

TEM Sample Preparation using Gallium & Neon Ion Beams

ZEISS ORION NanoFab

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Date: October 2017

Focused gallium (Ga) ion beams are well known for their use in sample preparation for Transmission Electron Microscope (TEM) investigations. In this application note, we report on integration of TEM lamella preparation hardware and software with ZEISS ORION Nanofab ^[1], as well as preliminary results showing that samples can be prepared using a reproducible workflow, with automated routines for milling and cut out. Relatively heavy metallic gallium ions can cause problems for certain materials. For example, gallium can induce phase transformations in stainless steel, embrittlement in aluminum, and actual microscopic depositions in ceramic materials ^{[2] [3] [4]}. There are ways to reduce the effects of damage, including lowering the beam energy and performing an additional milling step in a low energy argon milling instrument. This latter technique adds hours to the typical focused ion beam process. A recent publication, "Evaluation of neon focused ion beam milling for TEM sample preparation" ^[5] suggested that neon milling could alleviate certain issues associated with gallium damage in aluminum. Further work will be continued with lower energy neon beams to optimize the polishing process".

Introduction

Focused ion beams (FIB) are the conventional method to prepare ultra-thin samples for TEM analysis. A serious drawback of this technique is gallium ion implantation and damage caused by the beam. A TEM lamella prepared by both gallium and neon ion beams should have an advantage compared to a standard gallium focused ion beam for certain samples. The noble gases He and Ne, used as gas field ion sources (GFIS), do not form alloys with metallic samples.

For example, in the case of gallium sensitive materials such as GaAs and aluminum, implanted Ga can only be removed by low energy argon milling which can take several hours in a separate argon miller. TEM images show undesirable gallium segregating along grain boundaries in an aluminum sample after standard gallium FIB lamella preparation (Figure 1), and the microstructure of recrystallized gallium that has been deposited in a ceramic material during TEM preparation (which leads to confusion during the analysis) (Figure 2). Thus *in situ* neon ion beam polishing after gallium bulk milling is a promising technique for advanced samples.

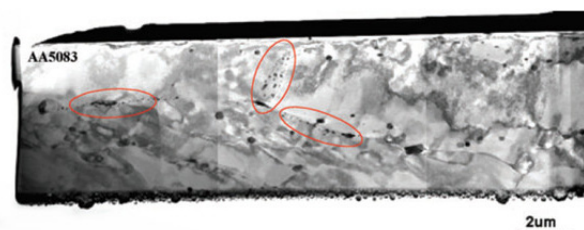


Figure 1 TEM Foil of standard AA5083 Aluminum after only Ga+ milling. Indicated regions are grain boundaries exhibiting discrete pockets of Ga. (Credit: Unocic, 2010)

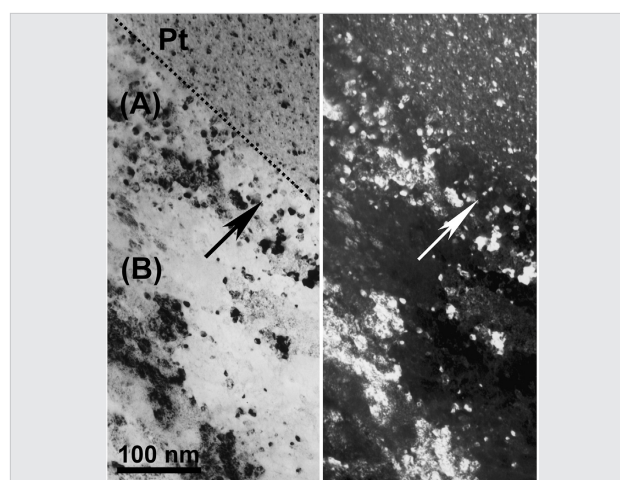


Figure 2 Microstructures of recrystallized gallium depositions visible in both bright and dark field images on a ceramic material with an ion beam deposited platinum coating (Credit: Munoz-Tabares 2013).

Method

ORION NanoFab is configured with He and Ne GFIS columns, a FIB (Ga) column attached at an angle of 54 °, an Oxford Gas Injection System (GISII) and an OmniProbe micro manipulator (OP400) which has four axis of freedom and is capable of in situ needle exchange capacity (Figure 3). TEM analysis was carried out on a JEOL 2100 TEM at 200 kV.

The fully automated cut-out process uses the following steps:

- Metal deposition on top of the targeted sample site of sample as a protection layer
- Trenching from bulk
- Medium polish
- Cut out

Following the gallium based bulk and medium milling and cut out steps, the lamella is lifted out using the OP400 and transferred to a standard TEM grid via the standard welding and cut off process. Preparing an initial lamella of 1 μm thick, 10 μm long and 5 μm deep with 600nm Pt protection layer takes 20 minutes. Lifting out the lamella and welding at the TEM grid takes another 10 minutes. The total sample preparation takes 2-3 hours depending on the user's experience. This workflow is reproducible and similar to other TEM lamella preparation workflows.

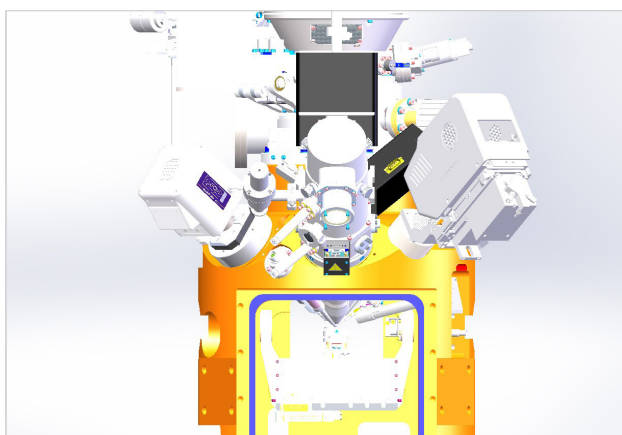


Figure 3 Physical layout of ZEISS ORION NanoFab chamber for TEM lamella preparation.

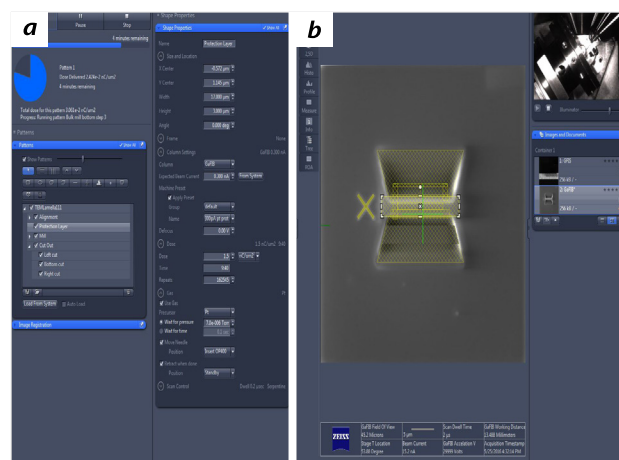
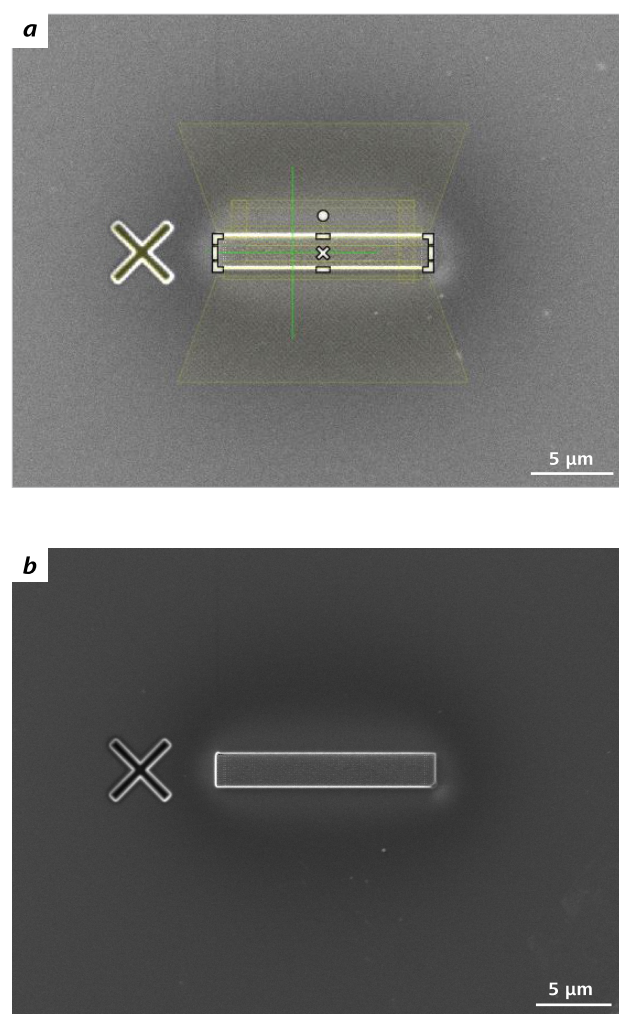


Figure 4 Software automatically controlling (a) lamella pattern on target sample (b).



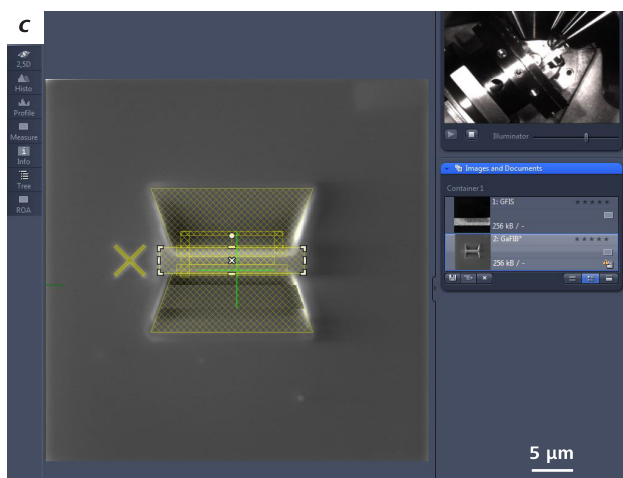


Figure 5 Metal deposition for protection layer (a, b), after trenced cut and during medium polish (c).

Lamella cut out, lift out & attach to TEM grid workflow

The Lamella lift out is carried out at 0 ° sample tilt with GIS probe inserted and bringing the OP400 to a eucentric height 300 μm above the sample. The OP400 needle is placed at the top of lamella corner by viewing the helium image and placing the tip of the needle on top (but 300 μm above) of lamella by manipulating the x and y axis movement.

The gallium beam is used to image and bring the needle down as close as possible to the lamella corner. The OP400 tip is welded to the lamella corner using a 10 pA Ga beam. The final cut to free the connected side of lamella with tip is done with a 100 pA Ga beam followed by a final lift out with z-axis movement (Figure 7). To attach the lamella to the desired grid position, both the GIS and OP400 needle are need to be at the inserted state.

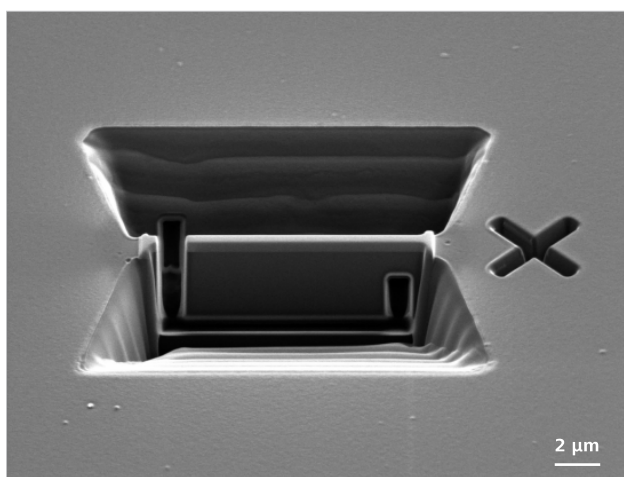


Figure 6 After lamella cuts, prior to completion of cut out and lift out

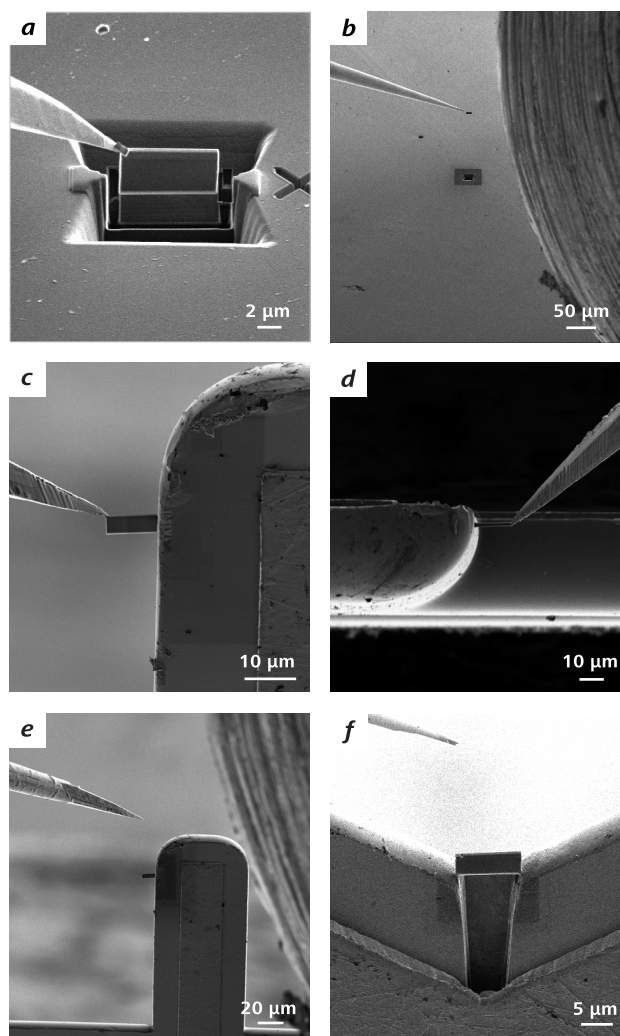


Figure 7 Final lamella lift out steps

Thinning with the Neon Beam

Thinning the lifted out lamella with Ne beam is done in several steps. Successive polishing with the neon beam thins the initial 1 μm lamella to 100 nm. Fine polishing is done to a final thickness of <50 nm. All the thinning/milling is done from both sides of lamella at a tilt of $\pm 3^\circ$. The thickness of the polished lamella is measured from the top view using a low kV Ne image.

Figure 8 shows a tilted view of low kV Ne polish and image of thin lamella. The measured thickness from a top view image is less than 50 nm.

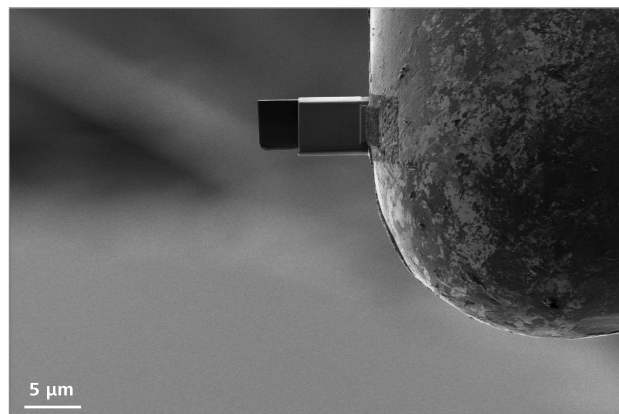


Figure 8 Ne image of Ne polished lamella.

Conclusion

TEM lamellas can be prepared using ORION NanoFab configured with gallium, helium and neon ion beams, platinum deposition, and OP400 nano manipulator. TEM lamellae can be prepared using a combination of rough and medium milling with gallium, followed by lift out and polishing with neon. Results of both pure silicon and semiconductor samples maintain atomic level crystallinity when visualized in a high voltage, high-resolution TEM, as seen in Figures 9 and 10. The TEM sample preparation workflow is very similar to traditional FIB-SEM instruments.

These very preliminary results indicate that neon has the potential to remove gallium implantation/amorphous layers and that the influence of beam energy needs improvement.

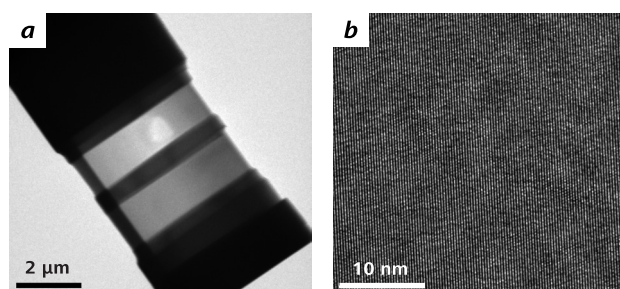


Figure 9 TEM image of the Si sample prepared with 10 kV Ne beam (a). TEM image (b) shows the Si crystal lattice.

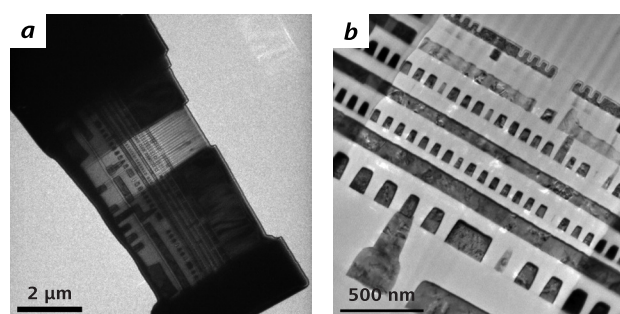


Figure 10 TEM images of a FIN FET sample (a) and (b) which shows Ne beam can thin down the lamella to electron transparency.

References:

- [1] "Focused Ne⁺ Ion Beams for Final Polishing of TEM Lamella prepared through Ga-FIB systems" D.Wei, C.Huynh, A.Ribbe, *Microsc.Microanal.* 21 (Suppl 3) (2015).
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