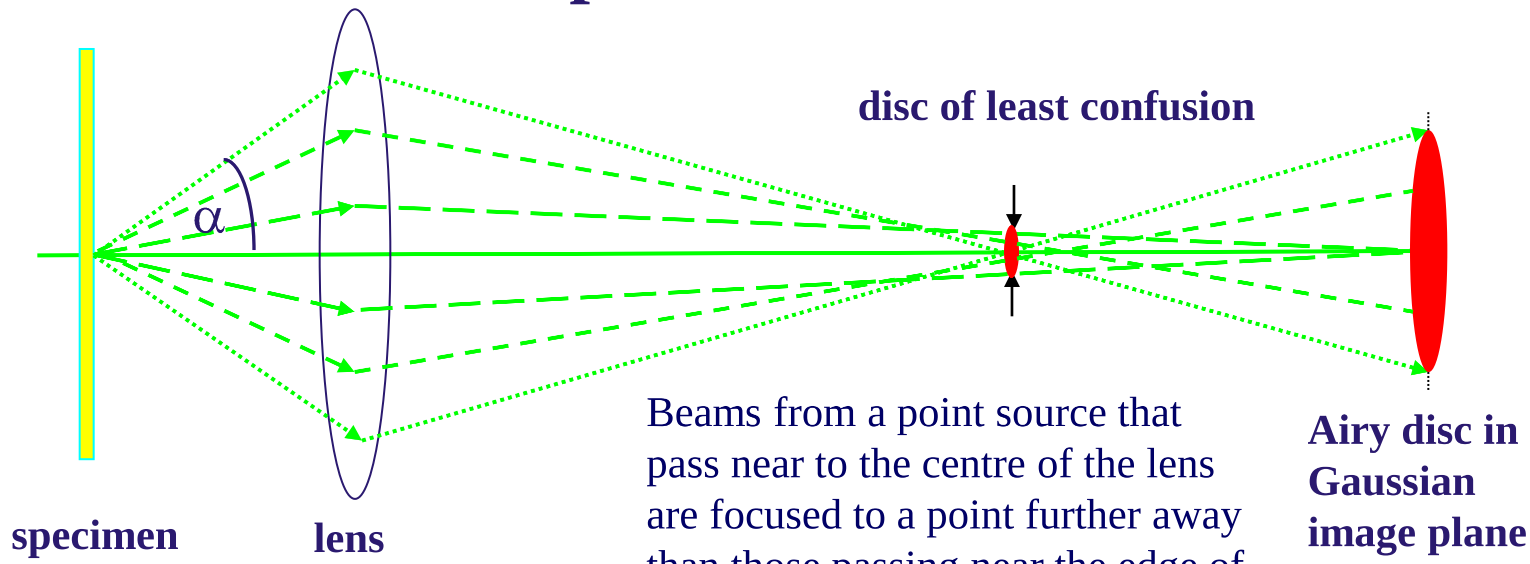
EEE6009 Advanced Instrumentation
Electron microscopy - Lecture 3

Recent advances in electron optics
and
an introduction to ion beam microscopy

advances in electron optics
aberration correction

In the lecture 2 we discussed how the ultimate resolution of the transmission electron microscope was governed by the aberrations in the electro-magnetic lens – the principle aberration being ***spherical aberration (Cs)***.

spherical aberration



Beams from a point source that pass near to the centre of the lens are focused to a point further away than those passing near the edge of the lens blurring out the focused spot

aberration correction

- **motivations for correcting spherical aberrations are therefore:**

  - **attain superior resolving power in TEM and STEM** – current microscopes have a resolution between 0.15 and 0.2nm, i.e. at or near important atomic distances – to answer the problems of emerging nano-structure technology better resolution <0.1nm is required

  - **provides design flexibility** – In order to minimise the spherical aberration of the objective lens (and hence attain the optimum imaging resolution) a conventional TEM/STEM requires the specimen to sit very close (1-2mm) to the objective lens poles. This places limits on the flexibility in positioning both the specimen (such as the degree specimen tilt allowed and the physical size of specimen holder). In addition the collection efficiency of detectors such as those for EDX chemical analysis can be reduced due to the limited space around the specimen in which to locate the detectors.

  By increasing the overall resolution of the objective lens system by aberration correction allows the use of larger objective pole piece gaps without significant loss of resolution and hence permits higher specimen tilt angles for tomographic (3D) imaging, larger specialised specimen holders, closer detectors and the **incorporation of additional detectors/manipulators for in-situ and dynamic studies**

aberration correction

- **motivations for correcting spherical aberrations - continued**

  - easier image interpretation in HREM by **reduced image delocalisation** effects

  - **higher STEM beam current for given probe diameter** – better for chemical analysis of nano-structures and for detecting foreign atoms

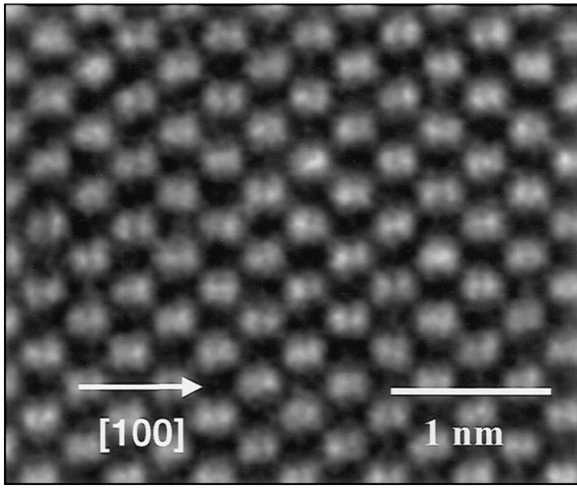
  - Cs correction has been theoretically understood for many years**,** in fact the means to calculate the aberrations in electron lens was presented by Otto Scherzer in 1947/8

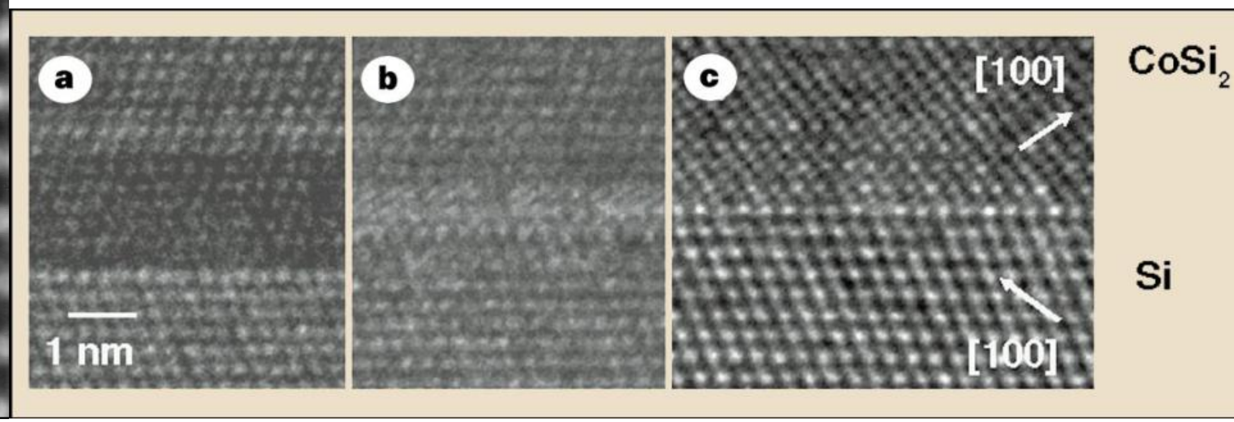
  O. Scherzer, *Optik* **2**, p114 (1947) and *J. Appl Phys*. **20** (1) p20 (1948)

  - However, it has only recently been recently achieved as a result of the availability of high quality electronics, stable power supplies and fast computing power.

aberration correction

first aberration corrected TEM in Jülich, Germany





improved point resolution (0.14nm at 200kV)

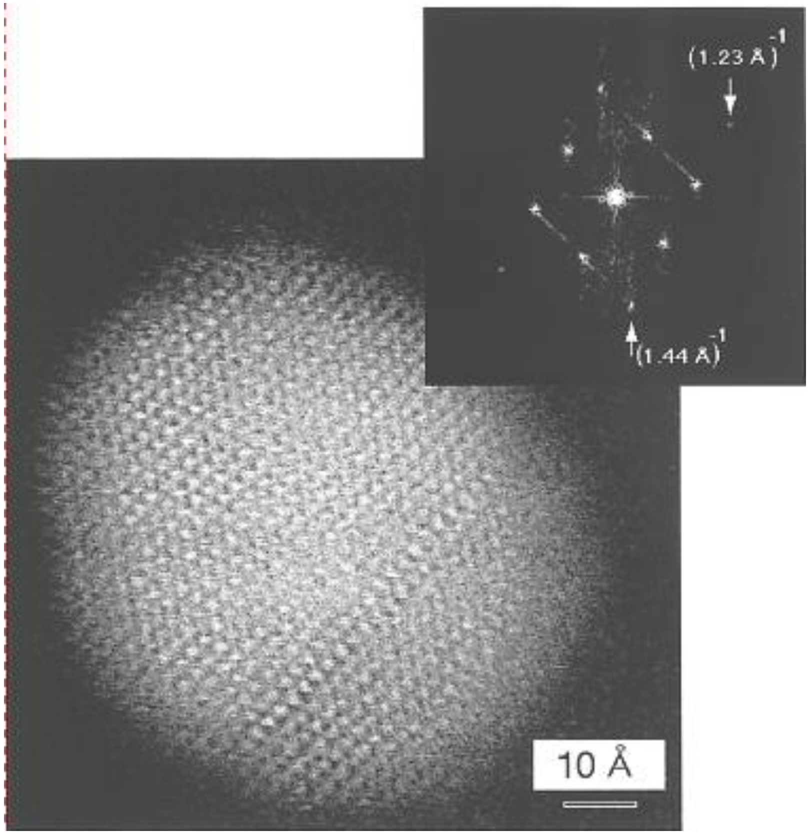
M. Haider et al., Ultramicroscopy 75 (1998) 53-60v

improved image delocalisation with aberration correction (right) compared to conventional HREM

M. Haider et al., Nature 392 (1998) 768-769

aberration correction

first aberration corrected STEM at IBM T.J. Watson Research Center, Yorktown Heights, USA

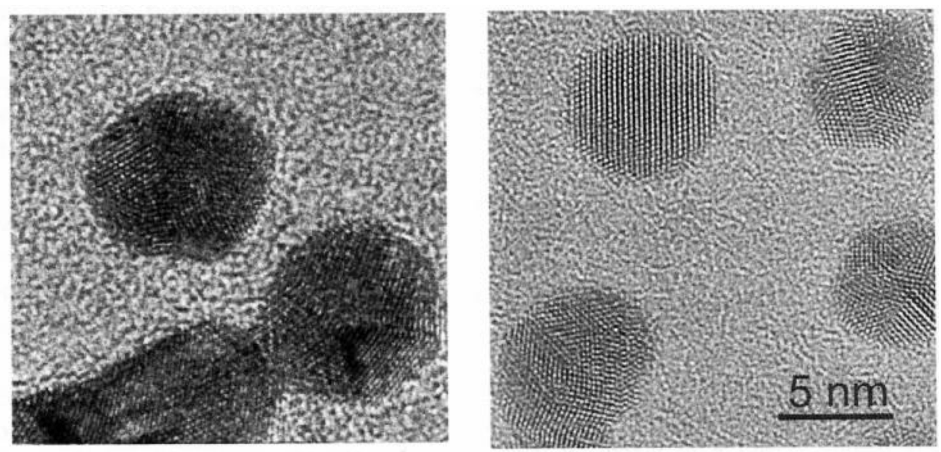


improved information transfer to 0.12nm at 100kV in an aberration corrected VG STEM, 160pA current

N. Dellby et al., J. Electron Microsc. 50 (2001) 177-185

aberration corrected TEM in the UK

All conventional objective lens systems have a ***positive*** value of the ***spherical aberration***. Aberration correctors apply a compensating negative aberration via several additional multi-pole lenses. Significant computing power is required to control these additional lenses.



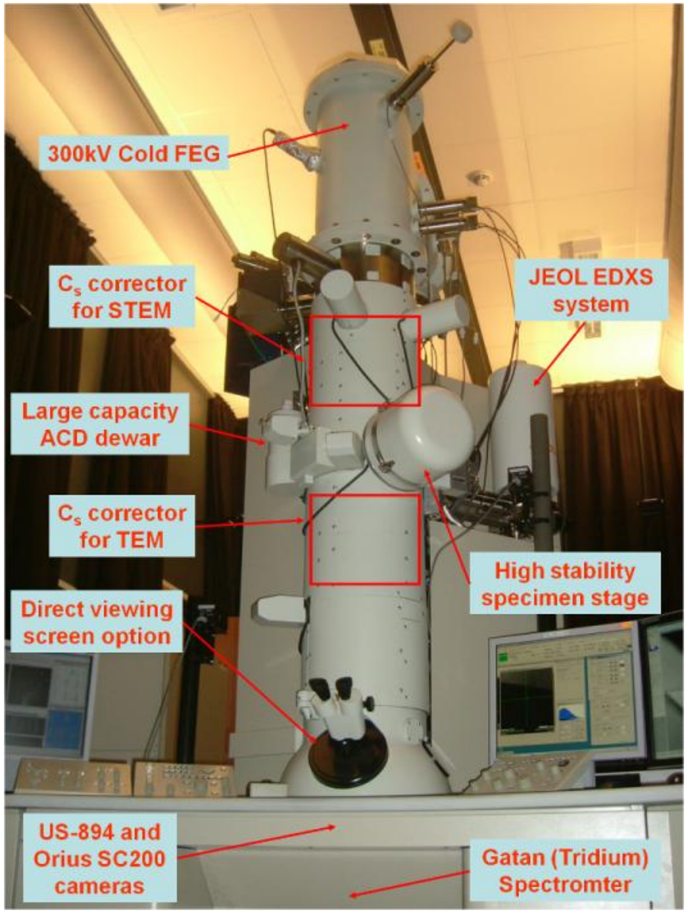
High resolution images of gold particles on amorphous germanium for (a) C_s= 0.5 (corrector off) and (b) C_s= 0.01 (corrector on).

H Sawada *et al.*, J. Electr. Microsc. 54 *(*2005) 119-212

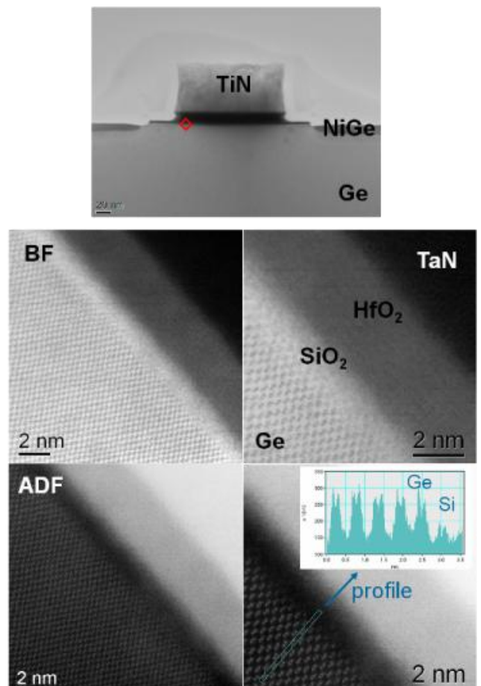
aberration correction

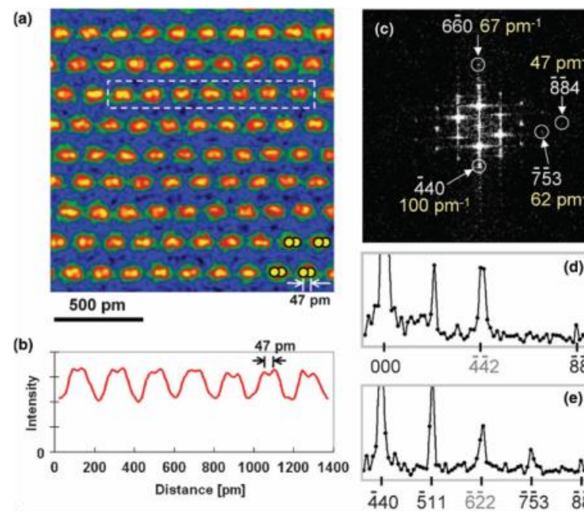
aberration corrected TEM/STEM at Sheffield

University of Sheffield 300kV Cold field emission gun double aberration corrected TEM/STEM produces high brightness sub-Å electron probes with improved energy resolution (0.35eV)



I M Ross et al *Proc. 17th Int. Conf. Microscopy of semiconducting materials (2011)*



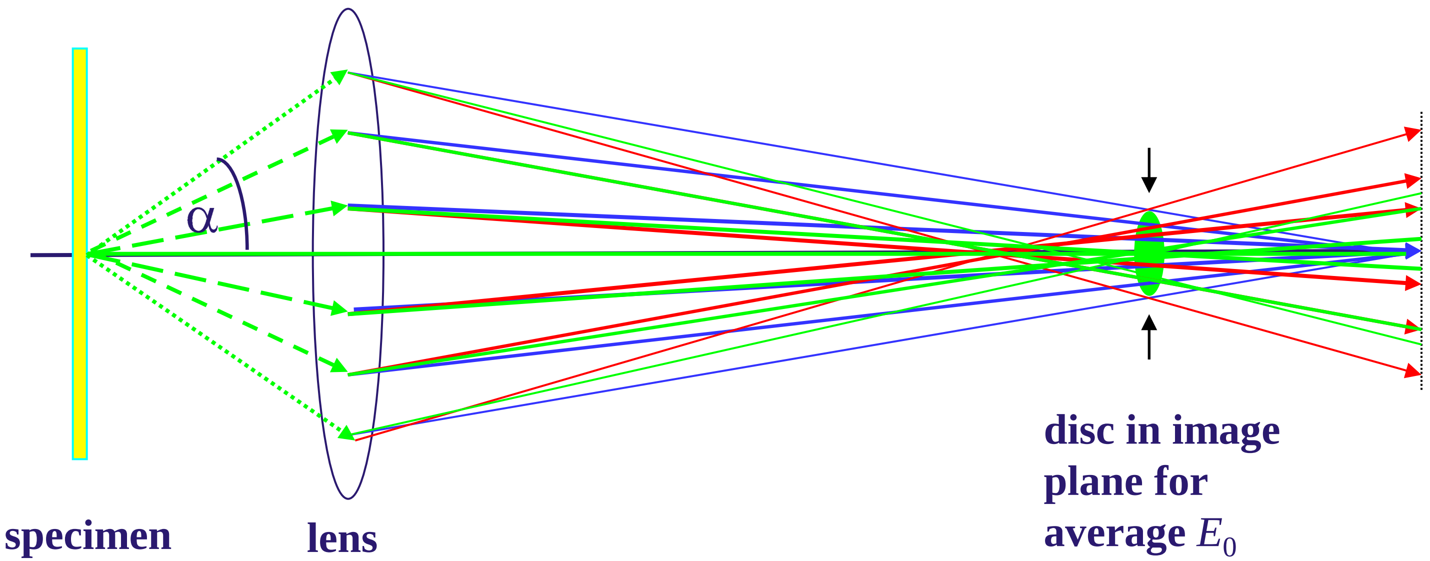


Germanium <114> H Sawada et al J. Electr. Microsc. (2009)

aberration correction

lens aberrations II

chromatic aberration



Waves of different wavelength are present in both the primary beam and those emerging from a thin specimen – ***chromatic aberration*** describes the effect that slower electrons (red) are brought to focus by a lens nearer the lens than faster electrons (blue). This chromatic disc diameter in the Gaussian image plane is given by: $d_c = 0.5\, C\text{c}\, \alpha\, \Delta E/E_0\, [1+E_0/m_0c^2] / [1+E_0/2m_0c^2]$

where ΔE is the energy loss and E_o is the energy of the primary beam and Cc the chromatic aberration constant.

aberration correction

- **what about chromatic lens aberrations?**

  - chromatic aberration (Cc) has a an influence on the **ultimate information limit** even after spherical aberration has been corrected.

  - to reduce or remove Cc would for example result in a **superior resolution in energy filtered images**

  - to reduce or remove Cc would improve ADF-STEM contrast and EELS-STEM quantification due to **reduced tailing in the electron probe** (as would a monochromator)

  - **Cc correction is technically more difficult to achieve**

  - ways to reduce the effects of Cc:

    - improved gun and electronics stability to reduce energy spread ΔE

    - monochromate incident electron beam – reduces the energy spread of the incident electrons to ΔE=0.1eV (typically 0.5-1.0eV for a FEG) – good for EELS, however, most current designs result in a large reduction in intensity

    - Cc corrector currently being developed by CEOS, Heidelberg, Germany

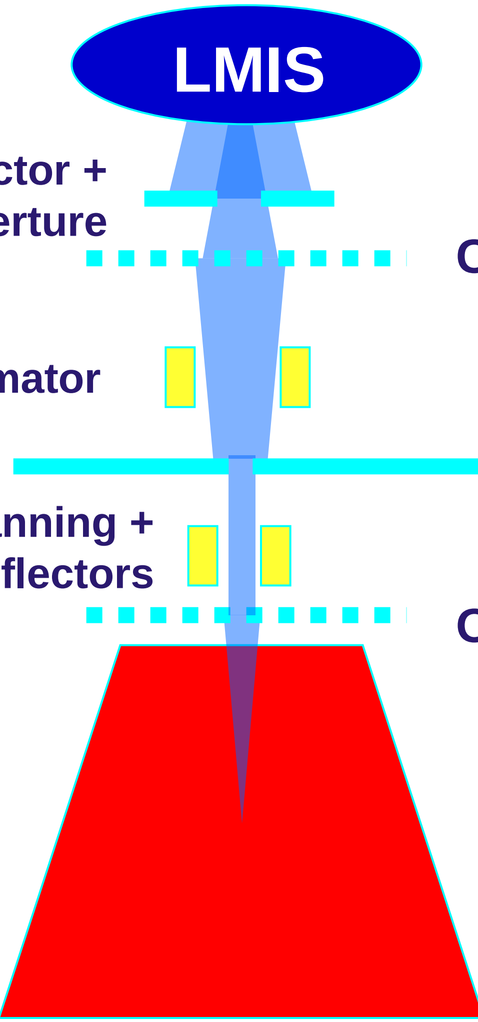
Ion beam microscopy

Focused Ion Beam Microscopy (FIB)

- ❖ **Nano Fabrication**:
  - ❖Design and repair of semiconductor devices
  - ❖Special geometries – AFM tips
  - ❖Selective etching and deposition of metals and insulators
- ❖ **Preparation of site specific SEM cross-sections**
- ❖ **Preparation of site specific TEM samples**:
  - ❖Lift-out technique – *ex-situ* and *in-situ*
  - ❖Trench technique
  - ❖Special geometries – 3D Tomography/Atom probe
- ❖ **Ion Contrast Microscopy**:
  - ❖identify grain boundaries, phases
  - ❖voltage contrast - electrical continuity

Ion beam microscopy

The Focused Ion Beam (FIB) System



Ion beam microscopy

Ion beam - material local interactions

Topology

ion sputtering, redeposition

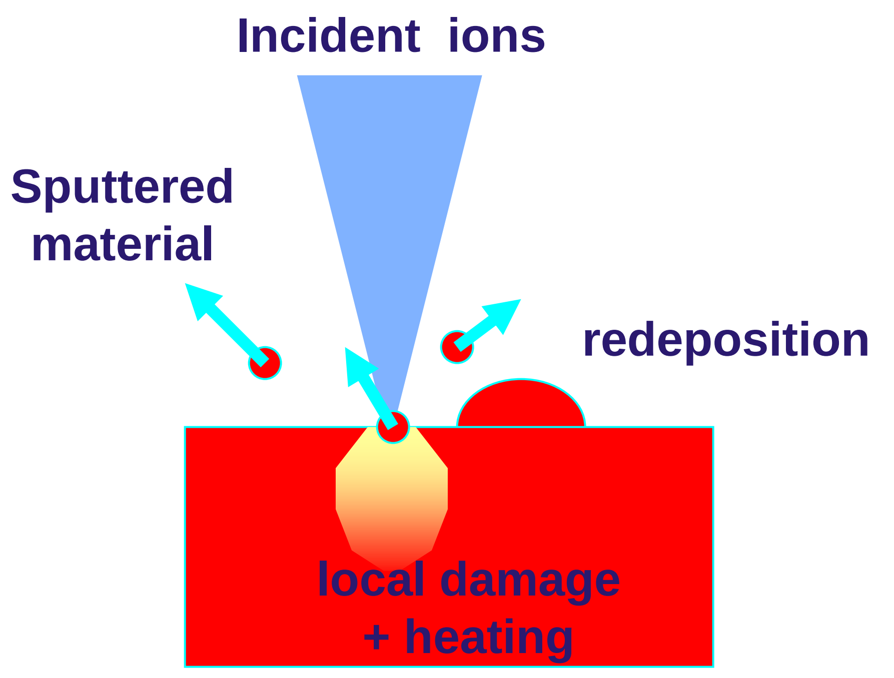
Structural defects

ion implantation - local strain
ion damage
(e.g. interstitials, vacancies)
surface amorphisation

Chemical changes

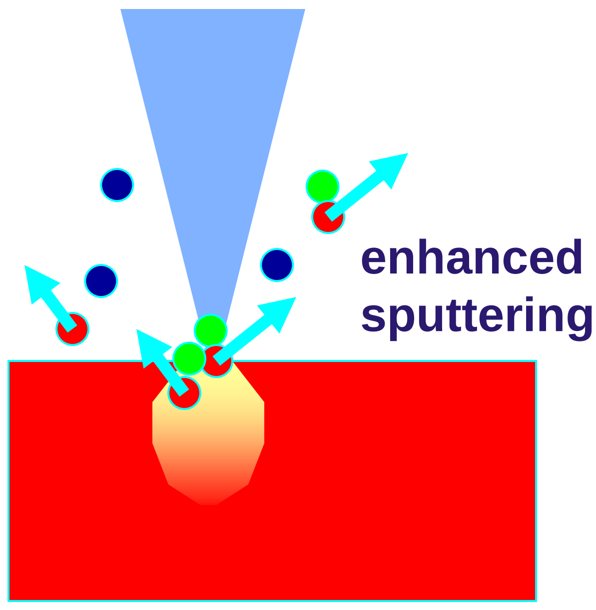
ion implantation, local mixing

Local heating

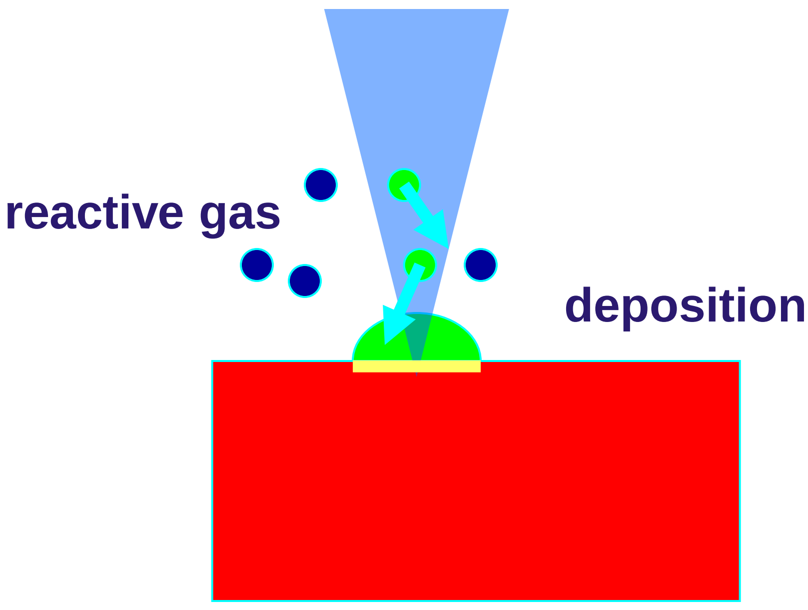


Ion beam microscopy

Reactive gas Ion beam nano-processing:



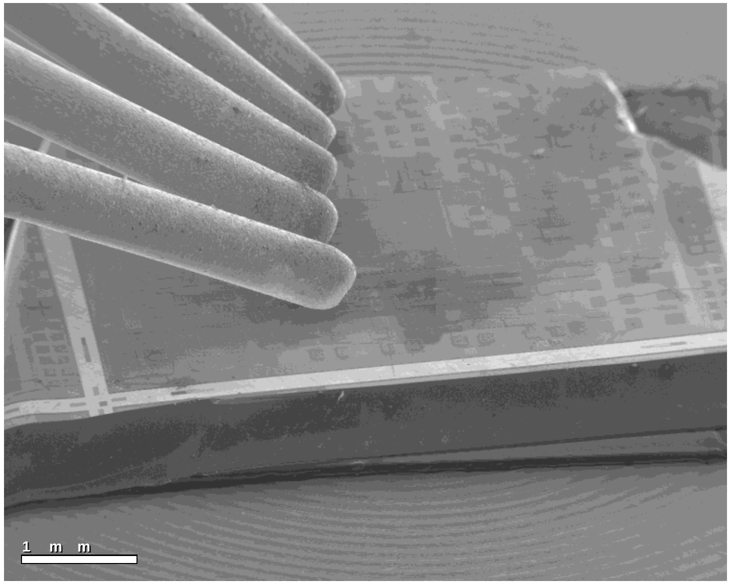
Local etch

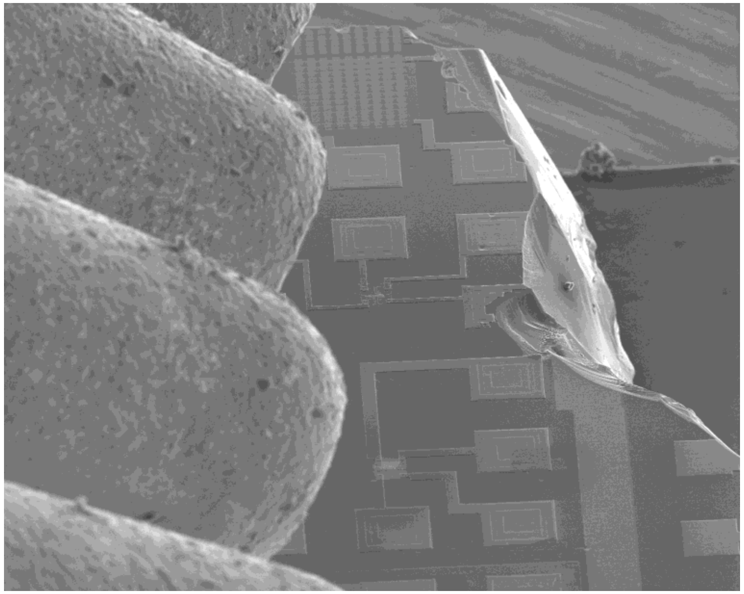


FIB-CVD

Ion beam microscopy

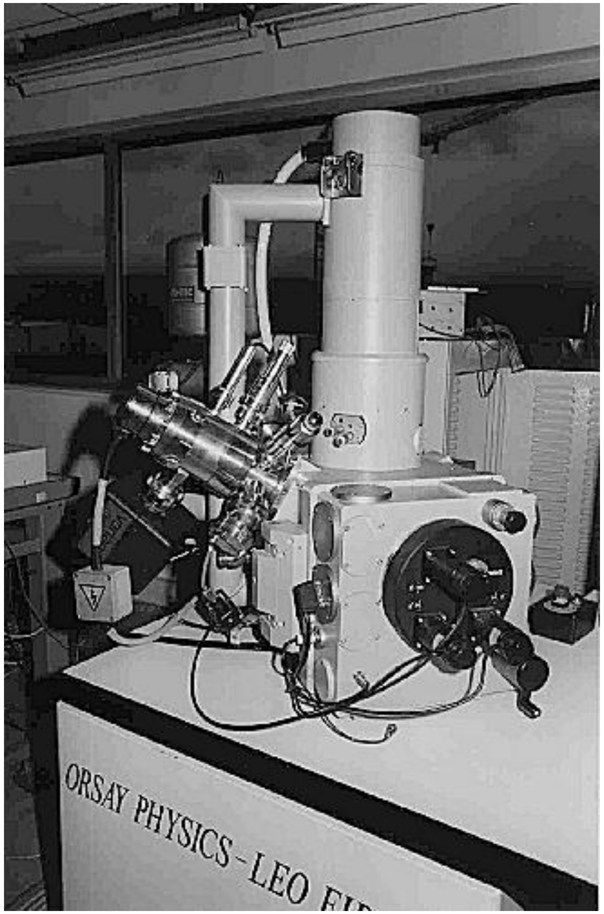
Ion beam nano-processing





Gas injection system – for selective deposition (Pt, W, SiO_2)
or reactive gas injection (F, Cl)

Ion beam microscopy

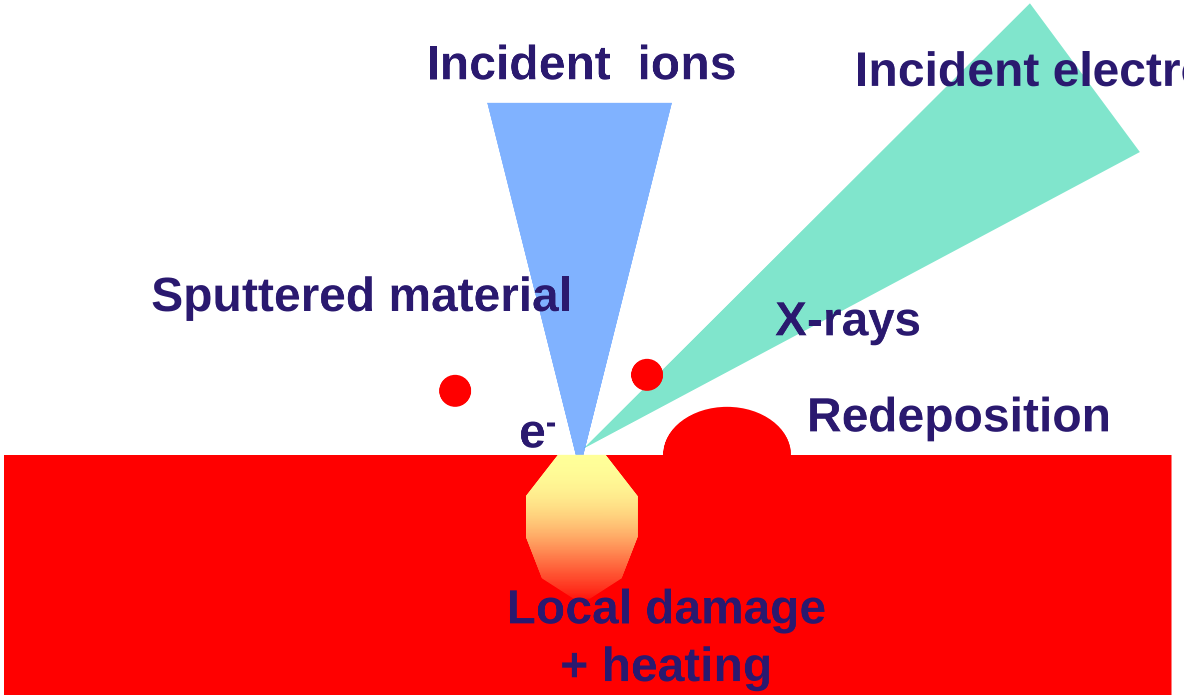


Ion columns available as single column instruments or dual column instruments combined with SEM imaging capabilities.

Scope for additional equipment; EDS, EBSD, CL, STEM and manipulators.

Ion beam microscopy

Focused ion beam nanoanalysis : dual beam machines



5nm SEM resolution at 15mm WD

9nm FIB resolution 30kV Ga^+ ions

Signals :
Ions, neutrals,
secondary e^- (ISE or ESE)
X-rays, BSE

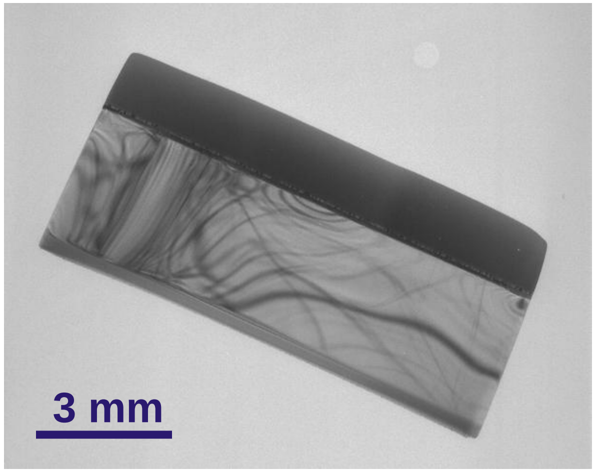
Imaging

In-situ detectors :
SE, SIMS
BSE/EBSD, EDX, TEM
Ex-situ : TEM, AFM

Ion beam microscopy

FIB machining of specimens for TEM nano-analysis

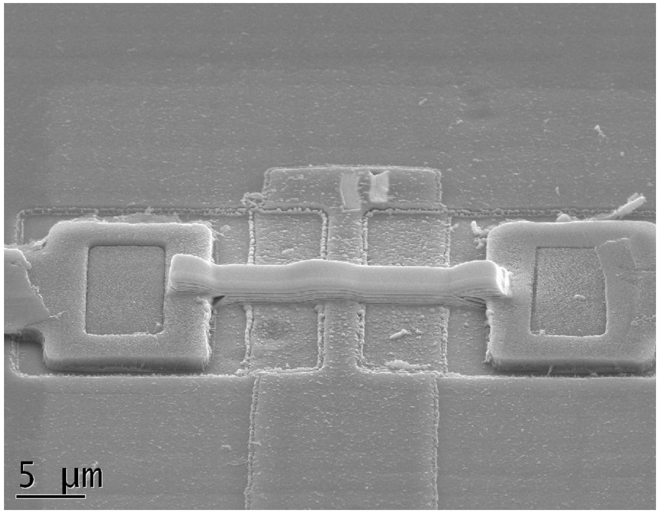
- Machining electron transparent sections
- Usually ‘2D’ (<150nm thick) slices through object, cut from a surface
- Can be other morphologies - needles for atom probe or tomography
- Seek to minimise surface damage/amorphization, e.g. final milling with lower energy ions (Ga^+ or Ar)

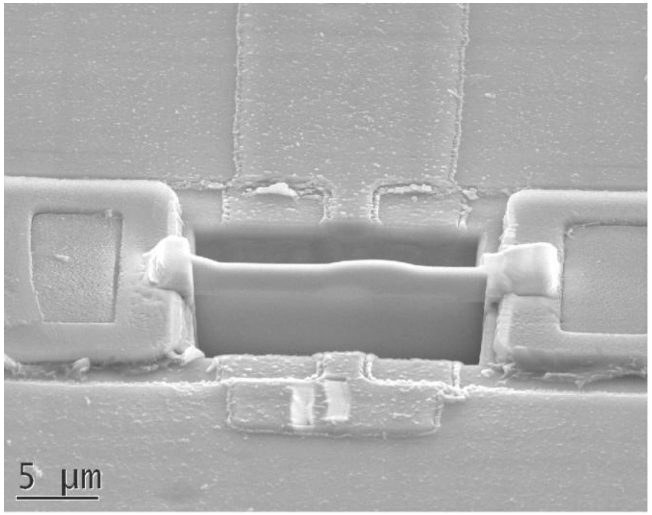


e^--transparent ‘plucked’ slice

Ion beam microscopy

TEM sample preparation via the *ex-situ* lift-out technique



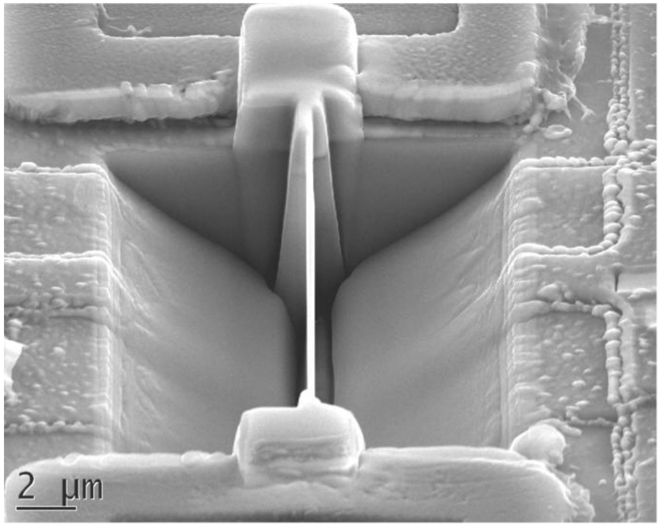


Pt or other protective layer deposited on device area by means of the gas injector.

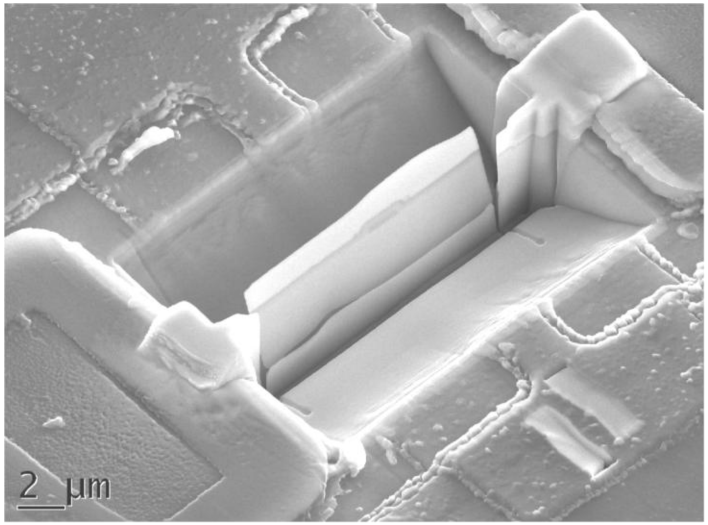
A wedge shaped cut is ion milled on each side of the device area with a probe current of ~ 60pA to 1nA depending on material.

Ion beam microscopy

TEM sample preparation via the *ex-situ* lift-out technique



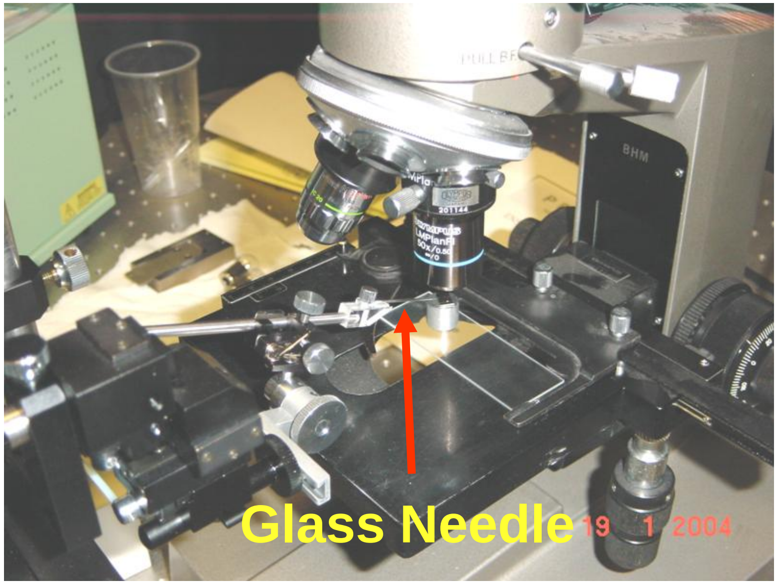
The intended lamella is polished down to electron transparency (~100nm) with a lower probe current in the region of 20 to 40pA, low kV if required.

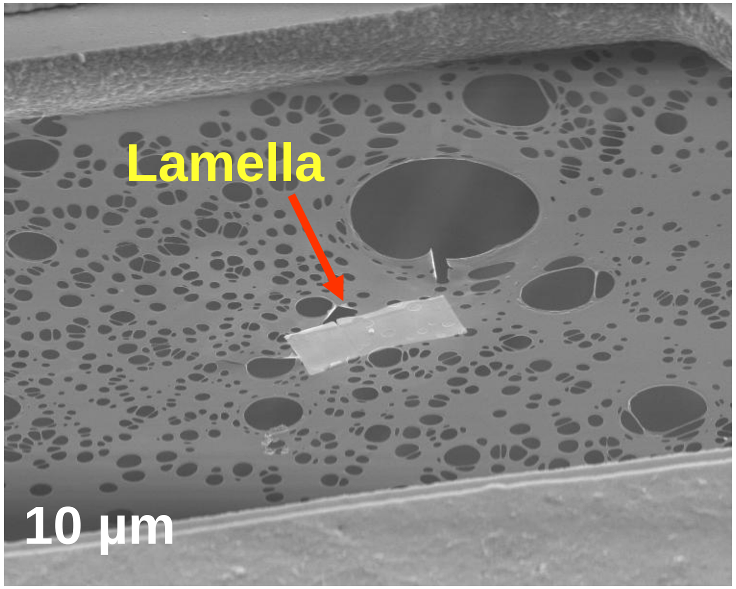


The sides and base of the lamella are cut and the lamella is dislodged from the wafer and ready to be lifted out.

Ion beam microscopy

TEM sample preparation via the *ex-situ* lift-out technique



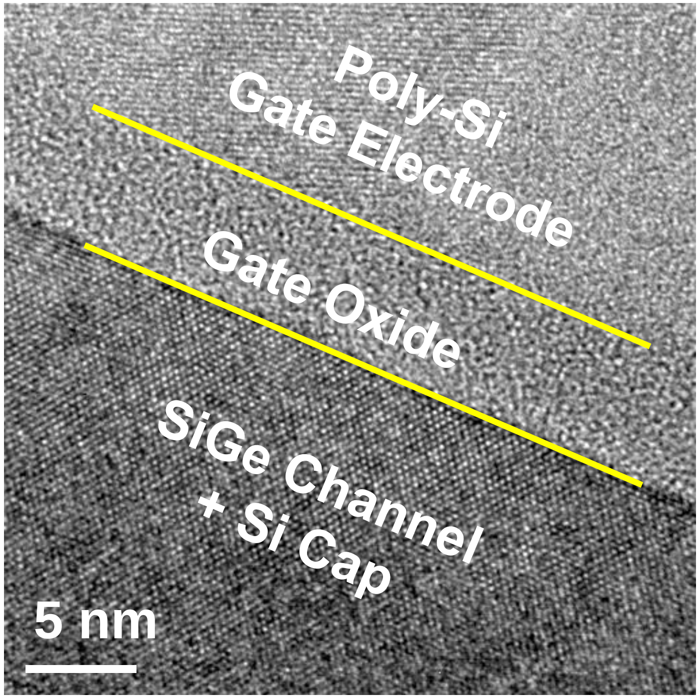


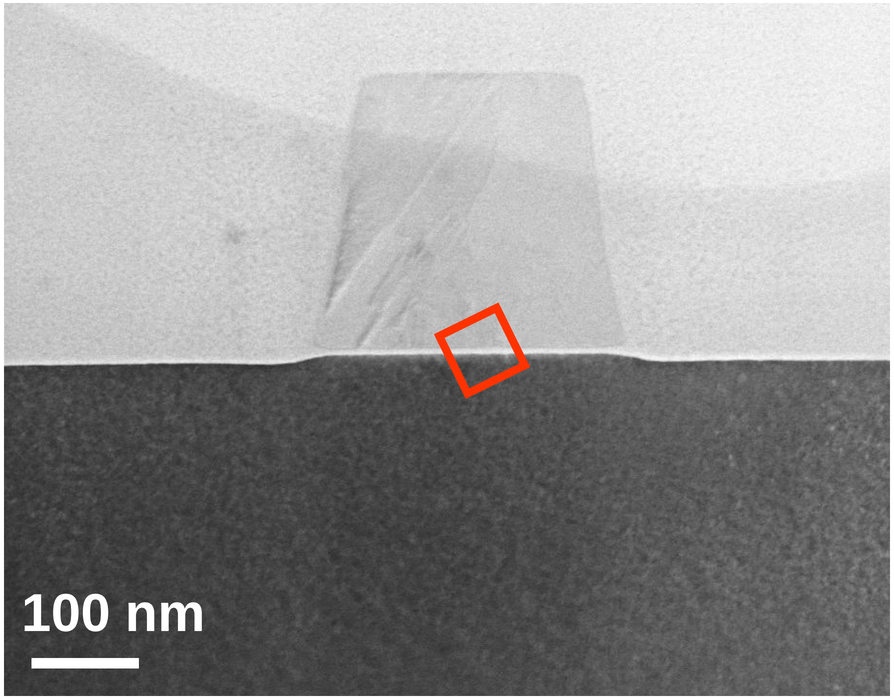
The lamella is lifted out by electro-static charge using a glass needle attached to an hydraulically-controlled micro-manipulator

The lamella is deposited on a carbon film grid used for TEM analysis

Ion beam microscopy

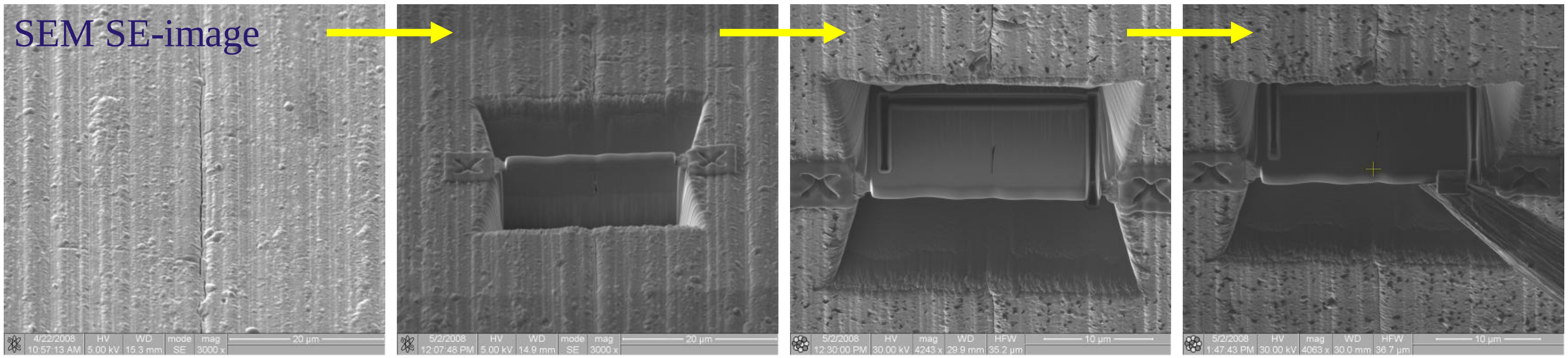
Gate Electrode and Gate Oxide Analysis – TEM specimen prepared via ex-situ lift-out technique

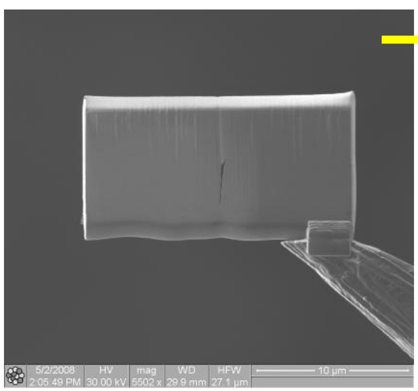


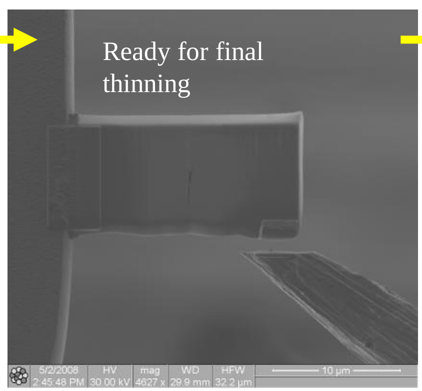


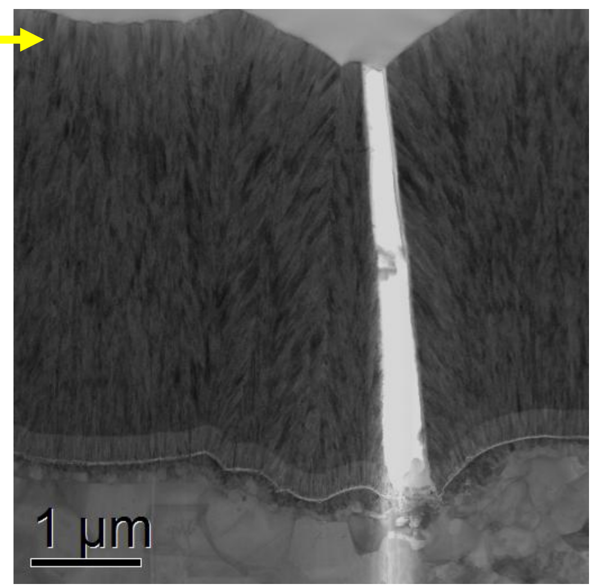
Ion beam microscopy

Cross section sample preparation via the *in-situ* lift-out technique





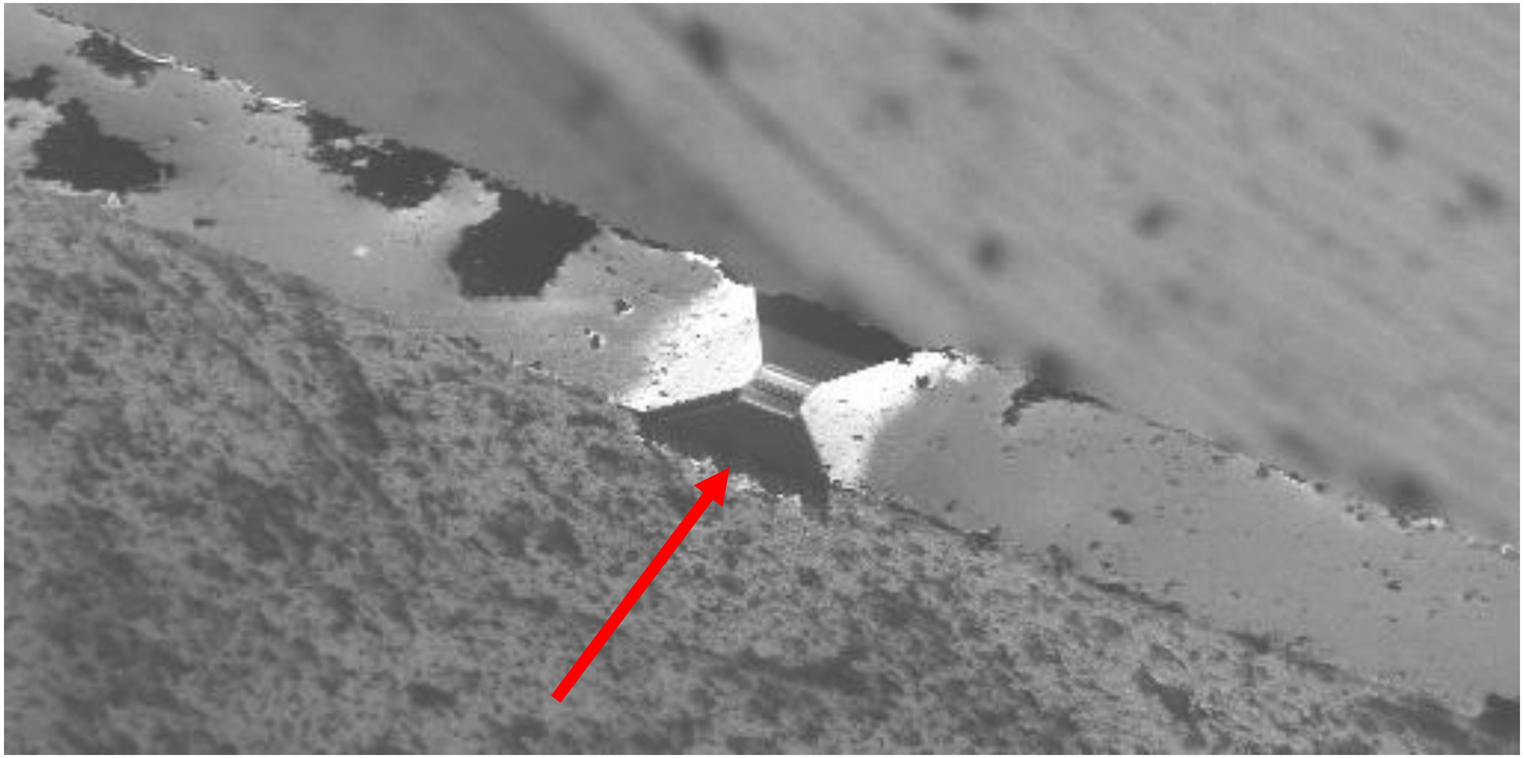




Using an in-situ lift out improves the reliability of the process and enables re-thinning of the specimen if necessary. Right BF-TEM image.

Ion beam microscopy

TEM sample preparation Via the "trench" or "window" technique



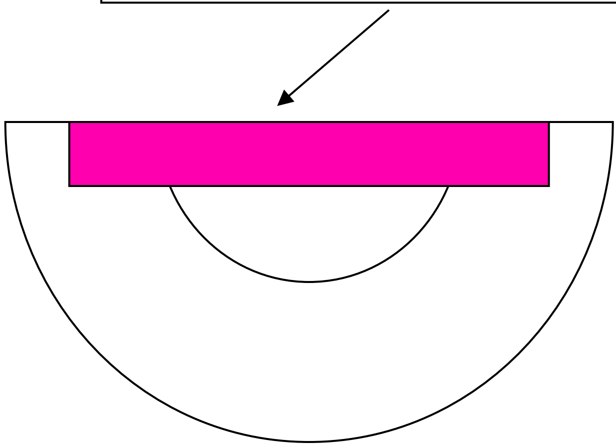
e^--transparent FIB cross-section

analysed volume ~ $10\mu m^3$

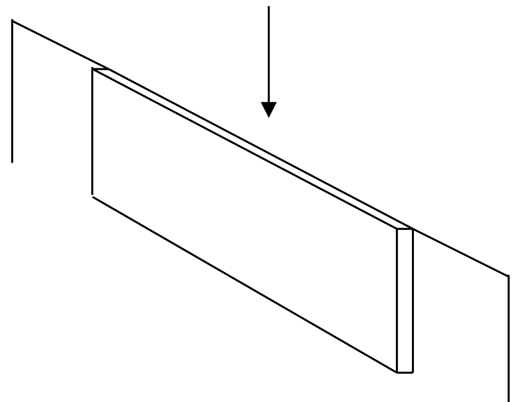
Ion beam microscopy

TEM sample preparation Via the "trench" or "window" technique

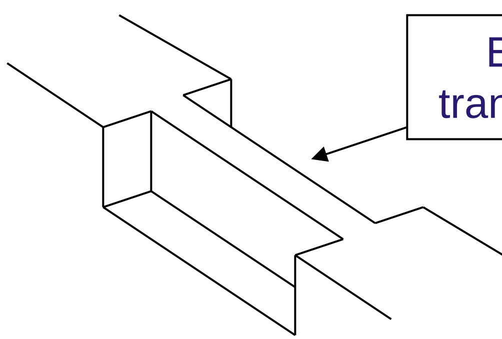
Mechanically thinned sample 30µm-50µm thick

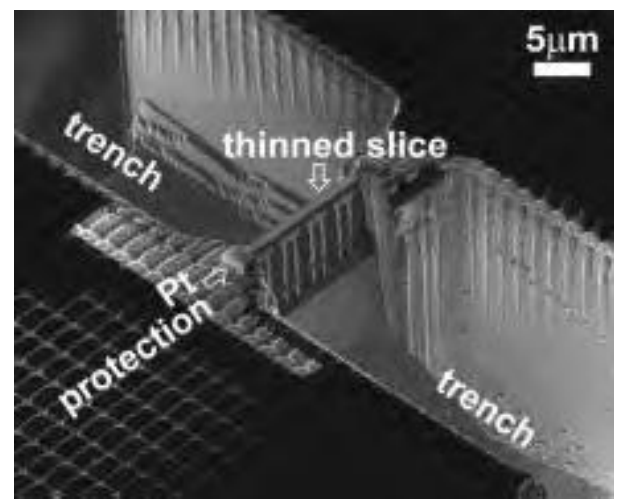


FIB milling direction



Electron beam transparent window





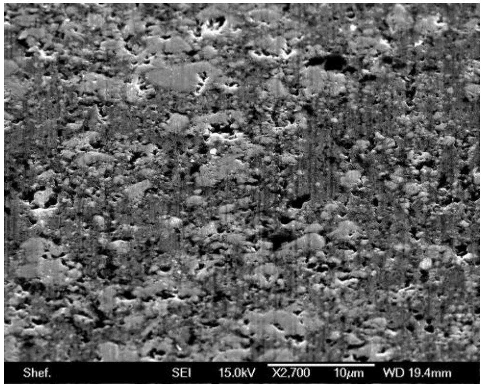
Ion beam microscopy

“Lift-out” technique versus “trench” technique

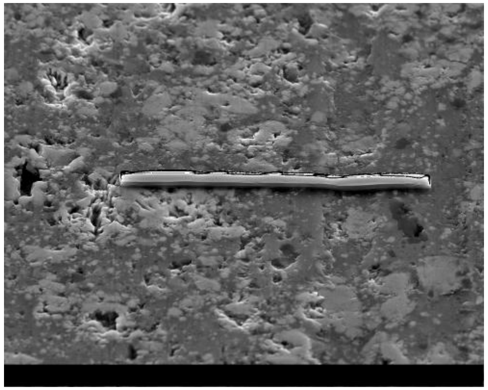
Lift-out technique	Trench technique
Site specific with high accuracy	Less site specific
No need for pre FIB sample preparation	pre FIB sample preparation required, cutting, mechanical polishing to 30-50μm
Success rate depends on the skill of the operator	Success rate higher as no lift-out stage.
Ex-situ lift-out requires some form of support and cannot be easily milled further	Sample already on support and ready for insertion into TEM
In-situ lift-out can be further milled if appropriate support used.	Easy to perform further milling in FIB or Ar^+ ions.

Ion beam microscopy - applications

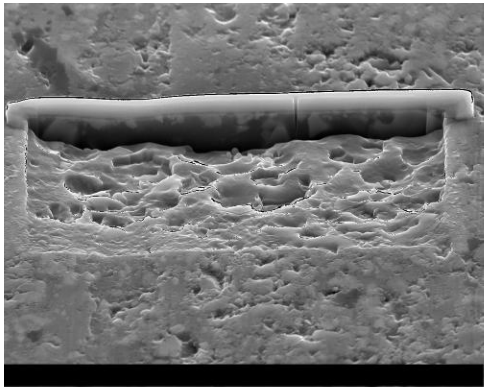
Preparation of site specific SEM cross-sections



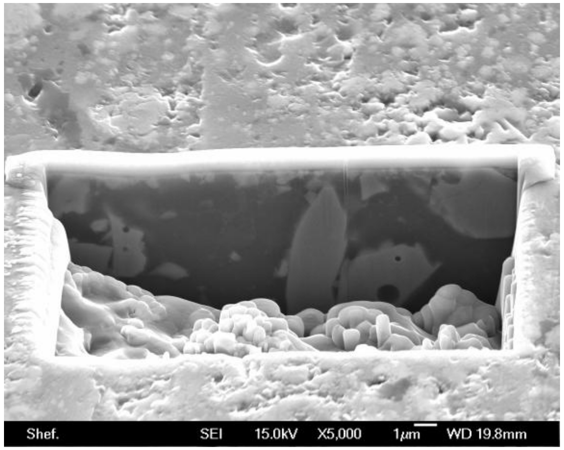
Worn Surface →



Metal Deposit →



Milling

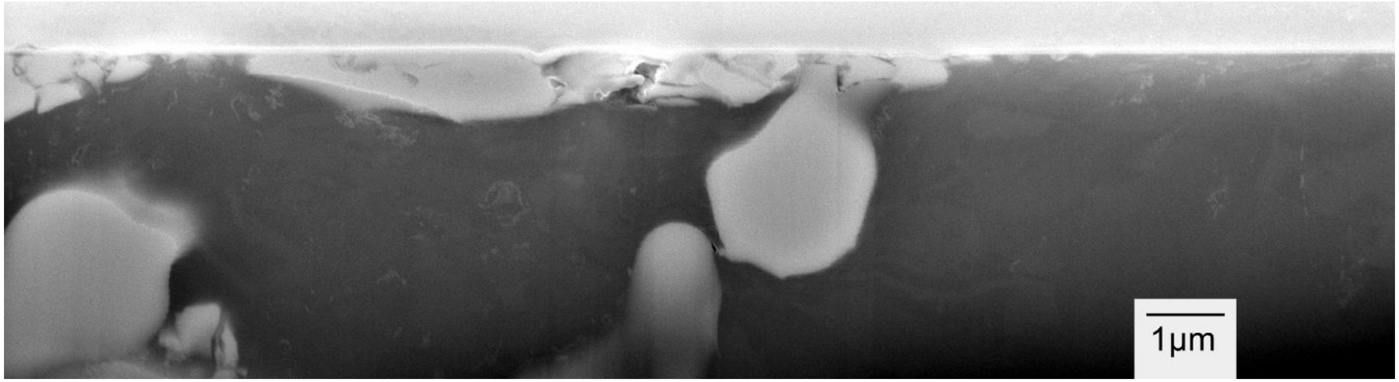


Final Trench

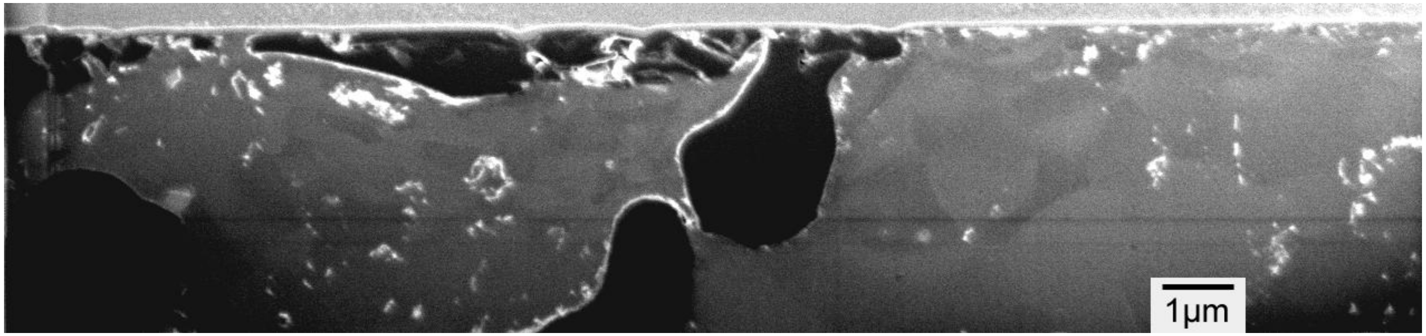
Images courtesy of J.Walker

Ion beam microscopy - applications

Ion Contrast Microscopy



SEM – Secondary electron Image

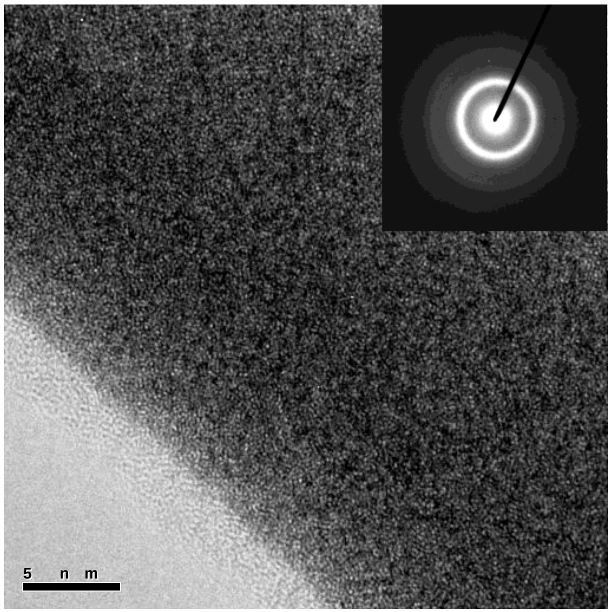


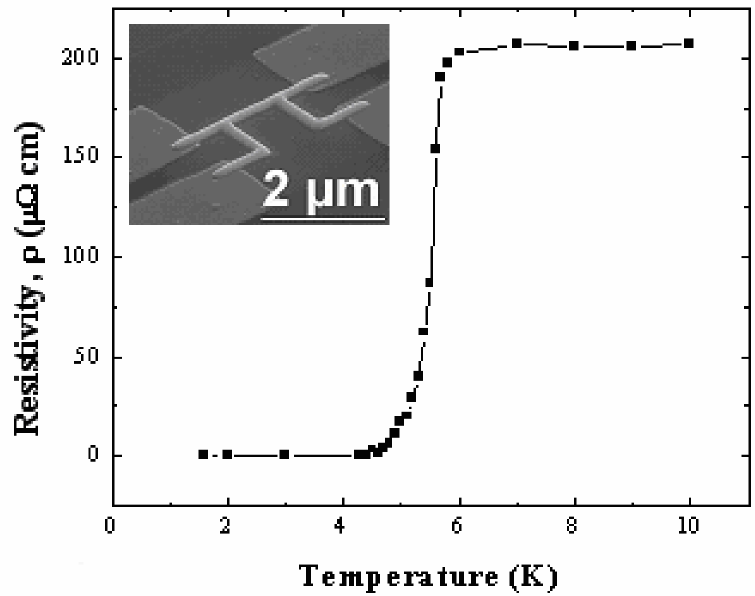
FIB – Ion Channelling Image

Longitudinal Section through a wear track in Al-alloy 5056 + 15vol% $MoSi_2$

Ion beam microscopy - applications

deposition of nano-wires/structures

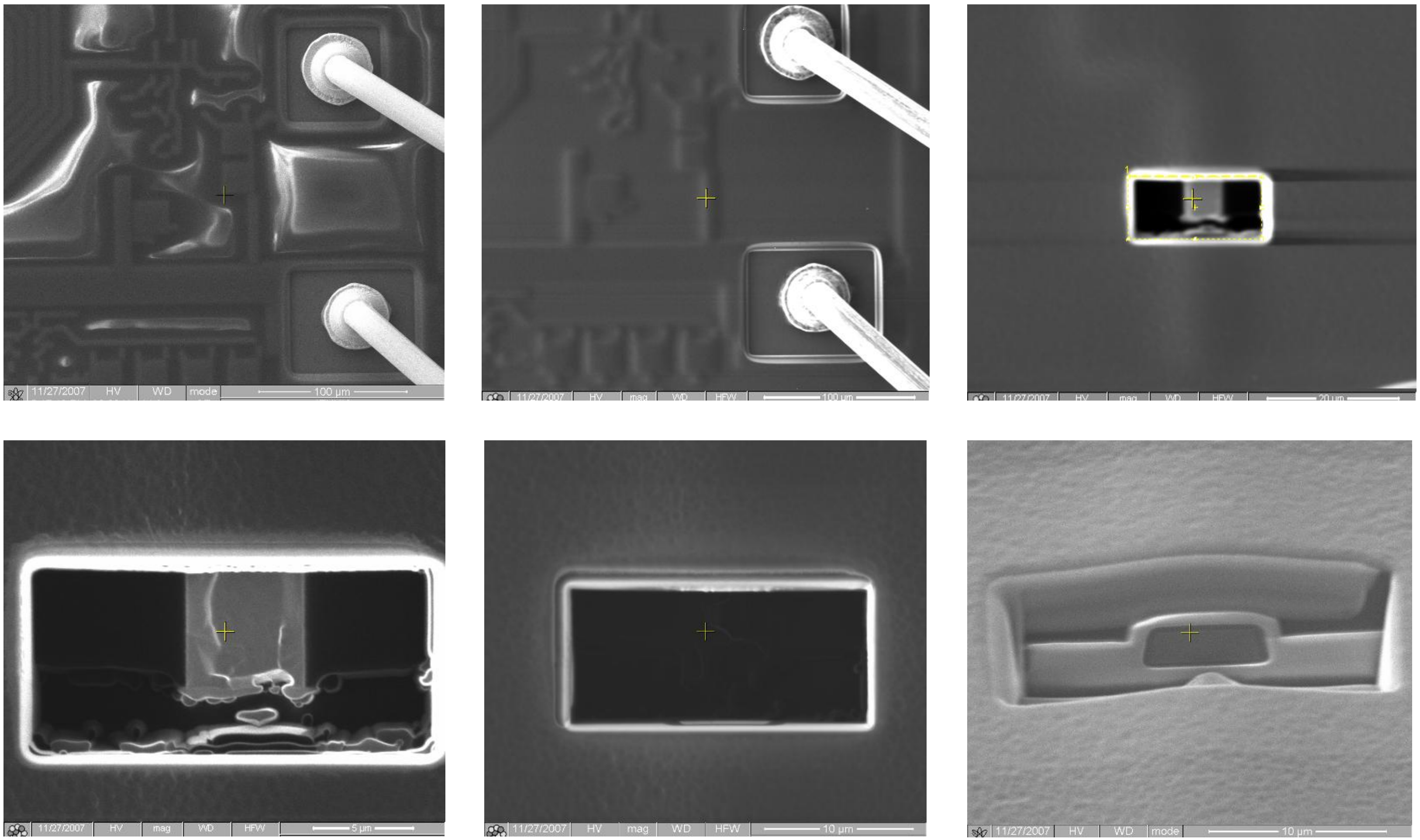




Tungsten nanowire deposited by ion beam induced chemical vapour deposition. Wire has an amorphous-like structure and exhibits superconducting properties below 5.5K.

Ion beam microscopy - applications

failure analysis, diagnostics and reverse engineering

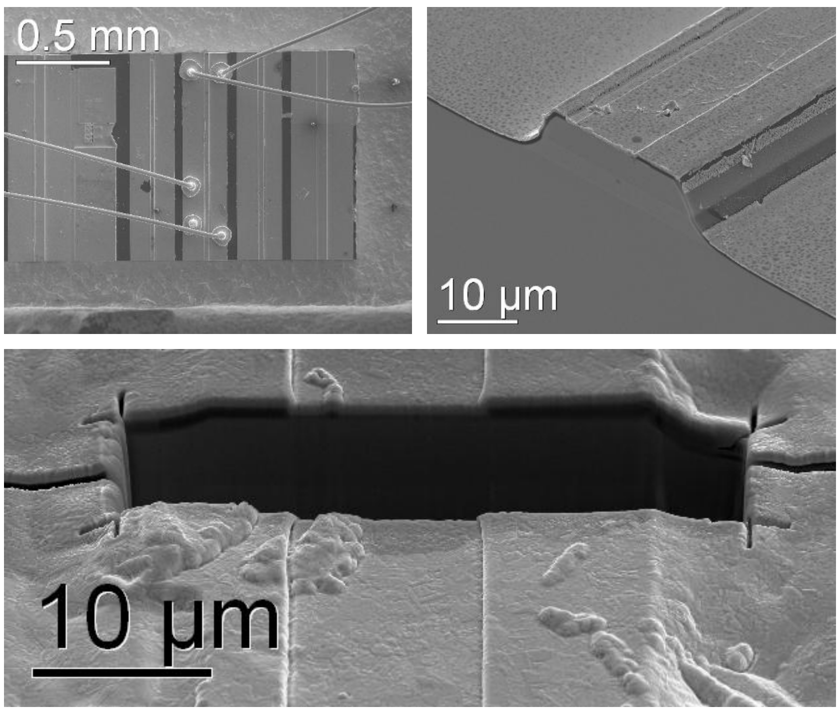


Site specific circuit modification of a packaged commercial component for development and diagnostic analysis.

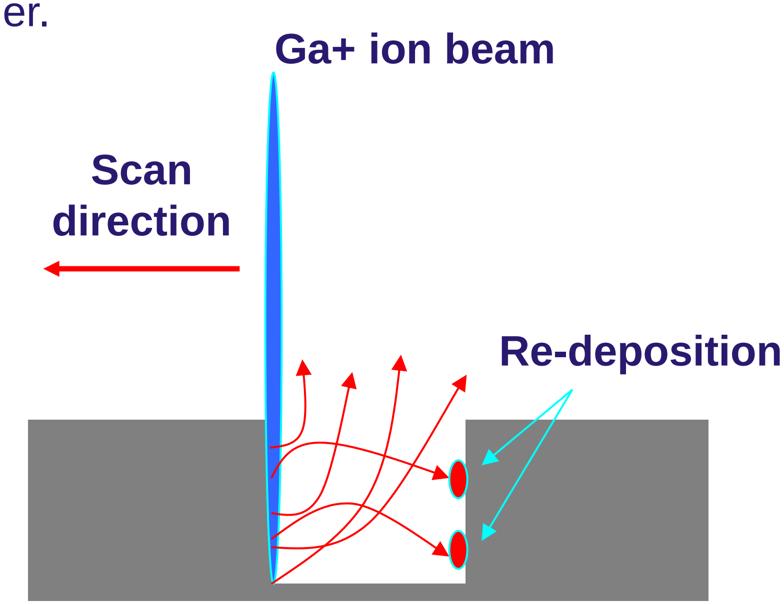
Ion beam microscopy - applications

device modification/fabrication

deep trench Bragg mirror for Infrared quantum cascade lasers:



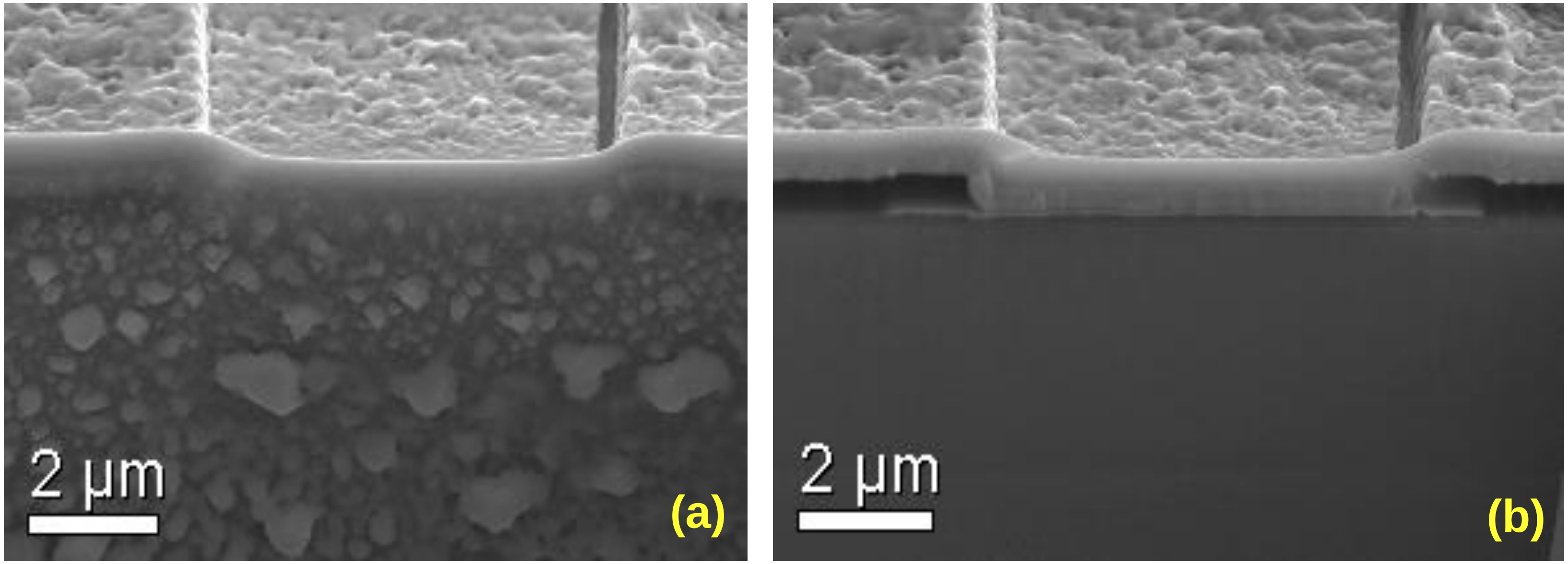
One phenomenon which can occur during the FIB processing of deep parallel-sided trenches is the undesirable re-deposition of sputtered material from one surface to another.



Specimen (cross-section)

Ion beam microscopy - applications

reactive gas assisted etching



Cross-section through the QCL device illustrating the difference in quality of the initially *clean* mirror surface after the adjacent surface has been milled: (a) by conventional ion milling showing the mottled surface due to re-deposition, (b) with XeF_2 gas assisted etching (all other milling parameters constant). (Ross et al J. Phys. Conf. Ser. EMAG 2007)

Ion beam microscopy

Further Reading

J Orloff, L Swanson and MW Utlant, High Resolution Focused Ion Beams: Fib and Its Applications: The Physics of Liquid Metal Ion Sources and Ion Optics and Their Application to Focused Ion Beam Technology, Plenum Pnb. Corp. 2002

J Meingaills Focus ion beam technology and applications, *J Vac Sci Technol.* B **5**, 1987

Special Edition of Micron: *Micron* **30**, 1999