MYSCOPE
MICROSCOPY TRAINING

Focused Ion Beam

Train for advanced research

Welcome

MyScope was developed by Microscopy Australia to provide an online learning environment for those who want to learn about microscopy. The platform provides insights into the fundamental science behind different microscopes, explores what can and cannot be measured by different systems and provides a realistic operating experience on high end microscopes.

We sincerely hope you find our website: www.myscope.training an enjoyable environment. In there you can explore the microscopy space and leave ready to undertake your own exciting experiments.

Module Contents

Credits

What is FIB? - FIB overview

As the name suggests, a focused ion beam (FIB) is a stream of energetic ions that are focused into a fine beam. The FIB instrument is based on scanning this highly energetic ion beam onto a target material. The interactions of the ions with the specimen can lead to sample atom removal with nanometre precision, making it into a nanoscale machining device. The fully controlled ion beam, combined with a high precision sample navigation system, along with advanced signal detectors in the FIB, creates a multifunctional analytical platform for imaging, sputtering and micro-fabrication.

Mass production of microelectronics in the 1970s required a specialised analytical tool to precisely locate invisible faults on the top surfaces of Si wafer semiconducting devices. This has provided the driving force for commercialising the FIB and laid the foundation for the design and manufacture of this type of instrument. Regardless of the application, the FIB is simply removing material to a high depth and spatial accuracy. In addition to the ion beam, most FIB instruments have an electron beam just like in an SEM, thus we refer to them here as FIB-SEM instruments.
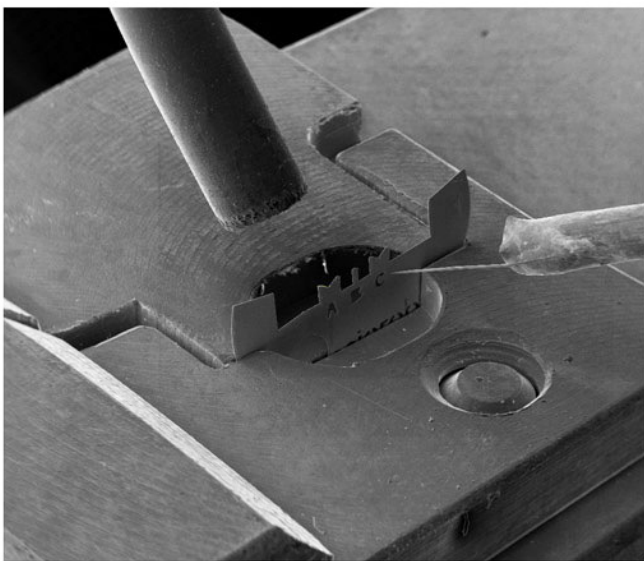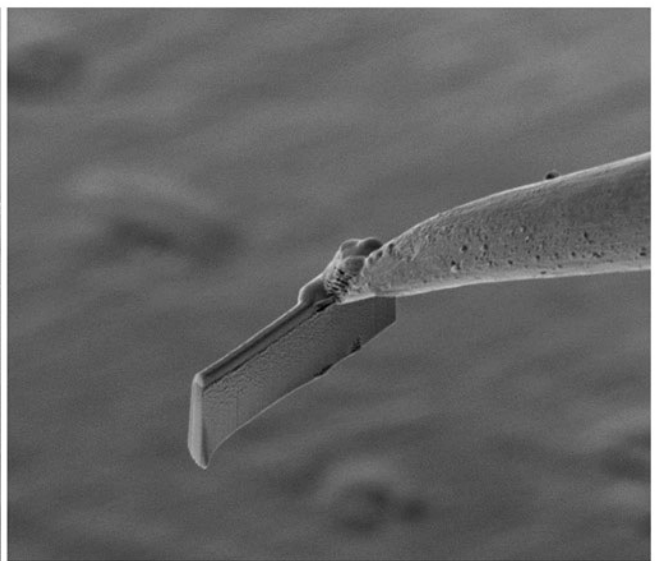
Common applications of FIB

The development of the FIB technique was driven by the needs of a burgeoning semiconductor industry, and then rapidly spread over many academic areas including advanced materials science and engineering, mechanical engineering, electrical and communication engineering, new energy, environmental engineering, geological science, mineral and petroleum engineering, medical and biological engineering. The development of Cryo-FIB promises to expand the application of FIB into biomaterial characterisation.

Focused ion beam systems are used for a broad range of applications, including but not limited to the following examples:

Micro-machining
FIBs are able to structure a wide range of patterns from the nanometre to micrometer scale. Examples include arrays of nanopores, plasmonic devices, microfluidic channels and complex 2D and 3D structures. You will see that you are only limited by your imagination! The FIB can also be used for applying grids and patterns to surfaces, and also to place fiducial markers on specific locations.
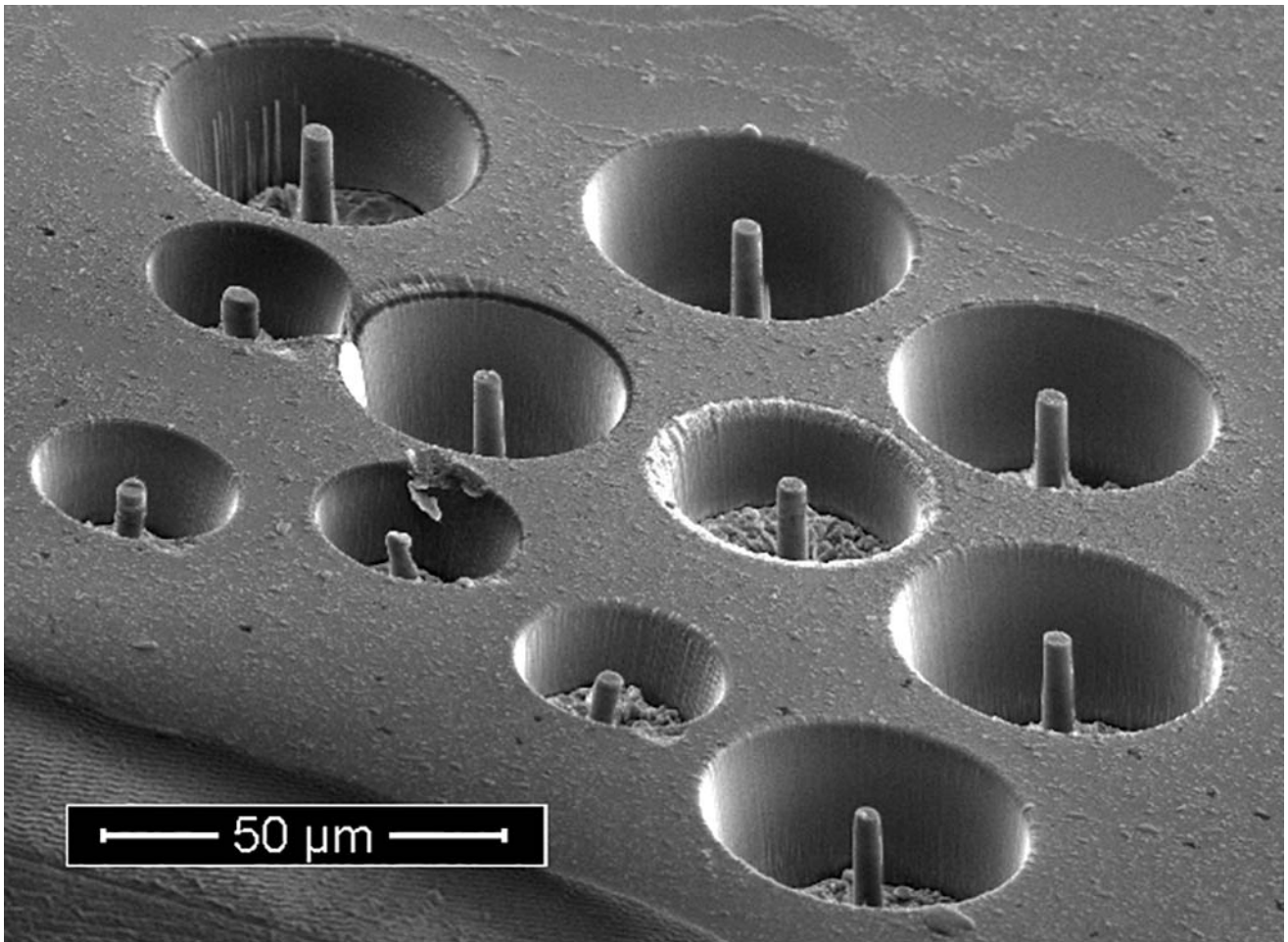
Image shows micro-pillars that have been made using a FIB. Image courtesy of Animesh Basak, Adelaide Microscopy.

Cross-sectioning
Features that are hidden underneath the sample surface can be exposed and analysed using cross-sectional analysis. The advantage over conventional cross-sectional analysis is that the area of interest (including nano-sized features) can be selected with nanometre precision. This high precision is impossible with any other current technique.
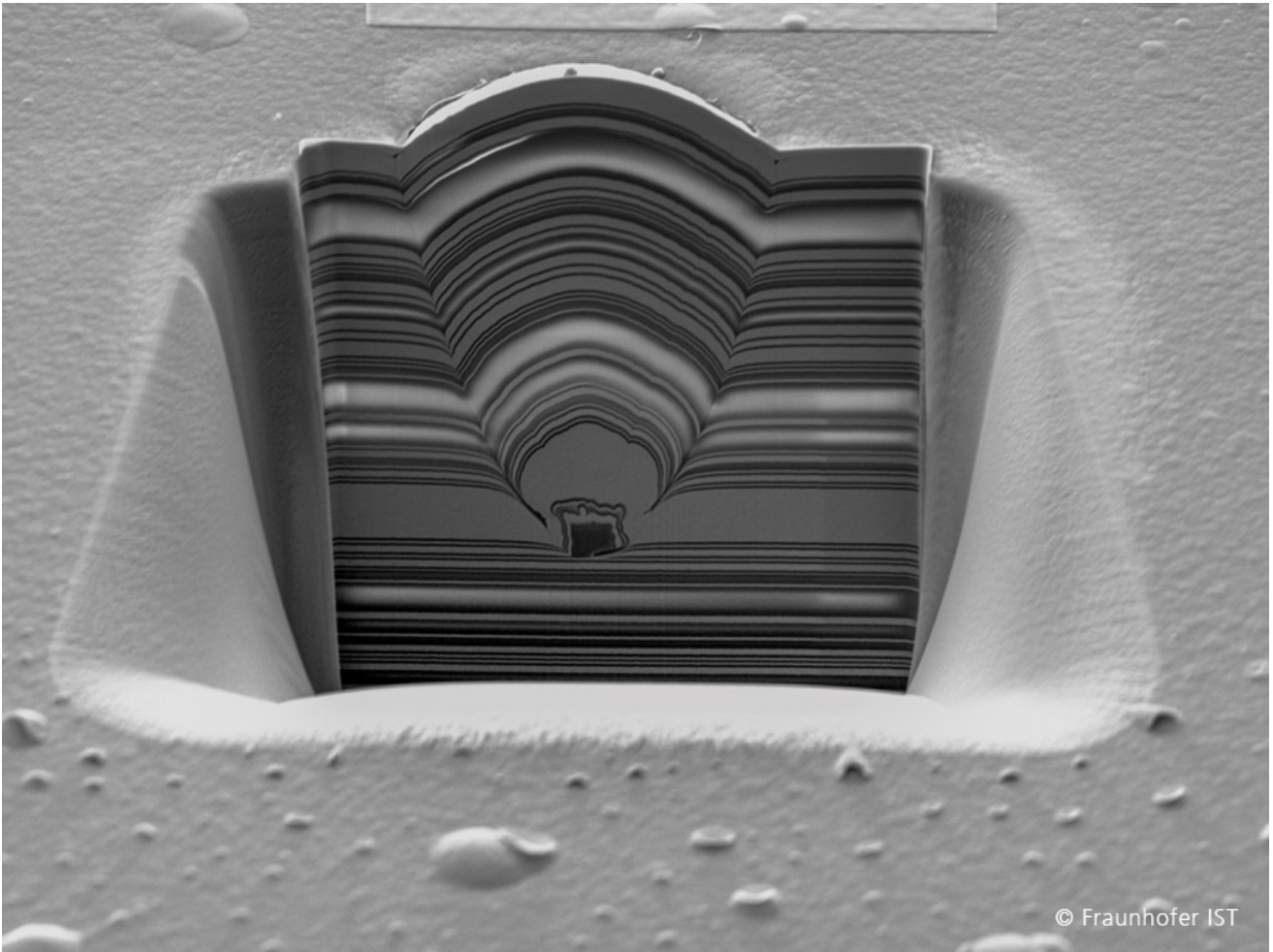
This image shows a cross section of a multi-layer film specimen made using a FIB. The cross section has revealed that under the surface there was a particle with a diameter of approximately 2 μm that was overgrown during the coating process. Image courtesy Dr Kirsten Schiffmann, Fraunhofer IST.

3D reconstruction

Samples can be sectioned and the acquired images/stacks can be used to reconstruct a 3D-model of the sample. The example here shows a magnetotactic bacterium.

Milling slices

Aquire images

2D image stack

3D reconstruction
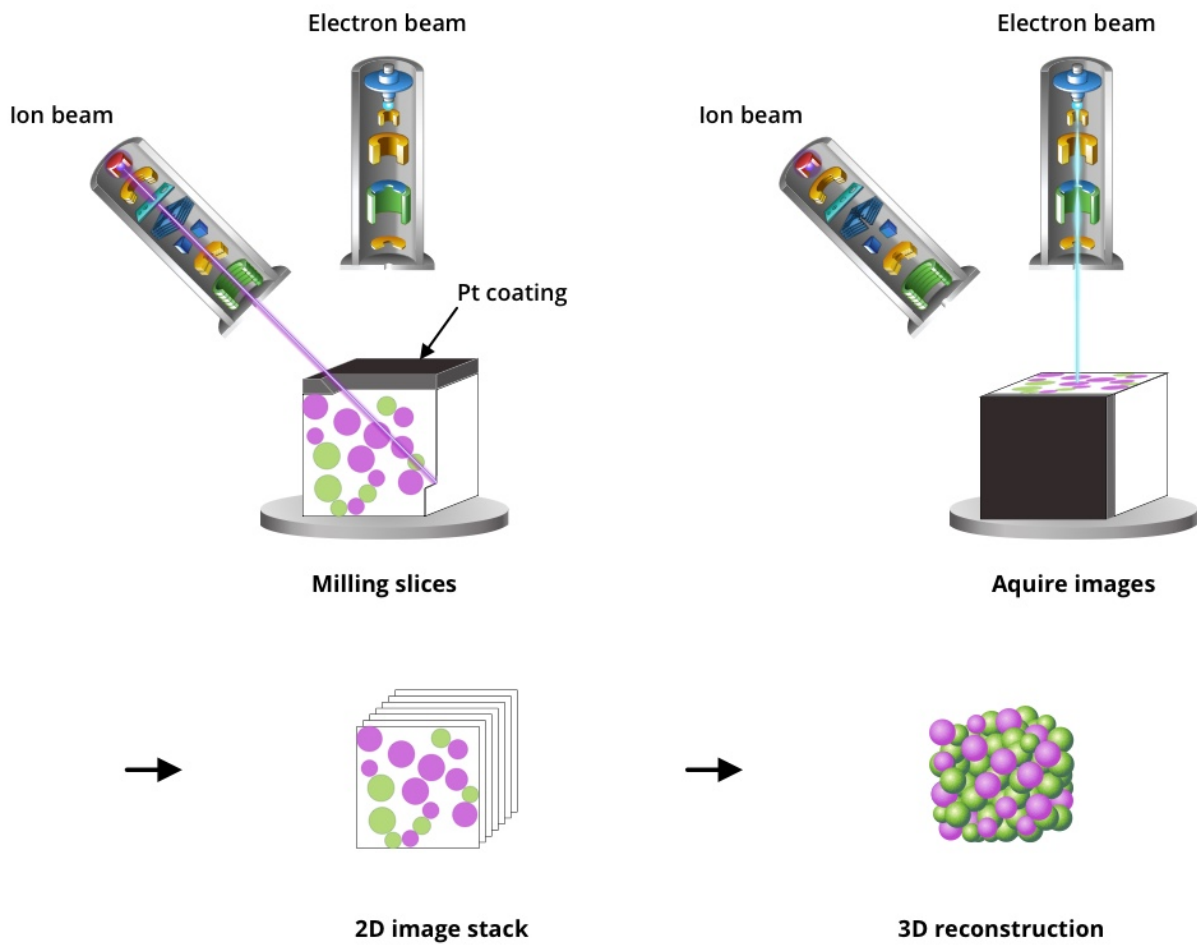
Site specific TEM sample preparation

One of the most common techniques used for the FIB-SEM is TEM sample preparation. The FIB-SEM is an ideal tool for this purpose because it allows you to choose a specific area on a specimen, such as an interface or particle, and then prepare a TEM section of this feature with sub-100 nm thickness.

mag ☐	curr	WD	tilt	HFW	⊢————— 30 µm —————⊣
3 000 x	27 pA	16.5 mm	0 °	85.3 µm	Helios D433
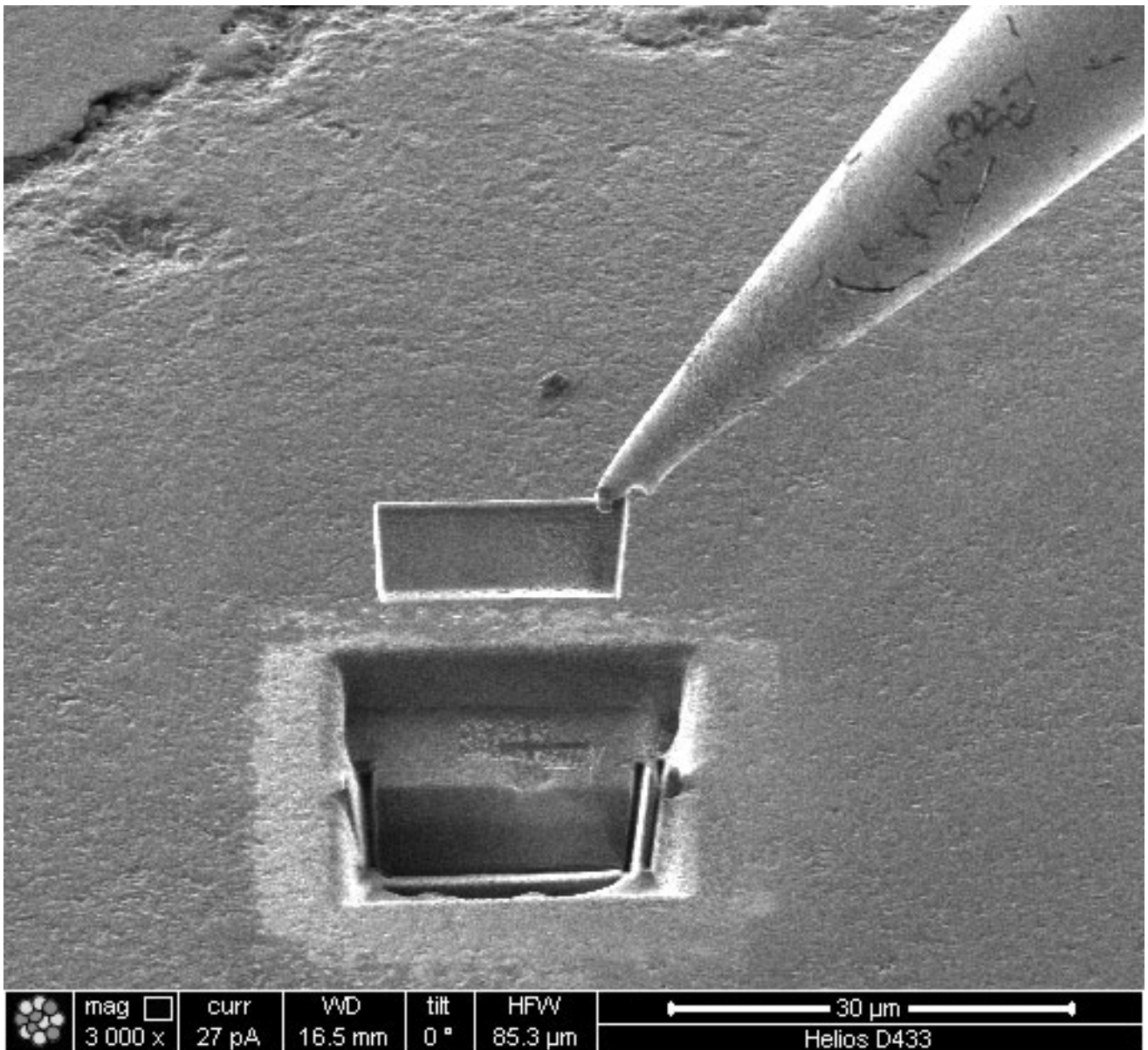
Atom probe tomography (APT) sample preparation

FIBs are used to prepare APT samples at precisely selected locations. The overall process is similar to TEM sample preparation. Whereas in the case of TEM samples, we are looking to make an electron transparent foil, for APT we need to make a sharp needle. The needle typically have a length of ~15µm and a tip diameter of less than 100 nm.

Image reproduced from: Roussel, M., Talbot, E., Gourbilleau, F. et al. Atomic characterization of Si nanoclusters embedded in SiO2 by atom probe tomography. Nanoscale Res Lett 6, 164 (2011). https://doi.org/10.1186/1556-276X-6-164
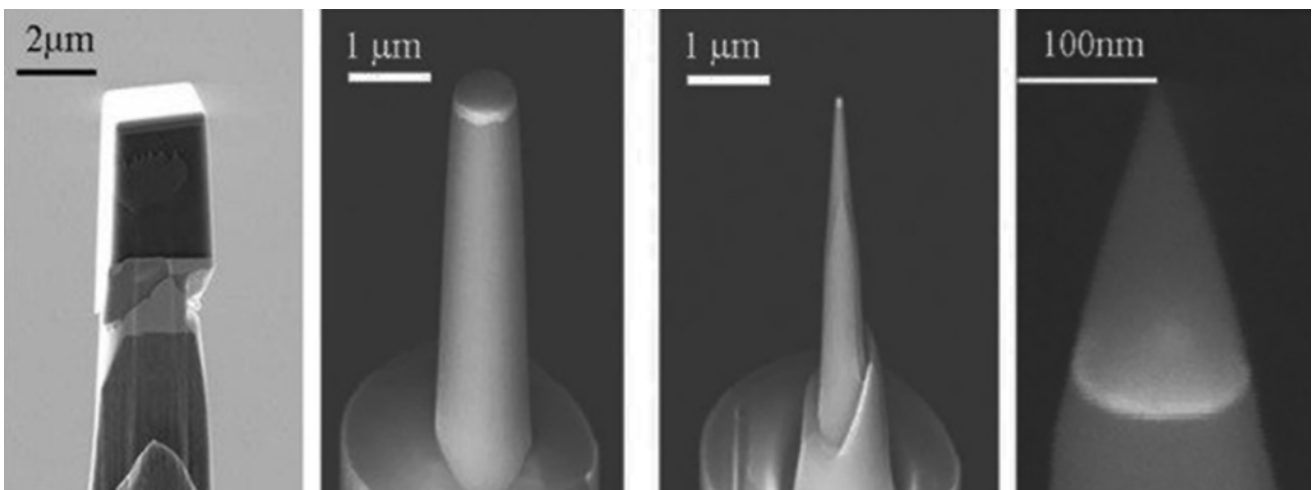
Analysis

FIBs allow researchers to analyse their samples. FIB-SEMs are conventionally equipped with a wide range of detectors such as backscattered electron detectors (BSE), in-lens detectors, electron backscattered diffraction (EBSD), and energy dispersive spectroscopy (EDS) detectors. These allow many kinds of sample analyses on the sample surface, in cross-section, or in a 3-dimensional volume through serial sectioning. In most cases, the ion beam is used for cutting or slicing, and the electron beam is used for analysis using the same tools and detectors as in a conventional SEM. Secondary ion mass spectrometry (SIMS) have been recently introduced into the FIB platform to compliment the analytical capabilities.
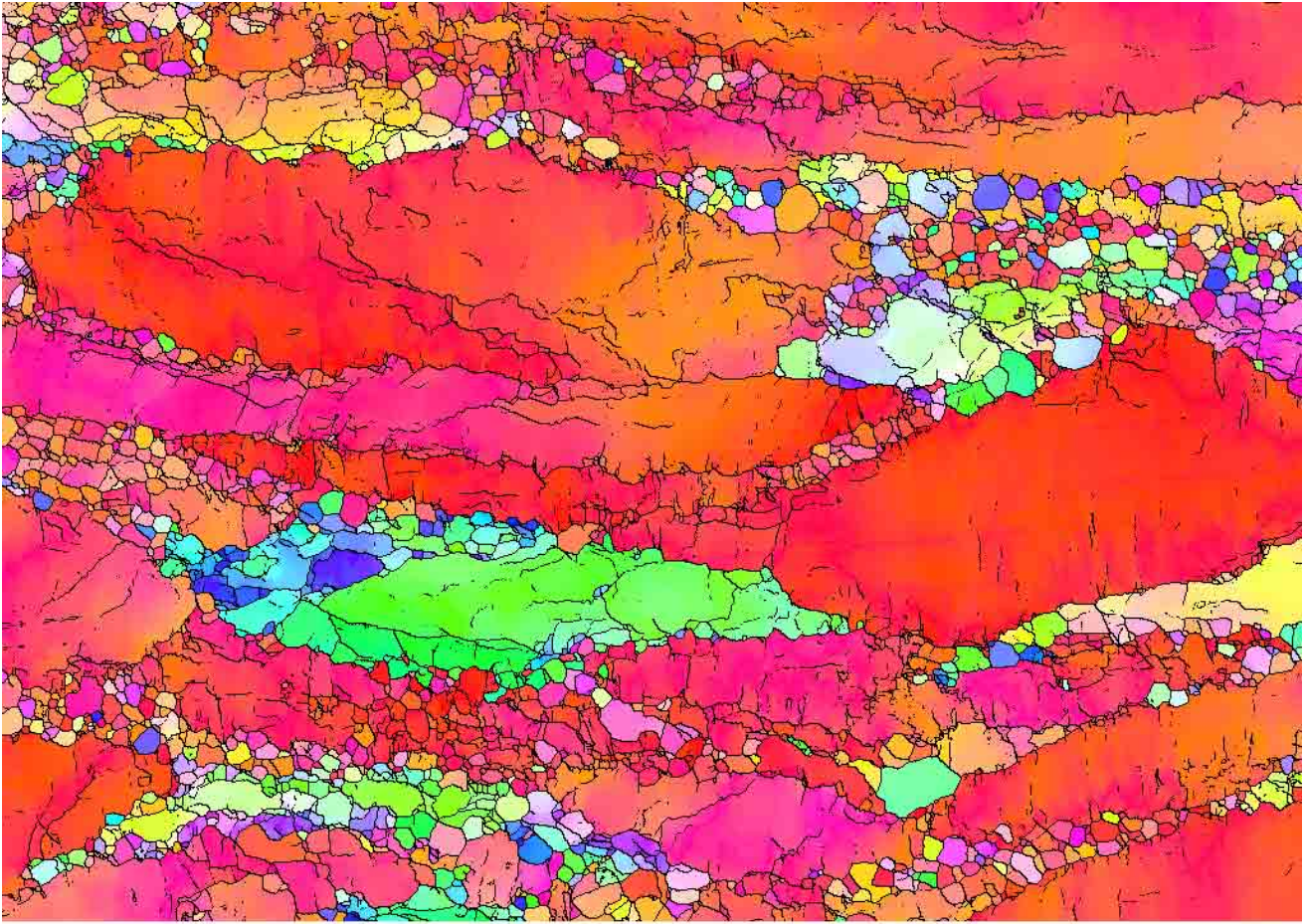
50 μm

Image shows electron backscattered diffraction (EBSD) pattern of hot compressed magnesium measured on a FIB, demonstrating the diversity of the dual beam platform. For more information on EBSD, please refer to the SEM module of MyScope.

Ion sources

There are many different ion sources used in FIB instruments. FIB columns typically employ a Liquid Metal Ion Source (LMIS), with the Ga+ ion source being the most common in microscopy applications. These instruments generally operate with energy ranges from 500 eV to 30 keV, beam currents from 1 pA to 65 nA, and image resolutions of ~ 5 nm are achievable. Other kinds of ion beam include: gas field ion sources (GFIS), such as He and Ne; inductively coupled plasma ion source using noble gases, such as Xe; Low Temperature Ion Source (LoTIS); magneto-optical trap ion source (MoTIs) and Liquid Metal Alloy Ion Sources (LMAIS).

The main developments in ion source technology are driven by the need for faster milling times and reduced specimen damage. For this reason, the plasma FIB technology is becoming increasingly common. These instruments typically use a Xenon source and have the ability to mill much more quickly than the Ga+ ion systems. One of the most recent developments is the incorporation of a femtosecond laser into the ion milling system, which increases the milling rate by up to one hundred times.

For high imaging resolution and reduced specimen damage, multi-beam FIB systems which have an additional ion source are utilised. These instruments, such as the helium ion microscope (HIM), provide a flexible platform for both high resolution ion beam imaging and nano-fabrication. The use of these different ion species extends the analytical range of FIB down to the nanometre level.

Ion–Solid Interactions -
Introduction to ion solid interactions

In order to effectively use a FIB, it is important to understand the physical features of the ion beam, and the interaction of the ion beam with the specimen. The following sections summarise the parameters that define the ion beam such as dose rate and energy, as well as the rather complex interactions that the ion beam can have with the specimen.

How many ions?

One of the most important instrument settings on the FIB-SEM is the ion beam current. The basic equations used to describe the number of ions in the beam are given below.

Number of Ions
The number of ions (N) that can interact with the sample during the time (t) can be calculated via:

$$N = \frac{I * t}{C}$$

Where N = number of ions
I = current in ampheres
t = time in seconds
C = charge

Ion Dose
We note here that there is a difference in the term "fluence" and the term "dose". The fluence is the number of ions that pass through an area prior to sample interaction. The dose is the number of ions that have travelled into the sample surface.
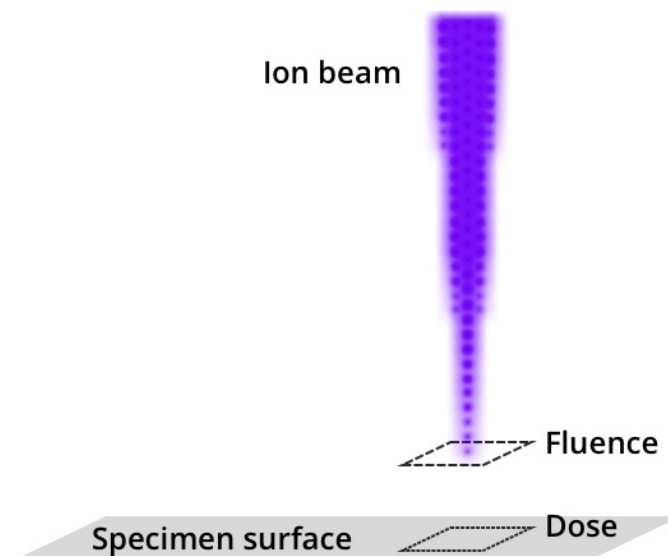
The ion dose is defined as the number of ions that travel through a specific surface area into the sample. Dose can be calculated via:

$$Dose = \frac{N}{A}$$

Where N = number of ions
A = area in square centimeters

Ion Dose Rate

The ion dose rate is often referred to as the Flux.

The dose rate describes the number of ions that are going through a specific area into the sample per unit of time:

$$Dose\ rate = \frac{N}{A*t}$$

Where N = number of ions
A = area in square centimeters
t = time in seconds

Interaction Overview

Several different interaction types occur when a beam ion interacts with the sample atom. The different possible interactions are shown in the illustration below.

The ion-solid interactions lead to emission of a variety of secondary particles such as secondary electrons and secondary ions (1), which can be detected using different detectors and are used for recording images. Photons are so rarely produced that they are neglected. In addition, sample surface atoms can be removed in a process called sputtering (2). This process is used to cut structures, features and cross sections into the substrate. Throughout the interactions, the sample is modified by the ion beam as the ion-solid interactions create vacancies, dislocations and interstitials (3) within the sample as well as polymerisations (4). The interactions take place until the ion has lost all its energy and is implanted in the sample (5) at a specific depth, often referred to as the projected range.
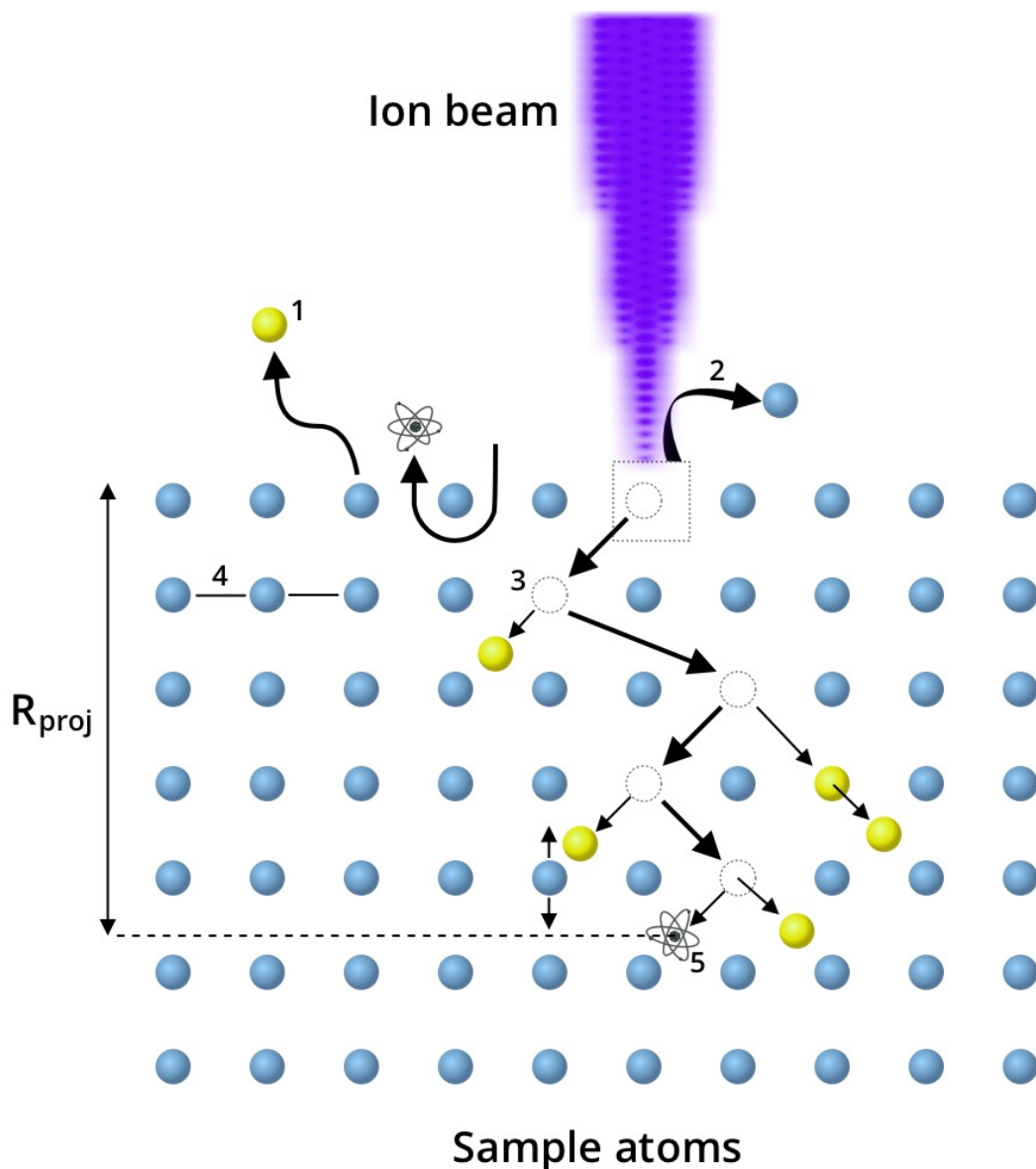
Dislocation/Displacements

A displacement occurs if the incoming ion transfers enough energy to the sample atom (the recoil energy is larger than the displacement energy) to remove the atom from its position in the lattice. The struck atom can either knock out another sample atom (and take its place) which is called replacement collision, or it can move into an interstitial site or jump back into its old position (anneal) or create a vacancy (if the original lattice site remains empty after the collision). Sputtering is a special case: a sample atom is removed from the sample surface. A single ion can dislocate several sample atoms. These recoiling atoms can then also dislocate further sample atoms, leading to a cascade.

Ion Implantation

Ga$^+$ ions come to rest in the sample after losing all their energy in the ion–sample interaction. The average depth at which the ions come to rest is called the projected range R_{proj}. This means that the beam ions implant in your sample. In the case of gallium, this can lead to changes in the physical properties of your sample.

Consideration needs to be made to the effect of parts-per-million implantation of gallium into the specimen. Gallium or Xenon implantation could potentially modify the crystal structure, grain boundary chemistry or some other characteristics of the specimen. This should be taken into consideration when deciding to use FIB, and should also be kept in mind during data analysis of specimens that have been prepared by FIB.
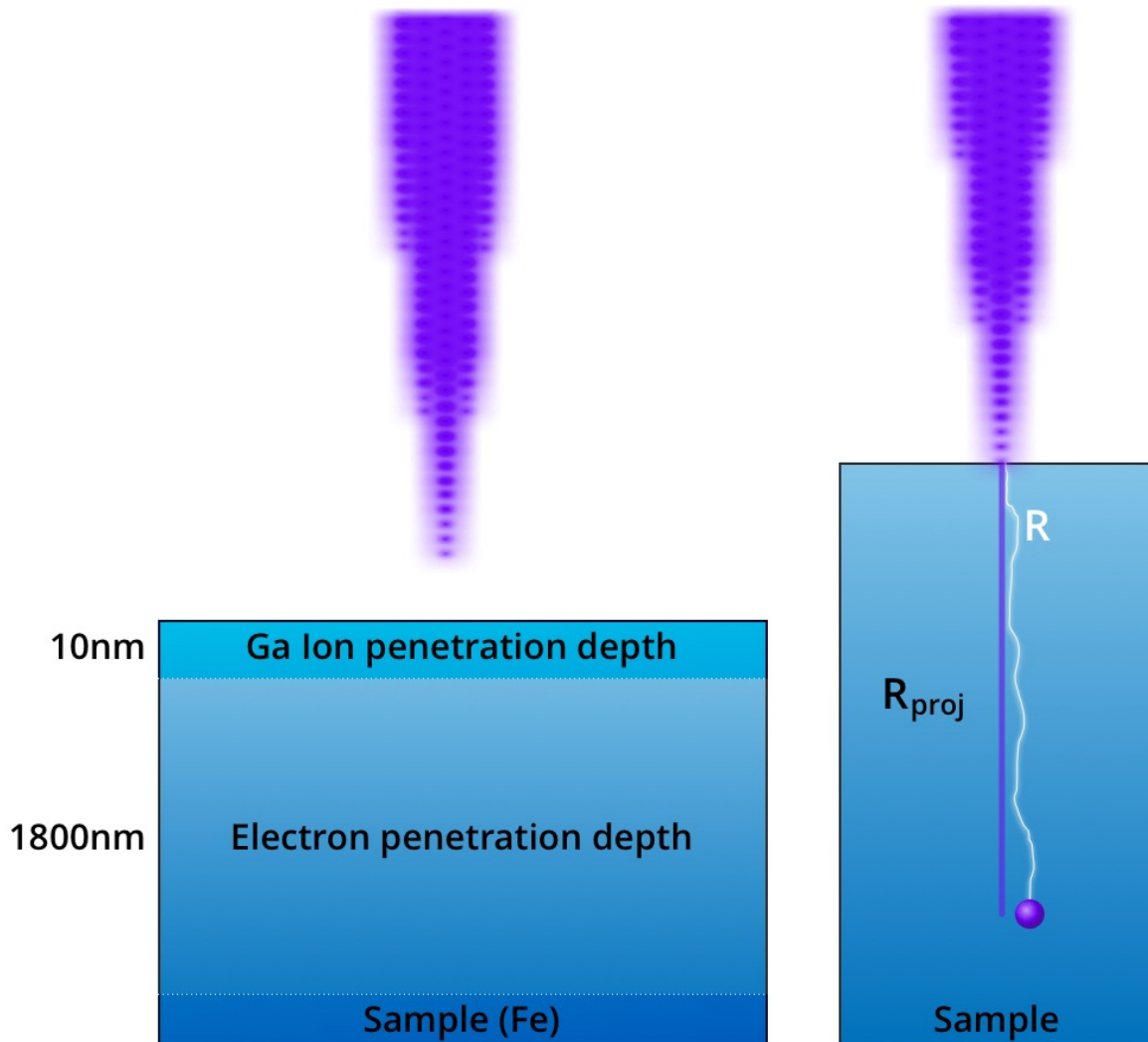
Secondary Electrons

The ion beam leads to secondary electron (SE) emission. A sample atom electron is ejected in this ion-beam sample interaction which can then be detected by a standard secondary electron (SE) detector. These SE create the signal that is conventionally used when imaging with the ion beam. The SE yield for ion beams is higher in comparison to electron beams: 1-10 SE are emitted per ion while only 0.1-1SE are emitted per electron beam electron.
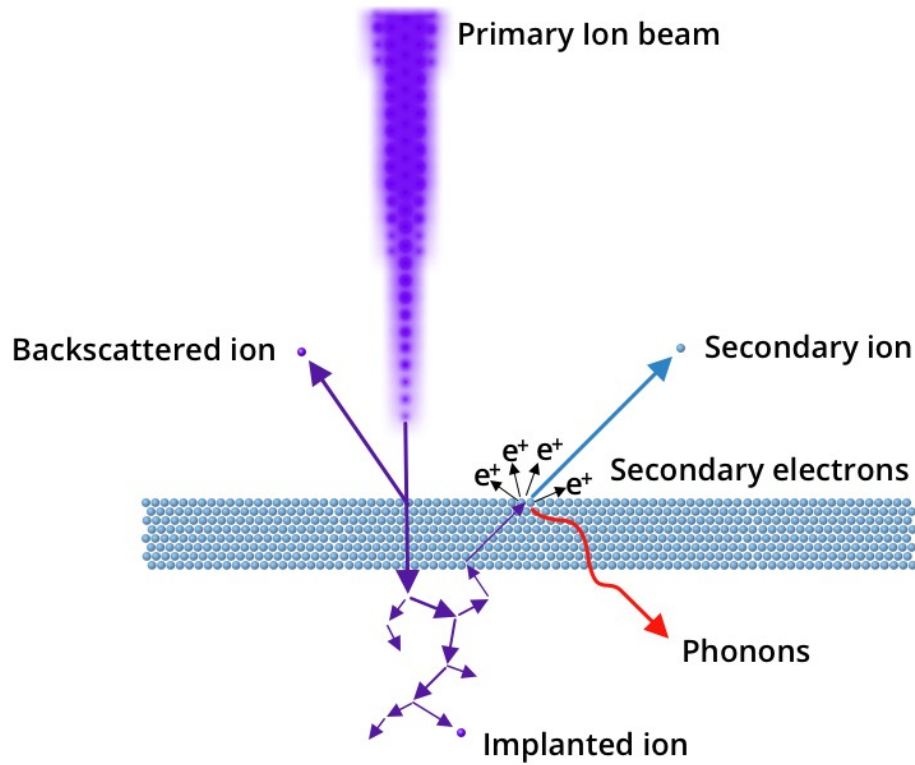
Secondary Ions, Backscattered Ions and Phonons

Secondary ions come from the sample. When the ion beam hits an atom within a specimen, this high energy collision can turn the samples atom into an ion, and this is usually accompanied by the ion being knocked out from the specimen surface. Thus a secondary ion is a sample atom which is ionised and emitted from the surface. Secondary ions therefore carry chemical information about the specimens surface, whereas primary ions have the chemistry of the ion beam source, usually gallium.

Backscattered Ions (BSI) are ions from the primary beam that "bounce" back from the sample surface. Since the backscattered ions come from the ion beam, they all have the same chemistry as the FIB source, usually Ga+. The BSI yield is low, usually only 0.1-10 % of ions are backscattered.

Phonons are atomic vibrations or waves within the samples crystal structure that are induced by the energetic bombardment of ions into the specimen. They represent a form of heat.
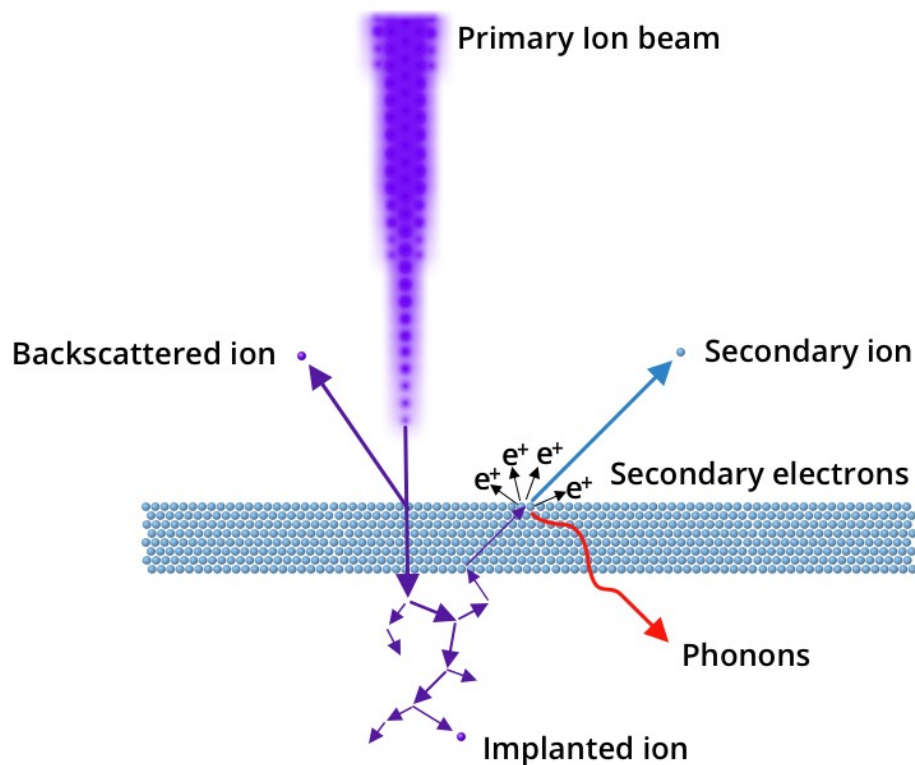
Energy loss

Energy loss from the ion beam occurs by two types of interactions: **elastic** interactions with the nucleus of atoms in the sample, or **inelastic** interactions with the electron cloud of atoms in the sample. Each time an ion collides with an electron cloud or nucleus, a certain amount of energy is transferred from the ion to the sample. The total energy loss is simply the sum of the elastic and inelastic interactions. The outcome of a collision between an ion and an atom in a sample depends on the energy that is transferred during the collision as well as the sample's crystallography and chemistry. Nuclear interactions can lead to sputtering, secondary ion emission (SI), dislocations, amorphization, vacancies and phonons. Electronic interactions can lead to secondary electron emission (SE), phonons, plasmons (in metals) and polymerisation.
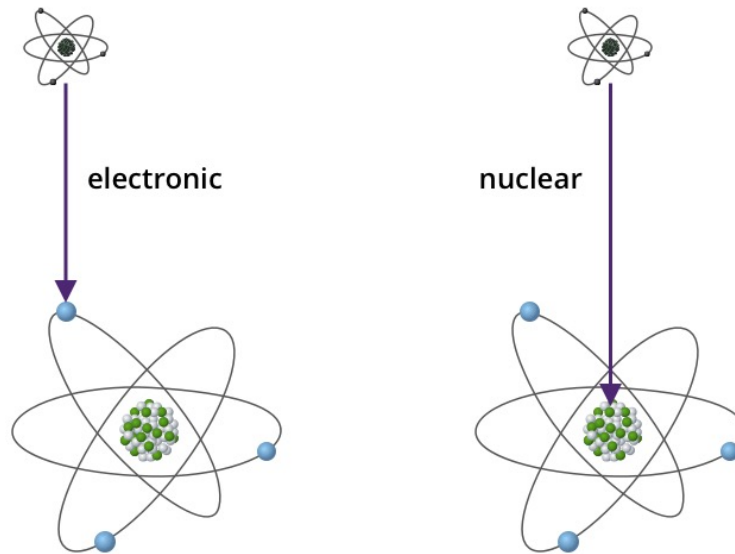
Collision Cascade

A single ion usually collides with multiple sample atoms and creates a cascade. This cascade can create vacancies, change the position of atoms within the crystal structure, and is usually associated with implantation of the incident ion from the primary beam. Collision cascade is commonly studies by Monte Carlo simulation, and a number of freeware packages are available for microscopists to study how the ion beam parameters effect the depth of specimen damage. An example of a Monte Carlo simulation is shown below. These calculation methods will also provide the microscopist with information about the energy of backscattered ions, backscattered electrons and a statistical analysis of the projected range.
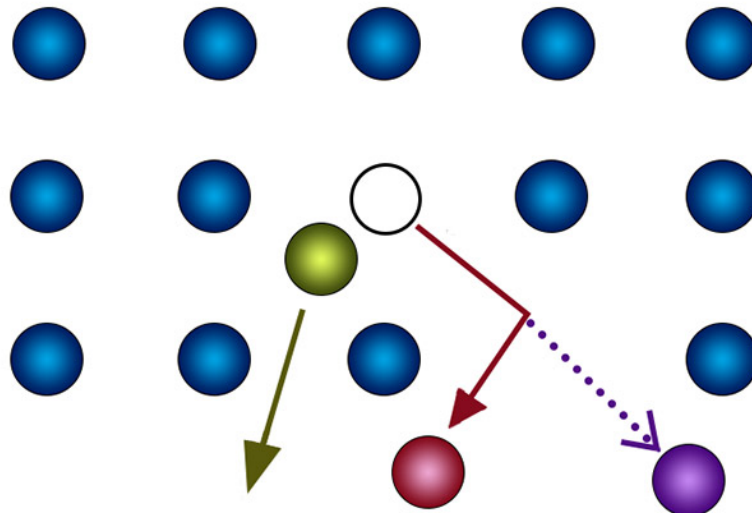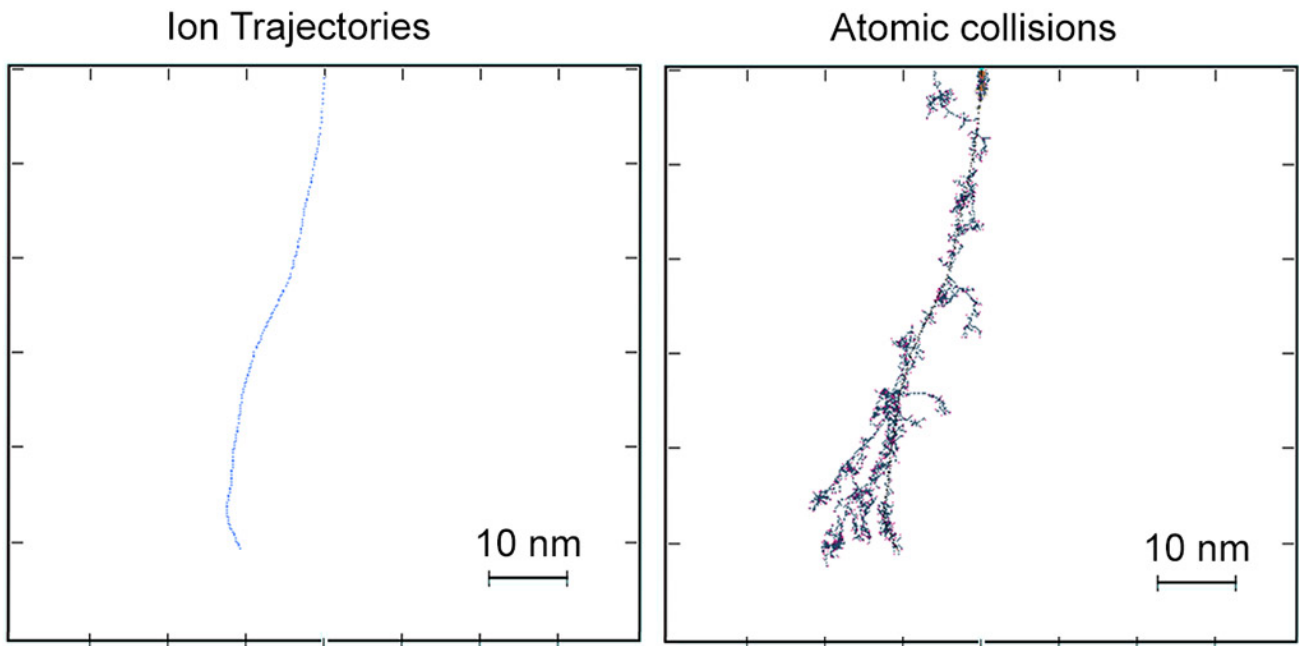
Sputtering - Sputtering Overview

Sputtering occurs when a sample atom is ejected from the sample surface. This is a special case of a displacement, only atoms near the sample surface are potential candidates for sputtering. The more collisions that occur on the sample surface, the more likely that sputtering occurs. Atoms that are located deeper within the sample are less likely to be sputtered because the ion beam loses energy as it penetrates the specimen.
Two conditions need to be met for sputtering.

1. The surface atom needs to receive enough energy.
2. The surface atom needs to be displaced into a position outside the sample

Parameters affecting sputtering
Sputtering is predominantly influenced by the surface binding energy, the incidence angle and the initial ion energy (acceleration voltage). The angle of incidence angle between the beam and the sample determines the trajectory of the collision cascade, and dictates how many atoms are near enough to the sample surface to be potential candidates for sputtering.
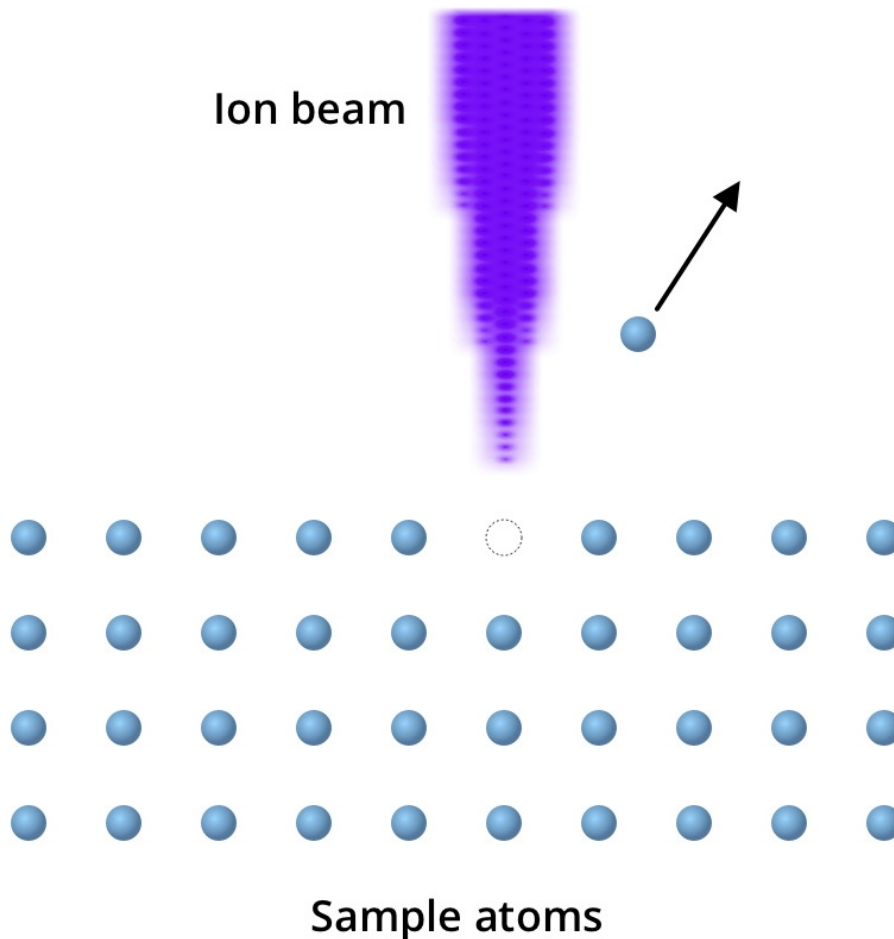
Sputtering Yield

The sputtering yield describes how many sample atoms are sputtered per incident ion

$$Sputtering\ yield = \frac{number\ of\ sputtered\ atoms}{number\ of\ incident\ ions}$$

The sputtering yield for Ga-FIBs is typically between 1 and 10 for an ion beam perpendicular to the specimen surface.

Effect of accelerating voltage

The following plot shows the effect of the acceleration voltage on the sputtering yield for a Ga+ ion beam in iron. At higher beam energies, more energy can be transferred from the ions to the sample atoms, sometimes referred to as recoils. The recoil atoms are therefore more likely to receive enough energy to overcome the surface binding energy and to be subsequently sputtered if they are close enough to the sample surface.
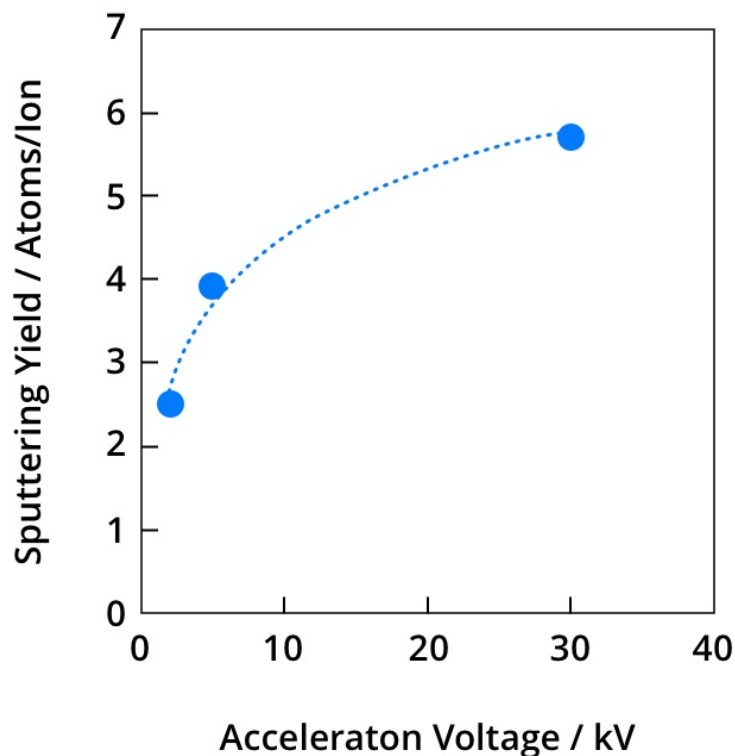
When using a FIB, the incidence angle (Θ) refers to the angle between the ion beam and the specimen surface. The incidence angle is 0° when the ion beam is perpendicular to the sample surface (indicated by the dashed lines in the illustration below).

Angle of incidence
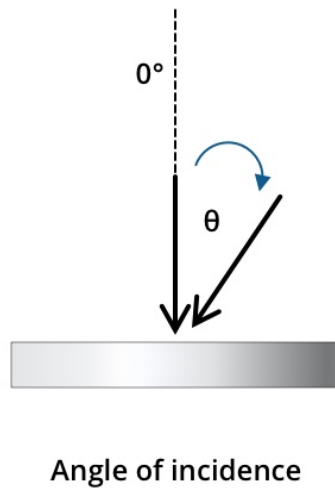
At larger incident angles, the collision cascade is predominantly located directly underneath the surface. This increases the probability of sputtering as more recoil atoms (sample atoms that have been struck by the incident ions) are close to sample surface. The maximal sputter yield can be obtained for an incident angle between 75°-85°, as shown in the graph below.
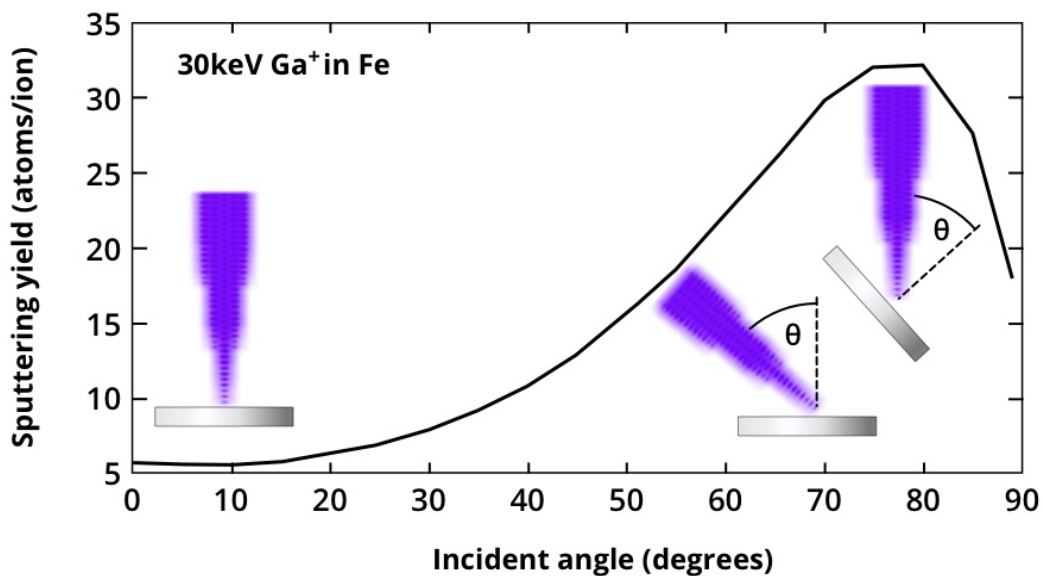

Material dependency

The surface binding energy is different for different materials, therefore different materials have different sputter yields. The difference in the sputtering yield gets smaller at higher angles, so the surface binding energy is less significant at high incident angles. Typical sputtering yields for a 30kV Ga+ ion beam are given below, where it can be seen that there are significant differences between the examples chosen here.

- Au: 17.6 atoms/ion.
- Fe: 5.7 atoms/ion.
- Si: 2.2 atoms/ion.
- Ti: 2.2 atoms/ion.

This difference in sputtering yields must be taken into consideration when using a FIB on a multi-phase system. For example, the image on the left shows the effect of cross-sectioning across a material interface between two different phases. Due to different sputtering rates, the material on the right has been milled to a significantly lower depth than the material on the right. When using the FIB to prepare thin sections such as those used for TEM sample preparation, breakage can occur at the interface, as shown in the image on the right.
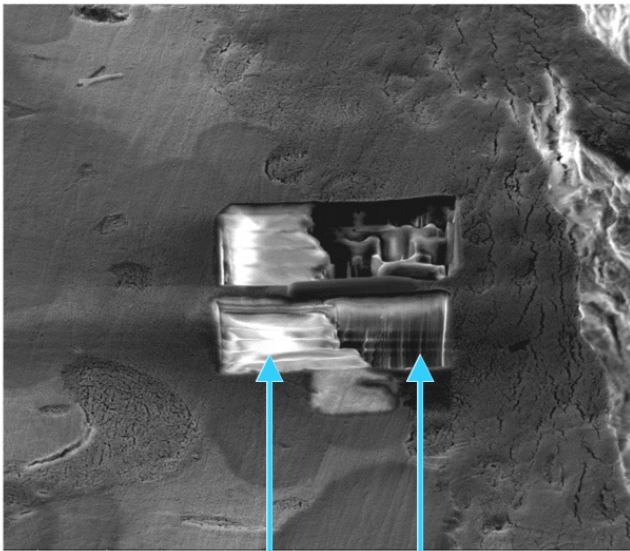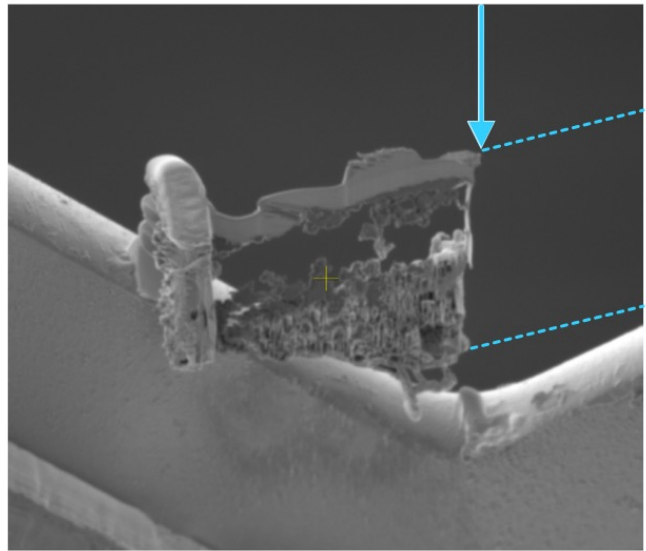
Material 1   Material 2

Image on the left shows the difference in sputtering yield, and therefore rate of milling, for different materials. Samples courtesy of Xiumei Zheng, Queensland University of Technology. Image on the right shows specimen breakage due to different milling rates across the interface between two different phases, image courtesy Jinying Lin, Queensland University of Technology.

Channeling

Channeling occurs for polycrystalline samples, such as copper, which has many different grains with different orientations. Ions penetrate deeper into low index directions (these directions have a lower density of atoms) and these will thus not produce as much signal or sputter as much, resulting in uneven milling as shown in the SEM images. Patterns that are structured across several grains often display different depths and structure qualities, as shown in the ion beam image on the left.

Slopes (redeposition)
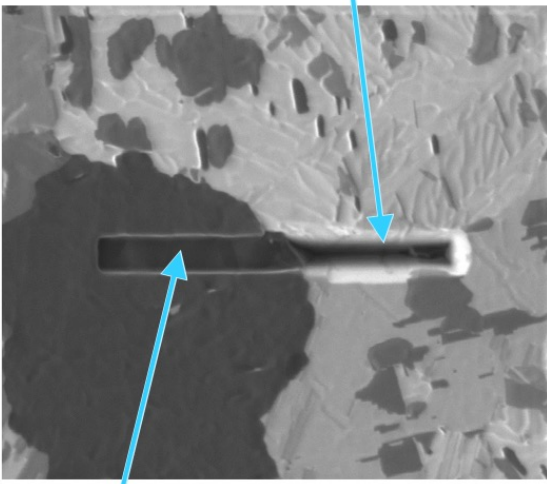
Higher structure quality
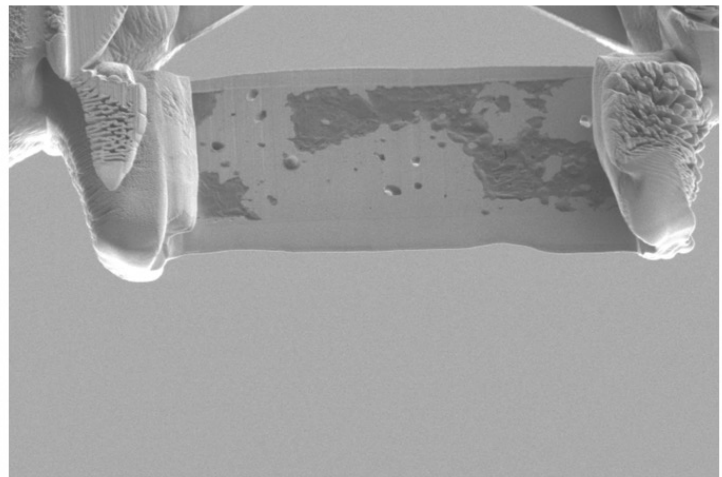
How does a FIB-SEM work? - Overview

A FIB is a focused beam of ions, and this beam is scanned (rastered) across the sample just like it is in the SEM. The ions have energy, and when they hit the sample they create a signal from this interaction. When the energy is high enough, the FIB can remove atoms from the sample surface. The removal of material is used to cut, trench or mill the sample on a scale of nanometers to micrometers.

The following sections will describe in more detail the complex intercations between the ion beam and the sample, and will also describe the design of the instrument, and some more common applications of the FIB-SEM.

Components of FIB - Introduction to components

Most FIB-SEMs have quite similar construction. They are built around a vacuum chamber, and have a vertical electron gun. The ion gun and the other selected attachments are placed around the electron gun at various angles and positions depending on the chamber geometry. Attachments to FIB-SEMs include Gas Injection Systems (GIS) for deposition or welding with different materials, or for patterning on the sample surface, a manipulator for in-situ lift-outs, and several types of detectors (often including secondary electron detectors, in-lens detectors, BSE, EDS and EBSD detectors) to allow a full-scale analysis of the sample and sample cross-sections. Please see the SEM and EDS modules for further information on how these detectors work.
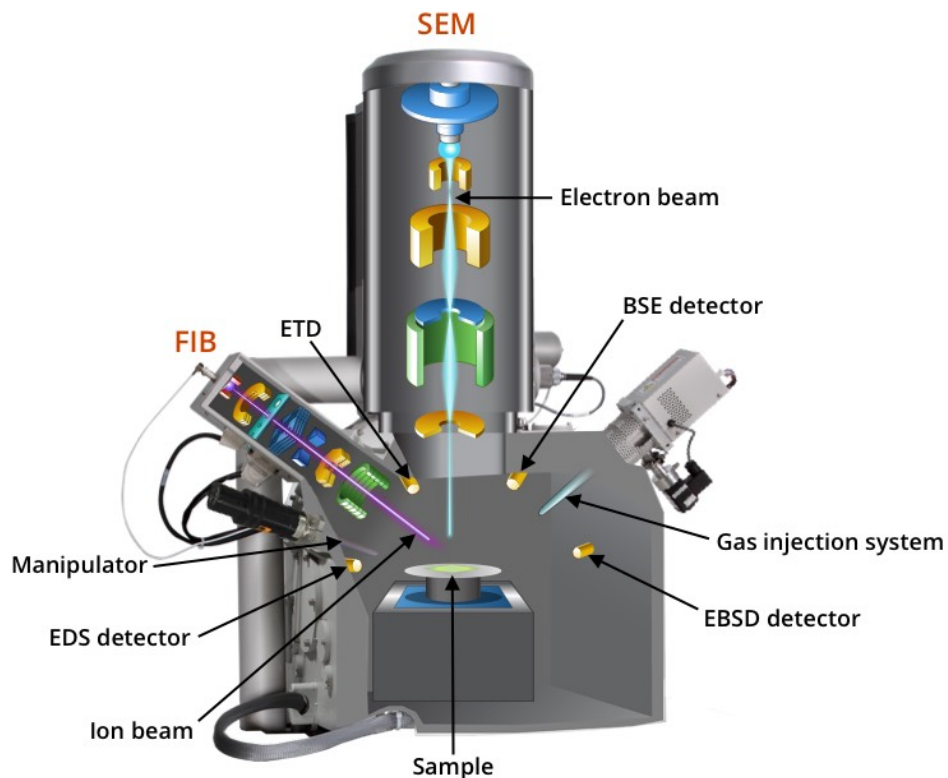
Vacuum system and Chamber

A high vacuum is required between the gun and sample surface to minimize collisions of the charged particles (electrons and ions) with air molecules. Vacuum is also very crucial for preventing contamination of the source and electrical discharge in the gun assembly. Ion pumps are normally used for the columns and a turbomolecular pump in conjunction with a dry pre-vacuum pump is commonly used for the specimen chamber.

SEM and FIB columns

SEM Column
Review the section 'Structure of the column' in MyScope's SEM module

Ion Column
The major components of the ion column are: the ion source that creates the beam; electrostatic lenses that control and shape the beam; a beam selective aperture that sets the ion beam size; and apertures that block out unwanted beam parts. An ion getter pump (IGP) keeps the column at a high vacuum ($\sim 10^{-5}$ Pa) to avoid contamination of the source and to prevent electrical discharges within the high voltage components of the column. Blanking plates in the FIB column are used to push the beam away from the optical axis when not required. Many different ion species and sources are available including the most commonly used gallium Liquid Metal Ion Sources (LMIS), the xenon plasma source, as well as liquid metal alloy ion sources using different ion species (LMAIS).
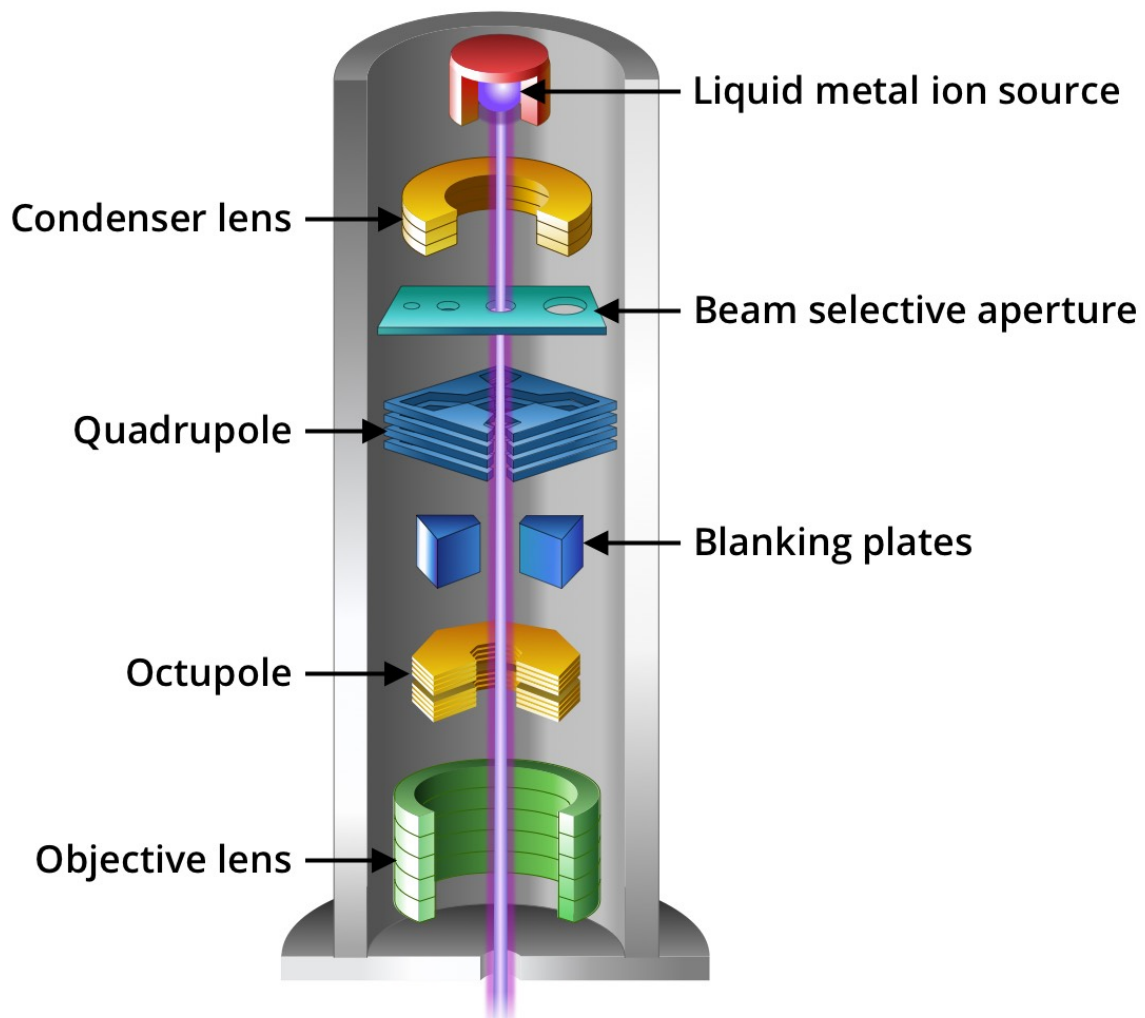
Gallium Liquid Metal Ion Source

The LMIS is a point source consisting of a tungsten needle with a liquid metal reservoir (conventionally gallium), a suppressor and an extractor. When in operation the LMIS is heated using a coil heater to reach the melting point of 29.8°C. The liquid gallium flows towards the tip of the tungsten needle which has a radius of around 2.5 μm. The extractor pulls the gallium into a small droplet called a "Taylor cone" which has a radius of around 2–5 nm. A tiny cusp forms at the end of the Taylor cone which is small enough to reach electric fields high enough for field emission (1.5×10^{10} V/m). The gallium Ions are extracted from the cusp of the Taylor cone by field ionization and are then accelerated down the column.

Gallium Reservoir

Gallium is most commonly used for the liquid metal reservoir since it allows room temperature operation due to its

low melting point of 29.8°C. This minimizes inter diffusion of the gallium with the tungsten needle. Gallium's low vapour pressure conserves the supply of the liquid metal and yields a long source life. Furthermore, gallium is sufficiently heavy for efficient sputtering of a wide variety of materials.
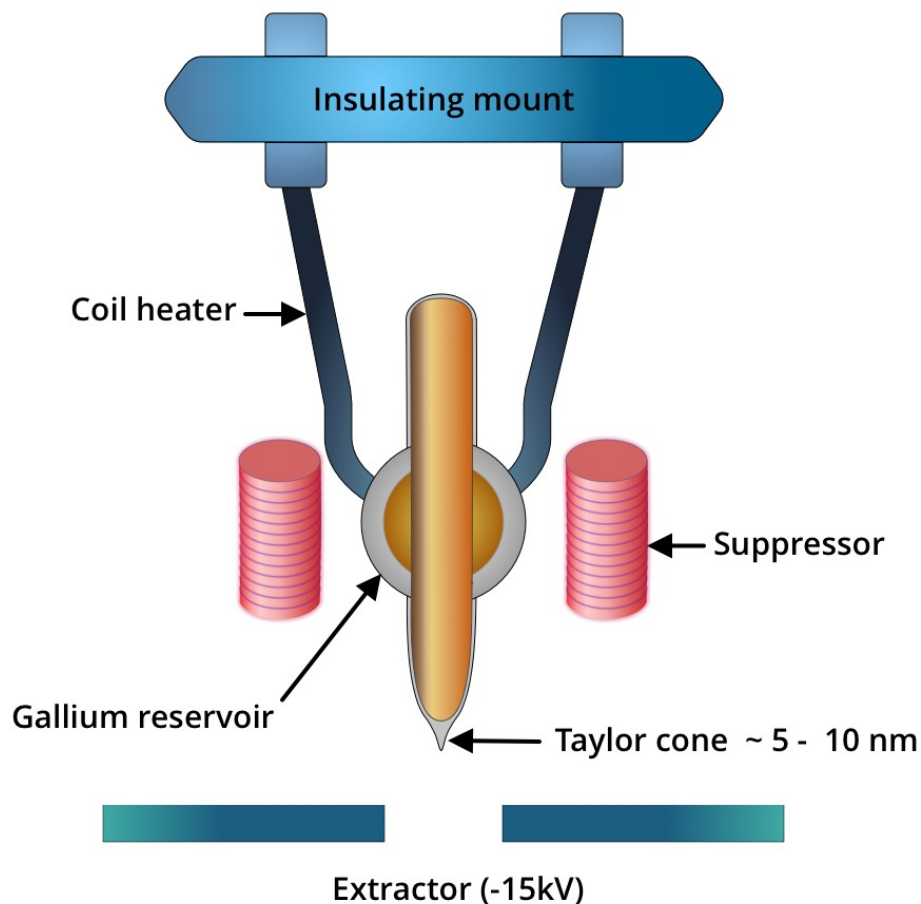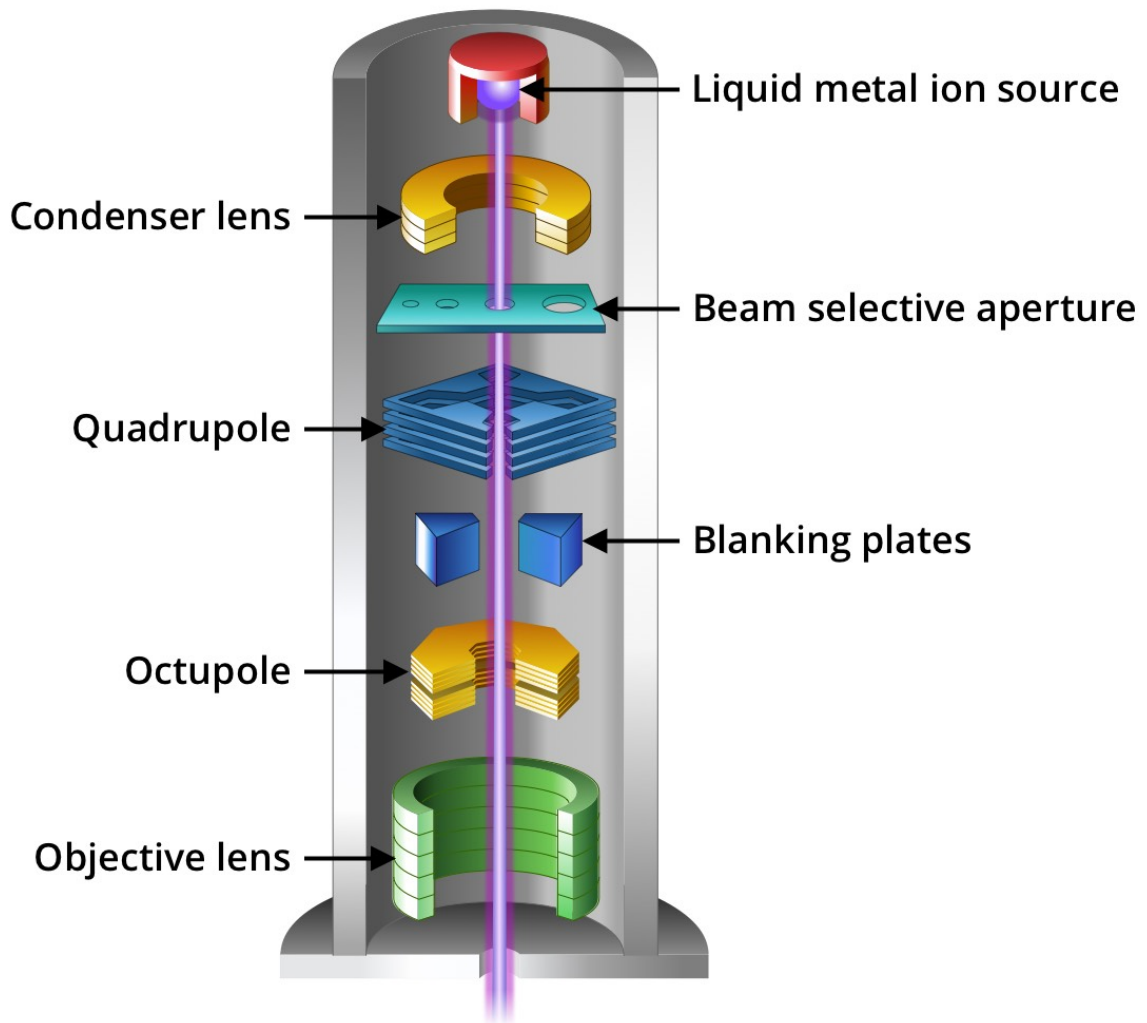

Ion emission

Ion emission is achieved by a complex mechanism involving field ionisation and field evaporation. The LMIS is placed close to the extractor (annular extraction electrode), which is held at a high negative potential with respect to the tip. The potential difference between the tungsten needle and the extractor creates an electric field and pulls the Ga from the needle tip, while the opposite surface tension of the molten Ga acts against this electrostatic force. Once balance is achieved between these two forces, the liquid Ga forms a conical shape called a "Taylor cone" with a tip radius of 2-5nm. The created field lowers (and distorts) the potential barrier of the gallium atoms in the Taylor cone. Electrons from gallium atoms at the cusp can now tunnel into the tungsten tip (field ionization). The ionised gallium atoms at the cusp are then field evaporated. Once ion emission starts, a constant emission current is maintained by the regulation of both the suppressor (actively changing) and extractor (remaining static once emission is achieved).

Tunneling process

Tungsten needle
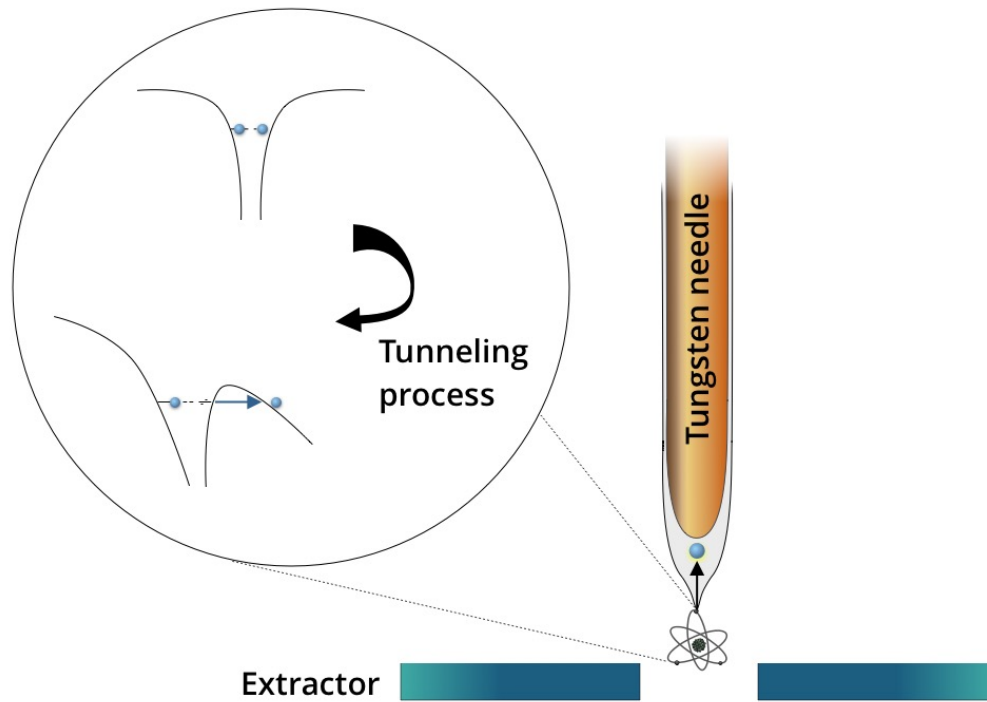
Extractor

Ion Optics

Once a beam of Ga^+ ions is extracted from the LMIS, it is accelerated through a potential and travels down the column. The ion beam will then go through a series of lenses and apertures along the beam path before reaching the specimen. The condenser lens forms the beam, which is then scanned across the sample surface by the octupole (often referred to as the scan coils). The objective lens focusses the ion beam onto the sample. Astigmatism is corrected by coils in the octupole region. The quadrupole controls the mid column steering of the beam during alignment. Electrostatic lenses are used for the ion column instead of the electromagnetic lenses which are found in the electron column. The velocity of ions is only 0.0028 of the electron velocity. Since the magnetic force in magnetic lenses is dependent on the particle velocity the magnetic lenses would be too large to fit in the ion beam column. Instead, electrostatic lenses which are independent of the particle velocity, are used in the FIB. The blanking plate deflects the beam away from the centre of the column to avoid unnecessary exposure of the sample during FIB operation.

Liquid metal ion source

Condenser lens

Beam selective aperture

Quadrupole

Blanking plates

Octupole

Objective lens

Beam Selecting Apertures

The ion beam current is selected by the beam defining aperture. This is an aperture strip with differently sized circular holes called apertures (see bottom schematic). The larger the aperture, the larger the ion beam current.

Large beam   Small beam

Sample
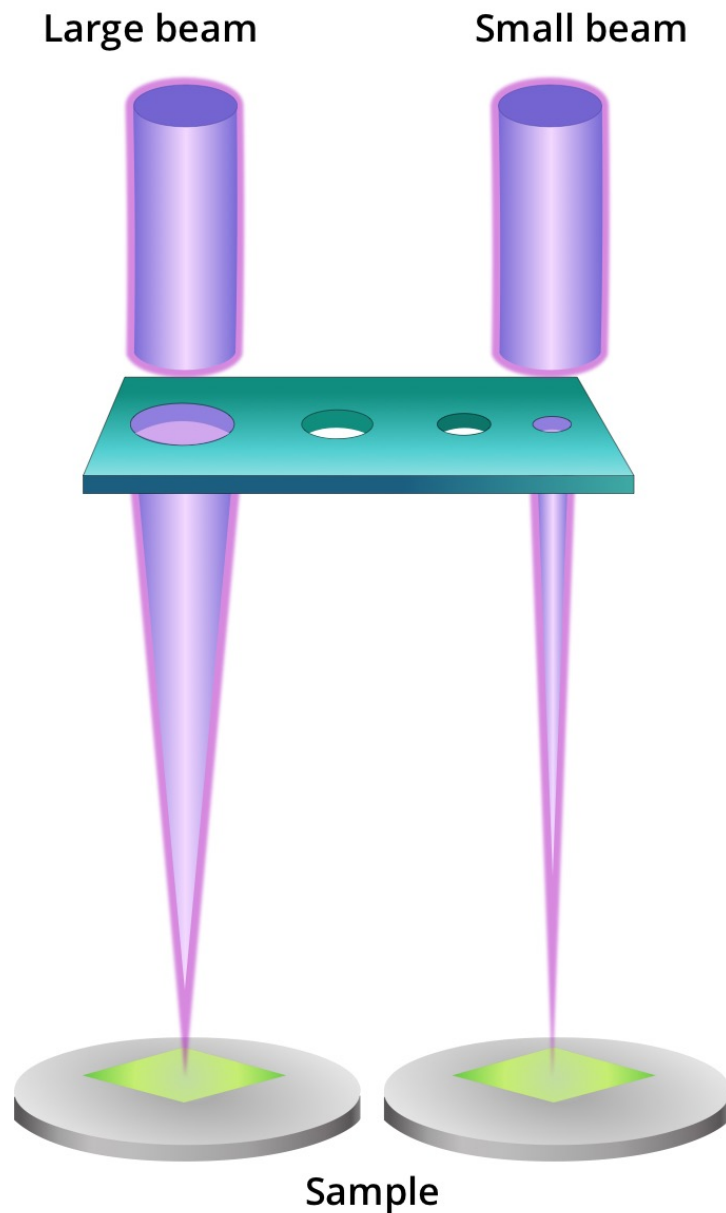
Imaging and Analytical Detectors

Imaging with the electron beam in a FIB-SEM is the same as in a SEM. Please refer to the SEM module for further information on electron imaging. FIB systems used for the characterisation of materials science specimens are therefore often equipped with the same set of detectors we would find on an SEM, for example, EDS detectors for chemical composition and EBSD for crystallographic orientation analysis. In addition, the ion-beam-created signals such as secondary electrons, and secondary ions can be detected via secondary electron detectors, secondary ion detectors, and time of flight secondary ion mass spectrometers.

Sample Stage

The stage is conventionally a five-axis (x-y-z-rotate-tilt) stage which allows precise and reproducible positioning of samples in the chamber. Specimen exchanges take place by venting the main chamber or through the attached small specimen exchange chamber for rapid specimen changeover.

Gas Injectors

Gas injection systems (GIS), introduce reactive neutral gases to the sample surface, are used for enhanced etching, preferential etching or material deposition (see below). GIS are usually inserted to within ~200μm of the sample surface. The gap between the inserted GIS and the sample surface corresponds to the thickness of a human hair. From that position, the GIS delivers a controlled flow of gas from the crucible (which contains the GIS precursor) to the sample surface through a long nozzle. As the precursor of the GIS is a solid, the GIS needs to be heated prior to operation to allow the precursor to transition to a gas phase.

Enhanced & Preferential Etching

Gas assisted etching can be used to enhance sputtering rates of materials or to preferentially mill one material over another. These applications are commonly used in the semiconductor industry. 26 different chemistries are available for a wide range of applications, for example Epsom salt. The Epsom salt precursor increases the milling rate of carbon and resist materials and reduces the milling rate of some metals, allowing the user to preferentially mill the resist and not the metals in semiconducting devices.

Material Deposition

GIS also allow the deposition of a broad range of materials including platinum-rich/tungsten-rich/carbon/insulating solid onto the sample. This is used for nano-fabrication and sample protection purposes. During deposition, a gaseous precursor (e.g. $(Ch_3)3Pt(CpCH_3)$ for the platinum rich deposition) is released onto the sample and is then cracked by the ion beam (or electron beam). The non-volatile products (here Pt-rich solid) adsorb on the sample surface. The volatile products are removed by the pumps that maintain the chamber vacuum.

For efficient deposition, the ion current used must be in proportion to the size of the deposition area. If the current is too high, the gas condensation is outweighed by the FIB milling process, leading to sample destruction rather than deposition.

Ion beam

Reflected ion

Volatile product

GIS

Sample stage

Nonvolatile product (Pt)

CH_3

$H_3C - Pt - CH_3$

CH_3

GIS
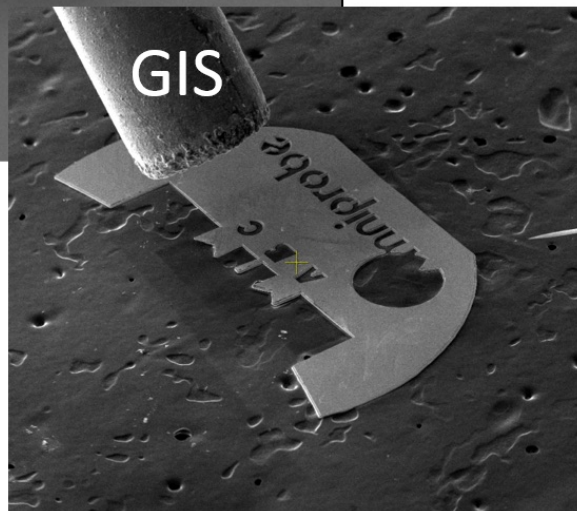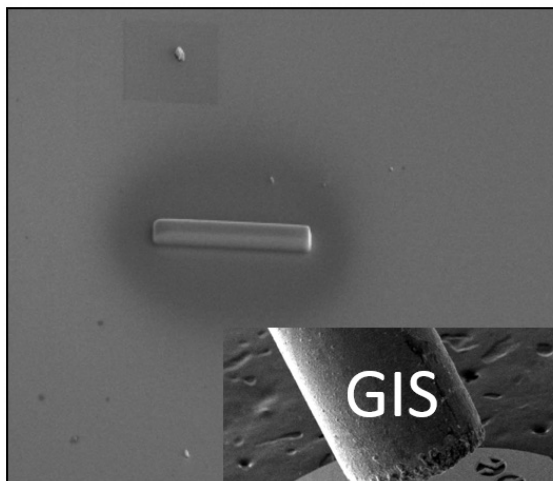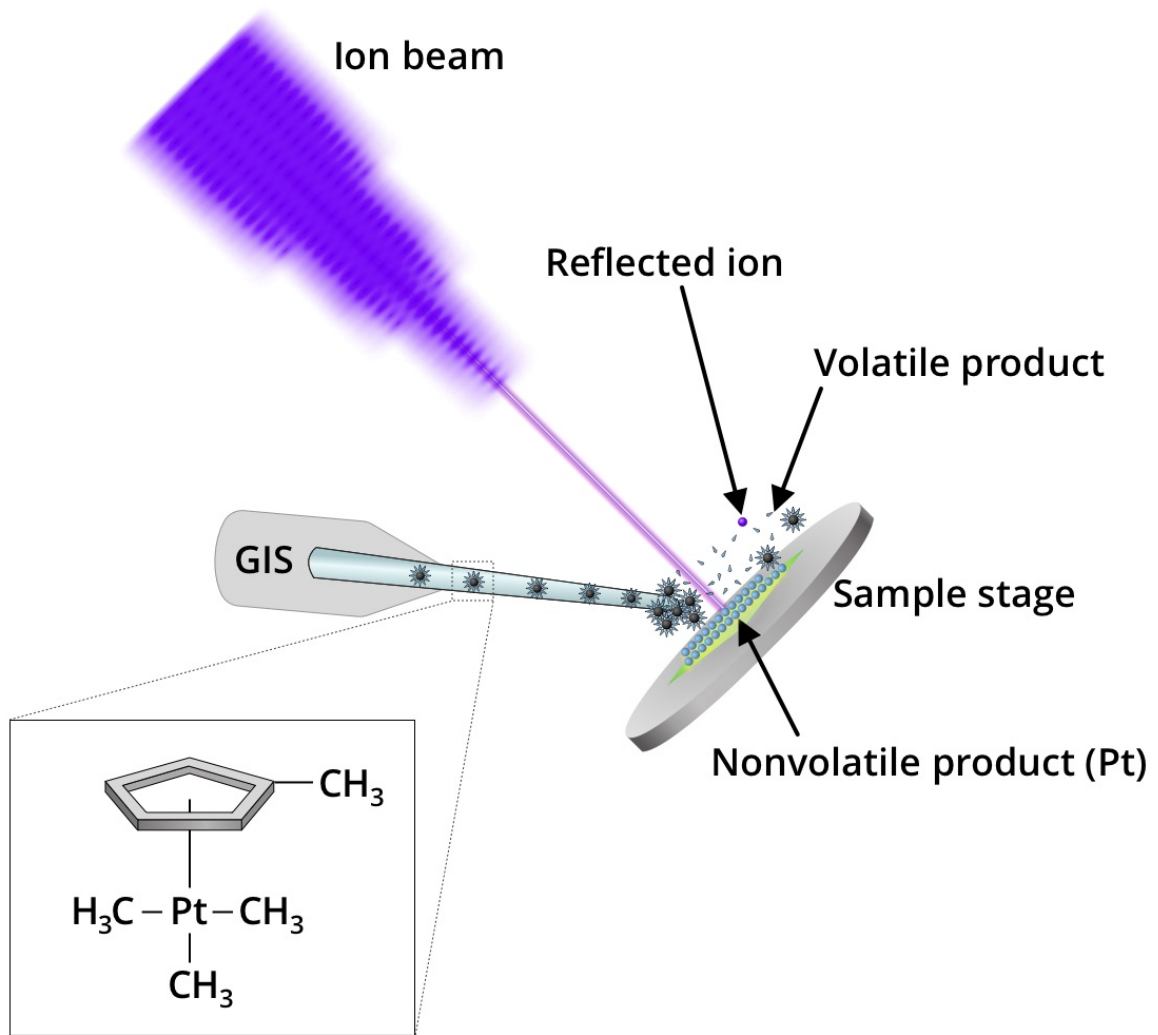
Manipulators

The manipulator is a sharp needle that allows sample sections to be lifted out and relocated to either a TEM grid (TEM lamella, APT sample) or another area of choice on the sample. Different manipulator systems are available for FIB-SEMs and their operation protocols vary for different devices. In general, both the electron beam and the ion beam are used to determine the manipulator position with respect to the sample and to assist in driving the manipulator to the sample area of choice with sub micrometer precision.

Manipulator
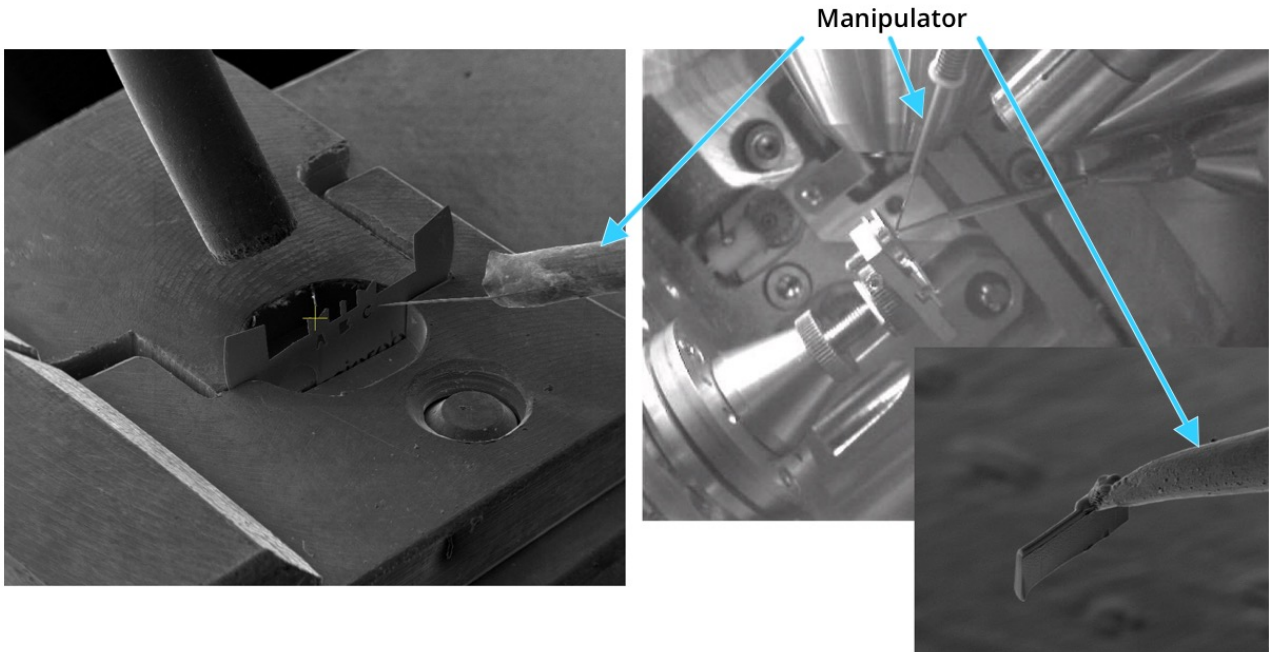
Geometry

Different FIB-SEM systems have different geometries. The angles between the FIB and SEM column differ for different manufacturers. Depending on the chamber geometry, the FIB-SEM angular offset ranges from 50-60°. The different attachments such as the GIS and manipulator can come into the chamber from various different ports on the chamber, as evident in the images. Understanding the geometry of the device is essential for safe FIB-SEM operations and should be checked with the instrument technician/owner.
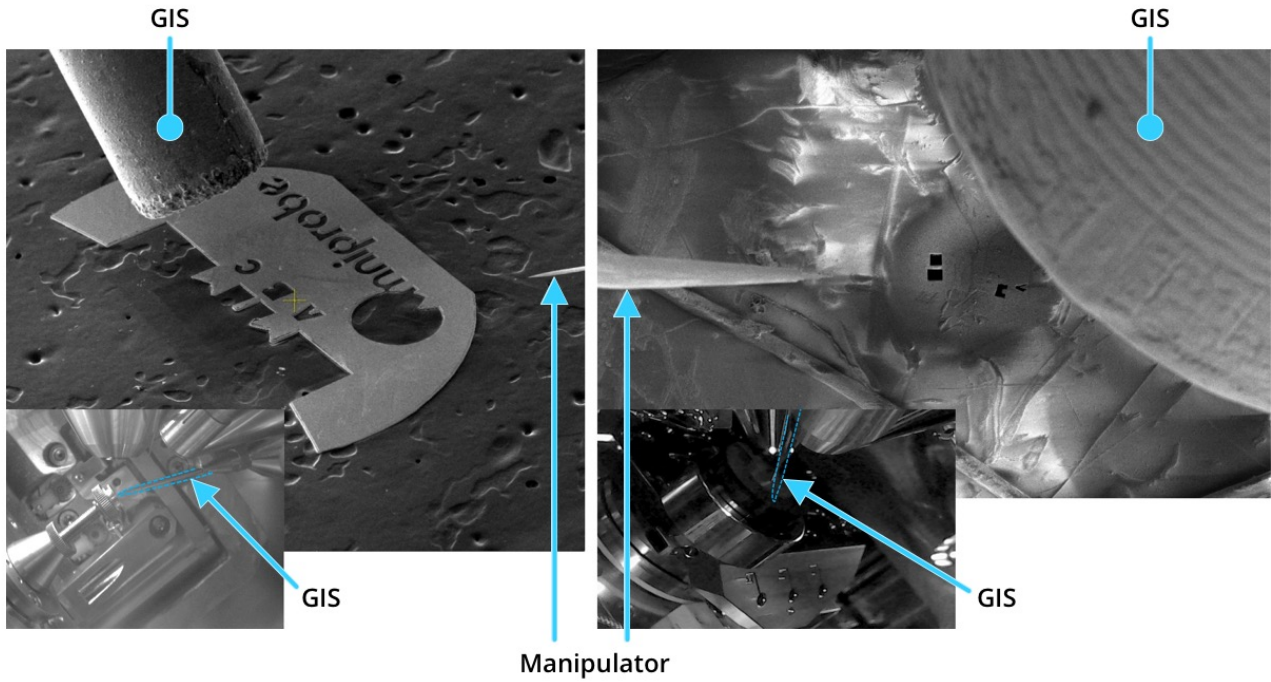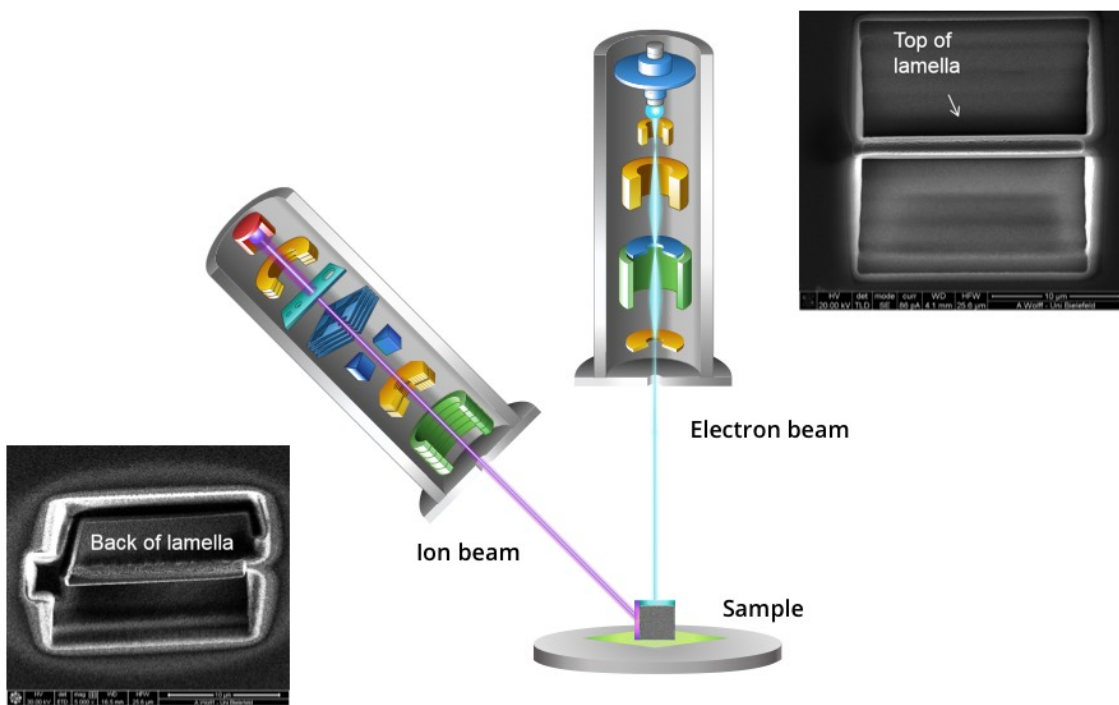
Concepts - Stage Tilt - Beam Geometry

Overview
Understanding the FIB-SEM geometry is important throughout operations. The stage tilt determines which surface area the different beams are hitting.

0 degrees
When the stage is tilted perpendicular to the electron column, the electron beam looks at the sample surface and the ion beam looks at the back of the sample.

Tilted
When the stage is tilted perpendicular to the ion column, the electron beam looks at the sample front (cross-section) and the ion beam looks at the sample surface.

Ion beam

Electron beam

Front of lamella
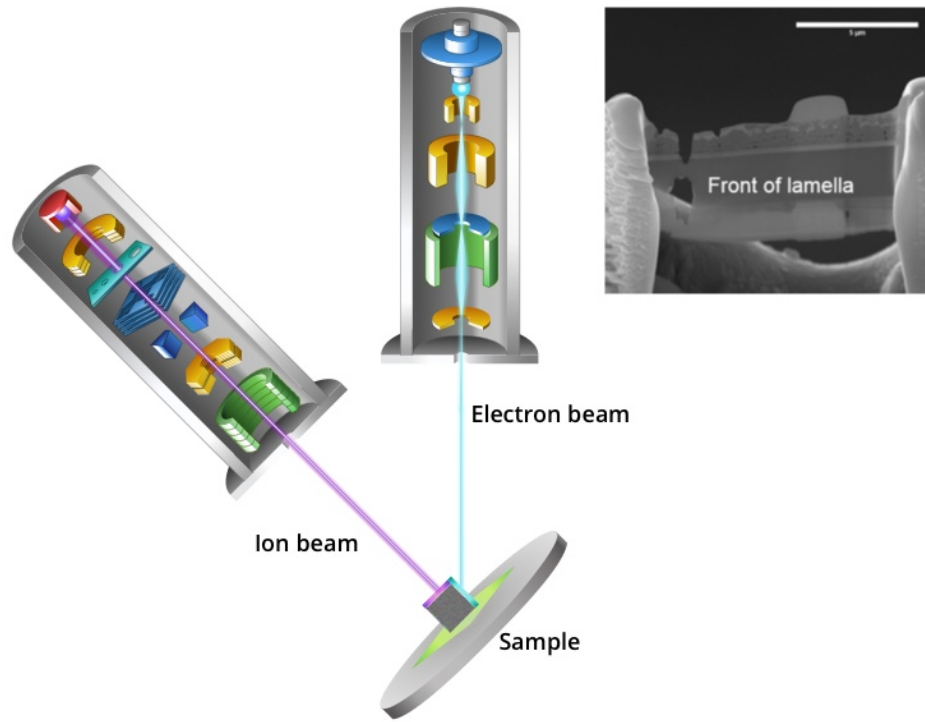
Sample

Imaging Artefacts

Tilting the stage allows you to image the sample from a different direction and thus to get a 3D perspective of the sample. Two effects need to be taken into account when imaging tilted samples:

1. The difference in height between the top and the bottom of the frame (left image).
2. The foreshortening of the y-direction

Correction algorithms are often implemented in the user interface of the FIB-SEM to compensate for these effects.

Working Distance
The schematic illustrates the difference in sample height when the stage is tilted. The top part of the sample (on the RHS) is closer to the pole piece and above the focus point (and thus unsharp) while the bottom part of the sample (on the LHS) is farther away from the pole piece and below the focus point (and thus unsharp).
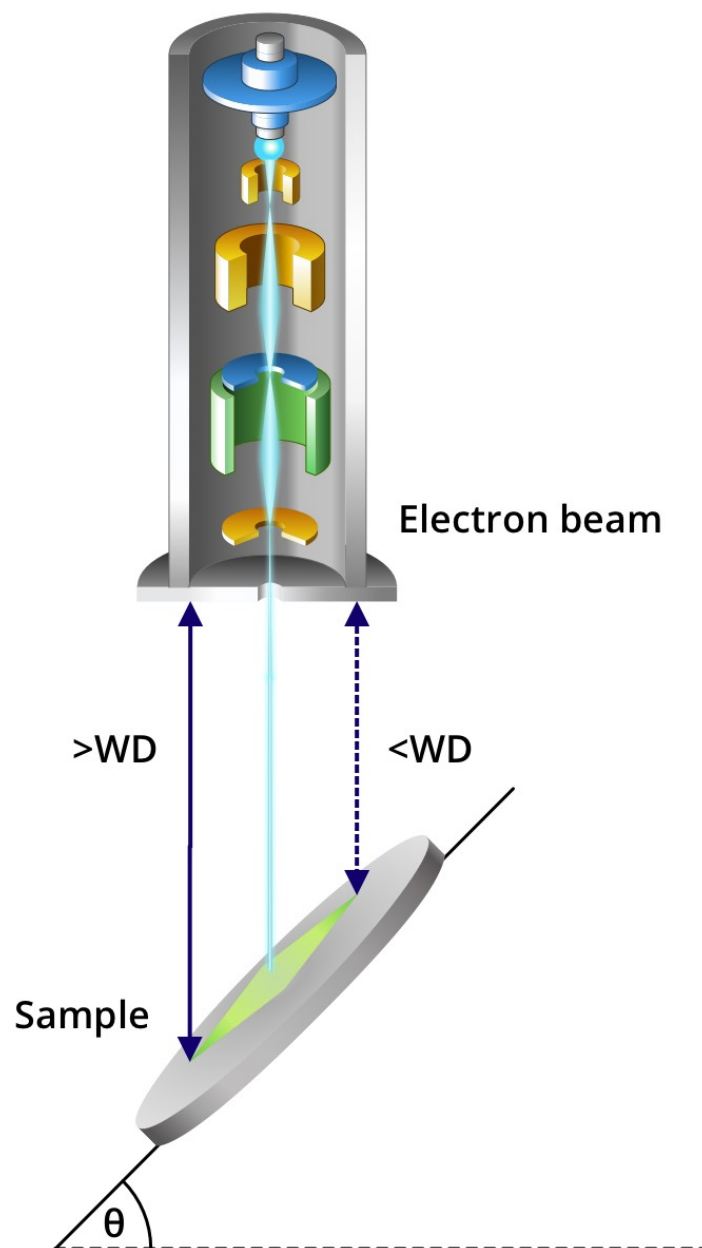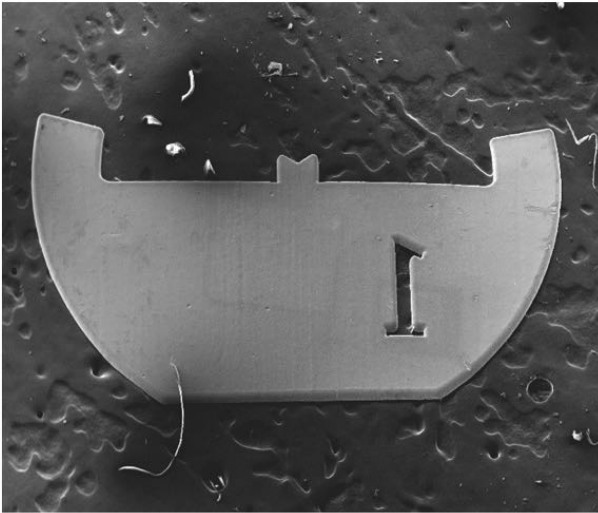

Forshortening
The y-direction appears shortened for tilted samples. This is illustrated in the middle schematic. The projection of

the sample is displayed on the image. The displayed image appears foreshortened as the projection is shorter than the actual sample length. This effect only occurs for the y-direction (as there is no tilt in x-direction).
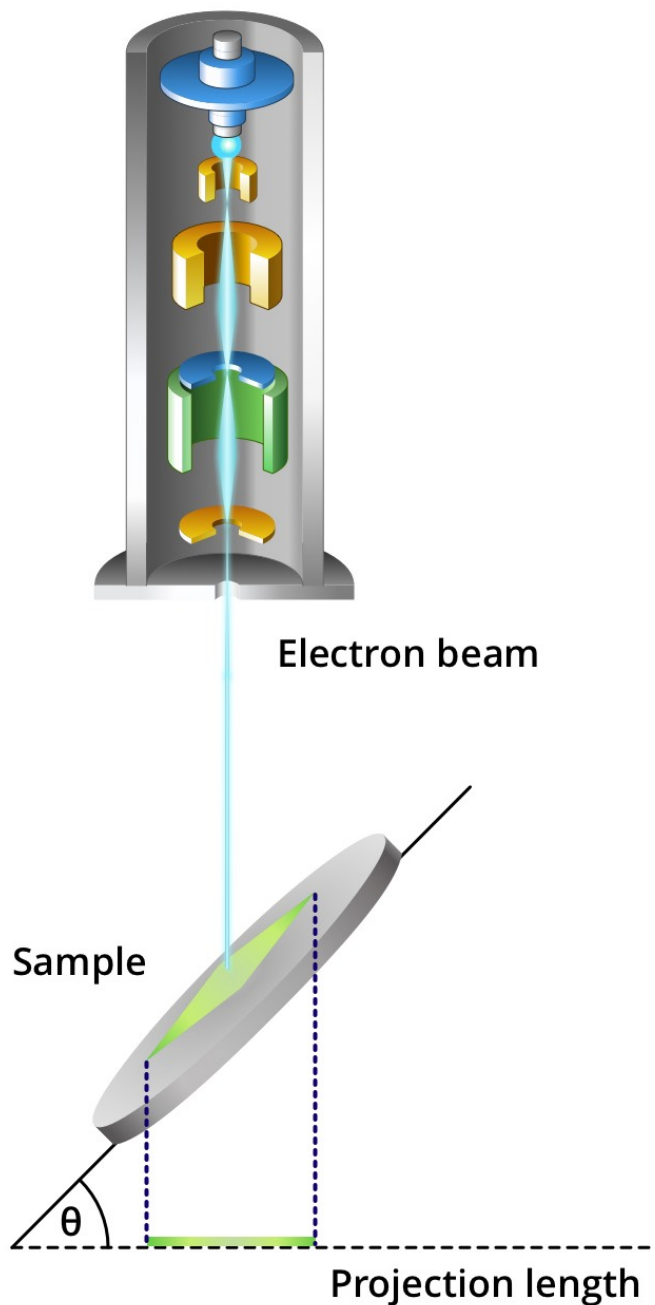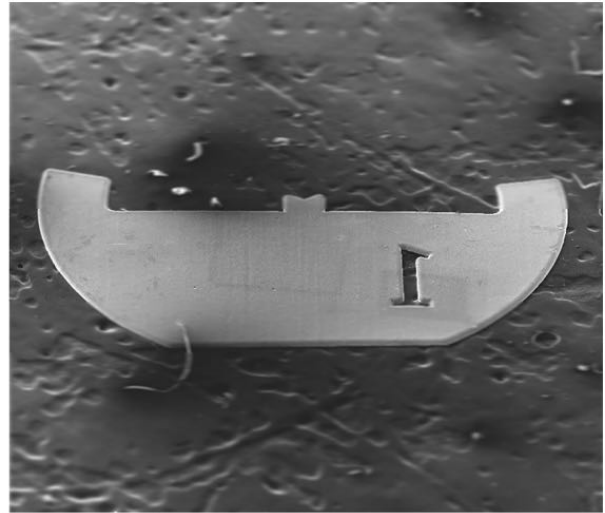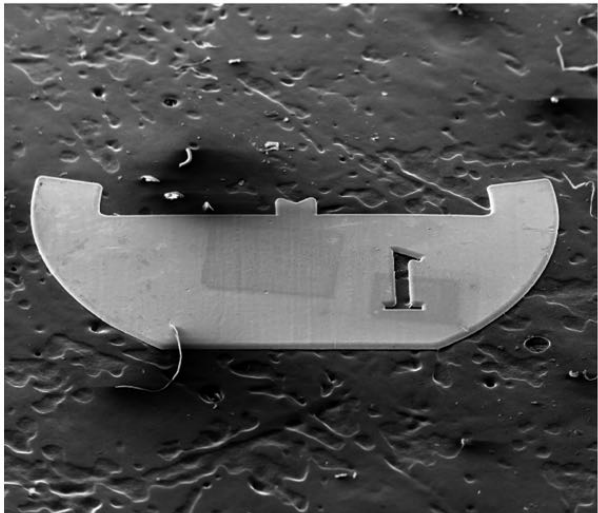

Visualisation of artefacts in SEM column
The images show the effect of foreshortening and working distance as well as the compensation.
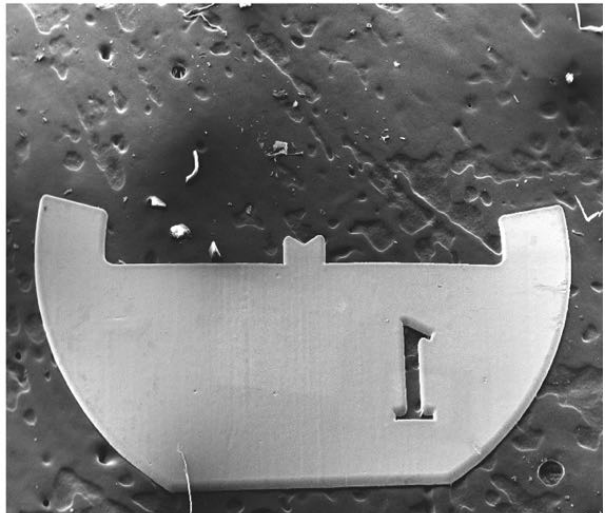
Electron image. Specimen is perpendicular to the electron beam.

Electron image. Specimen tilted to be perpendicular to the ion beam. Image has poor focus at the top and bottom of the image, and shows foreshortening


Electron image. Specimen tilted to be perpendicular to the ion beam. A focus correction has been made to account for the tilt of the specimen with respect to the electron beam, so the image is now in focus at all locations. This is often called dynamic focus. Image shows foreshortening.

Electron image. Specimen tilted to be perpendicular to the ion beam. Two corrections have been made, a focus correction and a tilt correction, to account for the angle of the specimen with respect to the electron beam. The image is therefore in focus at all locations and the foreshortening has been removed.
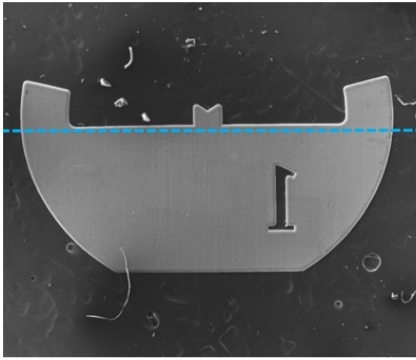
Eucentric Height

The eucentric height is the term we use to describe the location where the electron beam and the ion beam intersect. When at the eucentric point, the sample is on the tilting axis of the stage, and can be tilted to different angles without the feature of interest moving out of the field of view. This stage position is the recommended safe working distance for the majority of FIB-SEM systems.

Electron beam

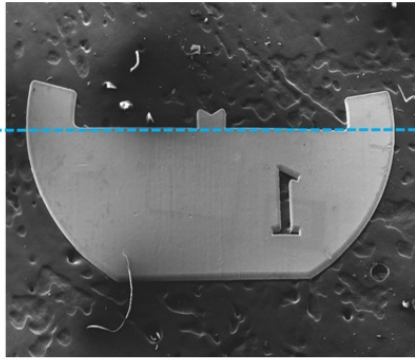**Ion beam**

Sample stage
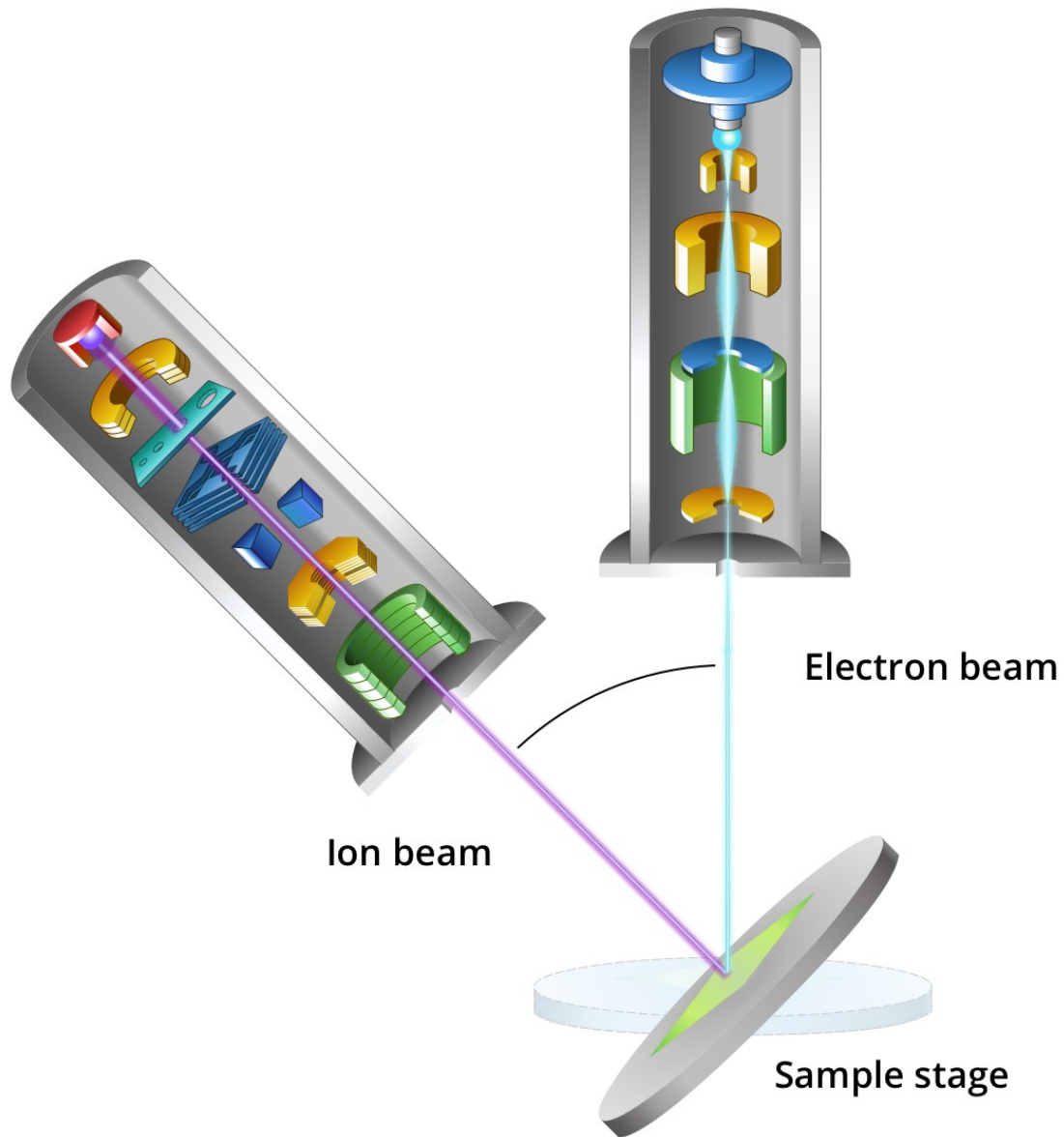
Effect in SEM images

The SEM images show the effect when tilting at eucentric height and not at eucentric height. When tilting at eucentric height, the feature of interest is in the same position when the stage is tilted perpendicular to the ion beam and perpendicular to the electron beam (left/middle image). The feature will be displaced in y-direction when the stage is not at eucentric height and tilted (left/right image).
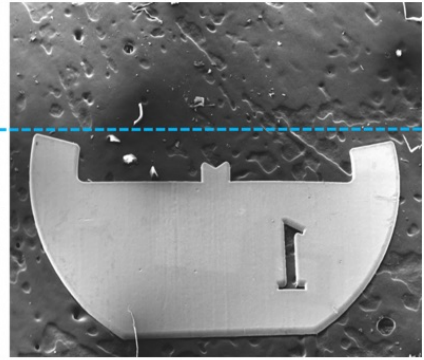
Tilt: ⊥E-Column

Tilt: ⊥ I-Column
Eucentric height

Tilt: ⊥ I-Column
**Not at eucentric height**

Coincidence Point

The coincidence point is defined as the point in which the intersection of both the electron beam and the ion beam hit the samples at the same spot. The same area can be seen when imaging with both beams at equal magnification. When the electron beam and the ion beam are not in coincidence the beams hit different spots. Setting accurate beam coincidence is of great importance during FIB-SEM operation. It allows the operator to use the electron beam for sample imaging instead of relying on ion beam snapshots or images that lead to sample degradation. Note the different viewpoints of the beams : At tilted stages, the ion beam looks directly at the sample surface (perpendicular). The electron beam looks at an angle at the sample surface.

Ion-beam not in coincidence   **E-beam**   **Ion-beam in coincidence**
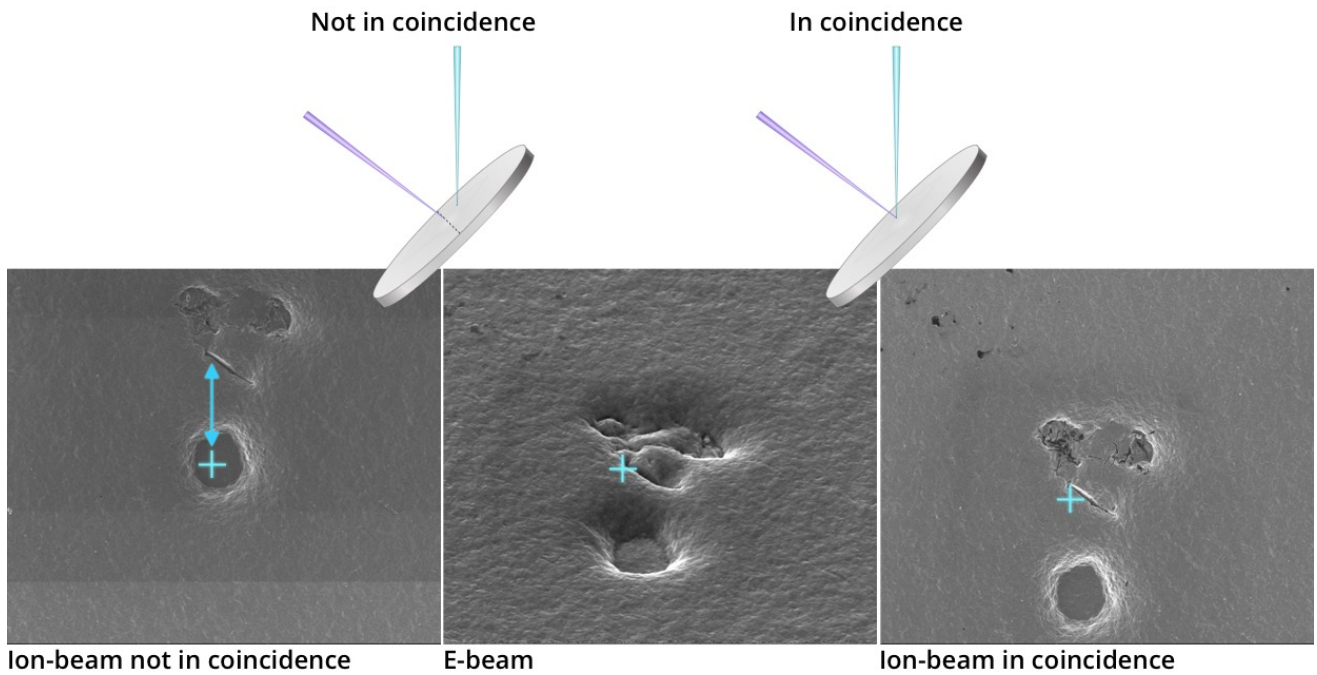
# Applications - Ion Beam Imaging - Image Formation

As we saw in the section on ion-solid interactions, the interactions of the ion beam with the sample atoms leads to secondary electron emission (emission of a sample atom electron) as well as secondary ion emission. A multitude of other emissions are also produced, but only the secondary electrons and ions are considered here as they can be detected and used for imaging applications. Secondary electrons that are created near the sample surface can be detected with a secondary electron detector.
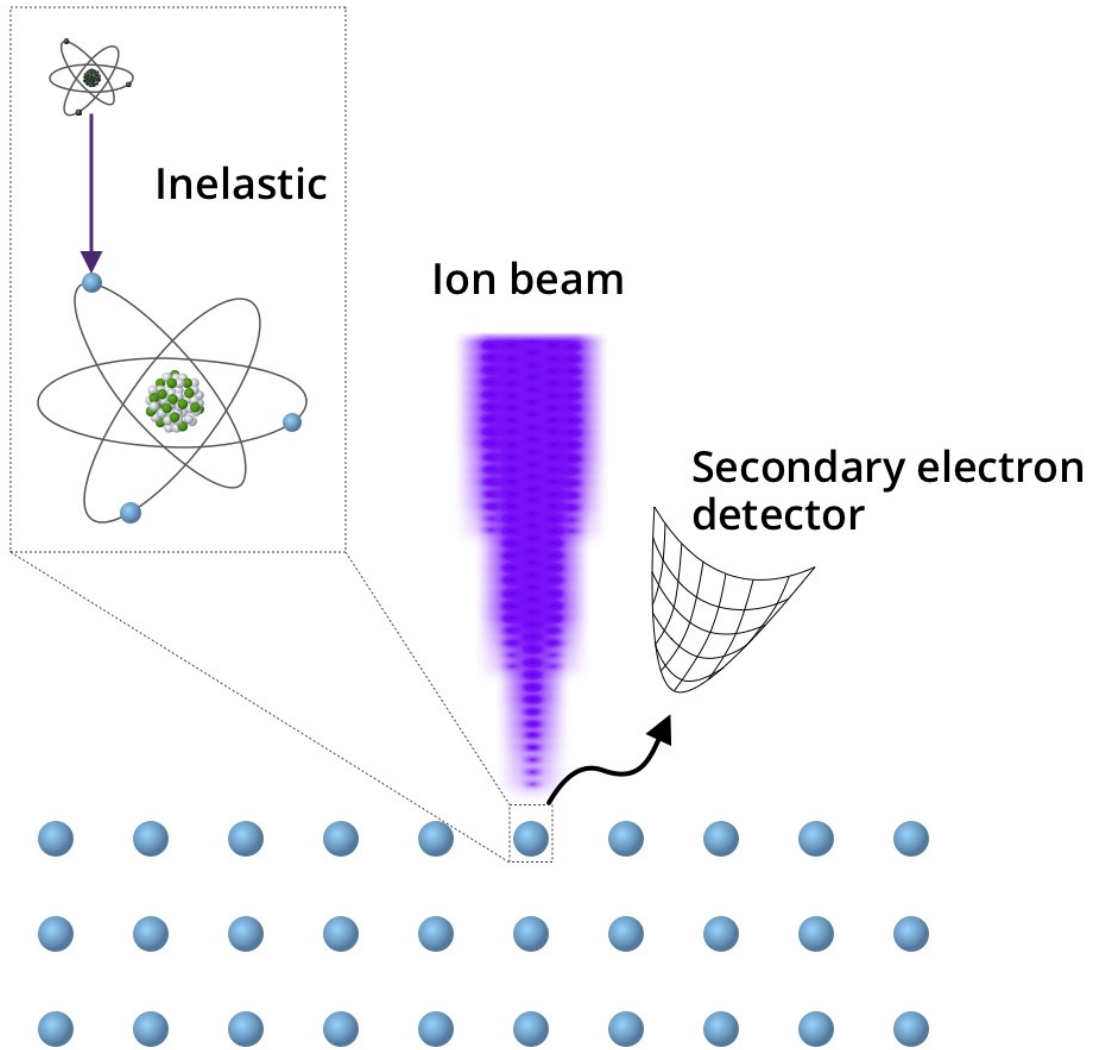
Charging

Charging in the ion beam image appears as (growing) black areas. The reason is illustrated in the right schematic: A positive charge (from the positive gallium ions) builds up at the surface in non-conductive samples due to gallium ion implantation and emission of electrons which also creates a positive net charge. The signal that creates the ion beam image is secondary electrons (we saw that earlier in the ion–solid interaction section). The secondary electrons have a negative charge and are back attracted to the now positively charged sample and do not reach the detector. The image appears dark since no signal is detected.

The charge can be compensated by using the SEM column as a floodgun. The negative electron beam is scanned over the sample while imaging with the ion beam. The electron beam compensates the build up of positive charge. This sample neutralisation allows the ion-beam-created secondary electrons to escape and reach the detector. Charge neutralisation can also be used when patterning and allows milling of semiconducting- and non-conductive samples. Low energy electrons are conventionally used for this.
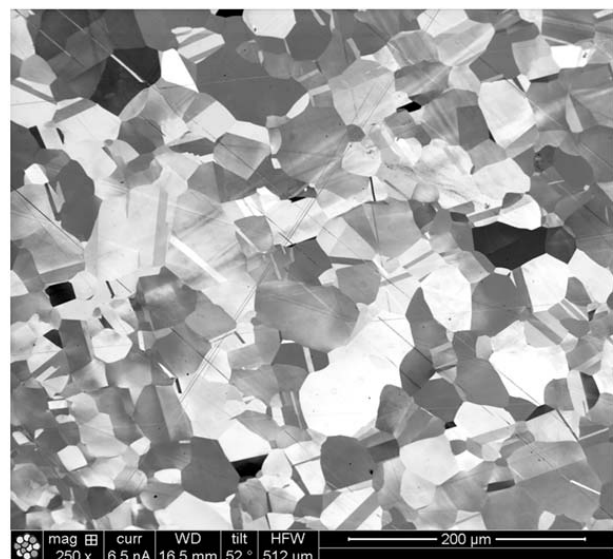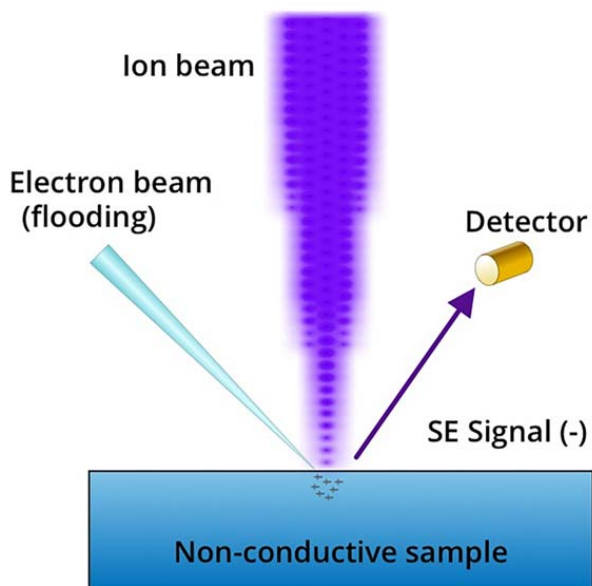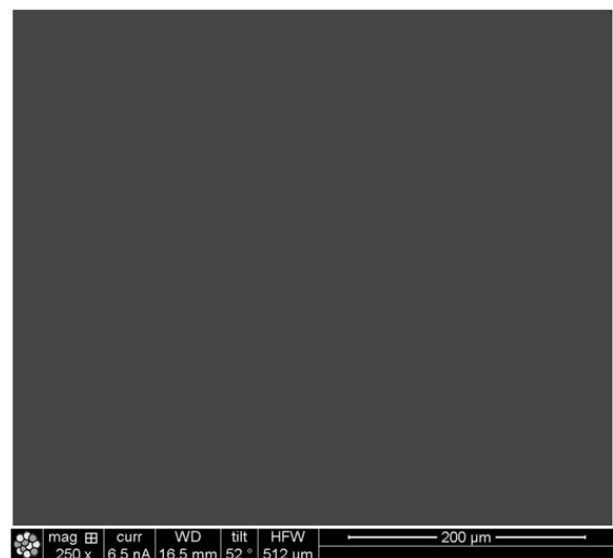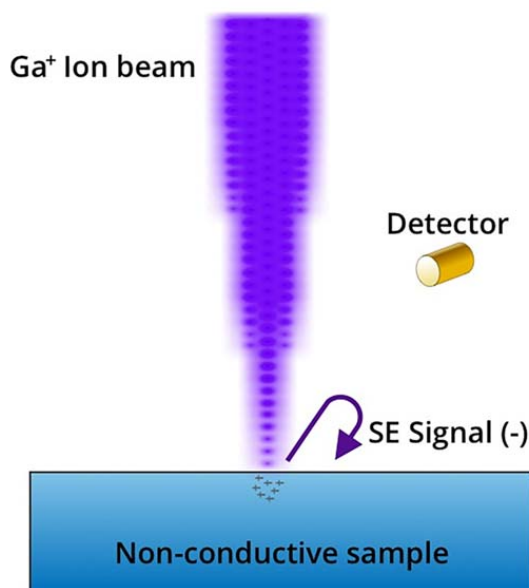
Channeling Contrast

FIB images show superior channeling contrast compared with conventional electron imaging. An example is given in the left image. Channeling contrast is created due to different ion–sample interactions in specific crystal directions. Ions penetrate deeper into materials in low index directions such as [110], [100]. In these directions, the collision cascade penetrates deeper into the material, due to there being a larger atomic distance (a wider gap) at these orientations, which leads to lower secondary electron yields. This means that these directions appear darker.

mag ⊞	curr	WD	tilt	HFW	⟵ 10 μm ⟶
5 000 x	27 pA	16.4 mm	0 °	25.6 μm	
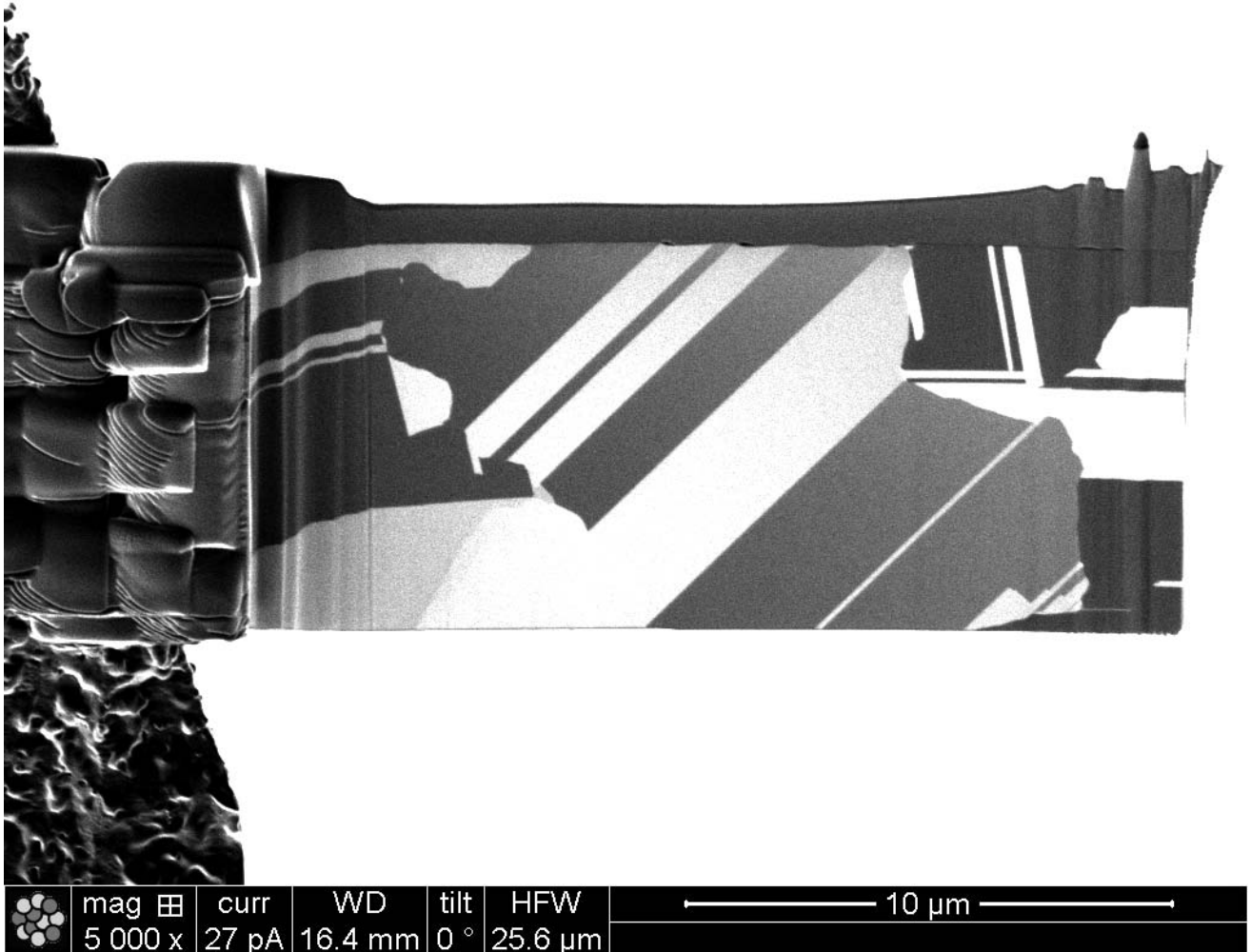
Ion channelling contrast image on TEM foil of Cu-Ni alloy. Image courtesy of Animesh Babak, Adelaide Microscopy
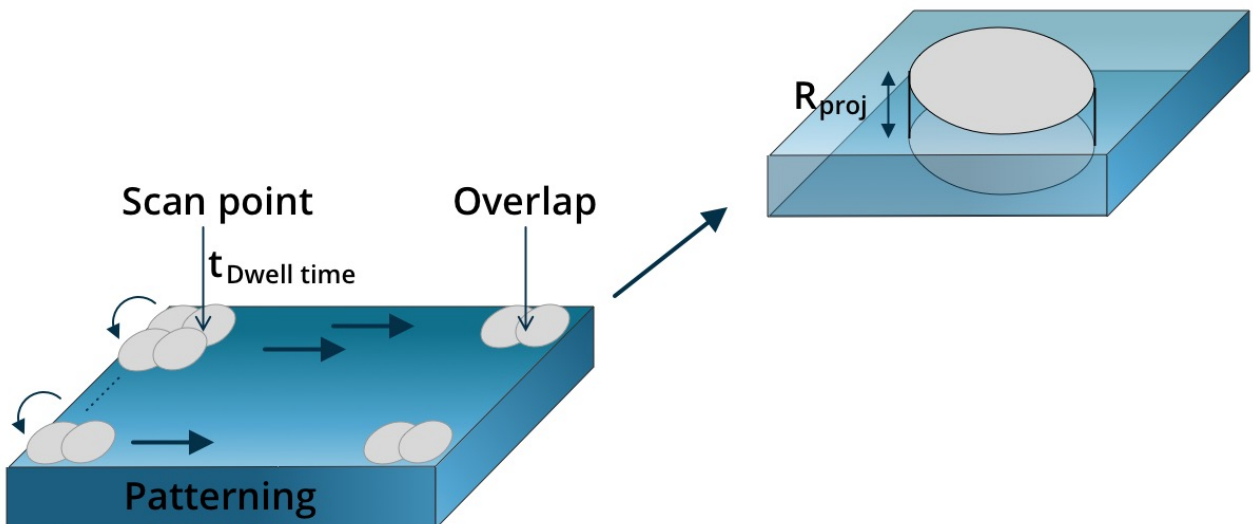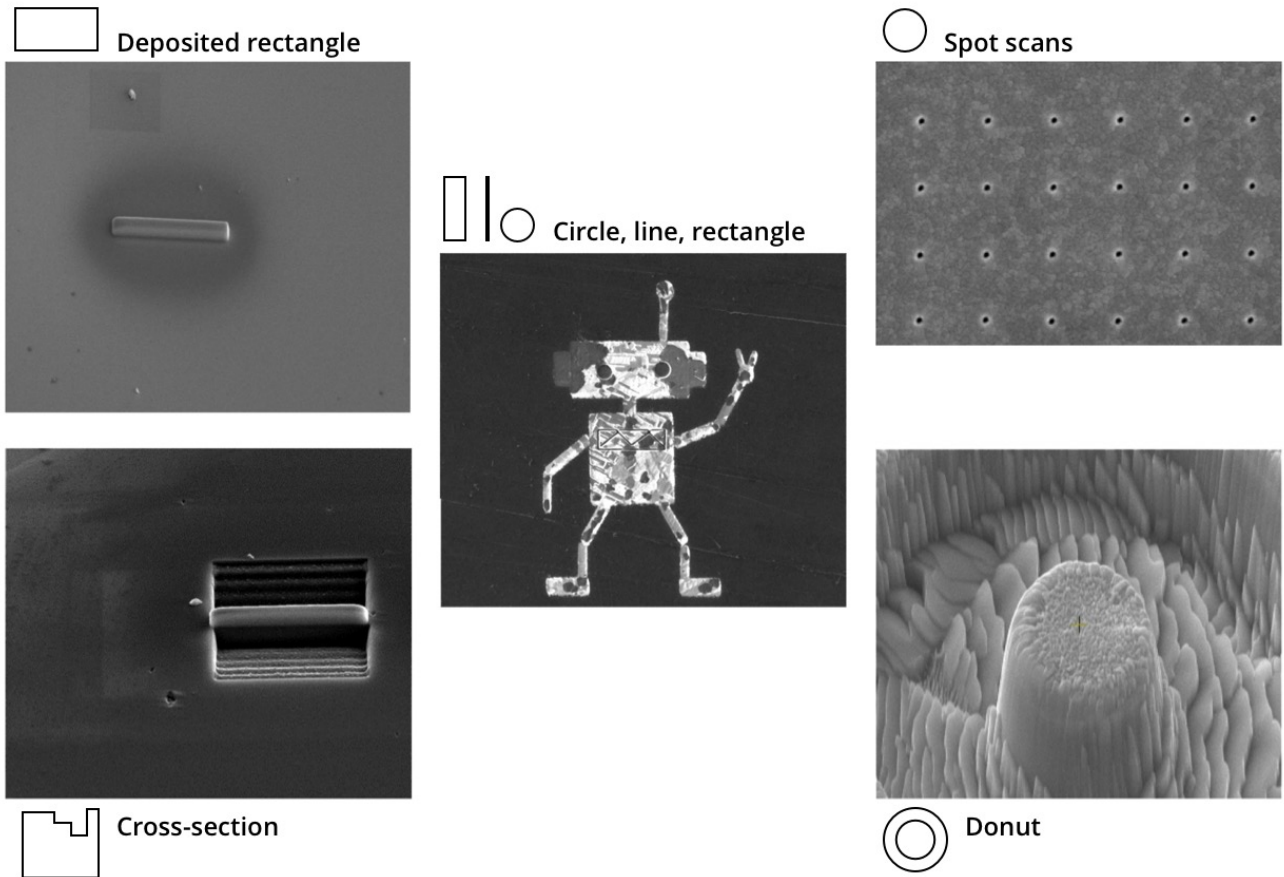
Destructive Imaging

Ion beam imaging is destructive! A single scan with the ion beam can severely damage the sample and introduce artefacts, as shown in the SEM image of a Au on C sample, imaged with Ga^+ ions, 30kV, 50pA, 30μs dwell time, 1024x884 resolution, 1 frame. How excessive the sample damage is depends on the sample. Sample damage can be reduced by limiting the ion dose (reducing the number of scans over the sample, reducing the ion beam current, or zooming out). Sample damage can be avoided by using the electron beam instead of the ion beam when possible, and utilising the beam blanker.
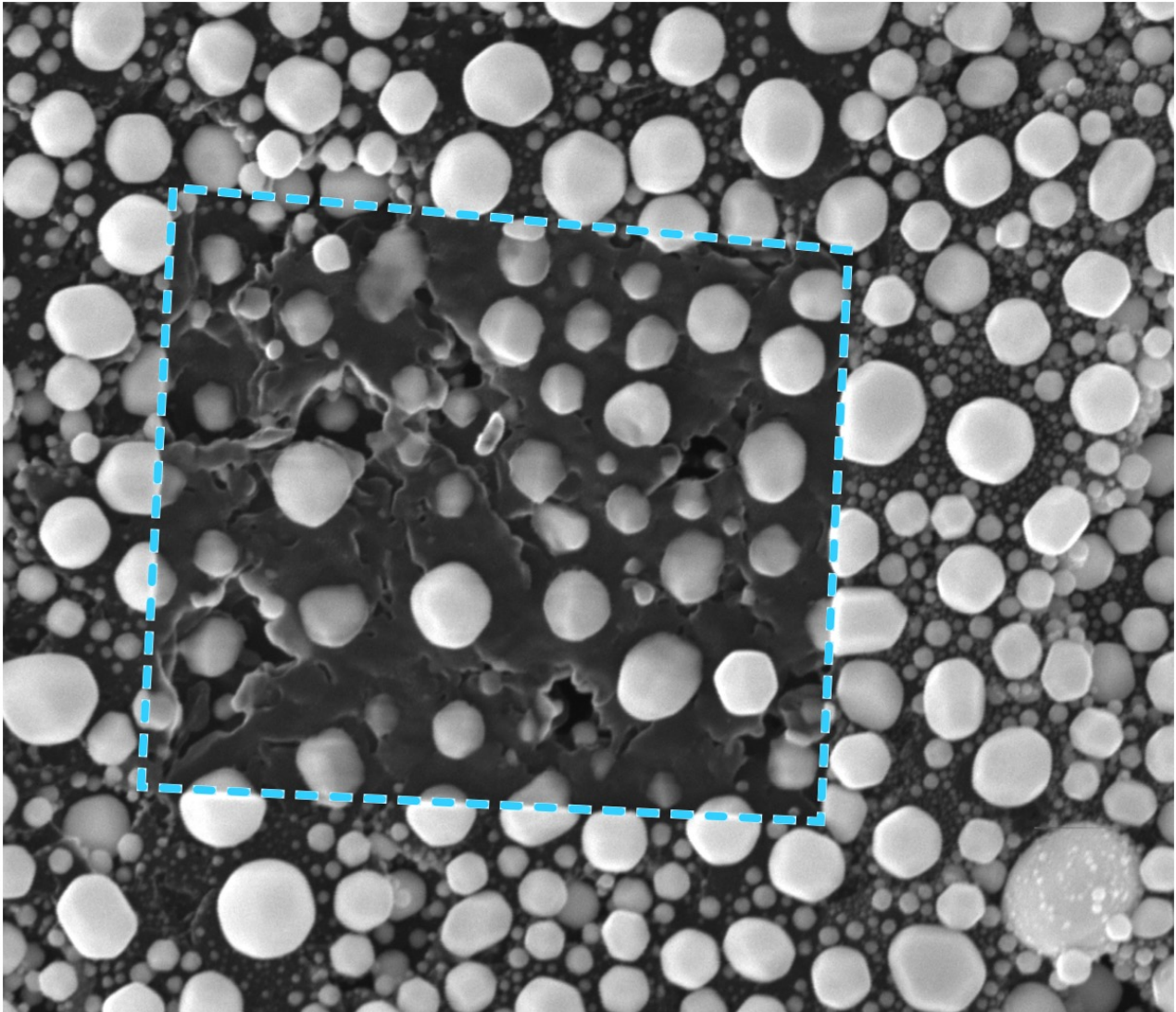
Milling - Patterning Mechanism

Patterning is the process of creating micro- to nanosized structures in/on the sample via ion beam milling or gas assisted deposition. Any complex structure can be created from basic building blocks (lines, spots, rectangles). In addition, stream files or bitmaps can be milled. Complex structures can be scripted and automatically performed by the system without an operator on site for a lot of FIB-SEM systems. Examples of structures, ranging from platinum rich deposition, to nanopore arrays, donuts, cross-sections and multiple structures like the robot are shown in the image. During patterning, the ion beam is (vector-) scanned across the sample surface. The beam stays in one scan point for a designated time (dwell time) before it moves on to the next spot (equivalent to the electron beam scanning). The rate and precision of the milling is determined by the ion beam current.

Deposited rectangle

Circle, line, rectangle

Spot scans

Cross-section

Donut

Scan point

Overlap

$t_{Dwell\ time}$

R_{proj}

Patterning

Patterning Parameters

Several ion beam patterning parameters must be set to achieve optimal results: acceleration voltage, current, dwell time/passes, pitch/overlap, scan direction, stage tilt, beam alignments and serial/parallel patterning for multiple patterns.

Patterning Parameters - Acceleration Voltage

The acceleration voltage determines the ion's energy. The higher the acceleration voltage (e.g. 30kV), the higher the ion's energy (30keV). At higher energy the ions penetrate deeper into the sample and result in a larger interaction volume (the volume in which the ions interact with the sample). The ion–solid interactions cause amorphisation. Therefore the larger the interaction volume, the thicker the amorphous layer. The TEM measurements illustrate the amorphous layers created depending on the acceleration voltage. 30kV acceleration voltage provides the best resolution and is conventionally used for nanostructuring/patterning purposes. Lower acceleration voltages (5kV, 2kV if available) are used for polishing samples (TAP and TEM-lamella preparation) to reduce the thickness of amorphous layers.
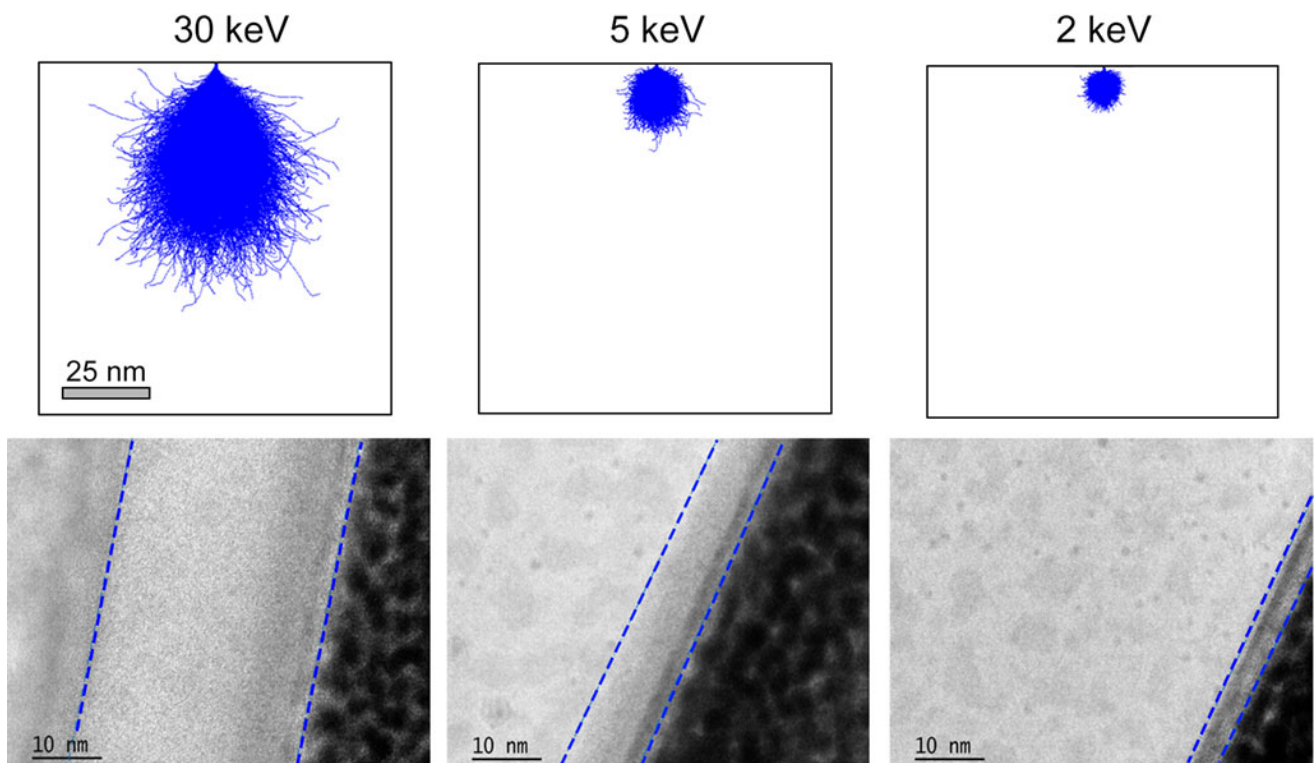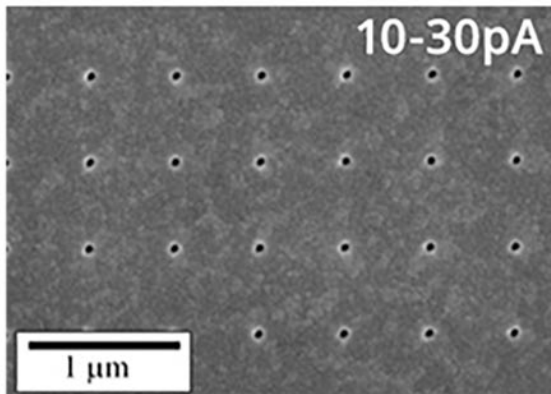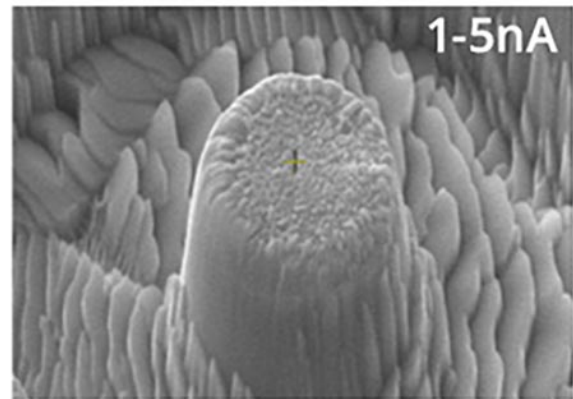
Aperture/Beam current

The ion beam current is chosen (via the beam selecting aperture strip) depending on the desired structure size. Smaller ion beam currents have a smaller final ion beam diameter and allow you to structure finer features such as precise nanosized patterns. Smaller currents, however, are insufficient to create larger structures and lead to excessive structuring times. Larger currents are used to structure larger areas.

The optimal beam current is one that allows you to mill features with high enough precision within a reasonable time frame with few artefacts!
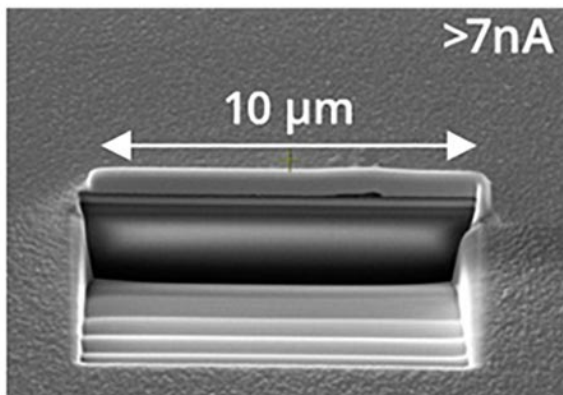
Passes/Dwell time

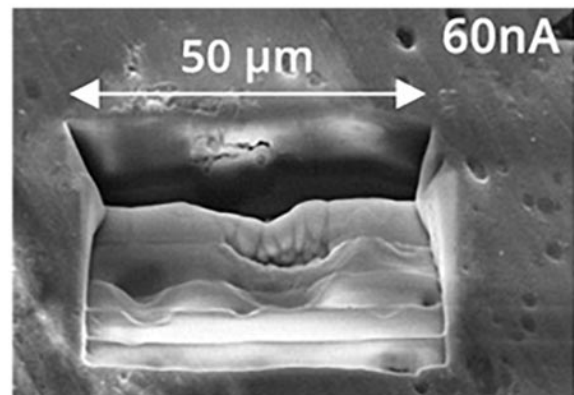The dwell time (or scan speed) sets the time that the ion beam remains in one spot. The dwell time is linked to the passes: the shorter the dwell time the more passes are needed to cut to a specific depth. Passes determine how often the ion beam is scanned over the entire area. A single pass only scans over an area once. Multiple passes allow the beam to have a shorter dwell time per spot but the area is scanned multiple times instead of just once. Shorter dwell times (~1μs) are selected when the sample shows heat damage or when redeposition needs to be reduced (ion beam mills over the redeposited areas and reduces redeposition). Longer dwell times are used when fast milling is desired or redeposition is not an issue. The long dwell time and single pass option means that each point is scanned until the desired milling depth is achieved. The next point is then milled to the desired depth and so forth. This means that the beam is slowly progressed along an edge and the resulting incidence angle (non-zero) leads to an increased sputtering yield (faster patterning). Redeposition into the already milled areas will occur and is more dominant for this setting. The multiple passes and short dwell times option means that the ion beam only mills each spot to a specifc depth and then moves on the next spot. The sample is milled down layer by layer.

$t_{= 30 \mu s}$ $t_{= 1 \mu s}$

1024   1024

884   884

1x   30x
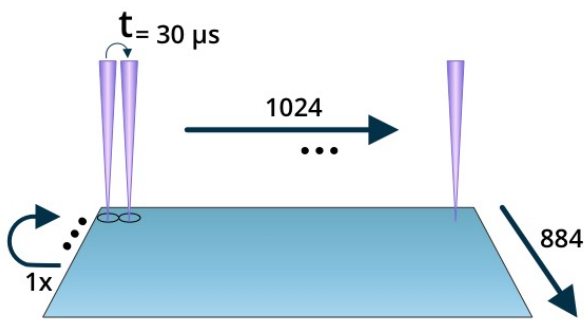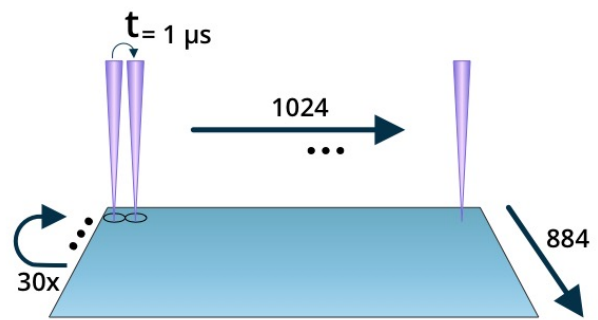
905216 points scanned 1x   **905216 points scanned 30x**

**Total time: 27s**   **Total time: 27s**
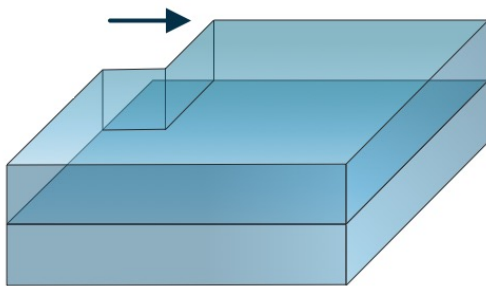
Single pass & long dwell time   **Multiple passes & short dwell times**
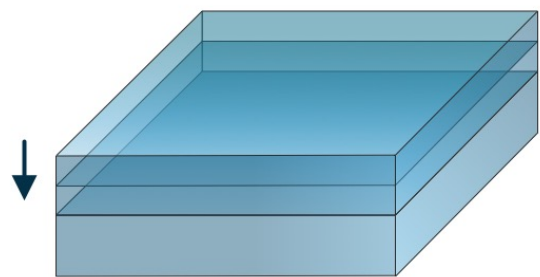
Overlap/Pitch

The ion beam conventionally overlaps 50% between the scan points to ensure a smooth milling edge (see top illustration and SEM image). When reducing the overlap (when using the GIS for deposition or to reduce the ion beam induced heat into the sample), the cutting edge is less smooth. It looks rippled, like in the bottom illustration and SEM image (highlighted by white arrows).
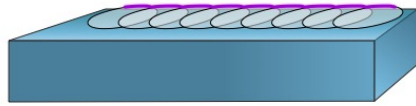
50% Overlap
(conventional)
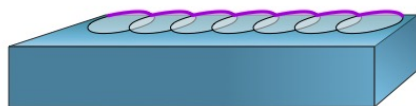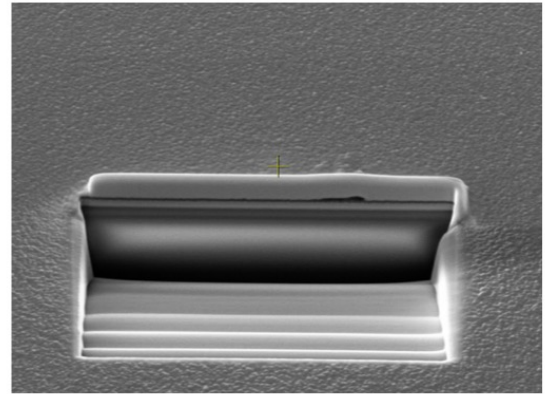
20% Overlap
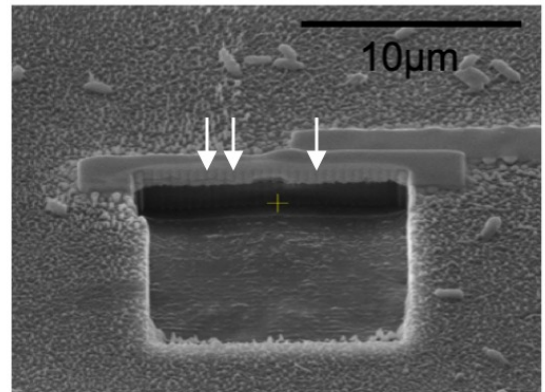
Scan Direction

The scan direction is used to control the direction of redeposition. Redeposition of the sputtered sample atoms occurs at the backside of the scan direction (where the scan started) and is indicated by the purple lines in the illustrations.

Scan direction: Top to bottom

Scan direction: Bottom to top

Beam Alignments

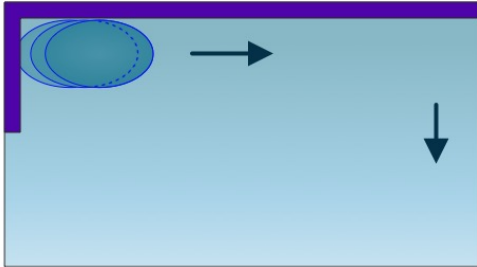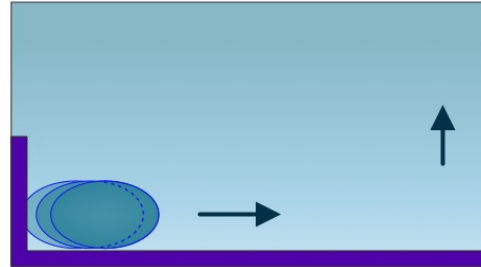When patterning, a well aligned, focused and non-astigmated beam is absolutely essential. The structures will turn out distorted otherwise. An example of a rectangle, milled with an aligned beam and unaligned beams shows the effect of beam alignments on pattern quality. Tips and tricks: after focusing the beam, perform a spotscan for a few seconds. The created spot pattern directly shows the beam shape. The pattern should be round and small (right image). If the pattern is distorted to an ellipse, the beam is still astigmatic. If the spot pattern is large, then the beam is not well focused.

Aligned beam

Unfocused beam   **Stigmated beam**   **Stigmated and unfocused beam**
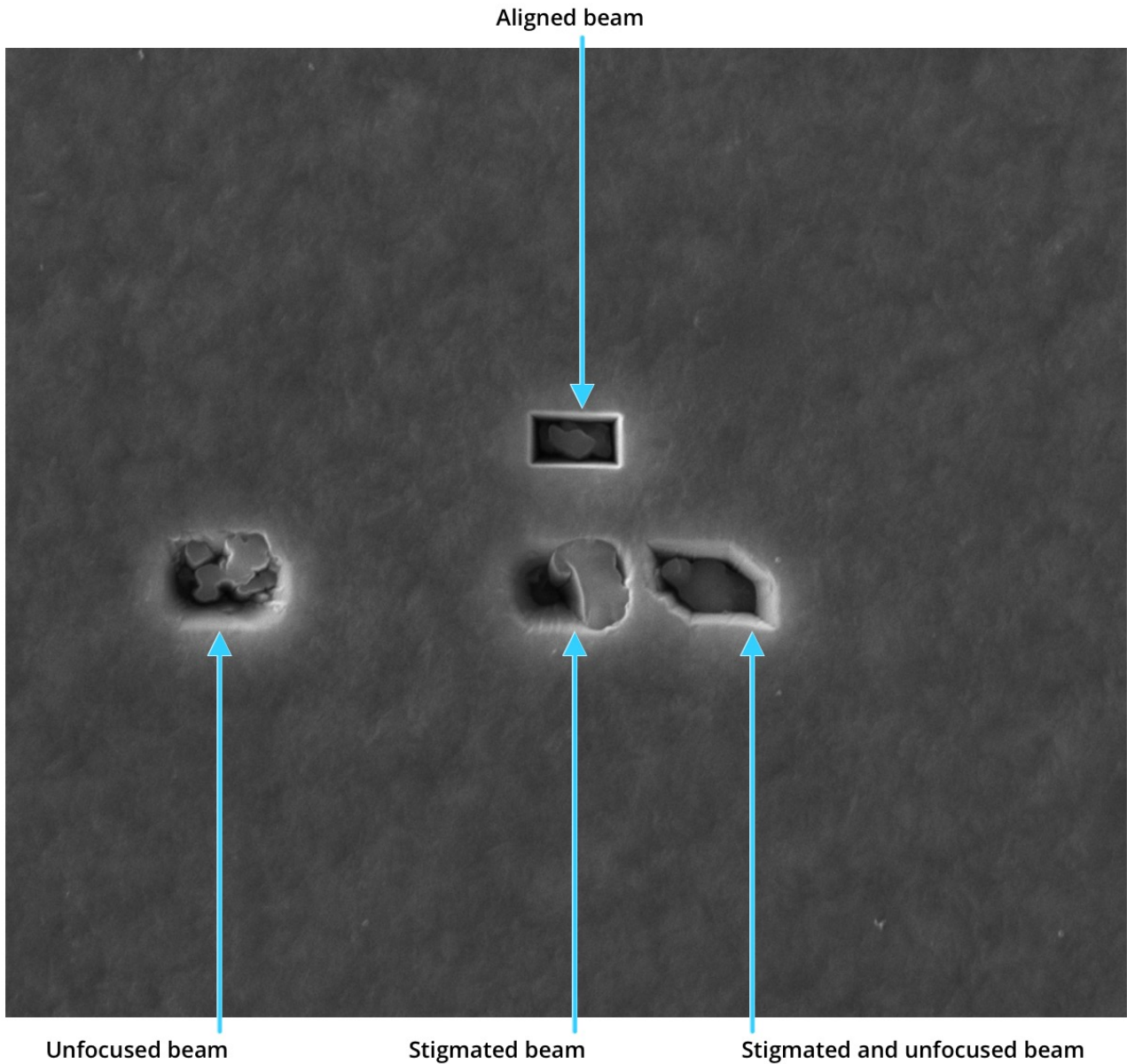
Artefacts - Introduction to Artefacts

Several artefacts occur during FIB operation that can affect structure quality, results and success. The most commonly observed artefacts are curtaining, redeposition, ion implantation, phase transformations, amorphisation, interface mixing, heating, uneven milling due to materials interfaces or grain boundaries.

Curtaining

The sputtering yield depends on the incident angle. Topographic features and voids within the sample create different incident angles for the ion beam, and hence different sputtering yields. This is illustrated in the schematic. The increase in surface roughness leads to an artefact called curtaining which produces vertical lines at the cross-section surface (see SEM image) due to the different milling speeds for different angles.

Curtaining can be reduced/removed from cross-sections by using GIS deposited smooth layers on the sample surface in addition to using lower ion beam currents when cutting cross-sections and by polishing cross-sections from different angles. Commercial rocking stages (that can be mounted onto the stage) tilt the sample during the cross-sectioning process and remove curtaining most efficiently. Curtaining, arising due to porous samples, can be reduced by additionally embedding the sample in resin, as the resin fills the pores that cause the curtaining.
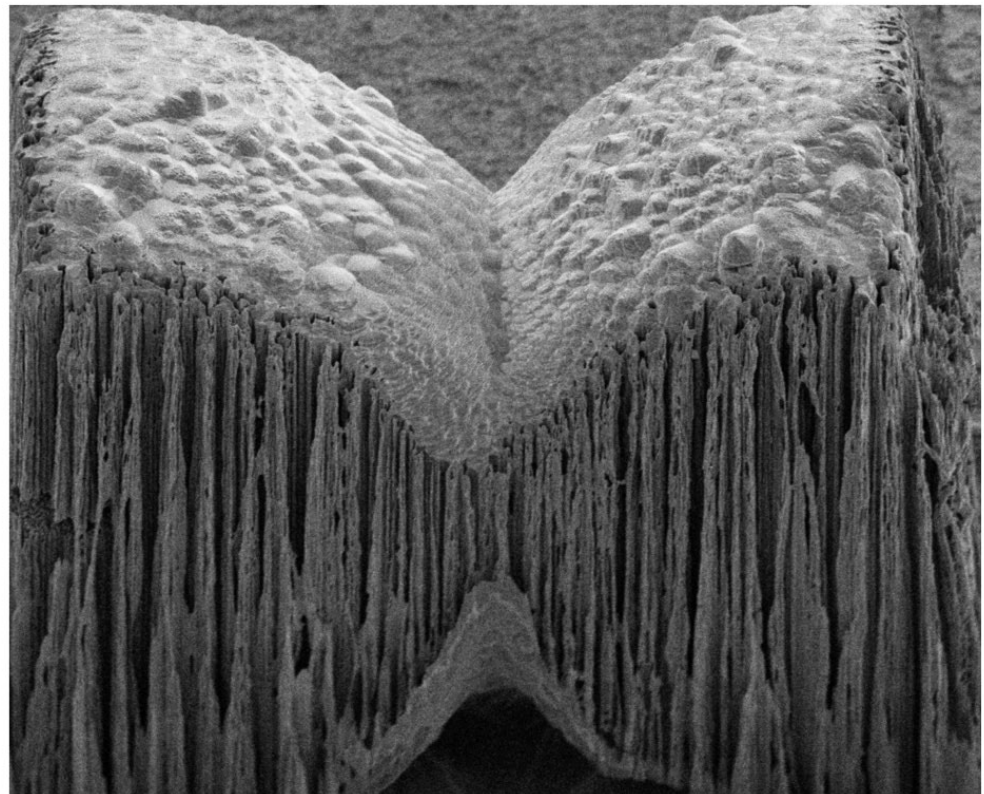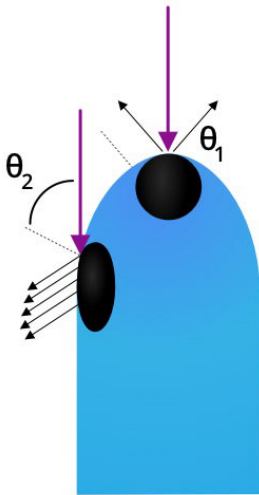

Sample courtesy Ruohui Lin.

Redeposition

Redeposition of the sputtered material occurs during the FIB milling process. Sputtered sample atoms can deposit back onto other sample areas, this can cause undesired topography and is particularly prevalent at high ion beam currents and voltages. Redeposition is enhanced by an increased sputter rate, and becomes worse when milling in confined areas or when milling high aspect ratio structures. This redeposition process limits the possible aspect ratio of nanostructures.

Redeposition is more dominant in soft, fast milling materials. A direct comparison can be seen in the image on the right where the faster milled material on the right shows slopes on the sidewalls that are caused by enhanced redeposition. This reduces structure quality.

Redeposition can easily be seen when milling high aspect ratio structures like nanopores or the J-cut in TEM lamella preparation. When milling high aspect ratio structures, the probability of sample atom removal deep within the structure decreases as the structure depth increases and eventually limits the possible structure depth that can be achieved. For TEM lamella preparation, the J-cut represents a confined area and the lamella can be glued back into the sample due to redeposition. To avoid this issue, J-cuts are usually cut in parallel mode (to avoid redeposition of a serial second cut in the previous cut). The most likely redeposition area can be anticipated via the milling direction. Redeposited material is most commonly found behind the milled pattern (if milling from left to right -> on the left, if milling bottom to top -> on the bottom).
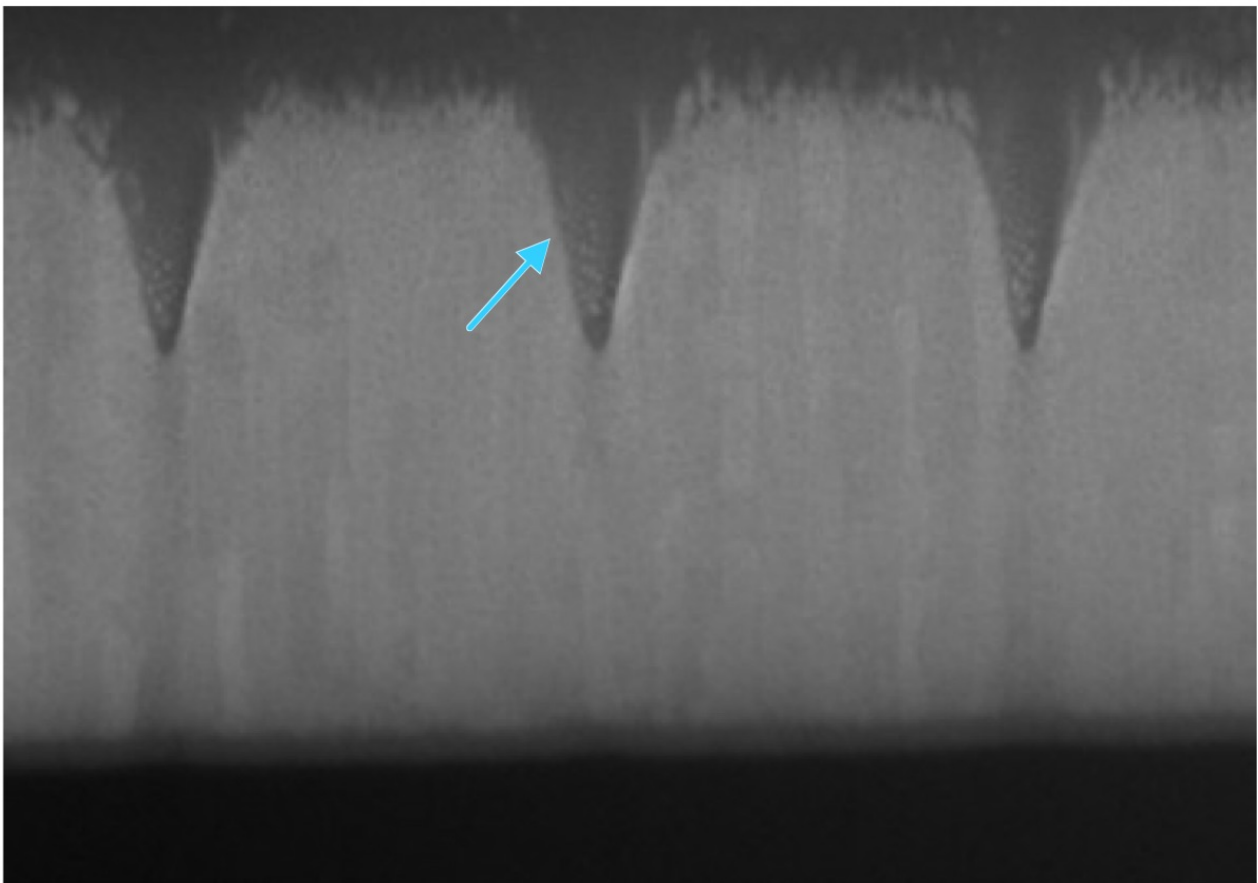
Implantation

The ion beam ions get stuck in the sample after losing all their energy in the ion–sample atom interactions. This is an undesired artefact. In semiconductors, the gallium ions are a dopant that strongly alter the physical properties of the sample (e.g. conductivity). The EDX spectrum of a TiSiSiO2 on multilayer TEM lamella on Cr/Fe/Ni/Al substrate shows the implanted gallium from the ion beam. The concentration of gallium dopant is limited in FIB-SEMs due to the simultaneous sputtering. 1at% to 50at% Ga are expected.

Different ion species, like xenon are now available to avoid the issues of gallium contamination. The different ion species are still implanted in the sample, but may not dope the sample and change the physical properties, like gallium does for semiconductors.


EDX Spectrum FIB TEM-Lamella

Implanted ions

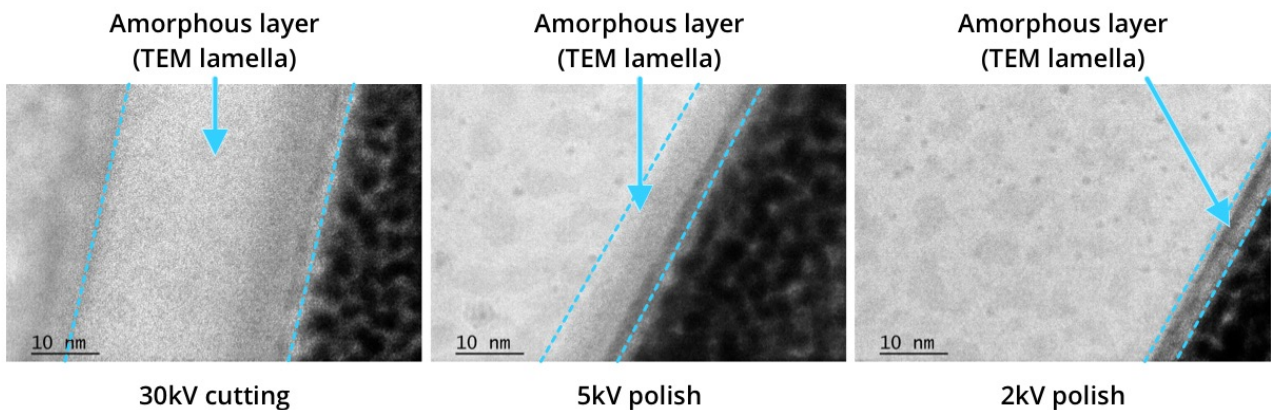Sample atoms
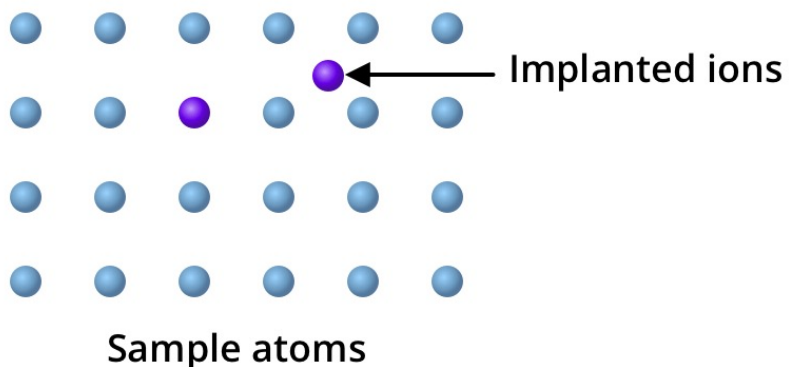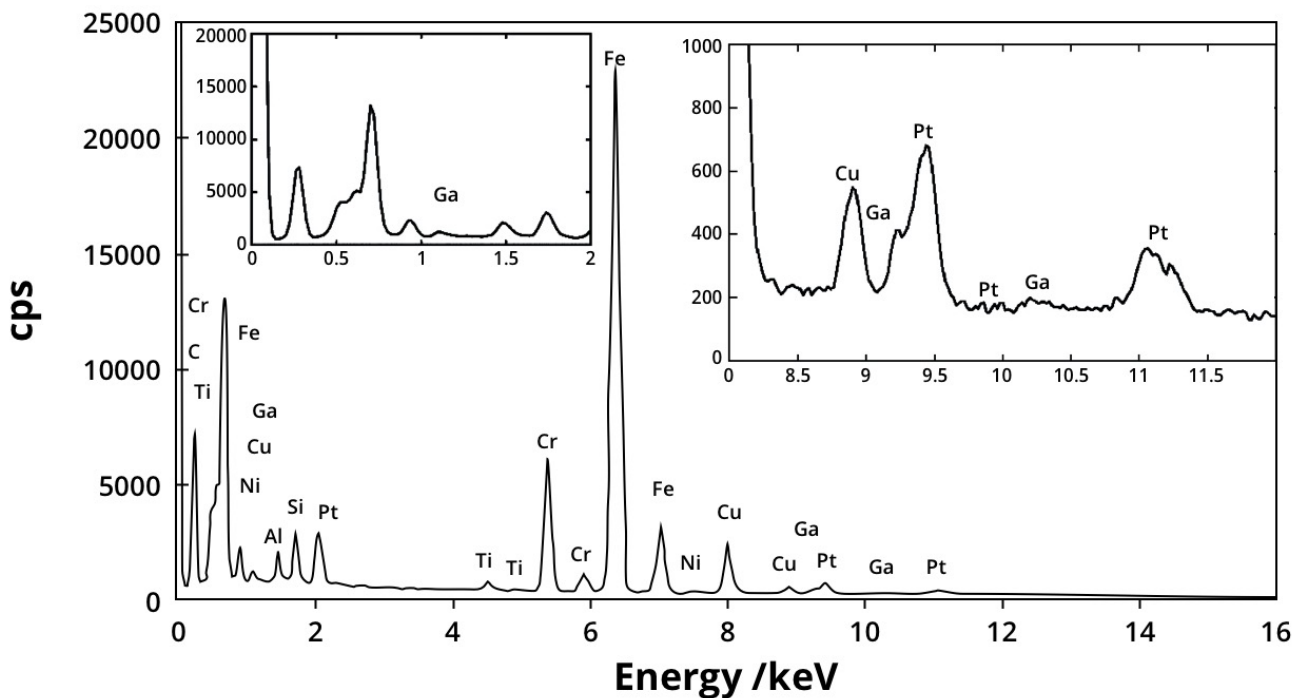
Phase Transformations

Ion beam irradiation can induce phase transformations in samples. An example of an ion-beam-induced phase transformation (appearing as dark patches) when working with copper is shown in the SEM images. It is worthwhile checking if these phase transformations occur especially when polishing for EBSD measurements to avoid misinterpretation. Transformation of austenite to ferrite is commonly observed in stainless steels during FIB milling.

t=0min   t~1min   t~3min
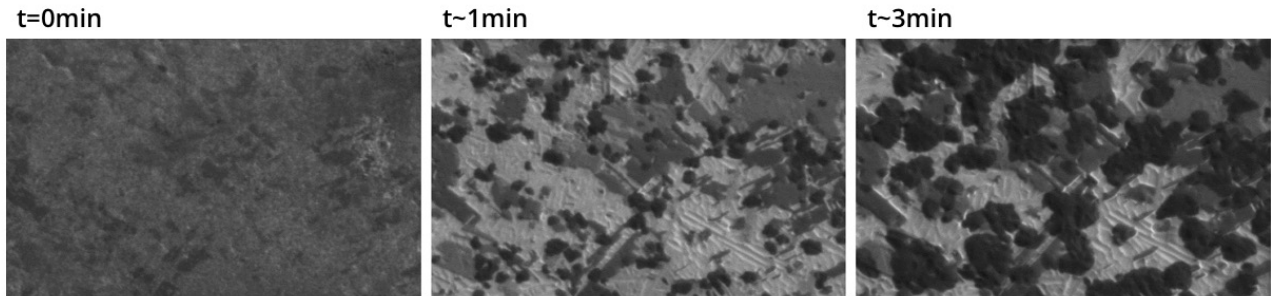
Amorphization

The ion beam–sample interactions lead to sample atom displacements, which means that sample atoms are dislodged from their original crystal lattice position. With sufficiently high doses (number of ions per area) a lot of sample atoms are displaced in the first few nanometres of the sample and the sample crystallinity can be lost, resulting in amorphous layers.

The TEM image shows an amorphous layer that formed during TEM lamella preparation when using 30kV Ga^+ ions in Si (left), after a 5kV polish (middle) and after 5kV and 2kV polish (right). The images show that the thickness of the amorphous layer can be greatly reduced when using lower energy ions. As lower energy ions have shorter ranges, the amorphous layer is reduced.

Amorphous layer   Amorphous layer   Amorphous layer
(TEM lamella)   (TEM lamella)   (TEM lamella)

30kV cutting   5kV polish   2kV polish

Interface Mixing

Interface mixing occurs in layered structures when atoms from 1 sample layer are transported into the next sample layer. Ions transfer enough energy to recoil atoms in layer 1 that can then move into layer 2, leading to layer contamination. Atoms can also be recoiled towards the surface.

The TEM image shows the intermixing of 2 Pt layers (electron beam deposited Pt and ion beam deposited Pt). The damage layer is ~50nm thick.
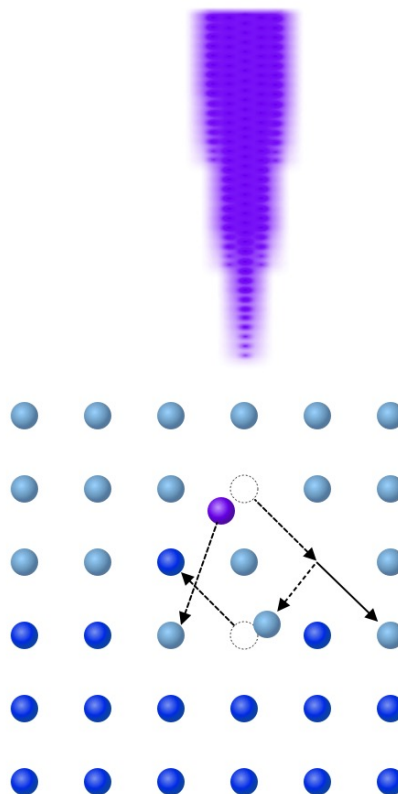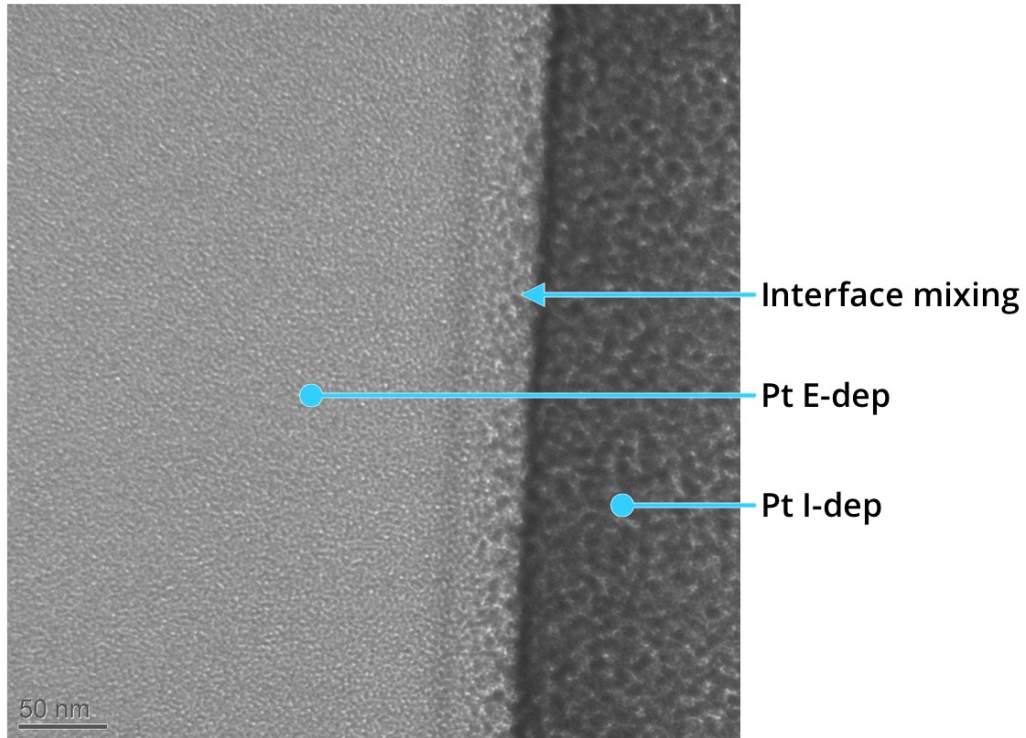
Heat damage

The incident ion beams energy is ultimately deposited in phonons and converted to heat, inelastic electronic energy losses also contribute to this. The temperature difference that is caused by the ion–solid interactions depends on the sample's thermal conductivity, size and geometry, and the ion beam characteristics. For Si, the temperature increases less than 2°C during a standard FIB milling procedure, so heating effects are often neglected by experienced FIB operators. Biological samples or polymers are more prone to heating and very high temperatures can be reached in such samples. Some of the damage issues mentioned here have driven the development of new technologies such as cryo-FIB which can assist in minimising these artefacts.
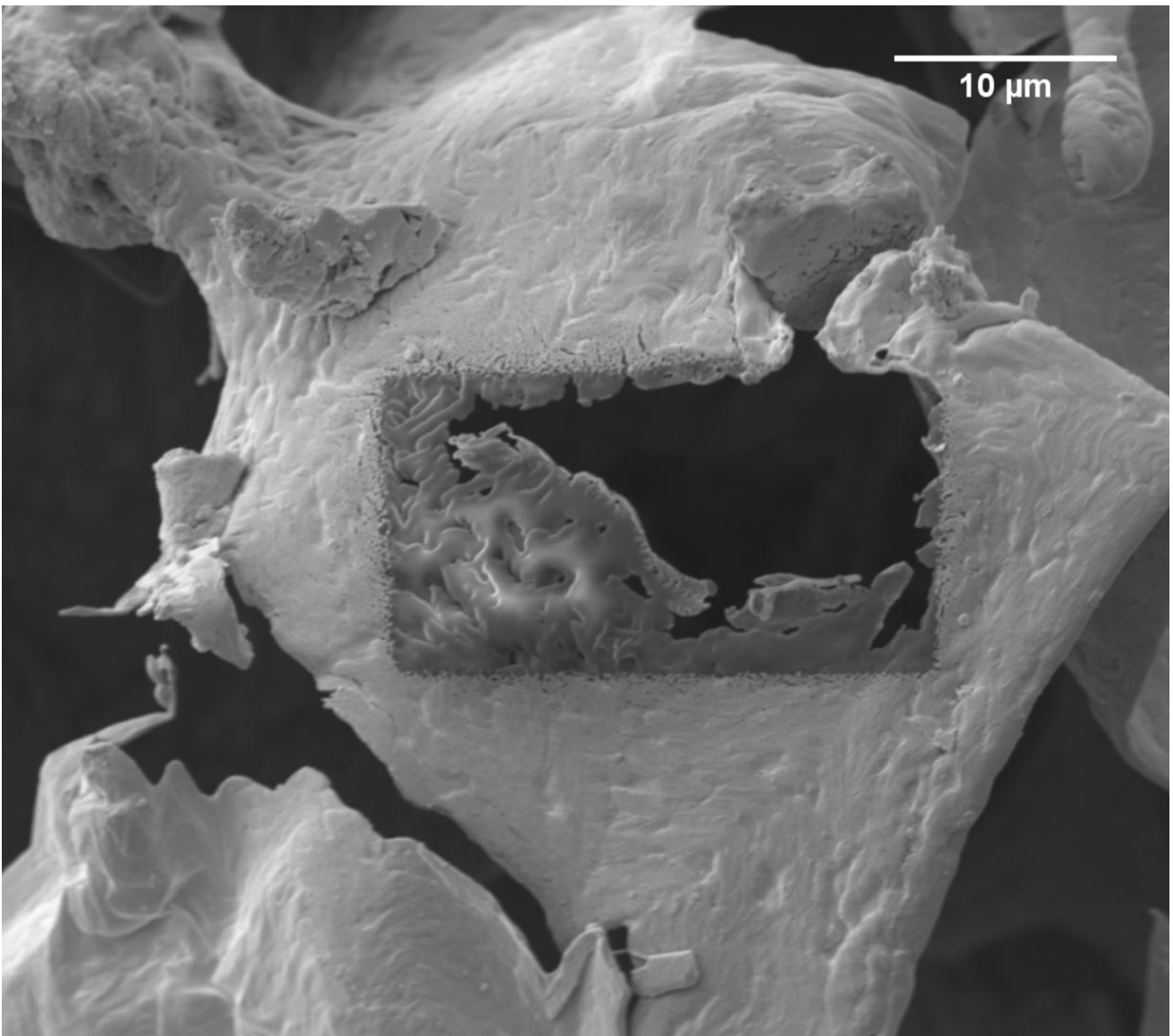

*Heat damaged collagen, cut with Ga ions.*

Channeling

Channeling occurs for polycrystalline samples, such as copper, which has many different grains with different orientations. Ions penetrate deeper into low index directions (these directions have a lower density of atoms) and these will thus not produce as much signal or sputter as much, the latter resulting in uneven milling as shown in the SEM images.

Patterns that are structured across several grains often display different depths and structure qualities, as shown in the ion beam image on the left. When preparing TEM lamellas of polycrystalline material, different lamella thicknesses (right SEM image) and loss of areas can result.

Faster milling grain.
Lower structure quality
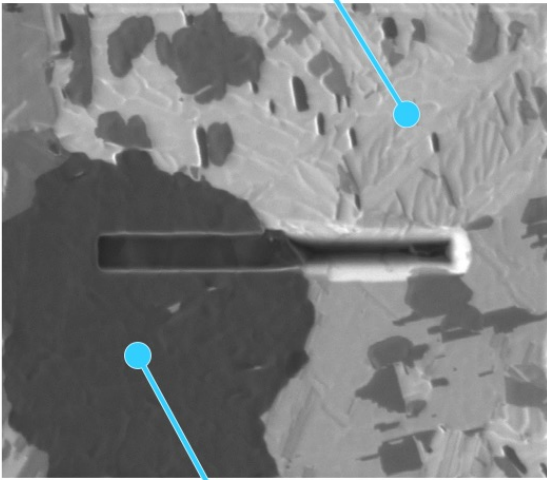
Slower milling grain.
Higher structure quality
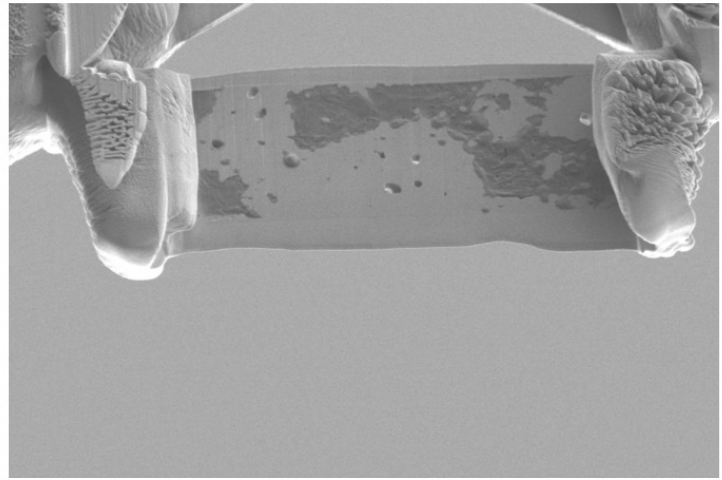

Uneven TEM-lamellas

Deposition

During deposition, the GIS releases a gaseous precursor close to the sample surface. The deposition of the platinum rich precursor is illustrated here as an example. The precursor molecules can be cracked with either the electron beam or the ion beam. The nonvolatile product (carbon rich platinum in this example) adsorbs onto the sample surface and forms the deposited structure. The volatile products (predominantly hydrogen) are removed by the pumps. To successfully deposit the precursor material, roughly the same amount of ions and precursor molecules need to be over the deposition area at the same time. To achieve this, the ion beam current needs to be selected depending on the deposited structure size (see Area 2 in the illustration).

Current too small:
If the current chosen is too low not all gas precursors are cracked which results in slower Pt-layer deposition rates, longer deposition times and leaves precursor molecules within the chamber (contamination). This corresponds to Area 1 in the illustration.

Current too high:
If the current chosen is too high, all gas precursors are used by only a few of the available ions and the remaining ions sputter the sample surface (milling a pattern instead of depositing). This corresponds to Area 3 in the illustration. When using ion beam deposition, some of the ions hit the sample surface and can damage the sample before the protective deposition layer is formed. To avoid this issue and to keep the sample surface protected, electron beam deposition is used. The precursor is cracked using the electron beam and the non-volatile part of the precursor deposits on the sample surface. This mode of deposition is less efficient, however, it protects the sample surface. Low energy electrons (~2kV) and high beam currents (~nA range) are usually used for this.

Rate (μm/min)

Area 1   Area 2   Area 3

$$\text{Current density}\left(\frac{pA}{\mu m^2}\right)$$
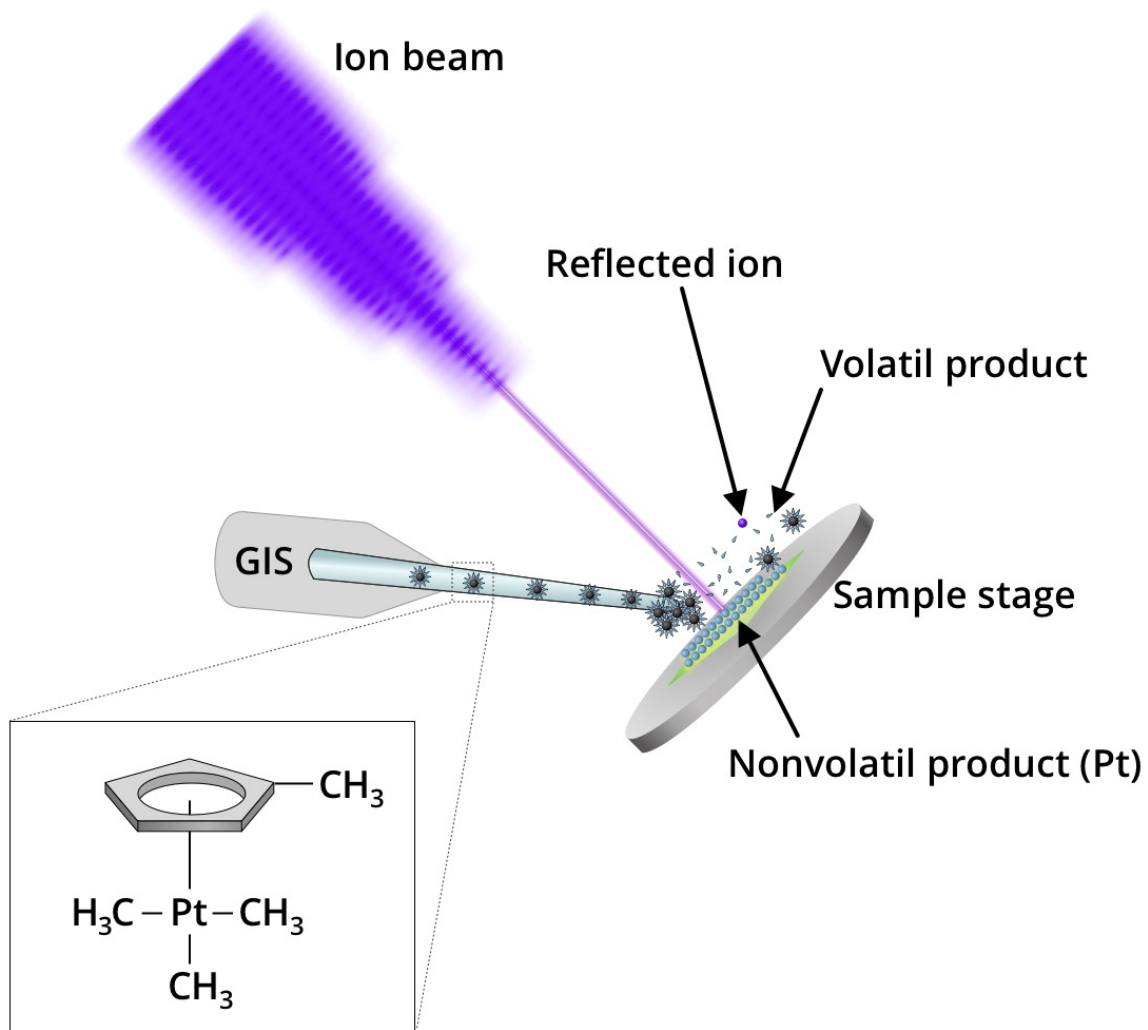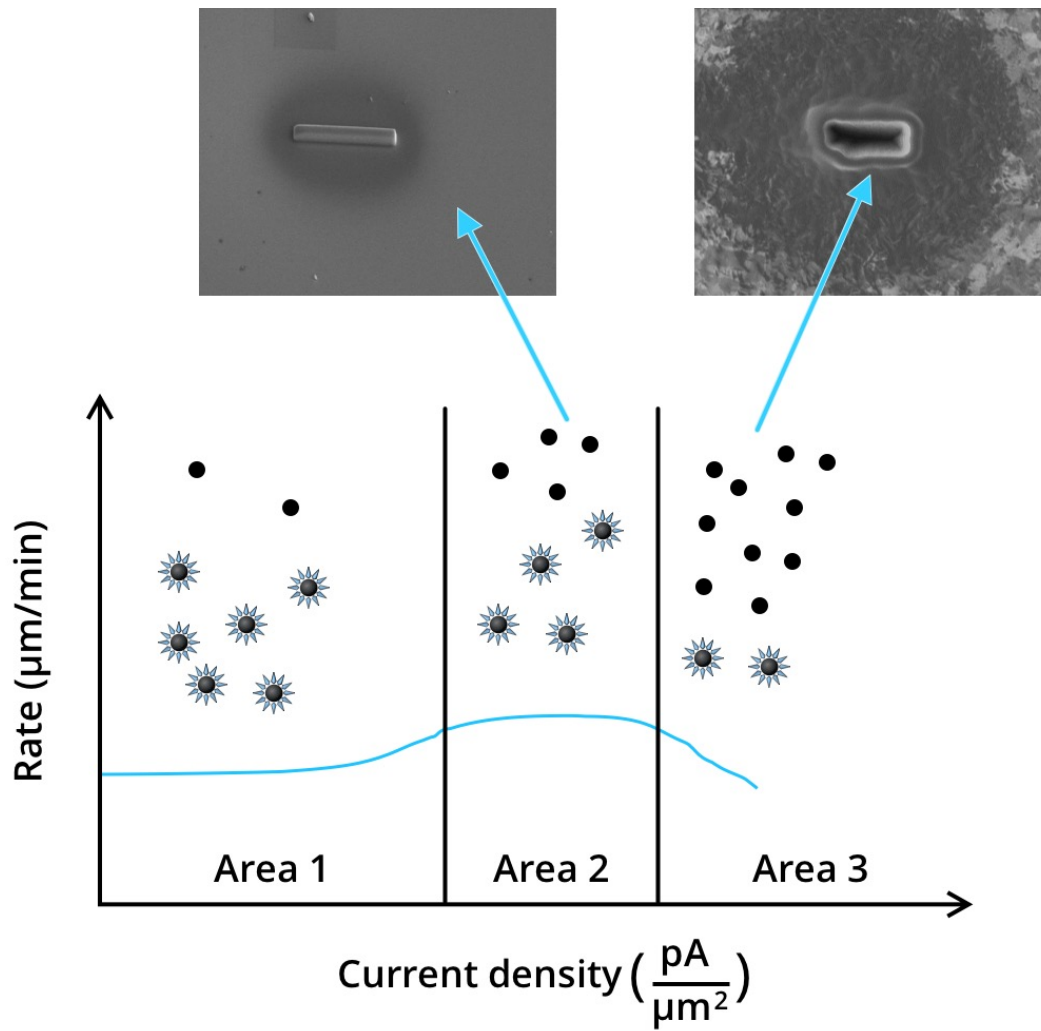
Cross-sectioning - Introduction to cross-sectioning

Cross-sections allow you to look inside the bulk sample and to reveal sample features that are hidden below the sample surface. FIB-SEMs have the unique capability to section and analyse samples at precisely selected points, which cannot be achieved by other techniques. For example, the SEM image on the left shows nanopores. The cross-section allows you to visualise the structure of the pores below the sample surface. When cross-sectioning, the area of interest is identified with the SEM. A protective layer (often tungsten, platinum or carbon) is then deposited onto the sample surface forming a smooth surface (to reduce curtaining) and protecting the sample surface from the ion beam during the cross-sectioning process. A cross-section is then cut with the ion beam to expose the sub-surface feature. The cross-section is finished off by polishing the cross-section surface with a smaller ion beam current.
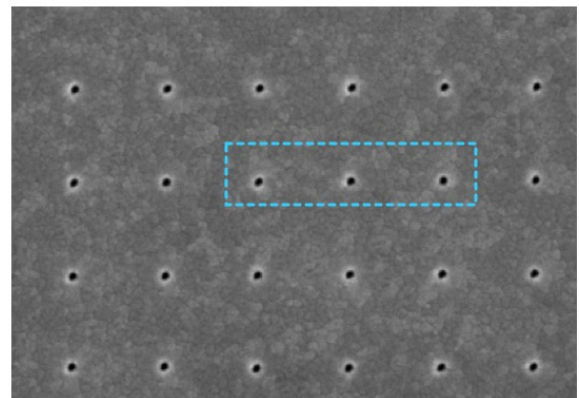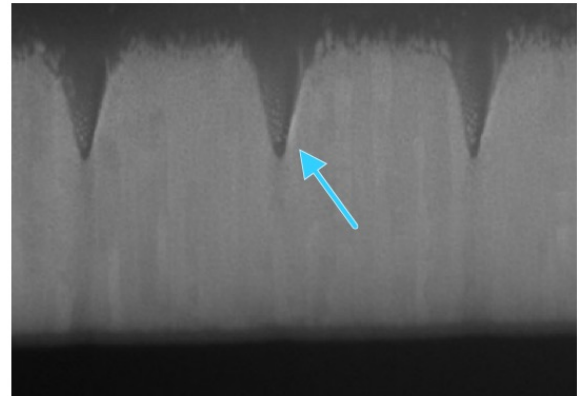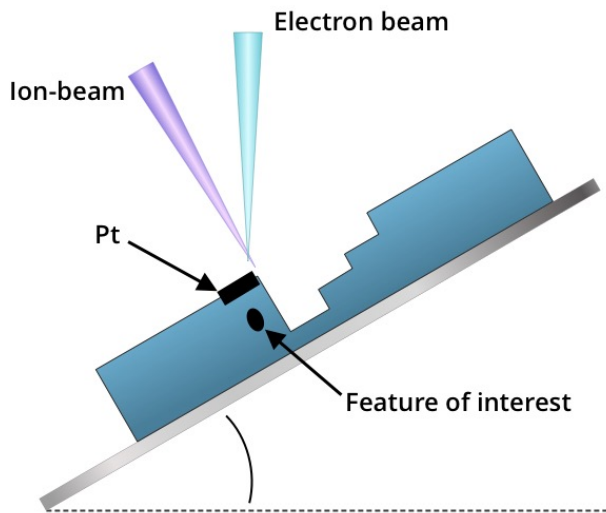

Tips and Tricks

It is important to cut the cross-section large enough to avoid shadowing and redeposition issues. As a rough guide the cross-section should be twice as wide in the y-direction than it is deep (z-direction). I.e. y = 2z

TEM-lamella preparation -
Intro to TEM-lamella preparation

TEM lamella preparation, preparing thin (<100nm) foils for TEM measurements, is one of the most difficult and complex applications for FIB-SEMs as it requires the operation of many attachments simultaneously. TEM lamellas are prepared with the FIB-SEM if the area of interest needs to be precisely selected with an SEM. The process can be separated into 6 individual steps:

1. Deposition of a protective layer (carbon or platinum deposition).
2. Prepare lamella via cross-sectioning.
3. J-cut.
4. Lift-out.
5. Thin-out lamella to <100nm thickness.
6. Polish lamella.
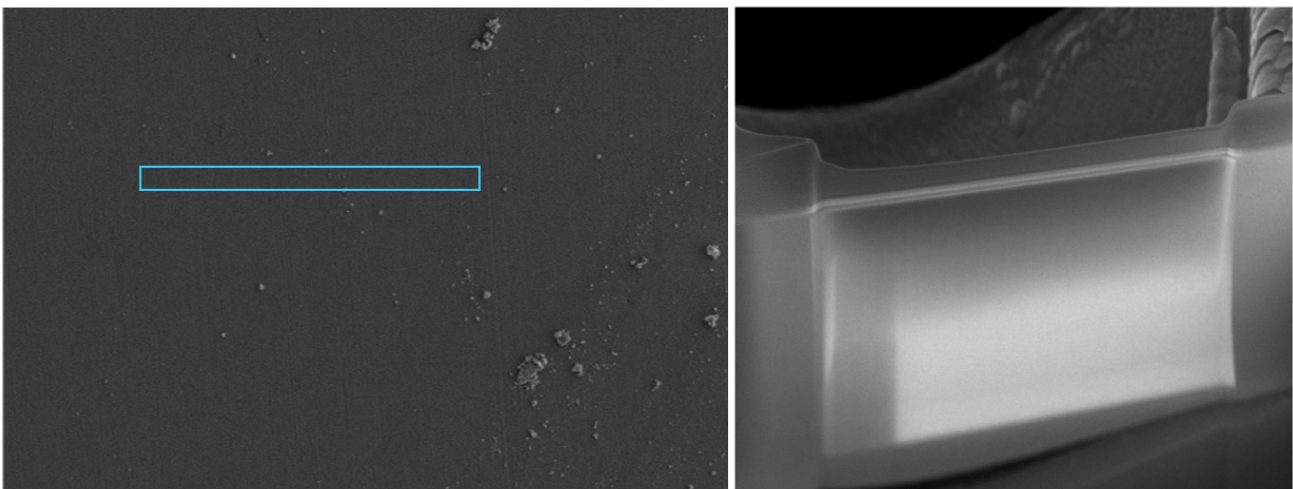
Process Steps - Deposition of protective layer

A thin protective layer (approx 500nm) is usually first deposited (if available) via the electron beam deposition. The sample surface is damaged during Ion beam imaging and this thin layer protects the surface area of interest during the ion beam imaging and ion beam assisted deposition process which are both for the lamella preparation. A thicker Pt protective layer (approx 1–1.5μm) is afterwards deposited on top of the thin protective layer via the ion beam (more efficient deposition)

Sample surface

3 μm

Pt deposit
(covers and secures area of interest)
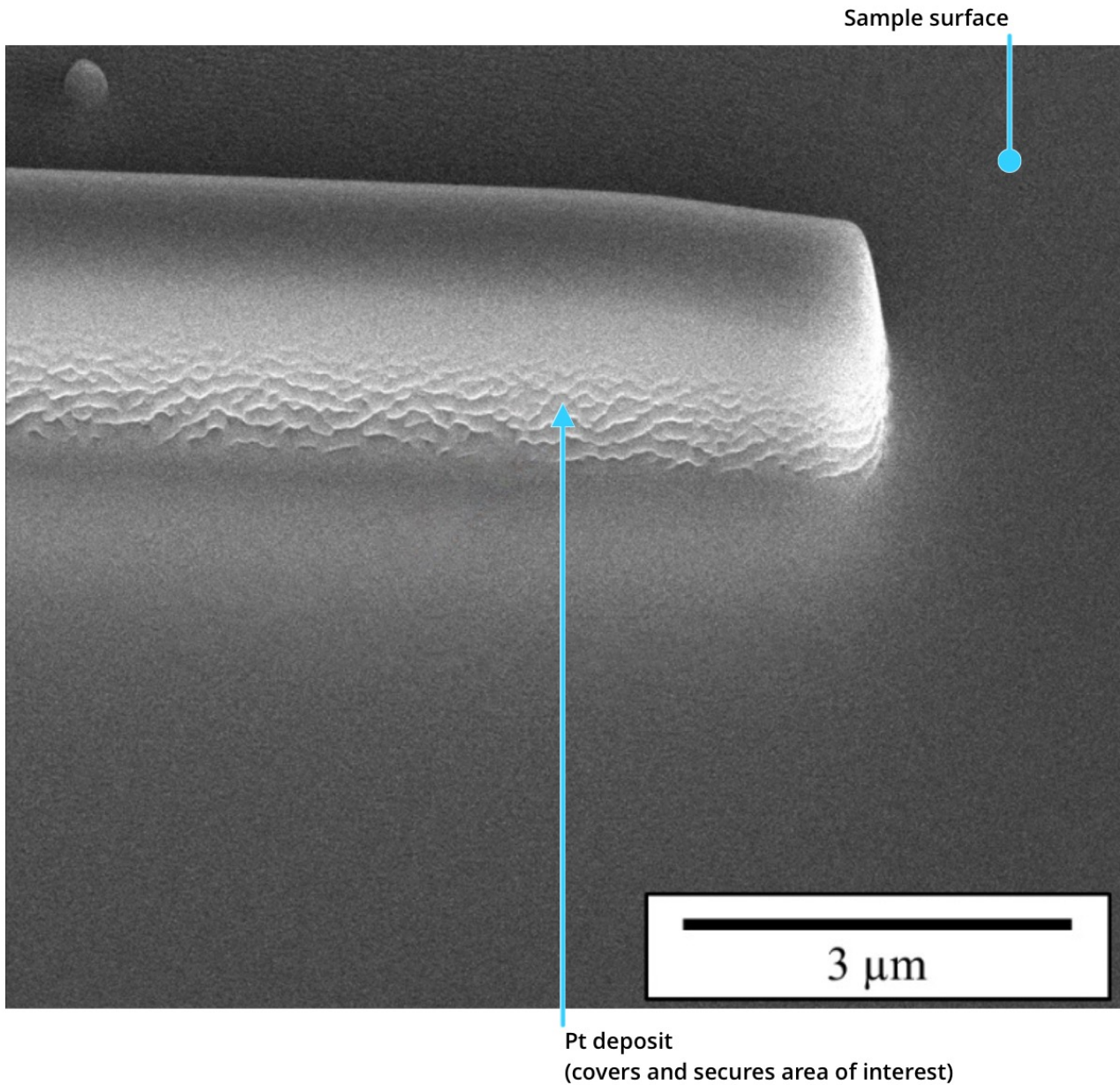
Prepare Lamella via cross-sectioning

Two cross-sections are cut above and below the deposited protection layer to cut out the area of interest from the bulk sample. This can be done in different ways, however, a common method is to cut the cross-section above the protection layer at slightly reduced tilt angles while cutting the cross-section below the protective layer at increased tilt angles. The slightly increased/decreased stage tilts are used to achieve more walls on the TEM lamella. Slightly sloped walls are created throughout the process due to the beam tails (and redeposition).

Cross-section 2

Cross-section 1

Area of interest below protective Pt layer
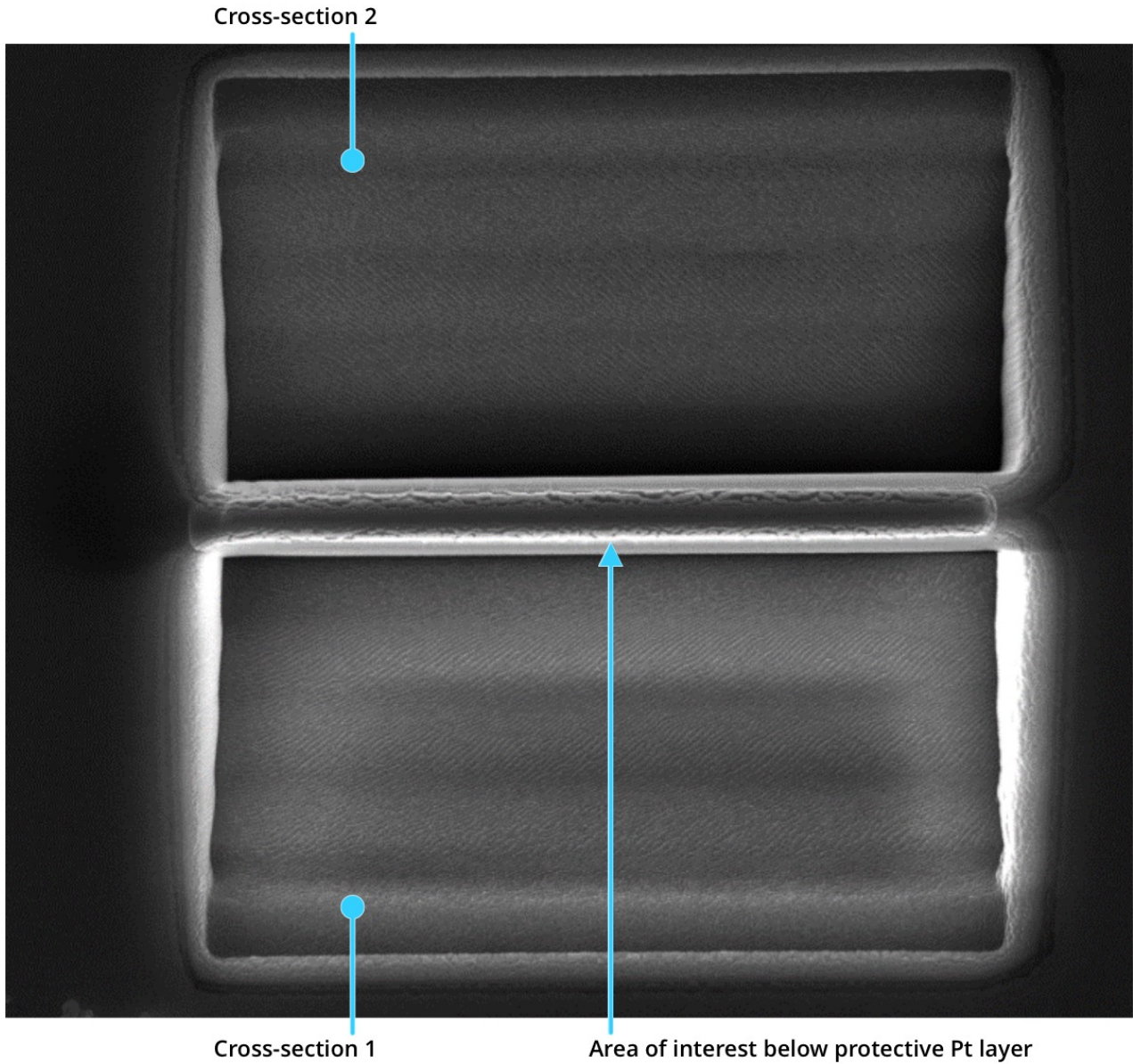
J-cut

A J-cut (as presented here) is used for in-situ lift-out to start cutting the lamella out of the sample, and must be made on the side of the specimen where the manipulator will be attached. Several rectangles are cut in parallel to achieve this. The quality of the J-cut often determines the success of the TEM lamella preparation: if the j-cut has not been entirely successful, the risk of losing the lamella during the lift-out is high. If serial milling is chosen then the rectangles are milled after one another and the previously cut rectangle can easily be filled again by depositing material (e.g. from a milling process close to the rectangle) which glues the lamella back into the sample. When all rectangles are milled parallel (simultaneously) the redeposited material is simultaneously milled away again and cannot glue the sample back together.

TEM lamella

Cutting edge

10 µm

20 µm
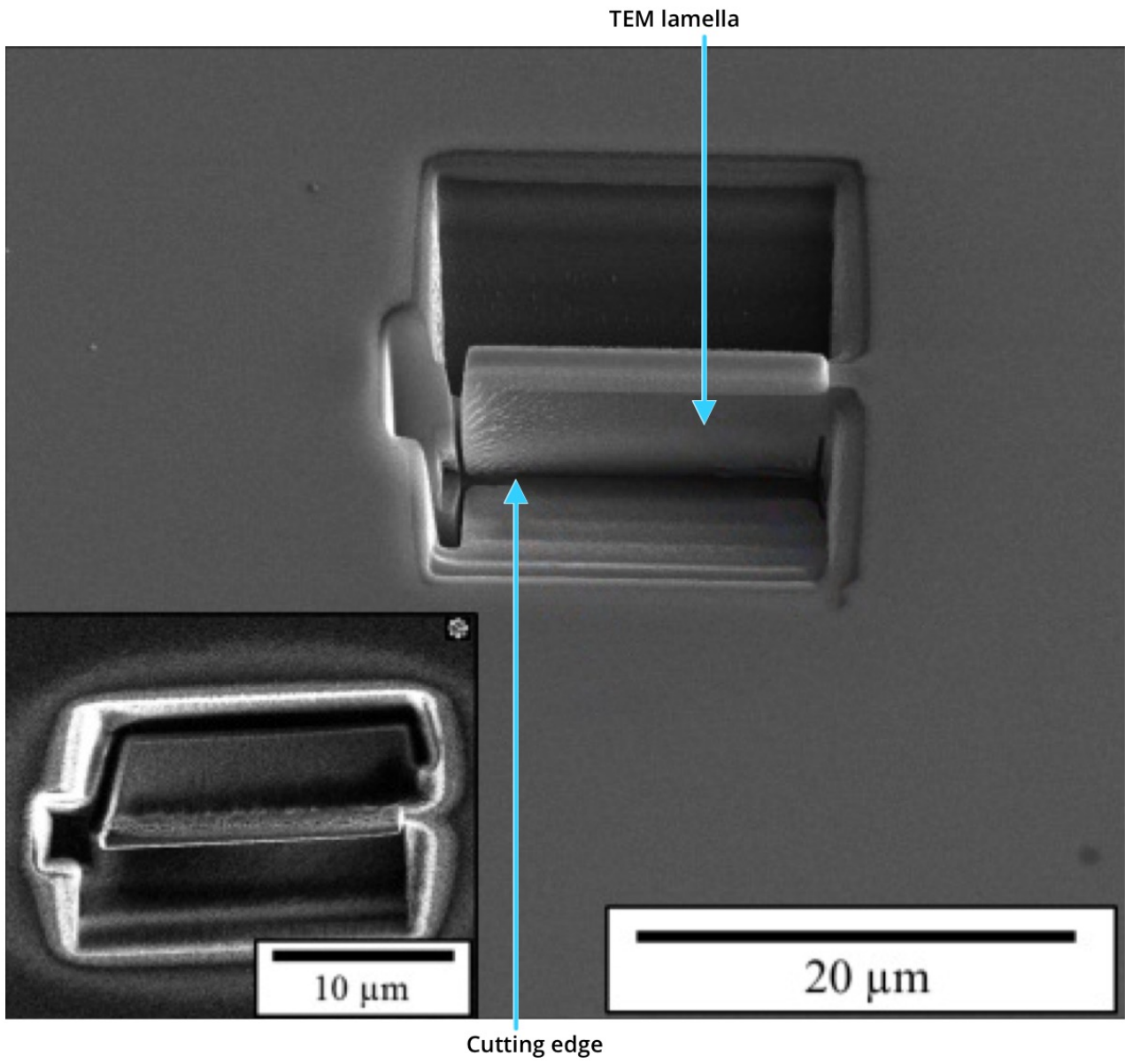
Lift-out

The GIS and the manipulator are used in combination to lift-out the prepared TEM lamella. During the lift-out process, a manipulator is inserted close to the prepared lamella and glued to the lamella using GIS platinum deposition. Once the lamella is attached to the manipulator needle, the lamella is cut out of the sample (left image). The prepared lamella can now be removed from the bulk sample by either rectracting the manipulator or by lowering the stage (centre image). The TEM grid is then brought into position and the the lamella is inserted into and attached to the TEM grid via GIS deposition (right image), while the lamella is cut from the manipulator using ion milling.
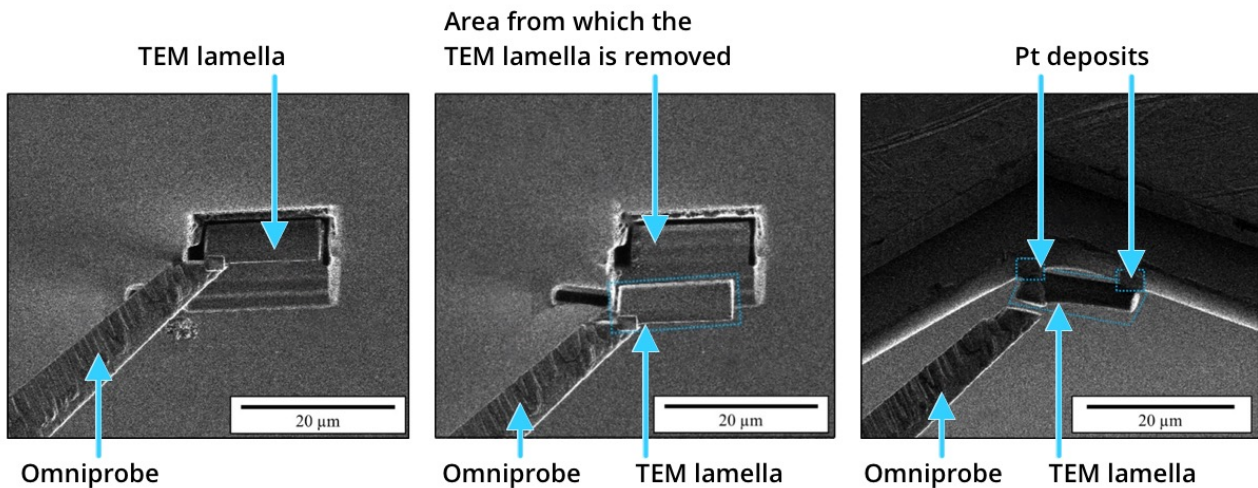

Operation procedures for the lift-out can vastly differ, depending on the FIB-SEM setup, and in some systems the manipulator may need to be sharpened before you begin. Detailed information TEM grid: Different TEM grid materials and designs are available including copper and molybdenum grids with either single post or triple post to attach lamellas. Detailed information TEM grid location: The TEM lamella can either be attached to the side of a post (preferred for energy dispersive spectroscopy measurements) or into a v-shape (more stable position, preferred for stressed/strained samples or long transports, redeposition of grid material on the TEM lamella can be a problem, especially when using a Cs corrected TEM for analysis).

Thinning

Once the TEM lamella is attached to the TEM grid, the sample is then thinned to <100nm thickness. A larger current is first used in combination with cleaning cross-sections to thin the front and then the back of the lamella down to roughly 600nm thickness. A smaller current is then used to thin the backside and then the frontside of the TEM lamella to electron transparency. At this stage, the majority of the protective layer has been removed in the process and a jagged edge is visible for the protective layer. Throughout this process, the sample is slightly tilted by a few degrees to minimise destruction of the protective layer during thinning/polishing.

The actual tilt angle depends on the sample material. If the top of the lamella is thinned but the bottom remains thick, the stage tilt is not large enough and should be increased. If the bottom of the lamella is milled but the top remains thick then then stage tilt is too large and the tilt angle should be reduced.
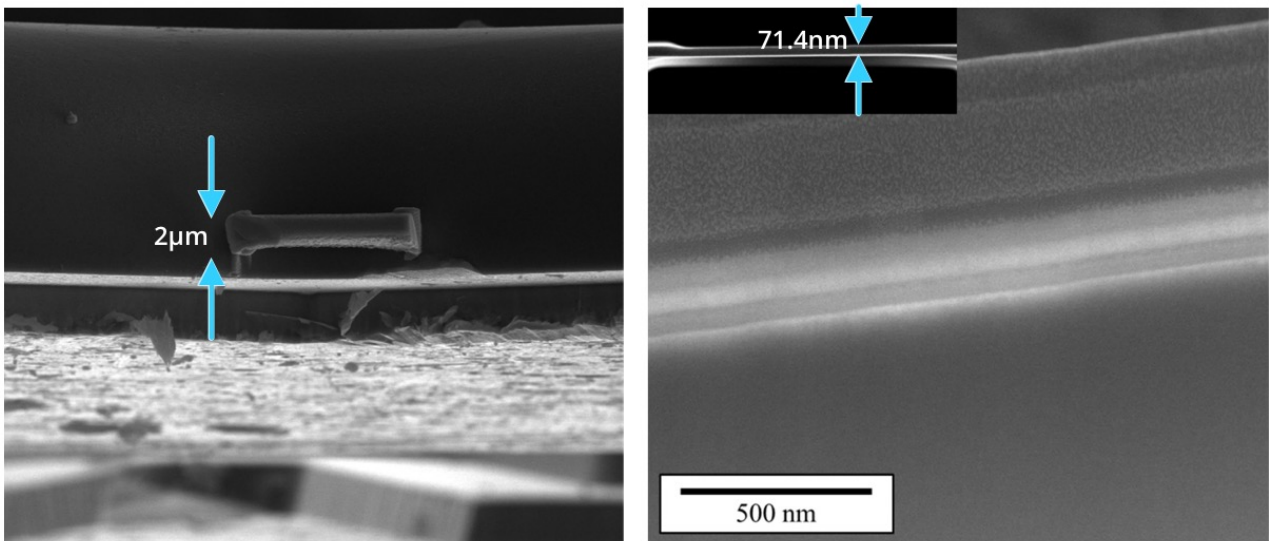
Polishing

Amorphous layers are created on each side of the TEM lamella during the TEM lamella preparation. The amorphous layers on each side are ~23nm for silicon. To improve the quality, the TEM lamella is first polished with 5keV ions which reduces the thickness of the amorphous layer to roughly 10nm. A 2kV polish (if available) further improves the TEM lamella quality by reducing the thickness of the amorphous layer to sub 5nm thickness.
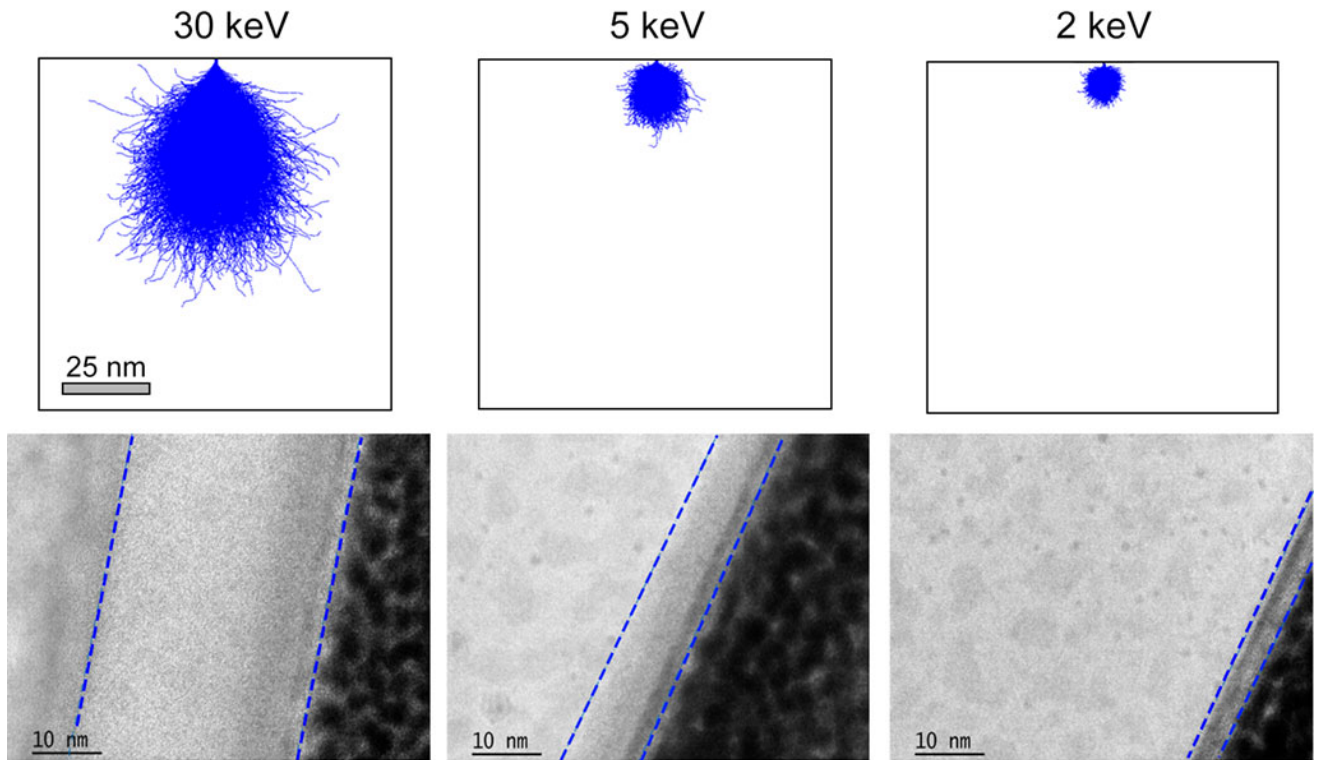
3D Tomography

3D reconstruction, often called 3D tomography, is the reconstruction of a 3-dimensional volume from a series of slices. This is sometimes called "serial sectioning". In this technique the sample is sectioned using the ion beam, making it a destructive technique. Before slicing, the area of interest is usually covered by depositing a protective layer using the GIS. The protective layer reduces curtaining effects during the slicing process. To avoid problems with redeposition, extra material around the area of interest is also removed. The slice thickness can be varied from ~10nm to several micrometres, depending on your sample feature size. A lot of FIB-SEMs allow the process to be automated.

Once slicing has begun, each slice is imaged using the electron beam, this could be done with conventional secondary electron imaging, or it could be a chemical map using EDS or a crystallographic map using EBSD. Each slice is recorded and becomes part of the image stack. The image stack is used to reconstruct a 3D model of the sample with a high performance PC. Conventional 3D stack sizes are < 50µm x 50µm x 50µm for Ga^+ ion instruments; <500µm x 500µm x 500µm volumes are becoming more common with the newer plasma FIB instruments that can remove material more quickly.
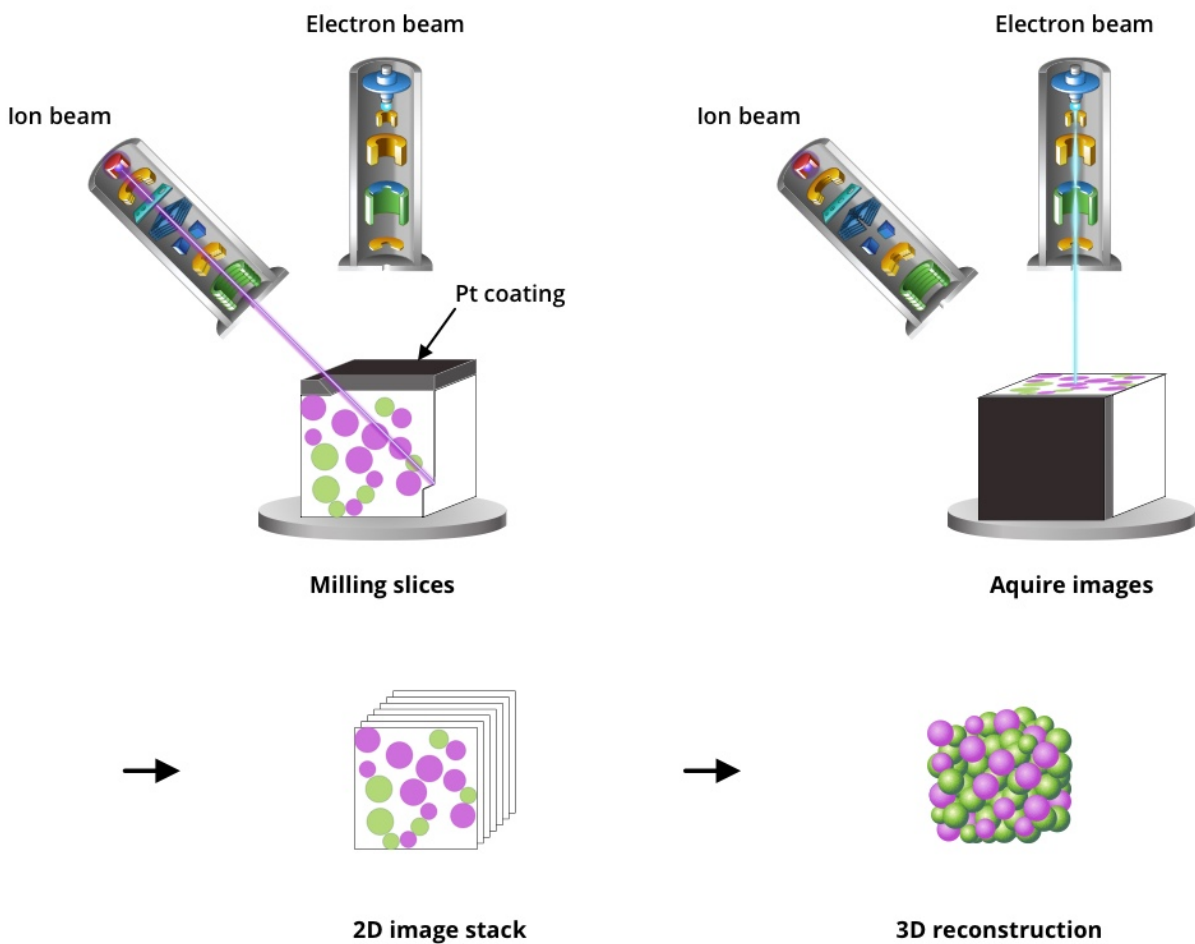
Credits

Microscopy Australia acknowledges the huge input of time and expertise by the many staff members and associates who have contributed to the development of MyScope over the years.

For the FIB module we thank: Annalena Wolff, Hui Diao.

Credits