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H2DRI Pilot Project Public Report April to October 2022



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SUMMARY

H2DRI PILOT PUBLIC REPORT APRIL TO OCTOBER 2022

O Milling, C Atkinson, M Allan

This is the Final Report for the H2DRI Pilot Project covering the period from April to October 2022. H2DRI (Hydrogen Direct Reduction of Iron) is a collaborative project between the Materials Processing Institute, Teesside University and C-Tech Innovation Ltd, with materials and expertise support from Rio Tinto UK and Swansea University Steel and Metals Institute (SaMI) a sub-contracted partner. This project was funded under Phase 1 of the BEIS £55m Industrial Fuel Switching Programme within the £1bn Net Zero Innovation Portfolio.

The project has succeeded in its aims to:

- Inform the operating conditions and scale up requirements required to design, build and operate a hydrogen direct reduction furnace for iron ore that can be coupled to existing steelmaking facilities in order to demonstrate tonnes per day hydrogen electric ore-to-steelmaking and establish an accessible national R&D focal point to accelerate adoption of the technology as a fuel switch from fossil coal and gas.
- Showcase and consolidate the consortium's collaborative capability in laboratory and small pilot hydrogen iron reduction, steelmaking and characterisation.
- Confirm feasibility of demonstrating and progressing the technology readiness level of secondary innovations during Phase 2, which would include the application of microwaves to the reduction process, and different approaches to carburisation of the reduced iron product prior to making it into steel.

The project succeeded in becoming a focal point for interest in UK hydrogen ironmaking possibilities from companies across the supply chain. The programme's UK Hydrogen DRI Interest Group now encompasses ore and pellet suppliers, iron makers and steelmakers, hydrogen suppliers, electrolyser developers and finished product producers, as well as key academic, trade and policy actors.

MATERIALS PROCESSING INSTITUTE H2DRI PILOT PROJECT FINAL REPORT APRIL TO OCTOBER 2022

1. INTRODUCTION

The “H2DRI Pilot” project started 28 March 2022 and was completed on 28 October 2022.

The project was funded under Phase 1 of the BEIS £55m Industrial Fuel Switching Programme within the £1bn Net Zero Innovation Portfolio.

1.1 Background: Carbon Emissions from Steelmaking

The steel industry is one of the largest contributors to worldwide industrial carbon dioxide emissions⁽¹⁾. Indeed, the sector has been reported to contribute 7 to 9% of the direct carbon dioxide emissions from the global use of fossil fuels⁽²⁾. Approximately 70% of global crude steel is produced via the blast furnace route, which largely relies on coke as the energy source and reducing agent for conversion of iron ore. Coke is a high carbon content coal-derivative and its use as a fuel and reductant generates carbon dioxide as a by-product, and thus the significant pollution caused by its use⁽³⁾. A 2022 study estimated 3.1 GT out of a total 3.6 GT carbon dioxide emitted globally by steel production in 2021 was from the blast furnace-basic oxygen furnace (BF-BOF) route, and that this output has doubled in the past 20 years⁽⁴⁾. As part of collective efforts to reduce global greenhouse gas emissions, more environmentally sustainable solutions are required. One such approach in the steel industry looks certain to be direct reduction of iron ore using hydrogen as the reductant, resulting in the formation of water instead of carbon dioxide as the main by-product⁽⁵⁾. Furthermore, electrification of the heating processes powered by renewable energy, in addition to direct reduction using hydrogen, has been proposed to offer carbon dioxide emission reductions of up to 95%, representing a highly attractive solution to the steel industry⁽⁶⁾.

1.2 Project Consortium

The project was a collaborative project between the Materials Processing Institute, Teesside University (TU) and C-Tech Innovation Ltd, Rio Tinto UK and Swansea University Steel and Metals Institute (SaMI), a sub-contracted partner.

The project was managed by the Materials Processing Institute (the Institute) with test work being carried out at Teesside University, C-Tech Innovation, SaMI and the Institute with materials and expert support from Rio Tinto UK.

The combined research resources of the partners proved a powerful tool for unlocking different aspects of the reduction processes and generated a level of understanding not available from literature, much of which has been corroborated by (unpublished) work presented by the HyBRIT programme in Sweden. The validity of a collaborative, inclusive network of facilities to support the core technology project was clearly demonstrated.

This report outlines both the experimental work carried out and the feasibility studies for a potential Phase 2 project for the creation of a pilot scale (more than 1 tonne per day) hydrogen iron production plant.

1.3 Industrial Interest Group

The 'UK Hydrogen DRI Interest Group' was started during the Phase 1 proposal preparation to ensure appropriate industrial and academic steer of the bid. There have been between 15-20 participants in the Group meetings representing the major UK iron and steelmakers, raw material suppliers, other supply chain actors and academic networks. The group expanded through the project.

Project publicity and dissemination activities were carried out throughout the project. A summary of publications is in the Appendix.

1.4 Project Outline

Section 2 describes the raw materials used for iron reduction and the reduction experiments using both the iron ore reduction (IOR) rig at SaMI and the lab based experiments at TU.

Section 3 describes microwave-enhanced reduction experiments carried out at C-Tech Innovation.

Section 4 outlines melting experiments and analyses of the steels produced by these experiments.

Section 5 provides an overview of the planned Phase 2 project bid and Section 6 conclusions.

2. IRON ORE REDUCTION FEASIBILITY

2.1 Characterisation of Materials

The analyses of the raw materials and the reduced products were carried out at the Materials Processing Institute. The techniques used to characterise the materials are described below:

- XRD analysis was carried out on finely ground samples using a Malvern Panalytical Empyrean X-ray diffractometer fitted with a copper X-ray tube and a Bragg-Brentano mirror.
- XRF spectroscopy was carried out on fused bead samples using a Rigaku ZSX Primus IV analyser. A quantitative analysis application was used to analyse the beads.
- SEM analyses were carried out using a ZEISS 300 VP Sigma instrument. This is fitted with Oxford Instruments detectors controlled by the AZtec operating system and is equipped with a Wavelength Dispersive X-ray Spectrometry (WDS) analytical package.

2.2 Sample Provision & Storage

The project consortium agreed on three types of iron ore feedstock to be studied for this project. They were lump iron ore (Lump Ore), blast furnace ore pellets (BF Pellet) and direct reduction ore pellets (DR Pellet). Ores and feedstocks vary between suppliers due to mine geology and ore preparation processes. These three types of feedstock are representative of the range of materials commonly used in the steel industry: lump ores, which are pure rock that has undergone only basic separation and crushing, through to prepared blast furnace pellets and high purity pelletised direct reduction ores.

Typically, direct reduction processes only use high purity pelletised ores. One of the aims of this project was to understand the implications of using standard grade materials in direct reduction processes and to understand how the slags and other by-products present in standard ores may require processing when steels are made directly from these direct reduced feedstocks.

Subsamples of the Lump Ore, DR Pellet and BF Pellet were taken by the Materials Processing Institute for compositional analysis and distribution to the partners. As the Lump Ore was a less homogenous material, the whole sample was spread out and subdivided systematically to allow representative subsamples to be taken for the experimental programmes of the partners (Fig. 1).



FIG. 1: DIVIDING LARGE LUMP ORE SAMPLE INTO REPRESENTATIVE SUBSAMPLES

The feedstock materials were subjected to X-ray fluorescence (XRF) and X-ray diffraction (XRD) analysis to identify the quantities of different iron phases and impurities in the samples. The XRD methodologies used at the Institute were refined to consider the range of components expected in the samples to ensure accurate and repeatable results.

Good correlation was found between supplier compositional analyses (not shown here) and the XRF and XRD analyses carried out at the Institute (Tables 1 to 3).

TABLE 1

COMPOSITION OF LUMP ORE – XRD SPECTROSCOPY

Mineral	Formula	Composition (wt.%)
Hematite	Fe ₂ O ₃	63.8
Goethite	FeO(OH)	32.3
Quartz	SiO ₂	3.9
Total Hematite with water		96.1

TABLE 2

COMPOSITION OF DR PELLETT - XRD SPECTROSCOPY

Mineral	Formula	Composition (wt.%)
Hematite	Fe ₂ O ₃	96.5
Katoite	Ca ₂₄ Al ₁₆ Si ₂₄ O ₉₆	1.5
Magnetite	Fe ₃ O ₄	1.4
Quartz	SiO ₂	0.6

TABLE 3

COMPOSITION OF BF PELLETT – XRD SPECTROSCOPY

Mineral	Formula	Composition (wt.%)
Hematite	Fe ₂ O ₃	93.5
Sillimanite	Al ₂ O ₅ Si	0.4
Magnetite	Fe ₃ O ₄	0.2
Quartz	SiO ₂	4.1

Analytical data were in good agreement, with only minor differences accountable by sample preparation and instrument factors, within the range of acceptable analytical reproducibility. These results gave confidence that subsequent XRD and XRF analyses of the reduced samples from the partners' experiments would be valid.

2.3 Iron Ore Reduction (IOR) Experiments

2.3.1 Objectives

Iron Ore Reduction (IOR) experiments were designed to investigate the potential variability of reduction reactions and final reduced products as influenced by the feedstock materials, and to demonstrate the difference between pure hydrogen reduction and conventional hydrocarbon reduction. Typically, direct reduction processes only use high purity pelletised ores. One of the aims of this project was to understand the implications of using standard grade materials in direct reduction processes and to understand how the slags and other by products present in standard ores may require processing when steels are made directly from these direct reduced feedstocks.

2.3.1 Experimental Procedure

The programme of iron ore reduction (IOR) experiments using the ISO standard test equipment (Fig. 2) at Swansea University Steel & Metals Institute (SaMI) was completed on Lump Ore, DR Pellet and BF Pellet samples. The standard reduction experiments used both pure carbon monoxide (CO) and hydrogen (H₂) gas.



FIG. 2: IOR FURNACE AT SaMI

An example of the experimental profile and the physical appearance of a Lump Ore particle before (sample mass = m_0) and after (sample mass = m_2) reduction is shown in Fig. 3. The metallised appearance of the Lump Ore after reduction was apparent.

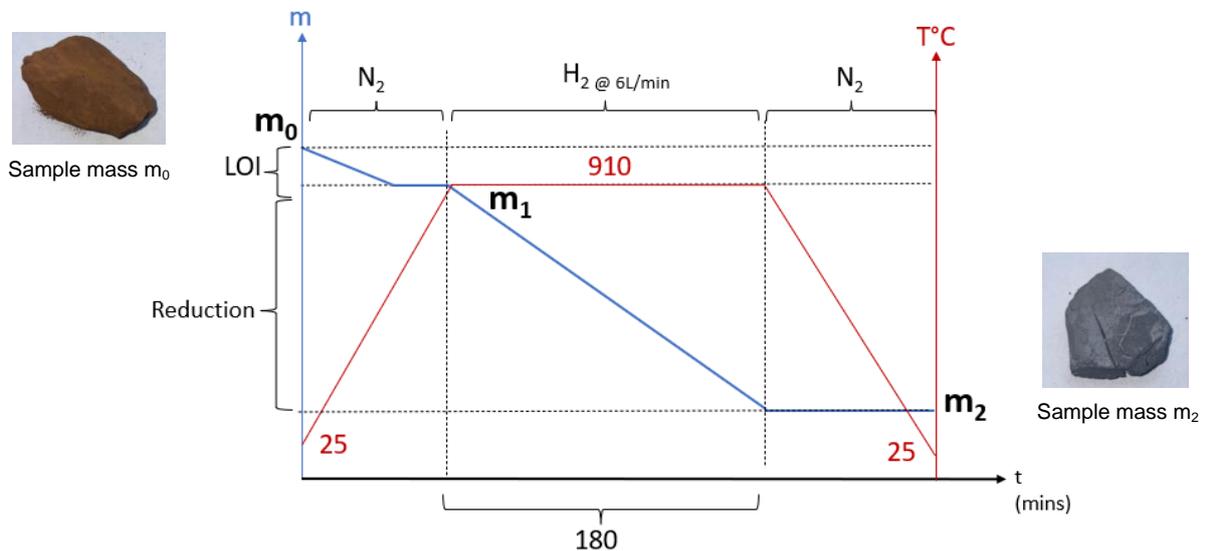


FIG. 3: EXAMPLE OF IOR EXPERIMENTAL PROFILE - LUMP ORE REDUCTION IN HYDROGEN

2.3.3 Results & Discussion

H2 v CO Reduction

The standard IOR experiments conducted on DR Pellet and Lump Ore samples showed significantly higher reduction rates with more complete reduction achieved when using hydrogen as the reducing agent than when using carbon monoxide (see Figs. 4 & 5).

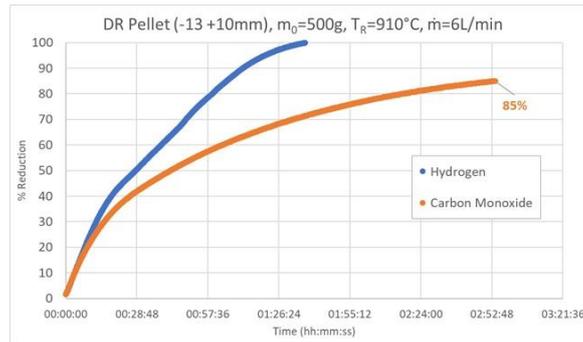


FIG. 4: REDUCTION EXTENT - DR PELLET IN H2 & CO

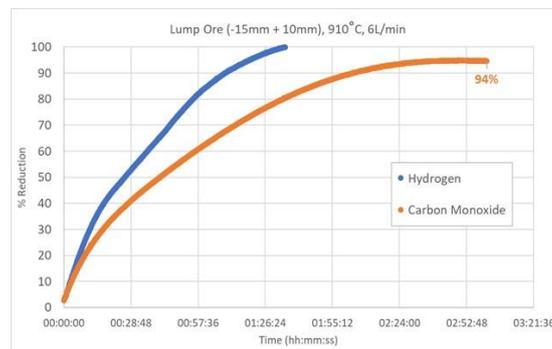


FIG. 5: REDUCTION EXTENT - LUMP ORE IN H2 & CO

When hydrogen was the reductant the sample mass loss, which indicates reduction rate, showed a rapid decline from the start of the experiment before levelling out after about 20 minutes. The reduction rate then showed a pronounced increase again after about 43 minutes, finally declining from about 48 minutes to the end of the experiment. This effect was observed with both DR Pellet and Lump Ore, although less pronounced with lump ore.

When pure CO reductant was used the reduction rate was slower than with hydrogen and reduced rapidly for the first 20 minutes of the experiment. The reduction rate then continued to reduce, but at a lower rate of change.

The inflection points in the reduction rates likely indicate changes in the ore phases being reduced throughout the experiments. The acceleration in rate seen at 43 minutes when using hydrogen reductant is of particular significance (see also the findings of the TU experiments, Section 2.4).

During the reduction with carbon monoxide complete metallisation was not observed with the DR Pellet samples. To investigate if metallisation could be increased with increased reductant gas flow, a set of experiments were conducted at higher CO flow rates of 10 l/min and 13 l/min (Fig. 6). The extent of reduction was only marginally improved by increasing the gas flow rate and there appeared to be a limit to the amount of reduction that could be achieved by CO at 910 °C.

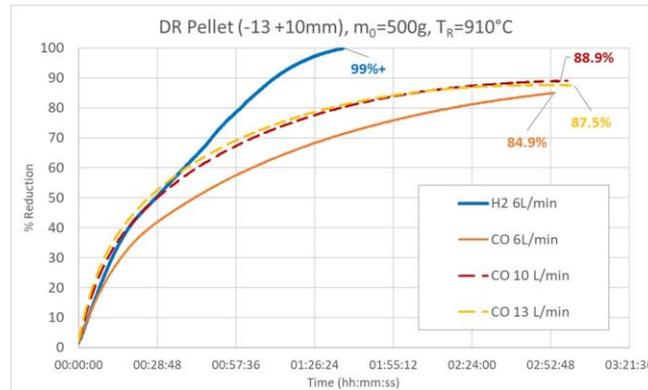


FIG. 6: REDUCTION EXTENT & RATE AS FUNCTIONS OF REDUCTANT GAS FLOW RATE

The mass loss results from a series of additional hydrogen reduction experiments on larger samples of DR Pellet and Lump Ore demonstrated that although sample mass influenced the rate of reduction, the overall extent of conversion was unaffected, within the limits of the experiments.

Strength Analysis

The reduced DR Pellet samples from the SaMI experiments underwent strength testing to discover whether there was a strength difference in the iron produced in the different test regimes. Tumble testing was carried out on the reduced samples with the degree of disintegration after tumbling providing a measure of material strength.

Figure 7 shows the variation in disintegration as the degree of metallisation increases. As metallisation proceeds, the material recovers and disintegration levels off, with the most pronounced effect seen with hydrogen reduction, the material also being stronger than the CO reduced material above 95% reduction.

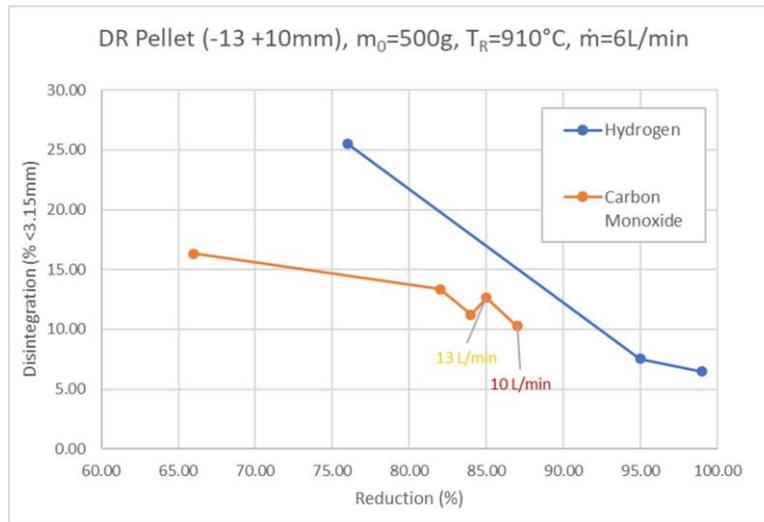


FIG. 7: DISINTEGRATION AS A FUNCTION OF METALLISATION: DR PELLETT

High levels of disintegration appear to be due to phase changes (FCC, BCC, etc.) in the iron crystal structure, that link to the degree of metallisation and the reaction time (Fig. 8). These cause volume changes that impart internal stresses on the material, leading to fracturing at all scales from the micro-level to the macro. At the longer reaction times of CO reduction, it could be speculated that some thermal annealing might come into play and that carburization of the iron could be influencing disintegration behaviour.

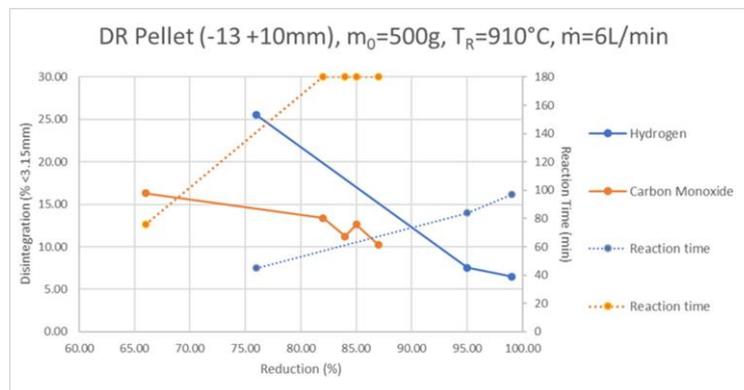


FIG. 8: DISINTEGRATION RELATED TO METALLISATION & REACTION TIME: DR PELLETT

To investigate the possibility of crystallographic phase changes adversely affecting the strength of the DR Pellet on reduction, experiments on BF Pellet under the standard IOR hydrogen reduction conditions were carried out. BF Pellet is a blast furnace feedstock, essentially pelletised fine ore with flux additions, which has a broader iron oxide composition than the more refined DR Pellet. Reduction of this material should provide greater opportunity to study the effect of crystallographic phase changes in more detail. Irregular behaviour was noted in the mid-region of the reduction profile for the BF Pellet sample where the hydrogen reduction rate is changing considerably. Cracks are likely to form due to stresses created during crystallographic phase changes as different iron oxide phases are progressively reduced and fresh oxide is exposed.

Strength testing of the 100% reduced samples recovered at the end of the experiments showed significantly higher disintegration values for the DR Pellet than the BF Pellet. It is proposed that the reducing BF Pellet recovers strength as metallisation progresses at the low rate toward the end of the experiment.

Hydrogen Utilisation During Reduction

The standard conditions of the IOR experiments were chosen to ensure that an excess of reducing gas was present to eliminate the influence of gas concentration on the reduction process. However, this raised the question of the level of gas utilisation during the experiments. Figure 9 shows the results for the DR and BF Pellet reduction experiments, with the mass loss rate converted to hydrogen utilisation.

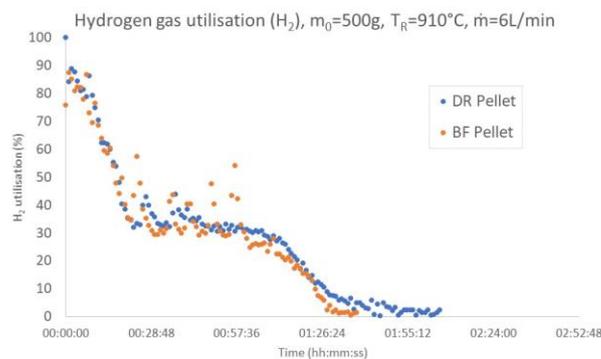


FIG. 9: HYDROGEN UTILISATION FOR ISOTHERMAL REDUCTION AT 910 °C

For both experiments the total hydrogen utilisation was close to 30%, giving an excess of hydrogen well above the requirements of the reduction process. However, it can be seen from Fig. 9 that hydrogen utilisation changed over the course of the reaction, from 77-79% over the first third of the reduction process to 15-20% for the final third, in line with the reduction rate and the degree of metallisation of the materials. As expected, hydrogen utilisation in the BF Pellet experiment was greater than that for the DR Pellet by virtue of the greater content of mixed iron oxide species present in the BF Pellet.

The reduced samples were sent to the Materials Processing Institute. Compositional analyses and melting experiments in the laboratory scale furnaces (Section 3) investigated the nature of the steel and by-products produced during the reduction experiments.

Comparison of IOR and TPRx Reduction Processes

A set of experiments were conducted to try to duplicate the experimental procedure used by Teesside University in their lab-scale TPRx rig (Section 2.4) to investigate if a similar level of detail of hydrogen utilisation could be achieved. Instead of the standard IOR experimental regime conducted isothermally at 910 °C, samples of DR Pellet, BF Pellet and Lump Ore were subjected to a temperature programmed heating regime.

Total hydrogen utilisation varied from 17.50% for the Lump Ore to 17.96% for the DR Pellet and 19.10% for the BF Pellet. The mass loss rate curves were similar in appearance to those produced for

water evolution in the Teesside University experiments, with the DR Pellet and BF Pellet showing more detail than the Lump Ore. The BF Pellet showed similar irregular behaviour over the 40-80% reduction range, as in the isothermal experiments.

It was noted that a high mass loss rate at low temperature was recorded in the Lump Ore experiment. This is believed to be due to the early dehydroxylation and reduction of the Goethite present in the ore (absent in the DR Pellet and BF Pellet). Similar behaviour was observed by Teesside University.

Overall, the reduction behaviour in the modified IOR experiments followed a similar pattern to the TPRx experiments giving confidence that the mechanism being observed by the different techniques were leading to very similar reduced products.

DR Pellet Carburization

One of the considerations for an H-DRI product to be fed into the electric arc furnace (EAF) is the value of inclusion or addition of carbon in the material. Conventional operation of the EAF when melting and refining large quantities of scrap often requires additions of carbon to provide extra heat for the melting of the scrap and to produce CO to aid foaming of the slag for oxidation protection of the melt. The possible benefit of carburization was investigated using the IOR to expose reduced DR Pellet to CO in an additional stage.

Fully reduced DR Pellet was heated from room temperature to 230 °C under nitrogen, then CO was passed at 6 l/min through the sample as it was heated to 830 °C in order to carburise it. Finally, the furnace was purged with nitrogen as it cooled back to room temperature. Mass gain was recorded throughout to allow calculation of carbon uptake. Figure 10 shows the results of the carburization experiment.

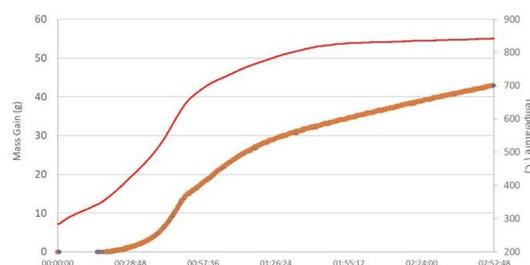


FIG. 10: CARBURIZATION OF REDUCED DR PELLETS

The total mass gain at the end of the experiment was 42.16 g. However, the question remained whether this was the result of carburization.

To provide some clarity, the carburized sample was subjected to a tumble test to determine the degree of disintegration for comparison to a fully reduced DR Pellet sample. By comparing the differences in disintegration between the two samples it is possible to estimate the potential level of carbides in the carburized sample as their presence promotes fracture of the material. By considering the mass gain observed in the carburization experiment, it can be estimated that there is a 1.7% uptake of carbon into the reduced pellets.

This level of carburization could be acceptable to the EAF operator for a carburized H-DRI feedstock, but the increased fines generation caused by higher friability compared with non-carburized H-DRI might reduce its usefulness. Further experiments are planned to refine the technique and end result.

Assessment of Potential Re-Oxidation

The hydrogen reduced material from the IOR experiments was highly metallised and, like all DRI material, could have the propensity to re-oxidise. This can be a serious fire risk as re-oxidation is an exothermic reaction and could make the H-DRI material difficult to handle and store. There is some evidence in the scientific literature that passivation of the H-DRI product is achieved when reduction by hydrogen is carried out below 600 – 800 °C.

To investigate if substantial re-oxidation of the material could occur, a sample of hydrogen reduced DR Pellet was periodically weighed to monitor potential oxygen uptake from the laboratory atmosphere. Figure 11 shows the mass gain curve for the sample over a period of 2 days, demonstrating only slight uptake of oxygen leading to an increase in mass of 0.6 g or 0.07 wt.%. This would be insufficient to cause any serious heat build-up by exothermic reaction in the sample (which remained at ambient temperature) and the level probably reflects re-oxidation at the surface of the metallised particles in the reduced DR Pellet.

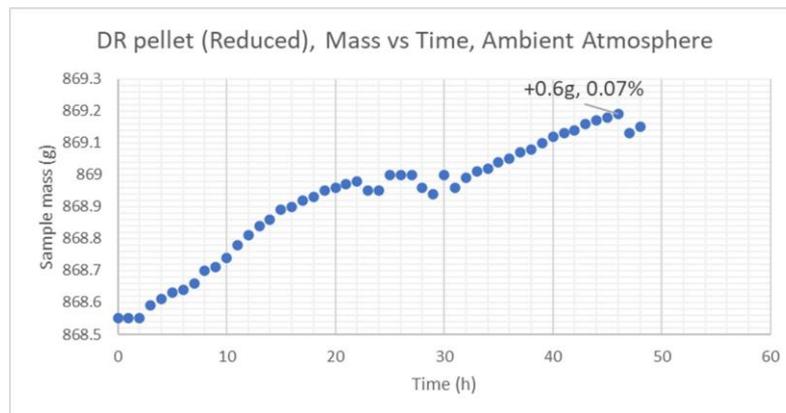


FIG. 11: MASS GAIN OVER 48 HOURS: REDUCED DR PELLETT

Development of ISO Standard Test for H-DRI

Currently ISO 11258 is used as a test to determine the reducibility of iron ores for gas direct reduction. One of the aims of this work is to determine, within the current limits of the test equipment at SaMI, whether a potential ISO test might be developed for hydrogen reduction of iron ores. The pseudo-ISO test carried out used lower gas flow rate than the standard tests due to current test equipment limitations.

A set of comparative experiments were conducted under pseudo-ISO 11258 test conditions to establish the feasibility of developing a test applicable to the H-DRI process.

Table 4 summarises the results, where the total mass losses in the pseudo-ISO test did not reach the theoretical level of oxygen loss, unlike the hydrogen reduction experiments. Not all the pseudo-ISO experiments continued to completion so direct comparisons are difficult. The BF Pellet experiment

showed that hydrogen reduction was more effective than the pseudo-ISO test and that it should be possible to develop a standard test similar to ISO 11258 to characterise the hydrogen reduction behaviour of iron ore.

TABLE 4
SUMMARY OF HYDROGEN REDUCTION V ISO 11258

Sample	Total Fe	FeO	Theoretical Oxygen Content	Total Mass Loss (g)		Reduction @ 180 min (%)		Time to 90% Reduction (min)	
	(%)	(%)	(g)	H ₂	ISO 11258	H ₂	ISO 11258	H ₂	ISO 11258
BF Pellet	65.03	0.076	139.6	139.8	130.2	100	93	71	100
DR Pellet	67.57	0.087	145.0	147.0	133.2*	100	90	76	128
Lump Ore	62.55	0	134.5	138.4	116.9*	100	n/a	85	144 [#]

Notes: * Did not complete (<180 min) # Time to 87% Reduction

2.4 Lab Based Reduction Experiments

2.4.1 Objectives

The Teesside University programme of work utilised a novel Temperature Programmed Reactor (TPR) to investigate hydrogen reduction behaviour. The objectives were:

- To observe / note the temperatures under which reduction of the ores (Lump Ore, DR Pellet and BF Pellet) occurs, in the presence of hydrogen and hydrogen / nitrogen mixtures.
- Sample characterisation by X-ray diffraction (XRD) to detect phase changes.
- To investigate carburization, by introducing methane towards the end of TPR hydrogen reduction experiments.
- To investigate the thermodynamic limitation of iron ore reduction by the presence of moisture.

2.4.2 Experimental Procedure

Samples were oven dried overnight before being placed in the TPR. The schematic in Fig. 12 shows the set-up of the TPR, with stainless steel reactor containment tubes used in the initial set of experiments to bring the sample temperature up to 800 °C. The concentrations of water, nitrogen and hydrogen in the exhaust gas of the reactor using a mass spectrometer (MS) enabled the reduction rates of the samples to be compared. Temperature profiles were also recorded upstream and downstream of the sample in the reactor and all TPR profiles showed minimal temperature perturbation across the samples.

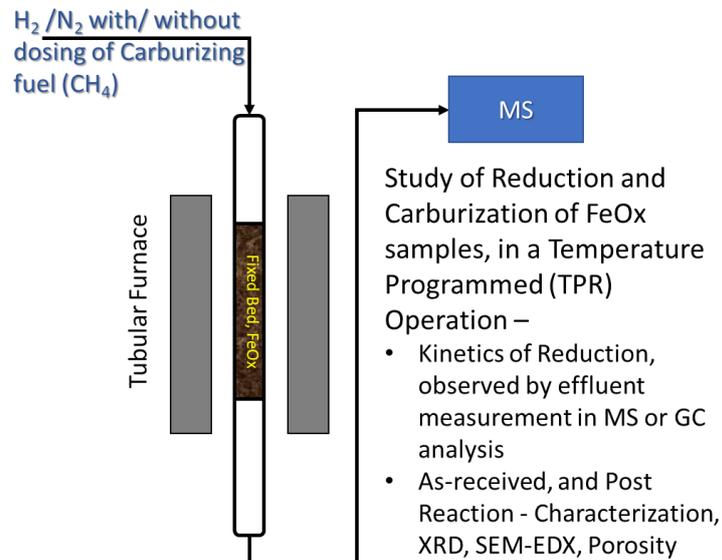


FIG. 12: SCHEMATIC OF THE GENERAL TPR EXPERIMENTAL SET UP

2.4.3 Results & Discussion

First Round Experiments

The first set of experiments determined the conditions required for sample reduction in a hydrogen atmosphere, with a view to gaining the maximum amount of quantitative data on the process. The experiments were:

- Reduction of Lump Ore by 100% hydrogen.
- Reduction of Lump Ore by 10% hydrogen in nitrogen.
- De-hydroxylation of Lump Ore by Temperature Programmed Desorption (TPD) in 100% nitrogen.
- Reduction of DR Pellet by 10% hydrogen in nitrogen.

A TPR experiment was carried out on Lump Ore in an atmosphere of 100% hydrogen. This experiment showed the evolution of water for about 120 minutes, indicating reduction of the iron ore, beginning at 230-250 °C and reaching a plateau around 400 °C. The high concentration of hydrogen produced a relatively flat profile and made it impossible to observe any detail in the change in water concentration in the exhaust of the reactor.

Further experiments were carried out using a mixture of 10% hydrogen in 90% nitrogen to reduce the partial pressure of hydrogen and hence reveal more detail of the reduction kinetics. These conditions going forward are referred to as TPRx. Reduction of a sample of Lump Ore (Fig. 13) showed clear peaks of water evolution and indicated reduction rate changes at approximately 140 and 170 minutes into the experiment (Peaks A & B, Fig. 13).

Peak A, early-stage reduction at a temperature of 380 °C, may in part be due to the freeing of water from hydroxides present in the ore sample. Peak B, second-stage slower reduction observed at 780 °C, reflects the different reduction profiles of the iron oxide phases in transforming to pure iron.

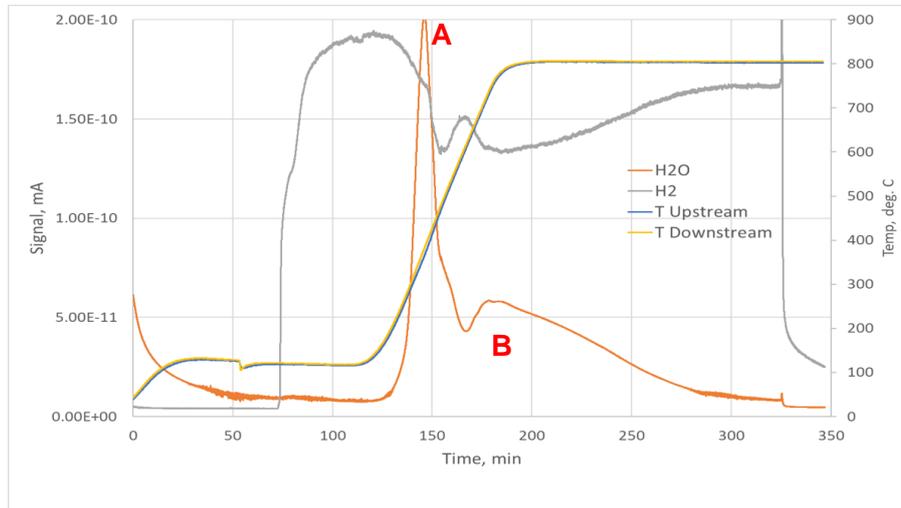


FIG. 13: REDUCTION OF LUMP ORE SAMPLE - 10% HYDROGEN / 90% NITROGEN

There was speculation about the transformation of significant quantities of FeO(OH) (Goethite) in the Lump Ore that would also undergo de-hydroxylation and could potentially contribute to the early reduction peak, Peak A.

The profile from a Temperature Programmed Desorption (TPD) experiment on a sample of Lump Ore where the standard TPR heating regime was followed, but with a pure nitrogen flow (90 ml/min) throughout showed a sharp peak in the region of 350 °C, with further minor peaks above 450 °C. This TPD experiment provided evidence that the early-stage moisture peak observed in the TPR experiment (Peak A, Fig. 13), was most likely due to moisture release by de-hydroxylation of Goethite.

Characterisation of Reduced Lump Ore

The reduction of the iron oxides in the Lump Ore samples was verified by X-ray diffraction (XRD). The resultant material in all three experimental regimes displayed similar XRD profiles, with alpha iron predominating.

Although the reduction of FeOx to alpha iron, Fe(BCC) (Body Centred Cubic crystal structure), is confirmed by XRD, given the limitations of the technique, there is no clear confirmation of 100% reduction of the iron ore. From earlier literature, in this area, there is evidence of non-stoichiometric iron oxide formation of a strongly refractory nature due to the heavy diffusional limitations on reduction under these conditions. It is likely that the reduction will be at least over 95% to the metallic phase at this temperature.

Figure 14 shows photographs of a Lump Ore particle before and after reduction. There was a clear metallic lustre associated with the metallisation of the ore on reduction, with the expected development of high levels of porosity that accompany reduction shown by SEM analysis.



FIG. 14: LUMP ORE PARTICLE: AS RECEIVED AND AFTER REDUCTION

Reduction of DR Pellet

Reduction of DR Pellet samples under the standard experimental conditions (TPRx) showed differently shaped peaks in the evolution of water. Figure 15 shows the TPRx profile of a sample of DR Pellet.

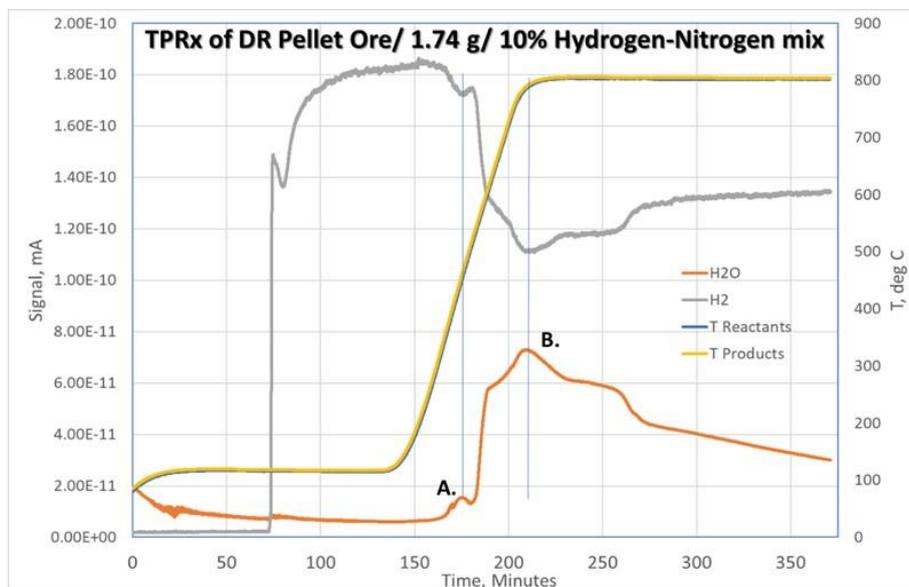


FIG. 15: TPRX PROFILE OF DR PELLET REDUCTION

The onset of reduction was observed at 350 °C, as water evolution began to rise, followed by an initial reduction peak at 450 °C (Peak A.). The main reduction peak was then observed (Peak B.) at 770-780 °C. It was noted that this peak had a shoulder at around 600 °C that warranted further investigation. A more precise experiment was conducted to resolve the origins of the shoulder.

In both experiments, the onset of reduction occurred at about 400 °C, although the peak reduction processes occur in the 600-800 °C region. The evolution of water continues over the isothermal

temperature soak region at 800 °C, which indicates that the reduction is inhibited at high temperatures and the reaction kinetics are limited, most likely by diffusion, as has been suggested in the literature.

Figure 16 illustrates a shrinking core type of reduction process in the larger size DR Pellet. The photographs show a cut-section of a DR Pellet that reveals incomplete reduction in the core, which can be related directly to the incomplete reduction seen in the XRD profile.



FIG. 16: PROGRESSIVE REDUCTION FROM SURFACE TO CORE IN LARGE DR PELLETT

Similar to the morphology of the reduced Lump Ore samples, the reduced DR Pellet samples revealed considerable porosity on examination by SEM. Wavelength Dispersive X-ray Spectroscopy (WDS) revealed negligible amounts of oxygen present after reduction, and predominantly Fe(BCC) as the iron species (Fig. 17).

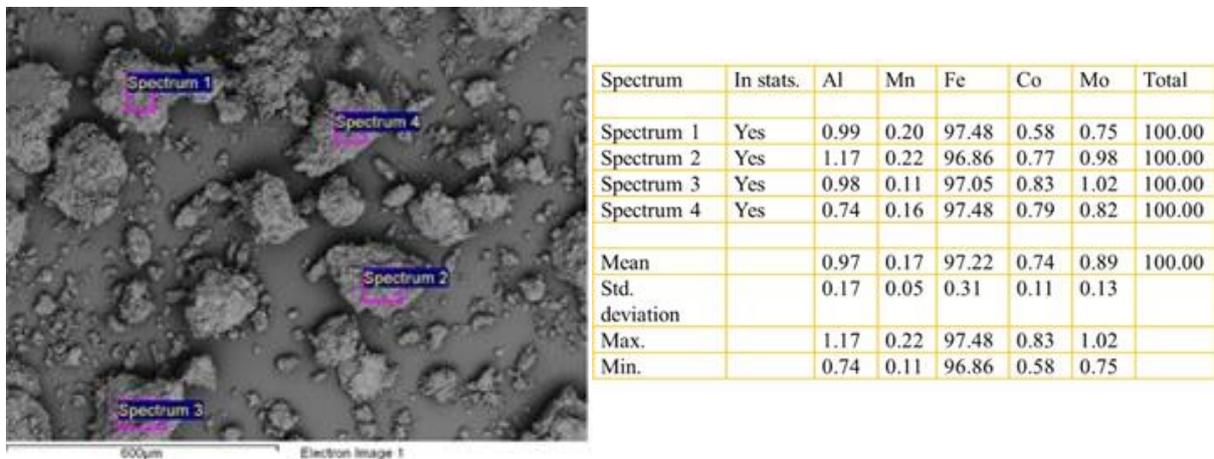


FIG. 17: WDS ANALYSIS OF REDUCED DR PELLETT

2.4.4 Second Round Experiments

The second set of experiments at temperatures up to a maximum of 975 °C were conducted on Lump Ore and DR Pellet samples using the TPRx profile of the first round, with a carburization step introduced after reduction was deemed to be complete. The experiments were:

- Reduction and carburization of Lump Ore by 10% hydrogen in nitrogen.
- Reduction and carburization of DR Pellet by 10% hydrogen in nitrogen.
- Effect of hydrogen concentration on reduction.

Reduction & Carburization of Lump Ore

Figure 18 shows the TPRx profile for reduction and carburization of Lump Ore. The sample was reduced initially under 10% hydrogen in nitrogen and then methane was added to the reduction gas stream to carburize the sample.

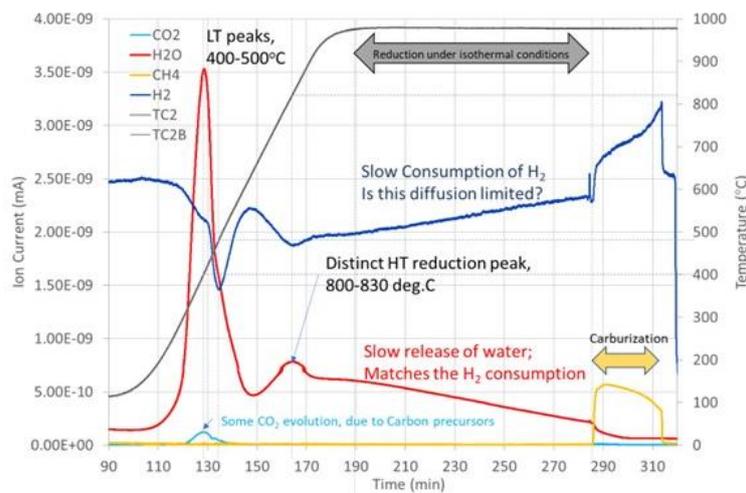


FIG. 18: TPRX PROFILE OF LUMP ORE REDUCTION & CARBURIZATION

As for the first round of experiments, with a higher proportion of Goethite present in the Lump Ore, there is de-hydroxylation with possible conversion of Goethite to Magnetite or Wüstite. De-hydroxylation peaks at around 400 °C, as seen in the TPD experiment. Hydrogen consumption is displaced to higher temperatures, which indicates that the ensuing process is one of reduction, releasing extra water that is seen as a shoulder at ~ 480-500 °C. A distinct high temperature peak occurred at 800-830 °C evidenced by both H₂ consumption and H₂O evolution, followed by a slow reduction under isothermal conditions at 975 °C that could be under diffusional control.

There is clear evidence of carburization on introduction of methane into the reducing gas stream demonstrated by the excess of H₂ produced. This increased as H₂O levels fell to near zero as complete reduction was neared. The carburization occurs as methane decomposes catalytically due to the presence of iron formed by the reduction process. This generates C and H₂ (visible due to the large signal increase) and the carbon will then react with the iron forming the carbide phases expected on carburization.

Reduction & Carburization of DR Pellet

Reduction and carburization of a sample of DR Pellet was carried out using the same procedure as the Lump Ore.

Evidence of carburization ($\text{CH}_4 = \text{C} + 2\text{H}_2$) was again apparent in the increased hydrogen signal corresponding to the methane decomposition.

Characterisation of Reduced & Carburized DR Pellet & Lump Ore

SEM WDS characterisation of reduced and carburized iron in DR Pellet and Lump Ore samples was conducted by the Materials Processing Institute. An analysis of carburized DR Pellet indicated the presence of carbon rich regions, Fe-rich regions and regions of Fe_xC or oxygen-carbides.

For the Lump Ore, carburization results in a large proportion of carbon filaments / nanotubes being formed throughout the sample, and there are still regions where oxygen has not been completely eliminated (e.g. Fig. 19).

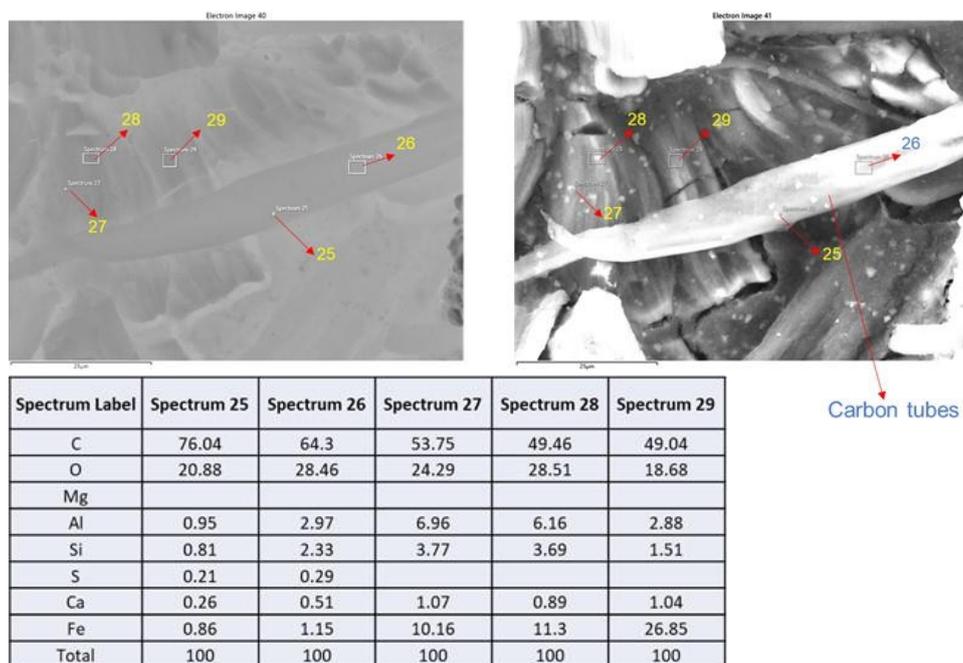


FIG. 19: SEM WDS ANALYSIS OF CARBURIZED LUMP ORE

Effect of Hydrogen Concentration on Iron Ore Reduction

In order to understand the temperature resolved reduction processes that convert Fe_2O_3 all the way to Fe, via Fe_3O_4 and FeO as the intermediates, an experiment in which the hydrogen content in the reducing gas (H_2/N_2) is varied, was performed.

The lower temperature “shoulder” / peak at 650°C did not vary much as the hydrogen content increased from 5% to 20%, but there was a clear impact on the higher temperature phase change shifting to higher temperatures. As the content of hydrogen was lowered from 20% to 5%, the high

temperature peak also shifted to higher values. This is consistent with the lowering of the partial pressure of the reducing atmosphere, which makes the reduction harder (leading to higher peak temperatures of reduction).

Further experiments were conducted to investigate the phase changes occurring at 650°C and at 850°C (isothermal reduction followed by XRD analysis) and identify any effect of moisture on the reduction process.

2.4.5 Third Round Experiments

The third set of experiments were conducted on BF Pellet samples to complete the TPRx investigation of the different iron ore feedstocks.

Reduction of BF Pellet: 10% H₂ 90% N₂

The BF Pellet (produced for direct feed to the blast furnace) is mostly Hematite and very similar to DR Pellet in composition. However, they have significantly lower porosity compared to DR Pellet. The TPRx behaviour (Fig. 20) was very similar to DR Pellet with a “shoulder” at 700-720 °C, and a sharper peak at about 850 °C. The peaks obtained were at higher temperatures compared to the DR Pellet, probably attributable to lower porosity.

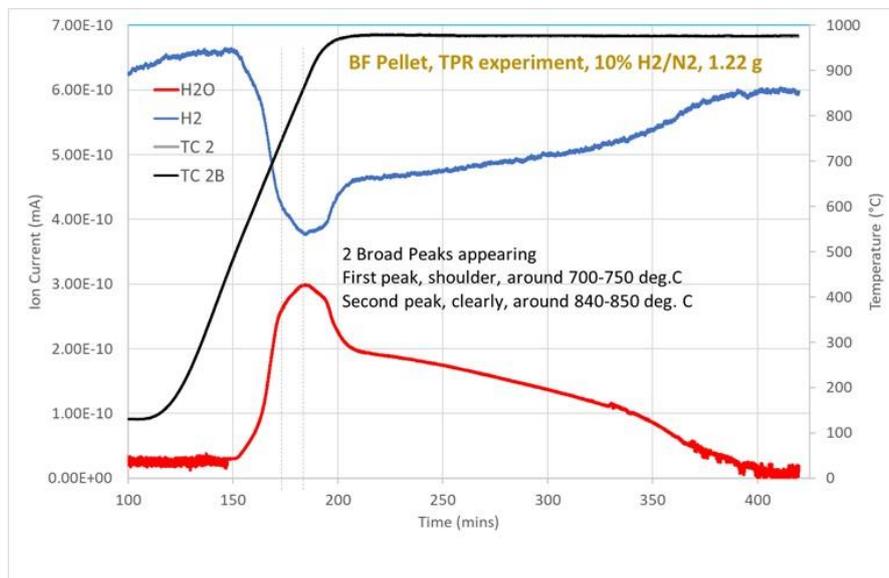


FIG. 20: TPRX PROFILE OF BF PELLETS REDUCTION

Independent porosity measurements were carried out on SEM micrographs using ImageJ software to map the images and evaluate porosity. The porosity of the as-received BF Pellet was about 11.5% and increased to 21.3% after reduction. Similarly, DR Pellet and Lump Ore particles were evaluated for porosity both as received and after full reduction for comparison. A summary of porosity measurements is shown in Table 5.

TABLE 5

POROSITY ANALYSIS

Sample	Porosity	
	As Received (%)	After Reduction (%)
Lump Ore	14.95	27.34
DR Pellet	21.30	34.40
BF Pellet	11.50	21.30

Isothermal Reduction of BF Pellet

The isothermal experiments were carried out on BF Pellet to observe the effect of adding moisture to the reduction gas feed and to determine if it had any influence on the extent of reduction (e.g., inhibition). XRD analyses of the as-received BF Pellet and the reduced samples after isothermal reduction were carried out at the Materials Processing Institute.

The as-received BF Pellet was found to be predominantly Hematite (94.3 wt.%), with a small proportion of Magnetite, Sillimanite and Quartz. After complete reduction in a TPRx experiment up to 970 °C, about 89.9 wt.% of the material was converted to Fe(0) with minimal amounts of Magnetite and Wüstite (<1.4 wt.%) remaining and the balance being Quartz.

As shown by the TPRx experiments (sections 2.4.3, 2.4.4 & 2.4.5), there was evidence of a first reduction peak around 650 °C and a later peak at approximately 800 °C. The BF Pellet samples after isothermal reduction at 650 °C revealed reduction to 42.1 wt.% Fe(0), 27.3 wt.% Magnetite and 27.1 wt.% Wüstite. The phase transformation from Magnetite to Wüstite was minimal, whereas significant Fe(0) was formed. In the presence of moisture, however, there was a strong inhibition of Fe(0) formation and the Magnetite remained extraordinarily stable. This indicates in all probability that Magnetite is stable at high partial pressures of water at lower temperatures.

After 4 hours of isothermal reduction at 850 °C (which corresponds to the end of the second peak in the TPRx experiment) XRD analysis reveals predominant conversion to 80.5 wt.% Fe(0), with only Quartz being the remaining significant phase and the levels of Magnetite and Wüstite having reduced considerably. At this temperature, moisture plays a major role in inhibiting reduction to Fe(0) (6.4 wt.%), with the reduction process stalling at Wüstite formation (90.1 wt.%) and proceeding no further.

It can be concluded that at lower temperatures, the reaction, which proceeds via the Magnetite pathway, is strongly inhibited by moisture at the Magnetite reduction stage. However, at higher temperatures, moisture impedes reduction beyond the Wüstite phase. Due to the thermodynamics, the presence of moisture strongly inhibits iron formation.

2.5 Time / Temperature Microwave FeOx Phase Curves

2.5.1 Microwave Assisted Processes – C Tech Innovation

The application of microwave (MW) energy has been shown to enhance chemical processes^(6–8). For example, closely related to the reduction of iron ore, the kinetics of the reduction of ilmenite (FeTiO_3) was found to be enhanced by application of MW and radio frequency (RF) radiation. Exposure of ilmenite to MW and RF energy gave a similar rate of reaction at 650 °C to an unassisted process at 950 °C^(9,10). This reduction in operating temperature of the furnace can offer savings in running costs.

The heating effect of MW energy on certain materials is well-known but can offer unique advantages over conventional processes. Indeed, MW fields are known to propagate through the volume of dielectric materials and offer non-contact fast heating, compared to radiative heating. Additionally, MW radiation can heat specific minerals such as magnetite more rapidly than gangue components. This process can induce stress cracking through a material and increase the surface area available for reaction^(11–14).

The application of MW radiation has been shown to induce additional phenomena in materials. For example, MW energy was found to aid the volume diffusion of oxygen in sapphire. The application of MW power was reported to reduce the apparent activation energy for bulk diffusion by 40% from 650 to 390 kJ mol⁻¹ and reduce the pre exponential factor by five orders of magnitude from 9.7×10^{-2} to 3.8×10^{-7} m²s⁻¹^(15,16).

The application of MW energy can potentially assist in the reduction of iron ore by offering enhanced kinetics and lower running costs for the industrial reduction of iron ore. This hypothesis underpinned the motivation for this body of work.

2.5.2 Objectives

The objective of this body of work is to inform on the suitability of MW fields in assisting the reduction of iron ore in a scaled-up furnace, as part of the collaborative H2DRI project.

C-Tech have reported on demonstration and quantification of any effect of MW radiation on the reduction of iron ore using the C-Tech Innovation Hybrid Kiln by mass loss measurements. This was conducted by examining any promotion of the reduction reaction rate using Direct Reduction (DR) Pellets. Reaction conversion after 5 min with and without MW assistance was studied to enable comparative analysis.

2.5.3 Equipment

A laboratory investigation of the direct reduction reaction of iron ore was carried out using the C-Tech Innovation Hybrid Kiln. The Hybrid Kiln is equipped with radiant heating, through electrically heated elements, and a 2.45 GHz, 2 kW MW generator (Fig. 21). The temperature of the furnace cavity is maintained by the radiative heating elements and controlled by a thermocouple located within the cavity away from the sample. The Hybrid Kiln has gas connections that allow experiments to be conducted under hydrogen or inert gases.

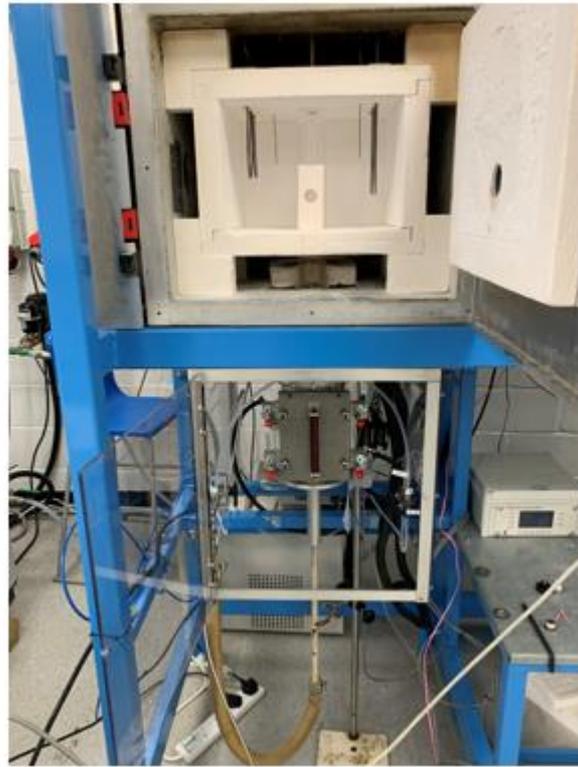


FIG. 21: PHOTOGRAPH OF C-TECH INNOVATION HYBRID KILN

The door of the Hybrid Kiln has a quartz window which allows for temperature monitoring using an externally mounted High-performance-Pyrometer Optris CSlaser 2MH (Fig. 22). The gas exhaust is connected to a Hiden Quantitative Gas Analyser mass spectrometer which allows for sampling of the reaction gas to provide additional insights into the reaction progress.

Characterisation and compositional analysis of compounds was carried out by collaborators at the Materials Processing Institute including X-ray diffraction (XRD) spectroscopy, X-ray fluorescence (XRF) spectroscopy, scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) spectroscopy.



FIG. 22: PHOTOGRAPH OF C-TECH INNOVATION HYBRID KILN DOOR WITH EXTERNALLY MOUNTED PYROMETER

2.5.4 Materials

Samples of DR Pellet used in this report were analysed by XRD spectroscopy (Table 6) with composition of reducible components used in mass loss calculations. Samples were crushed and sieved between 212-400 μm by collaborators at the Materials Processing Institute.

TABLE 6

COMPOSITION OF DR PELLETS SAMPLES BY XRD SPECTROSCOPY

Mineral	Molecular Formula	Composition in ore (weight %)
Hematite	Fe_2O_3	96.5
Magnetite	Fe_3O_4	1.4
Gangue components	N/A	2.1

2.5.5 Experimental Procedure

Experiments were performed according to the following procedure. A sample of iron ore (ca. 5 g) was loaded into a quartz holder. The quartz holder has a permeable frit that supports the sample and allows the supplied gas to flow through the sample. The sample and reactor chambers were purged with argon and the sample was raised into the furnace cavity at specified temperatures. Hydrogen was supplied to the sample at 3 L min⁻¹ for the defined reaction durations with concurrent application of MW energy at the specified power, where required. At the end of the reaction, the sample and reactor chambers were purged and the sample was lowered to cool under a flow of argon. The sample was removed and weighed immediately to avoid re-oxidation. Reaction conversions were calculated by mass loss upon net abstraction of oxygen (E1).

$$\text{Reaction conversion (\%)} = \frac{\text{measured mass loss (g)}}{\text{theoretical mass loss (g)}} \quad [E1]$$

2.5.6 Results and Discussion

Development and capabilities of the C-Tech Innovation Hybrid Kiln

C-Tech Innovation Ltd previously developed a technology on the enhanced pre-reduction of ilmenite ore through the application of electromagnetic fields to accelerate the kinetics of the reduction process. This furnace exploited a hybrid heating technology, where the conventional heating system such as radiant elements were used to provide the majority of the thermal input and control the temperature. A MW field was then applied to assist in processing of the materials by either providing volumetric heating or electric magnetic fields that may influence reaction kinetics.

Recognising the appeal and potential applications of hybrid heating technology, C-Tech Innovation has built and commissioned a fully instrumented laboratory Hybrid Kiln that utilises a combination of conventional heating and electromagnetic fields. This furnace is designed to operate as a flexible research tool to study the effect of dielectric fields on high temperature reactions. The following features allow the furnace to perform as a research and development system for this application:

- The radiative heating system is capable of operating at temperatures exceeding 1200 °C, with capacity for energy monitoring;
- The system possesses a fully programmable 2 kW microwave generator including electric field control and mode stirring;
- A gas control system can allow operation of reactions in atmospheres including 100% hydrogen or inert gas;
- The furnace is capable of data logging including temperatures, forward and reflected power phase monitoring of the induction coils performance;
- A flexible sample load capable of hosting up to 1 kilogram of material;
- A gas composition analysis system on the exhaust for monitoring inert, reaction or product gases.

2.5.7 Microwave Assisted Reduction of Ore Samples

Reduction of DR Pellet: Crushed and Sieved Material

Using the Hybrid Kiln, it was explored whether the hydrogen reduction of commercially relevant mined iron ore could be promoted by the application of MW energy. The DR pellet was a processed high hematite content sample (XRD analysis indicated a 96.5% hematite composition) and was selected for investigation. Notably, crushed and sieved DR Pellets exhibited enhanced initial conversion upon application of MW energy from 53% (1% standard deviation) to 74% (3% standard deviation) after 5 min (Fig. 23). This represented a promotion in reaction rate of approximately 50% upon exposure of MW energy.

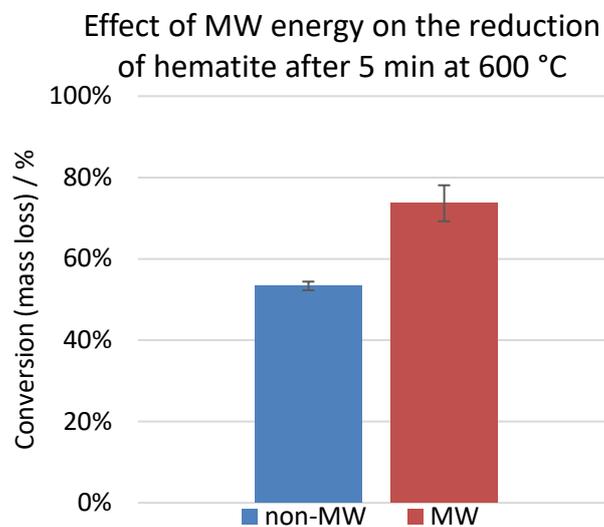


FIG. 23: CHART TO SHOW THE EFFECT OF MW RADIATION ON THE REACTION CONVERSION OF THE REDUCTION OF HEMATITE AFTER 5 MIN. ERROR BARS INDICATE $\pm\sigma$

2.5.8 Outlook and Further Work

This body of work has demonstrated that the reduction of iron ore at high temperatures can be conducted using the C-Tech Innovation Hybrid Kiln. The gas handling system permitted effective and safe manipulation of hydrogen as a reducing agent. The mass loss upon reduction proved to be suitable as a method of reaction monitoring. The attachment of a MW generator allowed for investigation of the effect of MW energy on reaction conversion, as per the objectives of this study.

This investigation found that MW radiation appeared to increase the initial reaction rate of the reduction of DR Pellets, where an increase in conversion was observed from 53% to 74% conversion after 5 min. In the context of the design of a scaled-up direct reduction furnace, it is apparent MW energy can promote the initial stages of iron ore reduction. This could offer significant energy savings and improvements to overall plant efficiency, and further investigations are recommended.

3. H2DRI MELTING FEASIBILITY

3.1 Objectives

The aim was to conduct a series of crucible melts in a kiln furnace and induction furnaces to determine the potential melting and product characteristics of reduced Lump Ore and DR Pellet. This was to demonstrate that hydrogen reduction of Lump Ore and DR Pellet gave H-DRI products suitable for further processing (melting and refining) using electrical heating (c.f., EAF processing).

Comparison was made with the melting behaviour of CO reduced material that would represent conventional DRI feedstock.

A chamber furnace and 2 induction furnaces used for the melting experiments. All 3 furnaces could be blanketed with argon to prevent re-oxidation on melting. The capacities of the furnaces are as follows:

- 45 kW Induction Furnace – 300 g scale.
- 16 kW Chamber Furnace (1600 °C max) – Multi 1 kg scale (up to 6 crucibles per run).
- 100 kW Induction Furnace – 6 kg scale.

In total, sixteen reduced DR Pellet and Lump Ore samples produced in the IOR (see Section 2.3) were received from SaMI, of which 11 were chosen for melt experiments. In addition, 10 kg of hydrogen reduced DR Pellet and 10 kg of hydrogen reduced Lump Ore were produced for the larger scale melt experiments.

3.2 Chamber & Induction Furnace Melts

Subsamples of each batch of DR Pellet and Lump Ore, reduced in hydrogen and CO, were prepared for melting.

The first two melts in the 45 kW induction furnace failed as the density of the DR Pellet (~1.5 g/cm³) was insufficient for the induction field to generate enough energy to melt the pellets and reach liquidus.

The third was conducted in an alumina crucible under an argon purge during heating and cooling. A graphite sleeve around the crucible was used as a susceptor to improve energy transfer in the 45 kW Induction Furnace. The maximum temperature achieved inside the crucible was 1589 °C, held for 10 minutes to achieve melting. The temperature profile and melt products are shown in Fig. 24. A slag layer formed on the surface of the metal and the two were easily separated to reveal a bright, relatively clean shiny surface to the metal.

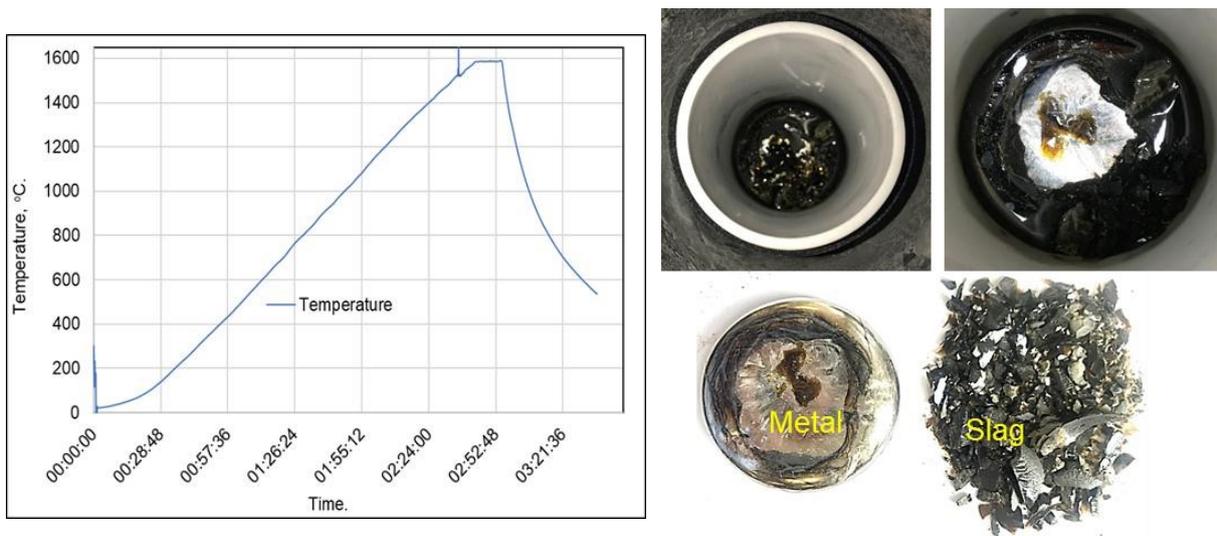


FIG. 24: TEMPERATURE PROFILE & PRODUCTS OF INDUCTION MELTING

Later samples melts were conducted in alumina crucibles heated to 1590 °C in the Chamber Furnace without any inert purging. Two 300 g samples were melted: CO reduced DR Pellet and CO reduced Lump Ore. Melt products are shown in Fig. 25.



FIG. 25: MELT 4 TEMPERATURE PROFILE & PRODUCTS

Slag and metal separation was good, but as can be seen in Fig. 64, the metal formed in air had a dull, pitted appearance, possibly indicating some surface oxidation on cooling.

A further induction melt was conducted on a mixture of CO reduced DR Pellet samples to demonstrate melting of a larger sample size and to increase the bulk density (to 1.63 g/cm³) for improved energy transfer. Liquidus was achieved, but the maximum temperature attained is uncertain due to a failed thermocouple.

Separation of slag and metal was not as clean as in the earlier melts, possibly due to the attempt to increase the quantity and bulk density (in the crucible) of the DR Pellet. The metal yield at 61.73% was the lowest of the melts conducted which was believed to be due to the long melting time required. A long melt time was experienced because of the poor transfer of energy to the sample by virtue of the relatively low bulk density of the DR Pellet.

DR Pellet and Lump Ore subsamples prepared from the bulk samples reduced in the IOR at SaMI were melted in the chamber furnace. The sample of hydrogen reduced DR Pellet gave good slag / metal separation and the alumina crucible showed no sign of attack by the slag. Melted samples of hydrogen reduced Lump Ore showed evidence of slag attack of the alumina crucibles, but with good separation of slag and metal. Melted samples of CO and hydrogen reduced DR Pellet, respectively, demonstrated different slag formation behaviour related to the reductant used. The CO reduced DR Pellet displayed foaming / splashing of the slag that was not seen in the hydrogen reduced DR Pellet melt and the slag was attached more strongly and was more difficult to separate for the CO reduced sample (Fig. 26).



300 g DR Pellet reduced by CO exhibited splashing
DR Pellet reduced by H₂ was "clean"

Slag of DR Pellet reduced by CO (left) attached to the surface of the iron more strongly than that reduced by H₂ (right)

FIG. 26: COMPARISON OF CO AND HYDROGEN REDUCED SAMPLES MELTED IN THE CHAMBER FURNACE

A larger melt was carried out in the 100 kW Induction Furnace on a 6 kg subsample of the hydrogen reduced Lump Ore produced in the IOR at SaMI. As earlier attempts to melt in both Induction Furnaces had been unsuccessful, a novel technique was adopted. The metal previously melted was added to the hydrogen reduced Lump Ore charge in the furnace crucible to function as a susceptor core to promote energy absorption (Fig. 27).



FIG. 27: MELT 9; 100 KW INDUCTION FURNACE, 6 KG LUMP ORE

Hand-held X-ray Fluorescence (HH XRF) and OES analyses of the metal from Melt 8a and Melt 9 showing almost pure iron.

TABLE 7

HAND-HELD XRF ANALYSES OF SELECTED MELT SAMPLES

	HH XRF ID No.	Al (mass %)	Si (mass %)	P (mass %)	Fe (mass %)
Melt 9 6 kg trial piece	9			0.158	99.84
	10			0.170	99.83
Melt 8a 1kg trial piece	11	0.088	0.291		99.62
	12	0.064	0.116		99.82

Table 8 gives the corresponding Optical Emission Spectroscopy (OES) analyses of the melt samples in Table 7. The electrical spark activation used for OES identifies elements in the first few microns of the surface of the sample. The concentrations of these were calculated by reference to metal standards using the OES programme Fe-10-M for low/medium alloy steel.

TABLE 8
OES ANALYSES OF SELECTED MELT SAMPLES

Sample ID	C (%)	Si (%)	Mn (%)	P (%)	S (%)	Cr (%)	Al (%)
Melt 8a DR Pellet (H ₂)	0.002	0.006	0.002	0.007	0.0005	0.003	0.004
Melt 9 Lump Ore (H ₂)	0.002	0.006	0.003	0.156	0.0008	0.003	0.004

Sample ID	Ti (%)	Fe (%)
Melt 8a DR Pellet (H ₂)	0.0005	99.9
Melt 9 Lump Ore (H ₂)	0.0005	99.8

The OES analyses show that in all experiments, whether in air or under argon, or whether the reducing gas was hydrogen or CO, the metal produced was almost pure iron with only traces of elements arising from the original ore or the slag on melting. Melting in the Chamber Furnace or the Induction Furnace produced identical results, even if the surface of the samples melted in air were dull and appeared to be heavily oxidised. The hand-held XRF and the OES analyses of DR pellet and lump one melts showed close agreement, indicating that surface oxidation was minimal, if at all.

3.3 Theoretical Energy Requirement for DRI Reduction & Melting

The theoretical energy requirements to produce liquid iron from hydrogen and CO reduced Lump Ore and DR Pellet were evaluated from the data of the Induction Furnace experimental melts.

The influence of the reduction degree (RD) of the DRI on the final reduction and melting in the Induction Furnace is illustrated in Fig. 28, showing the theoretical energy required to reduce and melt Lump Ore and DR Pellet with RD values of 95%, over the temperature range 20 °C to 1550 °C.

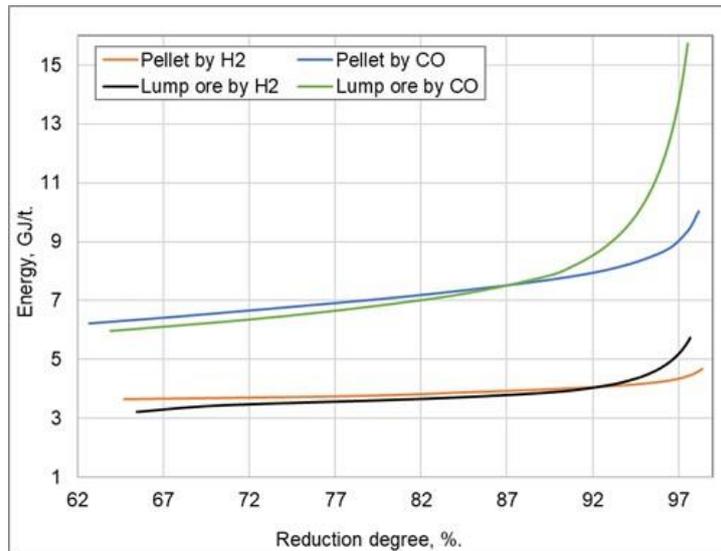


FIG. 28: THEORETICAL ENERGY REQUIREMENT FOR REDUCTION & MELTING

Comparing the theoretical energy required for final reduction and melting of the H-DRI and CO-DRI (Lump Ore and DR Pellet) with that required for melting alone in the Induction Furnace (Table 9), it is apparent that the reduction phase requires much more energy than the melting phase.

TABLE 9

TOTAL THEORETICAL ENERGY REQUIREMENTS FOR REDUCTION & MELTING

	Lump Ore (kWh/kg)	Lump Ore (GJ/t)	DR Pellet (kWh/kg)	DR Pellet (GJ/t)
H-DRI Reduction + Melting	1.237	4.454	1.163	4.188
CO-DRI Reduction + Melting	2.871	10.336	2.331	8.391
Melting Reduced DRI*	0.373	1.341	0.368	1.325

Note: * = heating fully reduced H-DRI or CO-DRI from 20-1550 °C alone

The theoretical energy required for final reduction and melting CO reduced DR Pellet and Lump Ore was illustrated by the temperature and power data from two experiments conducted in the 45 kW Induction Furnace.

Several process factors were apparent:

- The higher the RD of the DRI, the more energy required for final reduction and melting.
- H-DRI requires much less energy than CO-DRI for the same RD.
- Melting H-DRI or CO-DRI (heating from 20 to 1550 °C) uses half the energy of the reduction stage to produce liquid iron.

- At high RD (>93%), DR Pellet reduction uses less energy than Lump Ore reduction.

4. PROCESS FEASIBILITY FOR PHASE 2

The UK Hydrogen DRI Interest Group steered the Phase 2 proposal clearly in the direction of a vertical shaft furnace as the core reduction element, in order to replicate best conditions in the dominant technology (vertical natural gas DRI) which is transitioning to increasing hydrogen input.

A study of the state of the art makes clear, the ‘best’ technical solution for hydrogen ironmaking and converting the product economically to steel is not yet fully established and may differ depending on ore source, siting of the technology and end use.

4.1 Potential to Use a Microwave Rotary Reduction Furnace

The procurement of a rotary microwave furnace reduction furnace capable of reducing iron ore in batches of up to 50kg was funded by the BEIS Programme of Research and Innovation for the UK Steel and Metals sector (PRISM) grant to the Materials Processing Institute.

Although it wasn’t possible to complete the commissioning of this furnace during this project the principles of operation were established, the mechanical operation was confirmed and the hazard and risk mitigation requirements for operation with hydrogen was worked through.

Figure 29 shows the rotary kiln furnace in position at the Materials Processing Institute at the start of the installation and commissioning programme. The microwave generator and wave guide can be seen on the material feed input section on the right of the photograph. The reaction zone makes up the centre section of the furnace, where electrical heating for the process is provided externally, and product material is discharged from the output section on the left.



FIG. 29: ROTARY KILN FURNACE DURING INSTALLATION

Operability will be reported on once commissioning is completed under the PRISM project.

4.2 End Product Handling & Storage Assessment

Experiments conducted by SaMI to investigate the potential re-oxidation of H-DRI products reported negligible uptake of oxygen and limited surface oxidation. The extent of re-oxidation was noted to be insufficient to cause a temperature rise in the material that would lead to self-ignition.

DRI produced using hydrogen as the reductant produces appears to be a more stable material than when CO or a hydrocarbon gas is the reductant. Few issues with storage of H-DRI material are envisaged. This has been corroborated by the experience of the HyBRIT project in Sweden which has confirmed the routine handling, storage and transport of untreated uncompacted hydrogen DRI..

In terms of handleability, SaMI reported that as metallisation increases, the strength of the material also improves, with hydrogen reduction giving better results than CO reduction.

4.3 Energy & H₂ Demand Profile Assessment

Hydrogen consumption and energy demand have been confirmed as tracking each other closely in the iron reduction reaction, though there are variations depending on the composition of the iron ore. Since the TPR experiments at TU have been run with variable temperatures and the IOR reductions at SaMI operated isothermally, the combination and agreement of the two approaches with each other and with the energy balance measured at C-Tech Innovation has been satisfying and confirms the feasibility of a combined methodology for rapidly producing reaction equations for specific ore types and predicting the specific energy demand for a certain reaction profile.

The laboratory scale experimental programmes have gone some way to measure the hydrogen demand profile of the reduction process and give confidence in applying this to the scaled-up design concept. Further work at the pilot-scale will be required to ensure that the assessments are still accurate at larger scale, and to match them to the characteristics of different feedstocks.

4.4 Comparison Rotary Kiln Furnace v Vertical Shaft Furnace

The opinions of industry leaders were sought on the most likely furnace designs that would attract investment for developing hydrogen reduction facilities at integrated steelworks and raw material suppliers. Overwhelmingly, the tried and trusted vertical shaft furnace technologies were the favoured option as the established natural gas DRI furnaces have demonstrable capability for conversion to hydrogen as the reductant. The first generation of DRI expansion in Europe anticipates natural gas DR shaft furnaces in the initial years which transition to green hydrogen as supply increases.

The partners decided to meet the challenge of adapting a vertical shaft furnace design to allow the microwave assisted process to be studied. The concept for the pilot scale electrically heated microwave-assisted vertical shaft furnace at has been considered and the key design parameters developed based on lab scale studies of the kinetics . .

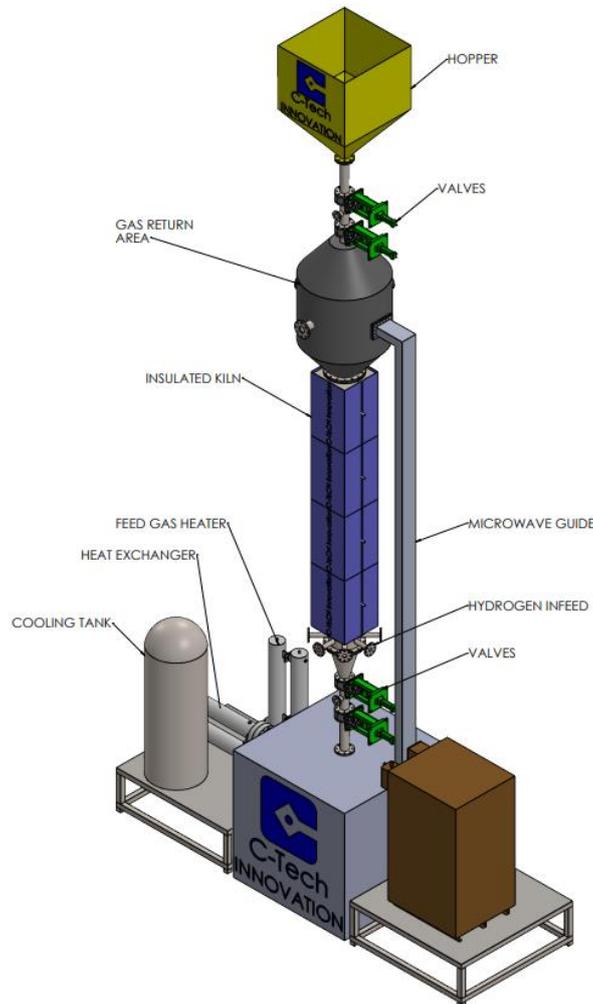


FIG. 30: CONCEPT FOR MICROWAVE-ASSISTED VERTICAL SHAFT REDUCTION FURNACE

4.5 Scale Up Pre-FEED Decision

Since the value of 'green' (hydrogen-electric) iron is only realised once it is made into marketable steel, the steelmakers and suppliers who are the core target group for Phase 2 must have confidence in the performance and value in use of the iron product during steelmaking and in the quality of the steel grades that can be subsequently produced. An accessible demonstration R&D furnace needs to be comparable in throughput to the 100-1000 kg per hour pilot units currently in private operation or under commission, such that pilot melts, casts and rolling or drawing of the steel end product can proceed to at least standard ingot or mini slab size (4-6 tonne).

The design concept was developed with the initial specification of a vertical shaft furnace capable of 250kg per hour DRI output. Preliminary CFD modelling by the Materials Processing Institute and anecdotal information regarding other pilot designs confirmed that a 400 m ID shaft would permit behaviour to approximate that in a larger commercial furnace (Fig. 30).

Having previously developed the microwave-assisted rotary kiln furnace, with external electrical and microwave heating, C-Tech Innovation applied the same approach to the development of this vertical

shaft system giving a potentially quicker and more efficient process by allowing the endothermic reactions to continue quickly without the need for excessive hydrogen preheating as the only heat carrier. The innovative use of microwave assistance is of interest to a lot of steelmakers. This is in contrast to shaft or fluidised bed furnaces, with process heat from hot reducing gas are the de facto standard technology in most steelmakers' opinion.

The current approach is to develop the process understanding in the pilot scale microwave-assisted rotary kiln furnace prior to scale up to larger system throughputs. There is a pressing need to evaluate the furnace performance against that of a shaft furnace design, to validate the economic advantages and demonstrate industrial viability.

4.6 Scaling & Integration – Upstream & Downstream Processes

Aspects of scaling of a H-DRI unit for upstream and downstream processes were discussed with the UK Hydrogen Reduction Interest Group in the context of integration of a unit into the Materials Processing Institute's electric steelmaking facility. The outcome of these discussions is to be used for the Phase 2 bid to build and operate a new H-DRI unit.

4.7 Hydrogen Demand & Supply Strategy

Ancillary services and a hydrogen supply network were installed on site at the Materials Processing Institute under the PRISM project to support installation of the Rotary Kiln Reduction Furnace and accompanying ancillary facilities. Operation of this network will guide the hydrogen supply and demand strategy going forward.

5 HYPOTHESES & CONCLUSIONS

The project has informed the operating conditions and scale up requirements required for the design, build and operation of a hydrogen direct reduction furnace for iron ore at tonne per day scale.

The work has shown that reduction with hydrogen is not only possible but that it is relatively fast, requires less energy than conventional reduction and produces iron of very high purity.

The collaboration and combined capabilities of the consortium partners has demonstrated the following, which is taken forward to a tonnes per day ore-to-steel scale-up concept under the working title of 'HEIDI' (Hydrogen-Electric Iron & steelmaking Demonstration and Innovation):

- Isothermal experiments showed hydrogen is able to reduce iron ores at significantly higher rates and achieve more complete reduction than was found possible using carbon monoxide.
- Hydrogen-based reduction is possible at lower temperatures than originally envisaged.
- It should be possible to develop a standard test similar to ISO 11258 to characterise the reduction of iron ore with hydrogen.

- Both isothermal and Temperature Programmed Reduction (TPR) experiments showed reduction rate changes that might be correlated to the phase changes. The experiments showed comparable results at different scales indicating that results may be scaled.
- Carburization of iron following full reduction has been demonstrated.
- Microwave assistance gave an increase in reaction rate of approximately 50% after 5 minutes to the reduction of DR Pellets, suggesting significant rate enhancements and running cost savings may be offered by application of dielectric fields.
- Melting of hydrogen reduced iron (H-DRI) was demonstrated in both radiant heated and induction furnaces.
- High purity iron, with good slag separation was produced from melted H-DRI.
- H-DRI required less energy than CO-DRI for the same reduction degree.
- Melting H-DRI used half the energy of the reduction stage to produce liquid iron.

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APPENDIX

Project Launch Press Release

Publicity was generated via press releases and widely replicated online including:

<https://www.iom3.org/resource/materials-processing-institute-to-lead-on-national-green-steel-hydrogen-pilot-project.html>

<https://www.pesmedia.com/materials-processing-institute-leads-on-national-green-steel-hydrogen-pilot-project>

<https://www.insidermedia.com/news/national/materials-processing-institute-leads-on-green-steel-hydrogen-pilot>

<https://www.businessupnorth.co.uk/materials-processing-institute-leads-on-national-green-steel-hydrogen-pilot-project/>

<https://neconnected.co.uk/materials-processing-institute-leads-on-national-green-steel-hydrogen-pilot-project/>

Conference / Technical Committee Presentations

Teesside University and the Materials Processing Institute representatives co-presented at the **H2 Supergen conference in St Andrews on 8th June**:

“High Temperature Electrolysis (HTE) - towards generation of hydrogen for Direct Reduction of Iron Ore (H2-DRI) for decarbonizing the steel industry “

WorldSteel Technology Committee TECO 54 – H2 13-14th July: H2DRI project was also highlighted in a presentation to the global steel collaboration forum in a presentation titled “Enabling practical decarbonisation pilots and site modification”.

The achievements of the project in producing the UK’s first hydrogen steel samples were also cited by Lord Redesdale (in the context of innovation investment needing to increase) during addresses to the first **UK Steel Expo at the NEC in Birmingham on 14th September** to which the Materials Processing Institute was a contributor and exhibitor.

Finally, project partners from the Materials Processing Institute and Rio Tinto were present at the second global **Hydrogen Iron and Steelmaking (HyIS) forum in Sweden on 12-14th October** and able to discuss the UK’s start-up H2DRI programme with technical and commercial leaders from across the supply chain, including the market leading OEMs for DRI technology and global leaders in the field from Sweden and South Korea. This has generated considerable interest in future access to an independent R&D facility as proposed in Phase 2 and confirmed the strategic value of this in accelerating the decarbonisation of primary steel. The forum also served to demonstrate the magnitude of the challenge and the critical mass now building to transform global steelmaking for a fossil-free future. Conversations with the senior researchers of the HyBRIT programme confirmed the interest

and value in future co-operation since the H2DRI project had independently demonstrated several very similar results as well as some approaches they had not yet investigated.

Industry Journal Article

Materials World May 2022 'The Energy Transition issue':



This article also raised interest in the project and in future collaboration with UK and European actors.



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