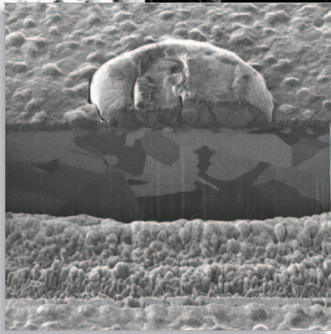

Focused Ion Beam (FIB)

- TEM/SEM sample preparation
- Failure analysis
- Cross-section analysis
- Sub-micron modification

A Focused Ion Beam (FIB) makes use of Ga-ions to remove material with a very high spatial precision. In this way cross-sections can be made on a specific location. The resulting samples can either be studied directly in the FIB or they can be transferred to a SEM or TEM for more detailed analysis. When both Ga-ions and certain gases are applied, it is also possible to deposit material. Therefore, the FIB can be used as a multifunctional tool in a broad range of applications.

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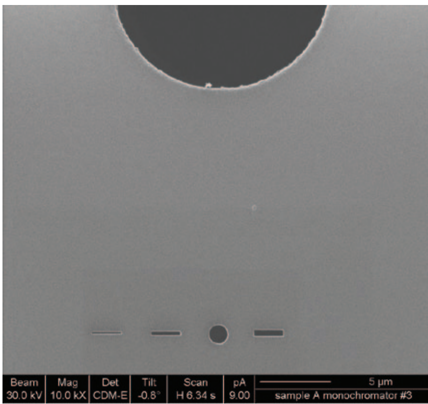
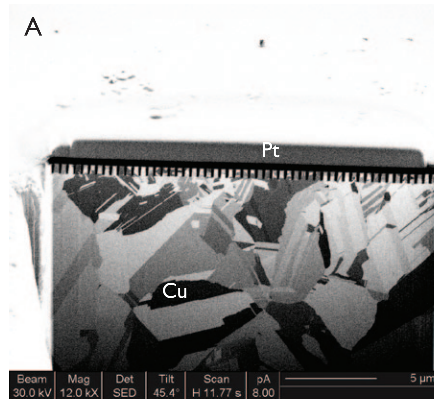


Fig. 1: Using the FIB, holes with various shapes were sputtered in a Mo-covered Si_3N_4 film. The large circle at the top part of the image was sputtered at the highest current. For the smaller features visible at the bottom lower currents were used.



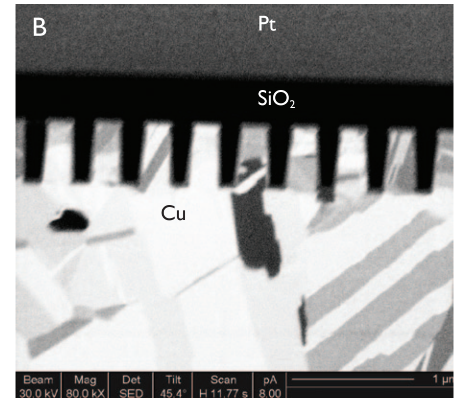


Fig. 2: (A) a three-layer system viewed in cross-section in the FIB. In the bottom part of the images a thick Cu-layer is visible: the different shades in grey correspond with different crystallographic orientations. (B) displays a detailed view of (A). On top of the Cu-layer a black comb-like structure is visible. This is a hole-pattern in the Cu filled up with SiO_2. The top (grey) layer is the Pt-protection layer.'

Basic principle

A Focussed Ion Beam (FIB) is in many ways similar to a SEM (Scanning Electron Microscope). The main difference is that ions are used, instead of electrons. Most FIB instruments are equipped with a Ga Liquid Metal Ion Source (LMIS). By combining heating with a certain extraction voltage, a Ga-ion beam is obtained. A set of magnetic lenses focuses the ions and allows scanning of the ion beam over a dedicated area. The ion beam can be used for local removal ("sputtering"), local deposition, as well as for imaging of material.

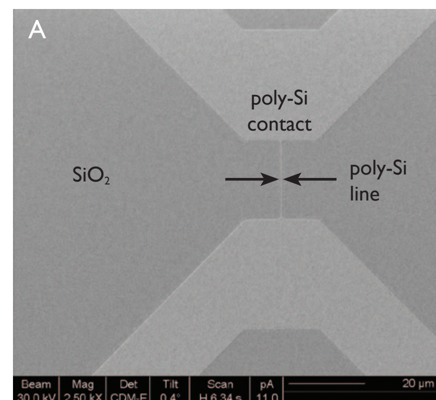
Sputtering

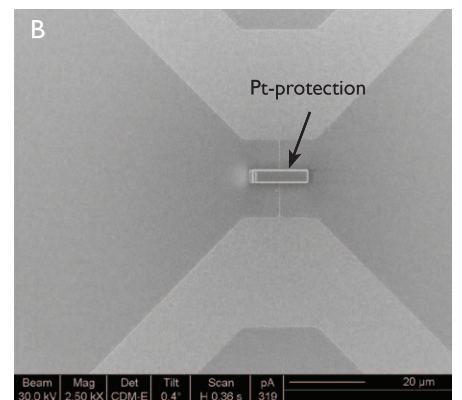
The most common application of the FIB is the removal of material by sputtering. A set of files with material-dependent sputter rates and pre-programmed shapes (i.e. donuts, rectangles, polygons) makes it possible to remove material at a desired location with desired dimensions.

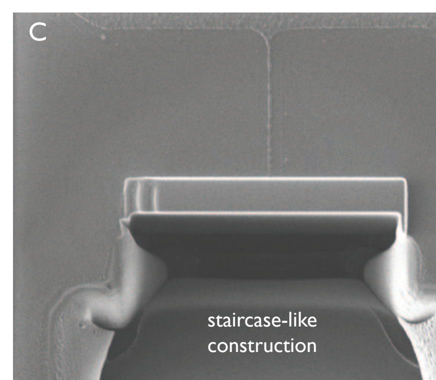
Varying the current of the ion beam influences the sputter rate. A higher beam current increases the sputter rate but decreases the resolution. Therefore, higher currents are used to remove large amounts of material, whereas lower currents are used for precise sputtering of features with small dimensions (see fig. 1) and for the final stage in the sputter process.

Deposition

By means of a needle a gas can be injected in the sample chamber. Depending on the gas components, sputter rates can be influenced or material can be deposited. For example, by cracking a volatile metal-organic Pt-precursor a thin strip consisting of platinum can be deposited. Similar to sputtering, the desired location and dimensions of the Pt-strip can be adjusted. Such a Pt-strip can be used as an electrical contact or as a protection layer. The electrical contacts can be used for IC-modification or for realising electrical test structures, e.g. to electrically address semi-conducting nanowires.







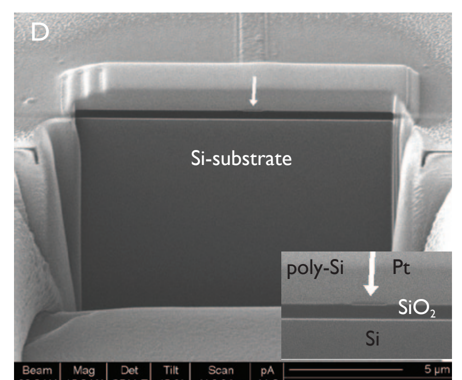


Fig. 3: The route for preparation of cross-section samples for SEM or FIB as illustrated by SE images acquired in the FIB. In (A) a patterned poly-Si structure on a SiO_2 covered Si-wafer is visible. The thin line visible at about the middle of the image had to be imaged in cross-section in the SEM. In (B) the Pt-protection layer is deposited on the feature of interest. In (C) the staircase-like construction is sputtered. In (D) the sample is tilted 45° and hence the cross-section can be imaged.

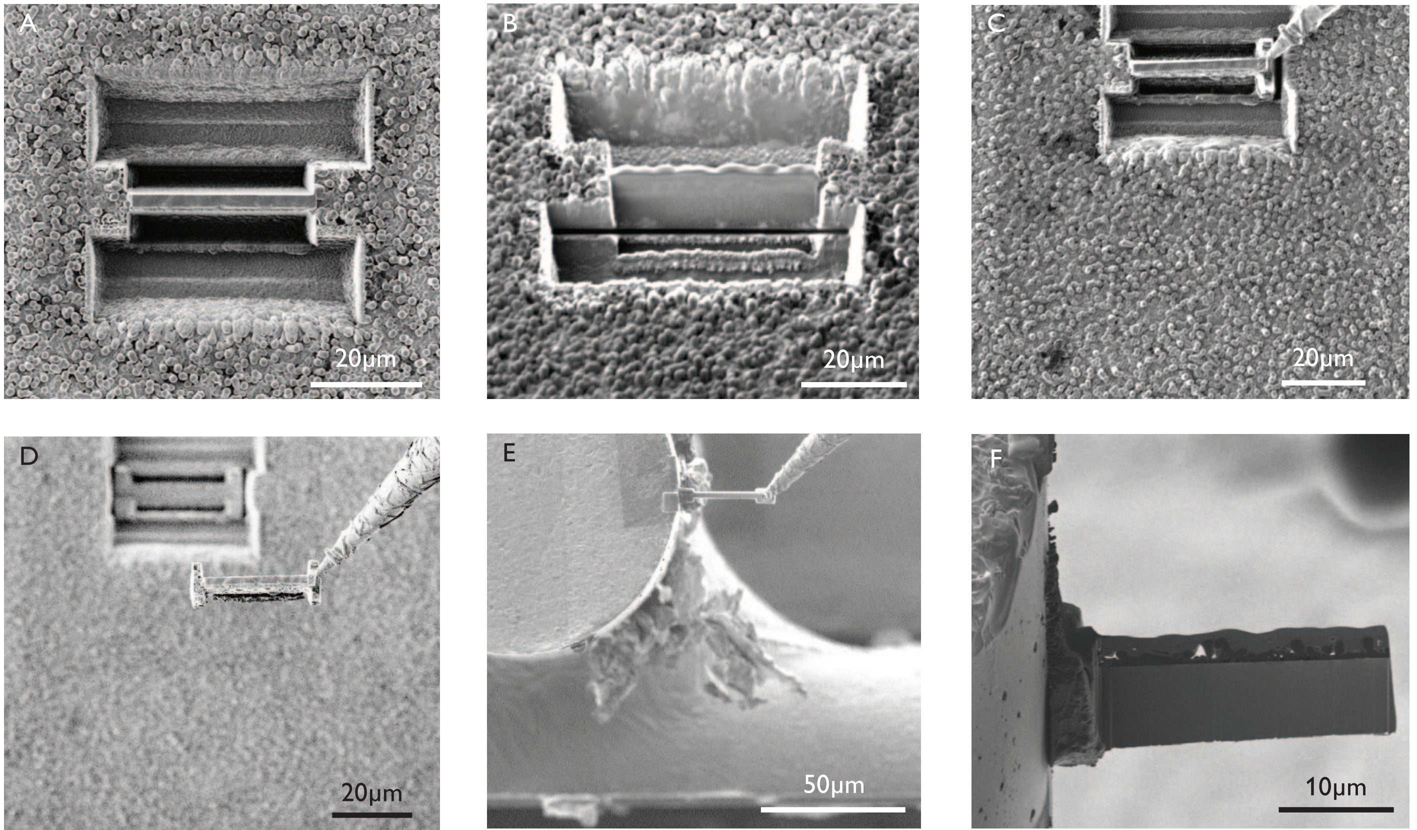


Fig. 4: Lift-out TEM sample preparation example. (A) displays the surface of a sample in which two parallel trenches are sputtered. In between the trenches a membrane is situated containing the feature of interest. In (B) the sample is tilted 45° and the bottom of the membrane is cut loose by sputtering. (C) displays the membrane after attachment (by means of Pt-deposition) to the lift-out needle: the membrane is cut loose from the right side now. After cutting loose the left side of the membrane the sample can be lifted-out, like shown in (D). In (E) the sample is attached to a supporting grid that fits in a TEM-holder. After cutting loose the lift-out needle, the membrane gets its final thinning to obtain optimal electron transparency. In (F) the supporting grid is tilted 45° and the cross-section made can be checked in the FIB. The sample is now ready for TEM study.

Imaging

When an ion beam interacts with material, secondary electrons are generated. These secondary electrons (SE) can be detected and used to form an image. The SE yield depends on the kind of material and crystallographic orientation. Grains that differ in their crystallographic orientation with respect to the ion beam will have different brightness in the SE image (see fig. 2). Hence, information on grain size and crystallographic distribution can be obtained.

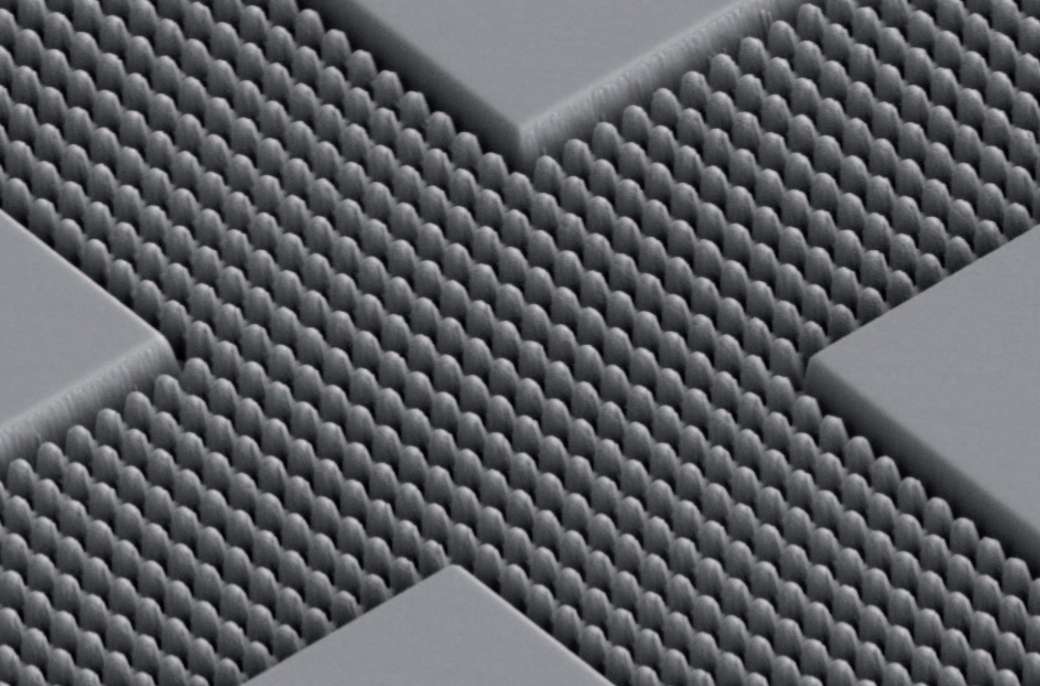
Sample preparation

Many methods exist to prepare cross-sectional samples for SEM or TEM studies. Amongst them are fracturing, cutting, polishing or the 'Schlieff'-method. However, the above-mentioned methods are not sufficient if either

1) the optical microscopy assisted preparation fails because the feature of interest is too small or hidden in between non-transparent layers, or
2) delaminating or smear out of features/ layers of interest occurs. In these cases, FIB-preparation is essential.

Sample preparation for SEM or FIB

Before sputtering is started, a Pt-based protection layer is deposited on the location of interest (see fig. 3A and B). Using the FIB it is possible to sputter a hole in the surface with one steep edge (oriented perpendicularly to the sample surface) at the region of interest and a step like construction at the opposite side (see fig. 3C). In this way it is possible to make a cross-section that can be studied by tilting the sample 45°. The cross-section can be studied either directly in the FIB (see fig. 3D) or in the SEM. Repeatedly sputtering and imaging the cross-section can reveal 3D-information. Advantages of a FIB cross-section analysis are 1) the good contrast in crystalline material and 2) the fact that the sample is not exposed to air after making a cross-section. However, limitations are the resolution of ~7 nm (depending on the materials present), the sputtering of the cross-section surface while imaging and the lack of possibilities to obtain compositional information. These might be reasons to choose for the SEM to perform the cross-section analysis.



Ni/P surface in which a pattern is milled based on a bitmap-file.

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Technical Note 19
May 2012

Sample preparation for TEM

For Transmission Electron Microscope (TEM) analysis a very thin sample (membrane) is needed (thickness ~100 nm). Such a thickness is necessary to allow transmission of the electrons through the membrane. At the desired location on a sample a membrane containing the feature of interest is lifted-out (see fig. 4A to D). This membrane is attached to a support grid that fits in a TEM specimen-holder (see fig. 4E and F). Both atomic resolution imaging as chemical analysis on nanometer scale are possible on such membranes in a TEM.

Small Dual Beam (SDB)

The SDB is a machine that is equipped with both a SEM and a FIB column. This combination has several advantages.

1) A cross-section made using the FIB can be imaged directly using the SEM without exposure to air.
2) Automation software enables the serial sectioning and imaging through a site-specific volume. The acquired image series can be merged into a movie or reconstructed to a 3D-image. In this way, 3-dimensional morphological information is obtained, for example on local defects, crack propagation or delamination.
3) In addition, electron beam assisted deposition of Pt, W and TEOS is possible.

Applications

- trouble shooting
- layer thickness determination
- cross-section analysis
- thickness modification
- nanometer scale feature fabrication

Characteristics

Sample type:
- Solid material; non-conductive materials have to be coated with a 20 nm conductive layer (Pt). Additional protection/contrast layers of Al or Si_3N_4 are deposited if required outside the FIB.
- Biological material (tissue) when it is chemically fixated.

Sample size:
- 4 x 4 x 2 cm (l x w x h) maximum

Magnification:
- 300x-800,000x (FIB);
- 32x-1,280,000x (SEM)

Imaging resolution:
- 7 nm (FIB), 1.1 nm (SEM)

Sputter characteristics:
- Lateral resolution: < 15 nm
- Largest dimensions: lateral dimensions up to 100 x 100 µm, depth: 30 µm

Deposition:
- Pt, W and TEOS can be deposited
  Layer thickness: 20 µm maximum
  Width: 50 nm minimum
  Length: 100 µm maximum
  (depending on width)

Final dimensions of SEM/TEM sample:
- Minimum feature size: 50 nm (in only one direction: the other direction needs to be bigger)
- Width: maximally 60 µm
- Depth: 30 µm for SEM; 8 µm for TEM

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