Site-specific sample preparation with the Helios Hydra DualBeam for advanced APT and TEM characterization

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High-resolution transmission electron microscopy (TEM) and atom probe tomography (APT) require high-quality samples.

These thin, precisely shaped pieces are typically extracted from larger macroscopic specimens. Focused ion beam (FIB) milling uses a high-energy beam of ions to selectively remove material from a sample surface, essentially carving out the lamella or probe tip for further analysis.

Different FIB ion species can have drastically different interactions with various materials, and ion species optimization can substantially improve sample quality. This process, however, can be time-consuming and resource intensive, particularly if multiple instruments are required. Thermo Fisher Scientific offers a novel plasma FIB (PFIB) capable of rapid switching between different ion species. The Thermo Scientific™ Helios Hydra™ DualBeam (focused ion beam scanning electron microscope, or FIB-SEM) features four distinct plasma ions: xenon, argon, nitrogen, and oxygen.

In this application note, we explore how researchers at the University of Sydney are utilizing this technology to improve their sample preparation, particularly for advanced atom-probe analysis.

What is atom probe?

In atom probe, a sample is shaped into a sharp tip, typically through electropolishing and/or focused ion beam milling. A high voltage is then applied to this sample tip, generating an intense electric field that causes atoms to be ionized from the tip surface; these atoms then impact onto a sensitive single-atom detector. The time of flight of the ion will indicate which atom it is, and back-projection calculations can determine where on the tip the atom came from. Combined, this information is used to produce high-resolution nanometer-scale 3D maps of the sample; with sufficient energy resolution, even light elements like hydrogen can be detected.

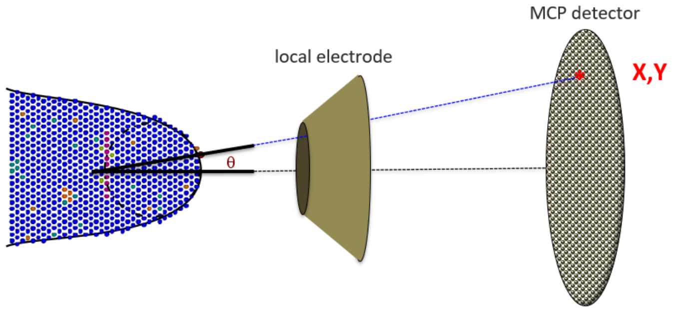

Figure 1. Schematic of the atom probe tomography process (adapted from B. Gault et al., Atom Probe Microscopy, Springer 2012).

Gallium FIB

Atom probe sample preparation has historically been the domain of gallium FIB, but there are inherent limitations to this ion source. Gallium FIB milling can be fairly slow, as it is only capable of removing small amounts of material at a time. Coarse milling must therefore be done by an additional tool before the tip is shaped with gallium. This makes tip preparation a significant bottleneck in a high-demand facility, which is often already running at capacity.

Sample implantation is an additional concern. Ions used for sample milling are not perfectly reflected off the surface; instead, some become embedded in the sample. Implantation depth is impacted by both the source ion and its energy, as well as the composition of the material. Implantation and surface modification can have considerable consequences for sample quality and, ultimately, the conclusions that can be drawn from the measured data. For example, gallium is particularly ill-suited for the milling of aluminum-containing samples, not just due to its high implantation depth, but also because gallium gathers along grain boundaries in aluminum, causing sample embrittlement.

Plasma FIB

To avoid some of the limitations of gallium FIB, researchers at the University of Sydney have successfully used plasma FIB for the preparation of a range of atom probe specimens across industries and materials. For example, PFIB was used to:

- Extract junctions from photovoltaic cells to study their distributions of lithium
- Further analyze alloy grain boundaries identified with electron backscatter diffraction (EBSD)
- Examine the 3D distribution of nanoparticles produced by corrosion

These samples were quickly and effectively prepared, allowing for reliable analysis with atom probe microscopy and/or tomography.

Generally speaking, plasma FIB mills samples at a much faster rate than gallium FIB, speeding up throughput across experiments. At the same voltage, xenon PFIB has also been shown to have a lower implantation length than gallium FIB, preserving more of the sample for contamination-free analysis. With multi-ion instruments like the Helios Hydra DualBeam, researchers can also compare the performance of different ion sources on the same sample to find the most appropriate milling ion for each material. Atom probe tomography, in particular, can be used to determine the precise location and depth of implantation, clearly showing the impact of sample preparation on the atom probe tip.

The signal from implanted ions can also overlap with sample measurements, convoluting results. The flexibility of a fast switchable multi-ions technology allows for the appropriate ion species to be chosen with minimal signal overlap.

Atom probe sample preparation

The parameters that make for a good atom probe tip can be quite exact. Critically, atom probe samples need to have a tip radius of 50–150 nm, and the area of interest has to be captured within the top 100 nm of the tip. This can be challenging, and researchers need to be highly familiar with the procedure to reliably produce tips that encapsulate their areas of interest.

At the University of Sydney, atom probe samples were generally prepared by a combination of coarse laser milling and subsequent electron polishing. This procedure can be fairly challenging, however, as it requires lift out and transfer of the laser-milled samples over to final electron polishing. Researchers would prefer to extract and shape the tips in a single tool, but gallium FIB milling is simply too slow to produce

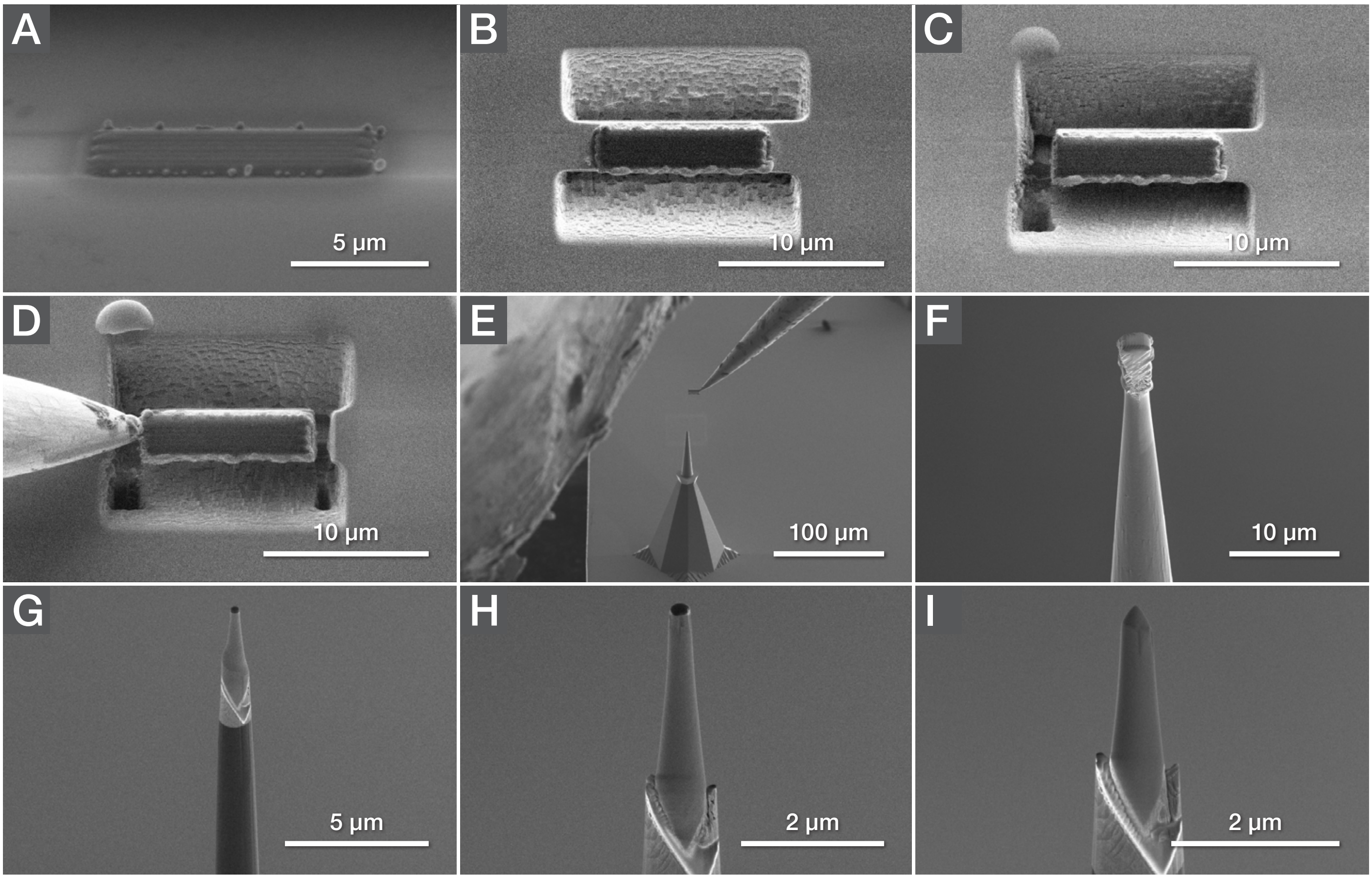

Figure 2. APT sample preparation process using the Helios Plasma FIB. (A) Protective deposition is followed by (B) trenching and (C) undercut. The sample is then (D) lifted out and (E–F) attached to a post where it is subsequently (G-L) thinned using an annular milling process that controls the taper angle.

these samples in a timely manner. High-current plasma FIB, however, can readily produce sharp tips for analysis.

Additionally, in order to minimize beam damage to the sample itself, specimens are typically coated with a protective cap before they are milled. These protective layers are created with electron or ion deposition and are designed to take the brunt of milling damage and implantation. Due to the nature of PFIB, it is important to have a capping layer density that is similar to the sample material. For example, researchers at the University of Sydney use the Helios Hydra DualBeam to mix precursors and create platinum/carbon (Pt/C) caps instead of pure platinum, resulting in improved cut-face quality.

Lasers are frequently used for bulk milling applications, but they leave a wide damage layer that can be tens of micrometers thick. Plasma FIB neatly compliments this technique by quickly removing this entire damage layer, providing a clean starting point for tip shaping.

While this was generally done with xenon PFIB, the flexibility of the Helios Hydra DualBeam allows researchers to experiment with alternative milling species, or additional shaping and polishing with other ions. For example, low-voltage nitrogen and/or oxygen have been used for polishing after xenon thinning.

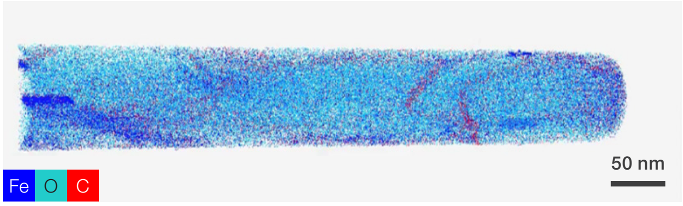

Figure 3. Atom probe data for a Cr_2O_3 protective oxide layer on stainless steel.

Overall, multi-ion plasma FIB has enhanced the speed and ease of use with which researchers at the University of Sydney prepare their atom probe samples. The application space of this technology is still being explored, not just by matching ion sources with various materials, but by sequential application of different ion sources for enhanced milling, shaping, and polishing.

About the contributors

Julie Cairney, PhD—University of Sydney

Julie Cairney is a professor of materials engineering and serves as CEO of Microscopy Australia, a national infrastructure project that provides open access to microscopy instrumentation across Australia. Professor Cairney leads a research group that specializes in advanced microscopy of 3D materials at the atomic scale. Her projects cover steels, light alloys, corrosion, geological materials, and biominerals. She serves as the vice president of the International Field Emission Society, which supports the atom probe microscopy community, and also serves on the advisory board of Ultramicroscopy.

Vijay Bhatia, PhD—University of Sydney

Vijay Bhatia completed his PhD on metallurgy and nano-coatings in 2013. This work relied heavily on scanning electron microscopy and Dr. Bhatia found a passion for the technique. Following his PhD work, Dr. Bhatia spent several years working for Thermo Fisher Scientific as a product specialist, working with industries around Australia to improve processes and workflows. He subsequently returned to the academic world for a postdoctoral position at the University of Sydney. Dr. Bhatia’s work focuses on the development and analysis of novel alloys for mining applications. He also manages the SEM and FIB facilities at ACMM, helping to create better research outcomes.

Limei Yang, PhD—University of Technology Sydney

Dr. Limei Yang is a lecturer at the University of Technology Sydney. She currently focuses on materials for energy conversion and storage in the fields of batteries, hydrogen, solar energy, and thermal energy. She has worked on various types of materials including metals, alloys, oxides, nitrides, and carbides, which exist in different forms such as nanoparticles, nanowires, thin films, and bulk. Prior to her position at UTS, she worked at the University of Sydney, where she focused on materials characterization using focused ion beam, atom probe tomography, and scanning electron microscopy.

Australian Center for Microscopy & Microanalysis

The Australian Center for Microscopy & Microanalysis (ACMM), based at the University of Sydney, is an open-access facility available to researchers across Australia. The ACMM offers a wide variety of instrumentation, serving researchers across disciplines, from materials science to the life sciences. Their offerings include 7 TEMs, 5 SEMs, and 2 FIB-SEMs, including the Helios Hydra DualBeam.

Additionally, they offer assorted light and laser systems such as super-resolution instruments, micro-computed tomography (microCT) tools, and 2 local-electrode atom-probe systems.

The ACMM is also the headquarters of Microscopy Australia, a collection of open-access microscopy labs across the country. These labs serve a range of different research areas, including functional and nano-materials research, advanced manufacturing, geology and biology research, as well as microscopy technique development.

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