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Sample preparation for HIM by Broad Ion Beam Cross Sectioning

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Introduction

Cross-sectional sample preparation for thin film and bulk samples is often challenging if large areas are to be inspected or if sample consists of porous or soft materials. Broad Ion Beam (BIB) cross sectioning is an advanced method for preparing accurate cross section surfaces for a wide variety of materials. With the use of an argon ion beam and a mask plate a clean planar cross-section can be achieved with minimal amount of artifacts on the cross-section surface. BIB excels in comparison to traditional methods such as mechanical polishing or microtome when cross-sectioning soft materials, very hard materials, porous materials or combinations of these. The BIB cross-sectioned surface area is approximately 1 mm in width and several hundred micrometers in length which is very large compared to those made with FIB (Focused Ion Beam).

Orion NanoFab in Jyväskylä, Finland

Finland got its first helium ion microscope when Zeiss Orion NanoFab tool was installed in the Nanoscience Center of the University of Jyväskylä in October 2015. This HIM tool is only second in Nordic countries. The acquisition was a joint effort of Departments of Physics and Biology and Environmental Sciences.

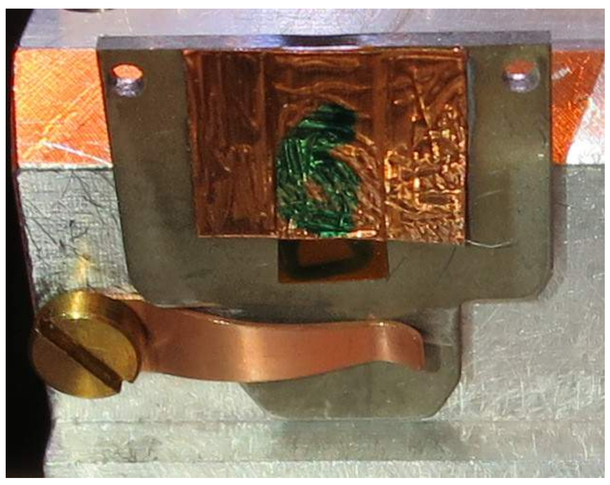
The HIM is equipped with a neon option and Raith Elphy pattern generator and it is installed in a cleanroom environment.

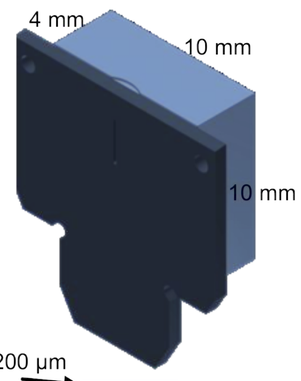
Broad Ion Beam Cross Sectioning

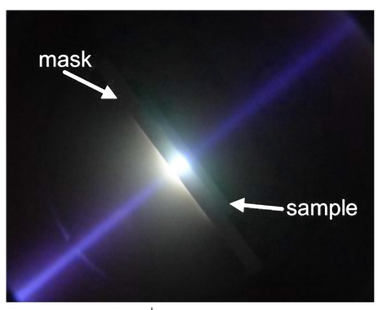
In broad ion beam (BIB) cross sectioning a sample is first cut and trimmed for suitable size and shape. It is then attached to a sample holder (titanium) which acts as a mask plate. A small part (20-200 µm) of the sample is left visible above the edge of the mask. The broad argon ion beam is aligned perpendicular on the edge of the mask so that half of the beam hits the sample and the other half hits the mask. The ions start removing material from the visible area of the sample while the mask plate protects the rest. Over time the ion bombardment results in a clean planar cross section area of the sample behind the mask plate edge.

Typical parameters of the Gatan Ilion system

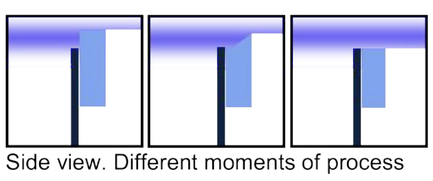
- Dual Ion Guns: argon ions
- Beam Energy: 100 eV to 6.0 keV Milling Rate: > 140 µm/hr (Si, 6 keV) Cut Width: 0.5 - 1.5 mm, adjustable Milling Angle: ±10 degrees, adjustable
- Sample rotation: Ion beam active for - 30 to +30 degrees from normal (plane direction). Decreases curtaining.
- Operating Pressure: <1x10^{-4} torr Titanium Mask
- Sample Cooling System: Liquid nitrogen — Minimum sample temperature: - 120 °C







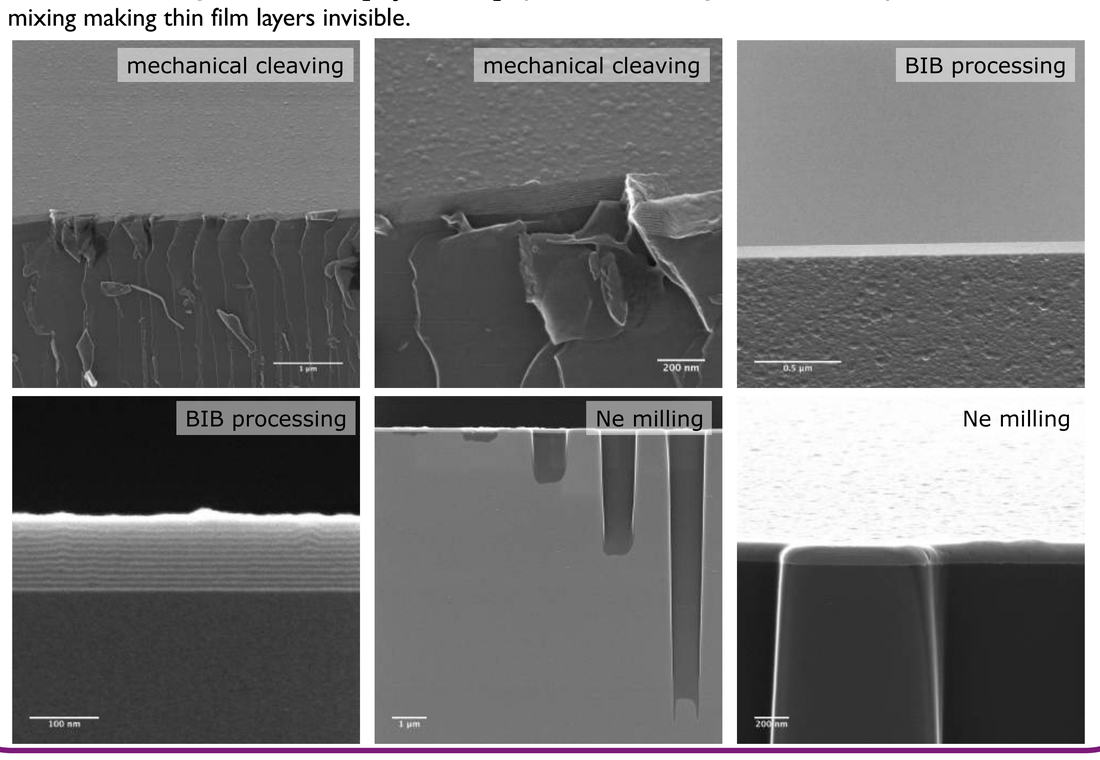
Top view of Ar^+-ion beam in action



Side view. Different moments of process

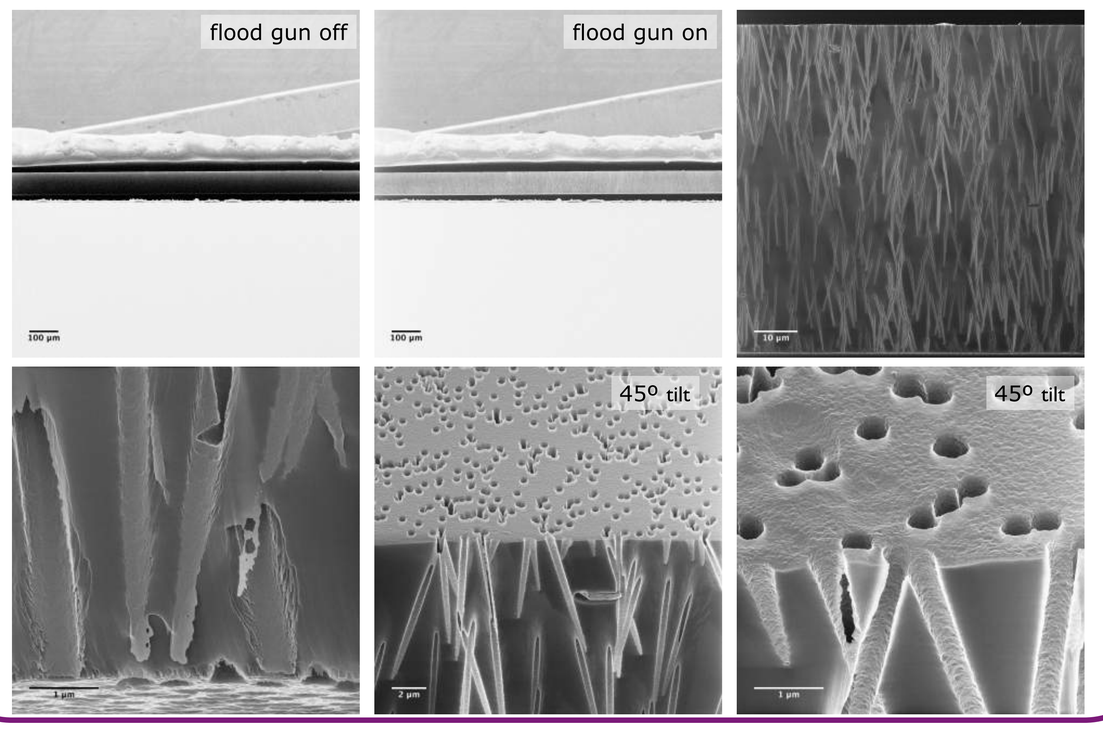
Thin film imaging

Mechanical cleaving of silicon wafers deposited with thin films results often in a rough edge where film imaging is difficult. We have used BIB processing for atomic layer deposited (ALD) nanolaminate films with 10 alternating 5-nm thick Al_2O_3 and TiO_2 layers. Neon milling with HIM usually results in ion beam mixing making thin film layers invisible.



Ion tracks in Kapton

Self-supporting 75 µm thick polyimide membranes (Kapton) were irradiated at the Accelerator Laboratory of the University of Jyväskylä with 600 MeV Xe^{25+} beam. The irradiated doses were 10^7 to 10^9 ions/cm^2, and single irradiations lasted from seconds to thirty minutes. Irradiation angle was continuously varied between ±15° and the foil was also rotated in order to get crossing ion tracks but still keeping the maximum pore diameter through the foil fixed. Irradiated foils were etched in sodium hypochlorite (NaOCl) and boric acid (H_3BO_3) solution to reach pore diameter of 300 nm.



IC devices

BIB processing was used for a IC memory chip from a USB stick. Cross-sectional imaging allows for observing very cleary different layers in the IC structure. Neon beam milling allows for further profiling of the structures of interest.