Chapter 6: Specialist imaging techniques

6.1 Scanning electron microscopy

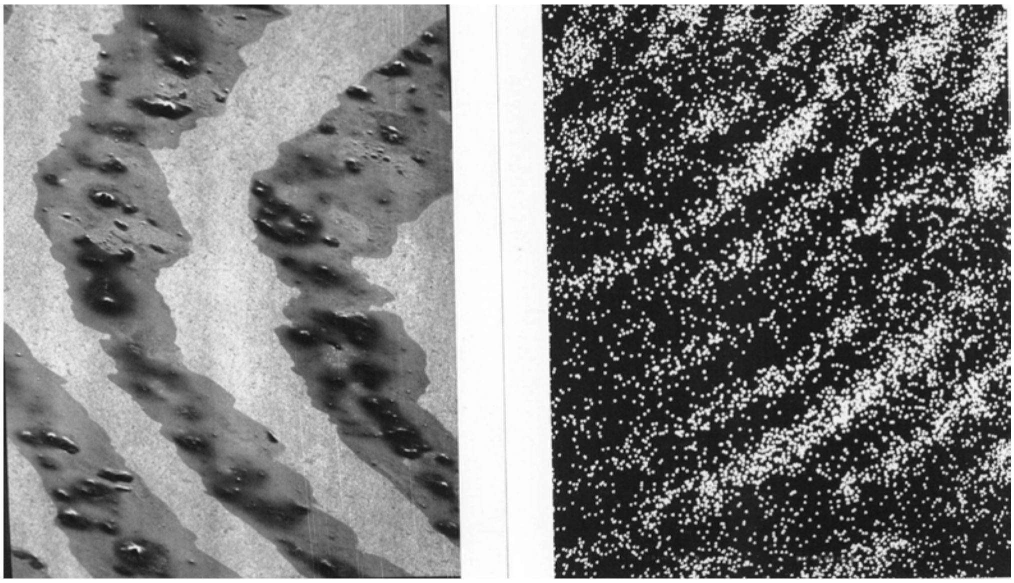
1. History

1.1 As early as the 1920s it was recognised that beams of electrons could be focused by means of electrostatic or magnetic fields and that the short wavelength of electron beams offered significant improvements in both resolution and depth of field compared with light microscopy [1].

1.2 It was not until the 1950s and 1960s that practical electron microscopes began to emerge, utilising both transmission and scanning modes to provide images of materials. The potential applications of electron microscopy (in particular the scanning electron microscope) in forensic science were first explored in the late 1960s. Van Essen [2] reported the use of scanning electron microscopy in combination with energy dispersive x-ray spectroscopy for the analysis of paint and metal fragments, ink composition and for studying hair and fibres.

1.3 It was also recognised that scanning electron microscopy could be used for imaging of fingerprints, and Garner *et al.* [3] demonstrated that latent fingerprints could be detected on both glass and metal substrates. On the non-conductive, glass surface a gold coating was required to prevent charging and it was also observed that older marks were more difficult to image than freshly deposited marks.

1.4 The Police Scientific Development Branch (PSDB) began studies into the use of electron microscopy in the late 1970s [4,5], and installed a JEOL scanning electron microscope with an energy dispersive x-ray spectrometer specifically to explore imaging of fingerprints. Various imaging modes were investigated [4] including specimen current imaging of latent marks and mapping of silver distribution in marks developed using physical developer. The microscope was also used as a research tool to study the secondary electron escape depth from fingerprints [5]. Both latent and treated fingerprints were used in these studies, the differences in elemental deposition occurring using vacuum metal deposition being utilised to reveal fingerprint ridges crossing print boundaries.



a) b)

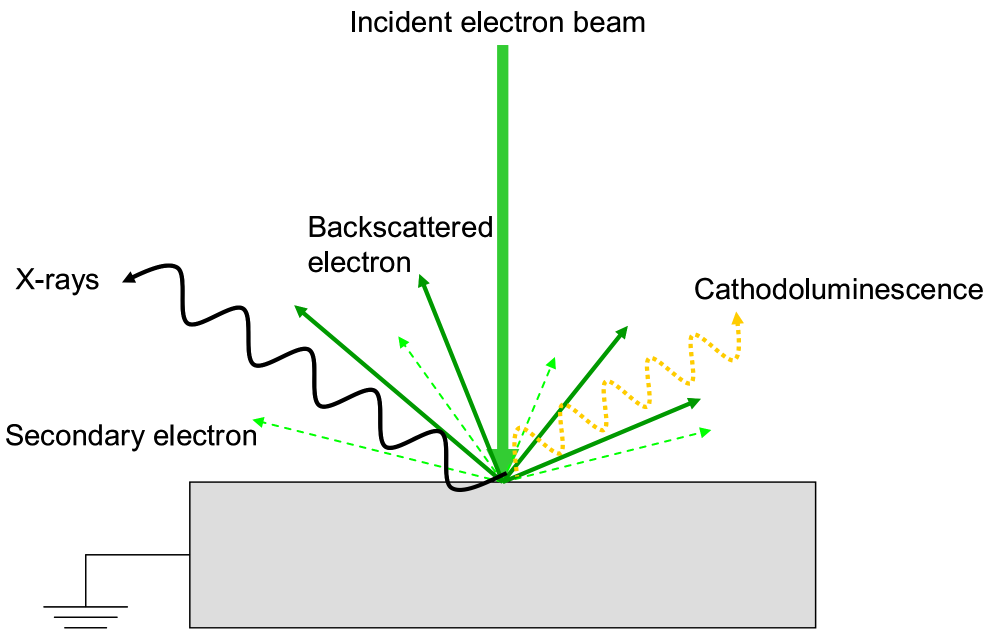
Early scanning electron microscopy images of fingerprints a) latent fingerprint in secondary electron imaging mode and b) mark developed using vacuum metal deposition in elemental mapping mode

1.5 Although potentially effective for distinguishing fingerprint ridges against obscuring backgrounds, scanning electron microscopy and its associated analytical modes have been more useful in providing information about the mechanisms of other development processes and the composition of powders and reaction products. Scanning electron microscopy has been extensively used by PSDB in the characterisation of fingerprint powders and brushes [6-9] and has also provided useful images of the reaction products from the superglue and physical developer processes.

1.6 In practical terms, scanning electron microscopy is little used for casework because its application often requires a small area to be cut from the exhibit and coated with a conductive material to prevent the sample charging. However, there are situations where it may provide additional information and it remains an invaluable tool for understanding the interactions between fingerprints and the surfaces they are deposited onto.

2. Theory

2.1 When an energetic beam of electrons is focused onto a surface there are a number of interactions that can occur [1]. These include transmission and diffraction, which are of most interest for transmission electron microscopy and are therefore not discussed further here. The interactions of principal interest for scanning electron microscopy are illustrated schematically below.



Principal interactions between electron beam and sample in scanning electron microscopy.

2.2 Discussing each mechanism in turn, backscattered electrons occur where the incident electron undergoes a series of inelastic collisions with atoms in the sample and are scattered backwards out of the surface and towards the detector. These electrons are relatively high energy and the number of them occurring will be related to the atomic density of the surface being examined.

2.3 Secondary electrons occur during the inelastic collisions between the primary electrons and the atoms and some have sufficient energy to escape the surface towards the detector. They are of lower energy than backscattered electrons.

2.4 X-rays are also emitted, in a process analogous to fluorescence in the visible region of the spectrum. Electrons promoted into excited states by the interaction with the electron beam decay into their ground states, with the emission of an x-ray of energy/wavelength characteristic of the element present.

2.5 For certain materials, the emission of energy as the electrons decay back into their ground state occurs at an energy/wavelength corresponding to the visible region of the spectrum. This is known as cathodoluminescence.

2.6 Of all these mechanisms, secondary electron imaging is most useful for examining the morphology of powders, brushes and reaction products. In secondary imaging, a positive charge is applied to the detector, which attracts most of the negatively charged electrons emitted from the surface. As a result, the signal received at the detector is relatively high and the image is not 'noisy'. The electron beam is scanned across the surface in a series of lines known as a raster, and the signal level recorded at each pixel represented on a screen.

2.7 Backscattered electron imaging is most useful where the elemental composition of the fingerprint ridge and the background differs, especially if one contains an element of a significantly higher atomic number. Because the number of backscattered electrons is a function of atomic density, areas of high atomic density will produce more backscattered electrons and appear brighter. Backscattered electron imaging can be carried out by biasing the detector with a slight positive charge, thus repelling the low energy secondary electrons and only allowing the higher energy backscattered electrons to reach the detector. Because fewer electrons reach the detector, backscattered images may be more noisy, but may be capable of resolving fingerprints developed using techniques such as vacuum metal deposition and iodine.

2.8 X-ray spectroscopy can be carried out in a static mode, to determine the elemental composition of a particular location on the sample. X-rays can be separated and analysed according to their characteristic wavelength or energy. In practice the energy dispersive detectors are more compact (although not as suitable for quantitative analysis) and are more commonly fitted to electron microscopes. Energy dispersive x-ray spectroscopy can also be used in mapping mode, scanning the beam across the surface and recording the types of x-rays emitted at each point. If a characteristic element is present in the fingerprint ridges, it is possible to resolve the ridges from the background in this way.

3. Reasons technique is not recommended by CAST

3.1 CAST does not recommend the process for routine operational work because it will normally be destructive to the exhibit, involving cutting an area small enough to fit inside the chamber of a scanning electron microscope and coating with a conductive element to prevent charging. These processes may be detrimental to subsequent analysis for other types of forensic evidence. However, recent advances in electron microscope design may mean that larger samples can be examined and a conductive coating may not always be required. In some circumstances scanning electron microscopy and associated analytical techniques may be capable of providing additional information about a fingerprint and its use should not be discounted. Suitable microscopes can be found in most universities.

3.2 Scanning electron microscopy is a useful research tool for investigating fingerprint development techniques and has primarily been used for this purpose in recent years, in some cases augmented by transmission electron microscopy and atomic force microscopy for cases where very high magnifications are required.

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6.2 X-ray imaging

1. History

1.1 The properties of the x-ray were first observed by Wilhelm Roentgen in 1895, during experiments into the effect of passing electricity through bottles containing gas. Roentgen observed that rays emitted from the bottles had the ability to take pictures of objects hidden under or within other objects, and took a picture of his wife's hand that revealed her bones and her wedding ring.

1.2 Although rapidly adopted for medical applications such as the imaging of the interior bones, x-rays were not seriously considered for forensic imaging until the mid-1960s, when Graham and Gray at the Victoria Infirmary, Glasgow, began experimenting with the technique of electronography [1,2], initially with the intention of revealing the watermark of stamps attached to documents. In the electronography technique a metal irradiated with a high energy, monochromatic x-ray beam emits its own characteristic x-rays, which cause a photographic film in intimate contact with the sample to darken. This is outlined in more detail in the 'Theory' section below.

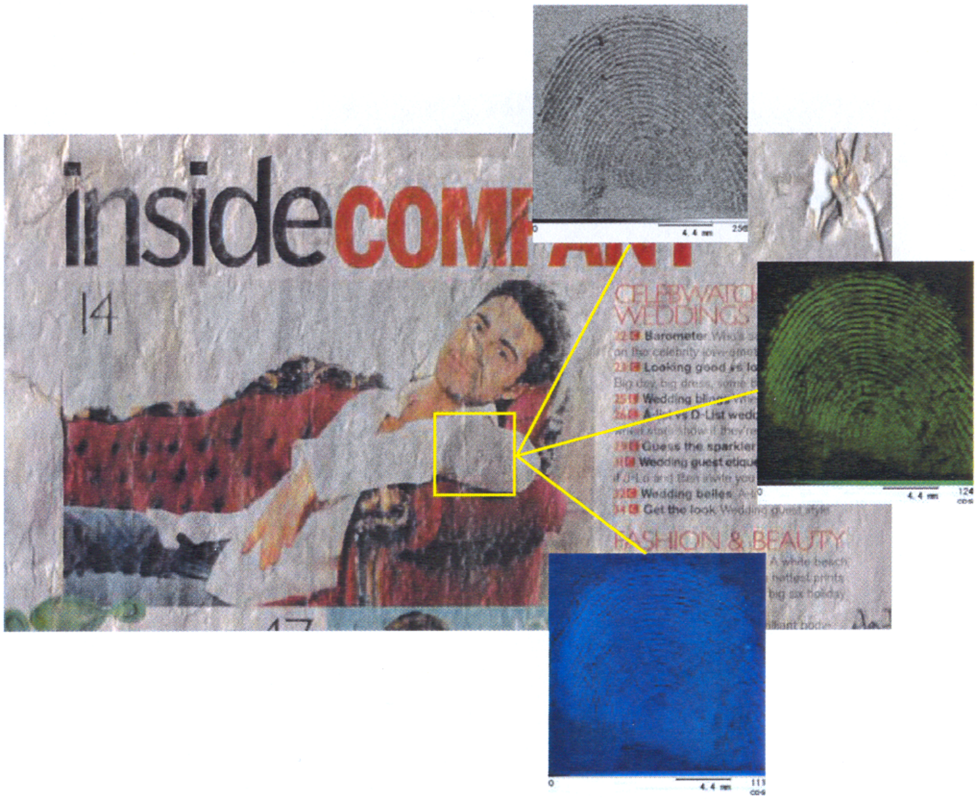
1.3 Graham [2,3] next considered using powdered lead to reveal indented writing, carrying out electronography to enhance the indentations the lead had preferentially settled into. Graham and Gray considered that fingerprints could be developed in a similar way [4], powders already being extensively used for fingerprint development. Magnetic powders with the Magna-brush were considered, but the emission from iron was not found as effective as that from lead and subsequent studies utilised lead powdering in combination with electronography. The first application proposed for electronography was the revelation of fingerprints deposited on patterned backgrounds. Once the fingerprint had been developed using the lead powder, only the developed areas emitted during subsequent electronography and the resultant fingerprint image was free of background. Test fingerprints were resolved on magazine covers and postage stamps.

1.4 Electronography was also proposed to image fingerprints on dead human skin, again using lead powdering to develop the mark and electronography to enhance the image and remove the background of skin texture, hairs, etc. [5]. There was reasonable interest in the technique for this purpose, with no satisfactory development technique being available at that time. The Police Scientific Development Branch (PSDB) placed a contract with Graham in the early 1970s to investigate the development of fingerprints on limbs using lead powder and electronography. The technique was adopted in some laboratories in the USA [6,7], and refinements were proposed to make the technique easier to apply both in the laboratory and in the field [6]. Later adaptations were proposed within the UK [8], and the use of lead powder with electronography was proposed as an alternative to vacuum metal deposition (VMD) for developing marks on polythene [9]. VMD was found to be far more effective than lead powdering for this purpose, and after the late 1970s the technique seems to have gradually faded from use.

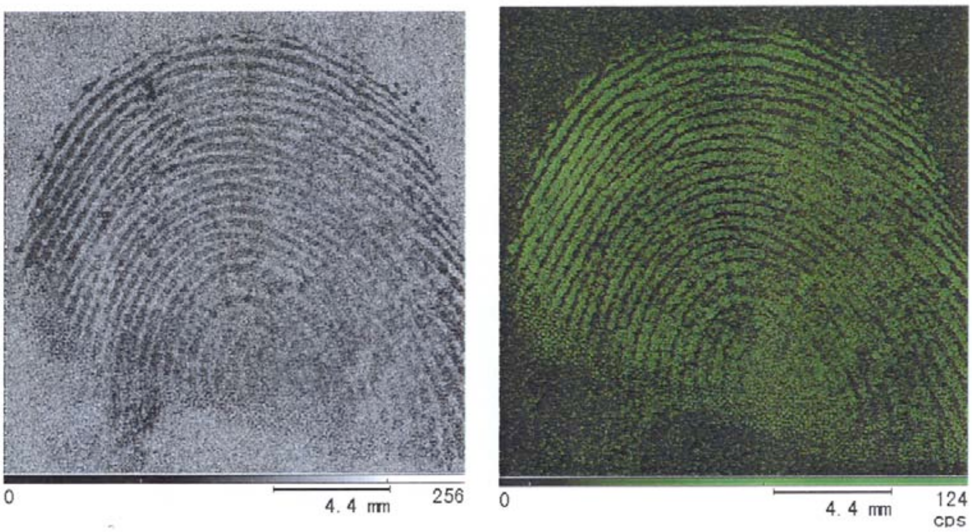
1.5 X-rays can also be used to image fingerprints in other ways. X-rays are also emitted from samples bombarded by electron beams in electron microscopes, and the characteristic x-rays thus emitted can be used to build elemental maps of a surface. This is described in greater detail in Chapter 6.1, Scanning electron microscopy.

1.6 Another way in which x-rays can be emitted is by x-ray fluorescence, irradiating a sample with monochromatic x-rays and causing characteristic x-rays to be emitted in a process directly analogous to fluorescence in the visible region of the spectrum. More recently, researchers have used an x-ray fluorescence instrument to scan surfaces and detect fingerprints by mapping characteristic elements within latent fingerprints and within contaminants that may be present on fingers such as sun cream [10]. Potential advantages of x-ray fluorescence over x-ray mapping within a scanning electron microscope are that larger areas can be examined, the sample does not have to be under a vacuum and the sample does not have to be coated with a conductive coating to prevent charging.

1.7 The Home Office Scientific Development Branch (HOSDB) has also carried out some initial studies into the x-ray fluorescence technique, in this case looking at fingerprints developed using techniques that result in characteristic elements being present in fingerprints ridges, such as physical developer, vacuum metal deposition and metal toning of ninhydrin [11]. It was shown that the technique had potential for revealing fingerprints on patterned backgrounds, such as magazines, and also on fabrics. The instrument used in these studies also had a transmitted x-ray mode and for fingerprints containing heavy elements, such as iodine, this was also found to be effective for distinguishing ridges from the background.

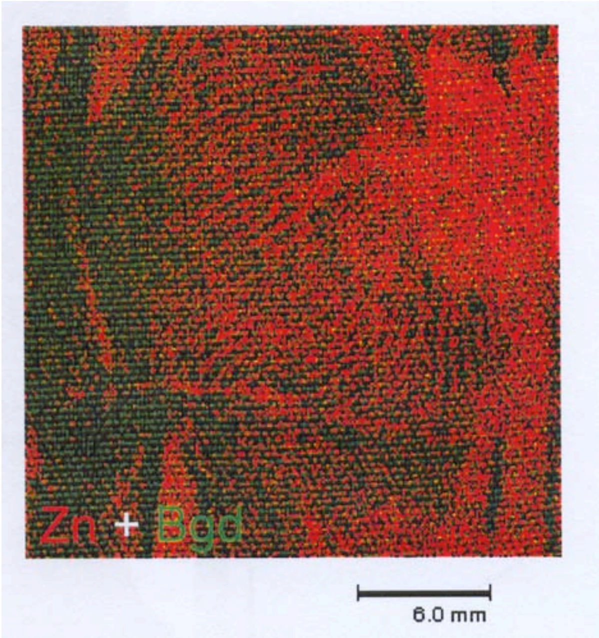


Overview of fingerprint treated with physical developer on magazine page, subsequently toned with potassium iodide.



a) b)

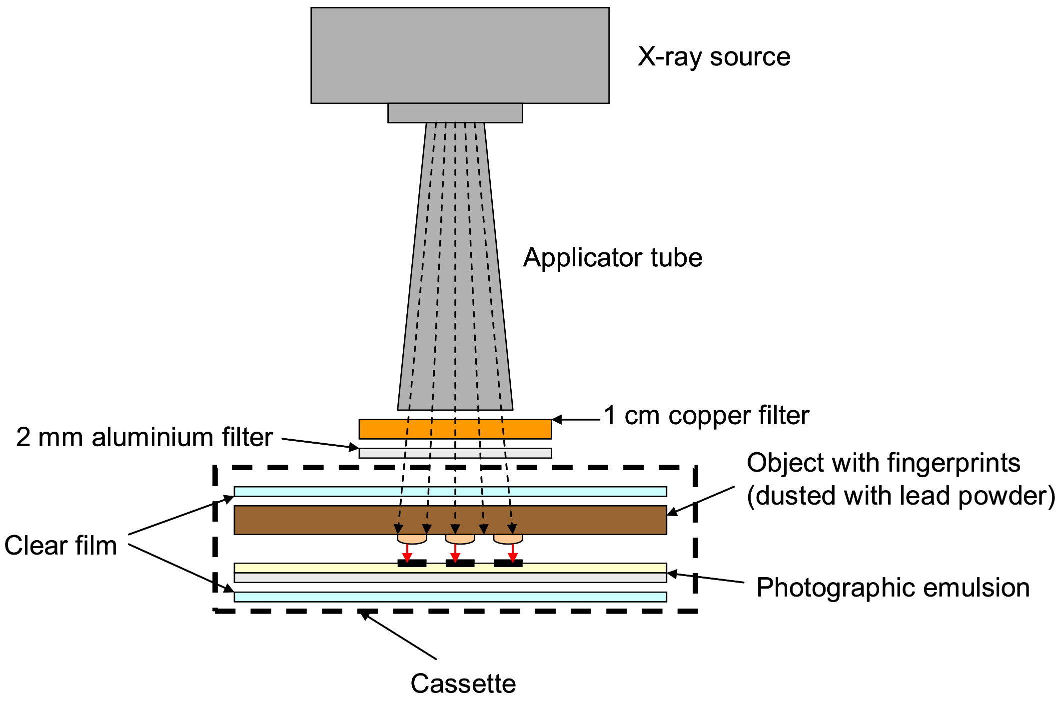
Closer view of x-ray images a) image of mark in x-ray transmission mode and b) image formed from characteristic x-rays from iodine.



X-ray image from mark developed on fabric using vacuum metal deposition, red signal = zinc from metal deposition, green signal = fabric background.

2. Theory

2.1 The practical apparatus used by Graham and Gray is illustrated schematically below, and the theory of electronography outlined subsequently.

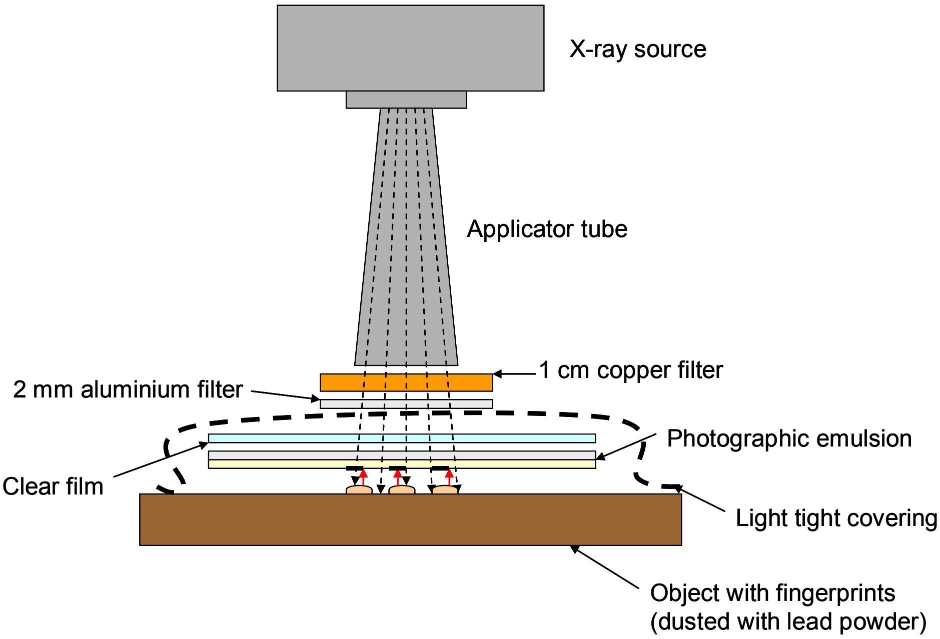


Electronography apparatus proposed for thin exhibits such as documents.

2.2 All metallic elements, when irradiated by a high kilovoltage beam, emit both electrons and x-rays characteristic to that element. These characteristic x-rays and electrons cause the silver halides of a photographic film emulsion to convert to silver, leaving a black image of the areas containing the characteristic metal element.

2.3 For this to be effective, the original, incident x-rays must have a negligible effect on the photographic emulsion and it is therefore necessary to filter the original broad spectrum of wavelengths emitted by the x-ray source. The longer wavelength x-rays that cause film fogging are filtered out by passing the beam through a 1cm block of copper. The characteristic copper x-rays emitted as the primary beam passes through the copper filter are in turn removed by a further 2mm aluminium filter, and the x-rays emitted from the aluminium filtered out by a clear plastic film. The short-wave x-rays pass through the object under examination and hit the lead particles adhering to the fingerprint ridges, promoting emission of x-rays and electrons that develop an image of the fingerprint on the photographic film in intimate contact with the surface. A further clear film is used below the photographic film to absorb scatter and emission from other areas within the cassette.

2.4 For articles that were not flat or could not be fitted inside a cassette, an adaptation of the method was proposed.



Electronography apparatus proposed for solid exhibits such as bodies.

2.5 In this adaptation, x-rays are allowed to pass through the photographic film and fall upon the surface being examined. The x-rays from the surface are emitted backwards onto the film and the clear film, film and surface are enclosed within a light-tight covering.

2.6 The theory of x-ray fluorescence is exactly analogous to fluorescence in the visible region of the spectrum. A short wavelength beam of x-rays is used to irradiate a surface, promoting electrons into excited states. As these electrons decay back to ground states, they emit x-rays at longer wavelengths with an energy characteristic to the particular elements present in the surface. By scanning the x-ray probe across the surface, a map can be produced of all locations where a particular characteristic element is present. If such an element is known to be specific to the ridges of the fingerprint, x-ray fluorescence can be used to reveal fingerprint detail.

2.7 X-ray imaging can also be carried out in transmission mode. In this mode it is the atomic density of an area that determines the intensity of x-rays transmitted through a sample. If a high atomic number element is present, fewer x-rays are transmitted and the area appears dark in the developed/collected image. If fingerprint ridges (or the background) can be preferentially doped with a high atomic number element, it may be possible to obtain contrast between the fingerprint and its background. This has been demonstrated using potassium iodide toning of a mark treated with physical developer and to a lesser extent with a mark powdered with bismuth salts.

3. Reasons technique is not recommended by CAST

3.1 CAST does not recommend electronography for operational use in police force fingerprint laboratories because of the hazards associated with the use of x-rays and the harmful nature of lead powder. In addition, no comparative studies have been carried out to demonstrate that electronography is more effective than other techniques for any of the applications for which it has been proposed.

3.2 X-ray fluorescence and x-ray transmission may be useful for practical application and the development of fingerprint reagents designed for x-ray functionality is feasible. However, the cost of analytical equipment is high and beyond the reach of most police forces. If the technique is to be used operationally it is likely that it will be confined to special cases, utilising equipment at establishments such as universities.

4. References

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6.3 Other specialised imaging techniques

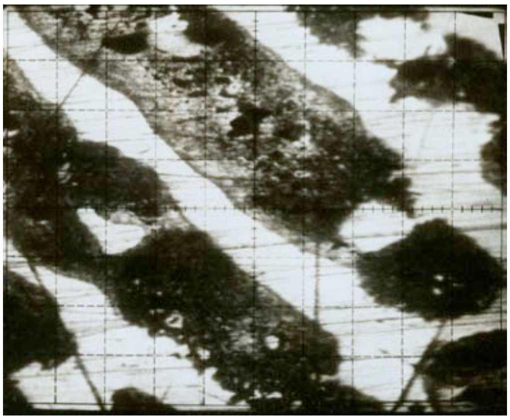
6.3.1 Secondary ion mass spectrometry (SIMS)

1. History

1.1 Secondary ion mass spectrometry (SIMS) has been used for many years as a technique for performing high sensitivity elemental analysis and operates by bombarding a surface with a high energy beam of particles and analysing the mass of the secondary ions emitted. The process was originally not suitable for surface analysis because the high energy beam progressively removed layers of material, but by the 1980s higher sensitivity detection systems were available that allowed the use of lower energy primary beam currents, and hence caused considerably less damage to the surface. Researchers began to explore the applications of SIMS for surface analysis, utilising the technique to identify the composition of surface coatings and small surface features [1]. SIMS was also used in an imaging mode, scanning the surface and detecting positions that specific molecular fragments were emitted from.

1.2 Bentz [2] applied SIMS to the analysis of fingerprint residues, in particular to the detection of traces of contaminants in the fingerprint. Many ‘natural’ fingerprints contained traces of silicones, and it was also demonstrated that small traces (nanograms) of illicit substances could theoretically be detected by the technique.

1.3 The Home Office Scientific Research and Development Branch (HO SRDB) funded an investigation of the use of the SIMS technique for both analysing fingerprint residues and mapping their distribution using the scanning mode [3]. These studies used fingerprints from six different donors, and confirmed the presence of sodium (Na^+) and chloride (Cl^-) ions in varying quantities. Spectra from all donors contained peaks indicative of the presence of both long- and short-chain aliphatic materials, and also peaks characteristic of silicones. Fragments representative of alkoxy and phenoxy groups were detected and, more specifically, spectra from all donors contained the main negative ion from myristic, palmitic and oleic acids. One donor also gave the stearate ion. The imaging mode was also successful in distinguishing between the composition of fingerprint ridges and that of the background.



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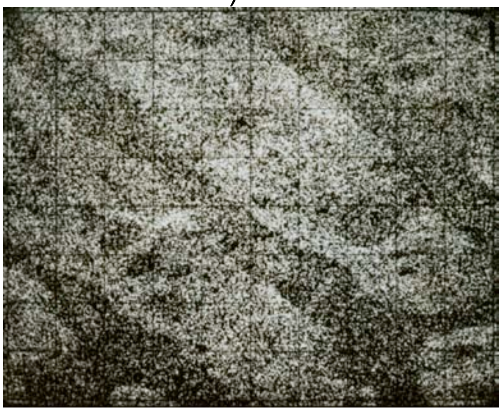


b)

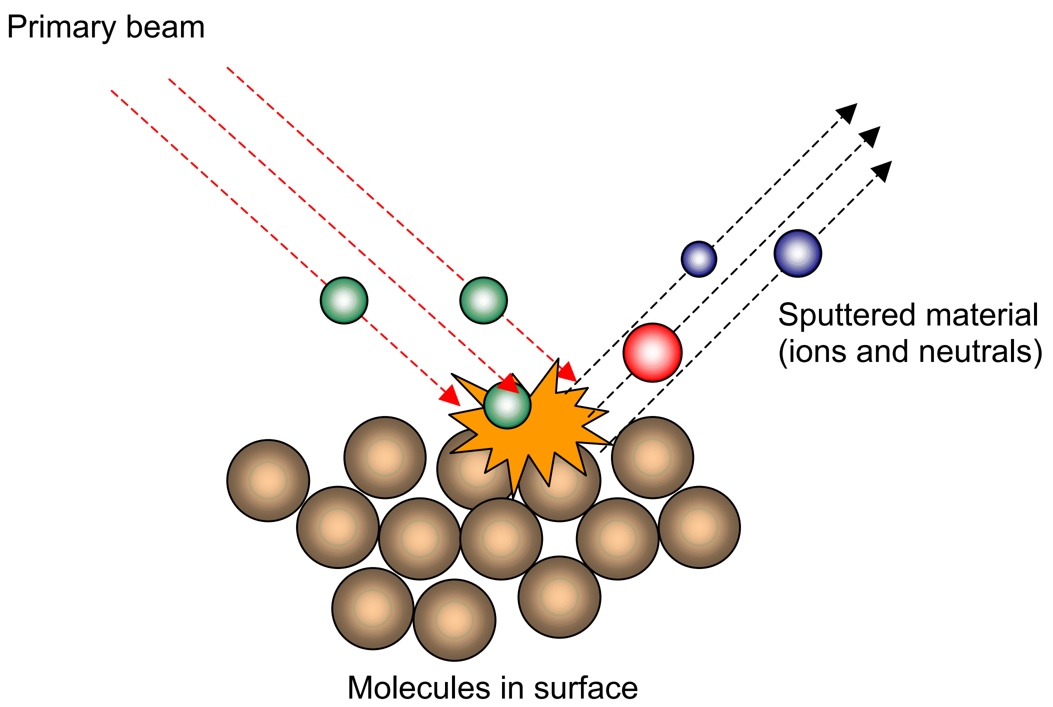
c)

Images obtained using scanning secondary ion mass spectrometry for surface analysis a) ion induced secondary electron image, b) Na^+ secondary ion image and c) $C_3H_5^+$ secondary ion image.

1.4 As the instrumentation available for SIMS has advanced, other researchers have also reported the use of the technique for fingerprint imaging and determining the distribution of principal constituents [4-6]. SIMS has also been used for depth profiling, examining the penetration depth of fingerprints into porous surfaces and determining whether printing or fingerprints were present first. The size of the instrument has also reduced and desktop systems are now available. It is unlikely that SIMS will become a primary fingerprint detection and/or imaging technique, but it can provide valuable information about fingerprint composition, contamination present in the fingerprint and contextual data. It may therefore be appropriate to use the technique in special cases.

2. Theory

2.1 The theory of SIMS is that an energetic beam of particles is used to bombard a surface in a vacuum. The collisions between the incident particles and the molecules in the surface layer produce a number of charged atoms, molecules and molecular fragments, which are ejected from the surface. This process is known as sputtering, and the ejected species are known as secondary ions. The secondary ions may be positively or negatively charged.



Schematic diagram showing secondary ions ejected from surface by the action of the primary beam.

2.2 The secondary ions ejected from the surface can be focused into a mass spectrometer where they are separated and identified according to their mass to charge ratio. Under appropriate conditions, minimal fragmentation of the surface molecules occurs and the molecular ions present can be more readily identified.

3. Reasons technique is not recommended by CAST

3.1 CAST does not recommend the process for routine operational work because it will normally be destructive to the exhibit, involving cutting an area small enough to fit inside the chamber of a SIMS instrument. In some circumstances SIMS may be capable of providing additional information about a fingerprint and its use should not be discounted. Suitable instruments can be found in some universities.

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6.3.2 Scanning Kelvin probe

1. History

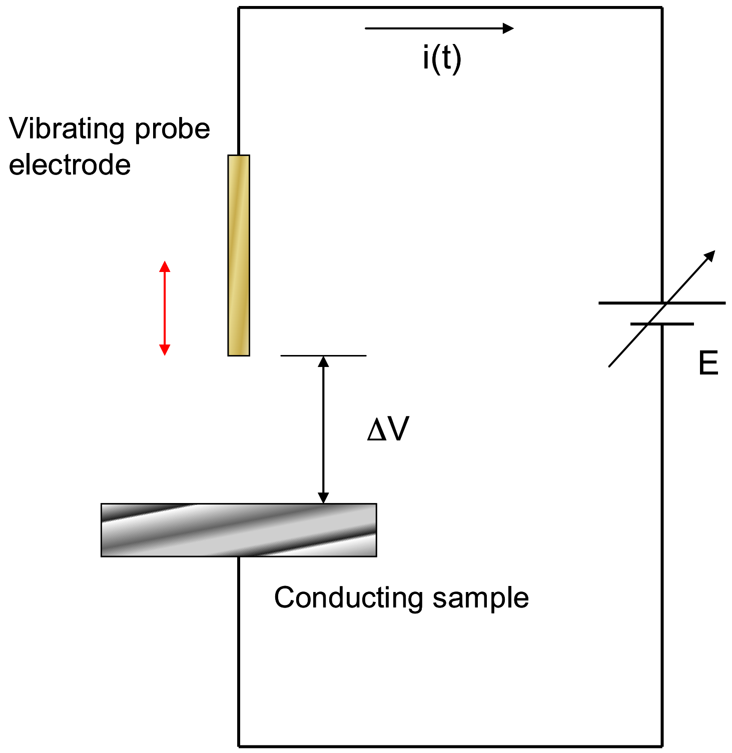
1.1 The scanning Kelvin probe technique was developed for the detection of corrosion occurring on metal surfaces. However, it was noted by Williams *et al*. [1] that electrochemical interactions may also occur between fingerprint deposits and metal surfaces and they subsequently investigated the application of the scanning Kelvin probe technique to fingerprint detection. Initial results were promising, with fingerprints being imaged on metal surfaces heated to 600ºC and beneath layers of insulating films.

1.2 Subsequent research by the same authors showed that the process was applicable to a range of metal surfaces and could still detect traces of fingerprints on surfaces where the residue had been rubbed away with a tissue. The technique was also applied to practical situations and apparatus was constructed for the scanning of cylindrical items such as cartridge casings [2].

1.3 The technique has the advantage that it is non-contact and non-destructive. It could, in theory, be used as the initial stage in a sequential treatment process. However, it has not yet been compared with existing processes in terms of sensitivity or effectiveness and the Home Office Centre for Applied Science and Technology (CAST) is currently (2011) funding a research programme to carry out this study with the University of Swansea.

2. Theory

2.1 The scanning Kelvin probe consists of a fine, vibrating gold electrode brought into close proximity to the surface being examined. The vibrating probe tip and conducting sample surface form the two plates of a parallel plate capacitor, with the space between them (predominantly air but possibly including any non-conducting layers on the surface) forming the dielectric. If there is a Volta potential difference (ΔV) between the probe and sample surface, the periodic capacitance change caused by the vibrating probe generates an alternating current, i(t), in the external circuit. The Kelvin probe measurement is made by applying a d.c. bias voltage E until the value of ΔV, and hence i(t). is zero. The circuit is illustrated below.



Schematic diagram showing principle of operation of the scanning Kelvin probe.

2.2 It can therefore be seen that any slight changes to the conducting sample or the dielectric between the probe tip and sample surface will result in changes to ΔV and therefore the resultant Kelvin probe measurement. Both eccrine and sebaceous fingerprints can change the surface and dielectric sufficiently for changes in ΔV to be detected, giving contrast between areas of ridge and background when the probe is scanned across the surface. In the case of eccrine prints there may be an electrochemical reaction between the print residue and the metal that changes surface potential, whereas in the case of sebaceous prints an additional layer of dielectric material is deposited on the surface.

3. Reasons technique is not recommended by CAST

3.1 CAST does not currently (2011) recommend the scanning Kelvin probe process for fingerprint detection because its relative effectiveness has not been established. However, the process is non-destructive, both for subsequent fingerprint development techniques, DNA recovery and examination of firing and rifling marks and there is no reason why it should not be utilised if the situation warrants it. The process is relatively slow, taking several hours to scan a single cartridge casing at high resolution, but for serious cases may provide valuable information. It is hoped that the planned comparative study will enable more detailed advice to be given on the use of this technique.

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