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Cryogenic Focused Ion Beam and Scanning Electron Microscopy (Cryo-FIB/SEM)

Cryo FIB uses traditional FIB techniques but with a sample stage that can be controlled to -135°C making it more suitable for soft materials.

Cryogenic focused ion beam and scanning electron microscopy (cryo-FIB/SEM) is a technique used to minimize ion and electron beam damage. Soft materials such as polymers and biological specimens, battery materials, and many other materials systems may deform, melt, or amorphize under room-temperature FIB milling and SEM imaging conditions.

Through cryo-FIB/SEM, the sample is cooled to cryogenic (-130°C) temperatures to minimize these artifacts. The cooling is done by running liquid nitrogen through the CryoMat cold stage installed in our ThermoFisher FEI Helios G3 system. This enables accurate FIB cross-sectioning and SEM measurements of beam sensitive materials with minimal deformation. This also allows for cryogenic FIB lamella preparation for cryo-TEM analysis.

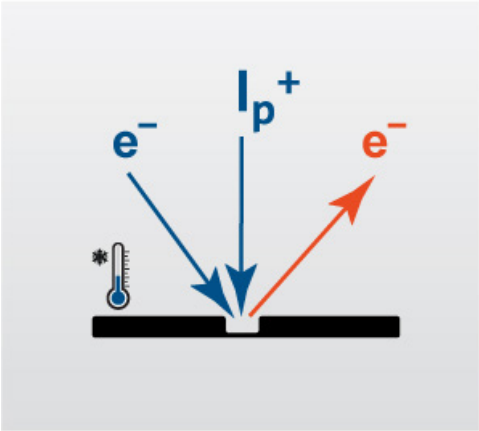

Figure 1. General schematic of how Cryo-FIB works

Strengths

- Minimize FIB artifacts (deformation, melting, bubbling, intermetallic formation)
- Enable the usage of higher ion and electron beam voltages and currents compared to room temperature FIB
- Minimal deformation during SEM imaging and measurements of soft materials

Limitations

- Maximum sample size is 20 x 20 mm wide, 7 mm tall
- Cooling the stage/sample and stabilizing at cryo-temperatures is time-intensive
- Maximum CryoMat stage rotation is +/- 20°
- Electron-beam and ion-beam assisted gas deposition is not possible at cryogenic-temperatures

Common Applications

- Polymers
- Li battery materials
- Photoresist
- Biological specimens
- Beam/heat sensitive metals

Industry Sectors and Technologies

- Energy/batteries
- Plastics/polymers
- Biotechnology

Case Study: Cryo-FIB of Li Metal

Li and its compounds are of great interest to the battery community. However, Li has historically proven difficult to prepare samples from using

room temperature FIB. Li metal is extremely beam-sensitive due to its low density and low melting temperature. These properties make it susceptible to local heating and Ga implantation. Here we show an example of cryo-FIB cross-sectioning and TEM sample preparation from pure Li metal.

In the room temperature FIB cross-section (figure 2), the cross-section of pure Li metal shows severe beam damage with the formation of many voids. These voids also cause heavy curtaining. In the cryo-FIB cross-section (figure 3), void formation is minimized and little to no curtaining is observed. The cryo-FIB is very visibly capable of reducing ion beam damage for beam-sensitive materials.

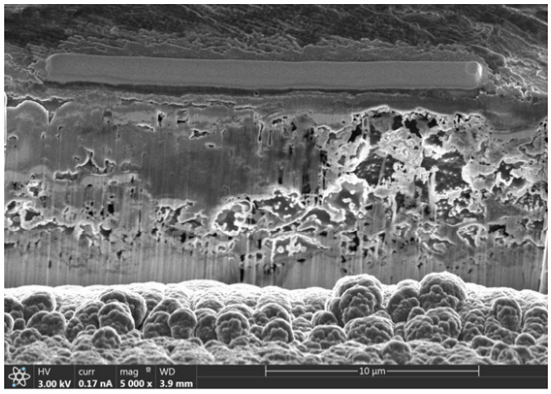

Figure 2. Room temperature FIB

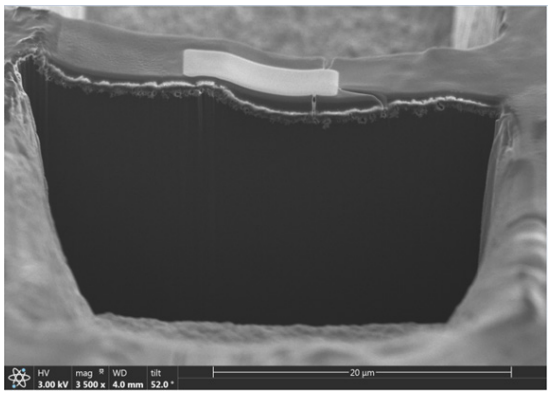

Figure 3. Cryo-FIB

The cryo-FIB was used to prepare an electron transparent TEM lamella from the pure Li metal (figure 4). This lamella was then loaded into a cryo-TEM for analysis.

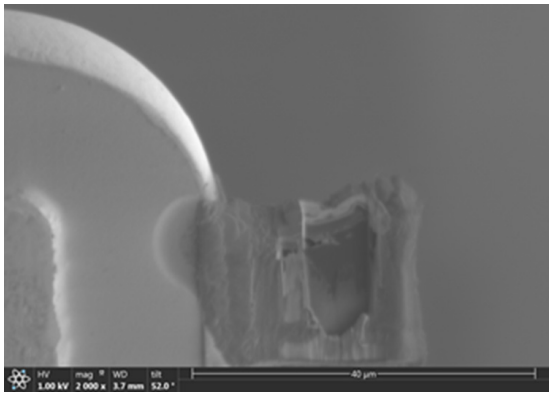

Figure 4. TEM lamella

Complementary Techniques

Cryo-TEM is also offered at Eurofins EAG Laboratories and is a great complementary technique to Cryo-FIB. Cryo-TEM involves performing Transmission Electron Microscopy TEM analysis while keeping the sample at cryogenic temperatures. The use of the low temperature allows for:

- The study of thin, frozen slices of suspensions, allowing for morphology studies of particles in their dispersed state.
- The reduction of sample heating by the electron beam and thus the reduction of potential beam damage of sensitive materials.
- The study of low-temperature phases of crystalline materials.

Cryo-FIB at EAG

EAG is your trusted partner in advanced microscopy analysis. Our SEM, FIB and TEM expertise is unsurpassed with our scientists having many years of experience across a multitude of materials and industries We have a diverse range of microscopy tools and services to meet your needs, from process development to failure analysis. We work closely with our clients to tailor the analytical plan to their needs. Contact us today to learn how we can help.