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INSTITUTE FOR NANOTECHNOLOGY
NanoLab Twente

TEM SPECIMEN PREPARATION

The art of TEM specimen preparation; an introduction:

Specimen preparation is an essential part of Transmission Electron Microscopy (TEM). Without a good specimen, no TEM. TEM specimens must be electron transparent and representative of the material one wants to study. In most cases it is desirable to have specimens which are uniformly thin, stable under the electron beam, conducting and nonmagnetic. Clear exceptions are of course magnetic materials for which magnetic properties have to be investigated, e.g., with Lorentz Microscopy, and metals or alloys in which crystal defects (dislocations) have to be studied; they should not be made too thin. There are many ways to prepare specimens for TEM. The chosen method will depend on both the type of material and the information one needs to obtain. Most important aspect to bear in mind is that the preparation technique must never affect what one observes or measures.

Specimen preparation is an extremely broad subject. Upon surveying the literature, numerous books have been devoted to this topic alone.

> For an extensive overview of TEM specimen preparation techniques, including corresponding literature references, may we refer to an excellent source of information on the web, created by Jeanne Ayache and coworkers (Ref. [1]).

MESA+ NanoLab has a well equipped set of facilities for inorganic TEM/SEM sample preparation, including the following techniques:

- Cutting, grinding, lapping, polishing
- Dimple grinding (TEM only)
- Material removal by Argon ion sputtering (TEM: GATAN PIPS, SEM: PECS)
- Standard metal carbon/metal coating
- High resolution carbon/metal coating (GATAN PECS)
- Wedge or TRIPOD polishing (TEM only)
- Focused Ion Beam (FIB)

For the observation of samples in cross-section, MESA+ NanoLab is specialised in the technique Dimple Grinding/Polishing and Argon ion sputtering, as depicted on the following pages 2 - 4. This is in particular applicable for „blanket“ films on a substrate. With precise control during the final stages of the thinning process, large, uniformly thin, electron transparent areas can be achieved, - with a minimum amount of damage - (see, e.g., Ref. [2]). Especially when care is taken that the Argon ion thinning stage is terminated just before an etch hole is created (see left XTEM image on page 2). In this case it is essential that the operator keeps his/her eye continuously on the ion thinning process. In Ref. [3] we have been able to show electron transparency beyond 6 micron.

For cross-sectional observation of more complex samples, such as „device-like“ systems, TEM specimen preparation by FIB is often the only viable alternative, and nowadays the preferred method. Applicable to a large variety of materials (virtually all than can be ion milled).

REFERENCES

[1] web site „Transmission Electron Microscopy (TEM): sample preparation guide“, by Jeanne Ayache and coworkers, http://temsamprep.in2p3.fr/

[2] E.G. Keim, M.D. Bijker, J.C. Lodder, J. Vac. Sci. Technol. A19(4), Jul/Aug 2001.

[3] E.G. Keim, L.T. Nguyen, J.C. Lodder, Proceedings Microscopy & Microanalysis 2002 Meeting, Quebec City, Quebec (CA), August 4-8, 2002, pages 1346CD-1347CD. ISBN 0-521-82405-2.

[4] Illustrations used on page 4, from/inspired by: M. Kawasaki, T. Yoshioka, M. Shiojiri, J. Electron Microscopy 48(2) (1999) 131-137. Associated text is however, adapted to our modified recipe of Dimple Grinding and Argon ion etching.

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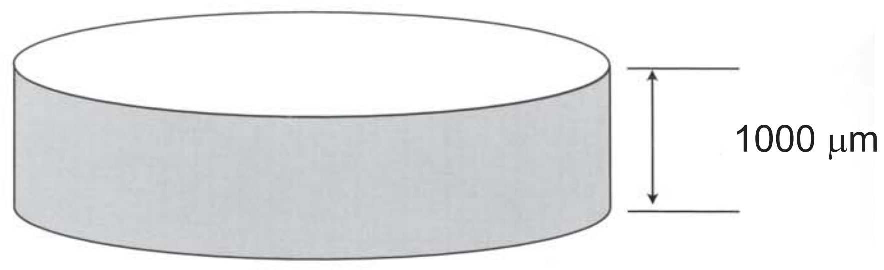
TEM SPECIMEN PREPARATION

The technique of Dimple Grinding/Polishing and Argon ion etching to prepare TEM cross-sectional specimens; a basic outline:

Step 1

The Gatan Ultrasonic Disc Cutter is used for coring or cutting TEM discs from brittle materials such as ceramics and semiconductors.

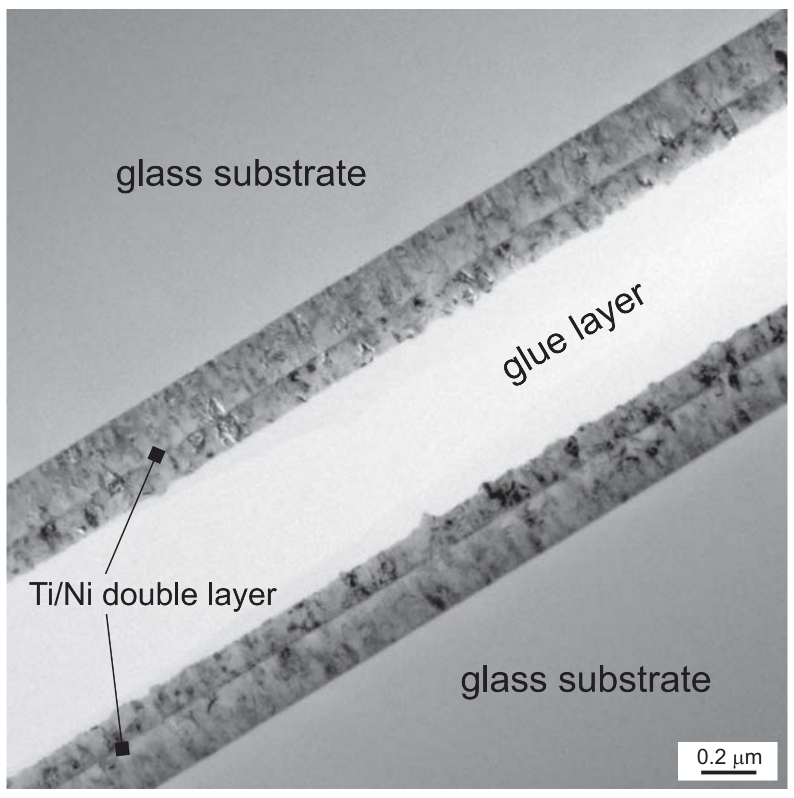


XTEM

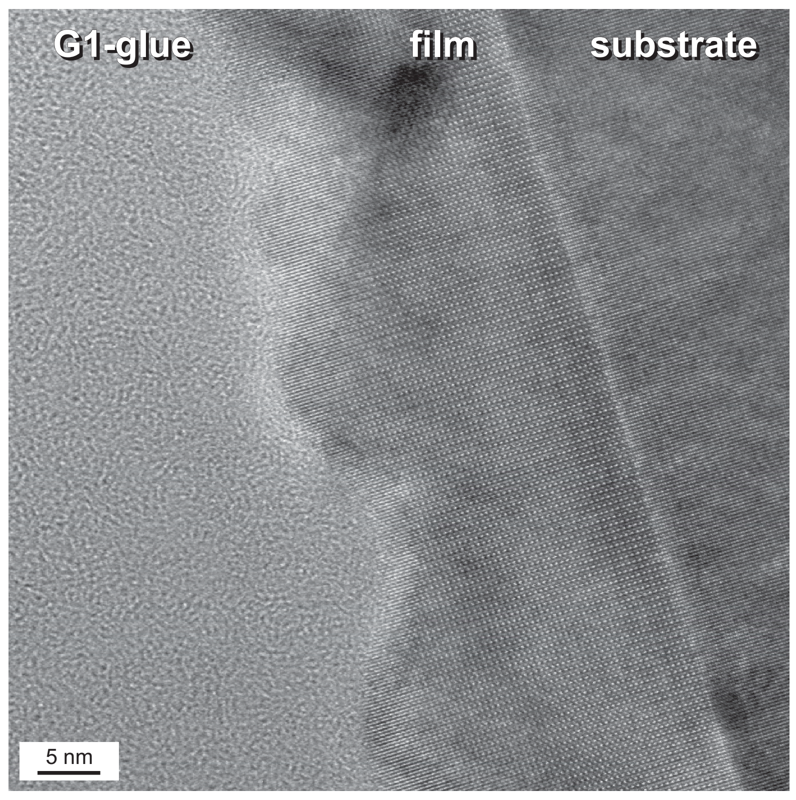
In conjunction with Gatan's other specimen preparation equipment, the XTEM kit is ideal for preparing cross-sections from almost any material encountered.

G-1 epoxy features:

- ideal for bonding ceramics, metals, glass, plastics, and semiconductors
- high strength
- resistence to solvents
- very fast curing times
- high temperature stability



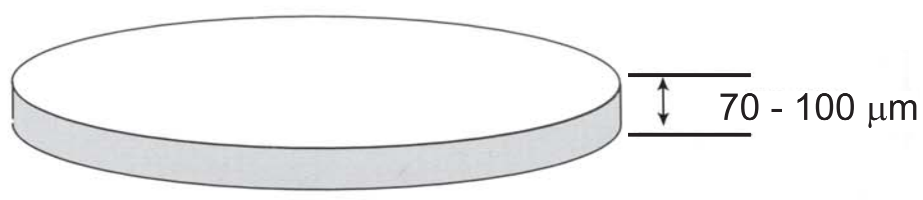
Fully preserved cross-sectional (XTEM) specimen in a symmetric sandwich shape, w/o physical etch-hole! Notice long-range.



High-quality high-resolution TEM lattice imaging feasible, a result obtained after dimple-grinding/polishing and Ar^+ etching.

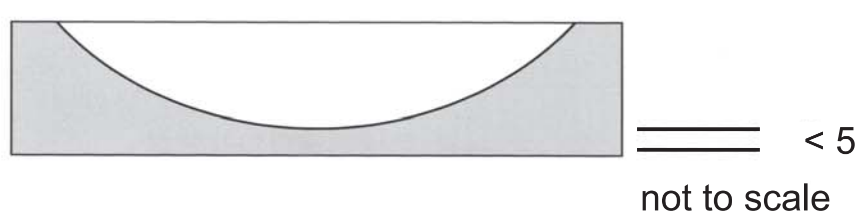
Step 2

Parallel-sided discs are prepared by using a precision disc grinder.



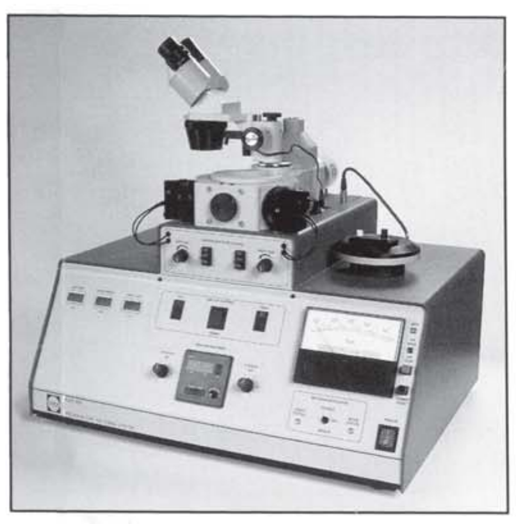
Step 3

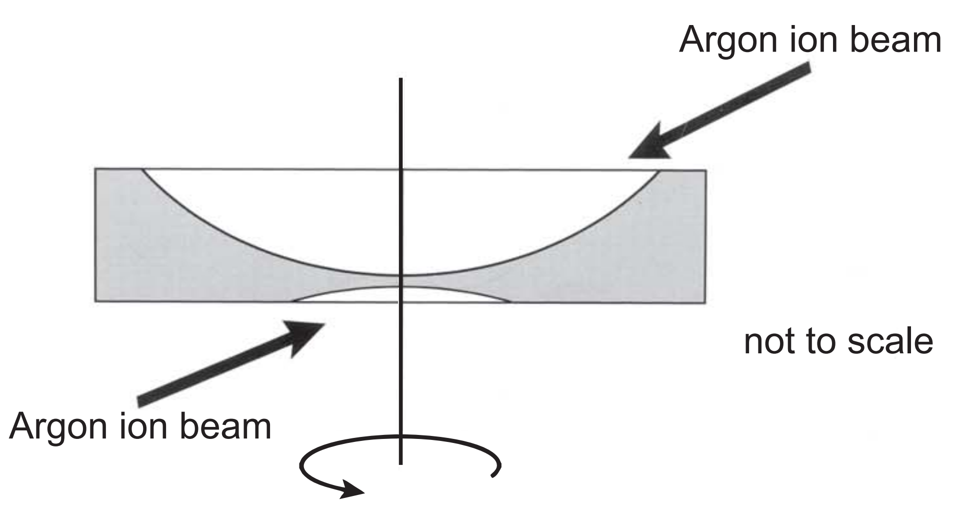
Mechanical dimpling thins the centre of the sample, yielding shorter preparation times and produces a larger thin area with thicker rims that makes fragile samples easier to handle.



Step 4

ion milling in the PIPS features variable low angle (< 1°), low argon ion energy, double-sided milling, with efficient thinning rates, and larger transparent areas with a minimum of damage/amorphisation.





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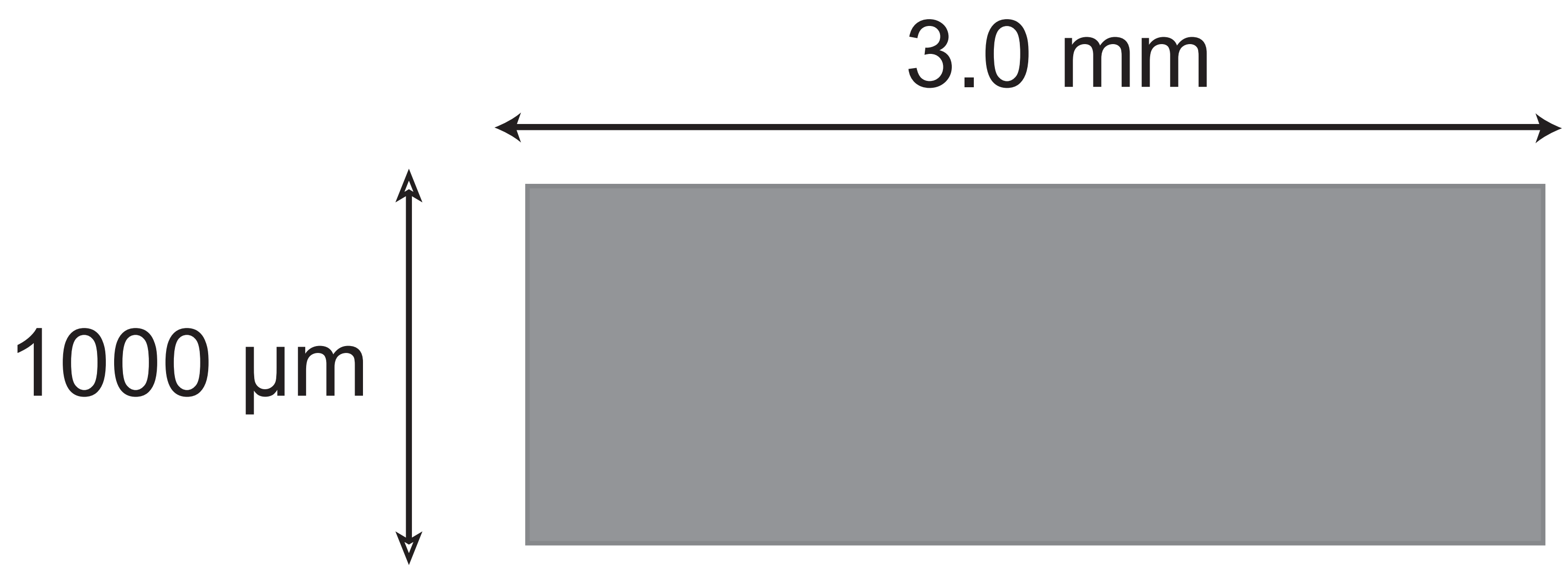
TEM SPECIMEN PREPARATION

The technique of Dimple Grinding/Polishing and Argon ion etching to prepare cross-sectional TEM specimens; shown in 4 easy Steps:



Step 1

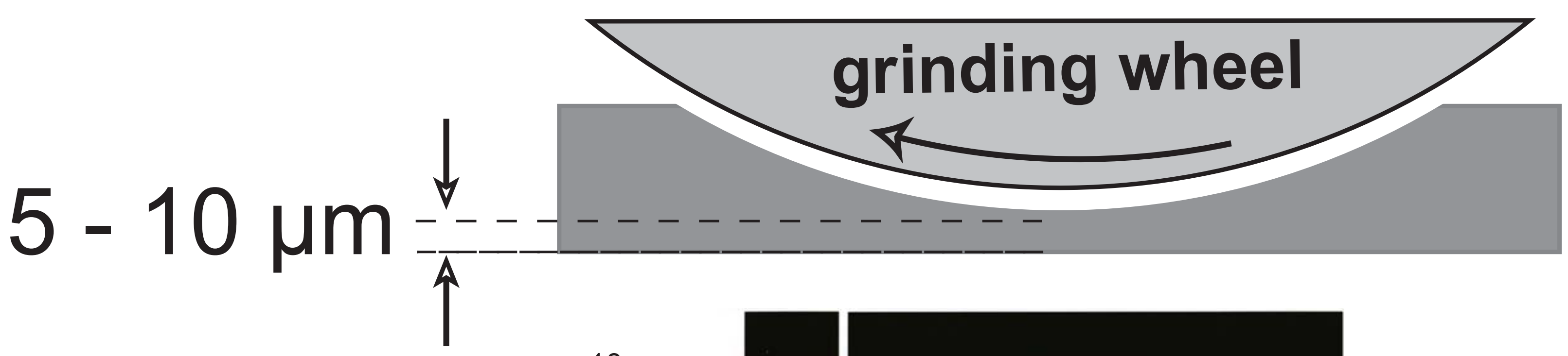
Sample Cutting:

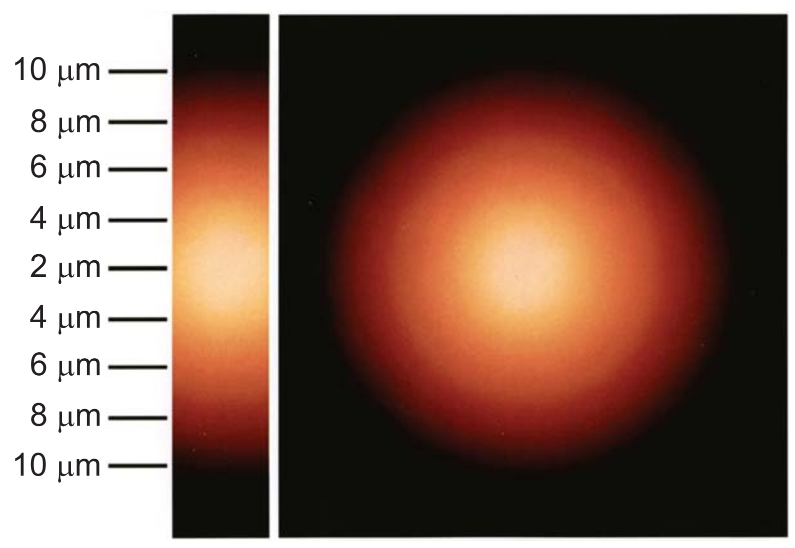
using an ultrasonic Disc Cutting machine, circular discs are extracted. The disc is then embedded into a re-enforcing Cu ring for enhanced ruggedness and stability.

Step 2

Mechanical grinding:

a sequence of wet diamond paper; using Grit size 500, 1200, 2400, and finally, 4000.





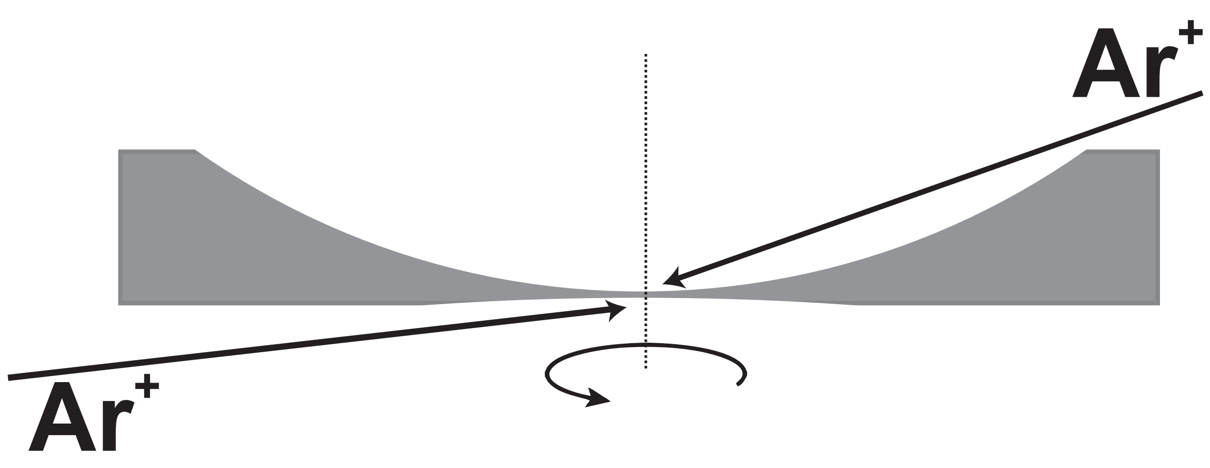
Transparency colours versus thickness in dimpled Si(100) single crystal (illustration, courtesy GATAN)

Step 3

Dimpling/Polishing:

One-side dimpling only, with a 15 mm diameter SS wheel for the grinding stage. Using oil-based diamond paste in the sequence 6, 1, 0.25 μm. Polishing occurs in 2 stages with a soft felt-wheel: extra grinding/polishing using 1 and 0.25 μm oil-based diamond paste as long as required (~orange/yellow colour in silicon), followed by a short polishing stage using a 0.05 μm alumina suspension.

Applying the same soft felt-wheel, the „flat-side" is polished only using a sequence of 1 μm diamond paste for 90 s, 0.25 μm 30 s, finalised by polishing with an alumina suspension for 2 min.



Step 4

Argon Ion Milling:

Electron transparency is achieved by low-angle argon ion milling: +4° down, -2° up. Ion energies, on average, typically, in steps from 4.5 down to 0.5 keV.

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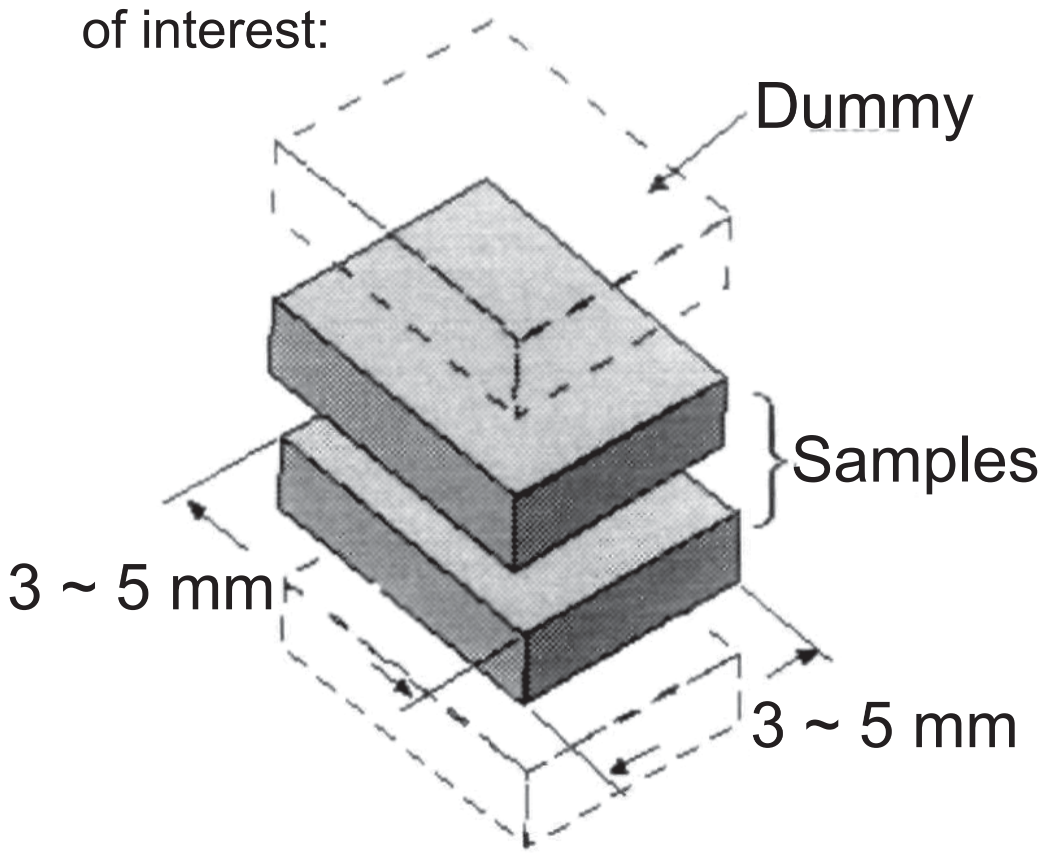
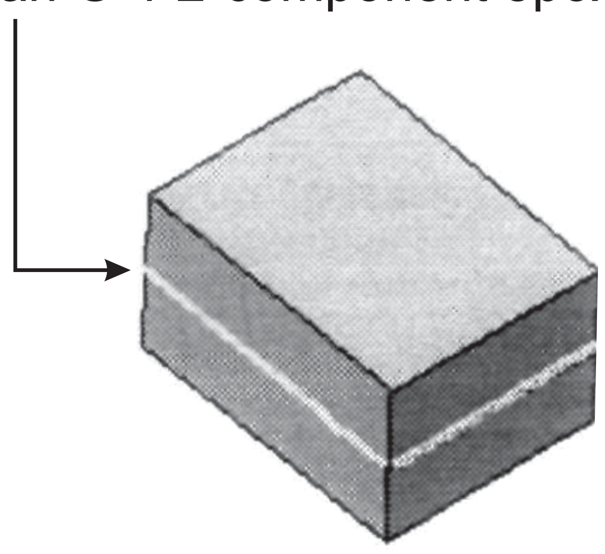
Dimple Grinding/Polishing + Ar^+ etching; a detailed schematic pictorial overview shown in 8 stages:

1. Cutting and assembling sample material of interest:



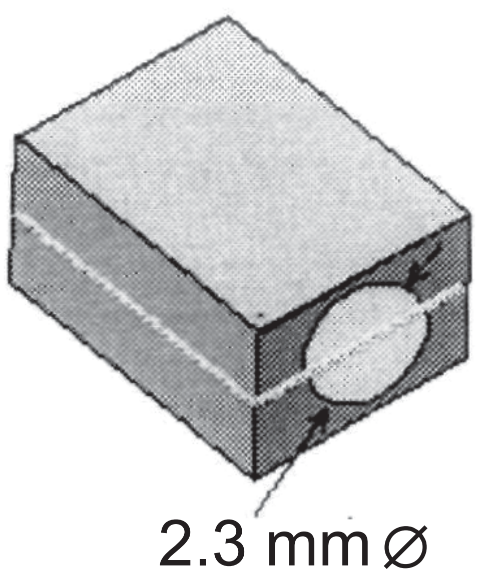
The dummy material is used to create a stack which is approx. 3 - 4 mm in height

2. Gatan G-1 2-component epoxy cement

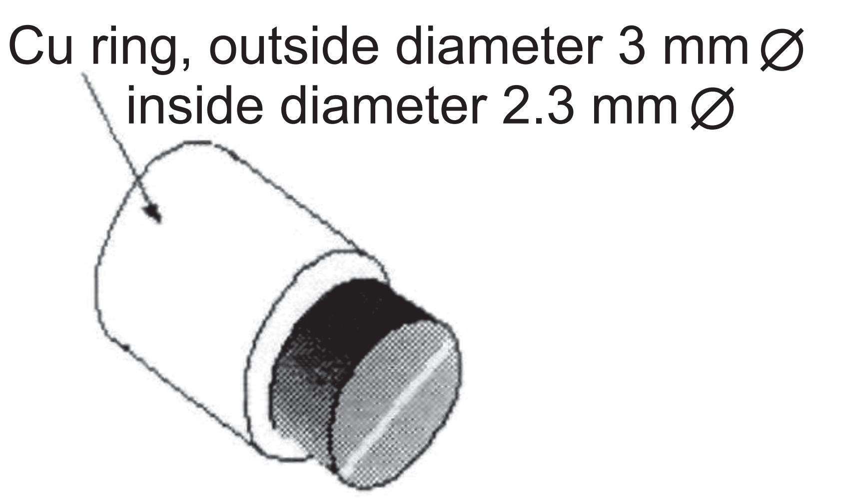


The sample material with the features-of-interest, together with the dummy material (preferably Si), is shaped into a stack using an appropriate epoxy resin.

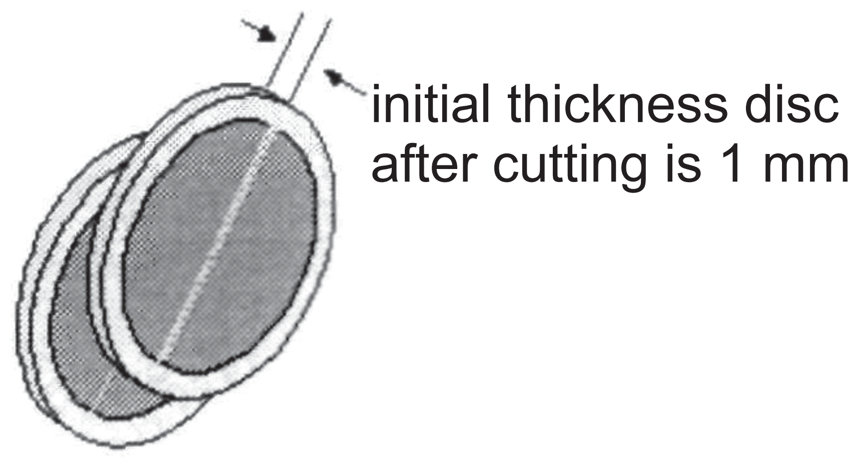
3. Retrieving cylindrically shaped sandwich by ultrasonic disc cutting (Step 1)



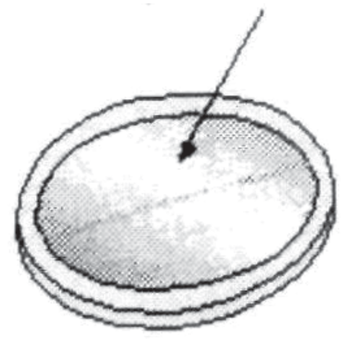
4. Re-enforcing Cu tube for enhanced ruggedness and stability



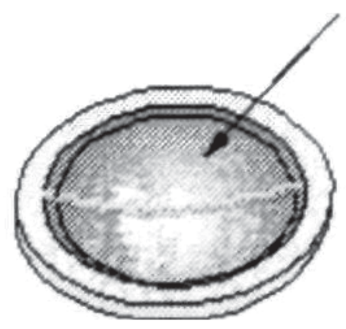
5. Step 2, cutting and grinding to ~ 70 μm



6. Flat side, one side, polishing only (= final phase of Step 2, followed by initial phase of Step 3: polishing only)



7. Other side, to be dimpled: by grinding + polishing, down to approx. 5 μm (Step 3)



8. Argon ion etching at very low angles of incidence (Step 4)