide predictive measurement of breakdown phenomena. Metrology challenges associated with breakdown measurements ferroelectrics (which are also piezoelectric) include transient currents, acoustic emissions, Barkhausen noise etc. In this project we will refine NPL’s capability regarding dielectric breakdown – based on our previous track record in a pan-EU project ‘Ceramelec’.

**Metrology Capability to be Delivered**

- Integrated system for (−40C to +200C):
  - PE loop measurement system for traceable measurement of energy density using programmable complex waveforms at high electric field.
  - Measurement of pulse power response (traceable measurement of power density) for charge and discharge cycles. This includes development of NPL’s dielectric and withstand voltage breakdown capability.
  - Measurement of fatigue response to rapid current transients and high electric fields.
- New high frequency (up to 1GHz) LIMM (Laser Intensity Modulation Method) for depth sensing measurement (10-100nm) of the effects of electrodes, space charge layers, trapped sites at the electrode interface on dielectric and breakdown properties. This approach is somewhat high risk but is backed by NPL’s world leading position with regards the LIMM technique. [see J. Am. Ceram. Soc., 91 [7] 2176–2181 (2008)].

**Exploitation/Spin Offs**

Integrated measurement of energy and power capacity will be of significant interest to the energy storage industry generally (batteries, super capacitors) and NPL’s new capability will be offered commercially.

Commercialisation of the output of the accompanying TSB project will require the facilities and expertise developed during this project thus providing opportunities for further collaborative R&D and direct commercial service provision.

Scientific expertise in the measurement of complex functional ceramic properties, fatigue and dynamic response in ferroelectrics will generate opportunities for participation in larger R&D projects e.g. FP7.

**Project Partners**

Details available on request.

**Co-funding**

TSB Technology Programme project Advanced Capacitors for Energy Storage (ACES) won.

**Synergy with Other Projects**

NMS FM04 (completed) measurement of piezoelectric properties of actuators in harsh environments. Applies the expertise to energy storage across a wide temperature range and conduction processes in ferroelectrics.

**Summary of Technical Work**

- Design and build integrated system for application of high electric fields and measurement of current and charge. Arbitrary waveform capability will allow loop measurement and transient / dynamic charge and current pulse response.
- Develop methods and analysis tools for assessment of energy and power density from the above.
- Extend the system to measure changes in energy and power density (fatigue) over complex cycling regimes.
- Develop new high frequency LIMM method and apply to the analysis of electrode interface and space charge effects in thin film ferroelectrics
- Multiphysics modelling of non-linear high field polar response electrode interfaces.

**Risk**

Time constant of thermal response in LIMM measurement may reduce signal amplitude at high frequencies. This may compromise the depth resolution of the new technique, although it is expected to provide ground-breaking new metrology for ultra-thin films and buried layers in multifunctional (polarisable) materials.

**Knowledge Transfer Plan**

The scientific progress will be reported in leading scientific journals and disseminated through Piezo Institute training, the KTN network and international conferences. Markys Cain is Director of the Piezo Institute and chairs the International VAMAS Piezo Technical Area and various Smart Materials and functional materials committees.

**Deliverables**

<table>
<thead>
<tr>
<th>Deliverable Title</th>
<th>Start: 01/05/2010</th>
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<tr>
<td>1</td>
<td>Integrated PE Loop and transient power measurement apparatus with switching cycle fatigue operational mode.</td>
<td>Evidence: Peer reviewed journal paper - metrology and characterisation of energy storage in new high permittivity ceramic materials</td>
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<tr>
<td>2</td>
<td>High frequency LIMM system – new metrology capability</td>
<td>Evidence: Peer reviewed journal paper – measurement of electrode and interface effects on energy storage in high permittivity ceramics</td>
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</tbody>
</table>
Vision
To extend the current capability of NPL and develop facilities, expertise and capability to characterise the performance of current and emerging materials for use in advanced cycle, zero emission power plant (ZEP) using advanced laboratory testing techniques.

Impact & Benefits
This project will deliver the capability to expose, test and characterise a range of materials in a range of controlled atmospheres, thereby improving the applicability of test results by conducting the test in accurately simulated conditions. Impact will be evident through the proving of new material systems for new ZEP based on austenitic and coating technology rather than expensive Ni-based super alloys.

Support for Programme Challenge
This project supports the UK energy challenge (for a low carbon economy whilst maintaining energy security), aligns with the strategic goals of NPL and forms the backbone of the high temperature degradation group’s road map.

The Need
There is a growing requirement to evaluate the performance of current and future structural materials and coatings in increasingly aggressive and novel atmospheres. These requirements not only relate to the oxidation and corrosion performance but also to the mechanical and wear performance of components. Improved test methods and facilities are therefore required to enable the measurement and characterisation of the mechanical, erosion and corrosion performance to enable UK industry to meet the low carbon economy challenge in as cost effective manner as possible.

Current State of the Art (NMI and elsewhere)
The current High Temperature team has a 10 year track record of developing metrology for characterising the performance of materials in aggressive conditions, and will build on their experiences with steam oxidation at ambient and high pressures, boiler corrosion in a range of gaseous conditions and mechanical testing to develop new metrology for these advanced operating conditions. Such developments will place NPL as the world leading NMI in this form of materials metrology and will strengthen our national and international reputation in this area. This work will also enable the team to maintain and foster new links with industry, such as Doosan Babcock, EON and RWE, and would provide opportunities to use large scale rigs and pilot plant, which would otherwise be beyond our own capability. With regards to the modelling aspects of this work, the metrology developed and used will help to further extend oxidation and spallation models such that they will account for geometry and heat flux affects. At present only Oak Ridge National Laboratory in the USA have such a model developed to the same extent as the NPL model. Further development of the NPL model will therefore maintain our position in this area. At present the UK possesses a high temperature solid particle erosion rig that operates up to 650 °C with speeds of 50 to 400 ms⁻¹. This project aims to look at the feasibility of moving beyond these conditions to more advanced operating conditions and will build on the solid particle erosion and wear expertise of Mark Gee.

The Solution
To address this requirement a suite of facilities consisting of new, modified and existing equipment will be used to develop test methodologies to characterise material performance in aggressive environments representative of those found in fossil power generation and aerospace. Existing mechanical test frames will be modified to allow for tests to be performed in controlled atmospheres under static and dynamic loading regimes. In addition static furnaces will be adapted to allow for testing under atmospheres representative of advanced cycles. In-situ methods for monitoring corrosion and erosion rates will be reviewed and the most promising methods will be developed and extended to high temperature operation.

Metrology Capability to be Delivered
- Measurement of the long-term mechanical properties at elevated temperatures, under static and dynamic loading in controlled atmospheric conditions (i.e. inert, combustion atmosphere, CO₂ rich etc).
- Measurements and characterisation of the erosion performance of monolithic and coated materials at temperatures in air and steam environments.
- Capability to expose and measure material performance under complex non-isothermal conditions and under novel gaseous atmospheres.

Exploitation/Spin Offs
Development of these facilities will provide added capability to the group extending the range of third party services available. This would also open up income streams from overseas where novel cycle technology is more advanced, enabling NPL to bid for more EU funds and strengthen its position with current European partners.

Project Partners
Details available on request.
Co-funding
Co-funding will be obtained for the completion of three existing projects: Surf700 high temperature turbine seals, IMMP3 materials modelling and AMBER modelling fireside corrosion, and three potential projects: MACPLUS – (£480k), AUSPLUS (£160k) and EFFOX (£100k).

Synergy with Other Projects
The work proposed in this project builds on recent activities of the group and extends our capability into emerging generating options for low carbon economies. It will generate better understanding of the effect of heat flux and strengthens the miniaturised testing theme. This project obviously links well with the co-funded projects and will also feed into UK/US and future COST activities.

Summary of Technical Work
- Deliverables 1 2 and 3 build on the expertise established at NPL.
- New methodologies for the measurement of fretting wear, and of brush seal creep, will be developed using equipment obtained through previous projects.
- A model for the degradation of fireside boiler materials will be developed using neural networks using data supplied by industry.
- Multi-variable data sets will be collated and stored in a newly compiled database.
- A thermodynamic database will be developed for IN625, IN617 and Alloy 263 Ni-Al-Cr-Fe-Mo-Nb-Ti to be used by partners to develop phase evolution/nucleation models.
- Uniaxial and multiaxial mechanical tests will support partners in development of failure mechanism models.
- Deliverable 4 will focus on the design and development and modification of existing and new equipment associated with operating tests in controlled and aggressive atmospheres. Commercial options for environmental testing are available, but NPL will need to judge whether these fully meet the requirements of the test programme.
  1. Assessment of mechanical properties and stress/corrosion performance in controlled atmospheres.
  2. Improved understanding of degradation processes in novel atmospheres and under heat flux conditions.
- Tests will be performed under a range of conditions, primarily on austenitic materials. Tests will include long-term mechanical properties in inert and two other gas mixtures; these will be performed under static loading and dynamic loading to examine the interaction of different damage mechanisms in these environments. During these tests load, strain and temperature values will be logged giving a fully instrumented test method.
- The feasibility of providing a high temperature, high velocity particle erosion apparatus will be conducted.

Risk
- The larger Co-funding projects are not funded. The proposals are submitted and the consortia are well established and the proposals have had positive feedback.
- EU funded projects extend beyond 2013: NMO funding not forthcoming. WG agreement on future projects to be sought early in current project.
- Aggressive environments prevent accurate measurement of material properties. A number of methods are available, which will provide some success, but any limitations may reduce the understanding of the degradation processes being developed.
- The number of variables required to model any processes becomes too high, making their formulation too complex for the systems planned. Data visualisation will help to identify key parameters before extensive analysis is carried out.

Knowledge Transfer Plan
The outputs of this project will be disseminated to UK industry primarily via interactions with the High Temperature Degradation IAG that consists of nearly all the UK power generation and material/equipment suppliers. Dissemination will also be sought through interactions with MAT UK, the Energy Materials Working Group and the Advanced Power Generation Technical Forum – Materials. Wider scientific dissemination will occur via international interactions resulting from the UK/US collaborative project and through future COST activities as well as through publication of scientific papers and international conference presentations.

Deliverables

<table>
<thead>
<tr>
<th>Deliverable Title</th>
<th>Start: 01/10/2010</th>
<th>End: 31/08/2013</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co-funding, for Facility for conducting time dependant thermo-mechanical testing in controlled atmospheres. Demonstrated via long-term tests on austenitic materials in inert and combustion gas atmospheres.</td>
<td></td>
<td></td>
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<tr>
<td>Evidence: Scientific paper in Materials at High Temperature</td>
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</tbody>
</table>
### Project Description (including summary of technical work)

1. **Guide to the expression of uncertainty in measurement.** The project will lead the editing of a new edition of the GUM and drafting Supplement 2 on general measurement models (due for completion in 2011) and Supplement 3 on modelling. It will support the practical use of these documents by metrologists by delivering software and case studies, maintaining a best practice guide (BPG), related software specification document and a training course. The project will influence, and contribute to, the work of the major standardization and metrology bodies, including JCGM, ISO, BSI and UKAS.

2. **Key comparison data evaluation.** Contributions will be made to (the Working Groups of) the CCs of the CIPM (particularly CCQM, CPPR, CCRI, CCAUV and others as required) to produce guidelines for key comparison data evaluation. Technical work will be undertaken to investigate (a) the use of KC reference curves in the context of KCs where the property of the artefact depends on a parameter (such as wavelength or frequency), and (b) approaches to relating the results of KCs (in appendix B of the MRA) to the CMCs claimed by NMIs (in appendix C of the MRA). A workshop will be run, in association with another metrology conference, such as in the series on the impact of IT in metrology co-organized by BIPM.

3. **Development of capability.** Work on topic (1) will build on that undertaken during an incoming secondment to NPL on the use of quantile functions to summarise the results of Monte Carlo calculations, and will further investigate kernel density estimation methods and the use of entropy to compare different summaries. Work on topic (2) will build on our knowledge of the methods of maximum likelihood estimation and asymptotic least squares to extend existing regression algorithms (such as those described in the series on the impact of IT in metrology co-organized by BIPM).
in an ISO TS, a previous deliverable of the project) to account for data with uncertainties based on few degrees of freedom.

**Impact and Benefits**
The need to evaluate and express measurement uncertainty applies across all areas of measurement, as well as in the application of measurement to standardization, calibration and testing, laboratory accreditation and decision-making, and in support of trade and regulation. The project will support the competitiveness of the UK NMS and UK industry in the global market by helping to ensure reported uncertainties are reliable and fit for purpose. The project will also ensure that the UK influences appropriately the work of the major standardization and metrology bodies, including BSI, ISO, JCGM and CIPM. Metrics to be reported for the project will include number of downloads of software, BPG, etc. from the S$\$/M web site, number of attendees on training courses, value of co-funding from other NMS programmes, and number of KCs and CCs supported.

**Support for Programme Challenge, Roadmaps, Government Strategies**
The project addresses the programme challenge “Credible and reliable values for measurement uncertainty” and through this provides underpinning metrology to support challenges addressed in NMS programmes and projects such as in the areas of dimensional metrology, acoustics, analytical science and optics. The project supports directly the S$\$/M programme roadmap on *Evaluation of measurement uncertainty*, as well as those on *Continuous modelling and Experimental design, data analysis and decision-making*.

**Synergies with other projects / programmes**
Other S$\$/M projects are underpinned by this project. Moreover, the project provides an avenue for codifying the technical outputs of other S$\$/M projects as documentary Standards and guidelines. The project underpins the support given by the S$\$/M team to other NMS programmes and many other projects, where those programmes and projects are concerned with modelling, uncertainty evaluation, calibration, traceability, experimental data analysis, KC data evaluation, etc.

**Risks**
- Dependent on continued membership of relevant committees: ensure visibility of the project team to these committees;
- Lack of control of the timetable for committee documents: maintain a leading role enables NPL to influence the timetable;
- Stakeholder acceptance of the proposed deliverables may take longer and more effort to achieve than expected: ensure continuity with accepted approaches and undertake continual consultation.

**Knowledge Transfer and Exploitation**
To include journal articles, and conference presentations and papers, new editions of NPL’s BPG on uncertainty evaluation and its related software specification document, software to support the application of the GUM and its related documents, and a training course run annually at NPL and off-site on demand. We will aim to collaborate with others, e.g., UKAS, with regard to training on measurement uncertainty evaluation. We will also continue to support particular conferences, including those in the AMCTM series organized by IMEKO TC21 and on the impact of IT in metrology co-organized by BIPM.

**Co-funding and Collaborators**
Details available on request.

**Deliverables**

<table>
<thead>
<tr>
<th>Deliverable title: Guide to the expression of uncertainty in measurement</th>
<th>Start: 01/04/10</th>
<th>End: On-going</th>
<th>Cost:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Contribution to JCGM/WG1, ISO, IMEKO, BSI, etc [on-going];</td>
<td>1.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Release and maintain software to support the application of the GUM and related documents [on-going];</td>
<td>2.</td>
<td></td>
<td></td>
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<tr>
<td>Case studies with other NMS programmes [one per year];</td>
<td>3.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maintain BPG on uncertainty evaluation, related software specification document and training course [on-going];</td>
<td>4.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Knowledge transfer in the form of journal articles, conference papers and presentations [on-going].</td>
<td>5.</td>
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</table>

<table>
<thead>
<tr>
<th>Deliverable title: Key comparison data evaluation</th>
<th>Start: 01/04/10</th>
<th>End: On-going</th>
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<tbody>
<tr>
<td>Contributions to the Consultative Committees of the CIPM [on-going];</td>
<td>1.</td>
<td></td>
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</tr>
<tr>
<td>Lead BIPM Director’s Advisory Group on Uncertainty (with particular responsibility for KC data evaluation) [on-going];</td>
<td>2.</td>
<td></td>
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<tr>
<td>Approaches to KC data evaluation where the property of the artefact depends on a parameter [01/04/10–31/12/11];</td>
<td>3.</td>
<td></td>
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</tr>
<tr>
<td>Update the 2002 paper key comparison data evaluation to bring in line with current practice [01/07/11 - 30/06/13].</td>
<td>4.</td>
<td></td>
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<tr>
<td>Workshop on KC data evaluation [01/07/12–30/06/13];</td>
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<tr>
<td>Develop good practice guide on KC data evaluation supported by a training course [new work item: 01/07/13–30/06/14].</td>
<td>5.</td>
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</table>

<table>
<thead>
<tr>
<th>Deliverable title: Development of capability (already contracted to 31/3/13)</th>
<th>Start: 01/07/11</th>
<th>End: 30/06/15</th>
<th>Cost:</th>
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<tbody>
<tr>
<td>Approaches to using Monte Carlo methods within measurement and calibration services [01/07/11–30/06/13];</td>
<td>1.</td>
<td></td>
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<tr>
<td>Determination of calibration functions from scarce data [new work item: 01/07/13–30/06/15].</td>
<td>2.</td>
<td></td>
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</tbody>
</table>
Summary
The project adopts a model-based approach to evaluating uncertainties in complex dynamic modern computer-based measurement systems, in line with the GUM and the philosophy of the SSfM programme, to ensure that all contributions to uncertainty that arise from definitions and models of the measurand, signal acquisition, conditioning and processing are identified and quantified in a rigorous, consistent manner. The project aims to provide tools that will ensure that best practice can be adopted by metrologists in an easily implementable manner to allow them to apply their measurement results in practice and develop new calibration methods with the confidence that the uncertainties arising from their chosen measurement techniques have been accurately quantified. We concentrate on uncertainty analysis methods and tools for complex dynamic modern computer-based measurement systems for mechanical metrology applications (e.g., force, torque, pressure), including the study and modelling of noise in real systems, and the use of system identification techniques to recover the impulse response of systems from measured data. The aim is to introduce traceability to an area of metrology where it does not currently exist.

The Need
For many dynamic measurements, including mechanical quantities such as force, torque or pressure, the provision of traceability is still based on static calibrations. This can lead to inaccurate measurement results and lack of understanding of the behaviour of sensors and processes, and therefore undermines confidence of end users, prevents compliance with EU directives and international standards, and limits the usefulness of current solutions for safety-critical applications in aerospace and automotive industries. It is necessary for experimentalists within NMIs to develop new primary standard methods and for NMI mathematicians to establish new tools for mathematical modelling, signal processing, deconvolution and uncertainty analysis to support traceable dynamic calibration. The project meets the requirements identified in the EMRP Outline document of 2008 for innovative set-ups for new industrial and societal needs in the area of mass and related mechanical quantities. The EMRP Outline recognised that mechanical quantities, including mass, force, torque, and pressure, are a key tool for industry, research and society because of their wide range of applications in aerospace, off-shore industries, robotics, micro and nanotechnologies and manufacturing, and to support diagnosis in medicine. Such requirements can only be met by NMIs, as industry is unable to provide the necessary primary standard methods.

The Solution
This project combines accurate and robust modelling and reliable evaluation of the uncertainties associated with the outputs of dynamic measurement processes. This requires:
- the development and identification of models of the complete dynamic measurement chain and its constituent parts;
- a consistent treatment of uncertainty evaluation and propagation both in NMI-based primary calibration procedures and in the secondary methods used for industrial applications;
- the reconstruction of the sensor input by the deconvolution of sensor and system effects from the sensor output to enable reliable evaluation of the dynamic quantity of interest and the uncertainty associated with that evaluation.

The project will develop mathematical and statistical methods to support scientists at NMIs who are establishing new methods of realising the dynamic quantities of interest and to provide industry with the tools and knowledge needed to take advantage of these newly available dynamic calibration methodologies.

Project Description (including summary of technical work)
The project provides mathematical modelling of the complete measurement chain and statistical procedures for the evaluation of the uncertainty contribution of each individual component. It will support experimentalists working in pressure, force and torque in the identification and definition of suitable calibration signals and the required signal bandwidths. Methods will be established and assessed to identify parameters of the dynamic models from dynamic calibration data provided by NMI experimentalists. To ensure traceability, consistent and reliable uncertainty evaluation is required. Since the current guidelines for uncertainty evaluation in metrology (GUM) do not address dynamic measurements directly, a crucial aspect of the work will be the development of uncertainty evaluation schemes in dynamic measurements that are consistent with these guidelines. The result will be an assessed dynamic model for the specific measurement accompanied by a reliable uncertainty budget. For industry-level measurements, deconvolution techniques will be provided and software published to assist in the estimation of the dynamic measurand, taking into account all calibration information provided to the industrial user by the calibration laboratory. The project will be carried out collaboratively with PTB (Germany), LNE (France) and INRIM (Italy).

Impact and Benefits
Dynamic measurements of force, pressure and torque are highly important for the automotive industry on which about 12
million jobs depend throughout Europe. As automobile use contributes greatly to CO₂ emissions, future improvements made possible will also help reduce carbon emissions. Dynamic measurements and generation of precise dynamic mechanical test signals are a prerequisite for reliable testing and hence it will be possible to improve the safety of many systems. For instance, aircraft safety requires accurate calibration of ADTS (Air Data Test Set). ADTS calibration is currently performed only by static pressure, and reliable calibration of ADTS in dynamic conditions would help improve aviation safety. In addition, areas of (metrological) research such as material testing will benefit substantially from the possibility of traceable dynamic measurements of force, pressure and torque. It will be easier for European manufacturers of sensors and instrumentation for dynamic mechanical applications to demonstrate product compliance with global standards and customer requirements following completion of this project. Research in the direction of this project is currently under way worldwide, and this project will help to strengthen the UK and European position. It will help ensure that Europe does not lose its leading position in this field of research. A consequence of failure would be that UK and European industry would be forced to seek dynamic calibration certificates outside the EU area.

Support for Programme Challenge, Roadmaps, Government Strategies
- Supports SSfM roadmap “Experimental Design, Data Analysis and Decision Making” by ensuring that the benefit of measurement processes are maximised by getting the best information from data.
- Supports SSfM roadmap “Evaluation of Measurement Uncertainty”, especially the driver “Consistency in national and international traceability”.
- Relevant to NMS and UK government strategies in energy, sustainability, global competitiveness and measurement infrastructure.
- Supports directly the objectives of the European Metrology Research Programme 2008 for “innovative set-ups for new industrial and societal needs in the area of mass and related mechanical quantities”.

Synergies with other projects / programmes
- Supports directly work in NPL’s Engineering Measurement team on Innovation R&D and Engineering and Flow NMS projects in dynamic mechanical measurements, especially dynamic pressure.
- Links directly to SSfM core project “Evaluation of measurement uncertainty” by extending GUM methods to dynamic measurement problems. The core project also provides a means of disseminating project outputs.
- Supports NPL contracts with European Space Agency for the provision of micro-Newton force measurements for satellite applications and for improvement of environmental control systems in research laboratories.
- Provides basis for future EMRP bids in relation to industry and advanced manufacturing.
- Experimentalists developing new methods of establishing traceability for dynamic mechanical quantities will need continuing support beyond the life of this project.

Risks
Project will require collaboration between four NMIs, each with different levels of experience in dynamic measurement. NPL will lead the mathematics and uncertainty analysis work required for the EMRP project. It will be necessary to manage the communication process between NMIs carefully and implement a flexible approach in which lack of progress in one technical area (especially by experimentalists) does not impinge heavily on progress.

Knowledge Transfer and Exploitation
- Main KT route will be via the impact work package of the associated EMRP project.
- Will also provide input to JCGM supplements to the Guide to expression of uncertainty in measurement (GUM)
- Project will produce downloadable software, reports, papers and conference presentations.
- Outputs will be taken up by experimentalists in NMIs and by industrial end users of new calibration facilities.

Co-funding and Collaborators
Project aligns directly with EMRP project 20i “Traceable Dynamic Measurement of Mechanical Quantities”, due to begin in July 2011 for which it provides matching funding (£158k). NMI collaborators are funded from the same source. Details available on request.

Deliverables
<table>
<thead>
<tr>
<th></th>
<th>Start:</th>
<th>End:</th>
<th>Cost:</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1st July 2011</td>
<td>30th June 2014</td>
<td></td>
</tr>
<tr>
<td>Mathematical and statistical modelling of dynamic measurements</td>
<td>mathematical definition of measurands and measurement chains to meet documented requirements; report on mathematical modelling of the measurement chain components and determination of (class of) models for considered transducers; report on statistical modelling of the considered dynamic measurements and evaluation of uncertainty; general methods and procedures for the mathematical and statistical modelling of dynamic measurements.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1st April 2012</td>
<td>30th June 2014</td>
<td></td>
</tr>
<tr>
<td>Implementation for NMI-level primary dynamic calibration</td>
<td>software for NMI-level calibration measurement analysis (pressure, torque and force); case studies for dynamic calibration of sensors and transducers (pressure, torque and force)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>1st April 2013</td>
<td>30th June 2014</td>
<td></td>
</tr>
<tr>
<td>Implementation for application-level measurement</td>
<td>software for application-level deconvolution, input prediction and uncertainty evaluation (pressure, torque and force)</td>
<td></td>
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</tbody>
</table>
**Summary**

This EMRP co-funded project will address fundamental aspects of metrology for tribology so that significant advances can be achieved in the long-term performance of surfaces in industrial applications. The issues that will be addressed are: the measurement of very small wear volumes, the measurement of low friction, the measurement of the temperature at wear interfaces, the measurement of changes in chemistry that occur at the contact interface between surfaces, and the development of methods for the assessment of the durability of tribological surfaces.

These are all challenging issues where novel measurement methods will provide industry with the ability to make step changes in the development of new materials and the design of new products. It draws on the expertise of a consortium of leading measurement institutes throughout Europe and is directly supported through collaboration with other research institutes and leading industrial firms.

**The Need**

Engineering of surfaces is recognized to be one of the ways to improve competitiveness of industry in sectors such as transport (air, sea and land), energy generation, manufacturing and mineral extraction.

Surface engineering solutions are now being developed based on complex composite coatings that have a multi-functional capability (i.e. low friction, high durability, and good performance in aggressive environments). Their adoption and implementation in industrial applications are being hampered by metrological issues concerning durability, and the assessment of their wear and friction performance that need to be addressed. Thus the measurement of small wear volumes is currently addressed by complex radioactive tracer measurement techniques that are only carried out in a few institutes worldwide, and only a few attempts have been made previously to address the very challenging measurement of temperature at tribological contact interfaces.

**The Solution**

This EMRP project will deliver new metrology to address major measurement issues in the assessment of the performance of engineered surfaces. The project consortium draws on the best measurement expertise from the NMIs of France, Germany, Finland, Italy and Denmark as well as the UK to address the metrological issues that have been identified. Thus the measurement of small amounts of wear and the low friction that is required in many applications for engineered surfaces will be addressed. The temperature that is reached through frictional heating with consequent chemical reaction at the interface between two surfaces is also critical to understanding the behaviour of engineered surfaces. Measurement methods will be developed for both of these issues. Finally, methods for the assessment of the loss of surface integrity with time will be assessed.

**Project Description (including summary of technical work)**

The measurement of small wear volumes will be addressed by traceable SEM stereology and optical measurements validated by fine probe profilometric measurements such as AFM. To improve the measurement of friction, particularly for modern low friction coatings where long term performance is critical, new measurement technology will be developed for intrinsically stable and self-calibrating friction measurement devices leading to a traceable measurement chain.

In-situ measurements in the wear contact will be a major focus of the project. Interface temperature will be measured by embedded fibre sensors, thin layer thermocouples, and thermal imaging with the use of infrared transparent counterfaces. In-situ measurement of wear will be addressed by the use of fast optical probes based on reflectance fibre optic and chromatic aberration technology. The feasibility of measuring changes to the chemistry of interface using In-situ Raman spectroscopy will be assessed.

**Impact and Benefits**

This project will provide UK industry with some of the tools that will enable them to compete worldwide with innovative products that have better performance than the competition, and with major reductions of impact on the environment. The direct beneficiaries from this project are throughout the production industry, and the minerals extraction, materials handling, and surface engineering sectors.

Improved metrology for tribological surfaces will enable the introduction of surface engineering technology that will significantly increase the efficiency of energy generation through better surface engineering to withstand the necessary higher operating...
temperatures, and therefore give reductions in the use of natural resources such as fossil fuel reserves. The efficiency of energy use will also be increased through reduced friction in manufacturing processes such as forging. Increased tool lifetimes will also enable a reduced environmental impact through savings in the manufacture of tools with consequent materials usage. Major fuel savings will also come from the higher operating temperatures that will be made possible for aero engines and reduced friction in internal combustion engines. Improved surface engineering will reduce health risks by enabling the introduction of better protective coatings that replace coatings that will be banned under safety regulation.

Support for Programme Challenge, Roadmaps, Government Strategies
This project fits with the NMO and NPL strategic aims for measurements and modelling to characterise atoms, molecules, bio-systems and materials, and science to support sustainable technologies in the areas of energy generation and usage, and high value manufacture, including life assessment. It also aligns with the TSB strategies for advanced materials and high value manufacturing.

Synergies with other projects / programmes
This project will build on the results of projects AM09 on the Development of Test Methods for Low Friction Coatings and T11 on Monitoring the Degradation of Surfaces Subjected to Wear. For the in-situ measurement of temperature, the project will also draw on the expertise of NPL in the thermal measurement area.

Risks
This is a challenging project with significant technical risks in most areas. The top two technical risks are to achieve successful in-situ measurement of temperature in the wear interface and small wear volume. The likelihood of success is considerably enhanced by the world leading expertise on temperature measurement and dimensional measurement in the project consortium. There is also a management risk with the large EMRP project consortium, but this risk is minimised by the experience of NPL as project coordinator and other project partners in project management.

Knowledge Transfer and Exploitation
An industrial focus group will be formed at the start of the project from collaborating firms that will act to steer the project and will also receive results of the project firsthand. The project will be targeted at the entire surface engineering supply chain including coatings suppliers, instrument manufacturers, research institutes and end users. Technical reports and papers will be written and targeted at those publications that are known to be read by industry concerned with the durability of surfaces with presentations made at appropriate conferences.

Good practice guides will be published reporting the new metrology developed during the project. A new Technical Work Area in VAMAS will be formed to carry out pre-standardisation work on the new measurement methods developed in the project. It is expected that these results will then be taken forward into standards through ASTM, CEN and ISO. Training workshops will be held on best practice in the assessment of the tribological performance and durability of engineered surfaces.

Co-funding and Collaborators
This project has agreed EMRP co-funding of £289k. The core EMRP project partners are PTB, BAM, LNE, INRIM, DTI and MIKES. Details available on request.

Deliverables
| Co-funding for EMRP project ‘Metrology to Assess the Durability and Function of Engineered Surfaces’ | Start: October 2011 | End: September 2014 | Cost: £ |
The project will develop and validate new measurement methods capable of high-resolution dimensional, mechanical property and/or deformational behaviour mapping based upon tactile probe (mechanical contact) methods. It will target physical properties of materials that can be equally accessed and measured by independent methods and in units traceable to the SI. The project will achieve a step improvement in the characterisation and control-by-design of the dimensional drift in metrological platforms and measurement systems. New and improved analysis methods will be developed and validated to describe contacts with viscous materials, enabling measurement of the mechanical properties of visco-elastic materials traceable to the SI. Industrial case studies shall include demonstration of the ability to distinguish mechanical property differences between products manufactured from recycled or virgin feedstock. The new validated measurement methods will allow the development of a specification for and identification of a list of candidate certified reference materials, for which there is a strong demand from the polymer and life science industries. This is an EMRP co-funded project.

The Need
The current state of the art is contained in ISO14577 “Metallic materials - Instrumented indentation test for hardness and material parameters” which is limited to the measurement of materials that deform only elasto-plastically. This “quasi-static” method succeeds by relying upon the indentation contact being stable. This approach is wholly inadequate for materials that are viscous or creep because the contact is by definition not stable (its size continues to increase with time, even at constant force). New measurement methods capable of independent validation are required because Industry is increasingly being driven to use viscous materials and requires high precision engineering parts with predictable and stable dimensions and QC ‘tools’ that are very stable in dimension and/or alignment. Material properties and construction design rules are needed to enable improved stability and traceability of QC methods. New sustainable products made from recycled materials require measurement methods to demonstrate equivalence of property and performance where lifetime and performance are limited by long-term deformation behaviour, e.g. car timing-belts. The measurement issues identified span many industrial sectors and require a coordinated government response and international standardisation to remedy current market failure through provision of internationally validated acceptance tests.

The Solution
This project will:
- Develop and validate a capability for measurement of structure/instrument dimension/alignment drift over industrially relevant periods using non-contact methods.
- Derive design rules for achieving excellent intrinsic structure or instrument stability and develop specifications to achieve metrology platforms mostly insensitive to environmental fluctuations.
- Provide underpinning metrology to enable measurement and prediction of creep rates in contacts and stressed materials
- Develop standard test protocols to demonstrate equivalence of complex modulus values obtained by different methods
- Demonstrate the new capability by conducting industrial case studies to solve industrial problems
- Develop a specification for and identification of a list of candidate certified reference materials.

Project Description (including summary of technical work)
Absolute drift and creep measurement traceability will be derived from the NPL X-ray Interferometer (XRI) through transfer interferometers (or closed loop stages) to contact based methods, which are an ideal candidate for the required high-resolution measurement solution. The key challenge is to develop methods of obtaining accurate time dependent properties of materials that are reproducible not only by indentation instruments but also by independent test methods, requiring new measurement and analysis protocols. The work will build on previous success by the project partners, and methods of tensile creep testing, indentation creep testing and dynamic (oscillating) instrumented indentation testing (IIT) will be used to develop models to obtain the viscosity and time constants of polymers. This will enable both improved measurement protocols and correction routines for contact-based dimensional measurement; the validated methods to obtain the mechanical properties of visco-elastic materials will provide values traceable to the SI. A specification for candidate certified reference materials, for rapid validation of testing instruments, will also be developed.

Impact and Benefits
The project outputs are directly applicable to many sectors, inter alia packaging, composites, manufacturing of polymer
components, degradation of polymers, (bridges, buildings, paints, white goods, double glazing, wiring etc), transport (Automotive, aerospace). Improved design and accelerated innovation will be enabled in the nano-composite and micro-moulding sectors. The project will also generate new metrology capability:

- Absolute drift and creep measurement traceable to X-ray interferometry.
- New/improved measurement methods for dimension, shape and mechanical properties of viscous materials.

The new capability will be demonstrated by case studies including: correction of dimension and shape measurement of aspheric lenses for compact cameras and sensor optics; measurements to reduce the long-term creep of automobile timing belts to improve the fuel economy of up to 60% of cars in the EU within 5y; measurements to determine the performance difference between use of recycled and virgin polymeric feedstock for drainage products for the construction industry.

International adoption and impact will be generated by supporting EN ISO standardisation and VAMAS pre-standardisation activities in cooperation with the relevant CIPM consultative committees.

It is anticipated that the project will enable increased polymer use for vehicle lightweighting— saving fuel for all vehicle users. If 20 million UK cars have a life of 10 years and average 15000 km per year per car; if the 2 million cars replaced each year are just 10 kg lighter as a result of switching to polymer use, this will result in a saving rising to 60 million litres of fuel per year (Yr 1 = 6M ltr, Y2= 12M ltr .. after 10 yrs 60M ltr per yr) in the UK alone. Engineering a timing belt to last the lifetime of a car will save car owners over £300 each in saved service costs. Redirection of polymer use from packaging to new applications in composites as used in wind energy technology, marine engineering, pipes, tanks, transport and construction will generate jobs as Europe captures 20% of the projected 5.8 billion USD market in polyester resin (Market research: “Global Unsaturated Polyester Resin Market 2010-2015”, Lucintel, Texas USA).

**Support for Programme Challenge, Roadmaps, Government Strategies**

The need for “stable materials / structures & design principles” was expressed in the iMERA roadmap 2006 on “dimensional metrology for micro-nano technology”.

This project will help industry to conform to the ‘Packaging and Packaging Waste Directive’ (94/62/EC); also Regulation (EC) No.443/2009 in which a sliding scale of adherence to the 120 g CO2/km objective is set out with financial penalties for failure; Together with the associated vehicle emission limits Euro 1 to Euro 6. Contributes to grand challenges on sustainable manufacture and energy generation and use.

**Synergies with other projects / programmes**

This project builds on work done in the Characterisation project SE02 “Dynamic Nano-Indentation Methods”. It links with the Dimensional programme (Nanotrace project). It builds a strategic relationship with Norbert Schwarzer for development of capability for application of contact based measurement in multiple dimensions, which will enhance the Materials project “MAXIMISE.” The improved measurement methods will feed into present and future polymer and nano composite projects.

**Risks**

The NPL XRI could fail or be moved to a new laboratory, which would delay the project and require switching NPL effort to PTB. The complexity of material model or analysis necessary may be too much for UK industry to understand easily and a ‘black box’ solution may be hard to accept. High noise levels or uncorrected drift rates may make long-term drift immeasurable in some instruments.

**Knowledge Transfer and Exploitation**

Publications (reports and scientific papers): [at least 3] in high-impact peer-reviewed scientific journals such as. Metrologia, Meas. Sci. Technol., APL, Appl. Phys. B, Prec. Eng. A. Presentations at leading international conferences Three workshops will also be arranged and be attended by the project partners, members of the user committee and representatives from a wide range of industry. Standard protocols will be submitted to ISO and CEN working groups addressing existing mandates in ISO/TC164/SC3 and ISO/TC61/SC2 to develop indentation-based methods for high spatial resolution polymer property measurement. A website will be created for the transfer of project results in the form of design criteria to industry and academia NMIs. Training in the use of new facilities, will be made available to Industry though an open public event

The projected impact for polymer product manufacturers will result, for example, in better QC, reduced scrap, and virtual prototyping with valid data. Also, SIO will provide software analysis tools ready for exploitation. These will be integrated into the current product range of SIO.

**Co-funding and Collaborators** EMRP 460k euro. Details available on request.

**Deliverables**

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<tr>
<th>Start: October 2011</th>
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**Co-funding for EMRP project MEPROVISC**
Summary
The overall objective of the project is the optimisation of thermal stability as well as the stability over time of ultra-precision engineering measurement and production tools. The project focuses on high precision applications, where temperature measurements and control in the millikelvin range are necessary. NPL’s contribution to this EMRP project is in the determination of the hardness and creep of materials as a function of temperature. NPL will also manage the generation of project impact.

The Need
Current trends in precision engineering are towards ever-higher accuracies for industrial high-end production and measurement equipment; especially from ICT or aerospace industries. Temperature effects and time-dependent drift are serious limitations on achievable system performance. A solution requires improved, temperature-insensitive designs of machine components and active compensation of thermal gradients caused by heat sources or drains.

The wide range and diversity of industry sectors, combined with the need for international standardisation requires a coordinated government approach. Furthermore, the same measurement needs directly impact National Measurement Institute’s ability to disseminate traceability of the SI length scale and for maintenance of length comparators at the required level of uncertainty.

The Solution
The project will develop knowledge and control of medium to long-term dimensional drift properties by studying aging, thermal expansion, hardness and creep of materials, joints and sensors; compiling a metrological knowledge-base for material properties, the measurement thereof and related metrological standardization.

Methods will be developed for reliable and low maintenance active control of temperature gradients. The use of thermocouples is a promising choice for the measurement of temperature gradients and their control using new control algorithms running on an embedded computer. Model-based control algorithms will be developed together with an easy method to define the control parameters. Cooling elements with low vibration and low thermal backlash will also be developed.

Thermal modelling of a prototype measurement machine will be carried out to develop a better design of the machine. Guidelines for practical application will be derived for industrial users. Additional improvements through active thermal control of unavoidable heat sources will also be demonstrated.

Key metrological capability developments include:
- Interferometric measurement equipment for the determination of dimensional drift with a measurement uncertainty down to 10 pm.
- Improved temperature measurement and control electronics in regard of sensor compatibility, control parameter determination and ease of use.
- Measurement uncertainty analysis and design rules for an improved indentation machine for the measurement of hardness and creep and their dependency on temperature.

Project Description (including summary of technical work)
NPL will assist in the characterisation of the mechanical and creep properties of materials and the drift of instrumented indentation instruments. Methods will be developed to evaluate instrument drift at higher than ambient temperature and instrument compliance as a function of temperature. Investigation of material creep rate as a function of temperature will examine the effectiveness of using time-temperature superposition for the acceleration of creep rate measurements in highly stable materials. The approaches developed will be validated by comparison with independent methods such as uniaxial tensile testing. The creep performance of different joining techniques will be investigated, including the effect of washers on bolted joints and a comparison of adhesive and wrung joints. The different stress states induced by different joining techniques will also be considered and assessed. Available models will be used, and developed, to predict the long-term behaviour of the materials tested.

Impact and Benefits
Precision tools and metrology systems form the basis of high-end industrial equipment in various industrial fields such as production technologies, process engineering, communication technologies, aerospace, automotive industry, semiconductors, opto-technology, and mechanical engineering in general. The semiconductor industry contributes €29bn directly to the EU economy and provides around 215,000 direct jobs, which rises to half a million jobs when indirect jobs are included. Metrological knowledge for the design of industrial equipment and standards for reference materials will improve control of production processes and will result in improved, more reliable, economical and safer end products and applications.

The project emphasis on dissemination will result in an improved networking of experts regarding temperature effects from different industrial and scientific areas.

Optical technologies are the key to green photonics, which is forecasted to play a major role in emerging megatrends such as energy, health and environmental surveillance. Precision alignment will improve optical efficiency and lifetime for LEDs. Thermal effects and drift are the main factors limiting the performance of measurement and production tools; helping industry to develop and use precision machine tools and lithography tools will boost competitiveness on the world market. Reducing the sensitivity of precision tools to temperature will save the energy otherwise required for air-conditioning to tight tolerances.

**Support for Programme Challenge, Roadmaps, Government Strategies**


The need for “stable materials / structures & design principles” was expressed in the iMERA length roadmaps 2006. Needs regarding dimensional stability and thermal stability are formulated by the CIPM WG on material property measurements as well as in the EuMaT Roadmap of the European Technology Platform for Advanced Engineering Materials and Technologies.

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<tr>
<th>Synergies with other projects / programmes</th>
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<tr>
<td>This project builds on the limited previous experience of measuring indentation properties of polymers as a function of temperature in project H05. This project will start the development of the ability to measure mechanical properties such as elastic modulus, hardness and creep using instrumented indentation at high temperatures. This requires a significant amount of new metrology in the calibration of indentation instruments as a function of temperature and in control of the environment to minimise drift due to thermal expansion. The future aim is to increase temperatures to over 1000°C and test in vacuum to enable materials testing at the temperatures relevant to industrial applications such as cutting, machining, gas turbines and combustion chambers.</td>
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</table>

**Risks (NPL segment)**

Possible oxidation of sample and indenter changing shape and properties of a contact. Testing in air will be limited to 300-400°C. Alternative indenter materials will be investigated (such as SiC).

Highly variable instrument compliance may generate measurement errors. Characterise compliance as a function of temperature.

**Knowledge Transfer and Exploitation**


Presentations at leading international conferences. Three workshops will also be arranged and be attended by the project partners, members of the user committee and representatives from a wide range of industry.

Standard protocols will be submitted to ISO and CEN working groups as NWI supporting documents. This will address existing mandates in ISO/TC164/SC3 and ISO/TC61/SC2 to develop indentation-based methods for high spatial resolution polymer property measurement.

A website will be created for the transfer of project results in the form of design criteria to industry, academia and standardisation laboratories.

Training in the use of new facilities, measurement methods and primary standards developed by the project for industry will be organised in the form of a public event, for which attendance will be open to companies not involved in the consortium.

**Co-funding and Collaborators** (Total project value = £2.26M).

NPL receives direct cash co-funding from EMRP of £203k. Details available on request.

**Deliverables**

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<tr>
<td>1</td>
<td>Co-funding for EMRP project 'Thermal design and time-dependent dimensional drift behaviour of sensors, materials and structures'</td>
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### Project Description (including summary of technical work)

- Develop finite element (FE) numerical scheme to solve the equations
- Check application of the FE analysis in the limit of 1D diffusion using established data for permeation in a martensitic stainless steel
- Demonstrate potential of the model in combination with partners

### Impact and Benefits

The potential consequences of a component failure due to hydrogen include destruction of major infrastructure, contamination of the environment, and potential loss of lives. Moreover, the continual monitoring, repair and replacement of hydrogen embrittlement-susceptible components, such as in off-shore wind turbines, is costly. The practical significance will be demonstrated within the overall EU project, of which the NPL work is a modest contribution, by 3 case studies with industrial partners: viz. coated components in the Ariane 5 system; advanced high strength steels in the automotive sector; wind-turbine bearings. The modeling tool that NPL develops will be among the most advanced of its kind and will lead to a scientific paper as well as maintaining NPL capability in hydrogen diffusion modeling.

### Support for Programme Challenge, Roadmaps, Government Strategies

- Lightweight transport; advances in renewable and alternate energy.

### Synergies with other projects / programmes

Historically, NPL has been among the leaders in research on modelling and measurement of hydrogen diffusion and trapping. The extended capability emerging from this collaborative project will sustain that position and will provide opportunities for extending the model to other applications, particularly in the oil and gas industry. Also, a Stage 2 EU project is envisaged as a follow-up in which the aim is to deliver an engineer-oriented software tool.

### Risks

We are not significantly dependent on other partners for success so, whilst a difficult technical project in terms of the challenges in 3D modelling, there should be no major extraneous impediments.
Knowledge Transfer and Exploitation

Knowledge transfer of the overall EU project will occur through diverse routes involving scientific publications, university courses and industry training programmes culminating in a modelling workshop targeted at a wide scientific and industrial community. NPL will participate in respect to the scientific publications and the modelling workshop.

Exploitation of modelling is a complex issue and it is perceived that engagement with software firms will take place though that is considered a development for a future Stage 2 project aimed at the engineer-oriented software tool.

Co-funding and Collaborators

EU FP project supported at a funding ratio of about 50:50. Project has already been awarded. Partners in 6 different countries led by the Fraunhofer Institute in Germany.

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<td>Co-funding for FP7 project MultiHy</td>
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The threat of climate change is the overwhelming issue in terms of driving forward clean energy technology. World Energy Outlook (WEO) 2008 edition claimed that ‘current global trends in energy supply and consumption are patently unsustainable – environmentally, economically and socially. It is not an exaggeration to claim the future of human prosperity depends on how successfully we tackle the two central energy challenges facing us today: securing the supply of reliable and affordable energy; and affecting a rapid transformation to a low-carbon, efficient and environmentally benign system of energy production’. To mitigate against the environmental impact of fossil energy CCS technologies are being implemented. However, CCS technologies are expected to have a high impact on the net efficiency of modern pulverised coal fired power plants, with an evident efficiency penalty. To enable the use of CCS technologies the overall efficiency of plant needs increasing through advanced material solutions. NPL is part of a large consortium of around 25 partners undertaking this work as part of a five year FP7 project, as such it is well positioned with other experts in the field to address the aims of this project and to maintain and raise the profile of NPL in this forum.

The Solution

Advanced material solutions are needed to offset the efficiency cost of CCS, this project aims to increase the net efficiency of coal fired plants by increasing the performance and reliability of some critical components identified as follow:
- refractory materials of the combustion chamber (especially for oxy-combustion application),
- headers and pipeworks (avoidance of weld Type IV cracking phenomena, working temperature increase),
- super heaters (optimised creep performance in high temperature oxidation/hot corrosion environments),
- coated pipes and boiler components able to withstand co-combustion conditions (high temperature oxidation/hot corrosion, erosion-adhesion and wear).

To address the aims of the project the following activities will be undertaken

- Characterisation of refractory material exposed to oxyfuel conditions (T~1740°C) before and after innovative laser melting surface treatment. This will mostly be performed using microscopy techniques applied to exposed and virgin material before and after surface treatment. Exposures will be performed by project partners in plant.

- Characterisation of long term mechanical properties of novel (MARBN) steels and Ni-based superalloys and their welds designed for future USC applications. Screening trials will be conducted to evaluate the creep properties of these specimens to assist in alloy development. The interaction of stress and corrosion will be examined.

- Development of a test procedure to evaluate reheat cracking of thick section components and the subsequent evaluation of candidate materials. The creep crack growth and creep fatigue properties of candidate systems will also be benchmarked.

- Characterisation of steam and fireside coatings (microstructure, TMF, adhesion and chemistry) and the subsequent thermal cycling tests under simulated plant conditions. In addition, steam oxidation tests of steam side coatings at atmospheric and elevated pressure will be performed and the data used in a multi-layered model to predict the stress state and heat transfer in the multi-layered structure.

Impact and Benefits

The International Energy Agency (IEA) have projected that, by 2030, steam coal utilisation will be around double current levels, and that 45% of global power production will be from coal fired plant. The projected value of the global market for new plant is...
around 900 billion Euros, and within Europe 500 billion Euros. Historically European Companies have supplied around 50% of the power plants worldwide, and the great majority of the plants in Europe. It is anticipated that European Companies will be able to secure a significant share provided that they can offer improvements in plant performance. For generating companies, the commercial benefits lie in pre-empting costly plant failures and potentially enabling more efficient or reduced, and so less costly, in-service weld inspection programmes. A power plant steam leak may currently cost 0.05 - 1 million Euros in unplanned outage time, while it is estimated that ~3 million Euros per year is spent on weld inspections on current UK power plant, primarily for Type IV cracking. These costs are likely to be transferred to the new advanced super-critical plant unless this problem can be reduced. In terms of the environmental impact, it is projected that increases in plant efficiency, through higher operating temperatures and pressures, is capable of reducing emissions by around 20%, with biomass co-firing potentially contributing a further 10-20% reduction. These measures could therefore be capable of meeting emission reduction targets to 2020, but, for the more aggressive targets beyond that date, Carbon Capture and Storage (CCS) will be required. The European Union is seeking to make it mandatory that all new fossil-fuelled power plants, installed after 2020, should be equipped with CCS, if a cost-effective technology can be developed. The projected value of the global market for new coal-fired power plants to 2030, both to meet increased demand and to replace a proportion of existing plants that are due to be retired, is around $1,450 billion, and within Europe, it is of the order of $400 billion. For NPL there is a clear benefit of being involved in this project, not only will it demonstrate and develop new metrology capability but also it will also maintain our profile with current contacts and also expose NPL to new contacts through this partnership.

**Support for Programme Challenge, Roadmaps, Government Strategies**
This project fits with the NMO and NPL strategic aims for characterisation of materials. This project clearly links and supports current policy for reduced carbon emissions and increased use of renewable energy. It also aligns well with TSB strategies for energy materials as well as European strategies on energy supply and reducing CO₂ emissions.

**Synergies with other projects / programmes**
This project builds on the work conducted on the degradation of materials used in power generation, and links with current activities both for third party customers and for grant funded projects such as AUSPLUS (RFCS) and current TSB projects (ASPECT) and future proposals addressing fireside corrosion under biomass combustion. The coating aspects also are directly relevant to the EMRP Energy project and the Grains to Structures project. This work also links to the UK-US collaborative works programmes and areas being funded by the US DOE and EPRI.

**Risks**
This is a large project with the work proportioned to the partners. These obvious increases the risk of delays especially where task are of a collaborative nature. This will have to be managed by the project management team. There is also a risk that NPL will not have sufficient capacity in its test equipment, this will have to be managed by the facility team who will be involved very early on to plan the test programme.

**Knowledge Transfer and Exploitation**
Dissemination will be performed through scientific publications and international workshops and EU information days. In addition there will be a MacPLUS website which will be used for dissemination along with press releases. Information will also be exchanged with other EU and International projects as detailed in the MacPLUS proposal.

**Co-funding and Collaborators**
Co-funding is anticipated to be through the FP7 project MacPLUS, this will be of the order of €400-500k. NPL will be collaborating with 23 partners consisting of range of European manufacturers, operators and universities. Details available on request.

**Deliverables**

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<tr>
<td>Co-funding for FP7 project MacPLUS</td>
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Project No. | MAT\2011\7  | Price to NMO | £1209k  
--- | --- | --- | ---  
Project Title | Facilities Support  | Co-funding target | N/A  
Lead Scientist | Eric Bennett  | Stage Start Date | July 2011  
Scientist Team | Materials Division  | Stage End Date | June 2014  
|  |  | Est Final Stage End Date | Continuous  
Sector | Underpinning Metrology  | Activity | Provision of Standards & Maintenance of Capabilities  

**Summary**

This project maintains a wide range of facilities that are routinely used by other technical research projects in the Materials and other NMS programmes and to disseminate measurement services to a wide UK community who rely on the traceability provided by the Materials programme. The underpinning nature of this project is the basis for providing and validating much of NPL’s materials characterisation and properties measurement capability to a high standard and as such supports the continued UKAS accreditation of Materials characterisation related measurement services.

**The Need**

A wide variety of underpinning material characterisation capability is needed to support the research in the Materials programme. Also, NPL provides a variety of measurement solutions to support the UK National Measurement System, which rely on utilising an infrastructure of measurement devices and facilities developed and run by the Materials Team. It is not sensible to support generic, underpinning facilities from individual research projects that are prioritised. Therefore, to ensure that these facilities are maintained, calibrated and operated to traceable standards and procedures over a wide range of conditions, and that their operation is peer reviewed by an external body, it is necessary for a separate funding route to be provided for this work.

**The Solution**

Support for the maintenance, calibration and accreditation of equipment providing optical and electron imaging of samples, including their metallographic preparation, heat transfer properties, magnetic properties, mechanical test facilities and for the licensing of critical underpinning software.

**Project Description (including summary of technical work)**

- To provide capabilities for the cutting, mounting and polishing of a range of materials covering the main interests of NPL, including metals, oxides, composites and polymers;
- To provide capabilities for examination of objects over a range of length scales from macro- to nano- scales using optical and scanning electron microscopy, and to retain the NPL’s UKAS accreditation for these services;
- Capabilities to measure heat transfer properties of materials groups and to retain UKAS accreditation;
- To provide capability for measurement of magnetic properties of materials and maintain NPL’s UKAS accreditation;
- To provide capabilities for mechanical testing of materials covering the main interests of NPL, including metals, oxides, composites and polymers.

**Impact and Benefits**

The benefits of providing these facilities are that all NMS programmes including Materials and IRD have available suitable facilities for underpinning material characterisation and that the wider UK user base has facilities that can provide traceability for materials measurement in the UK. In particular this project will provide:

- A materials preparation and measurement facility which underpins the Materials and Standards development work at NPL;
- Confidence that fundamental measurements in support of NMS projects and measurement service customers are made correctly through the accreditation of facilities by UKAS;
- A centralised approach to traceability of data from microscopes and mechanical testing equipment across NMS funded work at NPL;
- A unified solution to COSHH and ISO9002 for all metallographic and mechanical testing work at NPL.

The impact of these benefits will be measured through the successful completion and uptake of outputs of projects which utilise these facilities.

**Support for Programme Challenge, Roadmaps, Government Strategies**

This project underpins a diverse variety of projects which themselves support the challenges, roadmaps and strategies of the Government and National Measurement System.

**Synergies with other projects / programmes**

The project will support any work carried out within NPL for Government and UK Industry in the fields of microscopy, heat transfer, mechanical testing, modelling and magnetics for materials testing, thermal and analytical science.
Risks
- Major failure of equipment outside scope of service agreements, mitigated by training of staff and regular maintenance.
- Risk of injury of staff member using apparatus reduced by controlled use and staff training.
- Risk of building services failure outside scope of project, reduced by close working with Amey building services.

Knowledge Transfer and Exploitation
Direct exploitation will be by providing internal services to work at NPL which will then be taken up externally. The accreditation of techniques will be exploited in the sales of measurements and services by NPL to other customers.

Co-funding and Collaborators
Collaboration will be with, and through the other projects utilising the facilities. Details available on request.

Deliverables

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Summary

The primary goal is an advanced basis for plant life management leading to more efficient production with minimal uncontrolled plant outages associated with environment induced cracking. A current limitation, at root a metrology constraint, is the inability to account reliably for the growth rate of environment induced cracks in the short crack regime, from sub-grain size to about 1 mm (below the non-destructive evaluation, NDE, limit). An approach to through-thickness short-crack growth measurement was developed in the 2008-2011 project and provided a direct comparison with long crack growth measurements on similar specimens. However, while a key first step, the growth rate measured represents an average across 500 grains and may not reflect the early stages of crack development in the first few grains, near surface. Advanced measurement and analytical methods are required to bridge the gap. The main output will be a draft international standard on short crack growth measurement. In addition, an approach to prediction of time-dependent damage development will be set out. It is envisaged that there will be a further small extension of the project beyond 2014 to consolidate the draft standard through ISO.

The Need

- demand for longer design lives for structures in corrosive environments
- improved prediction of structural lifetime from short-term data
- guidance on maintenance schedules
- dating of cracks removed from service
- guidance on surface finish on key components

Life prediction assessment and intelligent plant maintenance in applications where the environment impacts on integrity are largely based on long crack growth data or on time/cycles to failure. The latter has inherent limitations where pitting corrosion or pre-existing defects impact on life, and size effects in laboratory testing lead to additional constraints. For long cracks, standards are in place for measuring crack growth rates and there is a degree of confidence in their laboratory and engineering application, the latter coupled with advanced NDE techniques. However, in the case of short cracks, there are no standards that guide the measurement process; simply recognition that growth behaviour can be different, that the rate will be sensitive to near-surface properties, and that the time spent in the short crack regime has an important impact on life assessment codes and inspection intervals. Guidelines documents on fracture assessment do not deal adequately with environmental effects and short cracks. The underpinning/generic nature of the research, with the goal to incorporate the methodology into an ISO standard, makes it applicable to wide range of industrial applications including steam turbines in conventional and nuclear power plants, flexible pipework and risers in oil and gas production, and pressure vessels in the chemical industry.

The Solution

- The metrology challenges that will be resolved are measuring crack extension when the crack size is small and growth rates are slow, characterising the evolution of crack shape, ascribing the mechanical driving force for the evolving crack, dealing with the distinct feature of cracks emerging in the strain fields associated with corrosion pits and, where appropriate, accounting for crack coalescence. The major output of this project will be a draft international standard that describes the process of short crack growth measurement and includes specimen type, methods of generating initial defects, measurement of crack size and crack shape evolution, and how to estimate the mechanical driving force.
- The through-thickness short-crack growth measurement developed in the 2008-2011 project provided a direct correlation with long crack growth measurements on similar fracture mechanics specimens. However, the growth rate measured is an average over at least 500 grains and may not be applicable to crack development in the first few grains. Hence, there is a need to develop a methodology for characterising the growth rate in the early stages of crack development and its dependence on near-surface microstructure, mechanical properties, stress state and environment.
- Complementing the establishment of measurement methods, a model of the crack evolution process will be developed encompassing short and long crack growth in a format accessible to industry.

Project Description

- Crack precursors will be produced in a steam turbine blade steel in the form of single pits of controlled size using electrochemical methods developed at NPL and as physical defects introduced by focused ion beam machining. The optimum method for measuring the growth of a crack in the first few grains (from pits and defects) will be established, based on...
combined electrical and optical techniques. These tests will be carried out in air first under fatigue conditions to optimise the approach. At that stage an interim report will be written.

- Short crack growth rates will be measured for both stress corrosion and corrosion fatigue conditions using defects of different initial size and compared with previous data on through-thickness specimens and long crack growth rates.
- Parallel tests will be undertaken to define the crack shape using a combination of crack staining and fracture and, in separate tests, X-ray tomography at U. Birmingham.
- Finite element analysis (FEA) will be undertaken to characterise the distribution of stresses for the specific defect geometries for comparison with previous work.
- The insights gained from the testing will provide the basis of a draft standard.
- A pragmatic model will be developed that encompasses short and long crack growth from defects.
- Using samples from service and specimens from laboratory test an evaluation of crack dating will be made based on the characteristics of the oxide with distance from the crack tip.

**Impact and Benefits**

In a power plant, an uncontrolled outage can cost £0.5m/d while the cost of a major failure as that at Aberthaw can approach £10m. An inadequate basis for prediction can also lead to an unnecessary outage and loss of power availability. NPL’s advice has been sought in the UK on the impact of water chemistry on the potential for cracking of steam turbine, on crack growth rates under different conditions, in interpreting the significance of pitting, and on understanding the origin of failures. This project will further underpin that process and support NPL’s world leading role in this area, which has attracted funding from the USA (EPRI), Germany (Siemens) and Korea (Doosan). The project of 2008-2011 led to an invitation to give a key talk at the Gordon Conference (Frontiers of Science) on Corrosion in 2010. The extension in the proposed project to meet the further challenges of short crack growth measurement at the grain size level will sustain our position at the scientific leading edge whilst developing enhanced measurement capability. Complementary to the specific focus on steam turbine, NPL acts as an advisor to BP on the programme of work that is being initiated at U. Manchester on measuring and predicting the growth of corrosion fatigue cracks from pits and defects in corrosion resistant alloys in flexible pipework and flow lines. The cost of a single failure is well known.

**Support for Programme Challenge, Roadmaps, Government Strategies**

Mat UK Energy Strategies on Fossil and Nuclear Power generation, Reduced carbon dioxide emission, Advanced Materials, Lifetime prediction/modelling

**Synergies with other projects / programmes**

The proposed project feeds off and contributes to the long-term “Grains to Structure” project and also is a direct complement to EPRI and Siemens funded projects on characterising crack development in steam turbine steels. In addition, an EPSRC grant has been awarded to Birmingham on XRT and crack-shape development that is designed specifically to link with this project. The project also has links to a major NPL-Serco-MOD project for pressurised water reactors, which is nearing conclusion.

**Risks**

There is an inherent challenge in dealing with the evolution of crack shape and derivation of a crack growth law that accounts for the mechanical driving force. In the latter case, consultation with experts on fracture mechanics will provide a perspective on the optimum approach. Crack coalescence may be important and is challenging to model but there is a framework for dealing with it.

**Knowledge Transfer and Exploitation**

The currently active IAG on steam turbines will be sustained and will provide the first route for dissemination to the major power plant operators. Presentations to the Steam Turbine Users Group meeting and the Parsons conference ensure extended dissemination to the wider UK Power plant industry. The link to BP and to the nuclear industry though separate funded projects will provide routes for further dissemination in different technological areas. Complementing this engineering focus, there will be a minimum of two high quality scientific papers and one science-based international conference as well as a draft ISO standard.

**Deliverables**

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<td>Draft scientific paper on “Environment induced small crack growth in a steam turbine blade steel”</td>
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39
A critical need is developing for electronics manufacturers to monitor the health of their products, as the business model for high value equipment changes and pushes responsibility onto them. This trend receives added impetus as safety critical systems are applied into increasingly hostile conditions in a number of sectors such as military, transport and power generation. Interconnects are a weak link on circuit assemblies and hence there is a need to monitor the wear out mechanisms and predict remaining lifetime. This project looks at identifying indicators and the mathematical tools needed to provide a prognostic health monitoring capability for electronic assemblies. The indicators will be based on the processed signal response to injected electrical signals. The new prediction systems will minimise both maintenance costs and risk of failure, and therefore enhance safety.

**State of the art:** This is a relatively new area, and there are two centres in US universities, but currently limited capability in Europe.

**NPL capability:** Developed controlled test environment for electronics, microstructural and lifetime characterisation of interconnects, use of electrical techniques to establish change in state of systems. Extensive mathematical experience of analysing and modelling signals from a wide range of applications using both time domain and frequency domain techniques, of uncertainty analysis for metrology applications. Hence, we are well positioned to extend this capability into prognostics.

**Summary**

The need for customers of complex electronic systems are increasingly buying a service, pushing back the hardware maintenance onto the manufacturer. These pressures are the drivers for lower maintenance costs and lower risks of failure. To meet these requirements there is increasing need to monitor residual lifetime in service, and hence avoid catastrophic failures by monitoring the “health” of a system, for example in the control system for aero engines. Whilst health monitoring deals with evaluating the health during operation, prognostic health management (PHM) aims at predicting the remaining useful life (RUL) by the use of damage proxies as indication of potential failures and by applying suitable methods of analysing measurements of these damage proxies. As such, it has a wide applicability, especially in applications requiring ultra-high reliability. Although prognostic techniques have been applied in mechanical structural systems for a long time, their application in electronics is still in its infancy. Electronic systems are complex structures where a large range of materials are used and hence they are susceptible to degradation (fatigue, creep, and corrosion) by mechanical, thermal and humidity factors, which can be experienced concurrently.

The research, developed in close collaboration with partners in the electronics industry, will start from a selection of critical parts of electronic systems. These could include solder joints, power electronic devices such as IGBTs or micro-electromechanical systems (MEMS). Initially, however, the focus will be on solder joints, where NPL has developed dedicated test facilities. The NPL developed instrumentation will provide an excellent platform onto which indicators for PHM can be added, and hence correlated with known performance indicators. An important step will be to build on the understanding of the main lifetime degradation mechanisms and to develop a range of sensing approaches that will be used as health monitors, or damage proxies. A possible list of damage indicators suitable for on-line PHM of electronic systems will be defined. The practical approach is to monitor the variation of several inputs (proxies) and use them as precursor indicators of impending failure (data-based approach). Alternatively, if the physics of the failure mode is known, a model to predict remaining lifetime can be incorporated in the procedure (model-based approach). The quality of individual approaches has not been validated. NPL plans to develop robust metrology method that can be validated, and look at the most suitable mathematical tools for analysing the measurements and evaluating uncertainties. These new developed capabilities will enhance NPL portfolio in the areas of reliability and health monitoring of electronics with immediate benefits to the UK industry.

**Project Description (including summary of technical work)**

The project will be steered with input from the industrial partners. The project flow will look to select suitable indicators, and in combination with various environmental stress develop mathematical tools to predict remaining lifetime. The project aim is to produce a procedure that includes sensing and prediction techniques for industrial applications.

- **D1:** A review and detailed project plan will be developed and the project partners engaged.
- **D2:** This will include design of an instrument for combined linearity and impedance testing instrument capable of measuring V-I hysteresis loops up to 10kHz, impedance frequency analysis and Time Domain Reflectometry (TDR). These systems will be used in conjunction with existing mechanical fatigue machines developed at NPL to establish relationships of prognostic indicators with mechanical indicators. A Printed Circuit Assembly (PCA) incorporating selected sensitive components will be constructed. The developed electrical techniques will be used to determine indicators suitable for prognostics and monitor them at different stages as the PCA is subject to vibration damage. Evidence: Published NPL Report / paper
- **D3:** Using suitable sensors that act as damage indicators from deliverable 1, and combining with temperature, humidity and...
acceleration sensors, a new PCA will be constructed which will log the harsh environmental situations the PCA experiences. Data collected from the PCA’s that have been subjected to different fatigue processes (shock, thermal cycling and vibration) will be analysed and related to damage within electronic systems. Evidence: Published paper
D4: Investigation on the most suitable mathematical methods to be used in conjunction with the instruments and sensors developed in deliverables 1 and 2. These methods will be either data-based or model-based and will be used to analyse the data acquired, correlate this with the damage accumulation and formulate a RUL prediction. It is anticipated that we will follow a data analysis route, which would also have wider applicability in larger systems. This will lead to the formulation of a well-defined procedure that will be integral part of the novel capability. Evidence: Published paper / NPL Report
D5: With the partners a simple but representative test vehicle will be designed and tested. A range of stress tests, which in addition to vibration will also include low cycle fatigue and shock testing, will be applied to test the applicability of the identified damage indicators and the mathematical tools to predict RUL. Evidence: Published paper and NPL Report

Impact and Benefits
The development of PHM for electronics systems will increase NPL capabilities in the field of health monitoring and reliability with knowledge and metrology tools that put NPL in a leading position in Europe. This will provide an important service to the UK electronics industry. In fact, the ability to predict more consistently lifetime of critical products will allow industry to reduce scheduled and unscheduled maintenance and retain effectiveness through timely repair actions. For example, prognostics can indicate when an IGBT within an offshore wind turbine is going to fail, facilitating replacement in good time, saving a significant amount of energy that would otherwise be lost. With further development where the electronics is part of a sensing or control system enhanced health monitoring methods will give confidence with advanced warning of critical failures and reduce the life-cycle cost of equipment by decreasing inspection costs and downtime.

Support for Programme Challenge, Roadmaps, Government Strategies
The TSB have identified several areas that this project supports: the most obvious is in the technology area: Electronics, photonics and electrical systems, but also innovation platforms of the Low Carbon Vehicles and other areas covering various aspects of transport. The project will help electronics systems that are protecting critical functions. This project will also help the UK electronics industry to remain competitive, which is one of the biggest of the European markets having a value of ca £40bn generating =10% of the UK’s GDP and =15% of UK trade

Synergies with other projects / programmes
Recent programmes such as AD22 and MAT/2010/04 have seen the development of mechanical testing machines for solders and adhesives. Use of these instruments will be invaluable as they will provide a mechanical damage indicator (normally the supported load) that can be correlated with the electrical indicator sought through prognostics. Future work could build on capability developed here for applications in MEMS, high density and 3D packages. These applications could in themselves be sensors for system integrity.

Risks: Project risk lies in achieving damage indicators with good signal to noise that are sensitive to degradation in the joint. The mitigation approach is to assess a number of probing techniques and the plan is configured to down-select to the strongest response. Similarly the most suitable mathematical approach for predicting lifetime will be adopted in collaboration with the modelling group at NPL.

Knowledge Transfer and Exploitation
There would be planned review and planning meetings with partners. We will prepare peer reviewed papers, submitted to Journal of Microelectronics Reliability and IEEE Transactions on Components and Packaging Technologies, through international conferences (e.g. EuroSIME conference). Present at an Electronics KTN reliability event, presentations at events organised by two trade organisations, including a webinar. The work will be presented to a wider IAG audience.

Co-funding and Collaborators
Details available on request.

Deliverables
<table>
<thead>
<tr>
<th>Deliverable</th>
<th>Start</th>
<th>End</th>
<th>Cost: £</th>
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<tbody>
<tr>
<td>1</td>
<td>Start: Oct 2011</td>
<td>End: Dec 2011</td>
<td></td>
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<tr>
<td></td>
<td>Review and detailed planning stage</td>
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<td>2</td>
<td>Start: Dec 2011</td>
<td>End: Dec 2012</td>
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<tr>
<td></td>
<td>Development of experimental techniques for identification of reliability indicators.</td>
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<tr>
<td>3</td>
<td>Start: Sept 2012</td>
<td>End: July 2013</td>
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<tr>
<td></td>
<td>Characterising response variables in harsh stress environments</td>
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<tr>
<td>4</td>
<td>Start: May 2013</td>
<td>End: Nov 2013</td>
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<tr>
<td></td>
<td>Mathematical tools for prognostic prediction</td>
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<tr>
<td>5</td>
<td>Start: Oct 2013</td>
<td>End: June 2014</td>
<td></td>
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<tr>
<td></td>
<td>Development and validation of a test methodology suitable for application to printed circuit assemblies</td>
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</table>
The existing SHM demonstrator based on the NPL footbridge has enabled NPL to develop its SHM expertise to the point where we have been invited to present papers at a key international conference “Structural Faults and Repairs 2010” in Edinburgh and have established a large IAG with more than 40 contributors /collaborators including many of the main UK asset holders. The lifetime of this demonstrator is now very limited as the testing of the structure reaches its final phase, but it has fulfilled its promise as the scale and types of instrumentation on this structure is state of the art in the Civil Engineering community. From interactions with the IAG it is evident that full Asset Management is the main driver for instrumenting structures and reducing costs. Asset management covers not only the structural health/integrity monitoring and assessment of the ageing infrastructure which might be subject to intensified use and/or adverse weather, but also monitoring of the performance of key functions of the structure, such as a floor vibration monitoring in hospital theatres; or pollution, temperature and humidity monitoring of the interior environment to prevent degradation (for instance, in historic buildings). Using data based decision making will improve energy efficiency and sustainability. To enable this vision needs knowledge derived, using processing techniques developed in this project, from data streams provided by a remote structural monitoring control centre.

The Need
Structural health/integrity monitoring has been developed to meet increasingly stringent demands on safety in extreme operational conditions. The main challenges are effective interpretation of experimental data for the life cycle asset management and reducing the costs of using monitoring systems primarily installation cost and in particular cabling on large-scale structures. To overcome these challenges requires new, technically very advanced and multi-disciplinary in nature activities. They include automated, wireless access to data using the most appropriate transmission and network protocols for sensing systems together with reliable, sustainable power sources based on various forms of energy harvesting. The demand for long-term secure data storage combined with fast access and reliable analysis for sensing applications is growing and, to our knowledge, there is no adequate solution available yet.

The Solution
To determine the benefits and limitations of different analysis techniques for use in asset management methods will be developed by NPL and collaborators. The novel data handling techniques will then be validated with new data streams from a remote structural monitoring control centre in NPL interfaced to the NPL Bridge and real structures. This will allow investigation of the various transmission types and protocols, broadened to include environmental condition monitoring and/or structural health/integrity monitoring of real structures. Other benefits of such a centre will be independent validation, certification, data management (storage) and analysis. The control centre will be equipped using equipment from an industrial consortium that will extend over the course of the project.

Project Description
The centre will be set up in a room in NPL equipped with servers connected to various wireless sensing systems installed on the NPL bridge and real structures. This control centre will provide data streams for further analysis, with the primary aim to develop reliable data processing techniques for aiding asset management. Issues that might effect the reliability of such techniques include:

- Cellular networks and wireless local area networks will be compared to identify if any issues exist specific to transmission of sensor data.
- Durability and reliability of the equipment and different ways of transmission under real life situations will be examined
- Assessment of data quality for large-scale networks of different monitoring technologies including handling and maintenance of archived and real-time data.
- Device power consumption in a series of scenarios will be evaluated and the effect of power control on the interference levels in the network will be investigated.

Impact and Benefits
The facility set up during this project will be used continuously for a number of years to maintain an important continuity of collaborations based on the bridge demonstrator and attract more interested parties, bringing co-funding and third party funding
to allow expansion of this technical area by creating new consortia targeting specific areas. It will strengthen the role of NPL as a focus for the structural health/integrity monitoring community and facilitate collaborative research to increase the role of sensor and monitoring technologies in the asset management process. It will provide a facility for training purposes covering a broad spectrum of participants such as UK and international students as well as technical specialists and to introduce new technologies and methodologies to asset managers and respond to their concerns. It will allow NPL to gain practical experience in technologies and work with providers leading to the capability to establish independent validation/calibration of new technologies and also long-term installations. This project will develop expertise in distributed measurement useful for future services like support to industry on applications, consultancy, training etc. An output of this project will be a paper in ‘Civil Engineer’, one of the main publications of the civil engineering sector.

**Support for Programme Challenge, Roadmaps, Government Strategies**

This project supports both the NMO Energy Challenge, as asset monitoring is vital in the life extension of Nuclear power and the economics of renewable energy supplies. This project also supports the Sustainability Challenge as it helps extend the lifetimes of civil assets and reduces their environmental impact by encouraging more efficient usage. The techniques that are developed will become important for the Digital Economy Challenge as greater control over structures operations becomes a possibility.

**Synergies with other projects / programmes**

Existing NMO funded projects such as SHM and DIC.
Innovation R&D Wireless Propagation and interference project.
TSB OWEL Marine Demonstrator: Specification of appropriate instrumentation and sensors.
SR Project Novel measurement techniques for radiation pattern characterisation of embedded radios emitting modulated signals
Bids/opportunities: FP7 - ICT for energy efficient buildings

**Risks**

The continuity of activities is essential to keep current collaborators community active. The current economic downturn may effect the development of new industrial consortia, but focusing to attract international partners and new markets will help mitigate this. Asset holders may not be prepared to share real data for research and demonstration purposes but we may be more successful by targeting the heritage/culture sector with existing government-funded projects. Funding decreases in higher education may limit initial income generated from training but it is not our main activity and running costs of the centre will be low. As chosen technologies may not perform as expected we’ll try to include as many as practical, but at least three. If this project doesn’t go ahead then there is a high probability that the current consortia and collaborative projects will not continue; maintaining funding in this area will allow existing collaborations to continue and new ones to be developed.

**Knowledge Transfer and Exploitation**

Using the facility to support live SHM, providing support in system design and with data handling and interpretation will develop UK skills in this area.
Using the facility for training/knowledge transfer purposes covering a broad spectrum of participants such as UK and international students, technical staff and introducing new technologies and methodologies to asset managers.
Developing international collaboration to take advantage of the EU and other international funding when opportunities arise and work with multi-national companies. Present conference paper at an international conference

**Co-funding and Collaborators**

The contribution in kind includes sensor networks and associated software (initially £5-10K each and about £20-30K in total). The contribution will also increase as the demonstrator reaches maturity in a similar way to the existing NPL bridge project. Free access to a medium/large-scale network of a real installation for research and demonstration purposes (to create one would cost up to £1M).
Seminars/meetings/training run through the centre are expected to bring in an annual income to cover running costs as the demonstrator reaches maturity.
When centre is established particularly with international presence a set of annual membership fees are planned to be introduced, for example, level 1 of £500 for access to various publication/presentations up to full membership.

**Deliverables**

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<th>Deliverable</th>
<th>Start</th>
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<th>Cost</th>
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<tr>
<td>1</td>
<td>Start: July 2013</td>
<td>End: Dec 2013</td>
<td>Cost: £</td>
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<tr>
<td>Set up a centre with wireless access based on two or three types of transmission technologies to a footbridge demonstrator and a real ‘in-use’ structure</td>
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<tr>
<td>2</td>
<td>Start: Dec 2013</td>
<td>End: March 2014</td>
<td>Cost: £</td>
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<tr>
<td>Develop generic training material for future use of Advanced Demonstrator as a training/knowledge transfer centre</td>
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</table>
Thermodynamic models for cement and aqueous phases and calculation methods based upon these models will be developed to create a tool for predicting the results of long term interactions between cements and aqueous solutions encountered in the natural environment, such as groundwater and seawater. These models will encompass high ionic strengths, elevated temperatures and wide composition ranges all beyond the scope of conventional calculations typically restricted to ideal solutions at room temperature interacting with stoichiometric compounds. The aim is to allow cement manufacturers, nuclear service companies and regulators to identify or design, in a swift and cost effective manner, new cement formulations suitable for use in nuclear waste management.

The Need
Current strategies for nuclear waste management focus on encapsulation in cement or cement like materials in metal canisters and disposal deep underground with cement backfill. In the long term, leaking containers, cracked backfill or other degraded structural elements of the disposal facility may lead to water ingress, opening up the possibility of radionuclides being carried into the environment in aqueous solution. The solubility of radionuclides is quite high in the high pH environments resulting from current cement formulations. This project aims to develop thermodynamic models for cement and aqueous phases and predictive calculation methods to underpin the identification and development of innovative low pH cement formulations able to contain radionuclides effectively. The long term (many thousands of years) stability and leaching behaviour of different cement formulations is currently investigated experimentally through short term “accelerated” tests which may not truly mimic long term behaviour. Current models tend to be valid only at room temperature, treating aqueous solutions as ideal and cements as collections of stoichiometric compounds. Although the feasibility of more sophisticated modelling techniques has been demonstrated at NPL for simple calcium silicate hydrate (C-S-H) gels,\(^1\) the comprehensive coverage of possible cement compositions and leachable ions necessary for nuclear waste management applications is not yet available. This project aims to address that deficiency. The generic nature of the planned underpinning model development work and the likely impact of the project in the drive towards low CO₂ energy production and the development of new and innovative materials makes it very suitable for Government support as does the aim of ensuring safe long term management of nuclear waste in the UK.\(^{2,3}\) J A Gisby, R H Davies, A T Dinsdale, M Tyrer, F P Glasser, J Hill, P Livesey, C S Walker C-S-H solubility modelling at high ionic strengths, 12th International Congress on the Chemistry of Cement, Montreal, 2007.

The Solution
The deficiency in current models outlined above will be addressed by developing new models for high ionic strength aqueous solutions and cement phases including ions (radionuclides), covering compositions scoped in initial discussions with the project partners. These models will be implemented within the NPL software for the calculation of phase equilibria, MTDATA and parameterised to produce a database which will be provided to the partners for testing, validation and ultimately use in making predictive calculations. IPR will be retained by NPL so dissemination to non-partners after an agreed lead-time will also be possible. The metrological aspect of this project lies in the development of a tool to predict the long term behaviour of materials likely to play a pivotal role in the UK’s future energy strategy, that of ensuring the safe management of nuclear waste. The long term nature of this behaviour, timescales of thousands of years, makes it difficult to address with confidence experimentally. Models and model parameters to underpin thermodynamic calculations, the best method for predicting long term behaviour, will be provided by this project, adding value to existing “accelerated” measurements by providing a yardstick for their suitability as indicators of long term behaviour, allowing future measurements to be targeted for maximum effect.

Project Description (including summary of technical work)
1) PROJECT SCOPING - Initial discussions with project partners, including academic experts in cement chemistry and representatives of cement manufacturers, nuclear service companies and regulators will be held to determine the cement phases, aqueous species and overall composition and temperature ranges which must be addressed to produce a useful tool. Generic test species and compositions will be identified for use during model development along with a wider range of species and compositions for consideration during final database production (model parameterisation).

2) MODEL DEVELOPMENT - Solution phases in cements and high ionic strength aqueous solutions will be modelled using the framework of the compound energy model a modified version of the Pitzer aqueous ion-ion interaction model. The choice of ions
to be introduced, their sublattice occupancy and the development of parameters to model the thermodynamics of their introduction and interaction, guided by experimental measurements either from the scientific literature or provided in-kind by project partners, will be the key modelling tasks. Work will focus on the system for model development identified in 1). The outcome will be a proven methodology for modelling cement and aqueous phases to underpin wider database development to follow. A scientific paper providing model details and results of comparisons between calculation and experiment will be written and submitted to a peer reviewed, Web of Science indexed, journal. This underpinning modelling work will be mainly Government funded.

3) DATABASE DEVELOPMENT - The modelling methodology developed in 2) will be used to parameterise cement and aqueous solution phases in the database development system identified in 1) to underpin “real world” calculations planned for database validation purposes in 4). This database development work will be mainly funded by cash contributions from project partners.

4) DATABASE TESTING AND VALIDATION - The cement and aqueous models will be brought together to predict the likely long term outcomes of interactions between cements and different aqueous leachates, each with “real” compositions, rather than simple idealised compositions. The exact nature of these calculations will be guided by the availability of experimental measurements for comparison with calculated results. An exploration of different calculation procedures (single step or iterative) is planned to identify that most appropriate for adoption in leaching calculations.

**Impact and Benefits** Uptake of the predictive capability produced by this project can be directly measured in terms of the number of companies licensing and using the database produced. Impact on the economy and quality of life will come through the development of innovative materials and an improved ability to quantify long term materials performance, specifically the long term suitability of new cements designed for use in nuclear waste management. This will lead to a safer environment and promote public confidence in nuclear energy, important in the struggle to control UK’s CO₂ emissions. Spin off benefits of the availability of improved predictive models are likely in other industries, such as building, hydro metallurgical processing (modelling of high ionic strength aqueous solutions will aid process optimisation) and the remediation of contaminated land (modelling will aid development of new speciality cements).

**Support for Programme Challenge, Roadmaps, Government Strategies**
The project addresses the science priority areas of “modelling to characterise materials”, “quantitative environmental monitoring” and “support for low carbon and sustainable technologies” identified in the NPL science strategy, which supports Government strategy. It is concerned with the prediction of macroscopic materials properties based upon atomic scale properties. Work of this type, spanning length and time scales, is targeted in the Thermodynamics TAR roadmap.

**Synergies with other projects / programmes**
The project has synergy with ongoing 3rd party projects to develop the NPL phase equilibrium calculation software and to develop the NPL thermodynamic database for oxide systems. Outputs from these projects will feed into the proposed project.

**Risks** The main risks are that incorrect choices of cement phases, aqueous ions or model structures may be made during the modelling process. The involvement of academic experts in cement chemistry will mitigate this risk and help to develop the expertise of NPL staff. The involvement of scientific experts from the industrial partner organisations will be of additional benefit.

**Knowledge Transfer and Exploitation**
Mathematical details of the models generated during the model development phase of the project and examples of their applicability will be disseminated to partner thorough discussions at progress meetings and to a wider audience through a scientific paper written and submitted to a peer reviewed, Web of Science indexed, journal. The final model parameters will be disseminated to partners through databases enabling them to make predictive calculations (when coupled with MTDATA) relevant to their own particular cement formulations and environmental conditions, probably resulting in further scientific publications. Non-partners will be able to lease the databases to perform their own calculations after a lead-time to acknowledge the cash input of the companies supporting the proposed project. Outside the nuclear sector, uptake from cement producers (to aid in the design of innovative products) and users in the building industry (to judge likely long term cement performance) and from hydro metallurgical processors (to use the high ionic strength aqueous model) is also possible.

**Co-funding and Collaborators**
Details available on request.

**Deliverables**

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<th>Deliverables</th>
<th>Start</th>
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<tr>
<td>1 Scientific paper describing model development and initial application</td>
<td>Start: Oct 2011</td>
<td>End: Dec 2012</td>
<td></td>
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<tr>
<td>2 Thermodynamic database for cement and aqueous phases</td>
<td>Start: Jan 2012</td>
<td>End: June 13</td>
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The Need - Future requirements for advanced materials and surface treatments are placing increasing demands on the tools available for characterising their performance and properties, particularly at the grain size level. Mechanical relaxation techniques such as hole drilling, slitting and curvature methods are well developed and have traditionally been used for measuring macro residual stresses on large testpieces and components, but the integration of the FIB/SEM with full field displacement measurements opens up new opportunities for using micro-relaxation techniques at much smaller length scales. It becomes possible, for example, using small holes, slits and sections to examine the characteristics of machining damage and simulate the development and mitigation of residual stress caused by local mechanisms such as crack initiation, plastic deformation and stress corrosion cracking. Initial studies using the FIB to investigate micro-hole drilling and slitting in the UK have been carried out at Manchester and Bristol Universities, and this is the current state of the art, but the experimental procedures, metrology and modelling of this approach are still at an early stage. Issues to be resolved relate to optimizing the conditions for ion beam milling, acquisition of FIB/SEM images, the DIC process and algorithms used to obtain reliable high resolution strain maps, and validation of the models themselves. Because the focus of the work is aimed at developing a new capability significant effort is required to develop and validate the test methodology, there is limited opportunity to leverage industrial funding and NMO support is essential to underpin the work.

The Solution - NPL has previously demonstrated the feasibility of using DIC with hole drilling as a non-contact method for monitoring full field displacements and measuring residual stress fields, and the methodology will be developed further in this project, albeit on a smaller scale in the FIB/SEM, with greater emphasis on modelling and the use of high resolution, full field displacement maps. The solution is to develop validated procedures and models for measuring residual stress at the mesoscale, based around micro-relaxation techniques (micro hole drilling and slitting) in the FIB/SEM, using full field strain mapping and modelling. This will be achieved through a combination of experimental work, modelling and validation, and disseminated as a Good Practice Guide. Metrology issues that will be addressed include the optimisation of the milling process, the influence of hole and slit geometry, optimisation of fine scale patterns/coatings for in-situ imaging, the validation of DIC strain mapping, and the development and validation of appropriate FE and analytical models. When milling holes it is difficult to control and accurately predict their dimensions, but this will be modelled and indeed exploited with the current approach, to investigate stress distributions associated with non-uniform shaped defects such as inclusions, and stress corrosion pits. Two systems/case studies will be examined in detail, examining the influence of different machining conditions on the residual stress state in surface layers in 304 Stainless steel, and by simulating and measuring in the FIB the competing actions of stress intensity factor enhancement and residual stress relaxation caused by the change in geometry and size of corrosion pits. An intercomparison exercise, using reference materials with a known, well-defined stress state, will support the technique development.

Project Description (including summary of technical work) The project is a combination of experimental work and modelling, carried out by mainly by NPL in collaboration with leading experts in the field, with a series of activities/tasks, focused on:

- Defining the optimum FIB milling parameters and procedures for generating accurate holes and slits, and reducing damage induced during the milling process
- Optimisation of the geometries/specimen configurations for drilling and slitting

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**Project No.** MAT\2011\18  
**Price to NMO** £488k  
**Co-funding target** £100k

**Project Title** Micro-relaxation Techniques for Residual Stress

**Lead Scientist** Jerry Lord  
**Stage Start Date** July 2011

**Scientist Team** Jerry Lord, Nick McCormick, Louise Crocker, Ken Mingard, Neil McCartney  
**Stage End Date** June 2014  
**Est Final Stage End Date** June 2014

**Sector** Advanced Manufacturing and Services  
**Activity** Methodology for New Capabilities

**Project Champion** Peter Flewitt
Developing procedures for image mapping in the FIB/SEM, including the application and optimisation of fine scale patterns for DIC imaging, alleviating potential problems with rigid body motion, out of plane movement, drift, etching and sputtering

Optimised procedures and algorithms for the calculation of accurate full field surface strain fields, based on DIC

Developing FE and analytical modelling capability and an experimental validation programme to support the above

An Intercomparison/collaboration with UK experts to validate the measurements and models using a set of “reference materials” with known residual stress fields

Validation with other residual stress techniques

Developing the world-first integrated DIC/hole drilling prototype instrument

Impact and Benefits
The main benefit is the availability to UK industry of validated procedures and a methodology for obtaining reliable residual stress measurement and strain mapping capability in the FIB/SEM; the impact will be measured by the uptake and level of collaboration developed. The techniques developed in the project will be used to provide a better understanding of the local variation in properties within a component, and provide the means for characterizing new materials and process treatments over a range of length scales, generating supporting data for the prediction of macro scale properties and performance with significant reductions in the time to market. The work will be multi-sectorial to ensure maximum impact, with uptake of the test methodologies expected within the project lifetime in the energy generation, materials manufacturing, aerospace, automotive and machining industries.

Support for Programme Challenge, Roadmaps, Government Strategies - OK
Aligns with the Advanced Materials and High Value Manufacturing strategies, directly supporting the development of advanced materials and processes, and the provision of data for modelling and design. It supports the MAT UK roadmap and Materials Division strategy for in-situ measurement and multiscale characterisation, materials modelling, structural integrity and NDT through the development of novel micro-scale techniques and modelling capability.

Synergies with other projects / programmes
The project:
- Builds on current work in projects A20 (property mapping), A22 (miniaturised testing)
- Supports work on SCC and short crack growth
- Interfaces with the proposed Materials 2011 “FE/DIC modelling” project
- Satellite project to the Materials 2011 “Grains to structures” Grand Challenge

The work has direct relevance to the current residual stress related issues associated with novel surface treatments and materials processes, high temperature degradation and corrosion studies, for characterising the performance of electronics, MEMS and nano composites and will complement the EBSD work on strain measurement. The project will also represent a significant enhancement/development in NPL’s residual stress modelling capability, and this will be developed further in future projects, through the integration of more comprehensive physical models incorporating grain size and microstructure effects.

Risks
- Availability of in house residual stress modelling expertise – review with key NPL personnel, develop collaboration with Manchester, Bristol Universities and Stresscraft.
- Limited access to FIB – collaboration with Manchester, Bristol & Surrey Universities will alleviate the demand
- IP issues related to DIC/hole drilling system – not aware of current issues, but a patent search will be carried out. Options to modify approach will be investigated with commercial partners

Knowledge Transfer and Exploitation
KT will be achieved through a combination of scientific papers (Mat. Sci. Tech or Exptal. Mechs.), contributions to the FIB GPG, presentations at relevant BSSM workshops and conferences, dedicated website pages and 3rd party work. Take up is expected by a wide range of industries with an interest in material development and experimental mechanics, machining, surface treatments and coatings, and portable residual stress measurement including Alstom, E.On, TWI, British Energy, Serco Assurance, Airbus, Stresscraft, Stresstech, LaVision and Imetrum. The licensing and future commercialisation of the DIC-based hole drilling system offers a significant opportunity for exploitation.

Co-funding and Collaborators
Details available on request.

Deliverables

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<tr>
<td>1</td>
<td>Start: July 2011</td>
<td>End: June 2014</td>
<td>£</td>
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<tr>
<td>Scientific paper on the metrology of micro-residual stress measurement, and input to FIB GPG developed in the Materials 2011 “Grains to structures” project</td>
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<tr>
<td>2</td>
<td>Start: Nov 2011</td>
<td>End: June 2014</td>
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<tr>
<td>Prototype DIC/hole drilling instrument and scientific paper</td>
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**Summary** - A model will be developed and parameterised to provide a tool for predicting the electrical conductivity of molten oxides, including glasses, as a function of composition and temperature. Such a tool is required to underpin optimisation of the design and energy use of processes in metals refining and recycling, glass production and recycling where electrical melting is important. Value will be added in the ability to predict the electrical properties of speciality glasses used in photovoltaic panels, for example.

**The Need** - The necessity to reduce energy usage and adapt processes to respond to new feed-stocks or product requirements is the driver for the need to know electrical conductivity for a wide range of liquid oxide compositions in the metals and glass industries. The time taken for and expense of experimental measurements slows progress. Existing models for electrical conductivity of high temperature liquid oxides and glasses tend to be empirical fits based on overall composition in terms of simple oxides with some theoretical input with regard to temperature dependence, single sets of parameters covering limited ranges of composition. The proposed model, if successful, will make predictions possible for a wide range of compositions, based upon the sound thermodynamic modelling of liquid oxides, in terms of non-ideally interacting associated species as well as simple oxide species, for which NPL is already held in high regard by industry. The generic nature of the model development work and its potential for underpinning reduced energy use and CO₂ emissions and speedier development of innovative products makes it suitable for partial Government funding. More specific model parameterisation work geared to particular oxide combinations will be funded by industrial sponsors. Hence the high cash co-funding target for the project.

**The Solution** - NPL has developed a world leading capability to predict equilibrium phase assemblages, compositions and thermophysical properties of oxide systems over many years. A model for calculating the viscosity of liquid oxides building upon the established thermodynamic model has also been developed \(^{(1)}\). The aim is to develop the mathematical framework for an electrical conductivity model underpinned by previous work on modelling the viscosity of liquid oxides and to implement this within the NPL software for the calculation of phase equilibria, MTDATA. The viscosity model will be used as an early test bed for ideas to be adopted in electrical conductivity modelling, to shorten the overall time taken for model development, so some additional work on viscosity is planned. Parameterisation of the electrical conductivity model to cover ranges of composition appropriate to the needs of the project’s industrial partners will then be undertaken. The result will be a database of parameters which will be provided to the partners for testing, validation and ultimately use with MTDATA in making predictive calculations. The partners are all current MTDATA users. IPR will be retained by NPL so dissemination to non-partners after an agreed lead time will also be possible. The metrological aspect of this project lies in the development of a tool to predict an important thermophysical property which is time consuming and expensive to measure. This adds value to existing measurements used as a basis for model development and allows future measurements to be targeted for maximum effect. \(^{(1)}\) P Taskinen, J Gisby, J Pihlasalo, T Kolhinen, Validation of a new viscosity database with industrial smelting slags, Proc. Int. Conf. EMC2009. Innsbruck Austria, Vol 3, 13pp.

**Project Description (including summary of technical work)**

1) **ELECTRICAL CONDUCTIVITY MODEL DEVELOPMENT** - Candidate mathematical model frameworks for predicting electrical conductivity, with an underpinning thermodynamic basis, will be developed, implemented within NPL’s MTDATA software and tested based upon calculations for systems containing just a few elements. Most promising candidate(s) will be developed further to demonstrate general applicability. A final choice will then be made (either a single mathematical framework or high and low temperature versions if necessary) for full parameterisation. A scientific paper describing the model and comparing calculation and experimental results will be written and submitted to a peer reviewed, Web of Science indexed, journal. It is envisaged that the predicted electrical conductivity values will be based upon contributions from various oxide species (single, double and triple oxides) weighted in some way by their equilibrium amounts calculated using existing thermodynamic models. Defining the precise method of combination and the way in which this might change with temperature will be the focus of the model development task. This work will be mainly Government funded.

2) **ELECTRICAL CONDUCTIVITY MODEL PARAMETERISATION** - Parameters will be developed to allow the final predictive model to cover as wide a range of composition and temperature as possible, determined in consultation with the industrial partners. This work will involve validation against literature data and measurements supplied by industrial partners. Some of the validation will be done as in-kind work by the partners, using parameter sets as they are developed. The final full set of parameters will be made available in the form of a database, validated and tested for quality at NPL and by industrial partners, for use in predictive MTDATA calculations. This work will be mainly industrially funded.
3) LEARNING LESSONS FROM VISCOSITY MODELLING - The development of the electrical conductivity model will be guided by earlier work on developing a viscosity model. Industrial users of the viscosity model have suggested changes to improve the predicted variation of viscosity with temperature which it is planned will be implemented and tested, through extension of the database, in the current project. Lessons learnt will aid in the modelling of electrical conductivity. This work will be mainly industrially funded and its inclusion will help in achieving the required level of cash co-funding. Parameters developed will be made available in the form of a database, appropriate to the use of a new method for calculating temperature dependence, validated and tested for quality at NPL and by industrial partners, for use in predictive MTDATA calculations.

Impact and Benefits - Uptake of the predictive capability produced by this project can be directly measured in terms of the number of companies licensing the databases produced. Impact on the economy and quality of life will be through reduction in energy usage and CO₂ emissions in industrial processes and the development of new materials such as specialty glasses for use in photovoltaic panels. The benefits will be difficult to quantify as companies will generally treat such advances as “confidential”. The NMI’s measurement capability is enhanced in terms of the development of a model to get best value from the limited measurements it is economic to make and to effectively target future measurements.

Support for Programme Challenge, Roadmaps, Government Strategies - The project uses models to characterise materials and supports low-carbon technologies in the areas of energy usage and high value manufacture, identified as key areas of science in the NPL strategy, which supports Government strategy. It is concerned with the prediction of a macroscopic materials property based upon atomic scale properties. Work of this type, spanning length and time scales, is targeted in the Thermodynamics TAR roadmap.

Synergies with other projects / programmes - The project has synergy with ongoing 3rd party projects to develop the NPL phase equilibrium calculation software and to develop the NPL thermodynamic database for oxide and sulphide systems. It will use outputs from these projects and its success will add to the attractiveness of these projects to future sponsors from industry. The addition of an electrical conductivity modelling capability is a part of a drive to supplement thermodynamic property prediction with thermophysical property prediction.

Risks: The main risk is failure to develop a suitable model framework. Even if that is the case it is valuable knowledge and a scientific paper to that end could result. The risk is low given that the NPL staff involved have considerable experience modelling for liquid oxide phase equilibria and viscosity and a variety of experienced industrial partners will be available to offer advice.

Knowledge Transfer and Exploitation - The mathematical framework of the model used will be disseminated to the partners thorough discussions at progress meetings and to a wider audience through a scientific paper written and submitted to a peer reviewed, Web of Science indexed, journal such as Calphad. The model parameters will be disseminated to partners (5 or 6 companies) through databases enabling them to make predictive calculations (when coupled with MTDATA) relevant to their own particular processes. These calculations will typically underpin process refinements to reduce energy needs, to cope with new raw material compositions or new product specifications or to develop innovative products and may result in further, more application specific, scientific publications from partners. Non partners will be able to lease the databases to perform their own predictive calculations after a lead time to acknowledge the cash input of the companies supporting the proposed project. Take up will be mainly in Metals and Minerals Processing industries but benefits will be more widespread as more efficient processing means environmental protection through better use of raw materials, savings in energy and CO₂ emissions, and reduction in waste. Impact in Energy and Environment will also, therefore, occur. Uptake from secondary metal producers in the UK, glass-makers and companies concerned with the development of new battery and energy storage technologies is also possible.

Co-funding and Collaborators - Proposed cash contribution of £10,000 per partner spread over two years. Details available on request.

Deliverables

1 Scientific paper describing model and model development

Start: Jan 2012
End: Apr 2013
Cost: £

2 Thermodynamic database to underpin electrical conductivity calculations

Start: Jan 2012
End: Dec 2013
Cost: £

3 Thermodynamic database to underpin viscosity calculations (New Model)

Start: Jan 2012
End: Dec 2013
Cost: £
Summary - This project launches the Materials Division in a new underpinning activity to develop and integrate measurement and modelling tools to characterise and generate fundamental understanding of mechanical properties and material characteristics at the sub-grain level. This bottom-up approach, focussed on 3D structural, compositional and mechanical characterisation, coupled with mathematical modelling and macroscopic property and performance measurement in complementary projects will provide the springboard for the development of materials with engineered properties and for prediction of material performance in service from fundamental principles.

The Need - The major drivers in our society include (though not exclusively) energy security, sustainability, reduced impact on environment, improved quality of life, competitive manufacturing and job creation, potable water availability, and information technology/communications. Underpinning the future requirements of all of these is the need to develop novel materials with tailored properties, to make radical advances in materials processing, to optimise manufacturing and engineering through knowledge-based design, and to establish innovative tools for predicting and characterising performance. Characterisation of processes at an increasingly localised scale is recognised as the basis for predicting, rationalising and manipulating macroscopic behaviour. Specifically, 3D sub-grain microstructural characterisation coupled with local mechanical property measurement and supported by multiscale modelling is required. Current capability for microstructural characterisation at NPL and elsewhere is largely based on 2D techniques. There is evolving work on 3D FIB/SEM at a small number of UK universities such as Manchester and Loughborough and centres for microscale X-ray tomography at Manchester and Southampton. Nanoscale mechanical property measurement can be made with nanoindentation and AFM systems but there has also been work on the use of FIB to produce localised small scale pillar and cantilever samples (Steve Roberts, Oxford) for mechanical property measurement. However, these varied studies are at an academic research level and need to be developed on a sound metrological basis with the focus on application to industry.

The Solution - Adopting a progressive long-term strategic approach, measurement and modelling tools will be established to enable understanding of material behaviour at length scales smaller than the grain size with a capability for integrating information to prediction of macro-scale properties and performance. Specifically, 3D characterisation of material microstructure, microchemistry, strain and stress distribution, and mechanical properties will be demonstrated and linked to modelling of crystal plasticity to provide the necessary physical insight. The information generated at this local level will then be correlated with macroscopic measurements undertaken in satellite projects; for example, on wear and environment induced cracking.

Project Description - Phase I, for which funding is sought in this round, is geared primarily to establishing the efficacy and reliability of using focused ion beam machining combined with scanning electron microscopy - energy dispersive X-ray spectroscopy and electron back-scattered diffraction to characterise the near-surface microstructure and microchemistry in three dimensions and its dependence on material history. Such 3D characterisation is still in it embryonic stage and a key output will be a good practice guide on the effective utilisation of the approach and potential uncertainties in measurement. Particular attention will be given to issues such as the accuracy and resolution of the processes used to determine the 3D information, control of ion beam damage to samples, and the integration of information from the multitude of analysis and imaging sources that are available. Complementing this activity, established nano-mechanical techniques will be used to determine mechanical properties on a sub-grain and grain to grain basis by high resolution mapping. Some work will also be carried out to explore the feasibility of making mechanical property measurements in-situ in the dual beam instrument using the ion beam machining of nanoscale compression pillars or cantilever beams, or in-situ nanoindentation of specific areas revealed during ion machining. This will enable direct correlation of mechanical properties with microstructure. Furthermore, sub-grain measurements of residual stress is being developed in a separate satellite project and the methodology will be applied to evaluate local variations in residual stress for direct correlation with other property data. Two systems will be studied with these techniques. In the first instance, WC/Co hardmetals will be investigated, for which there is substantial property measurement data available at NPL. The second system will be a corrosion resistant alloy commonly used in the oil and gas industry, the stress corrosion cracking resistance of which is being investigated in a parallel project. Crystal plasticity modelling of the strain distribution in single grains and in multiple grains will be initiated in order to provide the fundamental physical insight necessary to rationalise the measurement data, while at the same time the latter provides a test of predictions.

An important element of this work will be to ensure that the constraint that comes from small size of distinct phases in the materials is taken into account. This modelling work will be undertaken in collaboration with Imperial College through a post-doctoral fellowship with a view to developing an in-house capability in the long term.
Impact and Benefits

- Knowledge-based design of materials microstructure to give best and predictable long-term performance;
- Increased lifetime of components and structure and more efficient deployment of materials in the mining, mineral extraction, manufacturing, automobile, aerospace, rail, and power generation sectors;
- The project will provide UK industry with an integrated approach to prediction of materials performance based on a sound foundation of microstructural characterisation and mechanical property evaluation with sub-grain resolution. This will be aimed at supporting materials and component development and durability, extending in-service performance, and contributing to the validation of finite element models, structural integrity codes and material performance models.

This project will place NPL at the leading edge of international research in the area of materials characterisation, and long-term prediction of materials performance.

Support for Programme Challenge, Roadmaps, Government Strategies

This project fits with the NMO and NPL strategic aims for measurements and modelling to characterise atoms, molecules, bio-systems and materials, and science to support sustainable technologies in the areas of energy generation and usage, and high value manufacture, including life assessment. It also aligns with the TSB strategies for advanced materials and high value manufacturing.

Synergies with other projects / programmes

The project:
- Builds on projects A20 In-situ techniques for measuring stress and strain, AM09 Low friction coatings, P18 Traceable Microstructural Measurements, and T11 Monitoring the degradation of surfaces subject to wear
- Supports project on stress corrosion cracking of corrosion resistant alloys (P13)
- Underpins proposed Materials 2011 short crack growth project
- Links to proposed Materials 2011 micro-relaxation techniques for residual stress project

This project has a 6 to 9 year vision, so the work described in this proposal has detailed deliverables for the first three years only. In future work, increasing engagement with industrial partners is envisaged as the evolving capability is progressively shifted from the development phase to extended application.

Risks

The main risk is associated with significant technical challenges in achieving traceable 3D nanoscale characterisation of materials with dual beam FIB/SEM technology including registration of information, 3D reconstruction, and integration of the different sources of information in the instrument. There are also risks with the in-situ mechanical testing and modelling aspects of the project such as the derivation of mechanical property information to describe the behaviour of specific phases from simple tests with the high spatial resolution that will be required. All these risks will be mitigated by working in collaboration with established experts in the different field to ensure success.

Knowledge Transfer and Exploitation

A key output will be a draft Guide to Good Practice in 3D Characterisation of Materials Microstructure and Microchemistry developed in collaboration with leading researchers in the field. Application of the methodology will be highlighted in 6 scientific papers and 2 conference papers. Industrial sectors targeted will be mineral extraction, power generation, aerospace, materials supply and oil and gas sectors.

Co-funding and Collaborators

Details available on request.

Deliverables

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<tr>
<td>1</td>
<td>FIB - 3D characterisation of material chemistry, and structure. Good Practice Guide</td>
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<td>Correlating mechanical properties and material characteristics at the sub-grain scale with application to rationalising the performance of hardmetals and corrosion resistant alloys - scientific paper</td>
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<td>3</td>
<td>Report reviewing current capabilities and metrology</td>
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To realize the benefits and optimise the performance of micro and nano-structured materials and thin films, designers need to understand and thus be able to characterise their thermal, thermophysical and thermomechanical properties. For thermoelectric devices, for example, breakthroughs are required in the understanding of transport properties at small scales to stimulate innovation and development of efficient viable devices.

This project will establish validated, quantitative methods and procedures for the measurement of thermophysical properties of micro and nano-scale structured materials and products (e.g. thin films), focusing on the thermal transport properties (thermal diffusivity/conductivity).

The methods and procedures to be developed will be based on the use of thermally active AFM based probes.

The Need
To realize the benefits and optimise the performance of micro and nano-structured materials, thin films and products, designers need to understand and thus be able to characterise their heat transfer, thermophysical and thermomechanical properties and behaviour. However, existing established techniques are not suited for micro-scale measurements. Specifically for thermoelectric devices, breakthroughs are required in the understanding of transport properties at small scales in order to stimulate innovation and development of efficient viable devices. The need is therefore to provide a measurement platform to characterise reliably the thermal properties of materials on very small scales. Without such metrological capability, innovation will be hindered. For thermal properties measurement on the small scale, scanning thermal microscopy contact techniques based on the AFM have been developed, with some leading research done by the University of East Anglia. However, the approaches adopted vary considerably, and the techniques are largely qualitative. In particular, they do not provide reliable quantitative information on heat transfer properties. The main approach has been a qualitative measure of conductivity based on the heat loss from the probe on contact with the specimen. A nano-thermal probe facility has been established at NPL and qualitative thermophysical properties measurements have been made on composite and polymeric materials. As an early step in the development of the quantitative methods, modelling of the heat loss associated with the probe tip – specimen interaction has been carried out, highlighting potential limitations in the technique and also, potentially, in the modelling: both require further elucidation. Methods for temperature calibration of the nano-thermal probes using materials with known thermal transitions are currently being explored, and measurements of the glass transition temperature of polymeric materials have been made. However, there is need to improve on the quantitative aspects of the use of AFM based thermal properties measurements in order to provide meaningful data and confidence in their use, in particular for heat transfer properties.

The Solution
This project will contribute to establishing validated, quantitative methods and procedures for the measurement of thermophysical properties of micro and nano-scale structured materials and products (e.g. thin films) at those length scales. Specifically, it will focus on the thermal transport properties of thermal diffusivity/conductivity. The project will develop an AFM-based thermal probe facility for the measurement of the thermal properties of materials on the micro and nano-scales (at 100 nm resolution). The methods and procedures developed will be targeted at quantitative measurements of materials for thermoelectric technologies, organic electronics and micro-scale formed polymers, in order to support research and development and materials qualification needs. The techniques will be based on the use of thermally active probes mounted on an AFM instrument. The probe tip is used to either heat the sample locally (~100 nm for the AFM tip), thereby inducing thermal gradients and thus heat flux within the sample, or measure temperatures locally with heating provided independently. Both modes of operation provide opportunities for determining the heat transfer properties of the sample and will be investigated. Because of the very localised heating capability of the probe, variations in the thermal properties of the material on the micro and nano-scales (either its engineered structure, e.g. of very thin multi-laminate films, or the material’s microstructure, e.g. of blended materials) can be investigated.

The project will develop a validated AFM-based thermal probe measurement capability for thermophysical properties. It will encompass:

- Calibration and validation methods for temperature measurement using thermally active AFM probes.
- Calibration and validation of techniques for heat transfer properties measurement at the microscale (e.g. thermal diffusivity,
conducitivity).
- Industrially focused case studies on the use and benefits of the microscale thermal properties measurement facility.
- Good practice guidance for AFM-based thermophysical properties measurements.
- Peer reviewed publication on thermal properties measurement on the micro/nano-scale.

This project builds on the current project 1018, which is focusing on the use of thermally active AFM probes and their temperature calibration.

**Impact and Benefits**
This project, through a reliable ability to characterisation multiphase and micro-structured material on micro and nano-length scales, will further the understanding and enable the development of new and improved materials and products, in particular products of high added value (electronic devices). The capability will assist industry and R&D through, for example;
- the detection of hot spots in organic electronic devices (leading to improved design, thermal management, and improved reliability),
- evaluation of polymers used in photovoltaics (FP7 Sealsol proposal)
- identification of inhomogeneity in multiphase materials and micro-structured materials and products,
- and more specifically via the EMRP on thermoelectrics, enabling the design of improved solid-state thermoelectric materials for use in vehicles to increase mileage by up to 10% by the conversion of exhaust heat to electric power thereby reducing the output from and weight of alternators.

The project supports NPL’s strategy on nano-scale materials metrology: it will provide infrastructural measurement capability and will address the scientific questions of the effect of reduced length scale on properties of polymers that are, for example, highly relevant to performance issues in polymer thin film applications.

**Support for Programme Challenge, Roadmaps, Government Strategies**
This project supports the Energy, Advanced Materials and Underpinning Metrology challenges. The TSB strategy identified “High Value Manufacturing” and “Electronics, Photonics and Electrical Systems” as key technology areas. This work supports many of the areas identified therein, e.g. polymer electronics, where the device temperature is a limiting factor and improvements in cooling directly affect performance. It supports the Characterisation and Performance theme roadmap.

**Synergies with other projects / programmes**
The project builds on NPL’s world-class thermal properties activity, and on recent work on the heat transfer properties of polymers by transient methods. The project team is multi-disciplinary covering the Thermal, Nanomaterials, Temperature and Materials Groups at NPL. This project is aligned with the EMRP project “Metrology for Energy Harvesting” enabling access to a €3.2m project, and the EMRP project on nano-indentation of polymeric materials (2010 Industry call).

**Risks**
- Failure of identified techniques to provide sufficiently robust and accurate data: exploratory work on the use of AFM already carried out indicates that the various modes of operation of the AFM provide options for measurement route, thus enhancing probability of success.
- Insufficient instrument transparency to enable full understanding of its operation (re Wheatstone bridge): source independent manufacture of component.
- Project risk reduced as the major items of equipment required are already at NPL.

**Knowledge Transfer and Exploitation**
- Peer reviewed paper on calibration, validation and best practice
- Presentations at conferences, external and NPL Industrial Advisory Group meetings
- Materials World article on thermal analysis
- Provision of measurement services
- This project will further develop and validate micro and nano-scale metrology capability for thermal analysis, leading to the publication of future Good Practice Guides and Standards.

**Co-funding and Collaborators**
Co-funding will be sought from TSB (to engage with end users), EMRP calls (to engage with other NMIs) and FP7. Collaboration being established with Czech Republic NMI. This project will be carried out in parallel with, and provide underpinning metrology input to the EMRP Energy call project “Metrology for Energy Harvesting”. Details available on request.

**Deliverables**
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<th>Start: Jan 2012</th>
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<td>Methods for measurement of thermal properties at the microscale</td>
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Summary - This project will develop accurate methods and models for measuring and predicting levels of residual stress and strain in conventional composites, and metrology for characterising the physical, thermal and mechanical properties of composites containing auxetic constituents (fibres/fillers and matrix). The outputs from this project are expected to assist manufacturers and end-users to accommodate and/or reduce process-induced residual stresses that can have a detrimental effect on the performance of polymer matrix composite structures. Optical fibre Bragg grating (FBG) and incremental slitting methods are to be developed for characterising the residual stress and strain distributions within laminated structures and compared with predictive analysis (i.e. analytical and FEA) and results obtained from other techniques including the layer removal (curvature) method. The techniques will be used to verify residual stress reduction in auxetic laminates (to be supplied by industry) and to assess the accuracy and relevance of micromechanics models for predicting auxetic material properties.

The Need - A limitation of fibre-reinforced composites is that they can have high levels of residual stresses caused by cross-linking of the polymer matrix during curing and by differential thermal expansion between the fibre and matrix. Due to difficulties in quantifying residual stresses they are often ignored, which can be dangerous as residual stresses can significantly reduce the life expectancy of a structure; increase the likelihood of component distortion and in-service failure. If the magnitude of the residual stress is unknown, higher safety factors must be used which leads to overdesign of structures and increased weight. To date, the most widely used technique for measurement of residual stress in composites is the layer removal (curvature) technique. However, it can only be used to predict the macro-residual stresses within the composite and cannot cope with discontinuities at ply interfaces. Other methods that have been investigated include; incremental slitting, FBGs, chemical probe, photoelasticity, Raman spectroscopy and X-ray analysis. However, most of these techniques are of limited use. Incremental slitting is a promising method, but requires further development of both the experimental technique and modelling approach used to interpret measured strains. Optical FBG sensors can be embedded within the composite, with the advantages that the technique is non-destructive and can be used for on-line and in-service monitoring of residual strains. However, the experimental application, data analysis and associated modelling approaches need to be developed. The use of auxetic materials for residual stress reduction is state of the art and is several years from widespread utilisation. NPL recently worked with Global Composites Group to develop the first ever test to demonstrate that auxetic materials can be used in practice to balance carbon fibre laminates. NPL has the measurement, modelling and exploitation route expertise for developing residual stress measurement techniques and characterising auxetic materials, and thus is ideally placed to undertake this work.

The Solution - This project will develop incremental slitting and optical FBG methods for measurement of residual stresses in laminated composites. As well as experimental techniques, associated modelling approaches (analytical and FEA based), which aid the interpretation of measured strains, will be developed. The modelling approaches will be based on laminate analysis, inverse analytical methods and the use of high precision moulding (HPM) LUSAS FEA software. Optical FBGs will be embedded within laminates enabling on-line, real time measurement of residual strains/stresses during processing and in-service (e.g. cradle-to-grave) approach. Measurements will be compared with predictive analysis and results obtained from other techniques including the layer removal (curvature) method. Carbon and glass fibre-reinforced thermoset laminates (120 and 180°C cure systems), of various lay-ups, will be studied with varying degrees of process induced residual stress. The characterisation of auxetic materials will focus on thermoset composites reinforced with auxetic polypropylene fibres and, if available, those produced using auxetic matrix systems. The stress measurement techniques developed within the project will be used to verify residual stress reduction in auxetic laminates and to assess the accuracy and relevance of micromechanics models for predicting auxetic material properties.

Impact and Benefits - There are several benefits that the UK composites community will gain on completion of this project. The...
The main benefit is that a reduction in design conservatism in composite components is expected to be achieved (within 1-2 years of project completion) via provision of residual stress measurement and modelling techniques enabling more accurate measurement of residual stresses. Safety factors will be able to be reduced through more accurate characterisation of conventional and auxetic composites. Reductions in design conservatism will enable the use of lighter components through use of less material, with the knock-on advantage that fuel reductions for aerospace and surface transport applications will be realised. By developing the capability to characterise and model innovative auxetic materials, the opportunity to dilute high cost carbon fibre composites selectively with low cost extruded auxetic fibres will be achievable. This is expected to occur 3-5 years after project completion. It is estimated that a 10-30% reduction in wind turbine blade material costs is achievable through the use of hybridised composites containing up to 60% low energy, low cost, and locally produced auxetic fibre replacing high cost, high energy and imported carbon fibre. In addition, the use of auxetic materials will mean that tooling and component distortion will be reduced, components will be able to operate at higher temperatures, have improved damage tolerance and be of lower cost. This project will develop NPL’s capability in characterising; modelling and reducing residual stresses thereby promoting more sustainable composite components. The anticipated impact of these new capabilities will be evidenced by scientific paper citations, Good Practice Guide downloads and an increase in measurement service work in this area.

**Support for Programme Challenge, Roadmaps, Government Strategies**

This project aligns directly with the Composites TAR roadmap in that it is designed to address the market drivers of increasing the use of sustainable and multifunctional composite materials. It also aligns with the following:

(i) Programme Challenges: Energy, Transport and Environmental Sustainability: enhanced design, safety and reliability, and light weighting of structures for energy and transport infrastructure.


**Synergies with other projects / programmes** - The project builds on projects SM09 “Improving Quality in Composite Materials Through Provision of Traceable Measurements”, SM07 “Validated Measurement Techniques for Improved Design of Large Advanced Composite Structures Subjected to Multi-Axial Loading”, SM10 “Characterising Interfaces in Continuous and Dispersed Materials” and CU03 “Improving the Reliability of Materials Modelling”. These projects either reviewed or assessed residual stress measurement techniques and modelling approaches. The project outputs highlighted which stress measurement techniques offered most potential but did develop the methods significantly for use with continuous fibre-reinforced composite systems. Synergy also with MAT1024 “Development of NDE and SHM Techniques for Monitoring Defects And Chemical Changes in Composite Structures”. Beyond the current project, it is planned to progress standardisation of draft procedures for residual stress measurement.

**Risks** - The main technical risk is that the development of candidate residual stress measurement techniques and modelling proves too complex. This risk is mitigated by choosing two very different techniques at the onset of the project. There is also a risk that key modelling staff may not be available at NPL and this will be mitigated by partnering with LUSAS FEA Ltd. In-kind supply of suitable quality and quantity of auxetic material by Bolton University and Global Composites Group is a risk and will be mitigated by allowing sufficient funds to purchase material and identify other potential suppliers.

**Knowledge Transfer and Exploitation** - Principally through scientific papers (journals, conferences and trade articles), Measurement Good Practice Guide and draft procedures for future standardisation aimed at suppliers and end-users/designers of composite materials in multiple sectors (energy, aerospace, automotive, renewable energy, marine and construction). IAG, Materials KTN plus measurement services and consultancies are other alternative routes for dissemination and exploitation.

**Co-funding and Collaborators** –

Details available on request.

**Deliverables**

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<td>1</td>
<td>Start: Dec 2012</td>
<td>End: June 2014</td>
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**Development and Validation of Techniques for Measurement of Residual Stress**

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<th>Start: Jan 2014</th>
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**Characterisation and prediction of mechanical, physical and thermal performance of auxetic laminates**

**Formulation of a residual stress measurement toolkit**
The major drivers in society are the need for energy security through a sustainable manner with reduced environmental impact. A number of the large pulverised coal-fired power plant boilers currently operating in the UK are due to close in the near future after being opted out of the Large Combustion Plant Directive (LCPD), clearly affecting the security of energy supply. This loss in generating capacity leaves the UK facing an energy shortage whilst new nuclear options are investigated and built. There is, however, an opportunity to keep them operational by changing from fossil fuel to biomass firing. This would involve firing with higher levels of biomass, up to 100%, but at higher temperatures than conventionally used in biomass plant, since the existing boilers operate with final steam temperatures significantly above those employed for current biomass fired plant. It is anticipated that these harsh conditions will accelerate the rate of metal wastage and additional corrosion mitigation strategies will be required, such as coatings. However, industry is cautious due to lack of information as to the degradation rate of alloys in these complex atmospheres. Firing with biomass generates a very complex combustion atmosphere (consisting of alkali salts and raised levels of K, Cl and S) which would be further complicated with changing fuel diet, there is a need therefore for better understanding of the degradation processes of conventional and candidate materials under these increasingly complex and harsh conditions to ensure the reliability, maintainability and safety of these systems. Currently there are no controlled facilities in the UK to perform long-term tests of this type. NPL and Cranfield University are leading UK researchers that conduct research of this kind [1-3]; each offers distinct skills and expertise that compliment each other and as such often collaborate on grant-funded projects. Pilot plant and isothermal lab exposures are current state-of-the-art but do replicate plant atmosphere in a controlled, sustained and dynamic manner. The new capability developed in this project will advance laboratory testing to address dynamic changes in boilers and provide quantitative metrology of material degradation in these conditions.

1 “Metal wastage in low alloy ferritic steels exposed to multi-component gaseous atmospheres: data representation and predictive modelling”, Osgerby, Fry and Gohil, Materials at High Temperature.

Improved understanding of the combustion atmosphere and the effect on the degradation rate would alleviate some of the industrial concerns and provide greater confidence on the engineering solutions pursued. Thus the degradation rate and mechanism for conventional and candidate materials and coatings require characterisation under controlled complex combustion atmospheres and fluctuating atmospheres representative of proposed biomass fuels. Metrology of the complex layers of internal corrosion, external alkali layers and ash deposits will provide mechanistic information and corrosion rates. Previous work has shown the importance in understanding the impact of fluctuating conditions and solutions to do this in a controlled manner are required [1].

<table>
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<tr>
<th>Project No.</th>
<th>Project Title</th>
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<td>MAT\2011\27</td>
<td>Metrology of biomass derived fireside corrosion</td>
<td>£354k</td>
<td></td>
</tr>
</tbody>
</table>

**The Need**

**Summary**

This project will provide a mechanism to generate medium to long term corrosion data of alloys and coatings under constant and fluctuating biomass combustion atmospheres which are representative of those proposed for 100% biomass fired retro-fitted plant. Quick, easy and robust methods of specimen analysis will also be developed to increase the speed and information obtainable from metallographic sections. This will enable researchers to obtain much more statistically reliable data from complex environmental corrosion data, which by its very nature is difficult to analyse due to statistical variations. Such data will provide confidence to industry in the behaviour of materials in novel environments.

**Lead Scientist**

Tony Fry

**Scientist Team**

Jim Banks, David Laing, Dipak Gohil, John Nunn, Maud Seraffon

**Sector**

Energy

**Activity**

Development of Existing Capabilities

**Project Champion**

Peter Morris

**The Solution**

**Project Description (including summary of technical work)**

To ensure that this metrology is relevant and provides a true representation of plant degradation a new low velocity burner rig will be constructed which will enable multiple samples to be exposed to the controlled combustion atmosphere which will be computer controlled and capable of performing fluctuations representative of changing fuel diet. A series of coated and uncoated materials will be exposed in this rig under constant environmental conditions and fluctuating conditions to simulate the steady state firing and mixed fuel firing. In addition procedures for the optical examination of microstructural cross-sections for the precise measurement and characterisation of these complex multilayered scales will be developed enabling the rapid characterisation of uncoated and coated corrosion specimens. These data will be used to extend the database constructed as
part of a recent TSB funded project on fireside corrosion. Neural network models developed in this project will be modified to incorporate this extension in the data envelope. The reliability and adhesion of candidate coatings and scales will be evaluated using miniaturised test methods and microscopy, which, it is envisaged, will make use of the facilities and expertise generated in the Grains to Structures project as well as those in the EMRP Energy project. Coatings will be exposed to different atmospheres for different durations and the degradation of properties mapped, both in terms of through thickness and also as a corrosion map in terms of environment and time.

Impact and Benefits
A number of the major utility companies worldwide are in the process of considering the conversion of existing coal boilers to 100% biomass firing or high level biomass co-firing, and are concerned about the risks of excessive corrosion rates of boiler components. A method to accurately measure this rate using representative simulated atmospheres would address these concerns, and also add to the portfolio of measurement solution NPL can provide to the energy sector. British companies would then be in a strong position to win a number of the plant conversion contracts, and the ability to provide advanced solutions to the corrosion problems will have a positive impact on their competitiveness and help to maintain the energy generating capacity for the UK. The total capital expenditure on each of these fuel switching projects is of the order of £20-100 million, depending on the scale of operation. It is considered that the export potential in this sector is also significant; the overall size of this market may be significantly in excess of £500 million over the next few years. The results of the proposed project will help support the investment decisions required in order to implement the anticipated increased use of biomass as a fuel in existing pulverised fuel power stations. This will provide investment opportunities for plant operators, and plant modification and improvement work for equipment suppliers, whilst securing employment in existing power plant that would be otherwise be closed. The increasing large-scale use of biomass and the displacement of coal as a fuel source have obvious benefits in terms of reducing the CO2 emissions from burning fossil fuels.

Support for Programme Challenge, Roadmaps, Government Strategies
This project fits with the NMO and NPL strategic aims for characterisation of materials and their performance and is integral to the Surface Integrity Theme roadmap. This project clearly links and supports current policy for reduced carbon emissions and increased use of renewable energy. It also aligns well with TSB strategies for energy materials as well as European strategies on energy supply and reducing CO2 emissions.

Synergies with other projects / programmes
This projects builds on the work conducted on the degradation of materials used in power generation, and links with current activities both for third party customers and for grant funded projects such as MACPLUS (FP7) and TSB projects completed in the last year and future proposals addressing fireside corrosion under biomass combustion. The coating aspects also are directly relevant to EMRP Energy projects and the Grains to Structures project. This work also links to the UKUS collaborative works programmes and areas being funded by the US DOE and EPRI.

Risks
The main risk with this project is the ability to control the gaseous atmosphere within the correct limits such that the test is representative of plant conditions. Close links with industrial partners will help to tailor the atmosphere and interaction with other groups within NPL will help to resolve any issues. Otherwise as a fall back position premixed gas mixtures could be used. The extraction of corrosive gaseous also needs to be controlled to avoid damage to the buildings extract. Gas clean up can be used and the facility is likely to be housed in its own building (B24). There is also a risk that the dual-beam facility is not available when required, a fall back position of working with Loughborough University through the SuperGen II programme is available.

Knowledge Transfer and Exploitation
The outputs of this project will be used in the formulation of ISO standards as part of the activities of ISO TC 156. Energy utilities and OEM’s would be the target sector and they would use the data to guide design choices and could use the facility developed in third party exposure work and other grant funded activities. Dissemination would be through two focussed journal papers in journals such as Corrosion Science and Energy Materials, and through 2 key international conference presentations. The use of Biomass is increasing in popularity and decreasing the amount of co-fired material is attractive. Hence the take up of these results is likely to be by all the UK utilities and OEM’s and also internationally through initiatives such as COST and the UK-US collaboration.

Co-funding and Collaborators
Details available on request.

Deliverables

<table>
<thead>
<tr>
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<th>Start:</th>
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<tr>
<td>Procedure for the exposure of test coupons to biomass based fuels using a modified burner rig.</td>
<td></td>
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<td>2</td>
<td>May 2012</td>
<td>June 2014</td>
<td>£</td>
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</table>
The effect on the degradation rate would alleviate some of the issues that would involve firing with mixed fuel firing. These data will be used to extend the database constructed as part of a recent TSB funded project on fireside corrosion. Neural network models developed in this project will be modified to incorporate this extension.

To ensure that this metrology is relevant and provides a true representation of plant degradation a new low velocity burner rig will be required, such as coatings. However, industry is cautious due to lack of information as to the degradation rate and mechanism for conventional and candidate materials and coatings require changing fuel diet, there is a need therefore for better understanding of the degradation processes of conventional and candidate materials under these increasingly complex and harsh conditions to ensure the reliability, maintainability and safety of these systems. Currently there are no controlled facilities in the UK to perform long-term tests of this type. NPL and Cranfield University are leading UK researchers that conduct research of this kind [1-3]; each offers distinct skills and expertise that compliment each other and as such often collaborate on grant-funded projects. Pilot plant and isothermal lab exposures are current state-of-the-art but do replicate plant atmosphere in a controlled, sustained and dynamic manner. The new capability developed in this project will advance laboratory testing to address dynamic changes in boilers and provide quantitative metrology of material degradation in these conditions.

Summary

This project will provide a mechanism to generate medium to long term corrosion data of alloys and coatings under constant and fluctuating biomass combustion atmospheres which are representative of those proposed for 100% biomass fired retro-fitted plant. Such data will provide confidence to industry in the behaviour of materials in novel environments.

The Need

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Impact and Benefits

A number of the major utility companies worldwide are in the process of considering the conversion of existing coal boilers to...
100% biomass firing or high level biomass co-firing, and are concerned about the risks of excessive corrosion rates of boiler components. A method to accurately measure this rate using representative simulated atmospheres would address these concerns, and also add to the portfolio of measurement solution NPL can provide to the energy sector. British companies would then be in a strong position to win a number of the plant conversion contracts, and the ability to provide advanced solutions to the corrosion problems will have a positive impact on their competitiveness and help to maintain the energy generating capacity for the UK. The total capital expenditure on each of these fuel switching projects is of the order of £20-100 million, depending on the scale of operation. It is considered that the export potential in this sector is also significant; the overall size of this market may be significantly in excess of £500 million over the next few years. The results of the proposed project will help support the investment decisions required in order to implement the anticipated increased use of biomass as a fuel in existing pulverised fuel power stations. This will provide investment opportunities for plant operators, and plant modification and improvement work for equipment suppliers, whilst securing employment in existing power plant that would be otherwise be closed. The increasing large-scale use of biomass and the displacement of coal as a fuel source have obvious benefits in terms of reducing the CO2 emissions from burning fossil fuels.

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Synergies with other projects / programmes
This projects builds on the work conducted on the degradation of materials used in power generation, and links with current activities both for third party customers and for grant funded projects such as MACPLUS (FP7) and TSB projects completed in the last year. The coating aspects also are directly relevant to EMRP Energy projects and the Grains to Structures project.

Risks
The main risk with this project is the ability to control the gaseous atmosphere within the correct limits such that the test is representative of plant conditions. Close links with industrial partners will help to tailor the atmosphere and interaction with other groups within NPL will help to resolve any issues. Otherwise as a fall back position premixed gas mixtures could be used. The extraction of corrosive gaseous also needs to be controlled to avoid damage to the buildings extract. Gas clean up can be used and the facility is likely to be housed in its own building (B24).

Knowledge Transfer and Exploitation
The outputs of this project will be used by partners in the project and then made available to the wider energy utility and supply chain community through joint publications. Findings will support the formulation of ISO standards as part of the activities of ISO TC 156. The use of Biomass is increasing in popularity and decreasing the amount of co-fired material is attractive. Hence the take up of these results is likely to be by all the UK utilities and OEM’s and also internationally through initiatives such as COST and the UK-US collaboration.

Co-funding and Collaborators
Details available on request.

Deliverables

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<td>1</td>
<td></td>
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Summary:
This project co-funds the FP7 project Piezo Institute II a follow on from an existing supporting action that supported the establishment of the Pan European network for piezoelectric functional materials. The Piezo Institute (PI) is Europe’s centre of expertise within the field of piezoelectric multifunctional materials. The properties of these materials and recently developed technologies are enabling both miniaturisation and integration of key electronic components and the establishment of completely novel applications such as energy scavenging. The Piezo Institute acts as a focus within Europe for the gathering and dissemination of scientific research in this area.

The Need:
Piezoelectric functional materials and the technologies they enable are a rapidly growing in applications and installed devices. Europe and the UK have strong record in academic research and a growing community of companies developing and employing these materials. However, before the establishment of the Piezo institute sharing of knowledge and capabilities across Europe was fragmented and inadequate to support the promise of these new materials to nurture economic growth through the establishment of new industries. The Piezo Institute was established as a response to this need with start-up funding and an initial programme of knowledge transfer activities. The second stage of this activity is needed to maintain and build on the first stage success of the Piezo Institute.

The Solution
The project aims to establish The Piezo Institute as a European Centre for the development and harmonisation of the existing European know-how on piezoelectric materials. The application of these materials will have a strategic impact on European industry and scientific society by enhancing competitive advantage of Europe in advanced manufacturing The strategic impact of the proposed project will reinforce economic growth and sustainability of industry and society.

Through the results of this project, it will be possible to achieve world leadership in academic know-how and industrial production, to increase the level of the know-how on materials and devices and to start with new intelligent products or to increase quality and performance of existing ones. The perpetuation of an Institute capable of collecting and coordinating the wide (but fragmented) European know-how on piezoelectric materials, devices and applications is critical in achieving this solution.

Furthermore strengthening the already wide and diffused European studies on lead-free piezoelectric materials will give European industries an important advantage and leadership in the fabrication and use of green devices

Project Description (including summary of technical work):
During the next three years The Piezo Institute will build its network of contacts, establishing collaboration with additional Technology Platforms, both on materials and devices as well as the applications side. The Piezo Institute will further extend its education degree organisation, by postdoc positions exploiting opportunities within e.g. Erasmus and Erasmus Mundus programmes.

In order to increase its visibility and to facilitate knowledge of new technologies and opportunities for European industry, the Piezo Institute will add new industrial courses to its offering and intensify its presence at other professional conferences, giving presentations as The Piezo Institute and not as the individual members. Whenever possible it will also carry out tutorials on relevant topics, such as for example Piezoelectric MEMS or Processing of Lead-Free Piezoelectric Materials.

Impact and Benefits:
The Piezo Institute’s aim is to exploit its know-how to improve the competitiveness of European industry by supporting the transformation to a knowledge-based, resource conscious economy through provision of best practice and expertise within the field of piezoelectric, multifunctional materials and devices.

Support for Programme Challenge, Roadmaps, Government Strategies:
Functional materials are central to Materials Programme strategy as well as the Materials Strategy from the TSB.
The Piezo Institute project addresses key policy issues of the European Commission, which is to improve the collection and dissemination of European know-how from all countries.

**Synergies with other projects / programmes**
The Piezo Institute is an excellent vehicle for disseminating the measurement knowledge developed under the NMS Materials and IRD programmes to a broad audience in the UK and beyond. This activity will further develop our opportunities for collaboration across Europe in piezo metrology because of the outreach, marketing and dissemination activities being pursued in this project.

**Risks:**
The proposal for the Piezo Institute assumes that a certain level of income will be achieved for training courses, meetings etc. with the aim that at the end of this project it will be self-supporting. There is a risk that this will not be achieved. However, in mitigation the institute could be maintained but with a smaller remit with the cooperation of the member institutes and little resource.

**Knowledge Transfer and Exploitation:**
The Piezo Institute has developed a 4-point dissemination plan designed to engage with stakeholders principally from across Europe, but also the rest of the world. The dissemination plan is designed to:

1. Raise awareness of piezoelectric technology among potential end-user communities to increase use of technology and interaction with The Piezo Institute;
2. Consolidate and nurture network of existing contacts to enhance intra-European collaboration in the field of piezoelectric and related materials and devices in order to present a coherent European ‘cradle to cradle’ capability in the field;
3. Grow the network and collaborations drawing on European and Non-European R&D teams including academics, institutes and industrial R&D, production and end users.
4. The collaboration will cover research and exchange of scientists and their training, and will benefit European industry through knowledge transfer of new materials and devices development from other parts of the world.

In addition, The Piezo Institute has identified an additional need, existing mainly among SMEs, for a new business brokerage service. The Institute will develop a new service to deliver this need to European companies via new website tools. These tools will facilitate a paid-for service which aims to promote and organise technology transfer

**Co-funding and Collaborators:**
The work is co-funded through FP7 - Integration of technologies for Industrial Applications - support to networks of Excellence with durable integrated structures (supporting actions). Details available on request.

**Deliverables**

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<th>Start: Jan 2012</th>
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<tr>
<td>Co-funding for the FP7 project Piezo Institute II</td>
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</table>
This project addresses the “final frontier” of electrochemical measurement and modelling by tackling the major challenge of characterising the electrochemistry in harsh and extreme environments involving concentrated solutions, multi-phases, high temperature and high pressure. Such electrochemical insight is essential in replacing the semi-empirical approach to materials selection in harsh environments with one founded on fundamental understanding of the electrochemical processes that ultimately determine susceptibility to corrosion and cracking. A world-leading testing capability will be established, cementing NPL’s position at the forefront of measurement and modelling of the electrochemistry of corrosion. Application to specific challenges in the oil and gas industry will provide direct benefit to industry while development of an international standard (ISO) for measurements in such aggressive environments will enhance measurement quality internationally.

**The Need:** Many industrial applications involve elevated temperature and pressure and often potentially aggressive environments including multi-component, concentrated salts with dissolved mixed gases and acidic solutions. Examples include oil and gas production, chemical and petrochemical plant, geothermal plant, nuclear waste containment, and food processing. In most of these examples, corrosion and cracking are the primary integrity issues with the challenge being to confidently define and quantify the viable corrosion threats present and mitigate the risk of failure without undue conservatism in design and system management. Materials selection for these aggressive environmental exposure conditions is carried out usually on the basis of domain diagrams (e.g. pH-H2S) generated by pass-fail types of laboratory tests. However, this methodology is limited with respect to the wider range of variables (temperature, chloride, surface condition, stress state) and lacks a robust scientific framework. There is an increased awareness in industry (e.g. IAG meetings, Proactive Ageing Group for Nuclear Application, Japan) of the need for a more fundamental approach to have confidence in life prediction. From the viewpoint of corrosion and cracking, electrochemical measurement can provide the necessary understanding but is a major undertaking in concentrated solution at elevated temperature (e.g. 200 °C) and pressure (e.g. 250 bar). Measurement of potential has been carried out in the nuclear industry at 300 °C but the environmental conditions are relatively benign. Furthermore, when solutions are multi-component and concentrated, interpreting electrochemical measurements is also a challenge; for example, there is no consensus as to what pH means in concentrated solution and the significance for corrosion rates. This impacts directly in linking field measurements to laboratory testing. There is a clear need for generic standards for electrochemical measurement in harsh environments and for guidance in analysis and interpretation of data in complex media.

**The Solution:** To address these challenges the following developments are required:
- Validated methods for measuring potential and pH at high temperature/pressure
- Improved understanding and quantification of electrochemical kinetics in multi-component concentrated solutions.
- Development and validation of more reliable thermodynamic models of pH in concentrated salt solutions.
- ISO standard for high temperature, high pressure, electrochemical measurement in complex environments.
- Assessment of applicability of these new measurement and modelling tools to materials selection in the oil and gas industry.

NPL’s world-leading position in corrosion and electrochemical measurement and modelling and the successful implementation in engineering applications make it best positioned to deliver impact.

**Project Description (including summary of technical work)**
- Establishment of high temperature, high pressure flow loop for electrochemical measurements under controlled flow in oilfield environments.
- Development of a robust reference electrode for high temperature/high pressure measurement with extension to H2S gas systems (a major challenge) – include evaluation as a tool for field application.
- Validation of pH probes for high temperature applications, focusing on the temperature range 100 °C to 200 °C, where greatest uncertainty exists.
- Application of the experimental methodology to characterise the impact of surface treatment on the electrochemical properties of corrosion resistant alloys in simulated oilfield environments
- Selective measurements to underpin pass-fail domain diagrams in materials selection for oil and gas applications.
- Evaluation of the significance of pH in determining electrochemical reaction kinetics and corrosion noting that the ionic strength effect can result in the same pH for markedly different hydrogen ion concentrations.
- Establishment and validation of thermodynamic model of the pH and solution chemistry in concentrated solutions and
**Impact and Benefits**

- The short-term objective is to support UK based oil and gas companies by characterising the electrochemical behaviour of corrosion resistant alloys in simulated environments with respect to their surface condition, enabling optimum specification of surface treatment.
- Establishment of a more scientific framework for rationalisation and use of domain diagrams in materials selection.
- Validation of thermodynamic modelling of pH in concentrated media.
- A longer-term goal is to establish NPL as a world-leading centre for the measurement and characterisation of electrochemical processes in harsh environmental conditions (noting also the on-going in-situ measurements in operating fuel cells and the SECM-AFM measurements), benefiting UK industry through development and validation of experimental techniques and standards, supported by complementary thermodynamic modelling.
- ISO standard for Electrochemical Measurements in Harsh Environments.

**Support for Programme Challenge, Roadmaps, Government Strategies**
Enhancing sustainability and cost-efficient energy generation with minimised threats to environment and to personnel safety Reliable and efficient Energy Generation and production of Raw Materials Establishing a UK based strategic capability, via a centre of excellence in electrochemical measurements, to support deployment of materials in harsh environments (see Strategy from MAT UK Energy Review of 2007 and from NMS 2010-2014)

**Synergies with other projects / programmes**
There is a direct tie-in of this project with a proposal to BP for a NPL-University of Manchester-MIT collaboration, that NPL will lead, to develop a comprehensive capability for measurement and modelling of the electrochemistry of metals and alloys in harsh environment, including concentrated salts, H$_2$S, inhibitors, at varying temperature and pressure. Additional modelling will be conducted at UoM and MIT to characterise the mass transport processes in restricted geometries and concentrated environments that will link directly with the NPL programme.

**Risks**
There are challenges in developing robust pH and reference electrodes for in-situ measurement that will perform reliably over the range of temperatures and environmental exposure conditions. Mitigating that risk will depend on flexible design and electrolyte bridging methods.

**Knowledge Transfer and Exploitation**
The unique combination of advanced testing capability and scientific expertise will provide a major boost to NPL’s developing position as a world-leading centre for oil and gas testing and research. Business with BP and other oil and gas partners has expanded dramatically in the last few years and the new capability will support that further by responding to the increased demand for testing in harsher environments. Presentations at key conferences at NACE and Eurocorr will extend awareness to other stakeholders and this will be supported by focused scientific papers. The thermodynamic modelling will provide an additional module in the MTDATA suite of programmes. A further, longer-term, dissemination route is through the development of an ISO standard or standards on measurement of electrochemistry in harsh environments developed though ISO TC156 WG 11 (Electrochemistry) of which AT is the nominated UK expert. Any IP developed will be appropriately safeguarded.

**Co-funding and Collaborators**
Details available on request.

**Deliverables**

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<thead>
<tr>
<th>Deliverable title</th>
<th>Start: 01/07/12</th>
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<tr>
<td><strong>Validated high temperature reference and pH electrodes.</strong></td>
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<tr>
<td>Scientific paper outlining the experimental development and associated electrochemical data.</td>
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<tr>
<td><strong>Guidance on surface treatment of corrosion resistant alloys in simulated oilfield environments.</strong></td>
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<tr>
<td>Scientific paper on the impact of surface treatment on the electrochemical properties of corrosion resistant alloys in simulated oilfield environments.</td>
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<tr>
<td><strong>Draft ISO standard for high temperature, high pressure electrochemical measurements in complex environments.</strong></td>
<td>Start: 01/07/12</td>
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<td>Cost: £</td>
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<tr>
<td>Thermodynamic model of the pH and solution chemistry in concentrated solutions exposed to mixed gases.</td>
<td></td>
<td></td>
<td>Extension of MTDATA for concentrated solutions cross-correlated with both BP Cassandra and Shell models.</td>
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**Project Title**: Measurement of high temperature thermoelectric materials modules

**Price to NMO**: £590177

**Lead Scientist**: A. Cuenat

**Scientist Team**: Laurie Winkless, Andres Muniz Piniella, Clark Stacey, Tony Fry

**Sector**: Low Carbon Energy

**Est Final Stage End Date**: June 2016

**Summary**: Improving energy efficiency is the best approach to satisfy the ever-growing need of the world for energy. Recent advances in nanostructured thermoelectric materials have increased the conversion efficiency of the thermoelectric generators (TEG) to the point that they are considered cost-effective for widespread use, if they can be used at high temperature (for example 900 °C on the exhaust of a car). To facilitate the adoption of these promising new materials and stimulate the green economy, three important UK industry sectors require validated output power measurement from TEG: automotive[1]; power [2] and space sector [3]. This project will develop a new capability to measure their efficiency up to 1000 K. This will be a unique facility to measure simultaneously the temperature and heat and electrical flux in materials in a controlled, industrially relevant, environment. The capability will support the implementation of carbon capture schemes and underpin the aims of the Centre for Carbon Measurement (CCM): To accelerate development and assess performance of low-carbon technology and ensure confidence in environmental data. A rigorous uncertainty budget based on operating conditions such as pressure applied to the devices, surface finish and variation in temperature operation will be validated. This will translate to a considerable competitive edge for UK companies wishing to adopt these new thermoelectric materials. [1] UK Automotive Council, 2010; [2] Gao Min “Recent Concepts in Thermolectric Power Generation”, eh-network.org [3] ESA and UK-Space roadmap 2011

**The Need**: Reclaiming energy that is otherwise wasted as heat is an effective way to reduce CO2 emissions. For example, cars convert only about 25% of the chemical potential energy from fuel into kinetic energy; about 65% is converted to heat in the exhaust or the radiator. TEG have significant potential for increasing car energy efficiency through conversion of this heat to power. UK companies such as Jaguar-Land-Rover (JLR), are developing modules that can be fitted to car exhausts, which can reach temperatures up to 900 °C. There are however, significant measurement issues leading to 25% uncertainty in TEG specification [4,5]. While there are some efforts at NIST to produce reference materials, their research is focused on low temperature (100°C) application. Another industrial requirement that is not addressed is the accurate measurement of TEG power output in operating conditions. NPL is working with a range of companies (JLR, Johnson Matthey, European Thermodynamics, Siemens) to support the integration of these new materials. A number of industrial and academic partner have called for a national facility to be developed [6]. [4], Dresselhaus et al. Ener. & Environ. Science 2012 [5] Snyder. Nature, 2008 [6KTN Position paper on Thermoelectric-2012;

**The Solution**: This project will build a unique facility to characterise high temperature thermoelectric materials, and provide a focal point for UK companies that will enable them to create a competitive advantage. This will be achieved by bringing together NPL expertise in thermoelectrics and high temperature standard facility. The significant scientific and engineering challenges will be solved by building a controlled heat source in an environmental chamber incorporating high-precision thermal flux sensors (with an estimated sensitivity of 250 mV/ (Wcm2) at 900 °C, against 10 mV / (Wcm2) for actual commercial state of the art) and capable of measuring temperatures with an accuracy of ±1mK. Direct measurement of heat flux is usually accomplished with a differential temperature sensor. One type of sensor, the thermopile - often referred to as “total” heat flux sensors because they respond to all three modes of heat transfer - measures differential temperature directly using a series connection of thermocouples across a thermal resistance. Significant improvements are possible by using the thermocouple element of the sensor as the thermal resistance of the sensor. NPL has also developed significant knowledge in accurate measurement of thermoelectric modules in an on-going FP7 project on [7]. [7][Harman method - module efficiency measurement system]

**Project Description (including summary of technical work)** Heat flux measurement is a complex process that requires careful design and implementation of both sensors and calibration systems to ensure accuracy in the measurements. Novel methods for calibrating the sensor and other uncooled differential heat flux sensors at elevated sensor temperatures will be developed. Through an on-going project on low temperature measurement [8], we have identified a range of variables that affects TEG output power. The quality of contact between the hot and cold sources and the device – the “thermal contact resistance”- depends on a range of properties: the surface finish of the device, the “clamping” pressure, and the presence of thermal interface materials. The influence of all these parameters will be quantified and mapped using a Design of Experiments approach to manage all the variables, together with the influence of the controlled environmental chamber – variation in pressure, humidity and temperature: the thermal and electrical fluxes both inside and outside the device will be fully quantifiable. Ultimately, this unique facility will provide traceability in the efficiency measurement of TEG in a range of temperature 300 and 1000K. A rigorous uncertainty analysis will be carried out to determine the optimal configuration of the system. [8] TEG measurement facility up to 200 °C developed through the FP7 Nextec project.

**Impact and Benefits** Innovation: Heat flux is a critical parameter in many engineering systems. Improved direct measurement
capable of operation at high temperatures (> 300°C) will facilitate the development of, for example, thermal barrier coatings.

- Economic: It is estimated that validated measurements and standards will enable a potential market of $1B in sectors such as the automotive, space applications and combine heat power where the UK is a an EU leader [KTN Position paper on Thermoelectrics-2012]
- Science: Our measurement will enable the comprehensive characterization of high-temperature materials needed to advance commercially promising thermoelectric technologies.
- Quality of life: the inclusion of thermoelectric generators on cars is estimated to reduce the CO₂ emission by 30 g/100 km [source: JLR]

Our vision is to position NPL as the world-leading reference centre for the measurement of thermal energy harvesting devices. This is similar to the position of the US National Renewable Energy Laboratory (NREL) in the field of the photovoltaics.

**Support for Programme Challenge, Roadmaps, Government Strategies** The project addresses the Growth and Energy NMS grand challenges:

- Materials properties in harsh environments and measurement of the operational environment of energy systems and components.
- The measurement, and knowledge of the uncertainty of measurement for manufacturing [for thermoelectric modules]

The project is aligned with the harsh environment area of the functional materials roadmap at NPL and the Energy and Advanced Manufacturing sector of the NPL strategy.

**Synergies with other projects / programmes** Our existing funded projects (FP7 NexTEC) aims at developing new nanostructured thermoelectric materials for the car industry and measurement at the nanoscale of the Seebeck coefficient (EMRP-Energy harvesting). This project will extend the capability and knowledge of the area by extending it to high temperature. NPL is involved in a VAMAS TWA on thermoelectric.

**Risks** The high temperatures accelerate oxidation and sublimation; large power input for the heater; large cooling requirement. The risk is reduced by incorporating NPL expertise in the area of materials for harsh (turbine) environment and thermal conductivity at high temperature. We are also collaborating with Prof. David Rowe (Cardiff University), a world leader in the development of thermoelectric materials for energy generation [M. Rowe: Thermoelectric Handbook, vol 1 and 2, CRC press 2000, 2007]

**Knowledge Transfer and Exploitation** The innovative scientific solutions will lead to a number of publications papers (Ener. & Environ. Science, Impact factor 9.4). We will produce three case studies to address the most important UK sectors on board our IAG: automotive applications, space exploration and heat transfer for micro-generation. To enable the industry to apply the best practice, we plan to offer measurement service to

- Validate innovative modules to enable increase market share for the supporting company.
- Develop Good practice Guides to measurement of thermoelectric modules.
- Define Standard Operating procedures to qualify new modules.

We will offer a service at an early stage of the project, to establish a leading position and provide competitive advantages to our partners with world-wide sales. The results will be presented to the Automotive Industry thermoelectric conference (Berlin).

**Co-funding and Collaborators**
Details available on request.

**Deliverables**

<table>
<thead>
<tr>
<th>Deliverable title:</th>
<th>Start: 01/07/2013</th>
<th>End: 30/12/2014</th>
<th>Cost: £</th>
</tr>
</thead>
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<tr>
<td>Measurement service for commercial modules up to 200 °C with a repeatability better than 5%,</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Deliverable title:</td>
<td>Start: 01/07/2013</td>
<td>End: 30/09/2014</td>
<td>Cost: £</td>
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<tr>
<td>A controlled environmental heat source facility for thermoelectric materials up to 1000K,</td>
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<td></td>
<td></td>
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<tr>
<td>Deliverable title:</td>
<td>Start: 01/06/2014</td>
<td>End: 30/06/2015</td>
<td>Cost: £</td>
</tr>
<tr>
<td>New calibrated thermopile sensors with a sensitivity of 250 μV/ (Wcm2) at 900 oC,</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Deliverable title:</td>
<td>Start: 01/06/2014</td>
<td>End: 30/06/2016</td>
<td>Cost: £</td>
</tr>
<tr>
<td>Traceability of integrated facility and publication of three case studies.</td>
<td></td>
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</tr>
</tbody>
</table>
A novel set of test methods are proposed to characterise protective coatings used on electronics that are needed, both to meet developments in technology driven by Moore’s Law and for a wide range of industrial demands to locate electronics in increasingly harsh conditions. Within this technology there are a number of high level government strategies that are dependent on electronic systems working across a range of technologies in this commercially very important sector. This project will develop new functional metrology capabilities that can be exploited by NPL and industry to accelerate the qualification of systems into harsh conditions. NPL is uniquely suited to develop these technologies and can take the developed methods forwards internationally in the standards arena.

### The Need
The effect of Moore’s Law on microelectronics integration has pushed adjacent terminations on components ever closer together; in addition, in many application areas systems are required to operate in ever harsher conditions. These two factors give rise to an increased potential for short circuit and failure, driving a significant increase in the use of coatings. Current coatings are reflective of established technology and need to be developed to meet the requirements of harsher environments, requiring commensurate development of test protocols and measurement methods. New technologies; now require humidity barriers, and existing high reliability technologies in addition need a coating mitigation to tin whiskers. There are currently no tests: for coating penetration by whiskers, in situ moisture measurement within packages, & condensation testing.

Strategy as defined by BIS, TSB, and NMO include a number of high level objectives that need developments within electronic assembly systems: The Digital Challenge, Sustainability, Renewable Energy, Power Electronics in low Carbon Vehicles, Defence and Security, and Non-Destructive Evaluation (NDE).

IEC is generating new standards for coatings. NPL has a key role of advising on the test methods within these standards.

New coating technologies are being developed, ultra-thin coatings based on fluoro-carbons, right through to super hydrophobic chemistries, and nano particle filled coatings. These new advances require test methods to validate their use.

The leading research into coatings is currently being undertaken in the US by Sandia Laboratories, IZM Fraunhofer in Berlin, and IMEC in Belgium.

### The Solution
To solve the measurement challenges for insertion of advanced electronics into the above harsh conditions is the development of a suite of test methods. There are three measurement challenges: measurement of low level moisture, penetration of coatings by tin whiskers, and a measurement method for condensation conditions. There is currently no condensation test, reflecting the difficulty of controlling the condensation conditions within standard humidity chambers, this project plans to develop a modification to the sample configuration to achieve this test. Similarly with whisker penetration of coatings, both in and out of the coating, a test method will be developed. This is contingent on a unique capability that NPL has in plating tin that will grow whiskers in short time scales. For low level moisture sensitivity miniature high gain sensors will be developed, an IP opportunity, which will be applicable to component cavity packages. This will build on earlier projects where moisture measurement has been developed. These approaches will include novel electrical probing, development of mechanical probing of unsupported coatings, sensor arrays and micro-climate control around the test sample.

A multi-disciplinary approach is required for these challenges to which the expertise and resource at NPL is well matched. Such expertise does not exist to the same extent elsewhere in the UK. Project collaboration is envisaged for success, particularly with industry where the implementation problems lie, and NPL is well placed to leverage that support. Industry typically does not have the expertise for the challenges outlined here and will be keen to collaborate. Finally NPL is well placed to take the test methods forward internationally into the standards arena.

### Project Description (including summary of technical work)
Project aims to develop generic functional tests for coating performance in three areas: mechanical, electrical, environmental.

A coatings’ resistance to penetration by a tin whisker, ~5µm diameter and length from 1µm to 1mm, from under the coating and into the coating, presents a challenge from their high aspect ratio and flexibility. Penetration by fretting is a failure mode. Modification of equipment of penetration into a coating is planned by modification of existing equipment, but penetration out through a coating will require more effort in developing a support for the coating. We also plan to develop techniques for NDE testing of coating thickness, particularly close to small radius bends.

Electrically we plan to develop sensors with high gain, comb patterns on silicon, and work with partners on different packaging styles and compare with traditional Helium leak detection. The latest component geometries require testing methods that do not exist. The aim is to develop a very low cost sensor that is as sensitive as Helium testing but less awkward and can be easily
to components. Development of DC electrical probing will be developed using more sensitive impedance techniques.

To improve corrosion protection performance a condensation test will be developed, consideration will be given to the effect of pollutants from the atmosphere and those trapped under the coating. For condensation testing we plan to modify the microclimate around the sample within a humidity chamber. Hence we can introduce a cyclic condensation test. Since coating coverage is critical, various sensor configurations and arrays will be trialled to develop the most sensitive test.

In all of the above the introduction of new exciting coatings, beyond those traditionally used for electronics will be considered, superhydrophobic, nano-particle fillers, and self-healing coatings. Any test method needs to be sensitive to typical coating defects: pin holes, cracks, delamination, etc.

Impact and Benefits
The UK electronics market of £40bn is now one of the biggest of the European markets (£150bn) with BIS suggesting that growth in this market is between 2 and 6% annually. The UK IT, telecoms and electronics manufacturing industries (including blue-chip multinationals as well as early stage technology companies) generate around 10% of UK GDP (£40bn) and 15% of UK trade, and is enormously important to UK plc. It is unknown how much of this will be dependent on coating protection, but even a small fraction will be significant. Tin whisker failures for example can be extremely significant, and have been responsible for nuclear power station failures, health product failures, satellite failures, to name but a few. The Toyota unintended acceleration problem is widely understood to be in a major part due to whiskers, and this recall cost Toyota $2bn. Failed satellites, including launch cost can also represent $1bn. So high reliability failures can be spectacularly expensive. But other more business type scenarios can be challenging, large server companies with banking sector customers have found their products failing in Asian climates due to corrosion triggered by pollutants in the atmosphere.

Support for Programme Challenge, Roadmaps, Government Strategies
The project fits directly within overall strategies as defined by BIS, TSB, and NMO: The Digital Challenge, Sustainability, Renewable Energy, Power Electronics in low Carbon Vehicles, Defence and Security, and NDE. All these areas are dependent on electronics systems that can perform in a wide range of environmental conditions and material systems.

Synergies with other projects / programmes
This project is part an on-going set of projects that have been developed around advances in interconnect advances in arrange of new systems: PVs, printed electronics, nano-interconnects. Work from this project will also contribute to activities on prognostics of electronic systems. The work here will be coordinated with other public funded activities, like MTC and others.

Risks
The plan needs sensors to work over a range of conditions, to mitigate against failure we will develop alternative design approaches. We will also consult with other NPL areas of expertise and relevant industrial expertise.

Knowledge Transfer and Exploitation
Knowledge transfer will be through a number of targeted routes. NPL has excellent links with trade bodies, SMART Group, IMAPS, NMI and KTN, and will organise joint meetings with them on coating performance. Web based systems will utilised including LinkedIn thorough SMART & IMAPS and other as appropriate. NPL Facebook and Twitter can be used along with other streams. At least one webinar on the work will be broadcast. The developed knowledge and resources will be offered as consultancies and as a service within the Measurements Services portfolio and we will look for collaborative research opportunities, such as TSB. Furthermore scientific papers, standards, IAG, GPG will be delivered. Exploiting the sensor development will also be considered.

Co-funding and Collaborators
Details available on request.

Deliverables

| Deliverable title: Develop electrical functional testing and sensing techniques |
| Start: 01/04/13 | End: 30/12/14 | Cost: £ |


| Deliverable title: Develop mechanical functional testing: penetration, flexibility, and coverage |
| Start: 01/10/13 | End: 30/06/15 | Cost: £ |

Evidence: two peer reviewed papers on test methods: (i) Penetration methodology & flexibility of coatings and (ii) NDE coverage measurements with high spatial resolution.

| Deliverable title: Develop environmental and condensing test method and novel sensor design for coatings |
| Start: 01/04/14 | End: 30/12/15 | Cost: £ |

This project is the NMO-funded component of a TSB project that aims to address the interconnect challenges of achieving a high level of integration driven by Moore’s Law and delivering a high temperature interconnect needed for future power devices and technologies such as SiC. The project fits with BIS strategy and is in line with ITRS roadmap. The project will develop new measurement capability for assembling and testing sintered interconnects from nano-metal particles. Being state of the art, there is an expectation to be able to licence the technologies and instrument developed in this project. It is expected that metal powder sintering can overcome the current development road block of using SiC.

**The Need**

Many applications are now emerging for systems to be used in harsher environments where elevated temperatures, pressures and aggressive media are involved. Sensors for aerospace engines and engine management systems, oil and gas wells and under-bonnet automotive and power management for hybrid/electric vehicles, are some specific application areas. These have tough specifications that will command sustainable, high value manufacturing solutions and a strong UK manufacturing presence. As a result of emission legislation (eg CO2 emissions) and the drive for improvements in engine efficiency, the market for these applications is growing fast. As the sensing electronics moves ever closer to the source of combustion (in engines) or drill heads (down hole applications), normal electronic lead free soldering (melting 220°C) are unsuitable due to the ambient temperature exceeding solder melting points. The joining technology for these markets has traditionally been high lead(Pb) solder (melting point 300°C), however these materials are likely to be banned under the RoHS directive in the near future and viable sustainable alternatives are urgently required. Alternatives such as Gold–Tin alloys are extremely expensive (~£45,000 per kg). Recent innovations have led to the availability of nano-particle materials which show promise to be sintered at low temperatures to produce interconnection media capable of withstanding high temperatures of meeting the demands of the above markets. The UK is well positioned globally in the power and high temperature electronics industry. The BIS strategy of the Digital Economy and Sustainability assumes that very-large-scale integration (VLSI) interconnects and power electronics will be developed, this project helps support these aims. This is a very active area in academia and there are requirements for metrology in these systems. The University of Surrey is such a player and the connection with NPL is a strong one with EngD students.

**The Solution**

Solders can perform reliably up to 150°C, beyond which, new systems are required. NPL is well placed in the developments of fine scale interconnect characterisation. The work at NPL will develop a new tool for characterising nano interconnects which we plan to explore IP and licensing opportunities. The instrumental, materials and electrical skills are well suited to NPL’s expertise. A more immediate solution for high temperature interconnects is required, and hence today there is increasingly more interest in sintering of metal nano powders. The recent development in nano metal powders with low sintering temperatures has created an opportunity for their use as interconnects. A number of material companies are promising materials in the next 12 months, and universities are formulating experimental material systems.

**Project Description (including summary of technical work)**

This project would look at exploring the exciting new area of using metal nano silver powders as an interconnect solution. Interconnect processing routes, material and electrical properties and the time-dependent properties of nano silver interconnects are all unknown. A novel design, the Characterisation and Assembly of Nano-Interconnect System (CANIS), enabling the building and in-situ testing of interconnects has been proposed but has yet to be built. Using CANIS we would investigate these and develop the metrology methods.

**Impact and Benefits**

The global market for automotive underhood electronics is expected to increase to $4bn per annum and for high temperature electronics to $2.2bn by 2015. The potential market for assembly materials for high temperature electronic interconnect is very wide, covering a range of end applications from transportation (automotive, commercial, hybrid & electric vehicles, marine diesels and gas turbines) through oil and gas extraction to commercial and domestic boilers. There is pressure within all these industries to migrate the electronics closer to sensors working in high temperature environments. Better proximity of the electronics and higher operating temperatures will allow improved efficiency, more rapid feedback and reduced weight of interconnecting pipework and cabling. Direct chip attach (DCA) for insulated gate bipolar transistor (IGBT) market is currently estimated at globally £1bn and growing at 10%. UK manufacturing represents more than 10% of this, these measurement techniques will help them to grow. The interconnect performance is a road block to incorporation of SiC technology, and sintering can potentially unlock this. Power semiconductor and sensor manufacturers and a raft industries using these devices...
Sustainability benefits will be in 5 main areas: (i) High temperature electronics are currently fabricated using high Pb-content solders. The project partners estimate that the introduction of sintered Ag materials will significantly reduce the amount of Pb and Sb from entering the waste stream in the next 15 years by in excess of 10,000Kg. (ii) Electronics operating at higher temperature will enable sensors and monitoring electronics to be moved closer to their application positions, resulting in lighter, more efficient planes and cars that will reduce energy consumption and CO2 emissions. (iii) The increased operating temperature of ELCOSINT materials will enable improved performances in power applications such as DC-DC and AC-DC converters, power inverters, and power management electronics in renewable energy generation. Similar performance benefits will be realised in hybrid and electrical vehicles and other sustainable transport solutions. This will again result in energy savings and reduced CO2 emissions. (iv) Applications in the oil, gas and mining industries will allow improved access to hard to harvest, scarce materials resources. (v) Operating temperatures above 250degC are a current road block for SiC developments. Elcosint materials will assist in overcoming this and allow improved high-voltage switching in public electric power distribution and electric vehicles, resulting in energy savings and lower CO2 emissions.

Support for Programme Challenge, Roadmaps, Government Strategies
BIS and NMO strategies have identified the Digital Economy, and Sustainability, Renewable energy, and power electronics in low carbon vehicles. All these areas are dependent on electronics systems that can perform either high interconnection densities or under high temperatures. This project fits with the NMO thrust on nano materials and characterisation.

Synergies with other projects / programmes: This project is part of an on-going set of projects that have been developed around advances in interconnects. The work here will be coordinated with other public funded activities, like MTC and others.

Risks: Acquiring suitable samples is a risk, we have preferred suppliers but we continue to survey the market for alternative sources. There is a risk in delivering CANIS, but we can use expertise from around NPL to assist with any difficult problems.

Knowledge Transfer and Exploitation: Knowledge transfer will be through a number of targeted routes. NPL has excellent links with trade bodies, SMART Group, IMAPS, IeMRC, NMI and KTN, and will organise joint meetings to report progress. Web based systems will utilise LinkedIn thorough SMART & IMAPS and others such as NPL Facebook and twitter as appropriate. At least one webinar on the work will be broadcast. The developed knowledge and resources will be offered as consultancies and as a service within the Measurements Services portfolio. The developed instrument, CANIS, will also be explored for licensing opportunities. Furthermore the findings will be delivered in scientific papers, standards, and outreach through the IAG.

Co-funding and Collaborators
This project relates to a TSB proposal, ELCOSINT (£160k from TSB). We are also developing relationships with IGBT companies. Details available on request.

Deliverables

<p>| Deliverable title: Co-funding for TSB project ELCOSINT | Start: 01/11/12 | End: 30/10/15 | Cost: £ |</p>
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<tr>
<th>Project No.</th>
<th>MAT\2012\8</th>
<th>Price to NMO</th>
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<tr>
<td>Project Title</td>
<td>Rare Earths Phase Diagram Club</td>
<td>Co-funding target</td>
<td>£150k</td>
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<tr>
<td>Lead Scientist</td>
<td>Hugh Davies</td>
<td>Stage Start Date</td>
<td>July 2012</td>
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<td>Scientist Team</td>
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<td>Stage End Date</td>
<td>June 2015</td>
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<tr>
<td>Sector</td>
<td>Advanced Manufacturing</td>
<td>Est Final Stage End Date</td>
<td>June 2015</td>
</tr>
<tr>
<td>Project Champion</td>
<td>Prof Rachel Thomson</td>
<td></td>
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</tr>
</tbody>
</table>

### Summary
The project aims to start a multi-client club activity for the assessment and creation of underpinning thermodynamic databases for rare earth elements, oxides and other important compounds. Phase diagrams will be the main output and be calculated by manipulation of the underlying thermodynamic models by software such as NPL’s MTDATA code. This project will require the industrial co-funding to be in place to go beyond deliverable 1.

### The Need
Phase diagrams are of immense importance in determining the behaviour of materials during processing and use. In fact the introduction of a new technology (eg lead free solders) is often accompanied by significant interest in the fundamental thermodynamics of the relevant systems. In a world where materials security (geopolitical risks, poor recycling rates) is becoming of increasing concern, the use of phase diagrams to underpin choices in materials substitution and changes to processing routes will become increasingly important. The class of elements called Rare Earths is a likely case in point where there has been a lack of effort in compiling comprehensive thermodynamic and phase diagram information for systems except for the few that have achieved major technological prominence (eg magnets). Many other technologies (eg high-power lighting, catalysts, phosphors) could be varied to use alternative elements, however the underpinning thermodynamic and phase diagram information is often missing or too unreliable allow new processes to be evaluated and decisions made. Recent reports have highlighted seven (Dy, Tb, Eu, Nd, Y, Ce and La) rare earth elements (Critical Materials Strategy, US Dept of Energy, Dec 2011) as being at a critical or near critical supply risk in the short term (to 2015). The 2011 EU JRC report on Critical Metals in Strategic Energy Technologies supports the high risk status of the rare earths Dy and Nd. A recent House of Commons Science and Technology Committee report on Strategically Important Metals (HC 726, May 2011, Fifth Report of Session 2010–12) considered a wider range of elements; however rare earths were given a prominent position in evidence. We recognise there are other strategically important elements, in addition to the rare earths, however from our perspective as experts in thermodynamics and phase equilibrium this class of elements stand out in having a significant absence of assessed thermodynamic data for suitable for the calculation of phase equilibria.

### The Solution
Phase diagrams are commonly found in paper compilations. However NPL has pioneered the computer-based delivery of phase diagram information in terms of the underlying thermodynamic properties of the properties of the phases in the system together with software for Gibbs energy minimisation. The combination of these with specific parameters for the interaction of rare earth atoms in various structures (eg fcc, liquid) allow the rigorous calculation of low-order phase diagrams and rational extension into predictive calculations for higher order systems.

The project aims to start a club activity for the generation of parameters of interaction between rare earth elements in industrially relevant phases. The club members will provide the steer towards the specific rare earth elements to be studied. Databases of thermodynamic data arising from this work at NPL (Crown Copyright) would then be licensed on a non-exclusive basis but with a lead-time to protect the interests of the sponsors.

### Project Description (including summary of technical work)
- Formation of club and selection of scope of thermodynamic assessment activites with sponsors
- Selection of specific systems to be assessed
- Selection and testing of thermodynamic models for different phase types
- Assessment of model parameters
- Interim release of thermodynamic database for testing by club members
- Final release of thermodynamic database to club members

### Impact and Benefits
Support for “at risk material” substitutions by providing a rapid means to explore the effects of additions or replacements. Helps in increasing the efficiency in processing and/or recycling of rare earth containing materials. The creation of a rare earth phase diagram capability increases NPL credibility in equilibrium thermodynamics and phase diagrams.

### Support for Programme Challenge, Roadmaps, Government Strategies
This project strongly aligns with the recent NMO strategy aims to “bring confidence to the introduction of more sustainable products, materials and processes, by providing objective methods of process control and performance”
evaluation, including support for whole life cycle analysis, “and to “underpin the development and evaluation of novel alternative materials to replace those with limited reserves” and sits within one of NPL’s key areas of science, “modelling to characterise materials”. Recent reports from the UK (Commons Science and Technology Committee report on Strategically Important Metals), EU (2011 EU JRC report on Critical Metals in Strategic Energy Technologies) and US (Critical Materials Strategy, US Dept of Energy, Dec 2011) have highlighted a number of rare earth elements as being at a critical or near critical supply risk.

Synergies with other projects / programmes
The thermodynamics group at NPL has current experience with commercial multi-client projects on oxide thermodynamics and phase equilibria. Some of the outputs of this project may be used in this proposed project for non-rare earth components. There is a synergy with current and potential future projects on high temperature alloy corrosion, catalyst stability and activity and functional materials.

Risks
The specific risks identified are:
- (technical) difficulty in modelling the thermodynamic properties of rare earth containing systems. NPL has the necessary expertise in the thermodynamic modelling of a wide variety of relevant systems (alloys, oxides, aqueous), use of theoretical and estimation methods.
- (technical) difficulty in obtaining good quality basic thermodynamic and phase diagram experimental data for unary and low-order systems. Collaboration with SGTE (Scientific Group Thermodata Europe) and APDIC (Alloys Phase Diagram International Commission), sharing of information with commercial sponsors and users of rare earths, and potentially use of theoretical methods will be used to minimise this risk.
- (commercial) difficulty in forming the club. This will be minimised by collaboration with an external organisation (eg Mineral Industry Research Organisation, MIRO) that has experience in setting up and managing multi-client research projects. NPL has experience working with MIRO.

Knowledge Transfer and Exploitation
All IPR in databases of thermodynamic model parameters developed in this project will be held by NPL as Crown Copyright. Due to the high fraction of non-NMS funding in this project these databases will initially be exploited by licensing to the commercial sponsors of the project. Following a suitable lead time all databases will be made available for general licensing to non-sponsors and/or incorporation in other more general thermodynamic databases produced by NPL or its international partners (SGTE).

Co-funding and Collaborators
Initial discussions have also taken place with MIRO who have informally indicated that they wish to be involved in developing the applied aspects of this project. Details available on request.

Deliverables

1 Start: 1/07/12 End: 30/06/14 Cost: £
Deliverable title: Formation of club and selection of scope of thermodynamic assessment activities with sponsors, review and selection of thermodynamic models

2 Start: 01/12/12 End: 30/06/14 Cost: £
Deliverable title: Selection, implementation and testing of models

3 Start: 01/04/13 End: 30/06/14 Cost: £
Deliverable title: Assessment of model parameters for required systems and interim release of thermodynamic database for testing by club members

4 Start: 01/07/14 End: 30/06/15 Cost: £
Deliverable title: Final release of thermodynamic database to club members
Project No. | MAT\2012\9 Part A | Price to NMO | £317479
--- | --- | --- | ---
Project Title | Determining the uncertainty of in-situ strain mapping | Co-funding target | £200k won from TSB (see MAT\2012\9 PartB)
Lead Scientist | Nick McCormick | Stage Start Date | Oct 2012
Scientist Team | Ken Mingard, Nick McCormick, Tony Fry, Matt Brooks, Jerry Lord | Stage End Date | Feb 2015 Ext to June 2015
Sector | Advanced Manufacturing | Est Final Stage End Date | 
Project Champion | Peter Flewitt | Activity | Development of Existing Capabilities

Summary: This project relies on additional TSB co-funding for its successful outcome

The combination of in-situ testing and full field in-plane deformation measurement is a powerful tool for characterising the performance of materials and structures at many different length scales, supporting safety cases, providing material data and identifying maintenance needs to help enhance the lifetimes of critical structures. Digital Image Correlation (DIC) and Electron Backscatter Diffraction (EBSD) can make full field strain and displacement measurements, and are beginning to be adopted by industry, but the accuracy and uncertainty in the measurements needs to be better understood to predict the quality and resolution of the measurement, and extend their use to more challenging applications. NPL’s underpinning role in validating these techniques would enable development from isolated case studies to widespread adoption and create valuable IP.

The Need

Network Rail Justification for Civil Engineering Policy, March 2010 highlights that replacement of a rail tunnel is not affordable and that each tunnel must remain in a fit for purpose condition for an indefinite period. They suggest the use of innovation to restrict disruptive possessions is an essential area of improvement. DIC is a front runner for use as a low cost alternative to the current solutions which cost £100k for each site studied and will never be affordable for wide scale deployment.

At the other end of the length scale, measurement of strain at the microstructural level is critical to understanding failure mechanisms and for materials lifetime prediction in harsh environments such as nuclear power plants. NPL customers have made major investments in determining the accuracy and applicability of bulk strain measurements to stress corrosion cracking for nuclear applications (>£600K since 2005): fundamental work is now needed to demonstrate the accuracy and uncertainties involved in use of DIC and Electron backscatter diffraction (EBSD) at the microstructural level. This would enable further confidential third party work to be undertaken and ensure wider uptake by companies such as Rolls Royce and EoN.

The Solution

NPL’s expertise in the area of DIC has been recognised by Network Rail and Highways Agency for large state of the art structural measurements and by Magnox for measuring strain and deformation in nuclear graphite in projects totalling over £400k. Recent experience with full field strain mapping and surface deformation measurements has led to a good understanding of where errors arise in DIC and EBSD. A systematic approach in combining these errors will be developed to give an estimate of the total full-field error in advance of the measurement to enable greater confidence for industry, and will be applied to a range of applications over different length scales (nano – micro – macro – mega);

- By comparing DIC strain measurements at the scale of a material grain (nano) with EBSD strain measurements in the SEM, cross-validation of the techniques will allow a comparative error estimation to be made.
- To assess DIC measurement errors for determining strain at the microscale a case study will be carried out to examine layered structures such as oxide scales and thermal barrier coatings (TBC) needed for calculating damage accumulation and life prediction, as a function of thermal cycling, accurate robust measurements are needed to characterise increasing more complex materials and conditions.
- To explore errors in DIC measurements at the macroscale in extreme conditions, high temperature measurements will be made with the NPL ETMT and oxide scale test rigs.
- For megastructures the TSB Project on Accelerating Rail Innovation will provide solutions for DIC measurement of large scale structures, these will rely on a robust estimation of error.

Project Description (including summary of technical work)

By comparing DIC strain measurements at the scale of a material grain with EBSD strain measurements in the SEM, cross-validation of the techniques will allow comparative error estimation to be made. NPL has a state of the art technique for accurate calibration of EBSD systems needed to calculate absolute values of strain in polycrystalline materials. The use of the calibration method has been in a published paper but needs to be proven by application on real engineering materials. DIC measurements using the ETMT will be aimed at extending the application of the technique to thermal transients and to extending the temperature range to 1200°C. This will require novel solutions for minimizing errors and compensating for thermal currents and image distortion, this will build on a recently completed NPL Innovative Metrology project that indicated a way of determining the errors caused by atmospheric refractive index variation.

The TSB Project on Accelerating Rail Innovation will be concerned with understanding the errors introduced into the measurement process by identifying causes of general imaging errors and mitigating actions. It is expected that particular issues
There will arise during the course of tunnel examination due to the scale of the measurement, the type of environment (vibration at speed) and the possibilities of difficulties in control of orientation and position of the camera.

### Impact and Benefits

Work on DIC has already been used with success to help extend the working lifetime of current nuclear plant in material characterisation and it has real potential as a novel NDE tool for monitoring of the structure, particularly for identifying and measuring cracks in aggressive, inaccessible environments. A clear understanding of the in-situ measurement errors would ensure appropriate levels of accuracy increasing confidence in the measurements. This will lead to opportunities in nuclear decommissioning, waste management and nuclear plant life extension, currently an area of high national priority with a budget exceeding £42bn. This project will have a significant impact on the experimental mechanics community, particularly those involved in materials development and testing, design, FE modelling and validation and structural characterisation, through the development of the GPG and procedures for assessing accuracy and uncertainty in the measurement. Collaboration with LaVision, GOM, Airbus, Dantec, Imetrum and BSSM is expected to give high visibility and impact. The UK power generation industry and aero industry will benefit directly from the improved measurement capability and more accurate data generated on oxide scale kinetics and on TBC lifetime as a result of the new high temperature DIC capability. This project will support work for a TSB Accelerating Innovation in Rail Project currently in second stage development with support from TSB of £450k and industrial partners of £250k. The EBSD activity will have an impact on both manufacturers of equipment and end users, including manufacturers of advanced materials such as Tata, Sandvik, Rolls Royce and their customers from the power generation sector (nuclear and conventional) and the oil and gas industries for whom understanding and measurement of residual stress is fundamental to controlling component lifetime. SMEs involved in the EBSD market such as KE and BLG will also benefit from working with NPL to develop new products in their niche markets.

### Support for Programme Challenge, Roadmaps, Government Strategies

In-situ strain measurement is a strategic part of the NPL Advanced Engineering Materials group activity and directly underpins much of the materials characterisation work. At a recent road mapping exercise with NNL full-field deformation measurements were identified as being important during decommissioning of existing nuclear plant and for the monitoring of waste storage. This project supports the NMS in the areas of Sustainability, Energy and Digital Challenges, by developing in-situ monitoring techniques that can be used to improve the efficiency and extend the operational lifetime of fossil fuel plant and support existing Nuclear Plant.

### Synergies with other projects / programmes

This work aligns with Network Rail’s vision for the 2030 railway and the rail industry’s Technical Strategy Leadership Group (www.futurerailway.org) main challenges for UK rail and in other rail markets such as metro, light and high-speed rail and regional railways. This work will benefit from expertise from the NPL Freeform centre. This project also supports the TSB IMPACT project and expected follow on for applications in power plant.

### Risks:

Technical risks in identifying suitable measurement techniques have been mitigated, as previous work by NPL has developed a shortlist of techniques that are likely to work in extreme conditions. Risks arising from alignment control for the rail measurement platform will be mitigated using TSB project partner’s expertise in rail-based measurements. TfL and Network Rail have already identified and will provide access to the suitable test tunnel for measurement.

### Knowledge Transfer and Exploitation

A Good Practice Guide on using DIC, focusing principally on assessing the measurement errors will be developed in collaboration with equipment manufacturers and users this has been identified by the BSSM as a necessary step for the wider adoption of these technologies in many sectors and delivery will be via BSSM, BINDT and KTN’s. IP exploitation will be carried out in conjunction with the TSB project. Papers describing the outputs of the deliverables and associated news articles will be produced. A workshop on tunnel examination techniques will be held. A service to industry will be developed.

### Co-funding and Collaborators

Details available on request.

### Deliverables

<table>
<thead>
<tr>
<th>Deliverable title: Uncertainty of DIC and strain mapping</th>
</tr>
</thead>
<tbody>
<tr>
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</tr>
</tbody>
</table>
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### Co-funding and Collaborators

Details available on request.

### Deliverables

<table>
<thead>
<tr>
<th>Deliverable title:</th>
<th>Co-funding for TSBC Accelerating Innovation in Rail Project DIFCAM</th>
</tr>
</thead>
</table>

The deliverable will be a demonstration of the application of DIC and imaging techniques for tunnel measurement.
Summary
The metrology of crack initiation and growth in a range of advanced engineered materials (e.g. hardmetals, high strength steels and ceramics) will be studied with the development of a suite of novel in situ test jigs that will allow examination of crack – microstructure relationships during propagation combined with the measurement of the crack tip stress state. The in situ test jig development will build on NPL’s expertise with miniaturised testing and highly controlled application of loads needed to prevent run away growth. Licensing of the test jig designs to equipment manufacturers will be sought.

The Need
Failure of highly stressed engineering components by deformation mechanisms such as fracture and wear is a major cause of financial loss and in extreme cases fatalities. One estimate of costs puts it at 4% of GNP (Stephens et al, 2001) in the USA for fracture and fatigue while failure analysis of components by NPL alone, across aerospace, transport, electronics and nuclear industries costs these sectors over £0.5M. More proactively, the MatUK Energy Review #6 called for improved understanding of subcritical crack growth and the influence of residual stresses in materials for the nuclear sector, and tools for extending the predictive capability of fracture mechanics calculations. Future modelling requirements expressed by Rolls-Royce to NPL include the linkage of current macroscopic stress-strain measurements to mechanisms at the micro-scale.

To achieve this improvement the dynamic microstructural mechanisms that control failure modes such as fatigue, sub-critical crack growth and wear must be understood in combination with knowledge of both the local crack tip and bulk stress states. This requires in situ observation of the failure process and the material microstructure simultaneously together with measurement of local stresses. While many observations have been made after the failure event, in situ observations using commercially available rigs (e.g. Gatan/Deben, Kammrath&Weiss) are limited by the lack of test rig stiffness which often prevents precise control of the crack growth. This in turn limits measurement of stress field development at the crack tip (e.g. even in Al alloys (Lalpoor et al 2010)), and is particularly challenging for extremely stiff ceramics and hardmetals. Combining observation of crack growth and microstructure with the dynamic measurement of stress and strain by EBSD and DIC, and relating back to bulk measurements of, for example, residual stress is even more difficult.

The Solution
This project will deliver and apply a suite of novel in situ (in the SEM) test systems for the simultaneous observation of the degradation and failure modes with the materials’ microstructure to improve understanding of the microstructural mechanisms and the controlling effects of the materials stress state. It will build on NPL’s experience and reputation for stress and strain measurement across a range of scales, miniaturised test rig development and microstructural characterisation. In particular it will utilise knowledge gained under a strategic research project which developed an ultra-stiff test rig and modify this to observe controlled crack growth under both monotonic loading and fatigue in situ in the SEM. Further modification would also enable study of initiation and early/short crack growth to understand why some cracks start but do not propagate further. A range of methods of measuring of elastic and plastic strain in the bulk and at the crack tip will be evaluated.

Project Description (including summary of technical work)
A comprehensive programme of project work will be carried out that will include:
- Further development of the ultra-stiff test rig to allow cyclical stress application in hardmaterials, ceramics and metal alloys.
- The development of strain measurement methods with miniature clip gauges and resistivity measurements to enable mesoscale measurements to be related to microscale digital image correlation (DIC) measurements and Electron backscatter diffraction (EBSD) strain analysis.
- The development of new test protocols and a new in-situ rig to examine the initial stages of crack growth from intrinsic surface flaws developed naturally through surface preparation such as grinding and EDM, and from artificial flaws introduced by FIB-SEM. Measurements of residual stress in the sample surfaces before and after such testing will be essential to understand results.
- The examination of how crack tip deformation modes relate to the microstructure of the materials (and its distribution between two phases for hardmetals) and how this controls crack blunting. This will include the measurement of the crystallography of the crack path and determining the relationships between crack growth direction and phase boundaries, crystallography, grain size, contiguity and other stereological measurements of the microstructure.
### Impact and Benefits
This project will provide new understanding of the microstructural basis of the failure modes of materials from fracture and wear enabling development of new and improved materials with marked improvements in performance in demanding applications. A specific objective is to produce microstructural guidelines for development of hardmetal grades with greater fatigue and wear resistance, benefitting UK manufacturers such as Marshalls Hardmetals and Sandvik Coventry in the production of, for example, hardmetal mining “buttons”: with a worldwide market in excess of £100M for hardmetal mining components, a small gain in market share made possible by reduced failure rates will lead to substantially increases sales.

Lifetime modelling for safety critical environments in the oil and gas sector and especially nuclear power and propulsion plants will benefit greatly from the increased knowledge of stress field interactions between short cracks, microstructural and residual stress profiles and improved procedures for surface finishing methods will be defined for NPL customers in all these sectors.

### Support for Programme Challenge, Roadmaps, Government Strategies
This project addresses several challenges in the strategies of the NMO and the government (TSB High Value Manufacturing, Advanced Materials, Healthcare, Nanoscience, Sustainable Energy) by enabling the development of innovative and energy efficient products, addresses materials properties in harsh environments (e.g. rotating hardmetal seals, drill heads, oil and gas and nuclear power components), reductions of impact on the environment, increases in the efficiency of manufacturing and reducing the waste of materials because of reduced failure rates leading to lower consumption of strategic materials.

The project is a good fit with the roadmaps for the M4 and EMRP Programmes addressing the requirement for better understanding of the relationship between the failure mechanisms of materials and their microstructure.

### Synergies with other projects / programmes
This project will draw on the capability developed in previous projects in the Strategic Research Programme where the in situ stiff cracking test system was initially developed, and the IRD Programme where Project T11 developed techniques to monitor the degradation of surfaces from wear, in particular developing the in-situ microtribometer system that will be used in this project.

### Risks
The main risk of the project is the development of optimised in situ test systems which do not interfere with the imaging processes in the SEM (especially EBSD). This is technically challenging, but the risk is ameliorated by the expertise of the project team built up in previous work. Analysis of the large quantities of data that will be generated and defining the relationship between microstructure and failure mode also presents a risk which again is ameliorated by the experience of the project team.

### Knowledge transfer and exploitation
The test systems that are developed and utilised in this project will be licensed through firms such as Gatan, with whom discussions have already been held, to ensure that the technical developments in this project are fully available to academia and industry. Case studies will be required by Gatan to help market such products and will be developed in this project targeting the hardmetals sector and stainless steel users in both oil and gas and nuclear sectors. Future income will thus derive, through licensing, of equipment sales and probably to a greater extent from use of the equipment at NPL where expertise and background knowledge gives added value to results from the test rigs.

The already good links with industry through individual firms and industrial associations such as the British Hardmetals Research Group (BHRG), the European Powder Materials Association (EPMA) and British Soc. For Strain Measurement (BSSM) will be further developed so that the methodology and knowledge that is developed can be readily taken up by industry. An industrial stakeholder group will also be formed to ensure that industry has the opportunity to inform the project concerning industrial priorities, and to receive key output from the project. In particular links will be developed with Catapult AFRC and AMRC centres, and with the Rolls-Royce modelling group.

Dissemination of results will continue after the end of the project to ensure that the full impact from the project can be achieved. Scientific publications will be made, as appropriate, in peer reviewed journals to disseminate the project results to the scientific and technical communities. Results will be presented at a themed meeting of the Measurement Network.

### Co-funding and Collaborators
Direct co-funding from industry will be achieved to a value of £40k. There will also be satellite projects funded through the EPMA to a value of £60k. Suitable opportunities for co-funding through the TSB and the EU will be sought giving co-funding of £250k.

Details available on request.

### Deliverables

<table>
<thead>
<tr>
<th>Deliverable</th>
<th>Start</th>
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<td>Deliverable title:</td>
<td>Development of Ultra stiff test rig for fatigue measurement evidenced by case studies for the energy sector and hardmetal industries and a scientific paper.</td>
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<td>2</td>
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<td>Deliverable title:</td>
<td>Demonstration of strain measurement at crack tip and relationship to residual stress in sample evidenced by scientific paper on the initial stages of crack growth development at surfaces and the relationship to bulk residual stresses and good practice guide on in situ measurements reported to the BSSM and Forum for Eng.Structural Integrity</td>
<td></td>
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</table>
The Solution:

1. We will implement advanced manufacturing and process tools to engineer the degree of coupling coefficients by design, and in this way explore the influence of residual stress in thin films and interfacial effects in composites. We will explore the durability of magnetoelectric coupling with operational frequency in an attempt to relate electromechanical excitation to long-term microstructural fatigue and ME ageing. We have some critical IP based on MEMS processing routes [6].

2. We will advance the world leading measurement capabilities developed by NPL at the XMAS beamline at the ESRF, to measure crystallographic piezoelectric strain and magnetic properties in these devices. In situ, time resolved X-ray Magnetic Circular Dichroism (XMCD) measurements combined with an NPL commissioned piezoelectric ‘flipper’ device[7] will provide a direct measurement of the magnetisation, switched by the application of an external ac electric field, providing a clear link between the dynamic indirect ME effect & the microscopic origins of the strain mediated coupling.

3. Having optimised the ME coupling in thin film devices using MEMS processing techniques, we will produce a demonstrator based on an ultra-thin film ME wafer, redemonstrating piezoelectric ‘flipper’ device[7]. Having optimised the ME coupling in thin film devices using MEMS processing techniques, we will produce a demonstrator based on an ultra-thin film ME wafer, redemonstrating piezoelectric ‘flipper’ device[7].

Project Description (including summary of technical work): MEMS processing techniques will be used to manufacture ME heterostructures, with thin film (~100nm) PMN-PT and Co1-xFe3-x. Large ME coefficients (directly related to the sensitivity of the magnetic field sensing capability) have recently been reported in (Fe30Co70)Si3B10-AlN thin film composite structures of 737Vcm-1Oe-1 at resonance, higher than other reported values for bulk devices[8]. Thin film PMN-PT has an effective piezoelectric coefficient 20 times than that found in AlN[9]; recent reports of giant magnetostriction in Co1-xFe3-x show a magnetostrictive coefficient of 260ppm, with a low saturation field of ~10 mT[10], allowing for design of applications without the need for large external biasing fields. Further enhancements to the sensitivity can be achieved by back-etching of the silicon wafer, reducing the substrate clamping effects. Deliverable 1 concerns the verification and optimisation of ME coupling by

Summary: Multiferroic/magnetoelectric (ME) materials exhibit coupled magnetic and electrical polarisation, and are of interest because of their significant potential for novel applications in multifunctional devices, such as high-density memory storage, energy scavenging, security applications and oil & gas exploration. The functionality of these exciting new materials is critically dependent on the magnetic/electrical coupling, which is enabled by the transfer of strain between the two phases. Using MEMS (micro-electro-mechanical systems) technology we will design & build ME systems that inherently optimises magnetoelectric coupling and permits development of traceable metrology for their characterisation at the micro-scale. The work in this project will build on the current MEMS for Metrology project, and draw on NPL’s key strengths in ME metrology, to underpin and accelerate development of innovative new materials and devices.

The Need: For industry to adopt these materials there is a need for a much greater reliability of materials production & subsequent device durability than currently exist, & a better understanding of materials performance of the ME coupling at the micro- & nano-scale. To enable successful device design and integration of ME materials into miniaturised devices, manufacturers require a traceable measurement of ME coupling in thin films and micrometre-sized ME structures. The development of MEMS sensors has been identified as a major product driver across many industry sectors [1], with smart multifunctional sensors recognised as a rapid growth area for new applications[2]. Current state of the art ME multilayer laminar devices, comprise magnetic & piezoelectric phases and are typically tens of millimetres in size, exhibiting ME coupling coefficients up to ~40Vcm-1Oe1 with an equivalent magnetic field sensing resolution of 1pT/VHz[3]. Currently there are only two groups active within this research area: the Indian Institute of Technology[4] manufacturing single phase ME materials (with much lower coupling than we predict for out devices) on Si cantilevers, and a spin-out company from MIT[5] concentrating on wireless power transfer applications. In capitalising on our world-leading expertise in multifunctional materials characterisation, ME measurements and MEMS design, this project will accelerate exploitation of ME materials for the UK sensors industry. [1] ITRS 2010 Update p 16 [2] Piezo Institute 2010 Research Roadmap [3] Dong, APL, 87, 062502 (2005) [4] Prashanthi, Sens. Act. A, 166, 83 (2011) [5]ferrosoi.com

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<tr>
<th>Project No.</th>
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<td>Lead Scientist</td>
<td>Jenny Wooldridge</td>
<td>Stage Start Date</td>
<td>July 2012</td>
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<tr>
<td>Scientist Team</td>
<td>Markys Cain, Melvin Vopson, Mark Stewart, Mike Hall, Simon Brown (ESRF)</td>
<td>Stage End Date</td>
<td>June 2015</td>
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<td>Project Champion</td>
<td>Andy Marvin</td>
<td>Activity</td>
<td>Development of Existing Capability</td>
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</table>

Summary: Multiferroic/magnetoelectric (ME) materials exhibit coupled magnetic and electrical polarisation, and are of interest because of their significant potential for novel applications in multifunctional devices, such as high-density memory storage, energy scavenging, security applications and oil & gas exploration. The functionality of these exciting new materials is critically dependent on the magnetic/electrical coupling, which is enabled by the transfer of strain between the two phases. Using MEMS (micro-electro-mechanical systems) technology we will design & build ME systems that inherently optimises magnetoelectric coupling and permits development of traceable metrology for their characterisation at the micro-scale. The work in this project will build on the current MEMS for Metrology project, and draw on NPL’s key strengths in ME metrology, to underpin and accelerate development of innovative new materials and devices.

The Need: For industry to adopt these materials there is a need for a much greater reliability of materials production & subsequent device durability than currently exist, & a better understanding of materials performance of the ME coupling at the micro- & nano-scale. To enable successful device design and integration of ME materials into miniaturised devices, manufacturers require a traceable measurement of ME coupling in thin films and micrometre-sized ME structures. The development of MEMS sensors has been identified as a major product driver across many industry sectors [1], with smart multifunctional sensors recognised as a rapid growth area for new applications[2]. Current state of the art ME multilayer laminar devices, comprise magnetic & piezoelectric phases and are typically tens of millimetres in size, exhibiting ME coupling coefficients up to ~40Vcm-1Oe1 with an equivalent magnetic field sensing resolution of 1pT/VHz[3]. Currently there are only two groups active within this research area: the Indian Institute of Technology[4] manufacturing single phase ME materials (with much lower coupling than we predict for out devices) on Si cantilevers, and a spin-out company from MIT[5] concentrating on wireless power transfer applications. In capitalising on our world-leading expertise in multifunctional materials characterisation, ME measurements and MEMS design, this project will accelerate exploitation of ME materials for the UK sensors industry. [1] ITRS 2010 Update p 16 [2] Piezo Institute 2010 Research Roadmap [3] Dong, APL, 87, 062502 (2005) [4] Prashanthi, Sens. Act. A, 166, 83 (2011) [5]ferrosoi.com

1. We will implement advanced manufacturing and process tools to engineer the degree of coupling coefficients by design, and in this way explore the influence of residual stress in thin films and interfacial effects in composites. We will explore the durability of magnetoelectric coupling with operational frequency in an attempt to relate electromechanical excitation to long-term microstructural fatigue and ME ageing. We have some critical IP based on MEMS processing routes [6].

2. We will advance the world leading measurement capabilities developed by NPL at the XMAS beamline at the ESRF, to measure crystallographic piezoelectric strain and magnetic properties in these devices. In situ, time resolved X-ray Magnetic Circular Dichroism (XMCD) measurements combined with an NPL commissioned piezoelectric ‘flipper’ device[7] will provide a direct measurement of the magnetisation, switched by the application of an external ac electric field, providing a clear link between the dynamic indirect ME effect & the microscopic origins of the strain mediated coupling.

3. Having optimised the ME coupling in thin film devices using MEMS processing techniques, we will produce a demonstrator based on an ultra-high sensitivity magnetic field sensor. We will introduce a paradigm shift in sensor design via lateral separation of the piezo and magnetic phases that reduces the ME coupling uncertainty budget & provides for a step change in industrial exploitation, enabling NPL to characterise the coupling & create measurement artefacts of great precision. [6] Wooldridge et al., submitted to J. Micromech. Microeng. [7] Bouchenoire, Cain et al., AIP Conference Proceedings, 1234, 867

| Project Description (including summary of technical work): MEMS processing techniques will be used to manufacture ME heterostructures, with thin film (~100nm) PMN-PT and Co1-xFe3-x. Large ME coefficients (directly related to the sensitivity of the magnetic field sensing capability) have recently been reported in (Fe30Co70)Si3B10-AlN thin film composite structures of 737Vcm-1Oe1 at resonance, higher than other reported values for bulk devices[8]. Thin film PMN-PT has an effective piezoelectric coefficient 20 times than that found in AlN[9]; recent reports of giant magnetostriction in Co1-xFe3-x show a magnetostrictive coefficient of 260ppm, with a low saturation field of ~10 mT[10], allowing for design of applications without the need for large external biasing fields. Further enhancements to the sensitivity can be achieved by back-etching of the silicon wafer, reducing the substrate clamping effects. Deliverable 1 concerns the verification and optimisation of ME coupling by
traceable measurements of the induced voltage generated by magnetostrictive strain, using interferometric techniques with sub-pm resolution. In D2, we will work with our partners at ESRF, to perform time resolved XMCD measurements of the inverse ME effect. Following on from recent successful development of stroboscopic, synchronous electrical and XRD measurements for ferroelectric materials[11], we will make use of a piezoelectric switching device, built by NPL and already installed on the XMaS beamline, to capture the XMCD spectra at various points around the electrical loading cycle. Finally, in D3, a magnetic field sensor with sensitivity of 10fT/√Hz will be manufactured. Instead of depositing the piezoelectric and magnetic layers on top of each other, we intend to create 2D arrays of magnetostrictive beams to amplify the generated in-plane strains, and forces applied to the piezoelectric sensing component. The device will have near SQiUd levels of sensitivity, without the need for cryogenic cooling. NPL’s world-leading low magnetic field laboratory will be used to characterise the devices, to ensure high levels of magnetic field shielding. [8] Greve et al., APL, 96, 182501 (2010) [9] Herdier et al., Sens. & Act. A ,148, 122 (2008) [10] Hunter et al., Nature Comms, 2, 518 (2011) [11] Wooldridge et al., submitted to JSR (2012)

Impact and Benefits: The global market size for smart sensors is predicted to be $68 by 2015, & the use of MEMS technology is predicted to “revolutionise the smart sensors industry” [12]. The work carried out in this project will develop world leading traceable measurement standards for ME materials, required for the successful uptake of these new materials by industry. The potential range of applications in ME sensing is varied and numerous, including high-density memory storage, spintronics, magnetic field sensors & energy scavenging. Impact for the advanced instrumentation and metrology developed under this NMS proposal would include reduced time to market (2-5 yrs) for sensors in oil & gas exploration (magnetic sensing of nanoparticles for access to smaller reservoirs at reduced exploration costs), and extending device lifetimes and negating the need for secondary surgical intervention in replacing batteries for medical implants, for example. Other examples include enhanced bandwidth and data capacity for IT and comms based industries based on the development of ME filters, switches, and active resonators. These are the types of industrial applications and benefits envisioned following discussions held at recent workshops such as the IOM3/NPL workshop on nanorecification for energy harvesting, May 2011. See also Exploitation section below. [12]

Support for Programme Challenge, Roadmaps, Government Strategies: The multifunctional behaviour of MEs enable their use in a multitude of different applications, and as such are applicable to many of the key application sectors for smart materials identified in the TSB’s advanced materials strategy[13]. The development of high sensitivity uncooled magnetic sensors is strongly aligned with the NMS strategy on security, with potential applications for low observable technology in the magnetic detection of aircraft invisible to radar, infrared & sonar. Finally, the proposed work encompasses both the top level strategies outlined in the 2012 NPL Materials Division Strategy document, of materials metrology to sustain lifetime applications and materials metrology to enable emerging technologies, through the development of new and emerging materials and devices into UK business products. [13] http://www.innovateuk.org/ourstrategy/our-focus-areas/advancedmaterials.ashx

Synergies with other projects / programmes: ME materials and sensors are closely aligned to FP7 proposal Dynachip (modelling of high frequency multiferroics) and the development of NEMS in the EMRP Pathfinder project (Nanomagnetism & NEMS for multi-property single particle measurement). The measurement of nano-scale strain is also relevant to the 2012 IRD Nanostrain project, in evaluating the relationship between macroscopic and atomistic piezoelectric response of materials.

Risks: To enable production of successful MEMS devices, sufficient design iterations and manufacturing runs should be built into the project plan. Additionally, the efficiency of the ME coupling is entirely dependent on the availability active materials deposition. A commercial manufacturing route is sought to mitigate both these risks, in terms of quick times for process feedback and the quality of the functional thin films. In purchasing production services from a MEMS foundry, we are able to access extensive process libraries and manufacturing know how, without the need for major investment in development.

Knowledge Transfer and Exploitation: The advances in ME-MEMS generated by funding this work will be disseminated through publishing in high impact journals (Nature Physics, J Micromech. MicroEng., Sens. Act. A) & papers presented at global conferences (MRS, IEEE MEMS), and the development of a Piezo Institute training course. With the successful build of the magnetic sensor, IP will be patented, and a market review will be undertaken in collaboration with the Sensors KTN to assess the feasibility of a UK Ltd spin out company, to commercially exploit these new capabilities. A case study demonstrating the application of the magnetic field sensor will be carried out via the Technology Innovation Fund (& TSB) with one of the following UK companies: QinetiQ (military sensing), Bartington Instruments (magnetometers & gradiometers), Schlumberger (suppliers to BP for oil & gas exploration), TWI (non-destructive measurements), Seagate Technology (sensors in data storage).

Co-funding and Collaborators:
Details available on request.

Deliverables

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<th>End:</th>
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## Project No. | MAT\2012\17 | Price to NMO | £234485
--- | --- | --- | ---
### Project Title
Metrology for the growth in the use of and sustainability of permanent magnet materials
### Co-funding target
See MAT\2012\18 for co-funding already won
### Lead Scientist
Michael Hall
### Stage Start Date
Jan 2013

### Scientist Team
Stuart Harmon, Steven Turner, Brian Tate, Owen Thomas.
### Stage End Date
Dec 2015
### Est Final Stage End Date
June 15

### Sector
Advanced Manufacturing
### Activity
Methodology for New Capabilities
### Project Champion
Professor Andy Marvin

### Summary – Project part A to establish the wider metrology needed for MAT\2012\18 Part B
Existing NMS capabilities for permanent magnets (PMs) need to be developed for their future use. This project will establish a real world measurement method for PMs that will support green technologies and material security priorities. The metrology developed is a step change over traditional methods by making the measurements in air and using pulsed fields and is applicable to all magnet types. This provides measurement flexibility allowing actual magnet geometries and thicknesses, temperature behaviour, routes to economic recycling and high magnet throughput. These factors will help the UK capture more of the $17 bn (2020) market in NdFeB alone and lead International metrology in real world magnetic measurement methods. The growth in the use of permanent magnets (e.g. 9.9% CAGR for PMs in EV motors, wind turbines, metal recycling etc.) has produced an ‘unmet need’ within the supply chain as considerable volatility in the availability and cost of the magnets means industry must look for alternatives. Related to this are rare earth (RE) sustainability concerns and the efficient and timely development and use of alternative magnets requires the more complete characterisation offered by this method. For the method to gain industrial acceptance, the measurement uncertainty must meet industry needs and rigorous metrology is essential. The validation this requires will use and build on the existing capabilities and measurement expertise based in the same NMS laboratories.

### The Need
- The Critical Materials Strategy of the US DoE published in December 2011 showed the importance of permanent magnets to green technologies. To alleviate unsustainable demand for Dysprosium metal (increasing at 20 % per year) alternatives need to be found. Uses such as the traction motors for EV’s require 9 – 12 % Dy
- Massive investment in permanent magnet materials to establish a sustainable solution. US DoE announced funding of $31.6 million in October 2011. UK needs capabilities to support the UK PM industry growth potential this presents
- Growth has increased demand for an accurate (comparable uncertainty to existing standard method), high throughput measurement method with online potential (for recycling) for material and industrial R&D and QA
- The existing methods of electromagnet, VSM and SQUID cannot meet these demands
- Industry is using magnets with thicknesses of < 5 mm (necessary to reduce Eddy currents). Existing electromagnet measurement method requires ≥ 5 mm
- Measurements on shaped magnets used in machines are required. Not possible with the method of IEC 60404 part 5
- Thermal behaviour of RE magnets very important due to low Curie temperature (Decreases with reduced Dy). Not possible to modify the method of IEC 60404 part 5 without compromising measurement uncertainty too much
- More recycling of RE magnets required and online testing will be based on such an open circuit measurement

### The Solution
- The method of IEC 60404 - 5 available at NPL for over 30 years cannot be modified. A new method developed by NPL will build on these skills and provide fit for purpose validation to industry through an NMS led international study
- To overcome the thermal mass and AC losses of the magnetising yoke of this method, a step change in metrology from the static homogeneous flux to a rapidly varying (space and time) non-homogeneous flux condition is necessary
- This means developing an in air (open circuit) method with a target measurement uncertainty of 0.5%
- Establish a measurement methodology for final shape magnets with target measurement uncertainty of 1.0%
- Establish a method to control the temperature up to 200 °C that does not interfere magnetically with the measurement
- Implement low noise and high sensitivity magnetic sensors to enable measurement on test specimen volumes from 2 × 10^{-10} to 1 × 10^{-7} m³
- Establish traceable ways to calibrate the required magnetic field and sensor components
- Tools for Eddy current correction developed to obtain the DC properties needed for International trade
- Tools for self-demagnetisation correction developed to obtain material properties

### Project Description (including summary of technical work)
This project will consist of the following technical developments:
- Establish the test specimen geometries and temperature range through widespread consultation with stakeholders
- Design the solenoid and detection sensor arrangements that accommodates the range of dimensions
- Use FEA to obtain optimum uniformity for test specimen positioning, sensitivity and sensor coupling
- Establish temperature control without compromising the magnetic environment
- Calibrate the field source and magnetic sensors for the temperature range of interest
- Measure open circuit properties of a range of permanent magnet materials supplied by industry collaborators
- Correct for self-demagnetisation, eddy current and other dynamic effects that result from open circuit pulsed measurements and establish Material Property Analysis Routines (MaPAR)
- Compare open circuit material properties with results from IEC 60404 part 5 through an industry round robin
- Disseminate methodology to stakeholders and International community to promote NMS achievements
- Establish licensing arrangements with an instrument manufacturer for UK economic benefits

### Impact and Benefits
- Roland Berger Strategy Consultants forecast a global EV market size ranging from 0.75 to 2.55 billion Euro by 2020. Affordable and sustainable PMs are central to this and this project will enable UK SMEs to exploit this market
- Through a case study with the leading magnetic specialist Magnet Applications the improvement to their business through this measurement technique will be shown using metrics such as end user cost / kg of magnet material
- Support demand side initiatives like the UK government £400m programme for ultralow emission vehicles
- Delivering a ‘specification of an unmet need’ in providing the supply chain with the affordable magnets they require
- Lowing the cost of a key technology for electric vehicles will help overcome an affordability barrier for consumers, resulting in increased sales and the environmental benefits this brings
- Reduced environmental impact through recycling and remanufacturing using online measurements
- Measurement system requiring low skilled operation for industry
- 100% quality control possible for safety critical applications

### Support for Programme Challenge, Roadmaps, Government Strategies

### Synergies with other projects / programmes
Builds on the expertise in permanent magnet measurements established by the NMS. Meets the NPL strategy of establishing real world facilities and capabilities for the green economy. Capability will be part of the NPL Centre for Carbon Measurement.

### Risks
Main T risk is achieving an uncertainty low enough to achieve take up of the method by industry. This depends on how well corrections arising from making the measurements in air and using pulsed fields can be made. Mitigation is through comparison with established methods (in the same NMS laboratories) and collaboration with related project partners expert in modelling.

### Knowledge Transfer and Exploitation
New measurement service generating income of £30k per year. Offer a training course on magnetic measurements for real world conditions. MOTORE TSB project activities. Technical notes and articles to the Materials, Energy and Transport KTNs. International impact through IEC projects. Measurement system sales for other NMI’s. IP will be protected and licensed.

### Co-funding and Collaborators
Won MOTORE TSB project. EMRP Industry 2012 call for further co-funding. Details available on request.

### Deliverables

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<td>Deliverable title: Establish step-change in air metrology using pulsed fields to provide real world measurements. Use D1 as input to experimental arrangement: solenoid geometry; detection system arrangement for uniform coupling; detection method calibrations for operating temperature; self-demagnetisation correction process; dynamic correction processes; Material Property Analysis Routines (MaPAR).</td>
<td>01/08/14</td>
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<td>Deliverable title: Uncertainty for new real world measurements established through an NMS led international study. Through an NMS led international study compare, where possible (depends of magnet geometry and temperature overlap), new real world method with IEC 60404 part 5 to establish uncertainty. NMS report published and service opened.</td>
<td>01/01/13</td>
<td>31/12/15</td>
<td>£</td>
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D3 of Part B TSB funded project MOTORE (MAT\2012\18) activities. In addition, technical notes and articles to supply chain KTNs. UK industry case study. 2 UK Mag Soc articles. Presentation at UK Mag Soc seminar. Metrology input to IEC TC 68 projects. 2 journal papers. License MaPAR to industry.
The Critical Materials Strategy of the US DoE published in December 2011 showed the importance of permanent magnets to green technologies. To alleviate unsustainable demand for Dysprosium metal (increasing at 20% per year) alternatives need to be found. Uses such as the traction motors for EV’s require 9 – 12% Dy

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<td>Michael Hall</td>
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<td>Scientist Team</td>
<td>Stuart Harmon, Steven Turner, Brian Tate, Owen Thomas.</td>
<td>Stage End Date</td>
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<td>Sector</td>
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<td>Activity</td>
<td>Methodology for New Capabilities</td>
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<tr>
<td>Project Champion</td>
<td>Professor Andy Marvin</td>
<td>Est Final Stage End Date</td>
<td>June 2014</td>
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</table>
• Calibrate the field source and magnetic sensors for the temperature range of interest
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Support for Programme Challenge, Roadmaps, Government Strategies

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Risks
Main technical risk is achieving an uncertainty low enough to achieve take up of the method by industry. This depends on how well corrections arising from making the measurements in air and using pulsed fields can be made. Mitigation is through comparison with established methods (in the same NMS laboratories) and collaboration with related project partners expert in modelling.

Knowledge Transfer and Exploitation
Activities of the TSB project MOTORE including industrial and academic secondments to NPL to provide continuous training and KT. Technical notes and articles to the Materials, Energy and Transport KTNs. Industrial impact through International IEC projects. Exploitation through successful TSB projects, including RE recycling. Articles in the Magnews magazine of the UK Magnetics Society. Presentations at UK events such as UK Mag Soc seminars and International conferences. Progress announcements and feedback on Linkedin. Measurement system sales for other NMIs. IP will be protected and licensed.

Co-funding and Collaborators
Won MOTORE TSB project with partners Liberty Electric cars, Protean and Cranfield University. Details available on request.

Deliverables

| Deliverable title: Co-funding for TSB MOTORE project | Start: 01/07/12 | End: 30/06/2014 | Cost: £ |
The development of operational procedures for NDE inspection of composite components is critical for manufacturers to realise the full potential of composite materials. These materials offer high performance and reduced costs compared to traditional materials, but the detection, identification, and sizing of defects and damage that can reduce strength and stiffness are crucial for safe operation. Despite many advancements in NDE techniques, there are limited standards available, and perceptions of NDE techniques as too untried, costly, or complex hinder adoption.

**Project Title:** Development and Validation of Operational Procedures for Non-Destructive Evaluation (NDE) of Composite Components

**Lead Scientist:** Mike Gower

**Scientist Team:** Maria Lodeiro, Bill Broughton, Tim Young, Richard Shaw

**Sector:** Advanced Manufacturing

**Project Champion:** Roger Digby

**Activity:** Development of Existing Capabilities

**Price to NMO:** £738101

**Co-funding target:** £200k

**Stage Start Date:** July 2012

**Stage End Date:** June 2014 Changed to Dec 2013 due to the removal of 2 work pages

**Est Final Stage End Date:** June 2017

**Project Title Description:**

This project will develop and validate operational procedures for the inspection of a variety of composite components utilising microwave, pulse thermography and laser shearography non-destructive evaluation (NDE) techniques. The operational procedures will be developed from in-depth assessments of the probability of detection (POD) for different production and in-service defect types found in various composite material systems and formats, and will include sizing techniques equivalent to the 6 dB technique used in ultrasonic inspection. The suitability and robustness of each technique will be evaluated using a range of reference defect artefacts (RDAs) and where appropriate, round-robin (R-R) intercomparison exercises will be undertaken. In addition, ultrasonic C-scan procedures, developed in a previous programme, will be updated to reflect advances in ultrasonic inspection technology and capability. The key outputs from this project will be best practice guides containing operational procedures formatted as draft ISO standards for composite component NDE. The procedures will aid designers, manufacturers, and end-users to better detect and size defects within composite structures enabling more informed decisions to be made regarding defect criticality.

**The Need:**

The use of composite materials can provide manufacturers with a number of advantages including higher performance and reduced costs compared to more traditional materials. However, the detection, identification, and sizing of defects and damage that can reduce the strength and stiffness of a component and directly influence its safe working life is crucial. Composite structures are all issues that need still to be addressed before composites can be used to their full potential. Despite many innovations in the development of NDE techniques for the assessment of defects and damage, relatively few methods are commonly used. This is mainly due to the fact that standardisation operational procedures are not available and perceptions that NDE is too untried, costly, or complex. Whilst a plethora of composites and NDE ISO standards exist, there are currently no ISO NDE standards in existence that are specific to defect detection in composites. Several ASTM composite NDE specific standards are available, but these tend to be focussed on the aerospace sector and do not provide enough detail and validated data addressing issues such as probability of detection, size and location sensitivity. In order to increase industry adoption and realise the potential benefits of promising NDE techniques such as microwave, pulse thermography and laser shearography, there is a need to develop operational procedures that are based on a thorough evaluation of each technique for detecting a range of defects found across several sectors e.g. aerospace, wind energy, oil and gas, and civil infrastructure. The need for operational procedures/standards for NDE inspection of composites has been evidenced through consultation with industry (e.g. ESR Technology, Dantec Dynamics, LOT-Oriel and Evisive Inc).

**The Solution:**

The development and validation of operational procedures, that will act as precursors for standardisation, for microwave, pulse thermography and laser shearography NDE techniques for polymer matrix composite systems. These techniques are relatively novel and have shown promise (in previous projects TSB ‘IMAJINE’, F12 and 1024) for the inspection of composite materials that currently cannot be inspected using conventional methods such as ultrasonics or X-radiography. In the case of microwave inspection, the technique has seen limited use for inspection of HDPE water pipes, composites used in oil and gas applications, and assessment of bond integrity of fibre-reinforced plastic (FRP) strengthened structures such as bridges. The technique is ideally suited to the inspection of non-aerospace grades of GFRP that tend to have high levels of porosity (>2%) and/or that significantly attenuate ultrasonic signals. Ultrasonic C-scan procedures, developed in a previous programme (CARAD scheme of the DTI), will also be updated to reflect advances in ultrasonic inspection technology and capability. The formulation and standardisation process used for the ultrasonic C-scan methods, will be adopted for microwave, pulse thermography and laser shearography techniques. The metrology aspects of this project are as follows and will be addressed for each NDE technique: (i) comprehensive assessments of the probability of detection (PoD) for different defect types found in various composite material systems and formats, (ii) establish the limits of detection for each technique i.e. the minimum sizes of defects that can be detected and (iii) the development of damage/defect sizing techniques equivalent to the 6 dB technique used in ultrasonic inspection. The suitability and robustness of each technique will be evaluated using a range of reference defect artefacts and where appropriate, R-R intercomparison exercises will be undertaken. Funding is requested from the NMS to enable NPL to underpin the advancement and industry adoption of standardised NDE methodologies and procedures to the benefit of the UK composites community. NPL is uniquely suited to undertaking this work as it has an established track record in the development of operational C-scan procedures (published in INSIGHT) and numerous composite ISO standards. It also represents the UK’s interests on ISO standards committees and chairs the VAMAS TWAS Polymer Composites working group.

**Summary:**

This project will develop and validate operational procedures for the inspection of a variety of composite components utilising microwave, pulse thermography and laser shearography non-destructive evaluation (NDE) techniques. The operational procedures will be developed from in-depth assessments of the probability of detection (POD) for different production and in-service defect types found in various composite material systems and formats, and will include sizing techniques equivalent to the 6 dB technique used in ultrasonic inspection. The suitability and robustness of each technique will be evaluated using a range of reference defect artefacts (RDAs) and where appropriate, round-robin (R-R) intercomparison exercises will be undertaken. In addition, ultrasonic C-scan procedures, developed in a previous programme, will be updated to reflect advances in ultrasonic inspection technology and capability. The key outputs from this project will be best practice guides containing operational procedures formatted as draft ISO standards for composite component NDE. The procedures will aid designers, manufacturers, and end-users to better detect and size defects within composite structures enabling more informed decisions to be made regarding defect criticality.
through which pre-standardisation work of the procedures will be conducted. The group already collaborates with academic teams developing NDE techniques i.e. with Manchester University developing X-ray CT techniques for measurement of porosity.

**Project Description:** The project comprises of three key activities: (i) Design and manufacture of reference defect artefacts (RDAs) that are representative of the materials and defects typically found in, and of concern to the aerospace, oil and gas, renewable energy, marine and civil infrastructure sectors. RDAs will be designed in collaboration with suitable organisations in each sector. This activity will also update the ultrasonic C-scan procedures developed in a previous programme to reflect advances in ultrasonic inspection technology and capability. In particular the procedures will be updated to include guidance for the scanning of 3D components. The updated C-scan procedures will be submitted to ISO for standardisation, (ii) This activity will develop operational procedures for microwave, pulse thermography and laser shearography techniques. The procedures will be developed via a series of comprehensive trials using the RDAs developed in activity (i). For each NDE technique, key factors such as sensitivity (size of defect that can be detected), PoD, coverage and speed of inspection will be evaluated. The operational procedures will be written in the form of ISO standards, (iii) A series of R-R intercomparison exercises will be conducted in which participants will be required to inspect RDAs using in-house techniques and then using the operational procedures developed in (ii). The results of the R-R exercises will be used to refine the operational procedures and will be reported in a scientific paper.

**Impact and Benefits:** Manufacturers and asset owners of wind turbines, oil & gas plant, marine transport and civil infrastructure will benefit through validated operational procedures for a range of promising NDE techniques, resulting in more energy efficient, safer and longer life structures, with reduced operation, maintenance and repair (whole-life) costs. The procedures are expected to be adopted by industry within 2-3 years of project completion. Training is seen as one of the key factors limiting the uptake of NDE of composite components. The production of validated operational procedures will increase the knowledge base of composite inspection best practice and aid organisations to provide appropriate training, thus promoting the use of novel NDE techniques.

**Support for Programme Challenge, Roadmaps, Government Strategies:** This project aligns directly with:
- NMS Composites strategy and roadmap in that it is designed to address the market drivers of increasing the use of sustainable and multifunctional composite materials.

**Synergies with other projects / programmes:** The project builds on NMS projects F12 “Development of Test Methods for Determining the Criticality of Defects in Composite Material Systems Under Long-Term Loading” and MAT1024 “Development of NDE and SHM Techniques for Monitoring Defects And Chemical Changes in Composite Structures”, and Technology Strategy Board (TSB) projects ACLAIM (Advanced Composite Life Assessment and Integrity Management” and IMAJINE (Innovative Multi-materials Jointing and Integrity Management). Beyond the current project, it is planned to progress ISO standardisation of the operational procedures developed for microwave, pulse thermography and laser shearography techniques.

**Risks:** The main technical risk is that it will not be possible to source a laser shearography system (~£70k) due to insufficient capital. This risk has been mitigated by obtaining the support of Dantec Dynamics Ltd. who has agreed in principal to collaborate with NPL and provide in-kind contribution in the form of inspections of RDAs. Another perceived risk is that the inherent variability of non-aerospace grades of composite material may limit the suitability of candidate NDE techniques to certain material types. A range of material formats will be inspected and the limitations of each NDE technique (if any) will be identified which will form the basis of best practice for composite component inspection and the operational procedures.

**Knowledge Transfer and Exploitation:** Principally through the production of draft procedures that will be included as annexes in best practice guides (intended for future standardisation), scientific papers (INSIGHT – journal of the British Institute for Non-Destructive Testing (BINDT), ICNDT and associated conferences) and trade articles (NDT News). The intended audience will comprise of designers, manufacturers and end-users of composite components and structures in multiple sectors (energy, aerospace, automotive, renewable energy, marine and construction). In addition project outputs will be disseminated and exploited via the Composites IAG, measurement services and consultancies.

**Co-funding and Collaborators:**
Details available on request.

<table>
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Design and manufacture of reference defect artefacts for inspection trials undertaken in D2. Update of ultrasonic C-scan procedures and progression of standardisation through ISO.
Summary
New technologies are being developed that will enable reductions in energy consumption and improvements in efficiency and reliability in automotive, energy, process, electronics and medical industries through the use of new functional materials which operate at elevated temperatures up to 1000 °C. These applications exploit the actuation, sensing and cooling functionality of ferroelectric materials which result from strong coupling between electrical, thermal and mechanical properties. Degradation of materials properties at high temperature means that these applications are currently limited to operating temperatures below 200 °C. New solid-state cooling technologies are emerging based on the electrocaloric effect that will provide efficient cooling for computer chips and domestic and industrial refrigeration. New materials technologies are emerging to meet these needs, but are not currently supported by a metrological framework for traceable measurement of the coupling at high temperatures.

The Need
Reliable, accurate and traceable measurement of electro-thermo-mechanical coupling at high temperatures is essential to provide the data required for the development of new materials technology, effective design of new devices, reliability in characterisation and test, and to ensure quality in manufacture and reliability in service. This requires a robust measurement infrastructure that is traceable to national and international standards for SI units providing measurements under industrially relevant harsh environments which does not currently exist. This need is recognised by our collaborators from European aerospace, automotive, power, oil & gas and medical sectors and a manufacturer of test and measurement equipment participating as an unfunded partner.

The Solution
Develop the metrological infrastructure and facilities within Europe for the traceable metrology of coupling between thermal, mechanical and electrical properties in new materials technologies at high temperatures and high electric fields.

Project Description (including summary of technical work)
- Develop traceable measurement of linear and non-linear coupling between electric displacement and strain to the electric field, stress and temperatures up to 1000 °C based on non-contact methods.
- Develop system for the direct traceable measurement of the electrocaloric effect in bulk and thin film materials from 20 °C to 250 °C, and at high electric fields (the SRT call is for up to 1000 °C).
- Develop facilities for the traceable measurement of thermophysical properties up to 800 °C and radiometric properties up to 1000 °C.
- Develop models of the coupling between thermal, electrical, and mechanical properties in ferroelectric and related materials, and their application to the metrology of electrocaloric and piezoelectric properties. Models will include non-linear contributions such as electrostriction and effects of high electric fields.

Impact and Benefits
This project will provide the metrology required for the development of new technologies that will lead to more efficient and reliable transport and power generation e.g. through piezoelectric control of fuel flow in aero-engines and high temperature sensing and integrity monitoring in steam facilities. New solid state cooling technologies would reduce greenhouse gas emissions from refrigeration and enable faster electronics through on-chip thermal management. These innovations would provide a competitive advantage and stimulate growth in industries that are strong within Europe, thoroughly represented by our industry collaborators.

Support for Programme Challenge, Roadmaps, Government Strategies
This activity supports the NMO strategy particularly with regard to the objectives of supporting energy, sustainability and supporting innovation through excellent science applied to meeting these national challenges. The project supports the NPL multifunctional materials roadmap – extending functional measurements to high temperatures and EMRP 2008 Outline (new materials) and EMRP 2011 SRTn13 call.

Synergies with other projects / programmes
This project builds on capability developed in FM04 – actuators in harsh environments and TSB project APAHOE. Harsh environments, particularly temperature, are a factor in most practical implementations of functional materials and are a consideration in most projects undertaken by this group. This project will therefore inform existing and future projects in energy harvesting (current EMRP, future TSB/FP7), energy storage (current TSB / NMS hypercapacitors) and non-contact sensing.
Characterisation methods for multifunctional materials under extreme conditions & extreme environments has been identified by the Piezo Institute Roadmap (2009) as a key area for future EU framework activity.

### Risks

- **Electrodes.** Breakdown of conductive electrodes and electrical connections at high temperature or diffusion into the functional material is a significant risk. Mitigation is by specifically including study of electrodes both in terms of their effectiveness at high temperature and possible effects of the functional performance of the materials. Project partners have been chosen to include expertise and experience in this field. Solving this problem is a key technical challenge not just for metrology, but for commercial exploitation as well.

- **Conductivity.** Most piezoelectric ceramics are wide band gap semiconductors and become significantly conductive at elevated temperature. High levels of conductivity make high field measurement difficult and can increase uncertainties in the measurement of low field electromechanical coupling. As above, mitigation is by directly tackling this challenge within the technical work and appropriate choice of project partners. Again, this is a key challenge in the development of exploitable high temperature piezoelectrics.

- **Optical path disturbances.** Large temperature gradients in the vicinity of a furnace imply large gradients in, and potentially large fluctuations in refractive index due to convection currents. This will increase uncertainties in measurement and/or increase integration time thus restricting the frequency range achievable. Mitigation is through a staged approach with successively increasing temperature targets. Full capability could therefore be developed for a lower temperature range even if some compromises were required at the extreme high temperature.

### Knowledge Transfer and Exploitation

This will be achieved through the delivery of information exchange with our collaborators (including nine companies spanning this range of applications), training, workshops, web-based dissemination and publications as well as through our unfunded partner who will be able to develop new instruments for industry and research based on the outputs of this project. Scientific output which will be published in high impact factor scientific journals.

### Co-funding and Collaborators

Co-funded by EMRP 2011 New Technologies project NEW09. Project partners are PTB, LNE, MIKES, CMI, aixACCT Systems GmbH, University of Leeds. Details available on request.

### Deliverables

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**Deliverable title:** Cofunding for EMRP project NEW09 METCO
Exfoliation of corrosion products is a major concern for high temperature steam plant used in energy generation, chemical industry etc. Damage from exfoliation can take many forms, tube blockages, short/long term overheating, erosion and tube bursts. Where the scale does not exfoliate, it is used as an NDE metric of tube temperature via measurement with ultrasonics. Understanding the growth kinetics, thermal properties and scale morphology, particularly the formation of laminations, is vital to accurate measurement of material performance and life time management, in terms of understanding how the oxide thickens its insulating properties and also when to expect exfoliation. Within this project the cycling of materials as a function of temperature and pressure will be evaluated to assess the criticality of this in terms of oxide growth and morphological feature development. Additionally Thermo Gravimetric Analysis and high resolution microscopy will be used to examine the effect that surface condition has on the early stages of oxidation. A method of measuring the steady state thermal properties of scales will also be developed and demonstrated on a range of alloys and oxide thicknesses.

### The Need

The corrosion rate for high temperature materials in aggressive environments is a critical parameter which governs material performance, component lifetime and process efficiency. Steam plant remains key to energy security in the UK whether that is via fossil, biomass or nuclear steam generation. With renewable energy coming online there will be an increased requirement for fossil plant to two-shift (come on line quickly should demand rise, wind stop blowing etc.), placing structural components under increasingly demanding thermal workloads, which will affect plant life. The exfoliation of steam grown scales is likely to be accelerated due to increased frequency of thermal transients and metal wastage rates will increase, especially as more austenitic materials are used as the requirement for higher temperatures increases [1]. With the cost of an unplanned outage in excess of £500K per day (£1-2m to repair a tube failure [1]) there is an increased requirement for accurate models of oxide growth, void formation and the effect this has on heat transfer, strain distribution and exfoliation, which in turn require more accurate simulation within the laboratory of plant conditions, to improve the comparability of laboratory scale to service grown scales [1]. Recent work has shown that laboratory testing, is still not replicating the morphology of plant grown scales, and that significant differences still occur in the measurement of oxidation kinetics between different laboratories [2]. It is believed that this is due to initial surface specific phenomena during the early stages of oxidation. Improved methods to better replicate service scales are needed to fully understand exfoliation phenomena and improve modelling of the scale.

[1] 2nd EPRI-NPL Workshop on scale exfoliation from steam-touched surfaces  

### The Solution

It is widely agreed that simplistic tests will not produce the required morphologies and additional levels of complexities are required [1]. Attempts to address the additional complexities such as strain in the scale through growth strains or applied strain have been tried using test loops attached to boilers, but with a lack of precise control and stability of test conditions provide little definitive measurement of the kinetics and morphology. Strain can be applied axially or radially in tubes and this needs simulating under controlled laboratory conditions as it will impact on the defect structure in the scale. These defects also affect heat transfer and this will be quantified and the effect included in models. Development of a high-pressure exposure rig capable of thermal and pressure transients at much higher pressures, relevant to USC plant, than currently available, would establish the effect of static and fluctuating pressure has on the oxidation kinetics and morphological formations such as laminations in the scale. This would increase the UK capability from 75 to 250 bar and be a unique UK facility assessable to industry and academia. A method of measuring the axial heat flow under steady state conditions will also be developed and used to quantify the thermal resistance of oxide scales, and the effect laminations and voids have on the heat transfer.

### Project Description (including summary of technical work)

TGA experiments in flowing steam will be conducted to study the early stage oxidation of model and commercial ferritic and austenitic alloys. The microstructural evolution will be investigated using the dual beam FIB on samples exposed for varying durations. A facility for exposing materials under conditions of fluctuating temperature and pressure will be designed and constructed and used to determine the effect temperature and pressure transients (changes in radial strain) have on the exfoliation on flat, curved and tube sections. The morphology of the scales grown in this project will be critically compared to plant scales, and recommendations for more complex oxidation testing proposed. An axial heat flow meter will be modified and characterised. This will be used to generate data on the thermal properties of the scales as a function of morphological features.
**Impact and Benefits:** The improved exposure and test facilities from this project will lead to enhanced understanding of oxidation morphologies and greater comparability between laboratories in the generation of kinetics data. This will enable the development of improved models to provide better component lifetime prediction and plant management, in an industry where an unplanned outage costs in excess of £500K per day (£1-2m to repair a tube failure [1]). This work would also impact UK energy security by understanding and modelling material performance under more aggressive two-shifting and even three-shifting regimes and provide the UK with the scientific tools to support renewable energy sources using fossil energy as a flexible base load. Uncertainties in oxidation kinetics and TGA measurements will also benefit from improved measurement techniques and better comparability between laboratories, leading to future standardisation of TGA measurements. Impact will be demonstrated through linking the plant grown scales to those generated using these more complex cycling exposures and illustrating the similarities via the EPRI Oxidation Atlas [1].

**Support for Programme Challenge, Roadmaps, Government Strategies:** This project supports the need for clean and secure energy within the UK as outlined in the Energy White Paper, and aligns with Governmental commitments to the European targets for 2020. It also aligns with programme strategies regarding the reduction in carbon and the increased use and support for renewable energy, by supporting the conversion of coal plant to co-fired biomass. The development of these capabilities feature in the Materials Programme Strategy and the Advanced Engineered Materials Road Map.

**Synergies with other projects / programmes**
This project builds on the previous road mapped activities on oxidation and surface degradation and has synergy with the TSB project COMPACT and associated 2011 NMS project (subject to funding of COMPACT) and the proposal to develop a model of oxidation and void formation (proposed by Dinsdale et al.). This also links with activities with the SuperGen consortium and UK/US collaborative work on exfoliation and the Atlas of microstructures activity arising from the recent NPL-EPRI workshop [2]. The EU MACPLUS project evaluating steam side coatings and the work being performed within AUSPLUS also are relevant.

**Risks**
The main risk associated with this project is in the development of a flowing steam oxidation facility at higher pressures than currently available. Lessons have been learnt from the current lower pressure equipment and design experience from colleagues at NETL can be applied to produce a suitable rig. There is also a risk that suitable accommodation for such a piece of equipment could not be found, but this will depend on the working volume of high pressure steam being used. There are additional risks in the use of steam in TGA equipment but experience has shown that with sufficient care damage to equipment can be avoided.

**Knowledge Transfer and Exploitation**
Exploitation of the project and KT will be realised through conference presentations and journal papers. A key international conference in 2013 will be targeted for a high impact paper. Focussed working groups will be established to address the different aspect of steam oxidation and surface degradation. The capability developed will be demonstrated through a focussed series of tests and interaction with the SuperGen consortium and through case studies publicised through Measurement Notes and social media announcements. This will take the form of a ‘studio’ type project with industrial support. The facility will be available after the project to provide bespoke testing for industry and academia. KT to the wider EU market will be performed through interaction with the KMM-VIN, a follow-up to the COST network, which consists of over 50 EU organisations covering the complete supply chain for steam raising plant (material supplier, manufacturer, OEM’s and end user).

**Co-funding and Collaborators**
Co-funding from the ASPIRE and COMPACT TSB projects can be expected with additional contributions from MACPLUS and Supergen (in-kind). Details available on request.

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<td><strong>Deliverable title:</strong> Evaluation of microstructural features and the influence they have on the development of oxide scales, demonstrated through a journal paper.</td>
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<td><strong>Deliverable title:</strong> Thermal resistance data of corrosion layers demonstrated via a project report and peer reviewed paper.</td>
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The project is concerned with the development of a validated model for the corrosion of steel boiler tubes by high pressure steam. If successful this will lead to an extension of the lifetime of existing power generation plant, more efficient operation and reduced environmental impact.

The Need
Corrosion of steel boiler tubes used in fossil fuel power generation by high pressure steam leads to failure of the tubes, blockage from exfoliated material and damage to turbine blades. This results in plant failure, shutdowns and costly maintenance procedures. The cost of an unplanned shutdown is in excess of £500k per day. Use of reliable, validated models to predict formation of oxide scales and subsequent exfoliation would limit such shutdowns and allow plant to run towards optimal efficiency. At present there is no comprehensive model to describe how layers of oxide form on the surface of the steel under these aggressive environments. Current models are limited to simple stoichiometric oxide and ideal gas phases and do not take account of the mechanism of diffusion through the crystal lattice sites. There is a need to develop a much more detailed model which can predict which oxides can form under different conditions, and the conditions eg thermal cycling, pressure variation and pre-treatment of the surface of the steel which affect a loss of mechanical integrity leading to spallation [1]. Such a model could also be used to identify ranges of conditions and materials less prone to aggressive attack and develop criteria where exfoliation is likely to take place.

[1] 2nd EPRI-NPL Workshop on scale exfoliation from steam-touched surfaces

The Solution
The aim of the model is to predict the formation of oxides on the surface of steels under aggressive environments such as high pressure steam, how quickly these phases form and their thickness. The model will also provide information about the nature and composition of these oxides and how voids form at the interface between steel and oxide layer. Such a modelling programme will require a detailed knowledge of the chemical thermodynamics of the steel and oxides, the chemical diffusion of elements and ions through the phases and along grain boundaries and densities and expansivities of the various phases. The model should be validated against measurements and experience obtained in UK power generation plant for both austenitic and ferritic steels. NPL is well placed to develop such a model in view of its established expertise in the critical assessment of thermodynamic data and their use in calculating phase equilibria and in the experimental study of corrosion of materials. No other organisation in the UK has this breadth of expertise. The present project complements the proposed project “Improved measurement of high pressure and early stage corrosion rates” which will provide reliable data for such validation. Once validated the model will be used to identify ranges of conditions and materials less prone to aggressive attack and develop criteria where exfoliation is likely to take place. NMO funding is appropriate because of the precompetitive nature of the work and its potential to develop a facility at NPL for the prediction of corrosion by aggressive gaseous environments.

Project Description (including summary of technical work)
- Development of a detailed knowledge of thermodynamics of the steel and oxides, the chemical diffusion of elements through the phases and along grain boundaries and densities and expansivities of the various phases.
- Development of a kinetic model for the chemical diffusion of atoms and ions through the ferrite and austenite phases of steels, magnetite, haematite and iron chromite spinel before reacting with oxygen and water species present in high pressure steam. Implicit in the model is the generation of voids at the interface between steel and oxide.
- Validate model against typical ranges of conditions and materials used in current power generating plant.
- Explore ranges of conditions to identify where problems of corrosion and exfoliation would be reduced.

Impact and Benefits
The validated model would allow the power industry to identify possible surface treatments, ranges of operating conditions to avoid extensive corrosion and spallation. It would also help to predict when remedial action should be taken to avoid blockages and the transport of harmful dust to the turbines. The model would also help guide the choice of steels for future power generation.

Support for Programme Challenge, Roadmaps, Government Strategies
This project supports the need for clean and secure energy within the UK as outlined in the Energy White Paper, and aligns with Government, European targets for 2020. It supports the NMO strategy through supporting “the achievement of carbon reduction targets to address climate change by providing the underpinning measurement infrastructure needed to verify reductions in energy consumption and improvements in energy efficiency” and to “assist the drive towards greater energy efficiency through measurement research to improve energy management”. It also supports the Energy and Sustainability challenge by underpinning predictive models for improvements in efficiency and extension of the operational lifetime of fossil fuel plant. Sustainability of resources is addressed by creating a capability to aid in the design of new materials using less energy, controlled corrosion resistance and using elements with secure supply. This project sits within one of NPL’s key areas of science, “modelling to characterise materials”.

### Synergies with other projects / programmes

This model will complement the proposed project “Improved measurement of high pressure and early stage corrosion rates”. There are also links to an existing TSB COMPACT project, Supergen and to UK/US collaborative work on exfoliation and the Atlas of microstructures activity arising from the recent NPL-EPRI workshop [1].

### Risks

Identified risks include:

- Lack of experimental kinetic data - in this event missing data will be estimated
- The development of the model may be more complicated than expected - Initial work will be concerned with 2 or 3 component steels with simple diffusion model. The model can become more complex as confidence in the model grows.
- Available computation resources may be insufficient – initial work will be based on workstations. If calculations are too slow a multiprocessor blade-server (already purchased) will be used

### Knowledge Transfer and Exploitation

The results of the project will be disseminated through peer reviewed scientific papers and articles in trade journals such as Materials World. A case study will be developed in conjunction with the UK power generation industry. Progress and results will be presented at the biannual Power industries IAG meetings which will also provide some steer. At the completion of project it is envisaged that the model would be developed sufficient to offer a service of calculation to industry.

### Co-funding and Collaborators

Future co-funding as a result of the NPL-EPRI workshop is expected with joint projects coming from the recommendations from the workshop. Further co-funding will be sought through TSB projects. Details available on request.

### Deliverables

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<td>Deliverable title: Development of model for oxide growth including critical assessment of necessary kinetic and thermodynamic parameters, demonstrated by peer reviewed paper</td>
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<td>Deliverable title: Validation of model against experimental data and plant experience, demonstrated through article in trade journal such as Materials World</td>
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Erosion whether by high temperature solid particles (HTSPE) or water droplet is a major concern globally in high temperature plant across many sectors including power generation, aerospace or oil and gas. In the power industry only this form of degradation costs an estimated US$150 million a year in lost efficiency, forced outages and repair costs [1]. There is a requirement for improved test methods and mechanistic models for high temperature erosion. This project will develop HTSPE capability and use it alongside existing water droplet erosion and low and high frequency wear rigs to investigate the degradation mechanisms of three distinct erosion and wear phenomena. Using this information, the feasibility of developing a model based on microstructural and mechanistic observations will be explored.


The Need
Erosion and wear can dramatically reduce the efficiency and life of high value components across a range of industrial sectors. A recent EPRI (Electric Power Research Institute) survey [2] of HTSPE testing exposed a serious deficiency within the UK to perform HTSPE tests, an area of increasing importance in power generation and aerospace (for example with erosion of refractory materials, valve stems and turbine blades by exfoliated scale, volcanic ash and CMAS (calcium magnesium alumino-silicate)). Additionally recent novel work at NPL has shown that the presence of steam influences surface degradation during wear. The precise mechanism of this influence on the wear properties is not fully understood and the uncertainties in the measurement not fully quantified. Understanding these processes and evaluating the uncertainties inherent in the test is vital for understanding and characterising surfaces under these conditions. A recent NPL review [3] of modelling used in UK industry for wear and erosion has illustrated the need for more microstructural and mechanistic based models to better simulate erosion. This project will focus on two forms of erosion namely HTSPE and water droplet erosion. At present many of the models used are simplistic and rely heavily on experimental data and fitted parameters. Where more complex models do exist, these are developed for specific systems and are unable to predict the mechanisms of erosion in a generic manner.


The Solution
Whilst there are facilities which can perform HTSPE, they are not fully instrumented, becoming old and unreliable and most importantly the EPRI survey revealed that they are unable to perform tests at temperatures and velocities which are becoming of greater interest to a widening section of industry. The capability to perform these tests needs to be developed and reliably instrumented such that the measurements and the mechanistic degradation process fully understood. The fundamental underlying metrology required will be challenging involving temperature measurement of particles, velocity measurement, high resolution microscopy and 3D microscopy of wear scars, requirements which NPL is ideally equipped to address. There is also a requirement for a systematic study of the repeatability and uncertainties associated with low and high frequency wear testing in air and steam to improve the measurement interpretation and application. Such improvement requires quantification of the mechanisms and microstructural features of the processes. By understanding these, the feasibility of developing modelling approaches for erosion will be assessed for their capacity to be applied to a broad range of conditions rather than focussing on one industrial aspect. It is anticipated that models to perform this task will need to encompass the physical and mechanical properties of the scale, substrate and particulates, relevant failure criteria for element deletion (such as pressure stress thresholds) and fracture energies. Using NPL’s experience in both FE and mesh free methods, these techniques can be coupled to draw on the advantages of each method. For example using a mesh free area to overcome FE mesh distortion issues in the impact regions.

Project Description (including summary of technical work)
A HTSPE test facility will be designed and constructed to meet and exceed the requirements of industry as demonstrated through the EPRI intercomparison requirements. This apparatus will be capable of performing tests at 950°C with particle speeds of 250 m/s. Erosion (HTSPE and water droplet) and wear tests will be performed on representative model systems, including coatings, to determine the repeatability and uncertainty inherent in HTSPE, water droplet erosion and low frequency wear in steam atmospheres. Optical and high resolution electron microscopy will be used to monitor the evolution of the degradation mechanism which are characteristic to the different erosion and wear tests. This microstructural and mechanistic information
will be used to develop a model for erosion based on mechanistic phenomena rather than through curve fitting procedures.

Impact and Benefits Improved measurement procedures and understandable uncertainties in the test methods will increase the confidence and applicability of the results. This will lead to better understanding of HTSPE and water droplet erosion which will give improved measurement and characterisation of wear and erosion. This will allow for development of accurate models to enable better assessment of in-service components and allow the design of improved systems. This will lead to cost savings in industry through improved efficiency, improved design and as a consequence increased lifetime of components. For example reducing the erosion of the leading edge of turbine blades would result in the improved efficiency and thus avoid, in the case of a large power plant (~800 MW), the emission of an extra 250,000 tonnes of CO2 over the lifetime of the plant. Impact of the project will be demonstrated through specific case studies on HTSPE and water droplet erosion. The knowledge and capability developed in this work will additionally be beneficial to other programmes, such as land and marine turbines and aerospace, as a bespoke source of measurement and material characterisation.

Support for Programme Challenge, Roadmaps, Government Strategies This project addresses areas of strategic importance and aligns with the Advanced Engineered Materials road map and strategic direction. It addresses key programme challenges and governmental strategies outlined in the Energy white Paper and the Electricity Market Reform white paper 2011. It also addresses the Materials Programme Strategy key themes of: Growth: by improving the understanding, measurement and modelling of advanced engineered surfaces this project will aid high value manufacturing of components spanning the length scales, from low pressure turbine blades to piston heads and stem valves. Energy: addresses material properties in harsh environments (e.g. rotating hardmetal seals, drill heads, oil and gas exploration and recovery, steam turbines, aero engines and nuclear power). This project will also provide missing infrastructural testing capability to UK industry in terms of HTSPE capability.

Synergies with other projects / programmes This project links with recent activities on scale exfoliation and steam oxidation and complements fireside corrosion projects addressing fly ash erosion of coatings used in bio mass boilers (2011 NMS project, TSB project ASPIRE and joint Eng.D student with E.ON), and with revisions of the ASME design codes for boilers of which erosion plays a key part. The modelling work uses the expertise developed in the grains to structures project and will provide a case study as that work develops. This would also link to future EMRP proposals in which HTSPE will be submitted, allowing more interaction with EU NMI’s in the future.

Risks: The main risks relate to the development of the HTSPE equipment, and being able to achieve the necessary velocity of the particles. The team has a long history of design of this type of apparatus and good links with manufacturers so mitigation strategies could be found using alternative approaches to the current plans which would require alternative particle heat methods. Modelling may be too complex to achieve, modelling will start on a basic level with additional complexities added. EMRP proposal not successful, the team has good links with other NMI’s and experience of formulating successful bids.

Knowledge Transfer and Exploitation Exploitation of the project and KT will be realised through conference presentations and journal papers. NPL has already been invited to present at an EPRI workshop in Milan on this subject and will build on this with a view to the EPRI advanced materials conference in 2013. Focussed working groups to address the different forms of erosion are planned and will have contributions from the high temperature degradation IAG and Surface Engineering IAG, so that as representative a cross section as possible is addressed, these meetings will be publicised and will be open to other attendees. Measurement notes on the exposure and measurement techniques will be disseminated through appropriate special interest groups though platforms such as KTNs (Materials, Energy generation and supply), Linked-In, etc. The capability will be demonstrated through a focussed series of tests, which will form case studies for different forms of erosion and wear. Additional interactions through the UK/US collaboration and the SuperGen projects will also be sought.

Co-funding and Collaborators: Collaboration with EPRI through the intercomparison exercise is expected, with NPL already being invited to present at an International workshop of erosion in Milan in 2012, leading to a co-funding project expected to be in the order of £50k. Details available on request.

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Summary

Project is NMO-funded component of EMRP project NEW04 that was successful in the November 2011 EMRP funding competition. Objectives are to develop novel approaches to measurement uncertainty evaluation and to enable their consistent application, illustrated by appropriate case studies. The dissemination of these methods is ensured by providing input for future revisions of the Guide to the Expression of Uncertainty in Measurement (GUM), its supplements and other relevant documents and by providing algorithms and software. Focuses on three areas where new uncertainty analysis methods are needed: inverse and regression problems, computationally expensive model functions, conformity assessment and reliable decision-making. Nine other European NMIs (including LGC, PTB, LNE, INRIM and SP) are partners in the project. NPL leads work packages on Uncertainty Evaluation for Computationally Expensive Systems (Wright) and Creating Impact (Matthews) and provides substantial input to the Uncertainty Evaluation for Inverse Problems and Regression work package (Harris). Conversion factor for £: € is 1: 1.25.

The Need

New mathematical and statistical approaches are required to address uncertainty evaluation in many modern metrology applications that are not explicitly covered by existing GUM guidelines. For example, uncertainty evaluation in many emerging or rapidly growing metrology applications, such as biochemical measurements and nanometrology, often poses challenging mathematical and statistical problems. There exists an unnecessarily high risk of incorrect decisions without reliable measurement uncertainty analysis. Many applications demand guidance for conformity assessment beyond current standards. The specific needs in problems demanding inverse methods and regression or computationally expensive systems require a co-ordinated effort to cope with these challenges, to ensure harmonization and to develop a consistent application framework throughout Europe.

The Solution

Project focuses on the areas where a strong need for new uncertainty evaluation methods has been identified. Application areas include new analytical technologies for biochemistry and biotechnology (ELISA, PCR), transport processes (fluid flow, thermophysical properties of materials), industry and regulation (scattering, fire safety engineering, conformance testing for healthcare products). Case studies addressing these important areas were selected in such a way that their solutions generate a large immediate impact. They will also provide template solutions that will be easy to use for other applications. Proposal builds on the existing network of mathematical experts active in the EURAMET Focus Group Mathematical and Software Tools for Metrology in which NPL staff have leading roles, and will lay the foundation for a virtual European Centre for Mathematics and Statistics in Metrology that will disseminate state of the art methods to European industry and organizations.

Project Description

Uncertainty evaluation for inversion problems and regression work package will develop and provide improved methods for the evaluation of uncertainty in the context of parametric inverse problems and regression problems that arise throughout metrology, and also provide reliability and comparability for new analytical technologies, the determination of thermophysical properties of materials, and for challenging calibration procedures. This will include methods of exploiting prior knowledge and expert knowledge in Bayesian formulations of uncertainty analysis problems. Uncertainty Evaluation for Computationally Expensive Systems work package will investigate smart sampling methods and surrogate models for uncertainty analysis, with case studies in scattering, thermophysical properties of materials and fluid flow. Decision-making and Conformity Assessment work package will develop methods for decision-making and conformity assessment for multivariate cases, including identification of appropriate cost function models, using outputs from the tasks described above. Outputs from work packages include software, best practice guides, scientific papers and reports, articles in trade journals, conference presentations, and reference data sets. NPL also leads the EMRP project’s Creating Impact work package (See Impact section below) and contributes to the Decision-making and Conformity Assessment and Management and Co-ordination work packages.

Impact and Benefits

This project aims at a substantial extension of the mathematical infrastructure for metrology in Europe. Collaboration between European NMIs with mathematical and statistical expertise is essential to ensure wide take-up of the project outputs and to maintain Europe’s current leading role in mathematics for metrology. The results of this project will strengthen European capabilities for innovation by enabling traceability for modern metrology and measurement techniques. Product testing, safety regulations, medical diagnosis and drug testing will be significantly improved by the procedures for reliable uncertainty evaluation, decision-making and conformity assessment to be developed in this project. Training courses provided by the Creating Impact work package will allow European NMIs and DIs that are not part of the project consortium to develop their capacity in the application of mathematics and statistics to challenging uncertainty evaluation problems. Lays the foundation for a virtual European Centre for Mathematics and Statistics in Metrology that will disseminate state of the art methods to European industry and organizations and ensure that the momentum developed by the project is carried forward and that impact can be realized beyond the end of the project.
Support for Programme Challenge, Roadmaps, Government Strategies Aligns directly with Uncertainties and Continuous Modelling themes of Mathematics and Modelling for Metrology programme. Supports NMO themes for underpinning metrology to support measurement infrastructure through new developments in traceability, uncertainty analysis, standards and regulations. Supports EMRP aims of stimulating innovation (methods arising from this project will enable metrologists to improve the quality of data for policy-making and regulation), integration and efficiency (collaboration with other NMIs enables large-scale approaches that are beyond the capabilities of single NMIs), and further development of metrology capability.

Synergies with other projects / programmes Results of this project provide input to EMRP NEW06 project Traceability for computationally-intensive metrology. Project team both at NPL and at European level shares members in common with the team that is already working on the EMRP IND09 project Traceable dynamic measurement of mechanical quantities. The improvements to the Guide to the Expression of Uncertainty in Measurement that will arise from this project will provide the basis for work beyond EMRP NEW04 within the uncertainties and validation themes of the MMM programme. The proposed virtual European Centre for Mathematics and Statistics in Metrology will also ensure that the outcomes of the project have impact beyond 2015. All NMS programmes will benefit from case studies and from revisions to the Guide to Expression of Uncertainty in Measurement, in particular, Materials, Engineering and Flow, and Chemical and Biological.

Risks As the proposed work is being carried out as part of a Europe-wide collaboration, technical and operational risks for NPL are small as experience and knowledge (and project co-ordination responsibilities) are shared with colleagues from other NMIs.

Knowledge Transfer and Exploitation Project aims at a substantial extension of the mathematical infrastructure for metrology in Europe. Collaboration between European NMIs with mathematical and statistical expertise is essential to ensure wide take-up of the project outputs and to maintain Europe’s current leading role in mathematics for metrology. The results of this project will strengthen European capabilities for innovation by enabling traceability for modern metrology and measurement techniques. Product testing, safety regulations, medical diagnosis and drug testing will be significantly improved by the procedures for reliable uncertainty evaluation, decision-making and conformity assessment to be developed in this project. Training courses provided by the Creating Impact work package will allow NMIs that are not part of the project consortium to develop their capacity in the application of mathematics and statistics to challenging uncertainty evaluation problems. Key exploitation activities include software written in Matlab and R to be made available for download from the JRP web site. In the longer term updated software, or versions applied to end-users’ specific problems, may be made available commercially through sales or licensing. The virtual European Centre for Mathematics and Statistics in Metrology will continue beyond the completion of the project and will ensure continued dissemination of the work package outcomes beyond the lifetime of the project.

Co-funding and Collaborators: European Union through EMRP provides £213k of matching co-funding. EMRP NEW04 industrial collaborators and stakeholders provide in-kind support for the work. 22 organisations (industry, universities, professional societies, regulatory bodies, international organisation and NMIs outside Europe) provided letters of support. Details available on request.

Work packages

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<td>Co-funding for EMRP NEW04</td>
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Calculation in the digital era. This project will put in place, for the first time, a coherent framework for ensuring traceability in computationally-intensive metrology, a basis for ensuring the trustworthiness and fitness for purpose of metrology software for coming decades, providing the digital counterpart to the calibration of material artefacts. A paradigm shift, led by key case study applications. The power and operational efficiency of the approach will be demonstrated by complete, fully operational systems in case study applications. Partners Hexagon, Mitutoyo, Werth and Zeiss, will deliver this new capability to thousands of users, changing the traceability landscape for computation in coordinate metrology and providing a convincing and effective pathway for other metrology domains. Bringing clarity to computational elements in measurement. End-users will know exactly what computations the measurement system is performing. Enhancing confidence in metrology products. Users will be able to demonstrate to themselves that the computational software is appropriate for their computational task and their data. Metrology system suppliers will have an independent assessment of the validity of their software. Helping metrology software providers, accelerating their development and quality assurance activities through the provision of public domain reference data sets. Increasing the efficiency of the market place. Many disputes between suppliers and manufacturers are caused by different software packages giving different results. Without clear traceability for computation, these issues cannot be
Reduced unnecessarily scrap, reworking. By improving the quality of metrology software, users of measurement systems will make better decisions about part conformance.

**Support for Programme Challenge, Roadmaps, Government Strategies** The project extends the concept of traceability to cover computation and supports the NMO’s broader strategy: “The primary and national measurement standards, forming the core of the NMS, underpin the system of traceable measurement in the UK, on which measurements rely. Traceable measurement supports legislation, regulation, standardisation, calibration and testing.” At a European level, software is clearly identified as an emerging metrology area at page 14-15 of the *EMRP outline 2008* document, in the clause “Mathematics and ICT for metrology”, in particular stating “It is necessary, e.g., to develop reliable mathematical models, to enhance the capabilities for data analysis, to ensure the trustworthiness of complex software and to incorporate arising communication technologies. ... It is furthermore essential to enhance the trustworthy of software and measurement data by developing appropriate validation technologies and security concepts.”

**Synergies with other projects / programmes** The project will benefit all areas of metrology which rely on complex computation. Case studies in, e.g., engineering measurement, will provide in depth synergies. The ICT infrastructure will be designed to evolve into a traceability capability across many areas of metrology over the next 10 years.

**Risks**

Delivery of the complete project depends on effective collaboration of the project partners. As coordinator, NPL is well-placed to ensure project progress. A risk register for the project has been developed. Three hold point reviews are included in the project involving the stakeholder community. The stakeholder input will help ensure the project outputs are relevant and usable.

**Knowledge Transfer and Exploitation**

The central aim of this project is to provide the capability to validate metrology software at the point of use. This aim will be achieved by the unique and innovative internet-based software validation service that will be available worldwide. In particular, through the key case study relating to coordinate metrology, world-leading instrument suppliers Hexagon, Mitutoyo, Werth and Zeiss, as project partners, will help deliver this capability to thousands of users worldwide. The project will also include activities to bring the complete spectrum of outputs of the project (mathematical and statistical theory, algorithms, software, reference data suites, databases, web pages, specifications, protocols, guidelines and tutorial documentation) to the metrology community (standard bodies, NMIs, instrument suppliers and client users) in a timely manner using the most effective knowledge services.

**Co-funding and Collaborators**

This project represents the NPL element of a 20 man-year, 2.7 M€ EMRP project involving CMI (CZ), INRIM (IT), PTB (DE), UM (SL) and VSL (NE), four industrial partners and the Universities of Huddersfield (UK) and Zwickau (DE). The NPL cash co-funding from the EMRP is £297k. The project is in contract negotiations but the funding has been secured. Details available on request.

### Deliverables

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**Deliverable title:** Co-funding for EMRP NEW06 project
This collaborative project brings together a major UK manufacturer of energy harvesting technology (Perpetuum) with world leading expertise in energy harvesting system design (Dan Inman, Virginia Tech), composites and design optimisation (Bath) and metrology (NPL) to develop innovative new broadband energy harvesting technology at the macro/meso-scale to provide a UK lead in the $4.4bn energy harvesting market. New metrology of non-linear energy harvesting is required to support this UK led innovation, and to support the UK lead in development of non-linear energy harvesting technology.

The Need
The increased use of wireless devices such as sensor networks across industry sectors from oil and gas to healthcare is driving the need for novel power supplies that can be integrated within systems and that do not require periodic replacement. Energy harvesting devices capture energy from the external environment to power low energy electronics such as wireless sensor networks for industrial monitoring, transport asset monitoring and sensing in inaccessible locations. The energy harvesting market was worth $605 million in 2010 but is predicted to reach $4.4 billion by the end of this decade. One source of energy is vibration and piezoelectric materials are commonly used to convert mechanical vibration to electrical energy. The current state-of-the-art devices are designed to capture electrical energy at resonant frequencies. Whilst this works well in a well-controlled frequency environment, the harvesting performance falls dramatically outside the resonant frequencies and the resonant frequencies of a structure can quickly become unattainable as a device becomes small or large. This limits the application areas where vibratory energy harvesting devices can be used. For the market to reach its true potential products must be developed that can guarantee a greater energy yield across multiple frequencies. New metrology is needed to support the design and development of these products.

The Solution
A mechanism using bistable carbon fibre/piezoelectric composites, recently discovered by our collaborators, promises excellent harvesting performance over a range of frequencies due to its nonlinear dynamics. However, the complex physics of nonlinear bistable composites means that it is difficult to accurately measure harvester performance and therefore optimise the design. NPL will develop new metrology to measure the non-linear energy harvesting response and support the design and development of non-linear energy harvesting materials by its collaborators.

Project Description (including summary of technical work)
This collaborative project will combine expertise in bistable composites, design optimisation, mixed signal electronics, advanced metrology and industrial testing to design, build and test demonstrators based on real world applications. The NPL role is as follows:

- Construct test rigs for measuring the complex non-linear response to real world vibration profile. These will be based on existing facilities developed in NMS and EMRP projects that will be adapted to measure the macro-scale non-linear systems developed under this project.
- Measure energy harvesting performance of prototype bistable composite energy harvesting devices and apply these measurements to the development of our understanding on non-linear energy harvesting and device optimisation.

Impact and Benefits
- Support of UK-based companies developing novel energy harvesting systems to capture a significant portion of the emerging $4bn global market;
- Support the development of truly wireless systems with potential to benefit society globally in terms of improved safety, reduced costs to industry and transport, elimination of significant battery usage, and reduced energy consumption facilitating the transition to sustainable living;
- Cement Establish NPL’s reputation developed under EMRP and NMS projects as a world-leading centre for the measurement and characterisation of energy harvesting materials.

Support for Programme Challenge, Roadmaps, Government Strategies
This project supports the NMS strategic aims particularly with regard to the National Challenges of Energy, Sustainability, Health and Security. It also supports the TSB’s innovation challenge through industrial collaboration. The project supports the NPL
**Synergies with other projects / programmes**

This project extends capability developed in EMRP ENG02 “Metrology for Energy Harvesting” and NMS AM02 to non-linear measurements at a larger scale and applied to bistable composites.

**Risks**

We are dependent on the successful development of functional composite devices by our collaborators. This is mitigated by their experience in the preparation of composites and exploratory work conducted before the project indicating that this is possible.

**Knowledge Transfer and Exploitation**

Opportunity for UK-based collaborator Perpetuum who are the leading manufacturer of industrial energy harvesters, to exploit the technology and for NPL to license it. The development of NPL’s expertise and facilities in the measurement of energy harvesting materials and in particular non-linear energy harvesters could provide an opportunity for measurement services. Wider dissemination will also be provided through industrial training, workshops and a blog set up in the EMRP project, UK KTNS, EPSRC energy harvesting network, Piezo institute training. Scientific work will be published in leading peer reviewed journals and presented at international conferences.

**Co-funding and Collaborators**

This collaboration assembles a unique team which combines the world leading expert in energy harvesting (Dan Inman) with expertise in composites and design optimisation (Bath), NPL’s metrology. Details available on request.

**Deliverables**

Summary

This proposal describes NPL activity within the Nanoindent-plus project, an EU coordination and support action, which will further develop, validate and draft standards for nano-scratching, which is an important new tool to investigate the tribological properties of materials on a scale relevant to the fundamental mechanisms of wear. The new standard will facilitate the development of new coatings and engineered surfaces that will give impact through more efficient transport, improved competitiveness of industry and a better quality of life for EU citizens.

The Need

Micro and nanoscale tribological testing has been developed over the last few years, and is being used in the evaluation of some fundamental aspects of the tribological response of materials. However, the uptake of these test methods by industry and the science base is hampered by the lack of validation of the test methods, the need for the determination of measurement uncertainties, and the provision of internationally accepted standards on the method.

The Solution

This project will further develop and validate nanotribological testing by carrying out definitive tests on a small number of key materials relevant to applications. An interlaboratory exercise will be conducted and the results analysed to provide information on the uncertainties of the measurement methods. A draft standard will be written for consideration by the relevant CEN and ISO standardisation committees.

Project Description (including summary of technical work)

The work that will be carried out will comprise:

- Input into choice of test materials and agreement with other project partners. The emphasis of the project is to develop test methods for coatings and engineered surfaces, so two different coating systems will be chosen (probably carbon based and ceramic based), and a monolithic wear resistant material such as WC/Co
- Exploratory trials to determine best conditions for interlaboratory exercise looking at parameters such as contact geometry and materials, contact load, speed and number of scratch repeats
- Input into interlaboratory exercise
- Analysis of interlaboratory exercise results
- Writing draft standard for input into CEN and ISO committees

Impact and Benefits

This project will validate urgently needed new measurement methods for the surface engineering community that are needed to improve the ability of coating developers to design better coatings and surface engineering solutions.

Support for Programme Challenge, Roadmaps, Government Strategies

This project fits in the surface engineering area and underpins the provision of new measurement methods enabling the design and manufacture of better coatings and surface engineering solutions. The provision of better coatings will give improved efficiency of energy generation and use, improved sustainability through more efficient use of raw materials and higher durability of engineering components. It will help the manufacturing industry through the provision of better surface engineering solutions.
Synergies with other projects / programmes

This project will make use of the outcomes of the development of micro tribology tests systems from the IRD T11 project on the monitoring of surfaces due to degradation from wear. It will also draw on expertise being developed in the EMRP project MADES on the metrological assessment of damage to engineered surfaces.

Risks

The major risk of this project is that there is a breakdown in the structure of the project partnership through events such as withdrawal of a project partner from the consortium. This risk is mitigated by the fact that all the project partners have previous experience of EU projects and that there is sufficient capability in the project to continue even if a single partner withdraws. A second risk is that the interlaboratory exercise results are not conclusive, but here sufficient experience of the new test methods exists within the project partners to ensure that the interlaboratory exercise will be successful.

Knowledge Transfer and Exploitation

This project is inherently concerned with the dissemination of the results of the nano-tribology test method into new standards submitted to CEN and ISO as available. A scientific paper will also be written on the results of the interlaboratory exercise, and a workshop will be held towards the end of the project to get feedback on the draft standard.

Co-funding and Collaborators

This is an agreed EU coordination and support action project and £48k cofunding is accrued from this. Details available on request.

Deliverables

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Deliverable title: Nanotribology tests and interlaboratory exercise. Demonstrated by a collaborative scientific paper.
The objectives of this project are: a) to review the latest methods for building and selecting the right set of PDE models for a given physical system with high nonlinearity, b) to study how errors in the model formulation stage propagate and affect the behaviour of the corresponding mathematical solutions, and how uncertainties in physics- or materials-related properties manifest in the final solution behaviour, c) to identify 2 solvable case studies of practical importance, which can apply the developed methodology to metrological applications, such as i) PDE approximations for temperature variations and diffusion mechanics in fuel cells, ii) nanoscale heat transfer in nanomaterials, or iii) PDE-related methods for data assimilation, though the emphasis will be placed on the first two application examples.
The deliverables include 2 peer-reviewed journal papers and 1 top-notch conference paper, and 1 software tool.

Impact and Benefits

The impact of this project will result in a significant step change in NPL’s modelling capabilities. In fact, this will fill the gap between the physical modelling and mathematical/numerical modelling work at NPL. Therefore, new capabilities in fundamental research and modelling will be developed to solve problems of practical and theoretical importance that cannot be solved before. Modellers and designers in NPL and UK will benefit from the output of this work, leading to better understanding of how approximations, model uncertainty and nonlinearity can affect the ultimate behaviour of mathematical models and potentially help to gain more insight into metrology applications such as complex PDE models for fuel cells and heat transfer modelling in nanomaterials. It can also help to design better fuel cells in the ever-increasing green/clean energy solutions.

Outputs of this project will also have a wide impact in international science community with a better understanding of the advantages and disadvantages of different levels of approximations in both physical and mathematical models and how such approximation and uncertainties can be correctly treated both mathematically and numerically. This will open new research areas such as PDE model selection for multiphysics modelling and nonlinear PDE analysis for metrology applications.

Support for Programme Challenge, Roadmaps, Government Strategies

This project is in line with the NMS strategy on the “development of characterisation tools, methodologies ...”: The main activities in this project are primarily about modelling and characteristic of approximation techniques associated with nonlinear PDE models and their applications in metrology.

Synergies with other projects / programmes

Case studies will be selected so as to emphasize the problem of practical importance and also to benefit other research activities in NPL, including organic electronics and fuel cells where temperature variations, water and heat managements. We will also investigate the potential applications in materials modelling and uncertainties of materials properties. Applications to other materials and/or EMRP projects will be investigated.

Risks

1) Existing analytical methods may not be good enough for practical applications. This risk can be minimized by investigating alternative methods such as multiscale asymptotics, boundary-layer theory and Monte Carlo methods. 2) Implementation may not be efficient to produce good results. Fortunately, we have developed a stable multiphysics mathematical model for fuel cells modelling, which can be modified to serve as a basis for different degrees of approximations and model uncertainty studies. Collaboration with Oxford Centre of Industrial and Applied Mathematics and Thomas Young Centre will reduce the risks.

Knowledge Transfer and Exploitation

Knowledge transfer will include

- peer-reviewed journal articles, conference papers, and a technique report,
- presentations at conferences, workshops, NMS clubs and industrial meetings, and
- software implementation of key methods.

Co-funding and Collaborators

Other NMS programmes (organic electronics, fuel cell modelling), Oxford Centre for Industrial and Applied Mathematics (modelling and analysis), and London Thomas Young Centre (research on modelling). Details available on request.

Deliverables

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<td>- a draft report (recommendations for implementation and highlighting main issues)</td>
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<td>Deliverable title: Another case study, further comparison and writeup</td>
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<tr>
<td>- Another case study using the PDE-based models (mobility modelling for organic electronics)</td>
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<tr>
<td>- 1 peer-reviewed journal paper (for case study and comparison) and 1 conference presentation.</td>
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<tr>
<td>- Final report. (Total outputs: 2 journal papers, 1 conference paper, 1 report)</td>
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</table>
The aim of the project is to extend the quantum Drude oscillator (QDO) model, developed during the previous NMS project, to ionic aqueous solutions. This will be an important case study further validating the model and providing vital information about the degree of water restructuring and organisation of the hydration shells around ions.

The project builds on and extends the present collaboration with the University of Edinburgh and IBM Watson Research Centre.

Many biophysical applications require accurate knowledge of aqueous ionic solutions and rely on data provided by atomistic simulations using nonpolarisable force fields (e.g., CHARMM, GROMOS, Amber). There is an urgent need to improve the accuracy and reliability of theoretical predictions and the model. Full quantum calculations of this type are beyond the present capabilities. The QDO model, a renormalized one-electron model that encapsulates all many-body responses of a molecule, provides an attractive and computationally efficient way of treating many-body dispersion and polarisation effects in condensed matter. This class of models is of particular interest to IBM as part of their modelling strategy on massively parallel (exascale) architecture.

Following our work on QDO model for noble gases, which demonstrated that the model provides an accurate and transferable basis to study systems dominated by dispersion interactions, we will further extend the model to more complex systems that include ionic species. This will allow us to address the important structural features concerning the ion-ion interactions in water and to possibly resolve existing contradictions in their interpretation. We will also optimize performance on emerging IBM platforms such as the Blue Gene P and Q systems.

The developed QDO model for noble gases has been described in Phys. Rev. B 79, 144119 (2009) and in recently submitted to Phys. Rev. B, the water model is in preparation for Phys. Rev. Lett.

The project includes several linked tasks. First, the model for systems with permanent charges will be formulated with the decision to be made how to handle the charged systems in simulation. The QDrude code will be extended accordingly. Using small isolated clusters and diffuse Monte Carlo the short-range repulsion of the selected ions will be fit in the QDrude code. Results of high-level quantum chemical calculations or accurate empirical data. The obtained parameters will be used in Path Integral molecular dynamics (PIMD) simulation at ambient temperature to validate the developed model. The large-scale parallel PIMD calculations will be performed on the IBM’s Blue Gene supercomputer.

Funding covering two months’ time of Dr Andrew Jones, a PDRA at UoE is requested at £3300/month. Dr Jones has considerable expertise in QDO model and was a principal developer of the QDrude code.

Development of efficient atomistic modelling methods capable of accurate treatment of many-body polarisation and dispersion of aqueous solutions is of fundamental importance to broad scientific community in bio simulation, virtual drug design, and advanced materials modelling. QDO model of ionic systems is an important step towards more complex systems of practical importance for Materials scientists, including nanostructured functional materials and nanocomposites. NPL is developing its capability in molecular modelling to support major programmes through the SR Fellowship 2005-08 in molecular modelling and subsequent NMS-funded and third party projects. This project will help developing a general method that underpins all major science areas at NPL and has a potential impact on the whole area of classical atomistic simulation. It will also help developing strategic partnerships with UoE and IBM research headquarters.

In December 2011, BIS published a key document outlining its Innovation and Research Strategy for Growth, included in which important areas aligns well to the science and impact aims of this proposal. In particular, high performance computing as a key theme with a particular focus on its role as an enabler in ‘the modelling of complex systems and processes in sectors such as the life sciences’. A major international partnership such as this is directly aligned to these objectives. Key success measures on the 1-5 year horizon include adoption and inclusion into major open-source simulation packages such as Open Atom and NAMD with the developers of which the collaborating team have close links.

### Summary

<table>
<thead>
<tr>
<th>Project No.</th>
<th>MAT\2012\30</th>
<th>Price to NMO</th>
<th>£97K</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Project Title</strong></td>
<td>Molecular dynamics simulation of aqueous ions using quantum Drude model</td>
<td><strong>Co-funding target</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Lead Scientist</strong></td>
<td>Vlad Sokhan</td>
<td><strong>Stage Start Date</strong></td>
<td>July 2012</td>
</tr>
<tr>
<td><strong>Scientist Team</strong></td>
<td>Jason Crain, Andrew Jones, Glenn Martyna</td>
<td><strong>Stage End Date</strong></td>
<td>June 2013 Ext Aug 2013</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>Est Final Stage End Date</strong></td>
<td>June 2013</td>
</tr>
<tr>
<td><strong>Sector</strong></td>
<td>Underpinning metrology</td>
<td><strong>Activity</strong></td>
<td>Metrology for new capabilities; Knowledge transfer</td>
</tr>
</tbody>
</table>

### Activity
- Funding covering two months’ time of Dr Andrew Jones, a PDRA at UoE is requested at £3300/month. Dr Jones has considerable expertise in QDO model and was a principal developer of the QDrude code.

### Impact and Benefits
- Development of efficient atomistic modelling methods capable of accurate treatment of many-body polarisation and dispersion of aqueous solutions is of fundamental importance to broad scientific community in bio simulation, virtual drug design, and advanced materials modelling. QDO model of ionic systems is an important step towards more complex systems of practical importance for Materials scientists, including nanostructured functional materials and nanocomposites. NPL is developing its capability in molecular modelling to support major programmes through the SR Fellowship 2005-08 in molecular modelling and subsequent NMS-funded and third party projects. This project will help developing a general method that underpins all major science areas at NPL and has a potential impact on the whole area of classical atomistic simulation. It will also help developing strategic partnerships with UoE and IBM research headquarters.
benches and scaling behaviour on IBM platforms. Broader coordination among EU NMIs regarding simulation is a further strategic target to which this project may be a strong foundation.

### Support for Programme Challenge, Roadmaps, Government Strategies
This project aligns well with the NPL’s roadmap in molecular modelling and with NPL’s initiative to develop a strategic collaboration with Edinburgh University. It will provide a flexible basis to improve metrological traceability in various areas of nanometrology.

### Synergies with other projects / programmes
The project builds upon the recent work carried in the NMS multiscale project on coarse-graining and during Vlad Sokhan’s secondment to IBM Watson Centre. He has also won an EPSRC Industrial CASE studentship for 2012-13 ‘Electronically coarse-grained molecular dynamics’, which aligns well with the proposed programme. The results will feed the extended multidisciplinary project submitted to EPSRC grant jointly by scientists from the University of Edinburgh and IBM, where NPL will be sub-contracted.

### Risks
This is high-risk fundamental research project that primary depends on two factors: validity of the model and the suggested solution, and on availability of required resources. The first risk is reduced by our previous successes with the model. It might happen, however that the project will need more time to complete, in this case we will adjust the planned work and/or seek the additional funding from our collaborators. The project relies on the access to high-performance computing resources provided by our collaborators. The risk is mitigated however by the fact that the team has access to two independent sites in Europe and America, each providing sufficient resources for successful completion of the project tasks.

### Knowledge Transfer and Exploitation
All obtained results will be published in internationally recognised scientific journals and will be further disseminated via conference presentations, seminars, and personal contacts. The software developed within the project will be provided as ‘open source’. The output of the project will be of interest to wide academic community and a broad range of industrial end users in chemical, pharmaceutical, food, and oil industries.

### Co-funding and Collaborators
The project builds on and extends the existing collaboration with high calibre scientists at the University of Edinburgh and IBM Watson Research Centre. The project benefits from the HPC access at EPCC (HECToR Phase II) and to IBM’s Blue Gene/L and /P at Watson Research Centre, provided by our collaborators as in-kind contribution. In addition, Vlad Sokhan’s application for early access to STFC computer facilities at Daresbury Laboratory (12M core hours of Blue Gene/Q) is currently under consideration by the evaluation panel at DL. Details available on request.

### Deliverables

<table>
<thead>
<tr>
<th></th>
<th>Start: 01/07/2012</th>
<th>End: 30/06/2013</th>
<th>Cost: £</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Development of the mathematical model: based on the scheme used for noble gases and water develop a QDO model for systems including ions and parameterise it for specified ionic species.</td>
<td></td>
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<tr>
<td>2</td>
<td>Software development – extension of QDrude code: Implement the model in the code and test and validate the code against available experimental data using the test case of ionic solution.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Project No. | MAT\2013\1 | Price to NMO | £150k
---|---|---|---
**Project Title** | Programme Management and Formulation | Co-funding target | N/A
**Lead Scientist** | Rob Reid | **Stage Start Date** | July 2013
**Scientist Team** |  | **Stage End Date** | June 2014
**Sector** | Management | **Est Final Stage End Date** | Cont
**Activity** | Programme Management

**Summary**
This project will formulate a proposal of work for inclusion in the 2014/15 programme and engage with key stakeholders to ensure maximum impact is achieved from the science delivered by the programme. To achieve these objectives the project will:

- Maintain and develop the programme strategy and roadmaps;
- Consult with key stakeholders in government, industry, academia, regulators and other end users in order to determine future measurement requirements or other related issues that need to be addressed by the programme;
- Develop a series of project proposals for prioritisation by the programme working group;
- Develop and maintain a balanced scorecard for the programme as a measure of the impact of the programme.

**The Need**
New measurement requirements are constantly emerging from all areas of UK life. For example, new technologies require new underpinning metrology and standards, as do new regulations or environmental targets. To underpin areas such as growth in the economy, public health issues or mitigation of environmental impacts these measurement requirements must be successfully addressed as early as possible. In order to achieve these objectives effectively an overview of the research priorities and how to address them is required. Maintaining and developing a programme strategy and roadmaps achieve this objective and allow, in conjunction with knowledge of specific technical requirements obtained through stakeholder consultation, the formulation of a work programme that address UK measurement needs. Both the careful design of any programme of work coupled with the continual review of opportunities for increased impact are essential in order to make sure that the maximum value possible is extracted from the investment made in the technical projects.

**The Solution**
The views of a wide range of stakeholders from industry, regulators, government and other end users will be sort through a wide ranging consultation process in order to capture current and emerging measurement requirements. This process will include looking at independent evidence of measurement needs as expressed in government reports, foresight activities, industry roadmaps etc. as well as conducting meetings, surveys and interviews as required that focus on specific topics of interest. Collation and assessment of information from all sources will enable the programme strategy and roadmap to be developed which will guide the future direction of the programme. The detailed technical requirements will then formulated into a series of projects for prioritisation by the independent programme working group. Projects which receive the highest ranking will form a programme of work which will be initiated at the start of the next programme cycle.

In addition to the programme formulation, work will be undertaken to understand and maximise the impact of the research. This will be assisted by the establishment and maintenance of a balanced scorecard for the programme consisting of a number of key metrics.

**Project Description (including summary of technical work)**
- Horizon scanning, capture and analysis of Industry and Societal needs to feed into current and future programme direction;
- Development and updating of programme roadmaps and strategy;
- Engagement with programme stakeholders to:
  - Realise outputs and maximise benefits to the UK;
  - Ensure alignment of programme with UK Government, Industry and Societal drivers;
- Oversee preparation of project proposals for review and prioritisation by the programme working group;
- Submission of final programme proposal for contracting;
- Liaison with the NMO programme supervisor to deliver maximum impact and efficient delivery;
- Identification of exploitable material for increased impact through channels provided by the Pan-Programme KT programme and other KT avenues;
- Assessment of the impact of the programme through use of a balanced scorecard;

**Impact and Benefits**

Effective programme management will maximise the outcomes to key stakeholder communities from the outset of the technical work and ensure knowledge transfer activities in the programme are efficient and effective. The programme as a whole addresses many measurement challenges across the broad sweep of the UK economy and society. Therefore, the design of knowledge flows and exploitation plans in technical projects which occurs during the formulation process is essential for delivery of the wide benefits of the programme to the broadest possible audience.

**Support for Programme Challenge, Roadmaps, Government Strategies**

This project underpins the work of the whole programme through development of an overview of key societal drivers and measurement requirements as captured in the programme strategy and roadmaps. These key programme documents are utilised during development of technical projects to guarantee that all the technical work in the programme is aligned to addressing the metrology needs of the UK.

**Synergies with other projects / programmes**

This project will interact with the other NMS programmes so that synergies and common goals can be identified to ensure that the maximum value is returned from the investment in the NMS portfolio.

**Risks**

Not applicable.

**Knowledge Transfer and Exploitation**

The main functions of this project are to ensure the development of a new programme of work and to measure and increase the impact of the programme. Improvement of the programme impact will be achieved through proactive intervention in the technical projects within the programme rather than through direct knowledge transfer activity in this project.

**Co-funding and Collaborators**

Not applicable.

**Deliverables**

<table>
<thead>
<tr>
<th>Del.</th>
<th>Start: 1st July 2013</th>
<th>End: 30th June 2014</th>
<th>Cost: £</th>
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</table>

Deliverable title: Programme Management and Formulation
Summary

This project will deliver effective contract management for the programme. Work in this project will ensure timely invoicing and reporting to the NMO each month and delivery of an annual progress report to the NMO and programme working group.

The Need

Contract management is essential to ensure seamless delivery of the science projects in the programme while attending to all reporting and invoicing requirements of the NMO. A central point of control is also required for effective operational oversight and governance of the programme.

The Solution

This project will deliver effective contract management through a contract manager dedicated to this programme. They will have oversight of all:

- Project delivery;
- Invoicing;
- Contract status and variations;
- Monthly and annual reporting.

Project Description (including summary of technical work)

1. Attend meetings as necessary to support contract delivery and the needs of the NMO
2. Prepare reports monthly (invoices, progress report and financial forecasts)
3. Liaison with working group, industrial advisory groups & clubs
4. Manage delivery of the contract and submit change requests and contract amendments as necessary
5. Analysis of programme performance and revenue forecasts for the financial year
6. Ensure that the contract is managed to NPL’s ISO 9001 accredited quality system
7. Deliver annual report and present programme progress to working group and the NMO as required.

Impact and Benefits

This project will ensure that all operational, financial and reporting requirements for the programme are met. The work in the programme covers the oversight of delivery from all the technical projects and hence is where ultimate responsibility lies for the success of the programme.

Support for Programme Challenge, Roadmaps, Government Strategies

Not applicable.

Synergies with other projects / programmes

Not applicable.
### Risks
The main risks are the inability to deliver the monthly reports and invoices to the NMO and the failure to deliver the annual report to the programme working group. Both of these risks are mitigated by the availability of a large pool of senior managers who are available to step in to assist or take over delivery if adverse circumstances are causing problems with the completion of the key tasks of this project.

### Knowledge Transfer and Exploitation
Not applicable.

### Co-funding and Collaborators
Not applicable.

### Deliverables

<table>
<thead>
<tr>
<th>Deliverable title</th>
<th>Start: 1&lt;sup&gt;st&lt;/sup&gt; July 2013</th>
<th>End: 30&lt;sup&gt;th&lt;/sup&gt; June 2014</th>
<th>Cost: £</th>
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<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deliverable title</td>
<td>Contract management including production of monthly invoices and reports to the NMO</td>
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<td>2</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Deliverable title</td>
<td>Produce annual report and present progress to the NMO and working group</td>
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The Materials International project aims to propagate globally UK best practice, particularly that developed in NMS material projects, and to assess international best measurement practice to benefit UK industry competitiveness. The project has three themes, firstly, implementation of prior work as international standards and maintenance of NPL/UK owned standards; secondly, management of several UK led TWAs and secretariat of VAMAS and, thirdly, networking with other NMIs and Materials Research Institutes. It is important to use these networking opportunities to identify trends and needs that have relevance to the UK activities. During the previous periods extensive developments have occurred in the recognition and acceptance of materials metrology promoted by past Materials International projects.

The Need
It is important in order to obtain full value from the materials programme that research outputs are available in an easily adopted form and that the UK materials research programme has influence on the national and global stages. These needs are across all industry sectors with increasing importance as more developing countries become competitive. Many of the outputs are generic and apply across different sectors and applications and frequently appear as international standards.

The Solution
The UK through the NMS programme has a unique opportunity to lead international standards work. Although other NMIs are involved, the high degree of readiness of NMS outputs as completed drafts that have been used for interlaboratory trials and the resultant precision data increases both the speed of adoption of UK proposals and their progress through the balloting stages.

Through the alternating leadership (i.e. UK/USA) of the VAMAS Secretariat, through a high and sustained profile in International standards and other network groups, NPL has a major opportunity to encourage the adoption internationally of UK’s research outputs and preferences. The successful promotion by NPL of “materials metrology” has raised the awareness of the importance of accurate and relevant measurements of material properties in innovation, process control and certification and regulation.

The Versailles Project on Advanced Materials and Standards (VAMAS) provides a mechanism for global collaboration – secondly, management of several UK led TWAs and secretariat of VAMAS and, thirdly, networking with other NMIs and Materials Research Institutes. It is important to use these networking opportunities to identify trends and needs that have relevance to the UK activities. During the previous periods extensive developments have occurred in the recognition and acceptance of materials metrology promoted by past Materials International projects.

Project Description (including summary of technical work)

**VAMAS** - The Versailles Project on Advanced Materials and Standards (VAMAS) provides a mechanism for global collaboration and facilitates the adoption of its outputs by standards bodies. The participation is much wider that just NPL and other NMIs. NPL will be responsible for the shadow Secretariat for the next two years until fully responsible again in May 2014. NPL will continue to support the web-site on behalf of NIST as in the past year. The UK leads several Technical Working Areas and participates (including project leadership) in others. UK’s current TWA convenor responsibilities include: TWAS Polymer Composites, TWA22 Mechanical Properties of Thin Films and Coatings, TWA24 Performance Related Properties for Electroceramics, TWA31 Crack Growth of Weldments with Residual Stress (led by Imperial College), TWA32 Modulus Measurement and TWA 36 Organic Electronics.

**Standards** – Standards based on completed work requires continuing support that is not available without an on-going research project in the same technical area. The drafting, and revising, of a standard may take several years beyond the completion of the particular funded project. It is important that NPL expertise accumulated over the years in NMS funded projects is fully utilised to progress these drafts for the benefit of UK industry. NPL provides informed and independent expertise on International Standards that is recognised by industry as an important output of the research. NPL acts as project leaders in most cases based on the NMS produced drafts. Representation includes (n.b. varies depending on need and progress): ISO/TC206 Advanced technical ceramics; ISO/TC164 Mechanical testing; ISO/TC156 Corrosion of metals and alloys; ISO TC61 Plastics (SC2 Mechanical Properties, SC11 Adhesives, SC13 Composites) and CEN TC 249/GW15 Composites.

**International Networking** - Under the international networking agenda, several new initiatives are being developed. These include the VAMAS-BIPM MoU on materials metrology, implementation of the MoU signed with BAM (Germany) in 2008 to encourage interaction and use of facilities; continued involvement with the World Materials Research Institute Forum (WMRIF), which has signed also an agreement with VAMAS, and participation in CIPM CC intercomparisons, CEN and ASTM round-robin on a selective basis. Following the initiative led by NPL on metrology applied to the measurement of materials properties through the Working Group on Materials Metrology, CIPM have agreed a “MoU” with VAMAS. The on-going agreed interactions including new CIPM WGs on material aspects in existing CCs, organisation of “pilot” and “strategic” studies. VAMAS TWA32 includes a specific RR on modulus measurement using a known reference material aimed at supporting the BIPM-VAMAS MoU. The new MoU signed with BAM encourages interaction and use of facilities. Joint activities are to be planned, which may also include NIST where NPL has a MoU with the now Materials Measurement Laboratory. During 2007, NPL signed at Directorate level the agreement of the World Materials Research Institute Forum (WMRIF), directed at promotion of young material.
scientists (It is planned to enter 1 to 2 staff in this competition in Aug, 2012 –Dr Andy Wain (NPL) was the 2010 winner), promotion of databases, research on materials for energy and structural integrity, and material property simulation. NPL leads the WMRIF promotion of databases in WG4, which is of importance to Materials UK (WG4 will co-sponsor 3rd Asian Database meeting in Japan). n.b. NPL undertook a survey for DTI on the need and scope of materials property databases). VAMAS has a signed agreement to lead on standards for WMRIF with NPL providing the link between the organisations. Participation in the International Energy Agency (IEA), and in particular the Materials for Transport WG will be considered, as will participation in other relevant international studies where their value to UK can be demonstrated. Other representations on national committees, with international profiles, to be supported include High Temperature Mechanical Testing Committee (HTMTC, ESIS TC11) and British Society of Strain Measurement. The focus of joint activities will be BAM/PBT, NIST, and Far East NMI’s (e.g. NIM, NIMJ/NIMS and KRISS).

### Impact and Benefits
In all of the international projects, the aim is to bring UK developments arising from the NMS programmes to implementation as international standards or equivalents. UK global competitiveness will benefit through implementation in international standards of research outputs and standards initially identified by UK industry. The NPL’s position as the UK Centre of Excellence in materials metrology provides a unique opportunity to collaborate with technically based organisations in the UK, Europe and around the world, on measurements and standards supporting the competitive position of UK industry. It is well established that NPL is able to consistently deliver the required standards and other documentary outputs.

### Support for Programme Challenge, Roadmaps, Government Strategies
The provision of standards and regulations is critical to the success of new innovative products having economic and quality of life benefits, as recognised by the TS8 Innovation Platforms that span blue-sky research to procurement and other specific calls. As such, the projects particularly support the innovation and procurement phases in the roadmaps and strategy supporting the NMO Materials Programme and roadmaps and strategy more widely available (e.g. TS8). VAMAS aims to identify and provide the necessary optimised technical solutions prior to national solutions that need less-optimised harmonisation at a later stage.

### Synergies with other projects / programmes
This project has intrinsically considerable interaction with all the other NMS materials research projects in order to take their outputs through to implementation. Also to feed back into the project needs and measurement expertise identified globally. Increasing, the interactions with BIPM will cement existing connections with SI programmes such as engineering, chem-bio etc.

### Risks
This is a low risk project as it builds on existing well-founded interactions and uses the output of prior technical work undertaken in core projects in the Materials Programme, that was itself selected through extensive consultation with UK industry on their needs. The main risks is non-acceptance of UK work by the wider community, which is mitigated by ensuring up-to-date contacts with the wider stakeholders and to use a rigorous review process for acceptance of new work.

### Knowledge Transfer and Exploitation
Dissemination routes will include national standards committees, Industrial Advisory Group (IAG) meetings, national meetings, conferences and seminars; direct interactions with UK industry and academia, Measurement network and NPL publications. Initiatives to increase the profile and usefulness of the VAMAS web-site will continue, as NIST have requested that NPL maintains the web-site due to its excellence, even when NIST hold the Secretariat. It is also planned to develop an index to the high quality precision data collated in all VAMAS prior projects. In many cases testwork and consultancy services will be provided, principally to UK companies, providing industry with early experience and benchmarking opportunities of these new methods.

### Co-funding and Collaborators
Collaborating organisations include NMI’s (e.g. NIST, BAM, PTB, LNE, KRISS, NMIJ), standards development organisations (e.g. BIPM/CIPM, ISO, IEC, CEN, BSI), regulators (e.g. ILAC, UKAS, CAA, Lloyds Register), learned institutes (e.g. RS/RAE, IoP, IoM3) - and appropriate companies, trade organisations, IAGs and BSI committee membership. Details available on request.

### Deliverables

<table>
<thead>
<tr>
<th>Deliverable title: Leadership of VAMAS G15 Programme</th>
<th>Start: 01/07/13</th>
<th>End: 30/06/14</th>
<th>Cost: £</th>
</tr>
</thead>
</table>

Annual report on role of NPL representatives (e.g. Secretariat), TWAs lead and their work programme and outputs, meetings held and dissemination achieved to UK industries. Drafts for standards submitted.

<table>
<thead>
<tr>
<th>Deliverable title: Support to Implementing Research in New Standards</th>
<th>Start: 01/07/13</th>
<th>End: 30/06/14</th>
<th>Cost: £</th>
</tr>
</thead>
</table>

Annual report on role of NPL representatives (e.g. Convenor, Project Leader), work undertaken, standards balloted and published; together with an assessment of dissemination undertaken, especially to UK industries.

<table>
<thead>
<tr>
<th>Deliverable title: International Networking</th>
<th>Start: 01/07/13</th>
<th>End: 30/06/14</th>
<th>Cost: £</th>
</tr>
</thead>
</table>

Annual report on role of NPL representatives, work undertaken, standards balloted and published; together with an assessment of dissemination undertaken, especially to UK industries. New initiatives made or supported.
**Project No.** MAT\2013\5  
**Price to NMO** £183k

| **Project Title** | Efficient PDE model updating  
| **Co-funding target** |  
| **Lead Scientist** | Louise Wright  
| **Stage Start Date** | 1/08/13  
| **Scientist Team** | Minh Hoang, John Blackburn, Neil McCartney.  
| **Stage End Date** | 30/06/15  
| **Est Final Stage End Date** |  

| **Sector** | Use lexicon  
| **Activity** | Challenge Driven R&D  

**Summary**  
A partial differential equation (PDE) model is specified by a governing PDE, a material property description for each material in the model, and the boundary and initial conditions. This project will develop tools for updating PDE models and their associated parameters and uncertainties to achieve fitness for purpose. Recent advances in multiscale modelling and multiphysics simulations have led to many situations where a measurement process could be described by any one of a range of models of differing degrees of complexity. In many cases a change in complexity leads to a large increase in computational cost for a small increase in accuracy. This project will create a methodology for selecting a model that is sufficiently accurate and sufficiently low in computational cost as to be fit for purpose. Another aspect of model updating is driven by manufacturing challenges and process monitoring. The increasing demand for factory floor and real-time measurement, currently strongly driven by the aerospace sector (e.g. recent EMRP project i04 on Large Volume Metrology includes a work package on simulation for in-situ measurement of aircraft parts), requires a dynamic approach to model parameter and uncertainty updating. This project will develop methods and algorithms to address this challenge.

**The Need**  
Models are used to obtain parameter estimates from a wide variety of measurement processes. The typical model validation and parameter evaluation paradigm is shown in the figure below. Input variables (which may include previous parameter estimates), a physics-based model, and measurement data, all of which have associated uncertainties or errors, are combined in a validation process. This process either accepts the model and evaluates parameters and associated uncertainties, or identifies the need for updated parameter estimates, an updated model, or more measurement data (or a combination of all three). This paradigm is well suited to static measurement processes with simple models under controlled conditions.

Many measurement systems are best described by a PDE with associated boundary and initial conditions, and are typically nonlinear and require numerical solution and hence are not “simple” models. Updating such models generally involves choosing from a range of possible new models of varying complexity levels. A more complex model will give better accuracy at increased computational cost. There is a need for a generic objective methodology for choosing a fit for purpose model that balances accuracy and cost. A further complication arises if the measurement is taking place in an uncontrolled environment such as a factory floor or workshop. The temperature fluctuations in such a situation mean that the entire paradigm becomes dynamic, linking the “update” boxes on the right to the input boxes on the left. A dynamic approach for parameter updating and uncertainty evaluation is then required to ensure that the measurement data are interpreted correctly.

**The Solution**  
NPL has recently developed a conceptual methodology for model selection for PDEs during project MAT12025. A literature search has revealed a lack of PDE model selection methods, making our new development unique. To date, the methodology has been applied to a simple test case, described in an abstract submitted to a Joint Imeko TC1-TC7-TC13 Symposium due for presentation in September 2013, but needs extending to meet the challenges of real metrology modelling problems. This project will develop that methodology into a generic model selection process that will be applicable to any PDE model updating problem where the balance between accuracy and computational cost is a key factor. The project will also address the challenges of model and parameter updating in response to dynamic data by adapting data assimilation techniques, developed for PDE models in meteorology, to meet the requirements of PDE models in metrology under uncontrolled conditions. The main project outputs will be a methodology and a set of algorithmic tools, disseminated via case studies (in large volume metrology, organic electronics, and fuel cells) and journal papers.

**Project Description (including summary of technical work)**  
The project will further develop the existing methodology for PDE model selection and ensure it can handle the full range of types of model complexity by validating using data generated in areas of metrology of strategic importance to the UK. Possible factors causing complexity include nonlinear material models and
boundary conditions, pseudo-random materials (e.g. effects of dispersed particles, crystal orientation, etc.), dependence on an additional state variable (e.g. temperature), and dimensionality. The methodology’s ability to cope with the challenge of dimensionality (does a 2D model give significantly different results to a 1D model?) will be validated using data for carrier transport in organic electronics, where it is assumed that the through-thickness transport dominates any in-plane effects. The effect of the inclusion of additional state variables will be examined by looking at the effects of the removal of the assumption of isothermal conditions on fuel cell models.

The difficulties of updating PDE models under fluctuating environmental conditions will be approached by studying, adapting and applying data assimilation approaches. These approaches have been developed for updating meteorological PDE models and include time-dependent errors and uncertainties in the problem specification, making them ideally suited to metrology in uncontrolled conditions. The adapted approaches will be validated using a problem investigating the effect of changing temperature and temperature gradients on engineering structures subject to gravitational loading and fixture constraints, focussing on assimilating temperature measurements with FE models of structures of varying complexity. This case study will include aspects of model selection as well as parameter and uncertainty updating via data assimilation, and will involve collaborations with the Universities of Bath, Reading and Surrey.

**Impact and Benefits**
The need for in-situ real-time measurement is an important industry driver, and this project creates tools for accurate modelling and uncertainty evaluation under those challenging conditions that can be exploited via collaborations. The recent growth of interest in microstructure-to-bulk material property modelling means that models of ever increasing complexity are now available, but they may not be necessary for all applications. This project creates a methodology to choose a fit-for-purpose efficient model that can be disseminated via papers. The key areas that will benefit directly from the project will be those involved in the validation case studies as the project will create improvements in their modelling, and hence measurement, capabilities. In the longer term the techniques will be applied as required on any projects with NPL modelling involvement, and will be disseminated to academia, industry and other NMIs via papers, conferences, and collaborations.

**Support for Programme Challenge, Roadmaps, Government Strategies**
The project directly supports the (evolving) data analysis and uncertainty evaluation strategy relating to the evaluation of the uncertainty associated with models and determining data-model integrity for complex systems. The project will address some of the main issues associated with uncertainty evaluation for measurement systems whose responses are modelled by partial differential equations. Data assimilation is potentially important in four of the Metrology 2020s application areas: Monitoring the state of the planet, Energy efficiency and diversity of supply, A healthy population and Managing key resources and infrastructure. Data assimilation forms a key element in DBIS/EPSRC initiatives in Big Data and Data to Information.

**Synergies with other projects / programmes**
The case studies and real-world problems used to demonstrate the work in this project will be of direct benefit to other NMIs programmes. All the tools and methods generated will be sufficiently generic that they can be expanded and applied to other problems as metrological needs develop, whether within NPL or in the wider world. Areas that will benefit directly include the EMRP project i04 on Large Volume Metrology, and the MMMM programme projects on Fuel Cell Durability and Measurement and modelling for OPVs. Other projects and application areas with which this work aligns include: IRD project on verification of carbon savings, smart electrical grids, gas energy grids, environmental monitoring, and thermophysical property measurement.

**Risks**
Data assimilation methods may not be suitable for metrology problems: initial discussions suggest that this is very unlikely, but if so strong links with area experts (Met Office, Reading University, Surrey University) will enable development of new methods.

**Knowledge Transfer and Exploitation**
In addition to publications, the outputs of this project will be disseminated by the ongoing application of the tools and methods developed during the project to metrology problems. The use of these tools on NMS and third-party modelling work will promote the project outcomes through case studies and future publications that reference the outcomes of this project. Through its involvement as tier 2 partner in the Manufacturing Technology Centre NPL will realise impact through promoting project outputs to the High Value Manufacturing Catapult Centre members.

**Co-funding and Collaborators**
Internal collaborators on the case studies include groups working on dimensional metrology, fuel cells, and organic electronics. The project is expected to attract in-kind funding from its case study owners. The engineering structures case study will form part of NPL’s participation in a 5 year £5 M EPSRC project, The Light Controlled Factory, led by the University of Bath and involving the measurement of engineering structures in factory conditions. Initial discussions regarding data assimilation have taken place with the Met Office and the Universities of Reading and Surrey, leaders in developing data assimilation in meteorology. Details available on request.

**Deliverables**

<table>
<thead>
<tr>
<th>1</th>
<th>Start: 01/08/13</th>
<th>End: 30/07/15</th>
<th>Cost: £</th>
</tr>
</thead>
</table>

**Deliverable title:** Updating PDE Models

A report describing algorithms, supported by software where appropriate, for updating of PDE models and their parameters and associated uncertainties. Three peer-reviewed journal papers reporting the results of the application of these algorithms to specific real-world measurement problems.
Project No.  | MAT\2013\6 | Price to NMO | £300k
---|---|---|---
Project Title  | Uncertainty Analysis for Static and Dynamic Digital Imaging Systems | Co-funding target | N/A
Lead Scientist | Clare Matthews | Stage Start Date | Oct 2013
Scientist Team | Ian Smith, Peter Harris, Trevor Esward | Stage End Date | Sep 2016
| | Est Final Stage End Date | Sep 2016
Sector | Advanced instrumentation and sensors | Activity | Strategic capability building
Project Champion | K-D Sommer

Summary  At present there is typically no estimation and propagation of uncertainties within digital imaging analysis and consequently the accuracy of inferences drawn from images is poor, or often unknown. A standard approach to apply uncertainty analysis to imaging systems and applications does not currently exist. We aim to solve this problem by establishing new methods for applying uncertainty analysis to quantitative inferences from images, enabling users of image analysis tools to define with confidence the levels of uncertainty in the conclusions drawn from their analyses. Many high-level imaging applications are built on the same low-level tasks such as identification, segmentation or correlation/mapping. Similarly, the same sources of uncertainty are present in all digital imaging problems: pixelation, discretisation of pixel intensity, signal noise etc., and will each affect quantitative conclusions drawn from images, e.g., medical diagnosis or emission estimation. We will assess propagation of uncertainties through commonly used image processing algorithms and explore the application of signal processing and probability methodologies within new algorithms. Developed techniques will be validated firstly on simulated images, then using real data generated by key metrology areas at NPL and in collaboration with external partners at a major London teaching hospital. Outputs from the project will be published in scientific journals, developed into a Good Practice Guide, provided as supplements to the GUM and developed as software tools.

The Need  Image analysis is increasingly critical to applications in health, environment, manufacturing and materials performance. For example, the global medical image analysis software market alone is expected to grow at a CAGR of 7.2% from 2012 to 2017. However, a lack of accurate uncertainty analysis drastically limits the value and utility of the conclusions that can be drawn from images. This lack of confidence can impact the success of medical treatments such as the use of molecular radiotherapy in treating cancers (where at present absorbed doses in different patients can differ by two orders of magnitude, and quantitative imaging will improve dosimetry and therefore treatment efficacy) and the up-take of new techniques, for example the commercial take-up of LVM (accuracies in a factory environment are currently limited over ranges of more than a metre). Uncertainties in images are introduced at the point of capture (e.g. environmental effects on refractive index), by the imaging system itself (e.g. spatial and colour discretisation), and by subsequent algorithms employed to extract required information from an image (e.g. edge detection). Quantification and propagation of such sources of uncertainty must be considered to provide a complete uncertainty analysis and to associate probability statements with quantitative conclusions drawn from digital images. Providing a complete measurement and analysis view requires new methods, guidance and software tools, developed by NMLs with substantial experience in uncertainty analysis for metrology applications.

The Solution  The work will start by developing an understanding of the uncertainty that arises from the analogue to digital conversion process via spatial, colour and temporal discretisation. Firstly, through simulated images and subsequently through the use of data generated at NPL and by our external partners, we will examine how these factors influence image segmentation, sub-pixel resolution, and feature extraction results. We will then work to define how each component contributes to uncertainties associated with estimates derived and inferences made from the image, to enable uncertainty to be calculated and applied to the results of the analysis. The techniques established will, for the first time in many applications, enable quantitative conclusions with associated uncertainties to be derived from images. We will disseminate the outcomes of the work through publication in scientific and trade-specific literature, the development of software and the publication of a Good Practice Guide.

Project Description (including summary of technical work) We will adopt a systematic approach that can be applied to many digital imaging systems by concentrating on a range of lower-level features that are common across these systems, such as sampling effects, image bit-depth, correlation, image noise etc. The technical work will focus initially on understanding and quantifying these common sources of uncertainty in simple, simulated images, and then investigating their propagation through image processing algorithms used for common low-level tasks such as segmentation. For example, a photogrammetry problem may require an estimate of the centre location of a circular target. The resulting estimate will be affected by camera settings, image size, file size and noise, as well as different algorithms applied to locate the centre. These effects therefore all represent sources of uncertainty and a complete analysis of the estimated centre location can only be achieved by considering the contribution to and propagation of uncertainties at each stage. Analysis in the project will incorporate and develop our previous work on signal processing and uncertainties associated with digital filters, including recently published work (Measurement) on data fusion algorithms, and the use of prior information in deriving probability statements for measurement results. The project findings will establish guidance on application-dependent optimal image processing techniques. The work will be divided by three types of image uncertainty characteristics: static uniform (uncertainty uniformly spread across the image), static non-uniform (uncertainty non-uniformly spread, e.g. perspective scenes, images with camera aberration distortions), and dynamic
**Impact and Benefits**
The outcome of this project will be a method of expressing uncertainties associated with measurements derived from images, enabling quantitative analysis of measurement results from multi-dimensional imaging systems, which for the first time considers the complete measurement and analysis chain. This capability will change the current way of reporting results from many imaging algorithms, as at present uncertainties associated with conclusions drawn from images are not routinely quantified. A methodology for producing an accurate assessment of uncertainty in digital image based measurement will be developed for existing image analysis packages – a significant market in healthcare alone, and where applicable for specific applications, complete image and uncertainty analysis software will be developed. Direct impact will also be delivered through the validation exercises, perhaps most notably through the proposed hospital collaboration.

**Support for Programme Challenge, Roadmaps, Government Strategies**
- NMS strategies for digital economy, healthcare and security.
- NPL strategies for computer science related to statistical modelling and “trusted” algorithms for inference decision making, data analysis and reliability, and measurements and modelling to characterise molecules, bio-systems and materials.
- EMRP aims in multidisciplinary metrology (e.g. underpinning mathematics and ICT), metrology for environment (e.g. emission control) metrology for healthcare, and the move from qualitative to quantitative diagnostics, and evidence-based medicine.

**Synergies with other projects / programmes**
- Develops methodologies applied in signal processing projects in SSfM3 (digital signal processing Good Practice Guide) and SSfM4 (MST paper e.g. effect of signal to noise ratio on uncertainties) programmes and in current EMRP IND09 project on dynamic measurement systems.
- Builds on the CATMESS case study from MM programme 114405 (described in NPL report), calculating uncertainties associated with targets in an image, and provides close links to continuation of this area in new LVM EMRP project, led by NPL.
- Aligns with NPL-led, EMRP project: MetroMRT – Metrology for Molecular Radiotherapy.
- Provides input to core uncertainties project 114404 for GUM supplements and guidance documents on uncertainties associated with measurement results from multi-dimensional systems and specific image processing illustrative examples.
- Biometric systems work at NPL.
- Development of super-resolution imaging capabilities within Biotechnology Group. Single molecule detection techniques provide improving capabilities to visualise the structure of cells, leading to better understanding of disease.
- Grains to Structures within the Materials programme e.g., crack detection using images of material layers from FIB.
- Current work on carbon emission estimation based on Earth observation data.

**Risks**
Initial studies based on simulated images will diminish possibility of technical risks. The team proposed for this project has substantial experience in uncertainty analysis and in making software public.

**Knowledge Transfer and Exploitation**
- Potential for software licensing or bespoke software development
- Good Practice Guide
- A minimum of four papers in relevant scientific or trade journals
- Conference presentation at application-related conference and theory-related conference
- Input to GUM supplements and other JCGM documents on uncertainty analysis

**Co-funding and Collaborators**
NPL: Advanced Engineered Materials, Biotechnology, Mass & Dimensional, Radiation Dosimetry, Optical Measurement
External: major London teaching hospital working on quality assurance for medical ultrasound scanners; MRC National Institute for Medical Research for single molecule detection. Details available on request.

**Deliverables**

<table>
<thead>
<tr>
<th>Deliverable title</th>
<th>Start Date</th>
<th>End Date</th>
<th>Cost (£)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>1</strong> Establish electronic database of simulated images and images for validation (static uniform uncertainty, static non-uniform uncertainty, dynamic). Define measurement requirement from each image, and nature of uncertainty statement that is required and include this information in image metadata. Review activities after 1 year to identify optimal sectors and partners for collaboration to provide maximum scientific and business impact opportunities.</td>
<td>01/10/13</td>
<td>31/03/14</td>
<td>n/a</td>
</tr>
<tr>
<td><strong>2</strong> Static uniform images. Validation: biometric information fusion; Earth observation resolution scaling.</td>
<td>01/04/14</td>
<td>30/09/14</td>
<td>n/a</td>
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<tr>
<td><strong>3</strong> Static non-uniform images. Validation: medical ultrasound feature and change detection (collaboration with St. Thomas’); LVM target location. Submit abstract to relevant application-related conference.</td>
<td>01/10/14</td>
<td>30/09/15</td>
<td>n/a</td>
</tr>
<tr>
<td><strong>4</strong> Dynamic non-uniform images. Validation: dSTORM &amp; SIM for time dependent images; FIB defect detection for spatially related images. Submit abstract to a relevant application-related conference.</td>
<td>01/10/15</td>
<td>30/09/16</td>
<td>n/a</td>
</tr>
</tbody>
</table>

**Dissemination of findings.** Uncertainty analysis for digital images - Good Practice Guide.
<table>
<thead>
<tr>
<th>Project No.</th>
<th>MAT\2013\7</th>
<th>Price to NMO</th>
<th>£297K</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Project Title</strong></td>
<td>Time series techniques and evaluation of uncertainties in the analysis of anthropogenic trends and transitions in geophysical data</td>
<td>Co-funding target</td>
<td></td>
</tr>
<tr>
<td><strong>Lead Scientist</strong></td>
<td>Valerie Livina</td>
<td>Stage Start Date</td>
<td>Oct 2013</td>
</tr>
<tr>
<td><strong>Scientist Team</strong></td>
<td>Peter Harris, Alistair Forbes, Clare Matthews, Stephen Robinson, Michael de Podesta, Emma Woolliams, Paul Green, Stephanie Bell</td>
<td>Stage End Date</td>
<td>Sep 2016</td>
</tr>
<tr>
<td><strong>Est Final Stage End Date</strong></td>
<td>Aug 2016</td>
<td>Activity</td>
<td>Challenge-driven R&amp;D</td>
</tr>
<tr>
<td><strong>Sector</strong></td>
<td>Environmental Sustainability</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Project Champion</strong></td>
<td>Emma Woolliams, Paul Green, Stephanie Bell</td>
<td></td>
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</table>

**Summary** The work aims to develop mathematical and statistical tools for analysing time series data to identify trends and transitions in various Earth climate subsystems under anthropogenic influence as an application of the generic framework. The tools will be used to estimate long-term trends and detect possible tipping points in geophysical datasets, and to underpin the metrology for the protection of the environment from anthropogenic effects. The project will provide an evaluation of the associated uncertainties. It will compare various geophysical subsystems and attempt to establish their interconnections. The tools will be applied to observed data, including acoustic noise measured in the ocean, satellite-based sea-surface temperature (SST), humidity on land surface, and magnetic field fluctuations.

**The Need** Understanding components of a dynamic system, such as fluctuations and anthropogenic changes in the Earth, requires the analysis of vast amounts of data: to detect trends in data that is subject to instrumental and geophysical noise, which is recorded over decades, often involving multiple individual instruments. Developing a general stochastic framework for such applications is a challenging scientific task, which should also provide tools for analysing real geophysical data. Modern technology has provided the means to obtain large observed data sets, but their metrology and analysis is still relatively immature and needs to catch up with a rapidly evolving legislative framework. Furthermore, the scale of monitoring required, the changes due to the development of instrumentation, and the most appropriate analysis methods are the subject of current debate. The challenges in studying such time series include being able to handle a large dynamic range, non-Gaussian distributions at various frequency bands, the presence of high-amplitude transients, and processing of large data sets. The uncertainties to be associated with the trend estimates must account for the nature of the fluctuations as well as the influence of the choice of variables or metrics to be analysed; ideally, the developed metrics should also direct the climate metrology towards measuring intrinsic variables. The case studies selected here relate to EU legislative directives and datasets that are highlighted as significant in understanding anthropogenic climate change. The Global Climate Observing System has defined key “Essential Climate Variables (ECVs)”; land surface humidity and sea-surface temperature (SST) are both ECVs, the latter being one of the European Space Agency’s key ECVs for its Climate Change Initiative. These topics are being studied in other NPL projects, through the Temperature & Humidity and the Optical Group as part of NPL’s Centre for Carbon Measurement. Similarly, man-made noise in the ocean is classified as a form of pollution, and measures of Good Environmental Status specified by the EU Marine Strategy Framework Directive include the trend in the levels of low frequency noise from commercial ships, and the amount of high-energy impulsive sound introduced into the ocean from offshore wind farm construction and oil and gas exploration. Studying this data is particularly important for the NPL group working on acoustic measurements.

**The Solution** The analysis of geophysical time series data requires developing novel metrics that are robust to the choice of temporal averaging and sampling regime, and are capable of determining long-term trends in man-made effects in the presence of large short term fluctuations in the data, many of which have natural causes. The project will combine capability within NPL’s Data Analysis and Uncertainty Evaluation science area in time series analysis and uncertainty evaluation with the knowledge of NPL’s scientists in the areas of marine acoustic noise, SST, land surface humidity and earth observations to address metrological questions concerning time series data arising in these areas. The outcome of the project will be a unified framework for the analysis of geophysical data, providing quantification of trends, analysis of fluctuations, detection of tipping points, and evaluation of associated uncertainties. The project will provide a novel methodology for analysing separate components of geophysical variables: underlying natural variability (seasonal/diurnal), anthropogenic forcings, and detrended fluctuations. An important part of the project will be development of uncertainties estimators for the geophysical metrics in the considered subsystems.

**Project Description (including summary of technical work)** The project will establish a stochastic framework implemented using the tipping point toolbox (developed by Valerie Livina) with additional modules for trend analysis and uncertainty evaluation (to be developed). The tipping point toolbox is based on the Langevin-equation approximation of a time series and will be further developed and generalised to include additional noise and trend components, so that flexibility of modelling is ensured for different variables. Each geophysical subsystem will be studied to derive an appropriate metric for analysis. The choice of metric is important in order to obtain the essential statistics of each subsystem, where recorded data sets may be large and/or multidimensional. The chosen metrics will be studied using the developed project software providing full analysis and attribution of trends, identification of transitions or bifurcations, and evaluation of associated uncertainties. Finally, various climatic subsystems will be compared in their dynamics, and possible interconnections established using detrended cross-correlation analysis and cross-wavelet analysis. The data to be studied include ocean and atmosphere variables that are likely to be susceptible
to anthropogenic influence (marine acoustic data and the ECVs of humidity and SST). As a null-model, the dynamics of the variables will be compared with observed magnetic field fluctuations to establish objective interconnections between the geophysical subsystems and anthropogenic or natural influences. To investigate the connection of the studied subsystems with the current global warming, we plan to consider Mauna Loa CO₂ record and ECMWF reanalysis surface temperature. Outcomes of the project will be reported in scientific publications in peer-reviewed journals and conference presentations, as well as through International Committees, such as Committee on Earth Observation Satellites. Alongside the exploration of geophysical time series data, the project will provide a metrological software package for the comprehensive analysis of general time series of various origins and the evaluation of associated uncertainties.

**Impact and Benefits** The resulting project software may be used as a metrological tool with quantifiable metrics. The tool will be based on dynamical systems approach with stochastic modelling of its dynamics, and therefore will be general and applicable in areas beyond climatology. In recent pilot studies, the tipping point toolbox was applied to a system of harmonic oscillators on a lattice (statistical physics) and to a set of sensors records on NPL footbridge (structure health monitoring). If its generalisation is successful, the proposed project will contribute to better understanding of anthropogenic influences on climate. Particular benefits are expected for the climatological community and for the researchers in statistical physics and time series analysis. In the area of Earth observations, the developed tool may be later introduced as a metrological standard for the analysis of observed data. The SST work will be done in conjunction with UK-based academics involved in the ESA-CCI SST project, and will feed directly into the quality aspects of the project. Thus, the aims and impacts of the project are two-fold: generalisation and refinement of the existing initial framework and dissemination of the approach in the communities studying various dynamical systems.

**Support for Programme Challenge, Roadmaps, Government Strategies** The project will directly support the Metrology 2020 application “Monitoring the state of the planet”, which identifies the need for statistical models exploiting spatio-temporal correlation in data for trend analysis to support the validation of environmental monitoring networks. The project supports the NMO developing roadmaps on “Data analysis and uncertainty evaluation” in the areas of improved analysis and decision making, dealing with complexity, and feature extraction, and “Metrology for Earth observation” in the area of time series analysis.

**Synergies with other projects / programmes** The application to marine acoustic noise is relevant to acoustic metrology in the NMO AIR programme. NPL’s Acoustics Group already has access to data sets of in-situ noise measurements from a DEFRA-funded UK project on noise monitoring, and it is highly likely that NPL would gain access to long-term noise data recorded by the Comprehensive Test Ban Treaty Organisation (CTBTO), with whom NPL has already worked in this field. The output would also feed into the work of the EU MSFD Technical Sub-Group on marine noise (of which NPL is a member), and the work of ISO TC43 SC3. The application of the project tools to humidity data will continue the existing NPL’s collaboration with Met Office Hadley Centre, where the corresponding datasets are being developed. The SST fits directly into the existing NMS “Quality Assurance for Earth Observation (EO) and Climate Change” project, and relates to a Framework-7 project “Quality Assurance for ECVs” that has been bid for, and an expected EMRP project MetEOC2. If the project is successful, it may lead to policy-related solutions, in particular, within the ESA Climate Change Initiative.

**Risks** Interconnections between particular subsystems may not exist, and hence the corresponding parts of the analysis may not be informative. Particular datasets may not exhibit any trends, or those trends that are identified may not be attributable to anthropogenic influences. These research risks do not influence the development of the metrological framework as one of the main goals of the project. The project depends on access to time series data of sufficient length and quality for the particular applications to be treated. The proposed project may be taken as a longer-term research programme incorporating interests and expertise of several groups at NPL.

**Knowledge Transfer and Exploitation** The framework will be made open-access as a stand-alone software package available free at a public website (NPL or software portal, like Matlab Central), and the results of the analysis will be published in peer-reviewed journals. The developed metrics may be disseminated in the climatological community (conference presentations and publications), and implemented as a real-time early-warning system in climate monitoring (MetOffice).

**Co-funding and Collaborators.** Collaboration with Prof T.Lenton (Exeter), Dr C.Merchant (Edinburgh) and Dr G.Corlett (Leicester) will bring further links in academia and connections with the Earth system, oceanographic and ecological communities.

**Deliverables**

<table>
<thead>
<tr>
<th>Deliverable</th>
<th>Start</th>
<th>End</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Framework development. Development of a unified analytic framework incorporating various techniques of time series analysis (trend estimation, early warning signals, detection and forecast of tipping points) with evaluation of uncertainties. Software development and implementation.</td>
<td>01/10/13</td>
<td>30/09/14</td>
<td>£</td>
</tr>
<tr>
<td>Tests on artificial data. Simulations of artificial data with realistic properties equivalent to various geophysical data: atmospheric, oceanographic, solid earth time series. Development of a suitable statistical metric and implementation of an appropriate stochastic model for each geophysical subsystem. Application of the developed framework to the artificial data with evaluation of uncertainties.</td>
<td>01/10/14</td>
<td>31/05/15</td>
<td>£</td>
</tr>
<tr>
<td>Applications to real time series data. Ocean time series: marine acoustic noise (DEFRA and CTBTO). Atmospheric time series: HadCRUH specific humidity, HADISH land surface humidity. Solid earth time series: magnetic field fluctuations. Final synthesis of the results of the analysis with establishment of interconnections between the analysed subsystems.</td>
<td>01/06/15</td>
<td>31/08/16</td>
<td>£</td>
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</tbody>
</table>
**Project Title**  
Thermal characterisation of reactive energy storage materials for harsh environments.

**Project Lead**  
Lindsay Chapman
Angela Dawson
Petra Mildeova

**Project Team**  
Stage Start Date  
Sept 2013

**Stage End Date**  
Aug 2014

**Est Final Stage End Date**  

**Sector**  
Energy/High Value Manufacturing

**Activity**  
New Capability

**Project Champion**  
Peter Flewitt

**Contractor**  

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**Summary**  
This proposal addresses issues arising from feasibility studies carried out under the EMRP project ENG08 MetroFission focused on metrology for new generation nuclear power. This project is proposing to expand on the measurement capabilities developed in this previous work and focus on their application in increasing the safety of nuclear power. This is through addressing the harsh environments encountered under accident conditions (specifically extremely high temperatures up to 3000°C) and characterizing reactive materials, such as providing design data for the molten salts likely to be used in energy storage solutions. It is vital that traceable metrology is in place to provide confidence in the safety of both nuclear plant maintenance and design data for new build. This confidence would also be of benefit to high value manufacturing under extreme conditions where currently data for modeling is extrapolated from measured data.

**The Need**  
Nuclear Power has a significant role to play in UK energy policy with eight sites approved for new build. Apart from providing competitively priced energy it will also significantly contribute to the reduction of greenhouse gases. One major concern with this form of energy is safety, which has been highlighted by events at Fukushima. This project aims to provide metrology that will overcome the barriers to widespread adoption of nuclear power by providing reliable and traceable input data relating to accidents and how to prevent them from happening. This data would enable closer control of processes, and improved prediction of component lifetime performance including behaviour under accident conditions. It is crucial in any safety case to know that you are measuring accurately, with traceability and with known uncertainties. This capability would also benefit manufacturing industries, where there is an increasing demand for improvements in the traceability and uncertainty of thermophysical property measurements, to replace extrapolated data or provide data under conditions previously uncharacterized.

The current requirements for characterisation of extremely high temperature material properties already require a step change in what NMLIs can deliver in terms of validation facilities to provide confidence in the measurement data. For future components operating under more extreme conditions in all of the power generation, aerospace, and automotive industries, ever smaller measurement uncertainties will be required. Projects such as ENG08:Metrofission aim to increase the range of indirect thermal conductivity measurements to 2000°C. Indirect evaluation requires multiple property determination using a range of apparatus (for separate measurement of specific heat capacity, density and thermal diffusivity) by virtue of which the uncertainty can be assumed to increase. The direct method of thermal conductivity determination falls below the current requirements of industry, and the indirect method of determination currently offers no traceability above 800°C. If methods and data are applied to enable safe nuclear fission, this could have a significant impact on the industry and the economy.

**The Solution**  
The overall aim is to build a European metrology infrastructure to better characterize thermal properties at very high temperature (up to 3000 °C) for advanced materials used in nuclear power plants, and to improve the traceability of measurements such as thermal conductivity, diffusivity, and melting points to the SI, for the benefit of all institutes involved in data for this application. These objectives include opportunities for new science as well as newly developed science (MetroFission), pushed to another level (range of measurements) or being applied in a new way, such as to reactive liquid materials.

**Project Description**  
The overall aim is twofold. Firstly, to extend the temperature range of traceable thermophysical property measurements for the optimisation of industrial processes and lifecycle assessment and to apply knowledge developed for solid materials to development of suitable liquid reference materials. Secondly, to determine traceable measurements of thermophysical data for storage materials, and reference materials at high and critical temperatures. The project will support the implementation of facilities for determination of thermal diffusivity up to 3000°C, by systems and methods directly traceable to the SI via well characterised reference materials. It will provide an assessment of the uncertainty of measurement of current thermophysical property measurement methods at the highest temperature and adapt existing facilities for the measurement of specific heat and thermal diffusivity of molten salts (by using conclusions of the feasibility study of the project "MetroFission"). It will improve traceability of phase transition measurements (including melting point) and thermophysical properties measurements for the reactive materials used in energy storage in the high temperature range applicable to nuclear fuels. Thus, extending the scope of methods developed in EMRP ENG08:Metrofission but also exploring the application of methods for determination of melting point and enthalpy of industrial materials at temperatures above 2000°C.

**Impact and Benefits**  
This proposal is the first step in addressing a key capability gap with regard to the modelling of the
extremely harsh environments encountered in nuclear power stations under normal operation, and during accident conditions and in some manufacturing processes. Improved accuracy of these models will be achieved by being able to reliably and accurately measure materials to temperatures in the order of 3000°C, rather than by extrapolating from current data. Accurately modelling the physical behaviours of materials under the extreme thermal and mechanical loadings imposed by manufacturing processes requires the input of material property data which are difficult to measure reliably. Currently these are not generally available in the open literature and often extrapolations are used that have unknown levels of uncertainty associated with the predictions. If Industry is able to access traceable thermophysical property data with estimates of uncertainty, this will enable the development of more complex designs through predictive modelling, for the optimisation of both the operating process and designing to prevent accident scenarios. It is imperative to be able to determine accurately the thermal properties of the material used for this application, by precise, reliable and comparable measurements. This will extend the capability across a range of industries that require traceable data in a shorter time frame and at a significantly reduced cost compared with making multiple measurements, or producing bespoke samples required by direct methods. This will be done by comparing indirect measurements of established reference materials with data from NMI facilities, allowing evaluation of commercial apparatus and determination of errors associated with measurements on their particular materials. This will streamline the procedure for assessing new processing techniques and materials, resulting in more rapid adoption and at lower cost, without the significant lead time and cost associated with repeating the measurement of specific materials.

Support for Programme Challenge, Roadmaps, Government Strategies This project is proposing to expand on the measurement capabilities developed in a previous EMRP project MetroFission that has been looking at metrology for new generation of nuclear power. MetroFission demonstrated that safety is on top of the agenda for both new build and current plants and therefore a shift is required to focus efforts in applying metrology to safety issues. This project positions NPL for future EMRP projects in the Energy Call or other follow on projects.

Synergies with other projects / programmes

- The project offers opportunities for sharing best practice across industry, via secondment arrangements; by exchange of a wide variety of sample types in order to evaluate the uncertainty introduced by non-conforming materials or by development of uncertainty analysis models.
- The capabilities developed will be applicable across sectors, such that European aerospace, automotive and other manufacturing industries will be able to demonstrate the performance of their components compared with a traceable validation system, enabling them to gain a competitive edge in the marketplace.
- This project begins to identify the issues experienced with making measurements at extremely high temperatures. This will be applied in the main to solid materials but initial measurements will be made on liquid materials that will advise on future developments required to assess the uncertainty of liquid measurements.

Risks The risk of developing additional capability will be mitigated by working in collaboration with industrial partners. For example Mersen have indicated that they would be open to collaboration by making their thermal diffusivity apparatus available to NPL for use within this project. The risk is that the industrial partner will not be able to make the equipment available at times convenient for the project deliverables.

Obtaining suitable samples within the scope and timescale of the project is always a considerable risk, particularly when complex materials or sample dimensions are required. A significant challenge will be dealing with reactive materials and designing suitable containment systems to make measurements possible.

Knowledge Transfer and Exploitation Routes for dissemination are likely to include the high value manufacturing catapult, such as the AMRC Advanced Manufacturing Forum, the Warwick Manufacturing Group or the Nuclear AMRC. Report describing facilities and methods for measuring thermal properties of molten salts, including the applicability of a laser flash based technique for thermal diffusivity measurements on molten salts.

Co-funding and Collaborators Details available on request.

Deliverables

<table>
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<td>Application of thermophysical property reference materials to extremely high temperatures, including measurement of selected thermophysical properties of proposed reference materials in the liquid phase. Evidenced by paper.</td>
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### Project Title
Co-funding for TSB RESIST project (REsidual Stress and structural Integrity Studies using Thermography)

### Lead Scientist
Jerry Lord

### Scientist Team
Jerry Lord, Agnieszka Ratzke, Roger Morrell

### Sector
Advanced instrumentation and sensors

### Project Champion
P Flewitt

### Summary
The measurement and influence of Residual Stresses (RS) continue to attract considerable attention in engineering because of their impact on part distortion, service performance and the costs associated with residual stress related failures. Currently RS assessment can be expensive, time consuming, destructive and many of the techniques are lab-based or provide only limited, single point data. Industry has a need for a practical non-destructive method to measure the RS in components in situ. Common RS measurement methods such as incremental centre hole drilling (ICH) and layer removal are destructive and laboratory based. Non-destructive methods such as neutron diffraction are not portable and are very expensive, and novel methods such as ultrasound are yet to demonstrate any practical use or accuracy. In this project a new means of evaluating RS is proposed based on material and system models combined with data from a full-field, non-contact, non-destructive measurement technique based on ThermoElastic Stress Analysis (TSA). This approach will be particularly suited to large or expensive components, where material removal is undesirable and where contact is impossible.

### The Need
The holy grail for residual stress measurement is a portable non-contact residual stress mapping technique. The development of TSA for residual stress mapping goes some way to achieving this. It is a new approach that has been successfully applied to a case study on cold expanded holes, but further development and validation is required before it can be established as a reliable technology for residual stress measurement. An area where residual stresses are particularly important is in power generation plant, where residual stresses from welding have the capacity to strongly influence the structural integrity of plant components. In the nuclear industry witness the examples of so called reheat cracking (creep cracking driven by residual stress relaxation) in heavy section austenitic steel welds in AGR boilers, and primary water stress corrosion cracking in PWR dissimilar metal welds. Life extension of operating reactors can require more sophisticated structural integrity assessments of secondary systems than were envisaged during original plant design. The heat treatment history of welds in these systems can be unclear, and in the absence of firm evidence it is often necessary to make onerous assumptions about weld residual stress. Traditional methods of residual stress measurement are non-portable or semi-destructive, and normally require shut down of the plant to remove the affected component for testing, semi-destructive assessments or the construction of laboratory simulations to perform measurements. It is also normal to combine these measurements with finite element simulation of the relevant plant welds before making use of the resultant stresses in structural integrity assessments. This is a lengthy and expensive process. In all these cases, a rapid non-invasive technique that can reveal the presence of significant residual stresses, and potentially estimate their magnitude offers the prospect of significantly reducing the costs and time involved in assessing their impact on structural integrity.

### The Solution
The aim of the project is to develop and validate the TSA technique as a means of residual stress measurement. This will involve:
- The development of the physical understanding and a theoretical framework for RS evaluation by TSA
- Establishing the relationship between thermophysical properties, plastic strain and residual stress
- Developing material models for the calibration of data and system models for the determination of RS
- Devising a system that can apply a suitable load to a structure to generate a thermoelastic response.
- On plant measurements to demonstrate its industrial application and validation.

Measurements will be made on well characterised materials of relevance to the aerospace and power generation industries.

### Project Description (including summary of technical work)
The technical work will be largely split between the UoS, TWI and NPL. NPL will be responsible for the work supporting the determination of the thermoelastic constants $K_0$ and $K_p$. Three materials will be examined: a 316L stainless steel, and 7000 and 2000-series aluminium alloys, using existing specimens that have been machined and stress relieved (for the $K_p$ calibration) and a set that have been monotonically preloaded to different levels of plastic strain (for $K_0$). A limited number of additional tests will also be carried out at NPL on 316L specimens subject to a cyclic plastic strain path, to compare with monotonic uniaxial loading to provide a more realistic simulation of the strain in the weld HAZ, where cyclically accumulated plastic strain levels of up to 30% can be seen. The physical property measurements will focus on thermal expansion, specific heat capacity, thermal diffusivity and density. Initial tests will be carried out to optimise the test parameters and accuracy, and a Design of Experiments (DoE) protocol developed to refine and optimise the matrix of tests required. NPL and UoS will also develop microstructure-based material models to predict $K_p$ for the range of materials examined and relevant to the industrial application for 316L.

NPL will also carry out experimental work and provide validated data using conventional techniques such as hole drilling and XRD to quantify the level of plastic strain and will develop detailed material models and maps that relate the thermoelastic...
response to the plastic strain experienced by the component.

**Impact and Benefits**

It is estimated that RS related failures cost UK industry in excess of £1bn p.a., so the goal of the proposed work is to create a technology that directly reduces these failures and provides economic benefit to industry. Although initially aimed at the power generation industry, successful development of the TSA-based RS technique will have much wider application, facilitating rapid online measurements, near real time process control and improved quality management. The associated reduced cost and time for RS assessments will lead to more frequent use, hence higher quality and fewer failures. The work will impact on through life evaluations as changes in the stress distribution in structures can be monitored, enabling assessment of damage and cracks, as well as RS. The industrial benefits will come from implementation of this technology to, e.g., shot peening, laser shock peening and laser beam or friction stir welding. According to a 2012 study from IPPR commissioned by EDF Energy, investment in new nuclear power stations could raise UK GDP by over £5 billion, create 32,500 jobs, and increase nuclear industry exports by up to £900 million.

**Support for Programme Challenge, Roadmaps, Government Strategies**

Supports:

- NMS strategies for energy efficiency, advanced materials and manufacturing.
- NPL strategies for residual stress measurement, microstructural analysis, miniaturised testing

**Synergies with other projects / programmes**

- Builds on and extends the work started in MicroRS Materials 2010 project, and previous residual stress projects

**Risks**

Main technical risks associated with the overall project are related to the computational complexities of the modelling, but these do not prevent NPL from completing our tasks. Risks for NPL are that component geometries may not be suitable for conventional techniques and close discussion with the partners will be necessary to resolve this. Measurements and modelled results may not agree due to undefined instrumental errors such as surface effects, but the plan included time to study near surface phenomena and current work is indicating that the technical issues can be overcome. The management risk is that project partners do not provide materials expertise to plan, mitigated by our ability to provide materials collected from existing projects.

**Knowledge Transfer and Exploitation**

NPL provides national leadership in RS evaluation and its application. NPL also provide a widely accepted route to dissemination and exploitation and RS consultancy services to industry. We are well placed within the national infrastructure to provide services using the methods developed under this project. The technology will be exploited through the widening of a toolbox of NDE methods that NPL offers to resolve industrial problems.

**Co-funding and Collaborators**

The project provides co-funding for TSB project RESIST and work in the MicroRS project. Details available on request.

**Deliverables**

| Deliverable title: Thermophysical property measurements supporting TSA development | Start: 01/07/13 | End: 30/06/16 | Cost: £ |
Project Title | Co-funding for EU iSTRESS project “Metrology research for the development and validation of design rules for engineering of nanostructured and nanoenabled materials and devices” | Co-funding target
---|---|---
Lead Scientist | Jerry Lord | Stage Start Date | Feb 2014
Scientist Team | Jerry Lord, Agnieszka Ratzke, Sabrina Yan, Ken Mingard, Nigel Jennett | Stage End Date | Jan 2017
Sector | Advanced Manufacturing | Est Final Stage End Date | Jan 2017
Project Champion | Peter Flewitt | Activity | 

**Summary**  
Residual stresses resulting from manufacturing limit the performance and lifetime of thin films and coatings, but established techniques for micron-scale measurement of residual stress still have significant limitations. In this project, a European consortium is established to develop an innovative, highly reproducible and automated measurement protocol for the analysis of residual stress on a sub-micron scale, based on incremental focused ion beam (FIB) milling, along with high-resolution in situ Scanning Electron Microscopy (SEM) imaging and full field strain analysis by Digital Image Correlation (DIC). The activities will focus on the development, implementation, validation and pre-standardisation of automated FIB-SEM, DIC and inverse stress calculation procedures, through both CEN and VAMAS.

**The Need**  
Intrinsic (or residual) stresses, resulting from manufacturing or processing, mostly define the performance and limit the lifetime of nanostructured materials, thin films, coatings and MEMS devices. Residual stresses usually arise from the processing of the materials, caused by i.e. intrinsic growth stresses in thin films or due to the combination of materials with different thermoelastic properties. Residual stresses can be understood as internal stresses, which can act on the nanoscale and can cause damage and crack initiation on the local scale, leading to the failure of the whole coating system or device. The established techniques for micron-scale measurement of residual stress still have strong limitations, e.g. in terms of spatial resolution, lack of depth sensing, their applicability on non-crystalline materials or accessibility to industry.

**The Solution**  
The project aim is to develop and promote pre-standardization of an innovative, highly reproducible and automated family of protocols for the measurement and analysis of residual stress at the sub-micron scale, which affect the properties and lifetime of a wide range of micro/nanostructured and amorphous materials, thin films, MEMS devices and engineering coatings. The rapid expansion of nano-science and nano-technology in recent years has placed increasing demands on the development of appropriate nano-scale and sub-micron analysis tools for residual stress evaluation, but current techniques are lacking.

**Project Description (including summary of technical work)**  
The main focus of the scientific activities is on the development of new characterization approaches and experimental techniques aimed at defining the correlation between local microstructure and residual stress state and the mechanical properties of structured materials at the macro- and micro-scale. A key objective is the establishment of innovative, industry-oriented design tools, implemented via modelling and optimization, for the production of stress-controlled nanostructured and amorphous materials. Specific focus will be given to nano-layered coatings with optimized stress depth gradients, metal interconnects (BEOL and TSV), metallic and Si-based membranes (MEMS) and micro/nano-crystalline materials with tailored intra-grain residual stress distributions. Successful achievement of the scientific and technological goals will be accomplished through the exploitation of a synergistic research and development programme between the research and industry partners, based on the following four main concepts:

- Optimization, validation and pre-standardisation of a high-resolution method for the spatially resolved residual stress analysis on a sub-micron scale;

- Insights into the real ion-matter interaction mechanisms and evaluation of FIB induced artefacts;

- Direct access for industry, and SMEs in particular, to high resolution residual stress measurement protocols;

- New Design-rules implemented into industry design-tools.

Specific NPL activity will focus on the validation and assessment of uncertainties and errors in the FIB-DIC approach. This will be achieved through a combination of basic research, test method development and the organisation of round robin exercises (WP3) on selected reference samples to refine the test methodologies for a range of geometries and applications. Pre-standardisation of the methodologies will be achieved through the development of a Good Practice Guide and the collaboration and support of relevant CEN, ISE Technical Committees and VAMAS TWA22. This will ensure the traceability and reproducibility, thus providing both the scientific community and industry with an extremely versatile tool and new capability for residual stress assessment on a sub-micron scale. NPL will also co-ordinate and manage the overall dissemination activity in WP8.

**Impact and Benefits**  
The project is expected to realize a breakthrough in the measurement and modelling ability of the residual stress distribution at the (sub) micron scale, directly relevant to production/manufacturing processes, thus proving
industry with enabling technology for the design and production of innovative micro-devices with improved in-service performances, enhanced reliability and substantially reduced modelling and maintenance costs. A network of users including NPL and the University of Oxford will be established to provide access to the facilities and technique by UK industry. From a scientific viewpoint the project is expected to foster significant advances in the field of sub-nanometre resolution displacement measurement from electron microscopy imaging, by developing innovative FIB milling and SEM imaging procedures and repeatable DIC routines. It will establish the relationship between the residual stress distribution and the mechanical properties of amorphous and nanostructured materials and coatings, over a wide range of length scales, thus providing new insights into the actual mechanisms that controls the in-service performances of nano-coatings and micro-devices; and will form the basis of future international standardization of the FIB-DIC methods for micronscale residual stress analysis.

**Support for Programme Challenge, Roadmaps, Government Strategies** Supports:

- NMS strategies for advanced materials and manufacturing.
- NPL strategies for residual stress, full field strain measurement, microstructural analysis, miniaturised testing

**Synergies with other projects / programmes**

- Builds on and extends the work started in MicroRS Materials 2010 project, and previous DIC projects
- Takes developments in FIB machining from Materials 2010 project Grains to Structures and imaging capabilities from Materials 2008
- Is relevant to work on stress corrosion cracking, machining induced damage, surface characterisation, NMO Materials and directly funded projects

**Risks** Main technical risk is that the imaging methods do not have the stability or resolution to be able to analyse the strain field and that the models cannot be applied to derive the residual stress. Current work is indicating that the technical issues can be overcome and initial measurements at NPL and amongst the project partners have proved the potential of the technique. The management risk is that project partners do not provide materials expertise to plan, mitigated by our ability to provide materials collected from UK suppliers

**Knowledge Transfer and Exploitation**

- NPL leads the dissemination activities and will promote the technology to a wide range of UK industry, including the existing IAG memberships to aid better understanding of stress issues and to develop better designs and processes to improve product performance.
- A reliable test method and new capability will be developed and made available to UK Industry
- A Good Practice Guide will be produced to inform the practitioners and end users of this methodology
- Developments will be published in a peer review journal or conference
- The work will support the development of new international standards in the longer term

**Co-funding and Collaborators**

The project provides co-funding for FP7 project iSTRESS and work in the MicroRS project. The FP7 partners are: University of Rome “Roma TRE”(Uniroma3); NPL Management Ltd (NPLML); University of Oxford; Karlsruhe Institute of Technology (KIT); University of Erlangen-Nürnberg (FAU); Fraunhofer-Gesellschaft zur Foerderung der angewandten Forschung e.V.; Eidgenössische Technische Hochschule (ETH) Zurich; University of Brescia Italy University; Robert Bosch GmbH Germany; Berliner Nanotest und Design GmbH; TESCAN A. S. Czech Republic; Thales Research & Technology. Details available on request.

**Deliverables**

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Project Title: Micro-scale and nano-scale thermophysical and thermomechanical metrology

Lead Scientist: Martin Rides

Scientist Team: Martin Rides, Angela Dawson, Tony Maxwell

Sector: Advanced Manufacturing

Project Champion: To be confirmed

Summary
To develop metrological capability to investigate and characterise thermophysical and thermomechanical properties of micro and nano-structured materials to support their design and manufacture. NPL's emphasis will be on thermomechanical measurements. Supporting nanocomposites, automotive, microelectronics and instrument manufacturers. Further applications in a wide range of high technology sectors including medical and organic electronics.

The Need
To realize the benefits and optimise the performance of nanostructured or very thin film materials, product and tool designers need to understand their thermal characteristics and behaviour. There is a need to provide a measurement platform to reliably characterise materials at very small scales. Established methods for heat transfer properties, due to their approach and/or size cannot access properties on the micro-scale. These methods fail to identify local changes in physical properties and their causes, for example microstructural changes, critical dimensions changes, etc. Local probing techniques such as atomic force microscopy (AFM) are currently being developed to probe properties on the micro and nano-scales. Underpinning metrology work is required that is unlikely to be funded by industry to establish a quantitative, rather than merely qualitative, basis for such measurements.

The Solution
Scanning thermal microscopy (SThM), utilising a thermally active tip mounted on an AFM platform, can be used to probe the thermophysical and thermomechanical properties of materials on the micro and nano length-scales, yet is largely qualitative. This proposal aims to develop and establish validated, quantitative methods, procedures and reference materials for the measurement of thermophysical and thermomechanical properties of micro and nano-scale structured materials and products.

Project Description (including summary of technical work)
Metrology tools for the quantitative micro-scale and nano-scale thermophysical and thermomechanical characterisation of materials will be developed. The technical work is divided into 3 parts:

- Specifications and methodology – develop test materials and nano-scale terminology;
- SThM evaluation – SThM experiments and evaluation for metrology;
- Dissemination and evaluation – input into draft standard, Good Practice Guide for SThM technique, scientific paper on developed SThM measurement technique and uncertainties associated with technique.

Impact and Benefits
This project will enable the characterisation and design of improved multiphase, micro-structured and surface engineered materials, e.g. thermoelectric devices, enabling the development of new and improved products. It will enable device designers to model and manufacture reliable high-density electronics, polymer electronics, optical and telecommunications devices of improved performance and improved thermal management. Numerous industry sectors such as semiconductor, automotive, aerospace, information and communication technologies will benefit from this project. Accurate thermophysical measurements at sub-100 nm will enable industry to:

- Address the lack of understanding of the new generation of electronic components and failure mechanisms (reliability, life time),
- Characterize and optimize the thermophysical properties of:
  - new nanostructured materials such as nanoscale multilayered interphases used as thermal barriers in nuclear, automotive and aerospace industries,
  - nanoporous media used as insulating materials in building,
  - nano-objects such as nanotubes and nanowires integrated in electronic components or for the development of new generations of thermoelectrical devices, such as MEMS and NEMS.

The project will lead to the development of an International Standard for calibration of SThM probe measurements and the establishment of a world leading measurement facility for quantitative micro-scale and nano-scale thermal measurements at NPL.

Support for Programme Challenge, Roadmaps, Government Strategies
This project aligns with the Polymers roadmap for the development of “Thermomechanical metrology at the micro and nano scale” and with NPL’s strategy on “Measurements and modelling to characterise atoms, molecules, bio-systems and materials.”
The proposal addresses the NMS overarching strategy of Growth with a particular emphasis on high value manufacturing using polymeric materials, e.g. polymer electronics. It also addresses the challenges of Energy, for example polymer photovoltaics where characterisation of components is key. In particular encapsulation technologies can be more closely controlled using this technology and health sectors will benefit through micro-scale characterisation of medical devices and delivery systems.

Synergies with other projects / programmes
The project proposal will add value to the work carried out in current NMS project 115335 – MAT11022 by continuing further development of the quantitative thermal probe techniques for micro-scale and nano-scale characterisation of the thermophysical and thermomechanical properties of materials with development of standardisation for the techniques. The project proposal also adds value to the technical advances made in thermophysical properties on the micro-scale projects 113523_LS001_P12 and 114463_LS002 2010 18. The project would also compliment the EMRP projects on high temperature nanoindentation (T3D) and the on the nanoindentation of viscoelastic materials (MeProVisc). Beyond the life of the project this work will enable future developments in the characterisation of polymer electronics, additive manufacturing and nanocomposites.

Risks
- Difficulties in achieving consensus. The lack of consensus has a low impact on the project (consensus within the Standardisation activity will be more critical). Early and ample focused discussion groups have been scheduled to avoid misunderstandings and establish consensus.
- Sensitivity of methods to measured parameters is insufficient. Technically the consortium is strong. A British manufacturer of probes has been brought in to the consortium to produce probes. Modelling and experimental expertise has also been brought into the consortium to aid understanding of micro-scale heat transfer processes.
- AFM failure Alternative AFMs have been identified.

Knowledge Transfer and Exploitation
- Exploitation via the QuantiHeat project consortium directly via consortium members and their customers, e.g. IBM, Kelvin Nanotechnology and Thales.
- Scientific and conference papers demonstrating validity/applicability of methods.
- NPL Good Practice Guide (GPG) for conducting measurements using the SThM technique
- Input into the development of ISO Standards.
- Industrial Advisory Group, measurement services and consultancies.

Co-funding and Collaborators
Co-fund will be provided by FP7 QUANTIHEAT due to start 1 September 2013. The consortium is made up of 16 partners including NPL and other NMIs, manufacturers, and universities. There is a 60% chance of success as the QUANTIHEAT proposal has passed to second stage. Details available on request.

Deliverables

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Metrology for thermal protection materials

### Summary
Recent developments in polymer, aerogel and fibrous based composite systems used for thermal protection have shown the potential to provide thermal performance that is several-fold better than conventional insulation materials. However, there is currently no reliable metrology framework with which to evaluate their performance, including the validation of their use in safety critical engineering. The significant disagreement between reference laboratories and also different measurement techniques has resulted in over engineering or under performance, which causes an increase in financial costs and safety risks. Traceable qualification of improvements in high-performance thermal protection systems will have significant and far reaching impact across most of Europe’s Key Enabling Technologies and industries. This Joint Research Project will establish a thermal conductivity measurement infrastructure that addresses the whole traceability chain. It will aim to achieve three times better agreement between reference laboratories and it will evaluate the viability of improving transient and other industrial measurement techniques up to 800 °C. Reliable thermal measurements will then allow manufacturers to systematically demonstrate to their potential customers the performance of their products. This will also allow users, such as aerospace, power plants, and safety systems for industrial facilities and transportation to reduce waste, cost of production and disasters/incidents.

### The Need
The competitiveness of UK industry in a modern low carbon economy will rely on sustainable advanced manufacturing, which urgently requires reductions in cost, waste and carbon emission, and improvements in process efficiency.

- Measurement of thermal conductivity for the purpose of research and development can often produce scatter of over 100 %, (Ref. http://www.mersen.com/uploads/tx_mersen/calcarb-rigid-carbon-insulation-brochure-en_14.pdf) which means advanced manufacturing industries cannot reliably select or develop materials for their engineering applications. They are therefore obliged to make much more use of costly full-scale testing during the development of technologies such as aerospace components, structural fire safety systems and process plants. Today’s thermal modelling capabilities provide an opportunity to design industrial systems far more efficiently, but a lack of accurate thermal properties data for materials is the main limiting factor on the predictive power of thermal modelling.
- Despite European government having spent the last fifteen years putting into place new regulations (EU NO. 305/2011) and mandatory standards (including EN 14303 to EN 14309) with the aim of making certified thermal performance data available to industrial users, the current level of agreement between reference laboratories is still three times worse than the maximum 5 % allowed in these regulations. Implementation of these regulations urgently requires the science underpinning thermal conductivity measurements to undergo a step-change improvement.
- The latest EURAMET Roadmap for Thermophysical Properties identifies the need to develop new thermal metrology for advanced engineering. This is also in-line with the challenges facing Europe that have been identified by the Horizon 2020 Framework Programme: efficient use of resource, building industrial leadership in Europe, and a safe and secure society.
- BIPM have identified the metrology needs for dynamic measurements and stated: "...at present, a verification of dynamic measurement capabilities via key comparisons is a long way off, due to a lack of validated methods and accepted procedures".

### The Solution
A metrology infrastructure within Europe will be developed to provide reliable thermal conductivity measurements to support the implementation of the new European regulations and to enable the significant improvements in performance offered by new advanced thermal protection materials to have an impact on a broad range of industrial applications and to contribute to EU competitiveness. This metrological infrastructure will be achieved by bringing together expertise from across five EU national measurement institutes and a wider spread of Collaborators (NIST, USA and industrial laboratories). The significant improvement in the current level of metrology capabilities and capacities and the availability of traceable reference materials will enable the European aims for harmonised standards, fair trade, reduction in energy consumption and improved competitiveness to be achieved.

### Project Description (including summary of technical work)
The project will establish a thermal conductivity measurement infrastructure that addresses the whole traceability chain. It aims to achieve three times better agreement between reference laboratories and to evaluate the viability of improving transient and other industrial measurement techniques with a target temperature of 800 °C. The Joint Research Project will focus on the traceable measurement and characterisation of modern advanced thermal protection materials.

- WP 1 will develop new techniques for the next generation of national standard instruments.
- WP 2 will identify and develop the first-of-their-kind reference materials with a thermal conductivity in the range 0.02 W·m⁻¹·K⁻¹ to 1 W·m⁻¹·K⁻¹ and aiming at temperatures up to 800 °C.
- WP 3 will systematically investigate the limitations of the transient and other industrial techniques.
- WP 4 will for the first time theoretically and numerically determine the effect of radiant heat transfer on thermal...
Impact and Benefits

- The new measurement infrastructure established in this project will immediately provide the metrology that is urgently needed for the implementation of new European regulations (No. 305/2011) for insulation products used in industrial installations. It will allow European manufacturers to meet mandatory CE marking requirements under the new European regulations (No. 305/2011) and to market their high-temperature insulation products to European industry. The global high-temperature insulation product market was € 2.2B in 2011 and Europe is the largest consumer of products with 39 % market share.
- New composite thermal protection materials promise to be thinner, lighter, stronger and thermally more efficient than conventional materials. This offers enormous scope for improvements in performance, efficiency and in the competitiveness of European advanced manufacturing, including the aerospace and automotive industries. Traceable thermal conductivity data will enable engineering companies to rigorously demonstrate the performance of their new technologies to potential customers, which is now an essential part of the procurement supply chain within the energy, aerospace and defence sectors.
- The new infrastructure will also provide reliable thermal conductivity measurement data and will enable the designers of fire engineering (Structural Eurocodes EN 1990 to EN 1999) and transportation safety systems to select the best performance thermal protection materials. This will help to reduce the rate of industrial disasters/incidents and save lives.

Support for Programme Challenge, Roadmaps, Government Strategies

- TSB Strategy – Advanced Materials: materials to withstand more aggressive environments such as high temperature.
- The NMS Strategy - Growth: Knowledge intensive, high value manufacturing and emerging technologies where UK is strong: Measurement and knowledge of the uncertainty of measurement for manufacturing is a key area.

Synergies with other projects / programmes


Risks

- At 800°C the sensors and materials of the NPL High Temperature Guarded Hot Plate are much more likely to fail and will cause several months delay in the project. In fact, the surface of the existing NPL HTGHP has already warped and not meeting the flatness requirements of the measurement standard. This will reduce the quality of the measured data and will damage NPL’s reputation in this field. The mitigation will be to commission a new set of heater plates to replace the existing ones before the start of the project, but an extra £20k will be needed to cover the material and manufacturing costs.
- Based on the studies that will be carried out in WP1, NPL will provide a concept design and guidelines for a next generation of high temperature guarded hot-plates that can reliably measure thin advanced thermal protection materials. It is to the benefit of the competitiveness of NPL and UK industry (e.g. Rolls-Royce and Bombardier Belfast) that we immediately turn the design into a full set of engineering drawings. This is so that we’ll be ready for manufacturing or at least have solid information for the decision of manufacturing. However, this means that an extra £40k will be needed to cover the engineering design cost.

Knowledge Transfer and Exploitation

Knowledge Transfer: This project aims at thermal protection materials manufacturers and their users. Targeted workshops will be held at key conferences during the project, and the output of this project will be publicised in peer-reviewed journals, trade journals, conferences, stake holder committee meetings, standardisation and technical committee meetings and on-line. Good Practice Guides, training and secondment will be made available to manufacturers and users of HTGHPs. Technical feedback will be input to a new European measurement standard for high temperature Guarded Hot Plate (PrEN/TS 15548-1).

Exploitation: Provide European industry with more comprehensive high quality measurement facilities. New composite and insulation thermal conductivity reference materials up to 800°C will be supplied to industry to provide traceability. Provide consultancy to industrial and academic users on selecting the appropriate thermal conductivity measurement tools for research.

Co-funding and Collaborators

Details available on request.

Deliverables

<table>
<thead>
<tr>
<th>1</th>
<th>Start: 01/07/13</th>
<th>End: 30/06/16</th>
<th>Cost: £</th>
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Deliverable title: Co-funding of EMRP SIB52 THERMO
Summary
Recent developments in thin advanced thermal protection systems have shown the potential to provide thermal performance that is several-fold better than conventional insulation materials. However, there is currently no reliable metrology framework with which to evaluate their performance, including the validation of their use in safety critical engineering.
This project is an add-on to the parent EMRP SIB52 Metrology for thermal protection materials project. The aims of this add-on project are: (1) to prepare the key-equipment resource, the replacement heater plates for the existing NPL high-temperature guarded hot plates, to enable the successful delivery of the EMRP project; and (2) to immediately develop the engineering design drawings of a next generation high temperature guarded hot plate to meet industrial requirements of reliable measurement of thin advanced thermal protection materials.
Reliable thermal measurements will then allow manufacturers to systematically demonstrate to their potential customers the performance of their products. This will also allow users, such as aerospace, power plants, and safety systems for industrial facilities and transportation to reduce waste, cost of production and disasters/incidents.

The Need
• The successful delivery of the parent project (EMRP SIB52), particularly, the development of first-of-their kind high temperature thermal conductivity reference materials (WP2), relies on the performance and the availability of the NPL high-temperature guarded hot-plate (the key equipment). However, the surface of the existing NPL HTGHP has already warped and not meeting the flatness requirements of the measurement standard. This will reduce the quality of the measured data, hence the reference material data, and will damage NPL’s world leading reputation in this field. In addition, at 800°C the sensors and materials of the NPL HTGHP are much more likely to fail and will cause several months delay in the EMRP project.

• In the parent project (EMRP SIB52), design guidelines of a next generation of high temperature guarded hot-plate will be developed to meet industrial requirements of reliable measurement of thin advanced thermal protection materials. The JRP-Consortium has agreed that the EMRP project will not produce a full set of engineering design drawings. This is to ensure accurate measurements of thermal conductivities, and to avoid potential unknown systematic errors in the future which could be caused by apparatuses that use same engineering design drawings. This agreement means that there will be no full set of engineering design drawings available to manufacture and commission a next generation HTGHP for measuring high temperature thin advanced thermal protection materials for UK industries.

The Solution
• Prepare the key-equipment resource ready to enable the successful delivery the parent EMRP SIB52 project by manufacturing and commissioning a set of replacement heater plates for the NPL HTGHP. The replacement heater plates will ensure the quality of the thermal conductivity measurements, the reference data and hence NPL’s world leading reputation in this field. The new replacement heater plates will also significantly reduce the equipment down time from several months to within one month.

• It is to the benefit of the competitiveness of NPL and UK industry (e.g. Rolls-Royce and Bombardier Belfast) that we immediately harvest the output from the EMRP project by developing a full set of engineering drawings based on the new design guidelines. This will prepare NPL for manufacturing and commissioning of the next generation high temperature guarded hot plate in the near future. Reliable thermal conductivity measurements of thin advanced protection materials will enable UK industries to confidently select and develop new thin thermal protection materials.

Project Description (including summary of technical work)
• This add-on project to the EMRP SIB project will manufacture and commission a set of replacement heater plates for the NPL HTGHP before or at the start of the parent EMRP project.

• It will also develop a full set of engineering design drawings for a next generation of HTGHP to meet industrial requirements of reliable measurement of thin advanced thermal protection materials. It will be based on the new design guidelines that will be developed in the EMRP project.
Impact and Benefits

- The replacement heater plates will ensure the quality of the measurement using the NPLHTGHP and the reference data. It will significantly reduce the down time of the key-equipment and enable the successful delivery of the parent EMRP Sib52 project.

- The full set of engineering design drawings will enable NPL and the UK industries (e.g. Rolls-Royce and Bombardier Belfast) to immediately harvest the output from the EMRP project and gaining competitiveness by being ahead of the game.

- New composite thermal protection materials promise to be thinner, lighter, stronger and thermally more efficient than conventional materials. This offers enormous scope for improvements in performance, efficiency and in the competitiveness of European advanced manufacturing, including the aerospace and automotive industries. Traceable thermal conductivity data will enable engineering companies to rigorously demonstrate the performance of their new technologies to potential customers, which is now an essential part of the procurement supply chain within the energy, aerospace and defence sectors.

Support for Programme Challenge, Roadmaps, Government Strategies

- TSB Strategy – Advanced Materials: materials to withstand more aggressive environments such as high temperature.
- The NMS Strategy - Growth: Knowledge intensive, high value manufacturing and emerging technologies where UK is strong: Measurement and knowledge of the uncertainty of measurement for manufacturing is a key area.

Synergies with other projects / programmes EMRP SI Broader Scope Call 2012: Metrology for thermal protection materials.

Risks

- The limited staff resource at the Engineering Design office could cause the delay in the development of the full set of engineering design drawings.
- The cost for the full set of engineering design drawings is an estimate based on our experience in other designs. The real cost will depend on the complexity of the design.

Knowledge Transfer and Exploitation

- The full set of engineering drawing will provide a good start for the manufacture and commission a next generation HTGHP for measuring high temperature thin advanced thermal protection materials in a follow on project.

- Develop a commercial HTGHP apparatus in collaboration with instrument manufactures.

Co-funding and Collaborators

Details available on request.

Deliverables

<table>
<thead>
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<th>Start: 01/07/13</th>
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<th>Cost: £</th>
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<tr>
<td>1</td>
<td>Deliverable title: A set of replacement heater plates to the existing NPL HTGHP commissioned.</td>
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<td>2</td>
<td>Deliverable title: A full set of engineering design drawings for a next generation of high-temperature guarded hot-plates to measure thin advanced thermal protection materials.</td>
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</tbody>
</table>
dislocation modelling, will enable, for the first time, NPL to bridge the length scales that lead to the finite element
exploiting Tony Paxton’s expertise in the tight binding approximation to electronic structure in its application to
and stress corrosion cracking by developing an atomistic description of these phenomena. The s
very strong capability at the macroscopic length and time scales in corrosion especially in hydrogen embrittlement
established for dissemination into industry. The first exemplar project
assume a special position so that metrological standards for calculated electronic structure and total energy can be
functional theory and its impact on ther
of industry and within European funding streams. In some instances, such as computations within the density
special focu
within NPL. This will provide the springboard needed to complete the toolkit over the following 5
and to demonstrate effectiveness by completing two
establish a skeleton of the toolkit, encompassing the most essential tools as well as incorporating
modelling schemes, including software that

Project No. | MAT\2013\15 | Price to NMO | £552k
---|---|---|---
Project Title | Establishing a world leading modelling programme at the NPL | Co-funding target | £200k (post doc, student)
Lead Scientist | Tony Paxton | Stage Start Date | July 2013
Scientist Team | Vlad Sokhan, post-doc, student | Stage End Date | June 2016
Sector | Underpinning metrology | Activity | Establishing New Capability
Project Champion | Rolls Royce | Est Final Stage End Date | June 2016

Summary This project will begin a programme of work that will establish the NPL’s modelling team as a world leading
centre with the primary goals of conducting excellent and innovative science and delivering impact through UK
industrial growth and innovation. As a first step this project establishes the new science area lead, Professor Tony
Paxton, and his work on the atomistic modelling at the NPL; begins the process of identifying strengths and
weaknesses and formulating a strategy for the modelling team for the next 5-10 years; and develops key skills and
capabilities in strategically relevant areas and uses these to address real-world applications from across the materials
division in two exemplar projects.

The Need The importance of material development to growth strategies is widely recognised (EPSRC Materials
Strategy, TSB Advanced Manufacturing Strategy) and the key role that modelling plays in the development, design
and prediction of materials properties and performance is seen as the essential component of materials metrology
and advanced and sustainable manufacturing and energy generation (US Materials Genome Initiative, EU: Materials
for key enabling technologies). In order to support UK industrial growth, energy security and environmental
sustainability NPL needs an integrated facility for multi scale, multi physics materials modelling. This will allow NPL to
respond to modelling challenges thrown up both internally and by industrial and other customers, and to set an
agenda for modelling that will lead to high quality publications, close partnerships with academic institutes and a
continually growing portfolio of capability that will establish the NPL modelling team as a UK centre of excellence.

The Solution A toolkit of software, hardware and people skills will be developed in selected areas of modelling where
NPL can be world leading. Professor Paxton and his team will:
  • Establish an in-house compilation of modern modelling computer programmes spanning the length and time
scales from electronic structure through finite element and component modelling;
  • Bring in modern hardware, such as multithreaded CPU and GPU architectures to allow the development,
testing, validation and running of computations in all of its chosen modelling areas;
  • Retrain, where necessary, current staff and bring in new staff with key competences to establish a central
position in the UK and international materials modelling community.

NPL has initiated this programme of work in the first instance by bringing in Professor Paxton to lead the Modelling
science area. He will bring with him his own expertise in the areas of electronic structure, atomistic and Monte Carlo
modelling schemes, including software that he has authored and which will be brought in house. This project will
support this process by identifying the modelling areas where the team will focus and developing a strategy to
establish world leading expertise and capabilities in these areas. The goal will be to establish a materials modelling
toolkit comprising a triangle of interlinked components, namely software, hardware and skills. This project will
establish a skeleton of the toolkit, encompassing the most essential tools as well as incorporating existing schemes,
and to demonstrate effectiveness by completing two exemplar projects linked directly to current modelling needs
within NPL. This will provide the springboard needed to complete the toolkit over the following 5-10 years, with
special focus on bridging the length and time scales of materials modelling and to initiate new projects both on behalf
of industry and within European funding streams. In some instances, such as computations within the density
functional theory and its impact on thermodynamic modelling, including implementation in MTDATA, NPL will
assume a special position so that metrological standards for calculated electronic structure and total energy can be
established for dissemination into industry. The first exemplar project will build on NPL’s long standing interest and
very strong capability at the macroscopic length and time scales in corrosion especially in hydrogen embrittlement
and stress corrosion cracking by developing an atomistic description of these phenomena. The second exemplar, by
exploiting Tony Paxton’s expertise in the tight binding approximation to electronic structure in its application to
dislocation modelling, will enable, for the first time, NPL to bridge the length scales that lead to the finite element
modelling of materials deformation and fracture. This will be done using crystal plasticity finite element (CPFE) modelling, fed with data from atomistic simulation. We will carry out this programme in partnership with Professor Fionn Dunne at Imperial College Department of Materials. Both of these projects entail timely, adventurous, internationally leading research.

Project Description (including summary of technical work)
The project comprises three phases:

- Phase one is the establishment and incorporation of Tony Paxton and his work at the atomistic scale into the materials division and the development of a strategy for the development of a modelling tool kit;
- Phases two and three are each an exemplar project that will make use of and further develop NPL’s expertise and reputation in selected areas of modelling identified in phase one.

In phase one, an initial assessment of software, hardware and skills already in house at NPL will be made, focusing on the availability of system management skills and augmenting these with existing or new staff as needed. The target at the end of phase one is the skeleton of a modelling capability that incorporates Tony Paxton’s expertise and encompasses the principal length and time scales and the development of a strategy for the further development of a modelling tool kit. Specifically, this means that we have full capability in electronic structure and total energy employing more than one standard code (eg., LMTO and abinit), a working parallel implementation of LAMMPS for molecular dynamics, dislocation modelling using bond order potentials, kinetic and Metropolis Monte Carlo and free energy algorithms. These will be loosely integrated up into the existing finite element and thermodynamic modelling codes. The extent of this integration and the existence of additional bridging as well as other types of models will serve as a measure of further added value at the end of phase one.

Phase two is exemplar project number one and entails development of a modelling strategy for atomic migration under impressed electric fields. This is primarily aimed at an atomistic understanding of elementary processes in electrochemistry, especially corrosion. We will adopt the self-consistent polarisable ion tight binding (TB) theory developed and implemented by Professor Paxton in the last 10 years. As a first target for the end of phase two, we expect to have a self-consistent atomic scale modelling scheme to simulate transport across the Helmholtz double layer under conditions of uniform corrosion.

Phase three is a second exemplar project, the linking between atomistics and finite elements via CPFE. We will build a partnership with Imperial College through the employment of a joint post-doctoral researcher, funded under this project, who will transfer the technology to NPL. We will use atomistic modelling employing tight binding bond order potentials to create data including Peierls stresses, kink and jog formation energies and the energetics of dislocation-impurity interactions. These crystallographic specific data will be fed into CPFE models and application made to deformation and fracture in hydrogen embrittled materials such as steel and in Ti alloys.

Impact and Benefits Modelling of materials properties is essential to enable better design and prediction of material, component, device and plant performance and lifetime. The importance of this predictive modelling capability to design, manufacturing, energy generation, storage and usage efficiency, material security and ultimately industrial growth has been highlighted in key strategies from the US, EU, and UK governments. The development of a clearly defined strategy for NPL’s modelling team, informed through extensive internal and external consultation, and enabled by assessing, adapting and bolstering resources and skills will ensure that the NPL is well placed to deliver the impact in modelling required to meet these challenges.

Support for Programme Challenge, Roadmaps, Government Strategies This project will bring together the disparate threads of the current modelling team at NPL under a strategy designed to establish world-leading capability in specific aspects of materials modelling. The strategy will be informed by the NMS strategy, Metrology 2020, TSB, EU and other key stakeholder strategy documents and through consultation with UK industry as well as with scientists from across the NPL. The exemplar projects have been selected in consultation with science are leads from across the materials and modelling programme and as such represent opportunities for synergies.

Risks Exemplar project one: As yet there is no quantum mechanical scheme to describe mass and charge transport under conditions of uniform corrosion and so the work is timely and innovative. An extension into non uniform corrosion at either crevices or cracks is highly speculative and will form a longer term strategy beyond the time scale of this proposal. The scope in exemplar project two has not yet been attempted but parts of the work are already well established, namely the dislocation modelling in pure metals and the CPFE. In the detailed plans for both exemplar projects we will include risk management through fall back positions and intermediate milestones.

Knowledge Transfer and Exploitation
- Peer-reviewed journal articles, conference papers;
- Presentations at conferences, workshops, and industrial meetings;
- Development of software
- Collaboration with Imperial

**Co-funding and Collaborators:** In addition to the internal collaboration with materials scientists that will be established in the exemplar projects, collaboration will be established with Professor Fionn Dunne at Imperial College in the area of crystal plasticity. Further collaborations with industrial and academic partners are envisaged as the project progresses and areas for modelling capability are identified and targeted. Membership of the Thomas Young Centre will provide NPL with access to and a central role in the development of cutting edge modelling research.

**Deliverables**

<table>
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<th>Deliverable</th>
<th>Start:</th>
<th>End:</th>
<th>Cost:</th>
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<tr>
<td>1</td>
<td>July 2013</td>
<td>June 2014</td>
<td><strong>Deliverable title:</strong> Development of a clear strategy for modelling at NPL and identification of the skills and capabilities required to deliver this (includes Vlad’s time)</td>
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<tr>
<td>2</td>
<td>July 2014</td>
<td>June 2015</td>
<td><strong>Deliverable title:</strong> Delivery of exemplar project 1: Working modelling strategies for atomistic description of uniform corrosion</td>
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<tr>
<td>3</td>
<td>July 2014</td>
<td>June 2016</td>
<td><strong>Deliverable title:</strong> Delivery of exemplar project 2: Linking length scales between atomistics and FE; implementation of CPFE (needs to cover joint post doc with Imperial and case studentship)</td>
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<td>4</td>
<td>July 2013</td>
<td>June 2016</td>
<td><strong>Deliverable title:</strong> Membership of the Thomas Young Centre</td>
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The National Measurement System is the UK’s national infrastructure of measurement Laboratories, which deliver world-class measurement science and technology through four National Measurement Institutes (NMIs): LGC, NPL the National Physical Laboratory, TUV NEL The former National Engineering Laboratory, and the National Measurement Office (NMO).