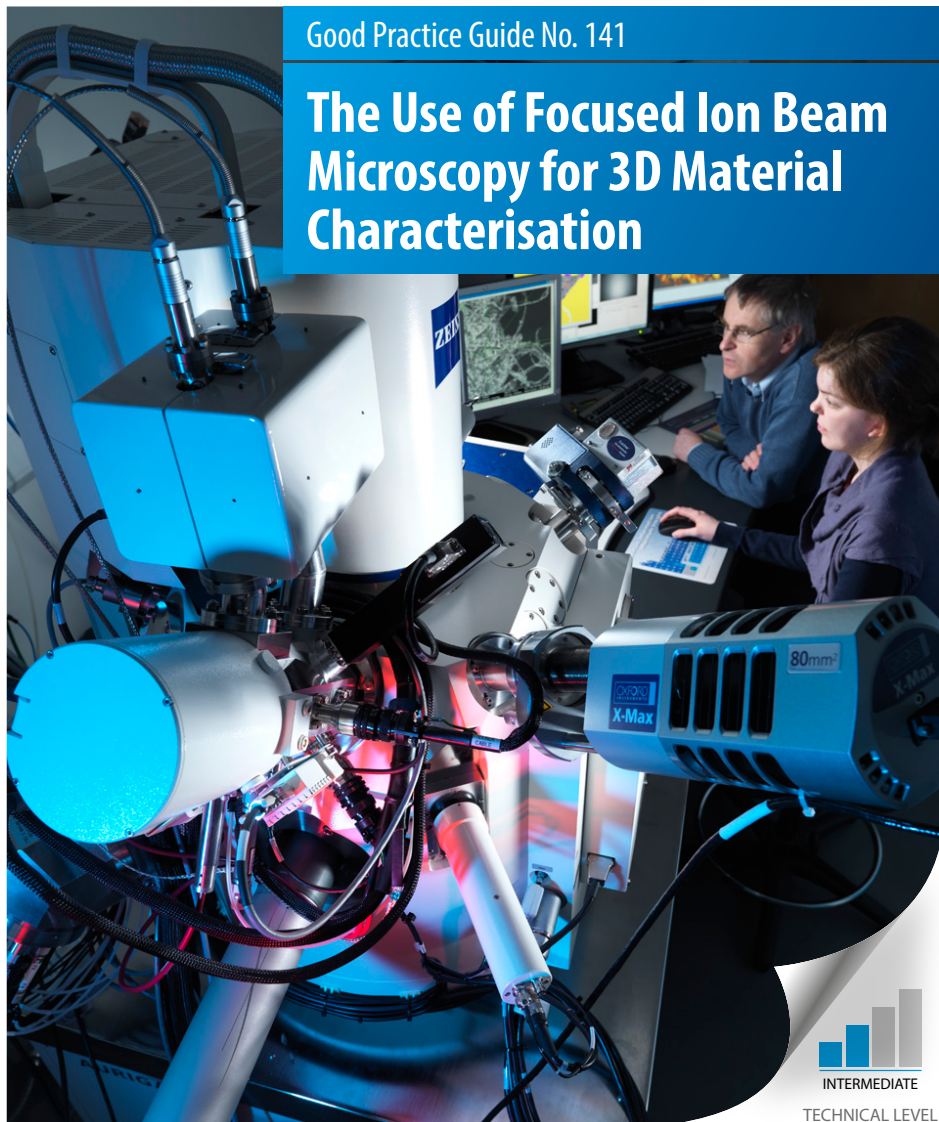


Good Practice Guide No. 141

The Use of Focused Ion Beam Microscopy for 3D Material Characterisation



INTERMEDIATE

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Front cover image: *NPL's Zeiss Auriga dual beam microscope. (Source: NPL).*

Guide Information

What is it about?

A Focused Ion Beam Scanning Electron Microscope (FIB-SEM) combines the column of a Scanning Electron Microscope (SEM) and a Focused Ion Beam (FIB) microscope in one instrument to enable both imaging with electrons and imaging and milling (local removal of material) with a beam of positively charged ions.

Who is it for?

This guide is most suited for users with existing SEM knowledge and new to FIB. Also, those who are working to improve their results, typically Masters/PhD students or users new to the field.

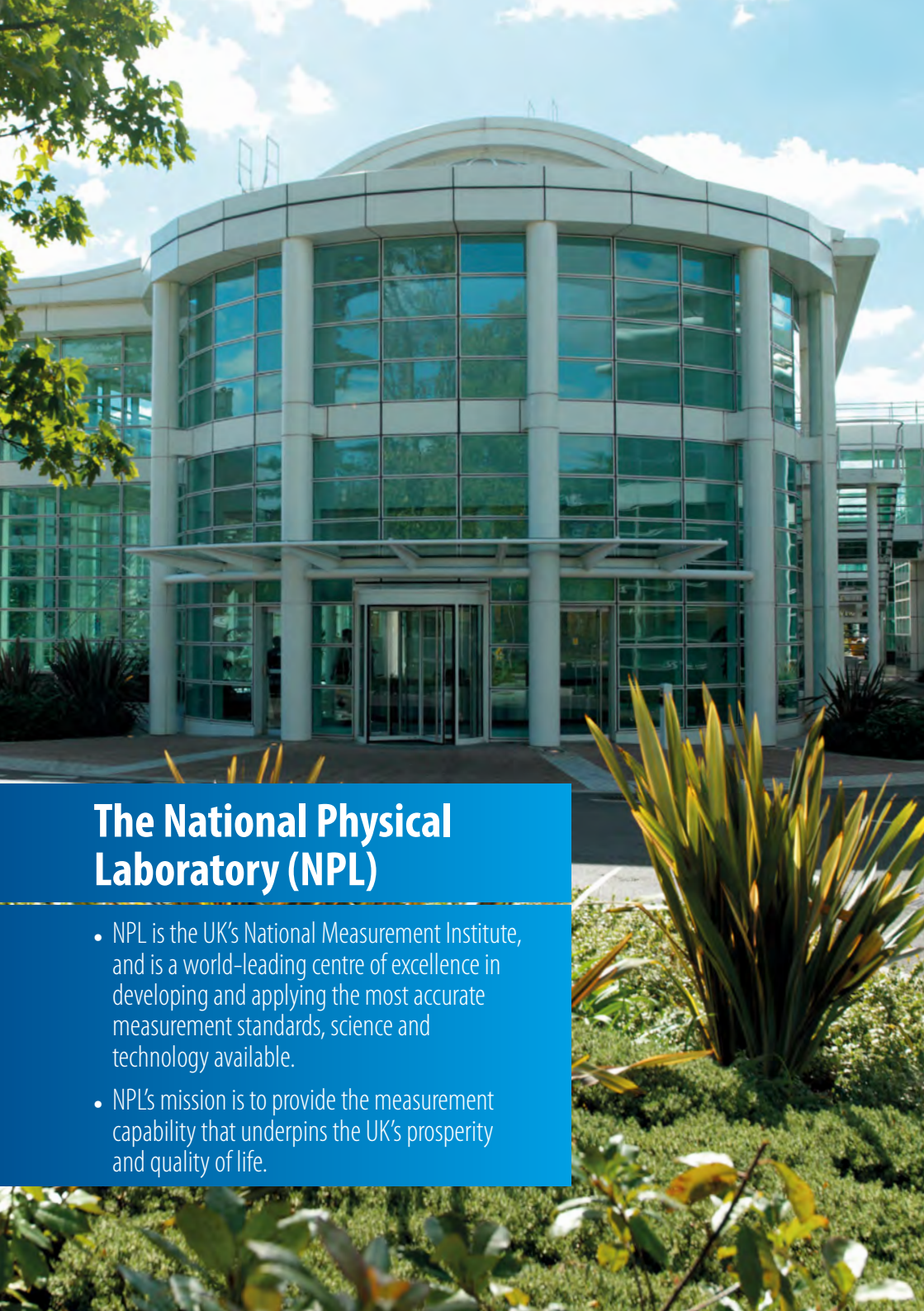
What is its purpose?

The purpose of this guide is to assist users to better understand the capabilities of a FIB-SEM for acquiring two and three dimensional microstructural information for most samples.

It aims to help the user to optimise the conditions for conducting experiments and the most suitable way to represent and analyse their data and assess the accuracy of this data. The guide does not intend to discuss in any great detail the other widespread uses for which the FIB-SEM is often used, such as preparation of samples for transmission electron microscopy, atom probe microscopy, or micromachining micro-devices. Nevertheless, the methods discussed for reducing ion damage and measuring uncertainties in milled materials can be applied to these wider capabilities.

What is the prerequisite knowledge?

Readers should be trained users of scanning electron microscopes and focused ion beam dual-beam system (SEM-only users can still use relevant parts of the guide as needed). Readers should also be capable users of and have access to image analysis and reconstruction software.



The National Physical Laboratory (NPL)

- NPL is the UK's National Measurement Institute, and is a world-leading centre of excellence in developing and applying the most accurate measurement standards, science and technology available.
- NPL's mission is to provide the measurement capability that underpins the UK's prosperity and quality of life.

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Foreword

This Good Practice Guide has been written by the National Physical Laboratory during the first phase of the internal project 'Grains to Structures', which ended in June 2014. It therefore covers work covered up to this time and will be updated in future Good Practice Guides as more knowledge is acquired and processes are improved.

The document describes recommended procedures to aid the operation of, and how to use, focused ion beam and scanning electron microscopes for specifically materials characterisation. Test procedures suggested in this document are general methods based on accepted good practice at the time of writing.

Introduction

- An introduction to microstructural characterisation in the FIB-SEM
- Capabilities and configuration of the FIB-SEM

An introduction to microstructural characterisation in the FIB-SEM

The need to characterise the microstructure of engineering materials in three dimensions (3D) has grown with the need to understand or predict their behaviour and properties under increasingly demanding conditions. This need includes not just the understanding of strengthening processes but also, for example, damage development such as pit or crack growth in corrosive environments. Modelling material's behaviour in 3D also requires accurate representations of microstructure if models of mechanical ^[1] or electrical ^[2] properties are to produce realistic property predictions; or, if simulated microstructures are used in models, the accuracy of the simulations requires verification ^[3].

Three dimensional characterisation of microstructures can be achieved by a number of different methods at different length-scales, of which the most common includes X-Ray Diffraction (XRD), X-ray Computer Tomography (XCT) and various serial sectioning techniques including ultramicrotomy, mechanical polishing, Broad Ion Beam (BIB) milling, and the subject of this paper: Focused Ion Beam (FIB) microscopy. XCT ^[4, 5] has the major advantage over the other techniques of being non-destructive, but to achieve resolutions of less than 1 μm needed for many microstructural studies has limitations of depth penetration and sample size in high density materials such as metals, or requires synchrotron X-ray sources ^[6, 7].

Microtome sectioning is widely used for biological materials ^[8], but for engineering materials the technique is limited to soft, ductile alloys such as aluminium ^[9]. Sections as thin as 10 nm can, however, be produced from aluminium to view volumes up to 50 μm across. Mechanical polishing is a very time consuming technique, but recent automation of the entire polishing and imaging process ^[10] has enabled analysis of very large volumes, several hundred times greater than is achievable by FIB sectioning, although with less control of the slice thicknesses. BIB can produce larger volumes ($> 200 \times 200 \times 50 \mu\text{m}$) having undamaged surfaces suitable for high-resolution Electron Back Scatter Diffraction (EBSD), but stacked layers are usually concave and non-parallel ^[11, 12]. FIB is limited to volumes generally less than $50 \times 50 \times 50 \mu\text{m}$, but can produce very high resolution images of almost all types of engineering materials.

The use of FIB to characterise the microstructure of materials in 3D by reconstructing serial sections has increased rapidly over the past decade as the capabilities to control the ion beam in order to machine and image a wide range of materials has increased. Focused Ion Beam-Scanning Electron Microscopes (FIB-SEMs) can be used for imaging and ion milling a large range of materials, from metallic samples and non-conductive materials such as ceramics and polymers to biological structures. Their use in materials science has been reviewed in a few papers ^[13, 14].

They are also widely used for micromachining and fabrication of micro- and nano-scale structures which can be tested in-situ if necessary. While fabrication is not an objective of this report, some aspects of common fabrication techniques, such as deposition of metal layers, are also needed for microstructural studies and will be covered briefly in the following section.

Capabilities and configuration of the FIB-SEM

Figure 1 below shows schematic diagrams of a possible set-up of an FIB-SEM: specifically it shows the instrument at NPL, which is currently more focused on material characterisation than fabrication.

Figure 1b shows the positions of standard imaging detectors in approximate relation to the electron and ion columns. The InLens secondary electron (SE) and Energy Selective Backscatter (ESB) detectors actually sit within the electron column while the Secondary Electron/Secondary Ion (SESI) detector sits lower within the microscope chamber. The 4-quadrant backscatter detector (4QBSD) is moved in directly between the electron column and sample when required, enabling it to detect high-angle backscattered electrons (BSEs). Chapters 3 and 4 describe the uses of these different detectors and imaging procedures.

Note that most FIB-SEMs are configured to produce a coincidence point, i.e. the common point at which both the FIB and electron beam will focus simultaneously at a working distance of typically between 4 and 5 mm. This is based on an optimum working distance to enable use of almost all the imaging conditions that may be required during or after milling. However, for certain applications, such as lamella preparation, the FIB column is positioned so that a longer working distance is achieved (up to 10 mm is common).

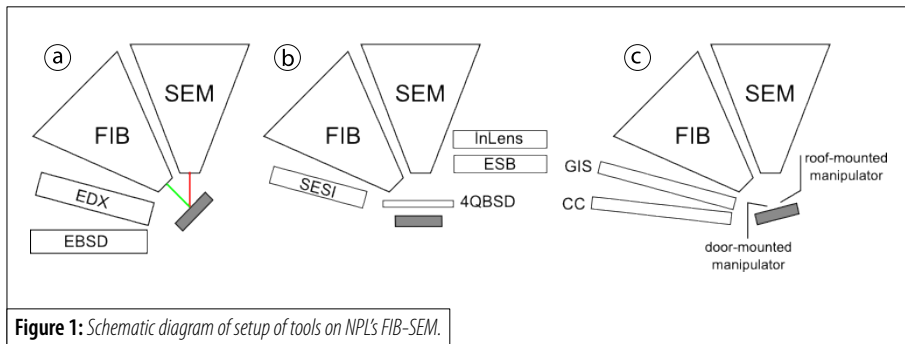
Figure 1a shows the positions of the Energy Dispersive X-ray (EDX) and EBSD detectors for, respectively, mapping of the chemistry and crystal structure of a sample. The sample stage is shown tilted perpendicular to the ion column. 3D image and EDX acquisition can be carried out in this position from a section milled in the surface without moving the stage, as reasonable electron or X-ray signals can be obtained with suitable sample preparation. To acquire 3D EBSD data, the sample needs to be pre-tilted and rotated 180° between milling and mapping in this particular arrangement, with milling having to be carried out on the edge of a sample. 3D data acquisition procedures are discussed in Chapter 7.

Figure 1c shows other tools that can be inserted very close to the sample; these are the Gas Injection System (GIS) needles, Charge Compensation (CC) probe and two micro-manipulators mounted in different positions.

CC – allows SE imaging of non-conducting samples

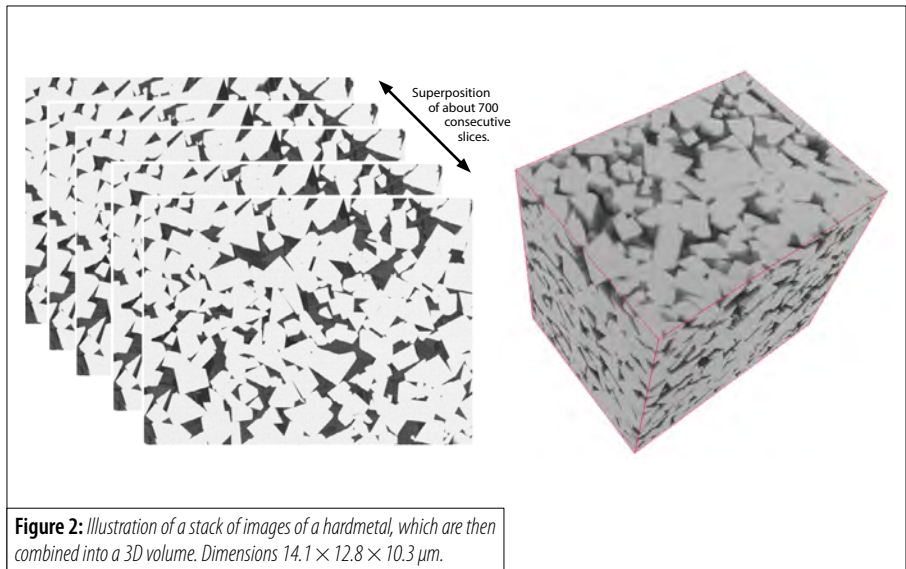
Electron flood gun – allows FIB milling on non-conducting samples

GIS – metallic precursors used for coating small regions of samples for better electron conductance,



Literature review

FIB microscopes produce 3D structures using a tomographic approach in which successive thin slices of the material are cut or milled by the FIB and a scanning ion or electron beam technique is used to image, or to map characteristics of the surface revealed by the removal of each slice. The images acquired can then be combined in a stack to produce a 3D representation (Figure 2), extrapolating between similar features on adjacent images, although the slice thickness is often of a very similar dimension to the pixel size within the images themselves.



In this type of ‘slice and view’ procedure, described in many papers ^[15, 16] it should be emphasised that the term ‘slice’ is used to describe the destructive removal of a layer of material by physical sputtering with ions, conventionally Ga^+ , and the imaging of the material revealed underneath the layer. This can be contrasted with the ultramicrotome technique which cuts a slice that is then physically removed and imaged.

Dual or cross beam microscopes combining an FIB with a scanning electron microscope have been made commercially for about 20 years ^[17], but initial studies of microstructure were made with single ion beam microscopes ^[18]. However use of the dual beam technique for material characterisation has grown significantly since about 2005, with a wide range of applications e.g.: the study of the microstructures of intermetallics in Mg ^[19] and Al-Si ^[20], steels ^[21], Ni superalloys ^[15, 22–24], oxide growth on zirconium alloys ^[25], ceramics ^[26–28], cast iron ^[29] and in fuel cells ^[16]. It is also used extensively to characterise microstructural damage resulting from,

for example, fatigue cracks ^[30], white etching areas ^[31], nanoindentation and sub-surface deformation ^[28, 32] and deformation of micropillars ^[33]. It has also found use in biological applications ^[34, 35] where serial sectioning using microtomes is already an established method, and for many other non-metallic materials such as cement pastes ^[36]. Recently FIB-SEMs together with Digital Image Correlation (DIC) analysis allowed for scaled-down conventional residual stress measurement methods, such as hole-drilling, ring-coring, slotting, etc. ^[37–40]

Quantitative and qualitative descriptions of the 3D structure thus generated can be obtained by measurement of a wide range of features, and often these will be compared with 2D measurements since the latter are far more easily obtained or routinely carried out. Measurement in 3D brings new possibilities for descriptions of the microstructure but also introduces scope for significant new errors in measurement, to add to the errors possible in 2D which have been investigated in depth over many years but are still regularly encountered ^[41, 42].

Electron-solid interactions and image generation

- Introduction
- Secondary electron imaging
- Backscattered imaging

Introduction

One of the most basic operations carried out on modern FIB instruments is the imaging of samples. Utilising both the scanning electron and ion columns gives rise to a large number of imaging modes giving different types of information about the sample. Turning firstly to electron-material interactions, the focused electron beam gives rise to a range of useful signals. These are derived from elastically scattered electrons: BSE imaging and forescattered electrons (primarily used in EBSD but are also used for imaging), inelastic or SE and X-rays (detecting either energy or wavelength information). Other less commonly used signals are based on Auger electron generation, cathodoluminescence and beam induced charge effects which are generated but rarely detected on FIB instruments. Most modern FIB instruments will be equipped with detectors for SE and BS, with EBSD and X-ray detectors being as-specified options. These imaging modes will be discussed in this chapter.

A modern electron column is capable of delivering a sub-nanometre beam spot of electrons accelerated across a potential in the range of 200 V to 30,000 V, with a typical beam current in the pA to low nA range (if using a Schottky source). The electron spot is usually rastered across the sample in a square or rectangular pattern dwelling for short periods (typically from 100 ns to tens of ms) at a number of points along a series of parallel lines interacting with the sample to generate different types of signals as it dwells at each point. A range of detectors collects these signals and they are usually displayed as signal intensity in a grey-scale image for SE and BS, with the option of colour images for X-ray and EBSD owing to the extra information available. Each point in the irradiated rastered region will correspond to one pixel in the image. Typically, these scanned regions will consist of the order of 1000 points per line and 1000 lines, although both significantly higher and lower numbers and non-square scans are easily achievable. The simplest description of the resolution for any given magnification can be thought of as the scanned area divided by the number of points in the scan giving each pixel an easily calculable size. However, as magnifications increase, or alternately the number of pixels is increased, a point can be reached where the pixel size corresponds to the size of the interaction volume of the beam, and this simple description breaks down as the beam spots begin to overlap. Regarding beam dwell times, this parameter will affect the time taken to perform a single scan of the area. Short times will lead to fast acquisition rates but noisy images, whereas slower scans will improve the signal to noise ratio but take longer to complete and, for very slow scans with dwell times > 50 ms, may often produce distorted images (image shear, stretch, etc.). Most modern electron or ion beam instruments have software based averaging and integrating methodologies to further improve the signal to noise ratio and suppress image distortions.

Although the beam spot may be sub-nanometre in size, high-energy electrons scatter on contact with the sample and can give rise to signal generation from significantly larger areas. The interaction volume of the electron beam will vary depending upon: (i) the energy of the beam; (ii) the density of the specimen; and (iii) the incident angle of the beam as it interacts with the sample. As electron energies are increased, the electrons can penetrate deeper into the sample and can also scatter and re-emerge at a significant distance from the incident point. However, as sample density is increased, the interaction volume decreases and electrons are either stopped or backscattered closer to the surface, owing to the increased stopping power of higher atomic number materials. The maximum electron depth will also be decreased as the sample is tilted away from orthogonal to the beam, however, the interaction volume will become elongated near the surface and a higher probability of electrons scattering back out of the sample will occur. (This last phenomenon is the mechanism used for EBSD analysis.) When imaging samples, the likely interaction volume of the beam should be taken into account and an appropriate beam energy used for the chosen detector. Monte Carlo methods are typically used to model electron sample interactions and quite accurate interaction volumes can be calculated with this method if the sample density is well understood^[43, 44].

In summary, the key parameters (assuming a properly focused and aligned beam) to be considered are:

Accelerating voltage

- Lower voltages are likely to give more contrast in images, and are better for low/non-conducting samples and to reveal surface information, but will give a smaller signal and hence may require slower scanning to achieve low noise images.
- Higher voltages give more signal and thus shorter scan times. They penetrate through thin contaminant layers and are essential for backscatter imaging, EBSD, and are also often used for EDX.

Aperture/spot size

- Smaller apertures will give better resolution but less signal.
- Larger apertures produce more current, enabling more signal and better EBSD to be acquired.

Pattern averaging, pixel binning and step size

- Increased frame averaging without pixel binning will give better signal to noise ratios and sharper patterns but with the increased risk of drift during acquisition. The same applies when using a small step size over a large area. This drift causes maps to consequently become distorted. Therefore, a compromise between map duration and resolution is important.

The next sub-section describes the different signals that can be detected and Figures 3 to 5 illustrate some of the differences between the signals, at a range of accelerating voltages with particular reference to FIB milling of microstructures.

Secondary electron imaging

As the beam impinges on the sample surface, loosely-bound low-energy electrons (typically less than 10 eV) will be ejected from the top few nanometres of the sample. These electrons, known as Secondary Electrons (SE), can be collected in two distinct types of SE detector, an Everhart-Thornley (ET) type detector, or an InLens SE detector.

Everhart-Thornley secondary electron detectors

The ET detector is often referred to as an SE detector (or Secondary Ion (SI) in the case of positively charged ions). Typically the ET detector will be chamber mounted and some distance from the sample. A grid on the front of the detector is positively biased to attract the low energy electrons as they are ejected from the sample surface. Secondary electrons originating from near the sample surface in the proximity of the beam spot are used primarily for imaging of sample topography and the different intensity recorded at each pixel is used to construct the grey-scale image. (Note that there are different types of secondary electrons.)

Owing to the chamber mounted location, ET detectors are prone to noise, as backscattering electrons from the sample can also generate secondary electrons from the other parts of the sample, or even parts of the instrument in the chamber. Furthermore, in an SE image there is often a component of the signal that is from BSEs entering the detector and it is this mechanism that gives rise to some compositional and crystal channelling contrast. It is possible to change the sign of the bias on the grid and exclude low energy electrons from the detector, giving only backscattered or diffraction contrast, although this is an inefficient mode of operation for these detectors, and image quality is almost always poor. The collection efficiency of ET type detectors also falls rapidly when the sample is tilted away from the detector and increases when the sample is tilted towards the detector, which should be considered when combining ion milling of holes and cross-sections with SE imaging. This mechanism also means that ET-SE detectors are good at revealing surface topography, due to the shadowing created by surface features blocking electrons from the detector.

Figure 3 shows ET-SE images of the milled nose of a cross section at the edge of a sample. Note the good topography visible at all accelerating voltages, particularly of the fiducial cross on the top surface which faces towards the detector. The signal

on the other face, while lower, and more significantly affected by lower voltages, is still good. The reduction in signal at low kV has the benefit of reducing contrast from the curtaining, while the sensitivity of the signal to orientation effects is increased because of a greater contribution to the signal by BSEs. The topographical contrast possible from ET-SE may be contrasted with the images in Figures 4 and 5 using InLens detectors.

InLens secondary electron imaging

The InLens SE detector is a far higher efficiency detector of secondary electrons. This detector is located within the electron column and is usually only used when a sample is at relatively close proximity to the pole piece (i.e. short working distance), as the efficiency falls rapidly once working distance is increased. The increase of the efficiency of this type of detector is due to an electrostatic field being established between the pole piece and sample when the detector is active, such that it effectively prevents secondary electrons from the sample escaping detection, but also prevents SEs generated by BSEs outside of the field from entering the detector (the field strength is insufficient to prevent backscattered electrons escaping). This field diminishes with distance, hence the drop in efficiency at long working distances. Fortunately the coincident point of the electron and ion beams in FIB instruments occurs at short working distances and so for conventional operation of these instruments the sample is usually in the range where InLens detectors are effective.

The enhanced efficiency of InLens detectors allows smaller beam currents and faster scan rates to be used while still obtaining better signal to noise performance than ET-SE detectors. The ability to obtain better signal to noise at lower beam currents (spot sizes) means InLens detectors offer higher resolution than ET type detectors. The exclusion of BSEs means the sensitivity of the signal to surface layers is enhanced, which can be an advantage for imaging thin layers and fine surface steps details, but conversely any thin contaminant layers which can build up under the electron beam will become much more prominent and may obscure detail.

Comparing the InLens images in Figure 4, with the equivalent ET-SE images of Figure 3, shows the increased signal from the milled face, showing the microstructure more clearly, and the reduction in topographic contrast. A greatly increased orientation contrast is also achieved at lower accelerating voltages: however, any curtaining on the milled surface becomes more pronounced.

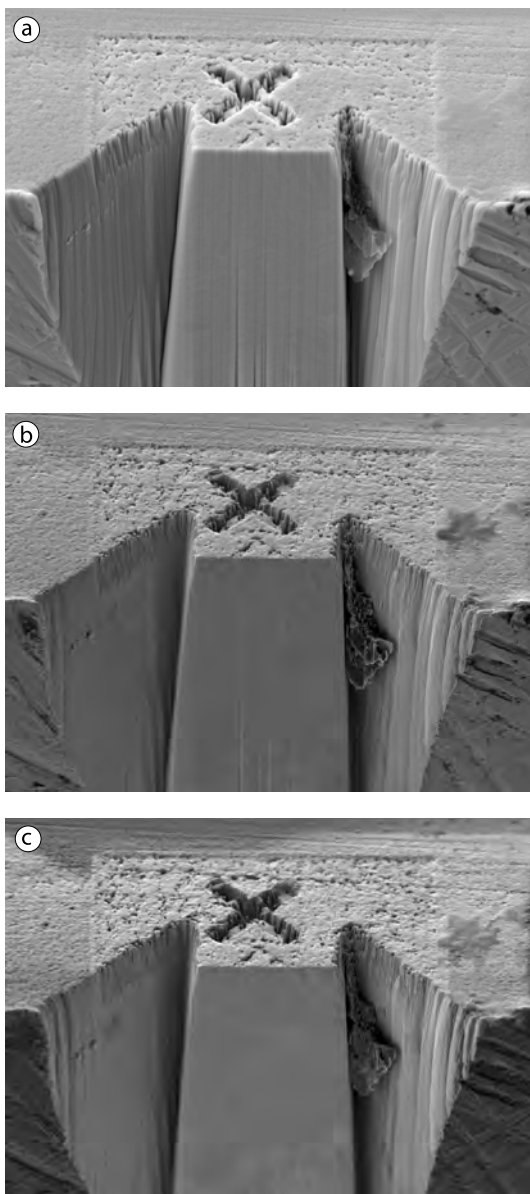


Figure 3: Secondary electron (ET) images of a tilted steel sample prepared for EBSD, taken with accelerating voltages of a) 20 kV, b) 5 kV and c) 1 kV.

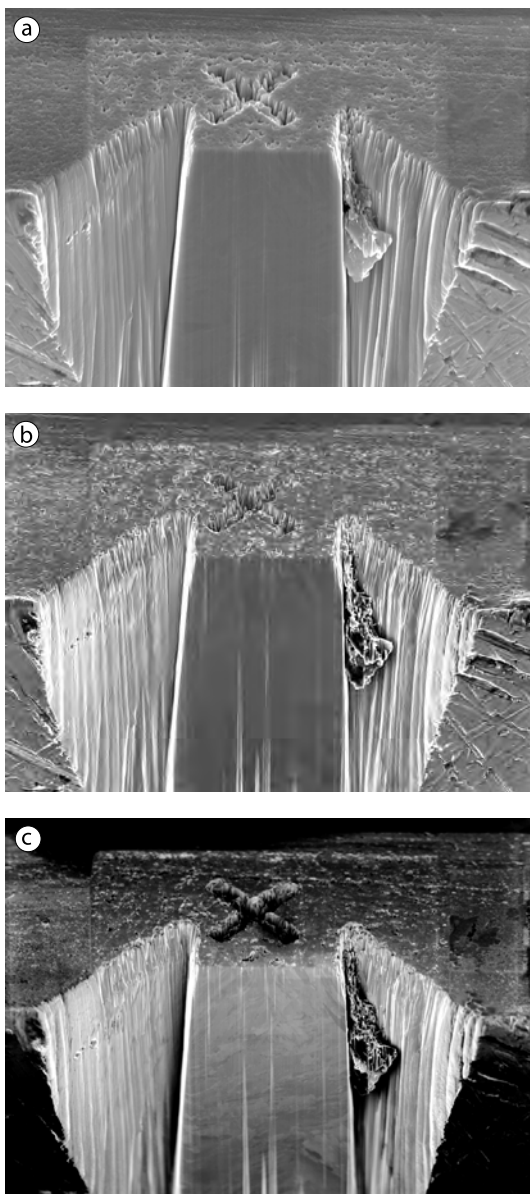


Figure 4: *InLens SE images of the same tilted steel sample shown in Figure 3, taken with accelerating voltages of a) 20 kV, b) 5 kV and c) 1 kV.*

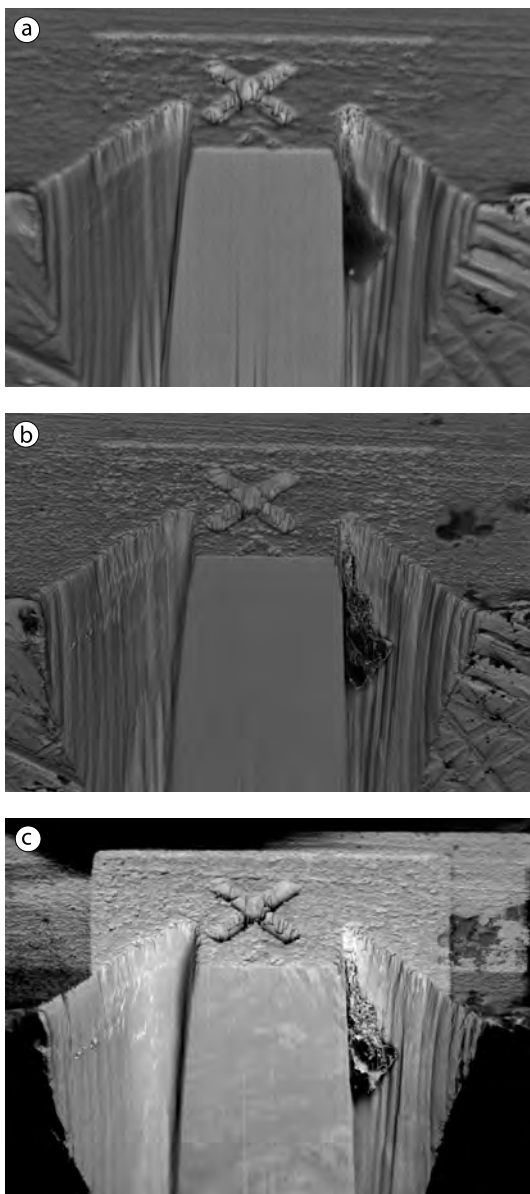


Figure 5: InLens energy selective BSE images of the same tilted steel sample shown in Figure 3, taken with accelerating voltages of a) 20 kV, b) 5 kV and c) 1 kV.

Backscattered imaging

Electrons from the beam interact with the sample and scatter straight back out into the detector. Owing to the fact that the electrons have much higher energies approaching the energy of the beam, BSE detectors tend to be inserted directly above the sample, in front of the pole piece.

Diode detectors

Two types of BSE detector are common. The first type is typically located on a retractable rod and is inserted when needed between the pole piece and the sample. The detector has a hole in the centre that the incident beam passes through, and can be made as one piece, as well as split into a two piece detector, a four segment detector or even concentric rings.

The high sensitivity of the BSE yield to atomic number means this type of detector can be very good for multiphase samples, and the ability to use individual quadrants allows for good topographical imaging and even 3D surface reconstructions. However, the need for the insertion of the detector below the pole piece means it is rarely used in conjunction with FIB milling because the latter requires short working distances and tilted samples.

InLens backscattered electron detectors

The second type is the ESB detector, which is located in the electron column in some microscopes, just above the SE InLens detector. It detects the BSEs at a range of energies, but has the capability of filtering out the lower energies to reduce the noise in the image. Due to its position with respect to the electron beam and the nature of its properties, it gives very low topography resolution but is very good at distinguishing subtle atomic number and orientation contrasts, as shown in Figure 5. These can be compared to Figure 3 and 4 where it is shown that at < 2 kV accelerating voltage, there is excellent orientation contrast on the milled section and minimal topography or curtaining contrast.

The ESB detector also gives low noise images during ion milling, since the low energy electrons produced by milling can be filtered. The voltage on the filter should be adjusted to get a good compromise between noise reduction and signal, and needs to be tailored to the accelerating voltage being used.

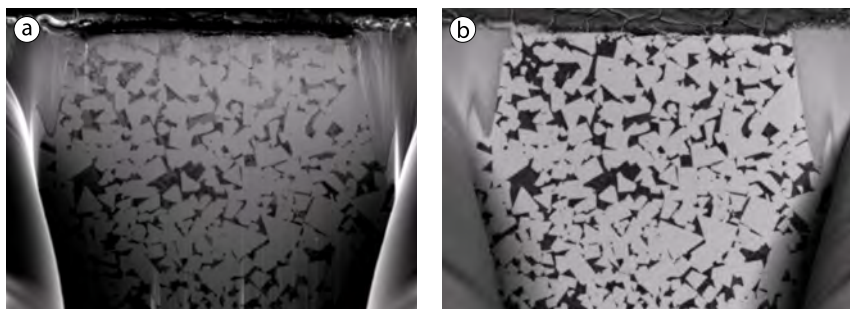


Figure 6: Comparison of InLens SE and backscattered detector images during slice and view of a WC/Co hardmetal where the light phase is tungsten carbide and the dark phase is cobalt, a) InLens SE showing contrast in cobalt phase but shadowing, and b) InLens energy selective backscattered image showing less shadowing but with less contrast within the cobalt phase.

The detector can also be less prone to shadowing than the InLens SE detector, as the two images shown in Figure 6 demonstrate. They were taken simultaneously towards the end of a slice and view process when shadowing by redeposition was increasing: thresholding of the two phases was much easier with the ESB image because of the more uniform brightness and contrast.

Ion-solid interactions

- Introduction
- Ion milling by sputtering
- Imaging with the ion beam

Introduction

The basic operation of an ion column is essentially very similar to an electron column. A source of ions is needed, a potential to accelerate them and a series of lenses and scan coils are used to focus and steer the beam around the sample. The most common form of ion source is a liquid metal ion source (LMIS) using the metal gallium (Ga). Other sources exist such as noble gases and even low melting point alloys with mass filters for selecting different species, but the type of source is exclusive to the particular instrument and cannot be interchanged. The scope of this document primarily concerns the use of gallium LMIS although the basic principle of operation is similar for the other sources.

A typical gallium LMIS ion column will accelerate a beam of ions across a potential of up to 30 keV. Ion columns are commonly operated at this potential but can be used in lower ranges to as low as 1 keV. The beam currents used will be wide ranging from as little as 1 pA to as much as 75 nA in some instruments. In the same manner as the electron column, the beam of ions is rastered around the sample, but often more complex patterns and beam paths are used, discussed in a later section.

Ion milling by sputtering

The main use of Ga^+ ions in FIB instruments is a process known as ion milling. Here the relatively large mass of the Ga^+ ion (almost six orders of magnitude heavier than an electron) is exploited to physically remove atoms from the sample surface. By carefully controlling the beam current, beam position, the step size of the beam spot and the dwell of the beam at any point, it is possible to remove sections of material very precisely to reveal internal structure of a material.

Each material will have a different sputter yield. This term describes the efficiency of the arriving Ga^+ ion in removing atoms from the sample surface. As an example of differing sputter yield, for a 30 keV Ga^+ ion impinging on graphite at normal incidence, the sputter yield is a little over one C atom per Ga^+ ion. By comparison, for pure Au the sputter yield under identical conditions is approximately 17 atoms per ion. Broadly speaking, the sputter yield increases with increasing atomic number of the specimen, although it is not linear and certain elements, for example Cu, have a considerably higher yield than their neighbours in the periodic table, whereas others are lower (see Appendix 2 for examples of pure element sputter yields). Furthermore, the sputter yield for a particular element will reduce if the energy of the incident ions are reduced by decreasing the accelerating voltage and it will increase significantly if the specimen is tilted, where the surface of the sample is not perpendicular to the beam. This is due to the available energy of the incident ions being released nearer to the surface as the angle is increased. Appendix 3 shows the effect of ion incident

angle on sputter yield, vacancy generation rate and ion backscattering.

Further variation in sputter yields arises when the sample is polycrystalline, as the incident angle of the ion beam may be aligned with open channels in the crystal lattice of some grains but not others. This will lead to ions being able to travel further into some crystals before they are able to scatter and release their energy with lower sputter yields. Grains that have no open channels will interact with the arriving ions much nearer to the surface and energy will be available to sputter atoms in the near surface region. Polycrystalline materials often show significant roughening during ion milling as some grains mill faster than others due to this effect.

For ion milling single-phase multi-element materials, the sputter yield will be determined by the average atomic number of the sample. For example, a material such as tungsten carbide has equal amounts of very low atomic number C and very high atomic number W; it would be expected that W would sputter with a much higher yield than C. However, this is only the case initially as the preferential removal of W will leave behind the lower yield C, changing the stoichiometry of the sample near the surface and reducing the overall yield as more C is present. In a real sample, this would give rise to a sputter yield approximately the average of W and C, but can still lead to subtle chemistry changes near the surface of the milled area due to some preferential sputtering. Strongly differing sputter yields can give rise to multiphase materials exhibiting very different milling rates for the different phases, making it very difficult to mill these materials uniformly.

Imaging with the ion beam

Secondary ion imaging

During the sputtering of atoms from the sample surface some of the ejected atoms will become ionised. These Secondary Ions (SI) can be detected in an SE detector with the biases reversed turning it into an SI detector. The SI image is created in the same way as an SE image. Predominantly the image will give information about surface topography, but the extra mass and size of the ions also give rise to very strong channelling effects in polycrystalline materials, leading to grain orientation contrast and contrast effects in multiphase materials. For SI imaging, an ET detector with reverse bias is used (often this is the same detector as the ET-SE) rather than the InLens detector in the SEM column, as it is not desirable to introduce ions into the electron column. Furthermore, use of the SI detector should not be encouraged unless necessary as the SIs will sputter away the phosphor on the detector, significantly shortening its life. It should be noted that SEs also degrade the detector phosphor, but at a massively reduced rate compared to energetic ions.

Secondary electron emission

As the Ga^+ ions arrive at the surface of the sample, electrons in the surface will be excited by the positively charged ions and forced from the surface. These secondary electrons are essentially the same as those derived from the electron beam and give rise to an imaging signal. This can be used to image the sample showing predominantly surface topography, but also generates elemental contrast, as higher atomic number elements give rise to significantly higher electron yields, just as they do with ion yields. Two important points to remember with SE imaging from ion beams are firstly, that whilst imaging is taking place the specimen is still being sputtered and even if the beam current is low, surface modification will be taking place (see Appendix 4 for examples of ion doses under different scanning conditions). Secondly, but beneficially, the interaction distance of a 30 keV ion in any solid material is very shallow, and is much shallower than even a 2 keV electron beam in the same material. This means that SE imaging from primary ions can give more useful surface information than electron beam generated SE imaging in some circumstances. See Figure 7 (compare with Figures 3-5) for ion-produced SE images.

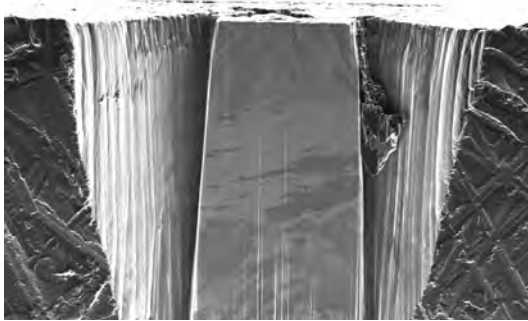
Ion damage and implantation

In all materials, a 30 keV Ga^+ ion arriving at the sample surface at normal incidence will implant into the material rather than recoil from the material. The statistical probability of recoil is almost zero, irrespective of the average atomic number. This means that even at modest currents, a very significant number of Ga^+ ions are being implanted into the near-surface region. With an energy of 30 keV, a typical average implant range will be of the order of 10 to 50 nm in most materials (see Appendix 3 for typical implant depths at varying incidence). Due to channelling effects in crystalline solids, the range can be increased by a factor of three for favourably oriented crystals.

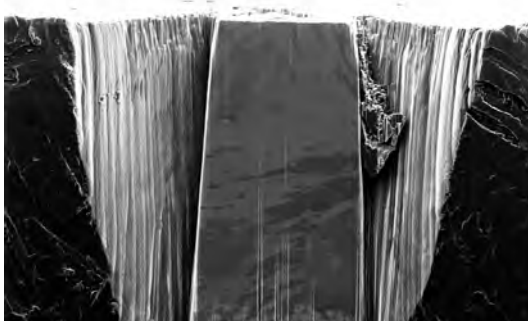
The arrival of a high energy ion into any solid material results in the liberation of the energy from the ion into the material. This energy can be transferred to atoms in the material that can either be ejected from the material (sputtered) if near the surface, or cause atoms to be displaced and move within the material. These mobile atoms can continue to collide with other atoms, triggering damage cascades and creating significant amorphisation of the crystal lattice.

In some materials the energy transferred into the sample by the ion implantation can prove sufficient to cause changes of phase, recrystallisation, and grain boundary movement. Such effects are difficult to predict, particularly in polycrystalline materials and complex alloys. The alloy chemistry may also be modified by the presence of the now implanted Ga^+ ions further driving these effects. This latter effect is very prominent in III-V semiconductor compounds where Ga has a very strong tendency to alloy with them, e.g. In, Al, As Sb, N and existing Ga.

(a)



(b)



(c)

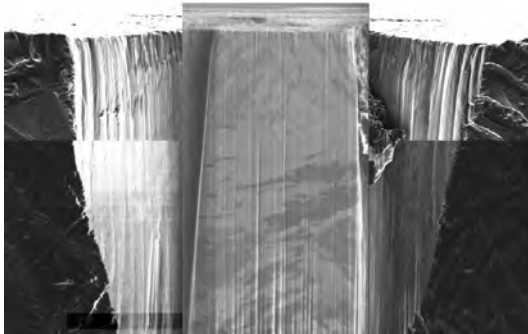


Figure 7: Secondary ion images of a steel sample (also in Fig 3-5) taken with beam currents a) 4 nA b) 1 nA c) 50 pA.

Phase transformations are readily observed in cobalt and alloys of Co with the face centred cubic (FCC) structure: Figure 8 shows an example of this in which low percentage additions of W and C stabilise the Co FCC phase, but after exposure to a 30 kV: 2 nA Ga^+ ion beam for 120 seconds significant hexagonal close packed (HCP) transformation can be seen, with some orientations more prone to transformation than others and some more resistant to milling than others. EDX mapping also

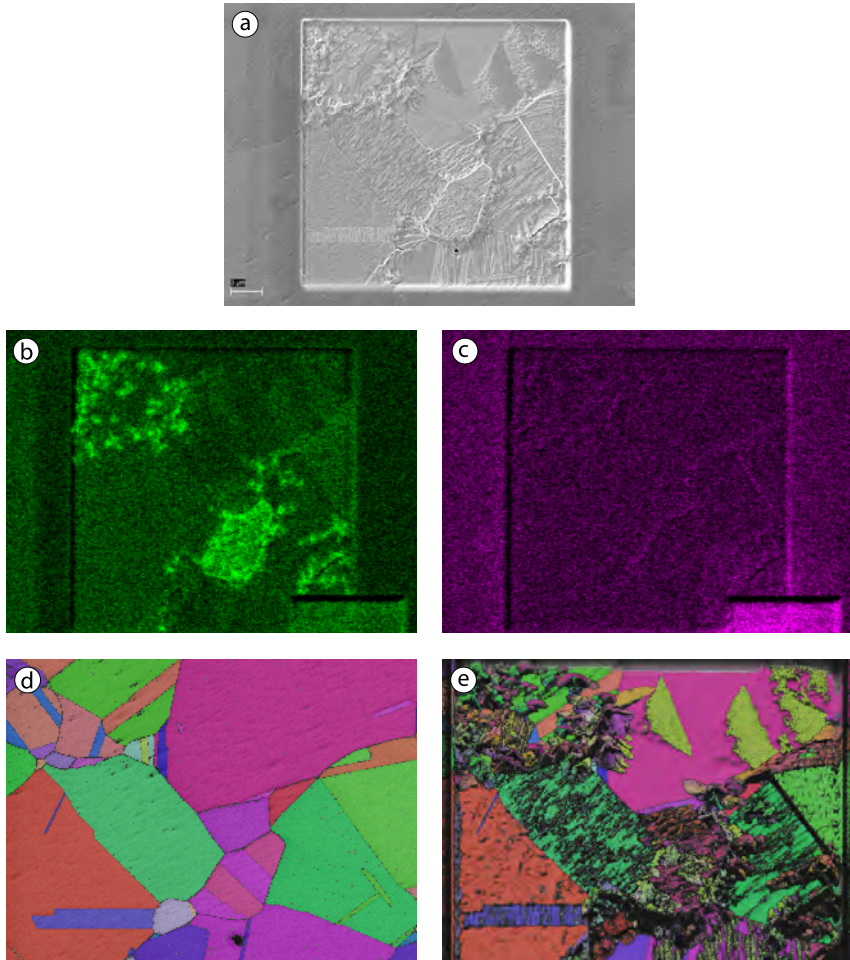


Figure 8: Transformation of FCC to HCP phase in cobalt: a) SE image of $25\ \mu\text{m} \times 25\ \mu\text{m}$ area of Co after exposure to 2 nA for 120 s, b) EDX map of a) showing gallium content, c) EDX map of a) showing carbon content, d) and e) EBSD orientation maps of, respectively, before and after milling of the area shown in a).

shows orientation dependence of Ga^+ ion implantation, with greater implantation in those grains milled away least. Figure 9 illustrates both phase transformation and amorphisation, this time with glancing angle milling of the face of a WC/Co hardmetal. Clearly the mechanically polished WC just visible at the edges of the map as red grains gave good patterns, but in the milled region no patterns were obtained from the WC because of amorphisation by the Ga^+ ion beam. By contrast good patterns were obtained from the Co in the milled region and a mix of FCC and HCP phase was detected, although the HCP probably resulted from the ion induced phase transformation seen in Figure 8, as earlier mechanical and argon ion polishing had shown only the FCC phase. The amorphised layer on the WC is, however, very shallow, as Figure 9c shows when mapping was carried out after a short milling run with a 15 kV:250 pA run. The equivalence of the Co region shapes shows that minimal material was milled away, but the WC could now be indexed; it is also noticeable that there was a substantial reduction in the proportion of the HCP phase produced by milling with this lower energy beam.

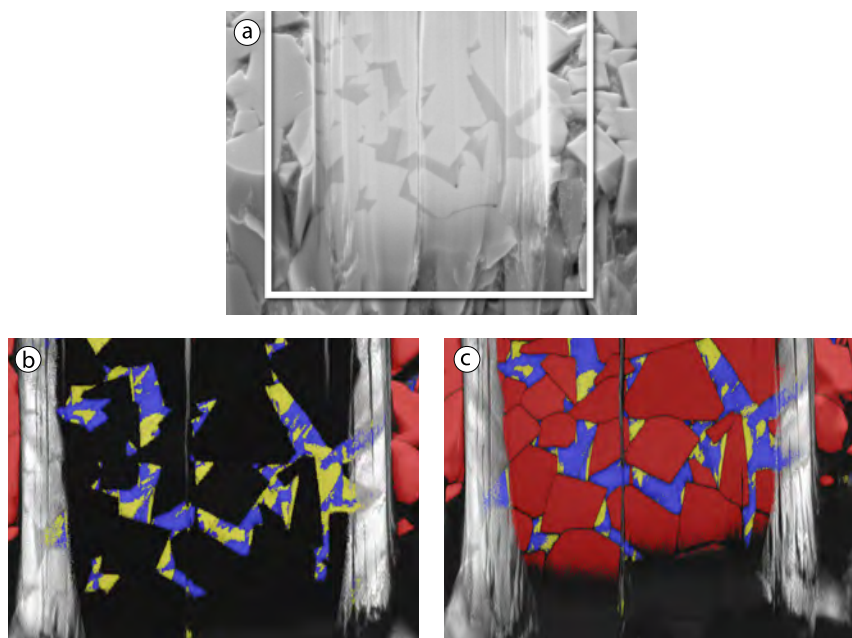


Figure 9: Images of an FIB milled area in a WC/Co hardmetal where the WC phase amorphises and the Co phase transforms. a) SEM image of milled area with a 30 kV:4 nA beam, with white box mapped by EBSD. b) EBSD map immediately after 30 kV:4 nA milling and c) after polishing with a 15 kV:250 pA beam. Points indexed as WC shown in red, FCC Co in blue, HCP Co in yellow, non-indexed in black.

Curtaining

Curtaining is frequently seen on milled faces. Examples can be seen in Figure 7 (especially the InLens image) and in Figure 9, where milling has produced irregular ripples or shallow trenches in an otherwise planar surface, running parallel to the beam. It results from a variation in the absorption/attenuation of the ion beam either as a result of an irregular top surface where the greater amount of material in a peak will require more milling relative to a trough, or variation in the sputter yield of different regions of a surface such as WC and Co, as seen in Figure 10. Where the proportion of Co (the darker phase) parallel to the beam is greatest there is more milling, producing a trench with the additional effect of reducing the EDX signal from this region.

The effect can be reduced by deposition with the FIB of a coating such as Pt on the top surface to smooth out ripples, or by using lower currents and milling more slowly.

In some materials, particularly carbon-based and ceramics, an uneven milled surface can also be produced which has more of the appearance of pillars. Figure 11a shows an yttria-stabilised zirconia material after milling with 2 nA. If, however, the milling is carried out without simultaneous electron imaging, this pillar effect can be reduced (Figure 11b).

Assisted ion etching

Two of the issues noted above can be minimised by the use of assisted ion etching using the GIS, where a gas is injected at the site of milling to increase the milling rate. In the case of WC, XeF_2 can be used to reduce amorphisation; this gas is also commonly used to increase preferential milling of Si. For carbon based materials, water can be injected which dramatically increases the rate of milling and reduces the tendency to form the pillars similar to those seen in Figure 11.

Ion beam resolution, beam spot size and beam tails

In normal operation, the beam of ions will converge on the sample in a focused spot which is as small as possible. Manufacturers often quote 'spot sizes' for given beam currents and beam energies assuming the sample is in focus. These spot sizes are seen to increase as the current is increased, and broadly the ion density per unit area of the beam is constant as the current is changed. At the lowest beam currents figures quoted are typically a few nm diameter, and a few hundred nm for the highest beam currents. See Appendix 1 for the spot size table for the Zeiss Auriga 60, as an example.

However, the quoted spot sizes are actually Full Width at Half Maximum (FWHM) values as the beam spot takes the form of a circular Gaussian distribution.

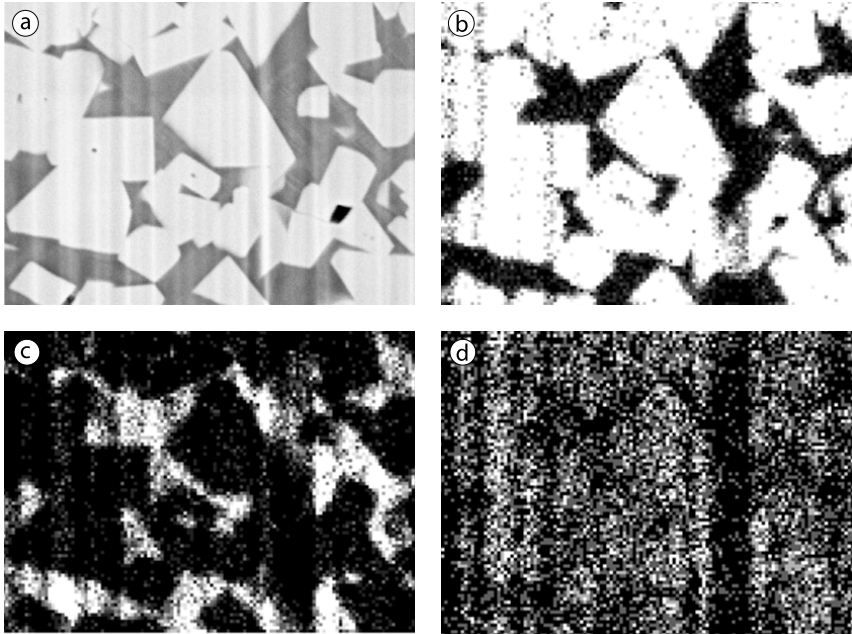


Figure 10: Images of an area on a WC/Co hardmetal that has been FIB milled with a high current and shows heavy curtaining. a) SE image showing curtaining grooves, b-d) W, Co and Ga⁺ EDX maps showing shadowing caused by the curtaining, as well as showing gallium implantation.

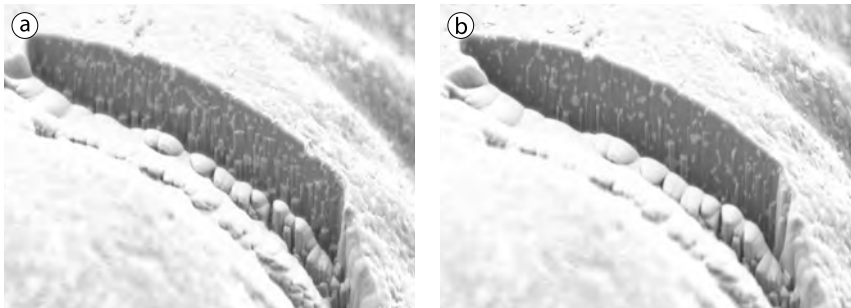


Figure 11: Yttria-stabilised zirconia sample milled with a 2 nA current, showing pillars on milled face a) with electron beam scanning during ion milling, b) with electron beam blanked during ion milling, showing a reduction in pillar formation.

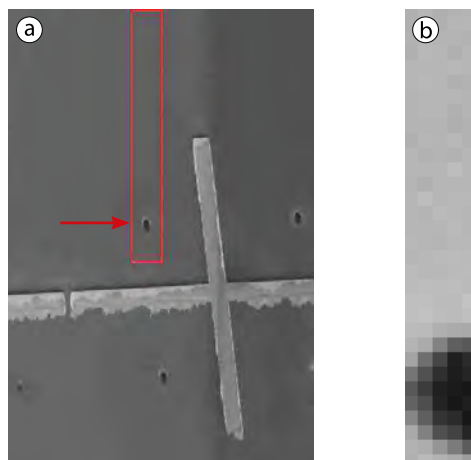


Figure 12: a) SE image of silicon showing points exposed to a 30 kV:5 nA beam for varying lengths of time, with the red box extending from one point denoting the region mapped by EBSD. b) EBSD map of band contrast produced from region in a).

The majority of the beam current will be contained in the FWHM portion of the distribution but a significant part of it will be contained in the tails and can extend well beyond the quoted spot size. At the smallest spot sizes the number of ions in the beam tails will be very insignificant, but at the larger beam currents the number of ions can easily exceed the amount required to amorphise the sample, and even to sputter significant material away from the focused beam spot. Frequently when attempting to ion mill vertical faces, such as cross sections or TEM foils, significant rounding of the top edge of the milled face of the sample is observed. This rounding is the contribution from the beam tails to the milling process and is largely unavoidable, although some protection can be afforded by the use of deposited protective layers using GIS.

Illustration of beam tail size measurement using EBSD

Figure 12a shows an SE image of an Si wafer on which a point was exposed to a 30 kV:5 nA beam for 100 ms. The immediate point of exposure is visible on the surface: the red box extending from it shows a region mapped by EBSD using a 1 μm step size. Figure 12b shows resulting EBSD map of band contrast, a measure of the average peak height background ratio derived from the Hough transform of the electron beam scattering pattern (EBSP) acquired at each pixel. The darkest region of the map with the lowest band contrast should be centred about the spot (the slight offset may be a result of drift). This procedure was repeated for other points exposed to the 5 nA beam for lengths of time between 10 μs to 1 s.

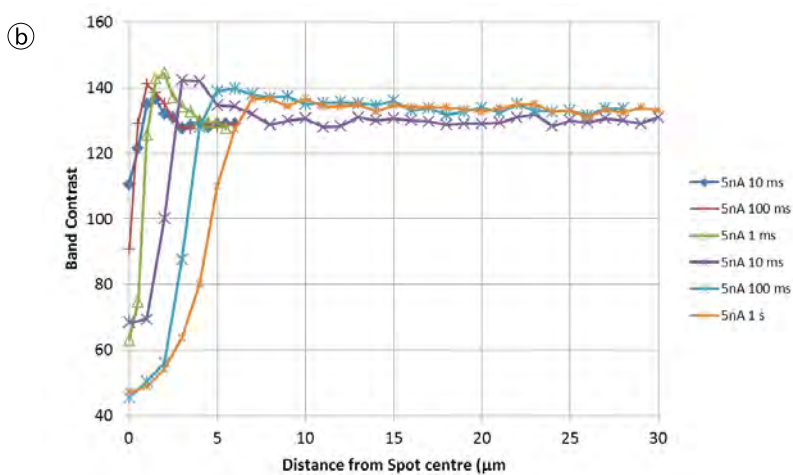
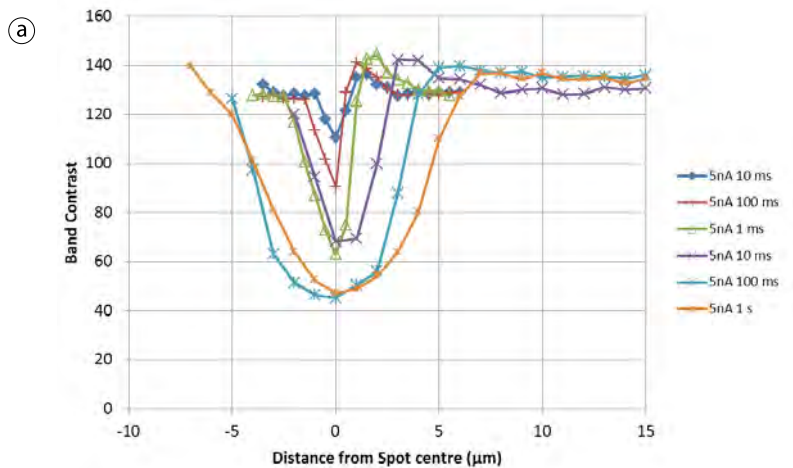
The variation in band contrast as a function of distance from the centre of the spot was analysed by averaging over a strip either side of the centre, in this case three pixels wide, and the results are plotted in Figure 13 for all the 5 nA spot exposure times. Figure 13a shows the variation either side of the spot. In general the band contrast reduced as the exposure time is increased, although not in consistent steps. The trough can be seen to be reasonably symmetrical as it rises from the minimum, but one side of this trough the band contrast rises to a peak before declining to the 'background' value obtained from unexposed Si. The peak is sharp for the short exposure times but for the longer times the peak is lower and declines relatively gradually, seen more clearly in Figure 13b. The distance over which the pattern is affected by the beam tails has been estimated from this decline in band contrast to the background level, although there is some uncertainty in the longest two exposure times because of the very gradual decline. The results of these estimates is shown in Figure 13c, which also includes a measure based on the band slope, a measure of the contrast gradient across the EBSD bands. The band slope variation as a function of distance is shown in Figure 13d; there is more variability in this measure of pattern quality which makes estimates for the shorter exposure times more difficult although the disagreement between the two measures is greatest at the longer exposure times.

Ion milling strategies for 3D imaging

In a Ga^+ ion beam system operating at 30 keV, Ga^+ ions have sufficient energy to sputter atoms from any solid material. As has been discussed in this chapter, all materials have a sputter yield associated with the specific ion species, ion energy, crystallography of the sample and incident angle of the ion. The amount of material sputtered therefore, is a function of these factors multiplied by the number of ions in the beam and the time focussed on the sample. The size of the beam spot, the distribution of ions in the beam spot and beam path over the sample will also affect the milling rate.

When milling a sample, the first consideration is the volume of the material to be removed and secondly what the sputter yield is likely to be. Typically, most materials have atomic densities greater than 1×10^{10} atoms per cubic micrometre and sputter yields typically ranging from one to twenty atoms per ion, where similar numbers of ions will be required to mill a one cubic micrometre volume. As a simple example with assumptions, a 1 pA beam will deliver 6.24×10^6 ions per second, and using Si as an example (atomic density 4.98×10^{10} per μm^3 , sputter yield 2.23), it would take almost an hour at this current to sputter 1 cubic micrometre, whereas if the current is increased to 1 nA the time drops to under four seconds.

However, in practice, the time taken to mill the volume even at 1 nA is greater than the simple calculation suggests because of the beam profile and pattern with



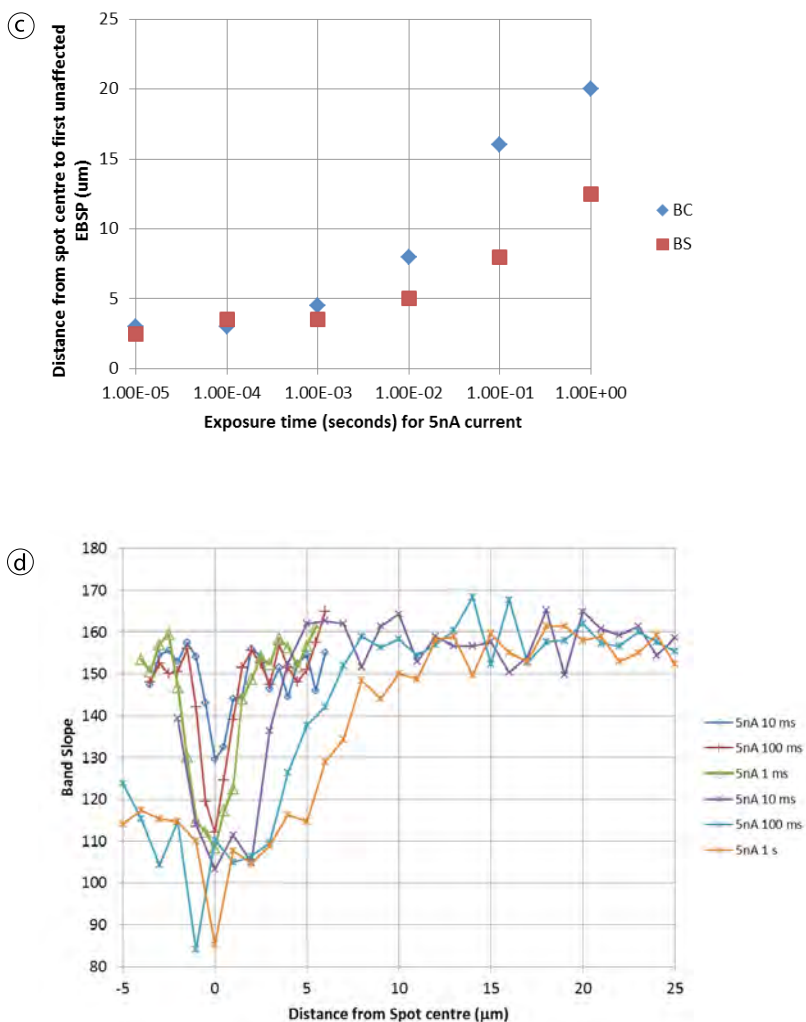


Figure 13: a) EBSP band contrast plotted as a function of distance from the centre of points on Si exposed to a 30 kV:5 nA beam for times between 10 μ s and 1 s. b) The data in a) plotted further from the origin. c) Distance from the beam spot required for EBSP patterns to achieve the background pattern quality, unaffected by beam tails, as determined from measures of band contrast and band slope. d) EBSP band slope as a function of distance from centres of beam spots.

which the beam is stepped across a milled surface. A focused ion beam has a FWHM diameter ranging from a few nanometres up to a few hundred, depending on the chosen beam current. The beam is scanned over the area to be milled with the beam dwelling at each point for a fixed period of time, similar to the electron beam. The step size of each beam movement is typically chosen to be half the diameter of the beam spot resulting in a 50% overlap of the spots.

Usually the beam path is either serpentine or rastered over the area to be milled with even size steps for both the point-to-point and line spacing. This overlap ensures even exposure over the area and should result in equally uniform milling of the exposed area. Furthermore, it is common practice to not attempt to mill the area in a single pass of the beam, usually thousands of passes are used. As the beam sits at one point and material is sputtered, a hole begins to appear beneath the beam spot. As this hole gets deeper so it becomes more difficult for material from the bottom of the hole to escape against the newly arriving ions from the beam. It has been demonstrated that multiple passes of the beam result in a far more predictable and uniform milling rate than only increasing the dwell time, due to this effect.

To summarise, increasing the number of passes increases the sputtered depth linearly, whereas increasing the beam dwell results in diminishing milling rate as the time is increased. Taking into account the need for scanning the beam and multiple passes often many thousands of times, the milling time can only be approximated by using a simple measure of beam current alone, however, it remains a useful initial indicator.

It is desirable to complete the milling in the shortest time by using the highest appropriate beam current. However, if the effect of beam tails causes unacceptably high rounding of the top edge, or damage to the structure revealed, or excessive curtaining is obtained, lower beam currents may be necessary to produce a finer milling geometry or to clean up the milled surface, resulting in longer milling times.

The factors that need to be taken into consideration when milling a material are:

- Material sputter rate
- Volume of material to be removed
- Size of both the largest and smallest feature
- Beam spot sizes and beam step sizes
- Beam dwell time vs passes

Three-dimensional data acquisition

- Preparation for producing image stacks
- Preparation for 3D EDX
- Preparation for 3D EBSD
- Recipes for milling example materials

To collect 3D information of a material, a sequence of sections or slices is milled by the ion beam through the region of interest. Images or maps are taken of the milled surface after each slice using any detector that gives the desired contrast, but it is also possible to acquire elemental maps (EDX) or crystallographic maps (EBSD) in addition to SE images.

Theoretically, SEM, FIB or EDX image stacks can be obtained from anywhere on a sample surface; this is useful since areas of interest can immediately be examined without requiring extensive sample preparation. However in practice, initial sample preparation is still important to ensure the milled surface is clean and the detector view of the surface is not obstructed (particularly for 3D EBSD). The biggest challenges in the preparations are to minimise FIB artefacts, as discussed in Chapter 4, and to prevent redeposited material from building up after milling many slices.

Preparation for producing image stacks

The initial trenches before image stack acquisition are shown in Figure 14, where a trapezium is milled in-front of the section to enlarge the view of the section. Large ion currents can be used to produce these trenches, but the larger the current, the greater the rounding of the corners between the top surface and the trench walls. This consideration is particularly important for preparing the front face; in practice, intermediate currents may be used to produce as plane and damage free a surface as possible before finishing with the current to be used for milling the multiple slices.

Figure 14b shows the sample after the nose has been partly milled, with redeposition left in front of the milled face. Figure 14 is at the same stage in the milling but the corresponding SEM image, with tilt correction so the section face appears perpendicular to the SEM beam.

Note that in Figure 14 no protective layer was used on the top surface since it was important to observe the sub surface image in relation to features on the top surface. However, an ion deposited layer (commonly Pt) is often used to reduce curtaining. No movement of the sample between milling and imaging is required (except for ion imaging). However, as milling progresses, the slice will appear to move up the image unless a software tool which, using the value of the programmed slice thickness, adjusts the electron beam position and focus (working distance tracking) to keep the slice in a fixed position in the image.

Image stacks can also be collected at the edge of the sample, where it is possible to mill to a much greater depth and therefore a larger cross section can be imaged. This is also beneficial because the redeposition is reduced. Figure 15 illustrates this geometry, and the possible milling strategies that can be used to minimise shadowing and allow for the redeposition that does occur.

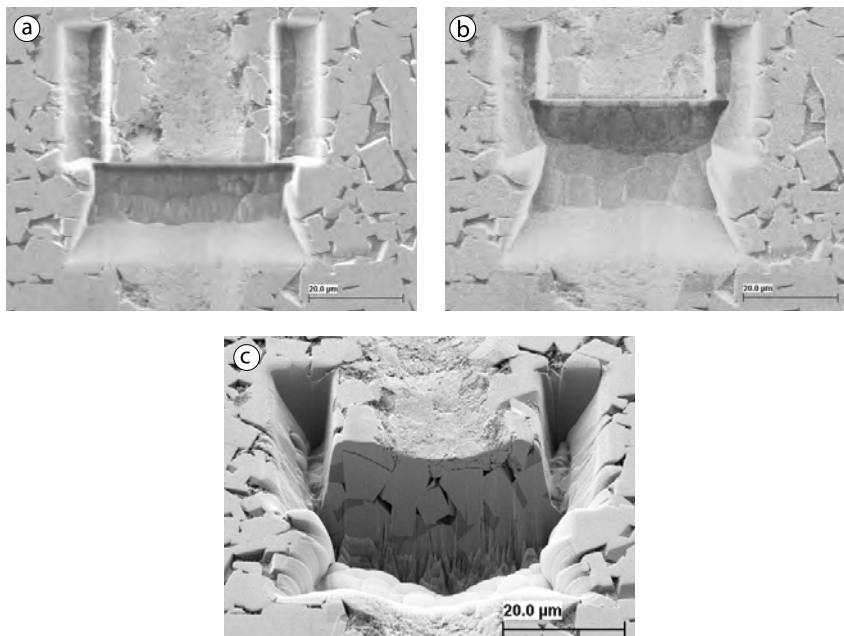


Figure 14: SE images of a cross section through a scratch on a hardmetal. *a)* Top (FIB) view of initial set up, *b)* top (FIB) view of half milled stack, *c)* SEM view of half milled stack.

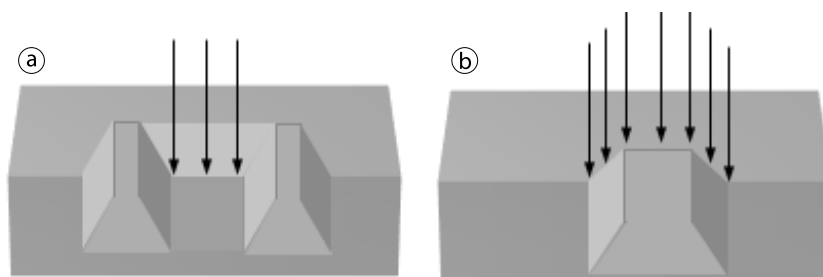


Figure 15: Possible geometries for slice and view, 3D EDX and 3D EBSD at the edge of a sample, where arrows indicate ion beam position. *a)* Cross-section of a nose with small trenches milled either side prior to image acquisition, *b)* cross-section of on a recessed area with wider sloping sides and base milled as part of the image acquisition and milling sequence.

Preparation for 3D EDX

Image stacks combined with EDX mapping can be acquired after preparation by a similar route to that described previously. Without stage movement this off-axis mounting will require a larger side trench to avoid shadowing if the area of interest is in the centre of the sample (Figure 16). For general structural investigations, as opposed to site specific studies, the edge of a sample is easier to prepare and it avoids shadowing altogether (as in Figure 15).

There is a large interaction volume associated with the X-rays that are emitted from the specimen after being hit by the electron beam. This resembles a bulb shape underneath the surface with a penetration depth and width depending on the beam energy. Therefore, at high magnifications, with pixel sizes of the order of 1 μm or less, the composition shown for any pixel will have a contribution from the surrounding material beyond this pixel. This means that there is limited lateral resolution, and on tilted samples this may be reduced further in the direction perpendicular to the tilt axis because the interaction volume is closer to the surface in this direction.

The cross section surface does not need to be perfectly flat after milling, so a slightly larger milling current can be used to save time. However, curtaining can still affect maps if there are defects in the material or phases of very different sputter yields (Figure 10).

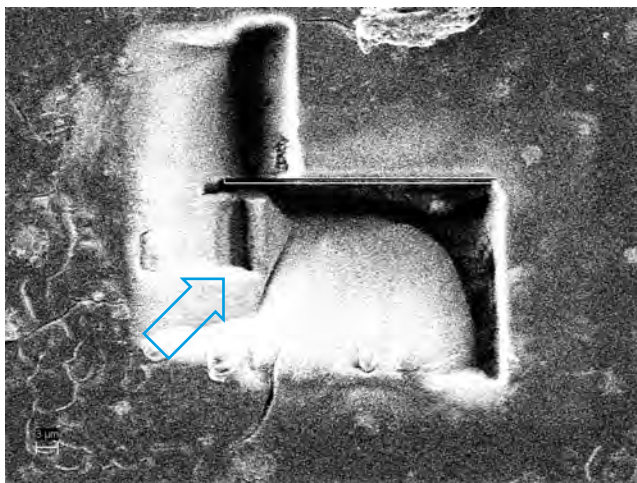


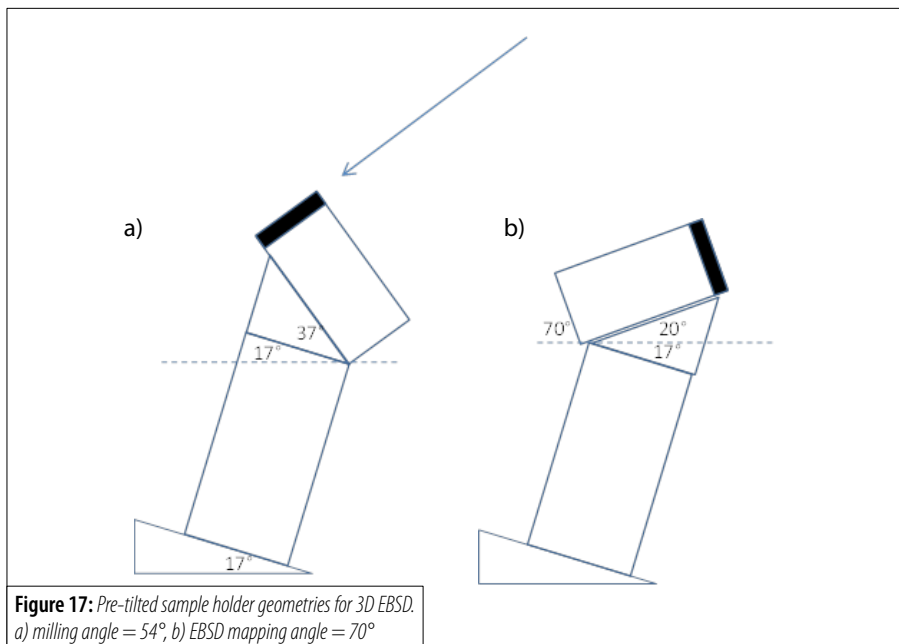
Figure 16: FIB SE view of region to be milled for EDX acquisition when the EDX detector is positioned on the lower left side of the milled region, as indicated by the arrow.

Preparation for 3D EBSD

The geometry of EBSD requires mapping to be carried out on sections prepared at the edge of a sample with side trenches as shown in Figure 15. (Milling at a shallow angle to a surface in the centre of a sample may enable a small region to be mapped but, in practice, shadowing by the lip of unmilled material rapidly reduces this region to a very small fraction of the milled face).

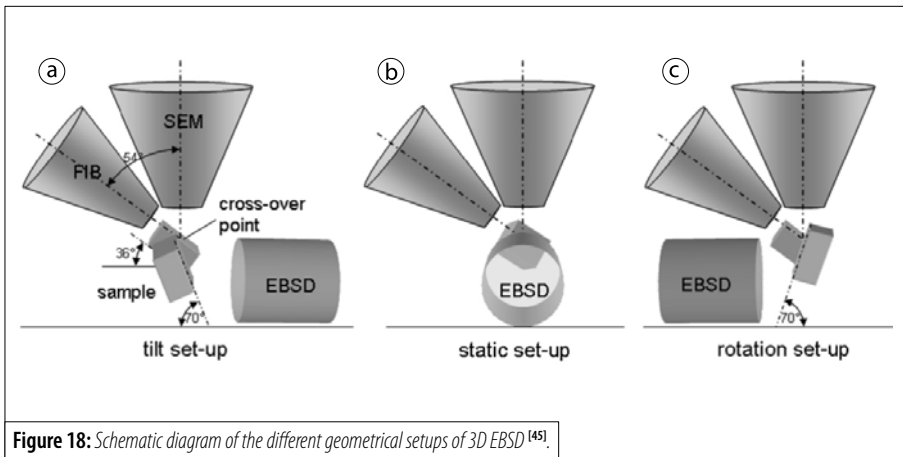
In most cases where the EBSD camera and ion column are positioned on the same side of the electron column, one above the other, the sample needs to be rotated 180° between the positions for milling a slice and acquiring a map. With a pre-tilted holder and a fixed tilt the need for tilting the sample as well can be avoided, which can be an advantage since the tilt axis is usually the least repeatable of an SEM's stage axes. This pre-tilt can be calculated using the angle to which the stage is tilted at the milling position and the 20° angle from the vertical required by the EBSD position as shown in Figure 17.

Two possible pre-tilt angles exist, depending on which of the two perpendicular faces on a sample is to be examined. In each case the sample is tilted to 17° so the sample can rotate 180° without extra stage tilt. After the rotation, the stage will have a small adjustment in X and Y to make and move a few millimetres between the milling



working distance (5 mm) and EBSD optimum working distance (12-14 mm). Absolute positioning of the sample for milling and mapping is then achieved by beam shifts determined by autocorrelation of fiducial marks.

For systems with the EBSD detector mounted on the opposite side of the chamber from that of the FIB, stage tilt instead of rotation between the two positions is required (Figure 18a). Either the sample can be mounted flat on the stage and moved between the milling angle (54°) and 70° , or a pre-tilted holder can also be used to move between them. The pre-tilt angle would typically be the same as the milling angle, so the same holder as in the previous geometry, as in Figure 17, would still work. If the detector is mounted at 90° to the ion column, as in Figure 18b, stage movement can be avoided altogether.



With either geometry, careful attention should be paid to the selection of the correct ion beam parameters to avoid excessive damage to the crystal structure as discussed in Chapter 4.

Recipes for milling example materials

This section gives some general recipes that have been found to work well with the Zeiss Auriga FIB-SEM, but they should be read in conjunction with the notes in Chapter 4 which describes some of the background to ion beam damage and means to avoid it.

Metals

The following has been found to work for slice and view tomography of metals including Cu, Fe, Co, Ni and even WC/Co:

- All can be milled with a 30 kV ion beam, with rough milling of trenches around the Region Of Interest (ROI) at currents up to 30 nA to minimise milling time, although reducing this to ~16 nA will give less rounding of the top edges of the ROI because of smaller beam tails.
- Multiple passes of the beam rather than a single slow sweep of the beam towards the ROI will reduce the amount of build-up of redeposited material in the trenches (which will cause problems of shadowing later during the tomography run).
- The front surface of the ROI to be imaged should be cleaned up with a lower current – typically 30 kV:4 nA and then for the actual slice and view, a 30 kV:2 nA beam will generally give a good result.
- Even for a relatively flat topped surface (such as a fine ground finish or even metallographic polish), a thin coating of ion-deposited Pt will improve the surface finish/minimise curtaining on the imaged surface, as Figure 19 illustrates. Despite this observation, Figure 20 shows that it is still possible to produce a good surface with a 30 kV:2 nA beam on a rough topped material with porosity, in this case in a BiTe alloy.

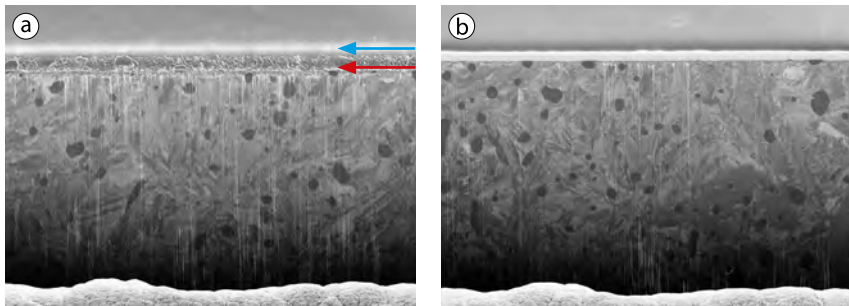


Figure 19: Images of a cross-section through a bearing steel. a) Milled section (30 kV:2 nA) where milling starts on the bare steel surface indicated by the red arrow, just in front of a thin Pt layer (blue arrow). b) Milled section produced in the same sequence as that of a) but milled through the Pt layer, resulting in less curtaining.

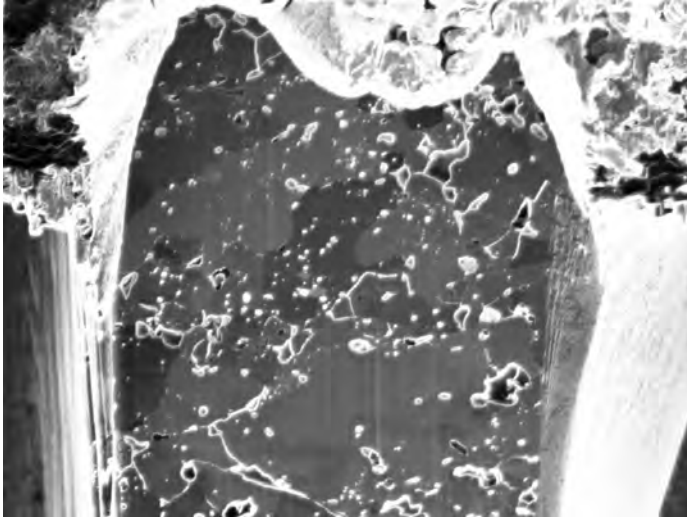


Figure 20: FIB InLens SE image of a surface milled with a 30 kV:2 nA beam on a BiTe alloy sample, showing a good surface finish despite a rough top surface and porosity.

More challenging materials

- a) Ceramics: despite limited conductivity, it is possible to mill many ceramics without additional measures for charge dissipation, as shown in the example of yttria-stabilised zirconia milled with a 2 nA current. Note the surprising effects of improved surface finish by not exposing the surface to the electron beam during milling. Significantly larger currents will however produce very rough edges.
- b) Polycrystalline diamond: can be milled with a 30 kV:600 pA to 1 nA beam, but the injection of water at the milling site greatly increases the milling rate and reduces the tendency for pillar formation.
- c) Polymers: Typically can be milled at 30 kV:240 pA current in conjunction with the electron flood gun operating with a 80 μ A emission current.
- d) Glass: It has been found possible to mill at 30 kV:240 pA to 600 pA with the flood gun (see Figure 21), but deposition of a Pt layer first can help reduce problems with imaging afterwards. Redeposited material charges particularly badly when imaging with the electron beam, so it is worthwhile aiming to minimise build up during milling.

Minimising Drift

A major problem during imaging, particularly with low conductivity materials, is drift. Charging of samples is however not the only possible cause, and other factors such as thermal drift, stage drift or sample drift can all contribute to the problem. Basic good practice for SEM specimens must be followed for all materials, even if of good conductivity, with samples stuck down with a conductive paste that has been allowed to dry fully. If samples have been stuck to an intermediate stub, this must be mechanically secured to the sample holder and SEM stage. Use of adhesive pads should be avoided as they do not generally provide sufficient mechanical stability. Other good practice includes allowing time for the sample and stage to stabilise thermally and mechanically after moving to the milling position, and allowing time for the beam to stabilise.

For low conductivity samples, a number of options exist to minimise problems beyond the obvious use of low electron beam currents and accelerating voltages. Deposition of a conductive coating locally to the milling site using a GIS may help, but to provide a complete conduction path, a micromanipulator probe can be moved to just touch the surface close to the site of interest. For imaging at low accelerating voltages it is also possible in some microscopes to inject nitrogen very close to the sample (i.e. operate in variable pressure mode).

During milling of low conductivity samples, equalisation of the positive ion charge can be achieved by the use of an electron flood gun: a tungsten filament is used to generate a diffuse beam of electrons that covers the milled region, but obviously prevents simultaneous imaging of the milling position. Figure 21 shows an example of the drift that occurs without use of the flood gun and the sharp features that can be obtained if one is used.

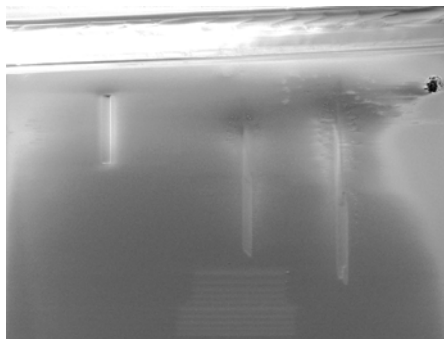
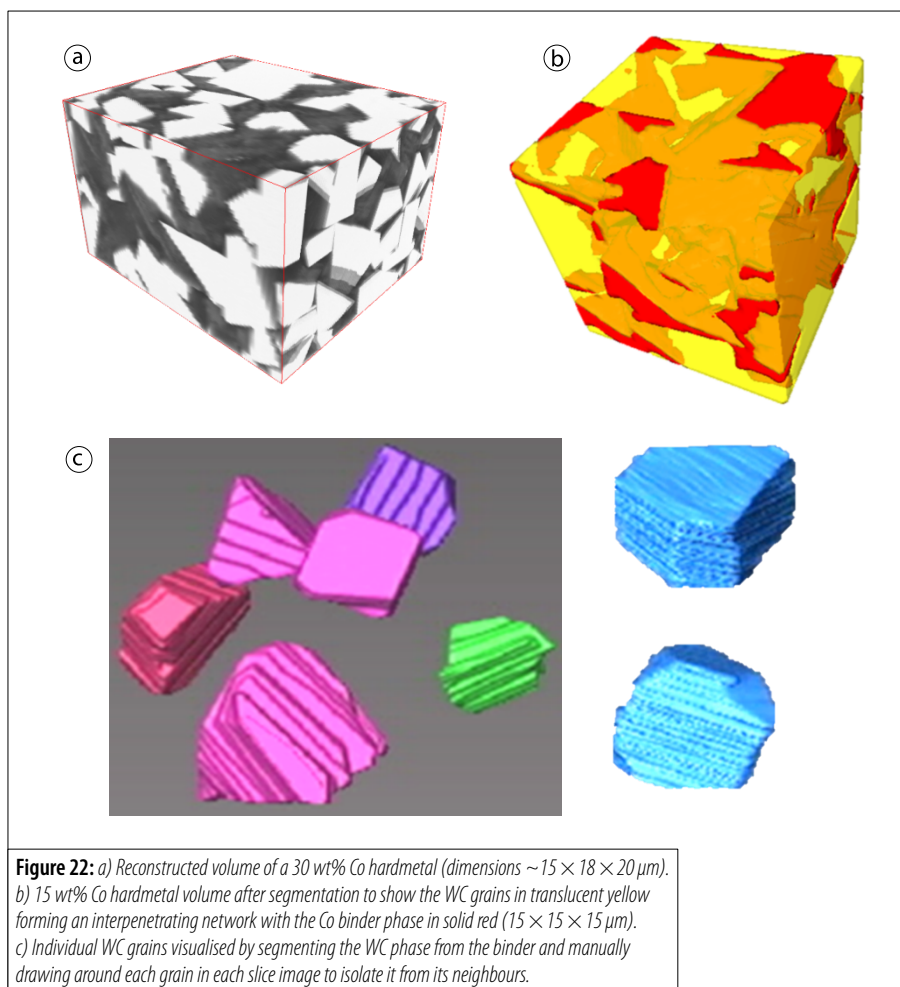


Figure 21: *Milling of a vertical feature on glass with and without the electron flood gun. The clearly defined rectangle on the top left was milled with the flood gun, while the two features on the right should have been of the same dimensions but without the flood gun show significant drift.*

Reconstruction

- 3D volumes from SEM images
- 3D volumes from EDX mapping
- 3D volumes from EBSD mapping

Using the pixel size of the images or step size of the maps with the slice thickness as the voxel depth, the slices can be stacked and viewed as a 3D structure (Figure 22). Segmentation of the data in the stack and 3D volume, using grey or colour values of pixels, allows examination of individual phases or grains within a single phase in 3D (Figure 22b) using thresholding on grey levels for SEM images or chemical or crystallographic data from EDX and EBSD mapping respectively. The segmented data can then be analysed in its reconstructed form by isolating individual grains or phases to gain information about characteristics such as volume, surface area or structure shape.



In many cases the slice thickness is assumed to be constant and either equal to the programmed value or to the average of the total distance milled. This is discussed further in Chapter 7. There are also often slight lateral movements of the imaged area between slices, either as a result of drift of the sample or the beam (charge deflection), or reposition errors if stage movement between milling and imaging is required. Software routines for image alignment can be used to improve slice registration, but different algorithms will produce different results as discussed in Chapter 7.

3D volumes from SEM images

Many phases can be segmented by thresholding SEM images alone based on elemental or orientation contrast, and sometimes even single grains can be distinguished (Figure 22c), but often grain boundaries can only be distinguished automatically from EBSD maps. In samples with damage or wear, it is also possible to reconstruct crack and void shapes within the structure (Figure 23) based on grey level values. Although 3D reconstruction is one of the main and most interesting analysis methods, it can also be useful to represent the image slice data as a sequence as the section is milled back to reveal smaller, enclosed features more clearly.

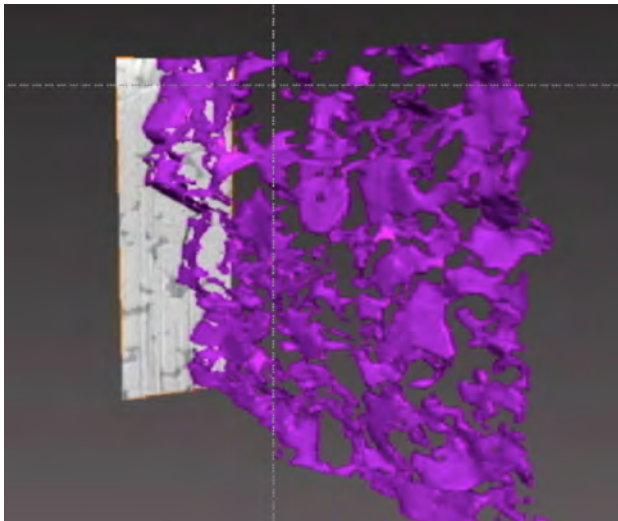


Figure 23: 3D reconstruction of a crack through a 6wt% Co hardmetal (shown in magenta) with SEM image of one of the slices at the end.

SEM imaging can produce high resolution definition of grain and phase boundaries, and low voltage imaging often gives very good contrast (Chapter 3) from a signal within a few nm of the surface and thus is free of any conflicting signals from depths greater than the slice thickness. However, since the chemistry or crystallographic properties cannot be determined from using SEM alone, the additional techniques of EDX and EBSD are often required for more detailed analysis.

3D volumes from EDX mapping

Three dimensional EDX data enables discrimination between phases with different compositions, and although the phases can often be distinguished in SEM images acquired at the same time, the EDX data sets are much easier to segment because they will not be affected by changes in brightness and contrast in the same way that images often are. The data may also distinguish between phases with very similar contrasts in SEM images. EDX mapping can generally be carried out in most places on a sample without stage movement as described in the previous section.

However, due to the interaction volume beneath the surface, the phase boundaries have an uncertainty associated with them that will affect the reconstruction and consequential measurements. The maps are also generally acquired with lower spatial resolution and are likely to be noisier than equivalent SEM images even though they take longer to acquire.

3D volumes from EBSD mapping

EBSD data acquisition usually involves stage movements which can contribute to problems of alignment as well as all of the issues involving mapping previously described. Obtaining orientation data means that grains within a single phase can be defined without ambiguity, which otherwise cannot be detected using EDX and may not give good contrast in SEM imaging. Due to ion beam damage, the patterns can be poor, resulting in poor mapping and ambiguous grain boundaries, as well as losing edge definition. This leads to added uncertainty at boundaries in reconstructions and false edge rounding.

A solution that reduces error in boundary definitions is to combine the imaging and mapping techniques to utilise the benefits of each. This could be to use SEM images along with EBSD maps to retain sharp phase boundaries in SEM with the additional grain boundary information from orientation mapping. The downside of combining these two methods is that the electron column requires a voltage change from 20-30 kV for EBSD mapping and <5 kV for good imaging. This is not an issue when simultaneous EDX and EBSD mapping is run, since both of these methods require the

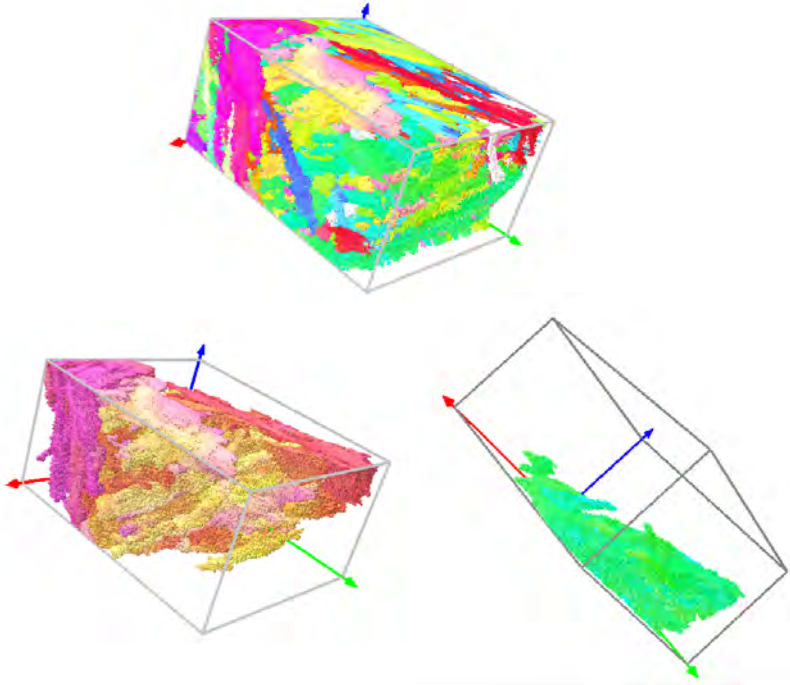


Figure 24: 3D EBSD reconstruction of a martensitic steel, with individual grains separated by orientation from the full dataset.

higher acceleration voltage. However, high resolution phase boundaries are lost and the acquisition time is significantly increased.

Accuracy of 3D reconstructions

- Segmentation errors
- Slice thickness issues

There are potential sources of errors associated with each step of the image acquisition and reconstruction process, and these can all combine to contribute to errors in any measurement made of microstructural features. These potential errors can broadly be split into two categories, shown with some examples below.

Segmentation errors:

- Segmenting images into different regions based on image brightness (grey level) where there is insufficient contrast, too much noise or gradients in image brightness.
- Boundary definition uncertainties for EBSD maps where diffraction pattern indexing is uncertain or there is poor sampling of small volumes.

Slice thickness issues:

- Errors in measurement (or because of lack of measurement and dependence on quoted values) of the actual slice geometry and thickness of material removed by milling across the whole slice.
- Sample or image drift between or during 2D imaging steps even when the sample/stage position is nominally held constant.
- Non-repeatable sample positioning between milling and imaging steps when stage movements are required between these steps.
- Image realignment – the post processing step of repositioning a sequence of images relative to each other, in order to overcome drift and positioning errors, may itself introduce errors.
- Artefacts induced by the ion beam in the images surface, such as phase transformations in metals or physical damage in materials such as plastics.
- Extrapolation errors or conversely (usually for EDX mapping) sampling of more than the slice thickness; these frequently lead to uncertainties in boundary definition mentioned above.

Segmentation errors

Image quality

There are many factors that affect image or map quality, which creates uncertainty and complications in data processing and analysis, in 3D data sets. An important part of image analysis is boundary definition between microstructural features such as phases, grains, precipitates or pores. Even at the highest resolution images, there are still challenges in identifying true boundaries using automatic segmentation software tools, due to complex greyscale structures that can only be truly differentiated by eye. Manual intervention to improve segmentation means there is a compromise between the time taken for data manipulation and the quality of the final dataset and its associated measurements.

Boundary definition

The main cause of poor boundary resolution is a contribution to the signal at a given point (pixel) from neighbouring grains and phases or the underlying structure resulting from overlapping interaction volumes when scanning for EDX and EBSD maps. The interaction volume is bulb-shaped and can be very large for EDX ($\approx 1 \mu\text{m}$) and $> \approx 50 \text{ nm}$ for light elements for EBSD. Since most work using a dual beam requires the sample to be tilted at high angles, this interaction volume becomes tilted relative to the sample surface, since its direction is still in line with the electron beam. This means the resolution is reduced in the Y direction (down the slope) but is typically 3x better in the X direction. The use of EDX and EBSD simultaneously is essential to discriminate between phases with almost identical EBSD patterns as Child ^[24] demonstrates. They show that careful simulation of the X-ray generation, taking into account the tilted geometry involved, is required to ensure a slice thickness is chosen sufficiently large that EBSD data is not conflated with EDX data generated from material below the slice of material removed.

Schaffer ^[26] also showed that porosity can result in X-ray generation from regions below the pore being taken as indicating the presence of material in the pore, and thus careful correlation of EDX maps with the corresponding SEM images are needed in such situations.

Slice thickness issues

Measurement of slice thickness

Although slice thickness is reported in most papers discussing slice and view type reconstructions, it is often as a nominal value ^[19, 20, 24] and few papers discuss measurement of this value.

The slice thickness is set in the microscope software and is obtained by deflecting the beam in increments perpendicular to this face, as it is scanned back and forth across the milled face, until it has been moved by an amount equal to the set thickness. The increments are usually pre-set according to the ion beam current selected, although more sophisticated lithography software can enable variable steps to be chosen by the user.

The repeatability of the slice thickness should therefore be determined by the repeatability of the beam shift and not on the material milling parameters, for a constant ion beam current. Even if the ion beam geometry is not parallel sided, then this should not affect the slice thickness, only the shape of the milled surface and its nearness to perpendicularity to the top surface. If the beam shape is poorly defined then there will be a tendency for a rounding at the top surface. If the beam diameter varies from its nominal value then the dose (i.e. Ga⁺ ions/μm², or nC/μm²) will differ also and this will presumably affect the depth achieved rather than the slice thickness.

Of the few papers which do report measurement of slice thickness Ni ^[25] states slice thicknesses (presumably averages) of e.g.: ~11, 14, 19 and 21 nm for different runs but does not state how these values were determined nor how they relate to the set values. Korte ^[33] reported using a nominal 40 nm slice thickness and subsequently measured average values of 45±22 nm and 47±26 nm; in order to get around the problem of pixel sizes similar to slice thickness, they obtained sub-pixel accuracy with cross-correlation and then centre of gravity matching methods of FIB images to determine these values. This range of about ±50% seems very large, but it is not clear whether this reflects real variation or uncertainty in the measurement of the thickness; this process involved stage movement for EBSD which could contribute additional variation compared with slice and view techniques. More generally, Uchic ^[46] suggests +/-10% for slice thicknesses as a general rule of thumb for all slice reconstruction methods, and also suggests a minimum slice thickness of 10 sections (or slices) per feature.

Measurement of slice thickness could be made for each slice by comparing successive images and adjusting for the tilt of the specimen, measuring the difference in position of the slice/top surface intersection ^[47] or difference in distance between the intersections and a fixed feature on the surface. However, unless the slice thickness is significantly greater than the pixel size of the image, this is unlikely to offer any

improvement in accuracy. Measurement of an average slice thickness over a larger number of slices is possible by this method but obviously will not give slice-to-slice variability.

Constant slice thickness may be a reasonable assumption in some cases but particularly on non-conductive materials such as ceramics, ion or electron charging effects may cause beam drift and thus variable thickness according to the degree of charge build up. Holzer^[27] note this in analysis of BaTiO₃ and compensated for it by the use of drift correction with pattern recognition of reference marks milled either side of the area of interest. Schaffer^[26] comments on this approach for (Ca) MgTiOx, noting that if large beam currents for large slice thicknesses are used (3 nA for 500 nm) (cf. ^[27] 50 pA / 17 nm), then drift correction cannot be applied frequently enough and reference markers may be milled away. They report an astonishingly large variation in slice thickness of between 290 and 600 nm for a nominal value of 500 nm measured from BSE images acquired after each slice. Payraudeau^[28], however, used 3 nA on a sialon ceramic coupled with drift correction, remilling the markers either side of the milled region as necessary, and comment that the marker positioning indicated a constant slice thickness of 100 nm.

If the precise shape or size of an object to be sectioned is known accurately beforehand, it should be possible to calculate backwards from an initial reconstruction to determine the actual slice thickness. This has been applied in biological investigations where cylindrical mitochondria were sectioned along the cylinder axis and the maximum size, measured in a series of slices, was taken to be the diameter and thus used to determine the slice thicknesses from the reconstruction at right angles to the measured dimension. Initially this was used with ultramicrotome sections but later with a FIB reconstruction^[48].

Use of milled fiducial markers on the milled surface itself can provide an alternative way of assessing slice thickness. Gholinia^[49] has shown how reconstructing sequential FIB images of the top (XZ plane) surface in which two lines, previously milled on the top surface to intersect each other at 90° and intersect the milled surface (XY plane) at 45°, showed the lines intersecting at less than 90°, from which it was deduced that the actual slice thickness was greater than that expected, but only by about 5%. Iwai^[16] also adopted this approach but used the markers largely for realignment. A US patent^[50] has even been granted based on this technique to quantify the slice thickness and the method is used in commercial software.

These methods have been adapted to measure the complete slice geometry from individual slice thickness measurements from top to bottom of an artificial structure manufactured in the FIB for this purpose^[51]. Multiple fiducial lines at 45° to the milled surface were milled on multiple ion beam deposited layers, alternating between Pt and an insulator on a Si base (Figure 25).

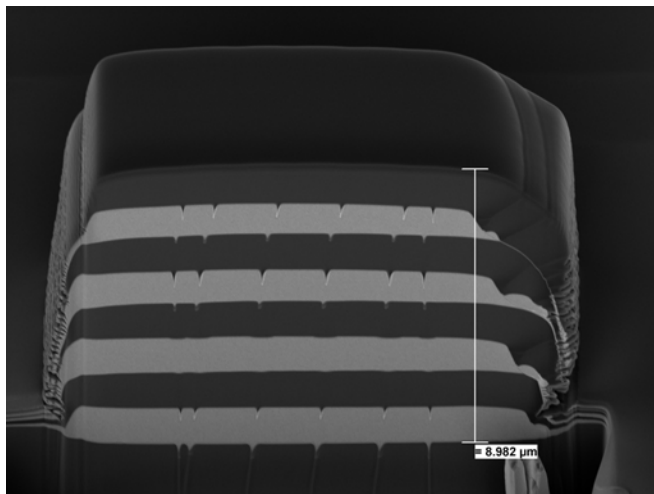


Figure 25: SE image of a silicon substrate with platinum (light contrast) and carbon (dark contrast) deposited with the ion beam on top in alternate layers. On each layer a set of diagonal parallel lines were milled, which are observed in the cross section as ticks between each layer.

This structure, approximately $10 \times 10 \times 10 \mu\text{m}$ in size was then milled with a conventional slice and view milling process. The alternating layers provided good contrast for the fiducial marks and the multiple lines in a given layer permitted measurement at multiple points across the stack to investigate how parallel slices were to each other and quantify measurement error. Straight lines on either side measured lateral drift and avoiding a cross-over of lines eliminated problems at the intersection of fiducial marks. Details of the measurement method are given elsewhere^[51], but the results of milling the stack with a 30 kV:4 nA beam and measuring the slice thickness from the changes in position of the fiducial marks between images are summarised in Figures 26 and 27.

The actual slice thickness can be seen to follow a transient behaviour, starting with thicknesses much smaller than the target of 100 nm, before overshooting and then settling down to oscillate by about $\pm 20\%$ of the target for the majority of the run. The initial under- and then overshoot was much more pronounced at the bottom of the stack, and the data from the different layers can be combined to show the effect of this on the profile of each slice as shown in Figure 27.

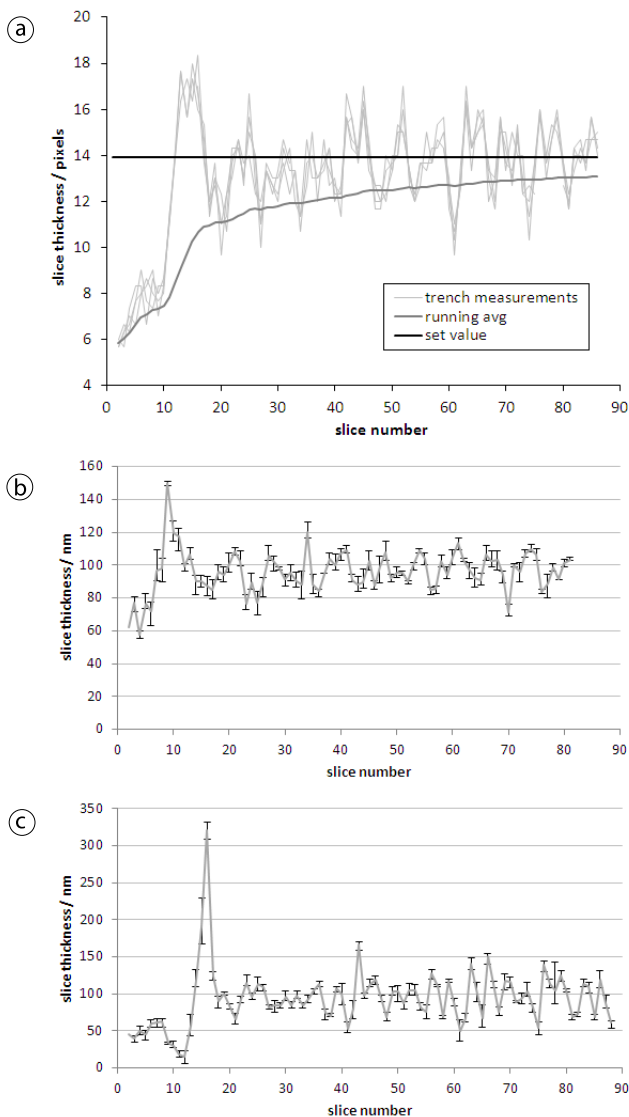


Figure 26: a) Measured slice thickness (in pixels) of slice and view milling through the stack in Figure 24 of each layer, with running average and target slice thickness also shown. b) Measured slice thickness (in nm) of one of the top layer with error bars indicating the variation in measured value across the width of the stack. c) As b), but of the bottom layer (on the substrate).

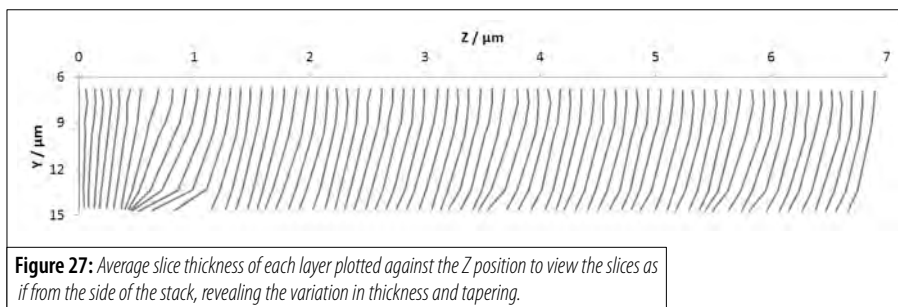


Image and sample drift

Even if the slice thickness is uniform and there are no stage movements during a ‘slice and view’ operation, sample or image drift between imaging steps (during milling) can result in misalignment between slices, and drift during imaging will cause distortion within an image/slice. See Chapter 5 for more about drift.

Drift is particularly a problem for EBSD mapping with its increased acquisition times and beam dwell times, so correlation of EBSD maps with electron images acquired before and after mapping should always be carried out if possible, prior to stacking images in a 3D reconstruction. For example, if there are phase differences that can be identified from contrast differences in a BSE image, these should be correlated with the EBSD map ^[12, 52].

Even without the use of EBSD, correction for sample drift has been noted in some papers where a slice and view method has been used with no stage movement ^[25, 26, 28].

Generally during slice and view or 3D EBSD an automated correction will be made after each slice for the shift in position of the imaged slice caused by removal of the slice thickness ΔT , with a beam shift of $\Delta T \cos \theta$ and a change in working distance of $\Delta T \sin \theta$, where θ is the tilt of the sample from the horizontal. This should maintain the intersection of the milled and top surface at the same point on the screen/image and obviate the need to align the images vertically when forming a 3D stack. The accuracy of this shift will depend upon the accuracy with which the slice thickness is maintained.

Figure 28a shows an example from a 15wt% Co hardmetal, in which prior to slice and view milling, the top surface was milled flat, coated with Pt and then milled with thin shallow lines at 45° to the front surface to produce crossed fiducial lines. Images were taken after milling every 60 nm (nominal/ defined by microscope set up) while the pixels in the XY plane were 32 nm in size. After scaling for these difference sizes and reconstructing the 3D dataset, the section on the XZ plane through the fiducial lines

in Figure 28b shows a number of issues with horizontal image alignment. Firstly, the right hand straight edge shows a lateral drift from left to right for most of the process which causes most of the distortion of the original straight fiducial lines crossing at 45° . Secondly, there is a noticeable drift during the initial stages in the opposite direction to the main drift noted above; this drift period corresponds with a relatively fast image acquisition scan speed (20 s acquisition time) compared with that used later (90 s) for the majority of the run.

In the YZ plane (Figure 28c), the reconstruction just using the slower scan section of the sectioning shows a slight drift downwards of approximately 8 pixels or 250 nm over $\approx 1.25 \mu\text{m}$ from the left hand edge of the section. This agreed with observations made by drawing a horizontal line on the first image (the XY plane) and following the movement of the Pt/hardmetal interface as slices were cut.

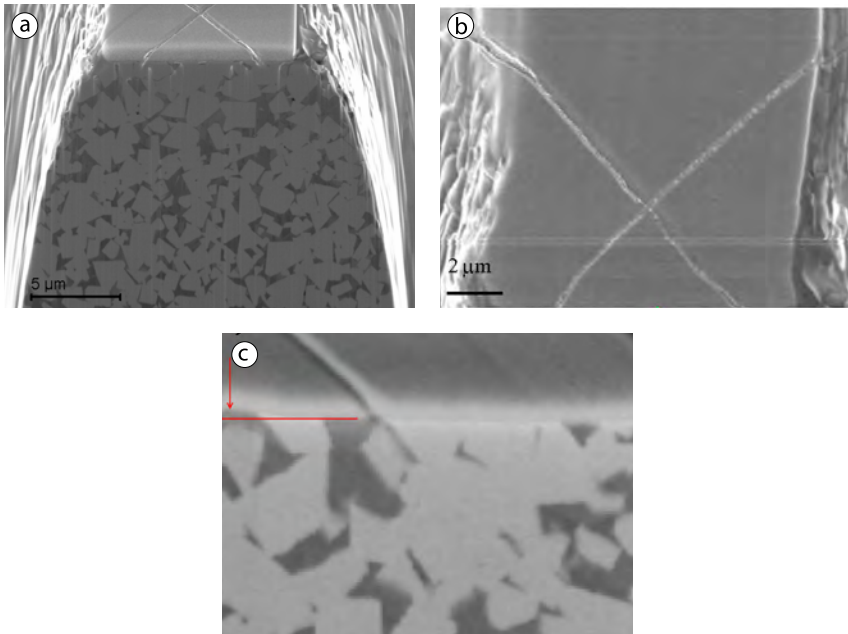


Figure 28: a) SE image of a prepared 15wt% Co hardmetal for slice and view milling, with a Pt layer on top and crossed fiducial marks milled at 45° to the image plane. b) Reconstruction in the XZ plane of the fiducial marks showing deviation from the 45° angles milled. c) Reconstruction in the YZ plane with vertical image drift shown by deviation from the horizontal of the interface between the Pt layer and WC/Co substrate.

Stage movement and realignment

Stage movement between milling and imaging positions has been required in almost all reported 3D EBSD studies. The one exception to this involves positioning the EBSD detector at 90° to the tilt axis, suggested by Day^[53] and recently reported by Gey^[54].

Zaefferer and Wright^[45] summarise the possible geometries for this static set up and for the two geometries requiring movement; either a rotation of 180° to move the milled surface to face the EBSD detector positioned below the FIB column, or a tilt to move to face the EBSD detector on the opposite side of the microscope to the FIB column. Microscope stage accuracy of positioning is usually only between 1 and 3 µm, which on its own is insufficient for the needs of repositioning, but methods outlined below have been developed to overcome this. Korte^[33] specifically measured average misalignments of 0.4 ± 0.3 µm (standard deviation) in the X direction and 2.0 ± 2.3 µm after repositioning without realignment.

Both geometries involving stage movement are widely used and have advantages and disadvantages. Fiducial markers milled adjacent to the imaged area can be used to compare the sample position each time it returns to the milling or EBSD position and then reposition the stage in the X and Y planes with an image correlation method. This does not, however, make any correction for rotation or for changes in Z which are usually also required to cope with the different working distances required for milling and for EBSD. Groeber^[22] reported positioning with fiducial markers to give sub-micron precision, later stated as 0.1 µm^[55] with a rotational accuracy of 0.1°. Zaefferer^[45] suggests 50 nm accuracy of positioning is achievable for images with pixel sizes of 15-20 nm, but this will only be true for imaging with a low current (e.g. 50 pA beam) with a resolution of this order. However, Korte^[33] reported the use of fiducial markers for EBSD but demonstrated significant need for alignment along the Y-axis (about 250 nm on a 5 µm pillar) but only 10s of nm in X. This was calculated from SEM images rather than EBSD, as the former contained more detail up to the edge of the sample.

The tilt accuracy of SEM stages is probably worse than their lateral positioning accuracy and the use of fiducial markers is not possible to improve repositioning accuracy. However, Zaefferer^[21] suggest that inaccuracies in the tilt, perhaps of the order of 1%, can be detected from elongation or contraction of features on the surface adjacent to the milled region and can thus be corrected. They also report that beam positioning and image recognition limits of 3-4 pixels in practice (cf. sub pixel reported elsewhere^[23] limit the practical slice thickness to 100 nm for EBSD). This compares with 25 nm^[15], 17 nm^[27] and 10 nm (NPL) for slice and view with no stage movement. Figure 28 shows an example of the misalignment possible even after use of fiducial markers and realignment. In this case, 50 slices of a nominal 100 nm thickness were milled from a hardmetal. An example of the 2D EBSD maps

acquired on each slice (with a 50 nm step size between pixels on the maps) is shown in Figure 28b, taken from the stack of maps in Figure 29a; the colours show the grain orientation using an inverse pole figure colour scheme. The sample was realigned after each movement between FIB and EBSD position using image correlation with fiducial marks. The section in the YZ plane in Figure 29c shows the result of stacking the EBSD maps; the boundaries between the grains, which should be approximately straight lines similar to those seen in the 2D map are clearly irregular as a result of the repositioning.

The replacement of stepper motors by piezo motors for repositioning may improve repositioning in future, but in general the latter have not been implemented for all axes because of limitations on the amount of travel or speed.

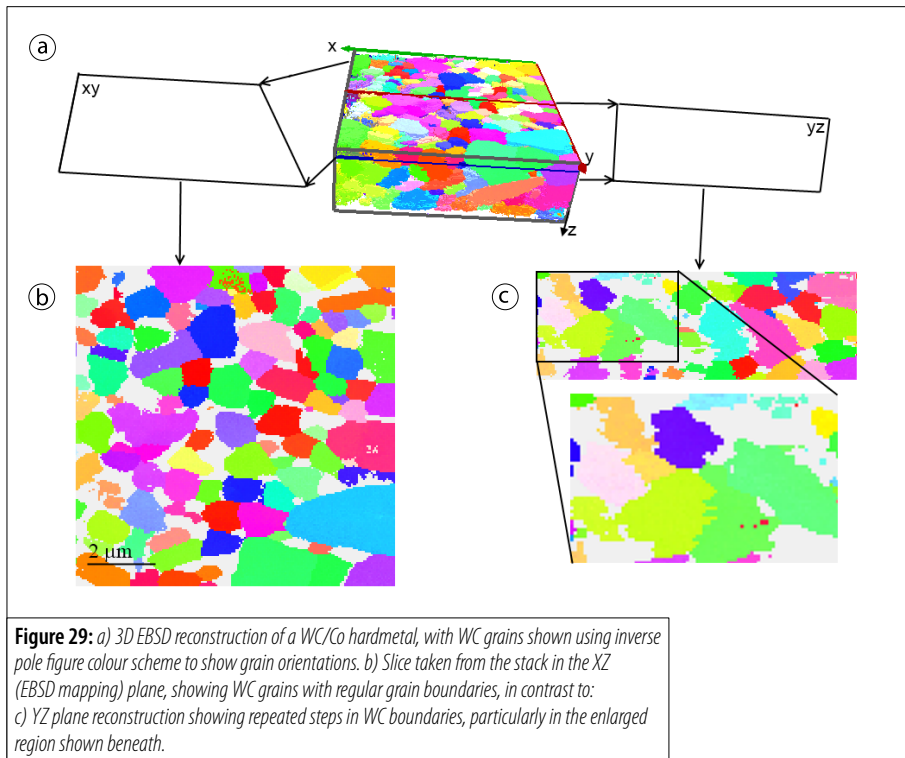


Image realignment

Methods of image realignment have been developed for many tomography applications. For FIB methods most papers report that image realignment is needed, although it is sometimes difficult to separate out descriptions of post-acquisition alignment and image alignment between stage movements. As noted previously, even if stage alignment procedures are carried out, post processing of images will still be needed.

With the exception of Korte^[33] discussed in the previous section, no other paper appears to discuss the degree of realignment needed or how the accuracy of any realignment processes have been verified. Zaefferer^[21] does, however, note imperfections are still visible in the reconstruction of pearlite cells.

Methods of realignment can largely be divided into external or internal feature based. External feature methods use features either outside of the milled region^[25, 26] or the outer shape of the milled region itself^[27], or fiducial marks^[16, 19]. These can suffer from disadvantages such as the need to view a larger area than necessary for the 3D volume itself (and hence with a lower magnification and larger pixel volume than might otherwise be desired), or lack of precise definition of the external milled region itself. Drift and distortion during imaging are also neglected, although Payraudeau^[28] describes how careful choice of imaging conditions can minimise this. The external method does however have simplicity to recommend it.

Realignment of EBSD data sets can utilise the orientation measurements associated with each pixel to aid an internally based approach, and this should in theory improve the accuracy of any realignment. However, it becomes difficult with heavily deformed structures^[45], and more fundamentally, a) an EBSD map may have much greater drift during the acquisition of each image that will distort any alignment process and b) is likely to have a larger pixel size than a conventional SEM image which causes noticeable difficulties with, for example, idiomorphic hardmetal grains^[56]. With the exception of Korte^[33], there appears to have been little if any discussion of the possibility of aligning the single SEM image and corresponding EBSD map for each slice before then aligning the combined datasets.

As an example of the degree of drift correction that can be required even during milling of a conductive material, Figure 30a shows an example section in the XZ plane (from the 'top' of the stack) from the reconstruction of the data set shown previously with the fiducial mark in Figure 28b after applying a rigid translation to the dataset using the Stackreg routine produced by Thevenez^[57] for ImageJ software.

Comparing the total shifts indicated by the vertical red lines in Figure 30a with the opposite shift of the edges of the Pt layer in Figure 28b confirms a reasonable accuracy of the alignment in the XZ plane, both with the noisier initial images and

the later sharper images. However, in the YZ plane there is a shift upwards towards the end of the image stack (Figure 30b) much greater than that justified in the uncorrected image of Figure 28c, and despite the apparent appropriate shifts in the X direction of Figure 30a if the fiducial mark is retained in the realignment, its reconstruction still appears to give a distorted cross, as seen in Figure 30c. Clearly this is just one example of one method of realignment, but it shows the importance of assessing the accuracy of any realignment using known external markers rather than just relying on subjective observation of the microstructure.

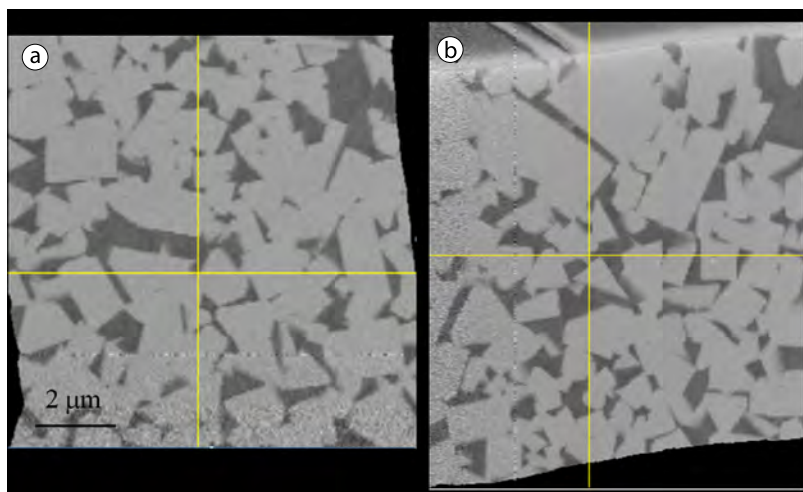


Figure 30: Corrections produced by image realignment assessed by a) Lateral shifts in the edges of the reconstructed volume equal and opposite to the shift in the edges of the Pt layer image seen in Figure 10b, and b) excessive vertical realignment compared with the image shift seen in Figure 10c.

Conclusion

- Overview
- Future developments

Overview

This Good Practice Guide discussed the capabilities of FIB microscopy for materials characterisation in 3D, as well as the associated uncertainties. The literature review and references show examples of FIB-SEM studies elsewhere, at the time of writing, indicating how the uncertainties are being identified and measured.

Depending on the manufacturer, or how a FIB-SEM instrument is predominantly used, different options will be available; this guide focused on the instrumentation available at NPL. These include electron and ion imaging with different detectors (almost all instruments will have similar or equivalent to those described), chemical analysis using EDX and crystallographic analysis using EBSD.

Suggestions for milling conditions on different types of materials were given in order to help users when milling their own materials. This included advice on how to reduce milling artefacts such as charging, amorphisation damage and curtaining.

In 3D routines, where a number of images are collected and reassembled into a volume, the uncertainty in individual slice thicknesses was discussed. It was found that there is very high variance in slice thickness; sometimes no material was milled and sometimes twice as much as the set value. A method to measure the true slice thickness was described for users to follow in order to reproduce the measurement on their own instruments. By measuring the slices directly, less time and data manipulation is required in post-processing.

Image realignment during reconstruction will almost always be necessary for any 3D dataset. In addition to errors made during acquisition, errors in the reconstruction can cause further uncertainties. Even though this is mentioned in the guide, data handling techniques which reduce uncertainty in reconstructions are not described in depth, as it was not investigated fully within the project timescale.

In conclusion, it is clear there are limitations with using FIB-SEM, such as analysis volume size and damage artefacts, meaning that it is not suitable for many materials. However, FIB still remains one of the most powerful techniques for materials characterisation, due to the combination with SEM to image the microstructure at nanometre resolution within a comparably large volume. There are currently related techniques growing in the field that complement FIB-SEM, as well as look to advance the current technology using gallium. Developments in metrology of the existing technology was discussed in the final section of this guide, as well as advances in the instrumentation itself.

Future developments

The FIB-SEM technique is continually evolving and being developed by many institutes all over the world. Following on from the project mentioned at the beginning of this guide, NPL will continue to work on metrological aspects of FIB-SEM data acquisition and processing in materials characterisation. Aims include:

- **Expand slice thickness measurement method to real materials and other types of 3D analysis**

The measurement method for slice thickness using deposition layers and tramlines can be used with any sample to get more accurate slice thickness measurements in real data sets. The uncertainty in slice thickness was high in the static slice and view procedure. The test described in Chapter 7 can be repeated with 3D EBSD experiments, which involves stage movements, to measure the uncertainty in this case as well as comparing it with the static setups.

- **More accurate determination of stage position and movement**

For instruments not already equipped with a piezo stage (as with NPL's microscope) interferometers can be used to measure the stage position and movement. Precisely knowing where the stage is adds another measurement parameter to the data from 3D acquisition routines enabling more accurate alignment during reconstruction. Alternatively, separate piezo stages can be retro-fitted inside the chamber to move between positions during 3D routines, bypassing the use of the stage altogether.

- **Static setup for all 3D methods**

Image alone 3D slice and view routines do not require stage movement between slices because the cross-section can be easily imaged with the electron beam in the same position as required for milling. However, when other types of analysis are combined, this position becomes less ideal or near impossible. There are certain geometries that allow a static setup for EDX and EBSD (e.g. Figure 17) without compromising on data quality. Future developments include exploring these options, which remove the errors produced by stage movements.

- **Expansion of software availability and capability for individual slice input**

Reconstruction software packages are readily available. Most perform at least basic routines for assembling slices, but others are continually being developed for more accurate reconstructions. They include individual slice thickness inputs and more advanced skewing registration routines. The open source package that can analyse orientation data as well as images is known as DREAM3D, available from <http://dream3d.bluequartz.net/> and developed as a result of a collaboration between many institutions (contributors are listed on the website).

- **Exploration of *in situ* temperature control of sample to reduce damage artefacts**

The damage artefacts such as amorphisation, curtaining and transformation are problematic during FIB characterisation. Freezing the sample may be beneficial in reducing these ion-induced artefacts using a cryo-stage intended for *in situ* SEM use. The use of temperature control also enables characterisation of sensitive biological and organic samples that otherwise are not possible to study using FIB-SEM. A better understanding of the damage mechanisms may also be gained by varying the temperature of the sample material and studying its behaviour.

- **Future advances in FIB-SEM instruments**

Most electron columns are close to resolution limit (Transmission Electron Microscopy, or TEM, has atomic resolution). However, even though gallium source FIBs have a similar resolution to SEM so that very fine machining can be achieved, the analysis volume for 3D is limited to $50 \times 50 \times 50 \mu\text{m}^3$ due to the sputter rates using gallium ions. Therefore, the next generation of instrumentation will use different ion species for milling larger volumes at the same time, and can also incur less amorphisation damage in the sample material.

Appendices

- Appendix 1: Ion beam spot size table
- Appendix 2: Sputter yields and damage rates for selected materials
- Appendix 3: Example of the effect of sample tilt
- Appendix 4: Ion dose look-up tables
- Appendix 5: Ion ranges in selected materials

Appendix 1: Ion beam spot size table

Zeiss Auriga 60 Beam spot table														
Probe current (pA) @ 30 keV	1	2	5	10	20	50	120	240	600	1000	2000	4000	16000	20000
Probe size (nm)	2	7	9	13	13	22	25	40	59	59	200	400	900	1100

- Values quoted are provided by Zeiss and are estimated FWHM of the beam spot values, not the maximum extent of the probe size.
- Spot sizes at lower accelerating voltages and alternate probe currents are also available, but in all cases at lower voltages spots sizes are significantly larger for any given probe current when compared to 30 keV.

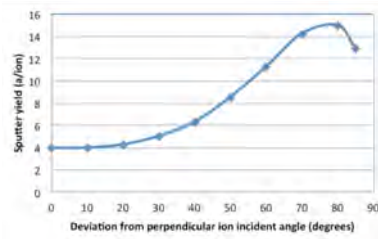
Appendix 2: Sputter yields and damage rates for selected materials

Sputter yields and damage accumulation for selected pure elements					
Z	Name	Density g/cm ³	Atom density a/μm ³	Sputter yield @30 kV	Vacancies/ion
6	C	2.25	1.13 x 10 ¹¹	1.36	317
13	Al	2.70	6.03 x 10 ¹⁰	3.86	487
14	Si	2.32	4.98 x 10 ¹⁰	2.23	746
22	Ti	4.52	5.68 x 10 ¹⁰	2.34	561
23	V	5.96	7.05 x 10 ¹⁰	2.54	540
24	Cr	7.20	8.34 x 10 ¹⁰	5.18	661
26	Fe	7.87	8.48 x 10 ¹⁰	5.97	699
29	Cu	8.92	8.45 x 10 ¹⁰	10.1	786
41	Nb	8.57	5.56 x 10 ¹⁰	3.90	611
42	Mo	10.2	6.41 x 10 ¹⁰	5.30	670
46	Pd	12.0	6.80 x 10 ¹⁰	13.0	736
47	Ag	10.5	5.85 x 10 ¹⁰	12.9	643
74	W	19.3	6.34 x 10 ¹⁰	7.56	698
78	Pt	21.4	6.62 x 10 ¹⁰	12.4	717
79	Au	19.3	5.90 x 10 ¹⁰	17.3	722

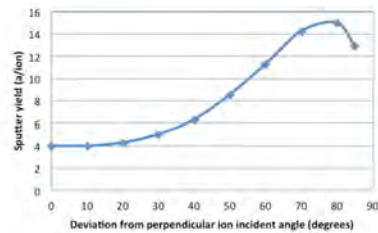
- Atom density is given in atoms per cubic micrometre as this unit is comparable with FIB length scales.
- Sputter yields are shown as sputtered atoms per incident ion.
- All values are calculated using full damage cascades and surface sputtering by SRIM 2011.
- No sample crystallography is assumed, as yields and vacancies will vary with incident angle for individual crystal orientations. These values can be thought of as average values for bulk polycrystalline samples where appropriate.

Appendix 3: Example of the effect of sample tilt

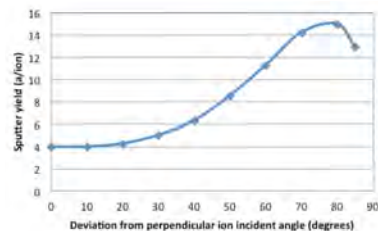
Effect of ion incident angle on sputter yield, vacancy generation and backscattering of Ga^+ ions for Niobium.



Sputter yield for Nb with 30 keV Ga^+ ions as a function of incident angle.



Vacancy generation per ion with 30 keV Ga^+ ions as a function of incident angle. Fraction of incident 30 keV Ga^+ ions backscattered as a function of ion incident angle.



- The examples shown are calculated using SRIM 2011.
- No sample crystallography is assumed as yields and vacancies will vary with incident angle for individual crystal orientations.
- Although only Nb is shown here the trends would be expected to be the same for any element. Alloys and compounds would be expected to show similar behaviour allowing for preferential sputtering of particular elements.
- As the incident angle decreases from perpendicular, although the vacancy generation rate is reducing, the vacancy density near the sample surface will be increasing as the ion interaction depth is also reduced significantly.

Appendix 4: Ion dose look-up tables

1 pA Beam Current (ions/ μm^2)						
x-y beam pitch	5 nm	10 nm	20 nm	50 nm	100 nm	200 nm
Dwell						
100 ns	2.50×10^4	6.24×10^3	1.56×10^3	249.6	62.4	15.6
500 ns	1.25×10^5	3.12×10^4	7.80×10^3	1.25×10^3	312	78.0
1 μs	2.50×10^5	6.24×10^4	1.56×10^4	2.50×10^3	624	156
10 μs	2.50×10^6	6.24×10^5	1.56×10^5	2.50×10^4	6.24×10^3	1.56×10^3
100 μs	2.50×10^7	6.24×10^6	1.56×10^6	2.50×10^5	6.24×10^4	1.56×10^4

20 nA Beam Current (ions/ μm^2)						
x-y beam pitch	5 nm	10 nm	20 nm	50 nm	100 nm	200 nm
Dwell						
100 ns	4.99×10^8	1.25×10^8	3.12×10^7	4.99×10^6	1.25×10^6	3.12×10^5
500 ns	2.50×10^9	6.24×10^8	1.56×10^8	2.50×10^7	6.24×10^6	1.56×10^6
1 μs	4.99×10^9	1.25×10^9	3.12×10^8	4.99×10^7	1.25×10^7	3.12×10^6
10 μs	4.99×10^{10}	1.25×10^{10}	3.12×10^9	4.99×10^8	1.25×10^8	3.12×10^7
100 μs	4.99×10^{11}	1.25×10^{11}	3.12×10^{10}	4.99×10^9	1.25×10^9	3.12×10^8

- These values are calculated for a single pass of the ion beam under the low and high beam current conditions shown.
- To calculate for other beam currents simply multiply the 1 pA values appropriately.
- A single pass of the beam is almost always never used. Simple imaging at fast scan rates will produce up to 40 passes per second, although with correspondingly short beam dwell times. Still ion doses can become very significant for even short exposures.
- When these tables are used with appropriate sputter yields and atomic densities, reasonably approximate ion milling rates can be calculated.

Appendix 5: Ion ranges in selected materials

Examples of Ga ⁺ ion range in solids and ion fluence required for onset of amorphisation			
Z	Name	Ion range (nm)	Onset for amorphisation (ions/μm ²)
6	C	17.6	9.73×10^6
13	Al	22.9	5.16×10^6
14	Si	26.1	4.89×10^6
22	Ti	16.8	3.58×10^6
23	V	12.9	3.47×10^6
24	Cr	10.9	3.37×10^6
26	Fe	10.2	3.21×10^6
29	Cu	9.60	3.01×10^6
41	Nb	11.5	2.56×10^6
42	Mo	9.90	2.53×10^6
46	Pd	9.00	2.43×10^6
47	Ag	10.2	2.41×10^6
74	W	7.30	2.03×10^6
78	Pt	6.80	2.00×10^6
79	Au	7.50	1.99×10^6

- The examples shown are calculated using SRIM 2011.
- No sample crystallography is assumed as range and vacancies will vary with incident angle for individual crystal orientations.
- The values shown are for ions arriving perpendicular to the sample surface. Values will change with change in incident angle.

References

- Glossary
- Contact NPL

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Glossary

4QBSD	four quadrant backscatter detector
BIB	broad ion beam
BSE	backscattered electron
CC	charge compensation - inserts a positively charged gas into the chamber (to alleviate charging when electron imaging)
DIC	digital image correlation
EBSD	electron backscatter diffraction
EBSP	electron beam scattering pattern
EDS/EDX	energy dispersive X-ray spectroscopy
Electron flood gun	inserts a cloud of electrons into the chamber (to alleviate charging during ion milling)
ESB	energy selective backscatter (detector)
ET	Everhart-Thornley (secondary electron detector)
FCC	face centred cubic
FIB	focused ion beam
FIB-SEM	general term used for a dual-beam system with an electron and ion beam
FWHM	full width at half maximum
GIS	gas injection system
HCP	hexagonal close packed
InLens	detector in the electron column for detecting backscattered secondary electrons
LMIS	liquid metal ion source (Ga^+ ions in this case)
ROI	region of interest
SE	secondary electron
SEM	scanning electron microscope
SESI	secondary electron and secondary ion (detector)
SI	secondary ion
Slice and view	serial sectioning method with combined imaging with electrons and milling with ions
TEM	transmission electron microscopy
XCT	X-ray computer tomography
XRD	X-ray diffraction

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The Use of Focused Ion Beam Microscopy for 3D Material Characterisation

Good Practice Guide No. 141

What is it about?

A Focused Ion Beam Scanning Electron Microscope (FIB-SEM) combines the column of a Scanning Electron Microscope (SEM) and a Focused Ion Beam (FIB) microscope in one instrument to enable both imaging with electrons and imaging and milling (local removal of material) with a beam of positively charged ions.

Who is it for?

This guide is most suited for users with existing SEM knowledge and new to FIB. Also, those who are working to improve their results, typically Masters/PhD students or users new to the field.

What is its purpose?

The purpose of this guide is to assist users to better understand the capabilities of a FIB-SEM for acquiring two and three dimensional microstructural information for most samples.

It aims to help the user to optimise the conditions for conducting experiments and the most suitable way to represent and analyse their data and assess the accuracy of this data. The guide does not intend to discuss in any great detail the other widespread uses for which the FIB-SEM is often used, such as preparation of samples for transmission electron microscopy, atom probe microscopy, or micromachining micro-devices. Nevertheless, the methods discussed for reducing ion damage and measuring uncertainties in milled materials can be applied to these wider capabilities.

What is the prerequisite knowledge?

Readers should be trained users of scanning electron microscopes and focused ion beam dual-beam system (SEM-only users can still use relevant parts of the guide as needed). Readers should also be capable users of and have access to image analysis and reconstruction software.

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