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APPLICATION NOTE

Atom Probe Tomography sample prep using Plasma FIB technology

Microstructural characterization for materials analysis

Atom probe tomography (APT) provides microstructural characterization at the highest spatial resolution for 3D compositional analysis of materials. Focused ion beam (FIB) microscopy is an essential technique for orientation and site-specific sample preparation for APT work.

For APT, dimensions of analyzed volumes are on the order of 50 nm x 50 nm x 20–500 nm; the depth depends on the quality of the specimen and position of features of interest. APT sample preparation is similar to TEM preparation, with criteria for every sample being fundamentally the same.

1. Needle-shaped specimen with tip radius typically less than 50 nm (to apply 20–40 V/nm electric field to allow field ion evaporation).
2. Uniform, circular cross-section of the tip to produce a radially symmetric electric field.
3. Correct taper angle to support robust tip for voltage plus or laser plus for significant evaporation events to occur.
4. Minimal damage introduced to the tip during specimen preparation; apex region of needle should represent original sample in terms of microstructure and composition.

Gallium liquid metal ion source (LMIS) technology has been the workhorse for FIB applications since it was introduced commercially more than 25 years ago. Over the years, Ga FIB beam properties and low-acceleration voltage performance have increased steadily. Today's most advanced Ga LIMS FIB probe size is 5 nm to 7 nm of full width at half-maximum, compared to nearly 50 nm in the mid-1980s. Samples prepared from metals and alloy bulk materials based on aluminium, iron, copper and titanium are very sensitive to Ga FIB damage. The damage affects sample surface amorphization, depending on beam energy and incident angle. Damage is also generated by introduced defects such as dislocation loops and formed intermetallic compounds in grain boundaries. Ga FIB can prepare well-shaped APT samples; however, Ga FIB-prepared APT samples can exhibit problems as shown in Figure 1.

The introduction of a Thermo Scientific™ DualBeam™ system with Xenon plasma FIB (PFIB) technology enables Ga-free sample preparation. Xe PFIB beam diameter is bigger than Ga FIB, which raises questions among FIB users. Can it be used to prepare samples for APT applications? If so, what are the benefits of using samples prepared by Xe PFIB?

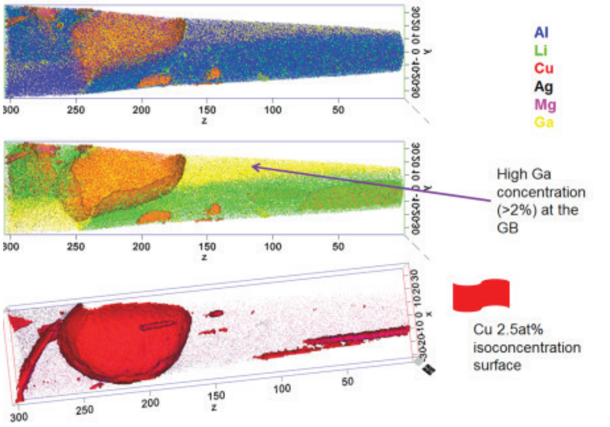

Figure 1: a) Gallium can decorate dislocation, b) Gallium concentration higher at apex of tip, up to 2% or even more, c) Gallium enriching at interface between matrix and precipitates for aluminium alloys, d) Copper alloy is very sensitive to Gallium damage, such as Cu-Ag alloy forming Ga-based intermetallic.

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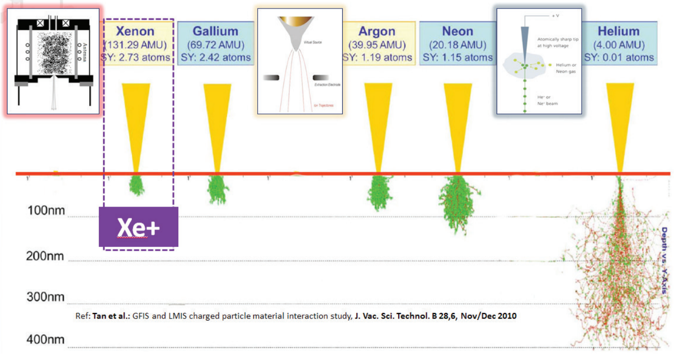


Ref: **Tan et al.:** GFIS and LMIS charged particle material interaction study, **J. Vac. Sci. Technol. B 28,6, Nov/Dec 2010**

Figure 2: Types of ion sources and simulations of ion sputter yield and implant depth of He+, Ne+, Ar+, Ga+ and Xe+ ions at 0-degree incident angle to single-crystal Si at 30 keV.

Figure 2 displays ion beam implantation and sputtering with simulation of ion species striking silicon at an incident energy of 30 keV using the TRIM Monte Carlo simulation. Clearly, the low-mass ions penetrate deeply into the sample but produce very low sputter yield.

The heavy element Xe, relative to lighter element ions such as helium, neon and gallium, penetrates to a shallower implant depth and produces increased sputter yield. The Xe plasma ion source is a broad area source with higher angular intensity. As such, it delivers good-quality high currents for large-volume material milling when compared to Ga FIB, where the classic Ga beam leaves the source with divergent angles, so the angular intensity is low.

The helium ion beam relies upon a cryogenically cooled, sharp tip in a UHV vacuum system, to which small amounts of helium gas have been admitted. It is point source, with a virtual source size of about 0.3 nm. This is good for imaging; however, it is impossible to use for APT sample preparation. Low-mass ion such as helium generate greater damage to the substrate materials if used in the same ion dose as Ga and Xe. Prior research results demonstrate that, in silicon, helium ions create amorphization in the near surface and also nearly 300 nm deep while also producing helium bubbles in the deeper substrate.

It is most important that FIB-prepared APT samples remain representative of the original sample in terms of composition. They should not introduce artifacts or change the microstructure. Because Xe ions originate from a noble gas, they are unlikely to bond with target materials while milling for APT. In this article, we describe APT sample preparation of an AlZrSc alloy using PFIB and present the results from APT analysis.

APT sample preparation and results

Workflow automation

Using Thermo Scientific™ AutoScript™ 4 or iFast™ Software, it is possible to automate the workflow, starting with the electron beam-protection deposition layer to annular pattern milling and, finally, to low-kV FIB cleaning. An example of the process workflow and part of the iFast Software recipe is shown in Figure 3. An instructional dialogue box pops up automatically in the process to help users make a decision for processing the next procedure. Simply clicking OK continues the process. This automation helps improve throughput and reproducibility while also bringing significant ease of use, allowing access to high-quality results by operators who use this technique only infrequently.

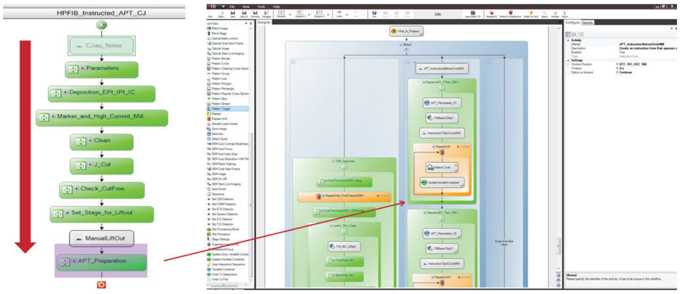


Figure 3: iFast Software-instructed semi-automatic workflow for Xe PFIB APT sample preparation.

µA beam high-current milling

The Xe plasma ion source is able to deliver beam currents higher than 2 µA, enabling fast material remove. Ga FIB systems generally provide typical beam currents from 1 pA to 65 nA at 30 kV. A crossover in beam spot performance between Ga LMIS and Xe inductively coupled plasma (ICP) ion source is around 50 nA, above which the ICP source offers better performance. This is because the Ga beam originates from a small virtual source with a higher divergent angle emitting the beam, and spherical aberration dominates beam performance when beam current increases to the 10s of nA current, where the large aperture opening angle is required to deliver high beam current.

Figure 4 is the first step of 2.5uA beam milling to create front and back trenches after deposition protection; mill time is less than 30s to create front and back 50um wide trenches. This is more than 20 times faster than gallium FIB mill. With a few tens second of 200nA beam current for cleaning mill, the sample is ready for taking cross-section images and for later bottom and side free "J-shape" cut with 15nA current.

There are many ways to do rough milling before lift-out for atom probe preparation. A main goal is to do the rough milling as fast as possible and the rough mill should not generate too much damage which cannot be cleaned by final milling. A routine method is trunk 'V" shape mill, it is suitable for low current gallium FIB since it reduces the amount of materials removing before lift-out, however, for high current PFIB it is unnecessary to do trunk "V" shape mill , which requires stage rotation relatively 180 degree and tilt. Lift-out using fully integrated EasyLift is much less challenging compared to other types of nanomanipulators. Since the lifted sample is about 40 to 50um wide, so it is way to make 5 to 8 pre-probe samples, as show in the Figure 4 a lift-out process.

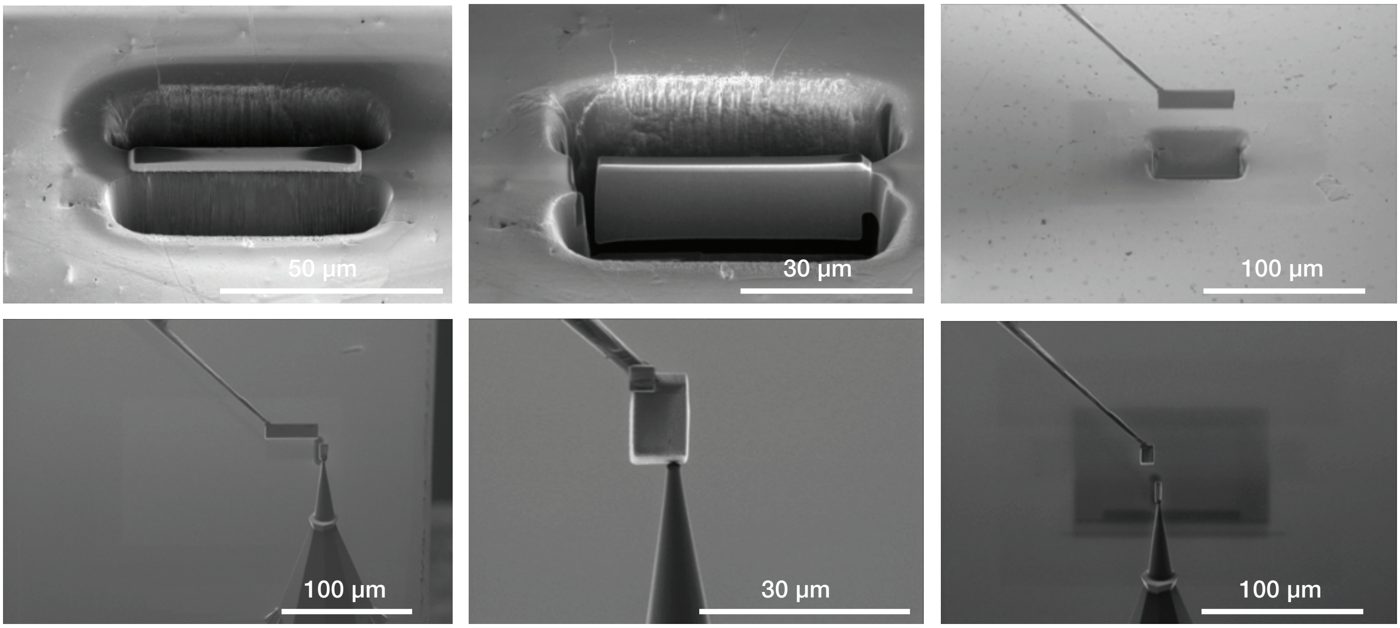


Figure 4: PFIB APT sample rough mill and lift-out. (a)–(b) SEM images of a sample rough milled by 2.5 uA FIB, with free J-cut completed on one side and bottom. (c)–(f) FIB images of lift-out process, with one lift for a few APT samples.

Annular milling process

Annular milling is a procedure for sharpening an atom probe tip containing the region of interest (ROI). It requires multi-circle patterns for milling with an outer diameter slightly larger than that of the needle and an inner diameter larger than the inner radius required for the tip. The outer diameter should be kept constant to prevent a projection being formed on the shank of the post, which will interfere with atom probe data collection. An automation recipe can generate multi-ring patterns automatically with defined step sizes for reducing inner radii and also defined steps with progressively lower ion currents until required dimensions are reached. The tip shape can be controlled by changing the step size of reduced inner radius, wherein the smaller the step size, the lower the taper angles. Figure 5 shows an example of how different step sizes created different tip shapes

The final stage of tip preparation is low-energy “clean up” milling at 5 keV or less, to remove the damaged layer. 5 kV milling can reduce such an amorphous layer to less than 5 nm. This milling is normally performed as a circle mill of the tip while being simultaneously monitored using the SEM with a DualBeam FIB.

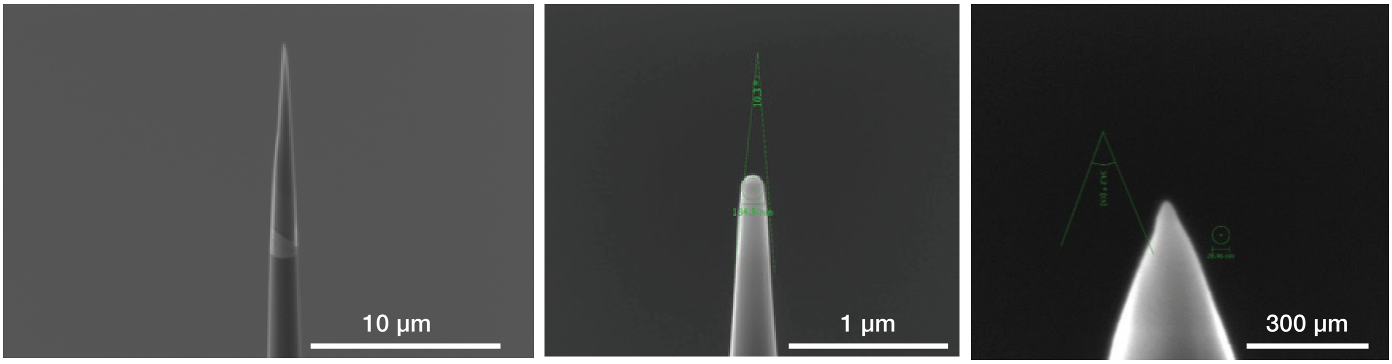
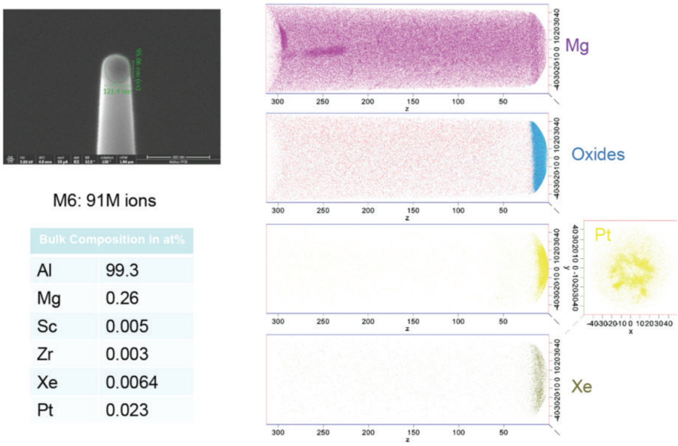


Figure 5: Shape of atom probe tips. (1) Tip is too sharp and too long; inner circle reducing step size of 100 nm. (2) Reasonable tip; inner circle reduced step size of 300 nm. (3) Good tip, sharp and strong; Inner circle reducing step size of 500 nm, and last circle with inner diameter set to 0.

APT results

Ga+ FIB milling is known to change the composition and structure of some type of materials. For a 30 kV annular mill, it has been observed to create an amorphous layer from a single crystal magnesium alloy sample 30 nm from the sidewall of a magnesium alloy post and implant significant amounts of Ga up to 50 nm beneath the surface.

APT can provide 3D mapping of all elements present in an AlZrSc alloy sample prepared by Xe+ PFIB. Local Xe concentration has been measured as close to detection limit (Figure 6).



Bulk Composition in at%	
Al	99.3
Mg	0.26
Sc	0.005
Zr	0.003
Xe	0.0064
Pt	0.023

Figure 6: 3D mapping of all elements present in AlZrSc alloy sample prepared by Xe+ PFIB.

Summary

Xe PFIB is a very promising technique for APT sample preparation. PFIB's high current provides fast throughput for rough milling; one 50 um wide lift-out specimen could provide 8–10 APT samples in a very short time. The high-current density of Ga+ FIB provides faster final milling; however, this can sometimes prove too fast to control the sample's final shape. The low-current density of the Xe ion beam allows for better control of APT final milling. Because Xe ions originate from a noble gas, they do not react with target materials during APT preparations; therefore, Ga contamination is avoided for Ga FIB-sensitive materials.

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