

Application of Low Energy Broad Ion Beam Milling to Improve the Quality of FIB Prepared TEM Samples

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Introduction

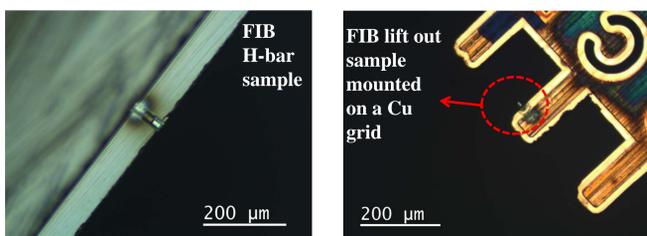
Focused ion beam (FIB) is one of the most common techniques for TEM sample preparation. FIB is especially viable when specific sample orientation or site specific positional accuracy is required. On the other hand, some of the damage types caused by this method are amorphization, redeposition, implantation, vacancies, sample alteration due to beam heating, surface roughness and selective abrasion [1].

Amongst all these problems, FIB induced surface amorphization is the most extensive and limiting. The reason is two fold. Firstly, the amorphous material reduces the signal to noise ratio [2]. Secondly, in order to avoid geometrical blurring for high resolution transmission electron microscopy (HRTEM) applications, the thickness of the sample must be reduced by the square root of the feature size [3]. A 25 KeV Ga+ FIB column can generate ~20 nm per specimen side in damage for a Si sample [4] and this could be specifically limiting in the case of modern Si based semiconductor devices with less than quarter micron features.

Experiments

In this study, the quality of FIB prepared TEM samples was improved and the FIB induced amorphous layer thickness was reduced by the use of low energy (<300 eV), broad beam ion milling in *Gatan's Precision Ion Polishing System II (PIPS II)*. Multiple new features in PIPS II such as focused ion beams at low energies, X-Y alignment stage, optical camera along with Digital Micrograph imaging software and stationary milling mode, make this possible.

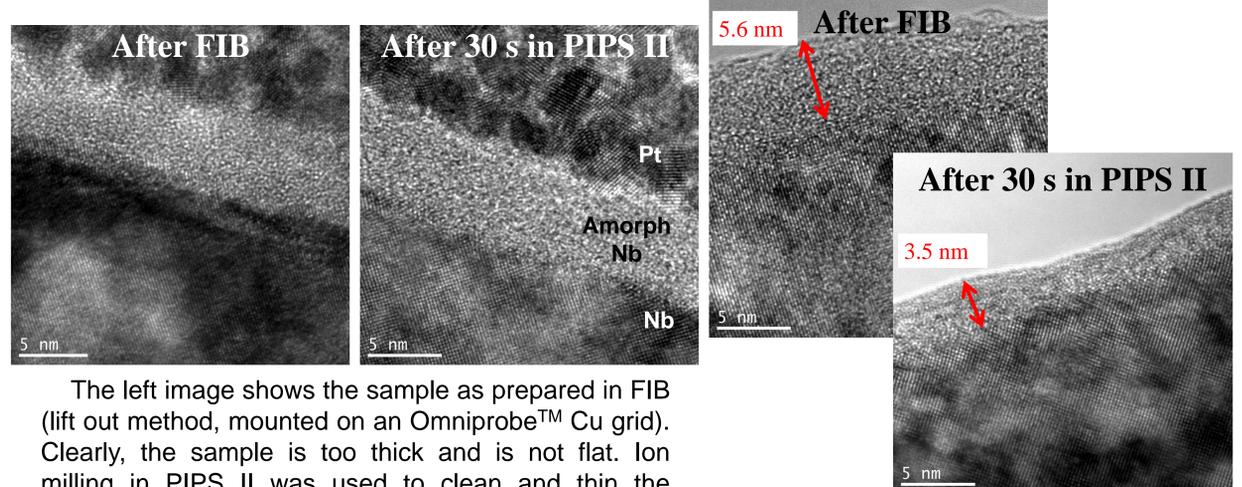
Several FIB prepared specimens made of different materials were polished in PIPS II. In the case of lift-out samples, they were mounted on either Cu or Mo OmniProbe™ grids. Specimens were imaged before polishing in the PIPS II, as to have a reference of the sample condition after preparation in FIB. Samples were then polished using low energy Ar ion guns in stages with sequential observation in a TEM. Argon Beams were set to mill at incident angles <5° from either top or bottom. Samples were cooled during polishing to reduce heat induced damage. Samples were positioned using the PIPS II X-Y stage with respect to each gun and the sample was stationary during milling. Milling the samples in Stationary Mode and focused ion guns at low energies made it possible to polish the FIB sample efficiently at <300 eV, without redeposition of the holder material (Al) or the grid they were mounted on (Cu or Mo).



Considerations

- **Beam energy:** Low energy ions have smaller penetration depth, which reduces the ion induced surface damage.
- **Milling time:** Because ion beams are well focused in PIPS II, the current density at the milling area is high, thus material removal rate is high. Milling time must be optimized to remove enough material to improve sample quality, but not to over thin.
- **Milling angle:** A higher beam angle results in increase in ion induced surface damage. In these applications, low angles are desired, within any constraints from mounting grid geometry.
- **Sample temperature:** Due to ion bombardment, the sample temperature at the mill area increases. This can result in heat induced damage unless cooling is applied.
- **Type of supporting grid:** Mo has a lower sputtering rate compared to Cu, which reduces the chance of re-deposition from the grid onto the FIB lamella.
- **Position of FIB lamella on the supporting grid:** the lamella should be carefully positioned to reduce re-deposition.
- **Ion milling mode:** it was found that keeping the sample stationary and milling with only one gun provides the best results (PIPS II Stationary milling mode).

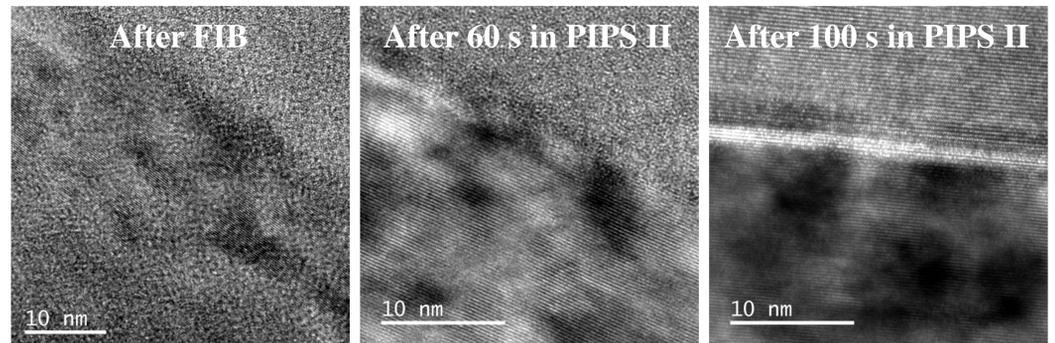
Sample 1: Cut from Nb Sample



The left image shows the sample as prepared in FIB (lift out method, mounted on an Omniprobe™ Cu grid). Clearly, the sample is too thick and is not flat. Ion milling in PIPS II was used to clean and thin the sample; sample was cooled to -80 °C. Stationary milling mode was selected and the gun was set at 300 eV and at 3° from the top side, to mill for 30 s. The image on the right shows the sample after it was milled in the PIPS. The Nb layer is thinner and flat.

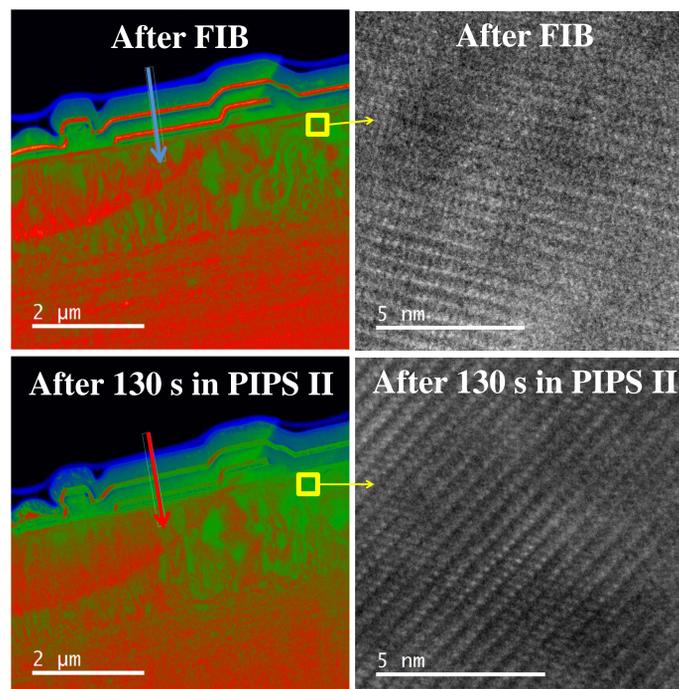
The removal of FIB produced amorphous surface layers in Nb sample can be readily seen in this sample. The images above show changes after only 30 s milling at 300 eV. In this time, the amorphous layer was reduced from about 5.6 nm to about 3.5 nm (~40% reduction).

Sample 2: Multi Layer GaN



TEM micrographs of a multi layer sample. The image on the left was captured as prepared in FIB (H-bar). The image in the middle and on the right were taken after polishing in PIPS II for 60 s and after polishing for additional 40 s, respectively; due to FIB induced amorphization, the thinner interlayer is not visible in the first and second images, but after sufficient Ar ion polishing it is clearly visible in the last image.

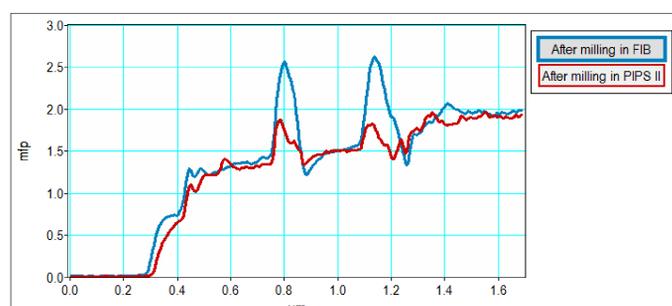
Sample 3: Semiconductor Device



The color images on the left show the thickness maps* of the same area in the device, captured after milling in FIB (H-bar) and low energy milling in PIPS II (300 eV beam, stationary milling mode, 130 s). Sample thickness can be compared in these two images using the profile lines along the arrows shown in the graph below.

TEM micrographs on the right, show high resolution images taken before and after low energy ion milling in PIPS II (taken from the areas shown by yellow squares on the thickness maps). By reducing the sample thickness, parts of FIB induced damage on the sample surface has been removed.

*Rainbow color scale on the thickness maps varies from 0 (black) to 4 (white) mfp.



References

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