How-To Guide

Advanced Ion Milling Systems

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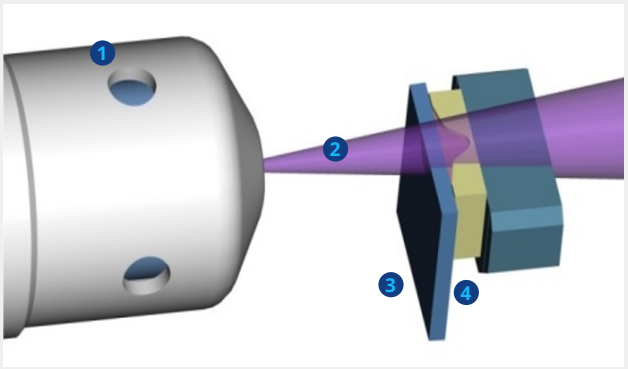
Introduction

Achieving precise SEM analysis requires advanced preparation methods like Broad Ion Beam Milling (BIB), which excels where traditional techniques fail, especially with composites and layered structures of varying hardness. Ion milling is a refined sample preparation technique widely utilized in research fields such as materials science to produce smooth, artifact-free surfaces essential for high-resolution imaging and analysis.

Broad Ion Beam Milling is an advanced sample preparation technique for Scanning Electron Microscopy (SEM), leveraging accelerated Argon ions to sputter material from a specimen surface. This process avoids mechanical force, thereby preserving the specimen's intrinsic structure and revealing its true nature for SEM analysis.

Two primary methodologies are employed: cross-section and flat milling:

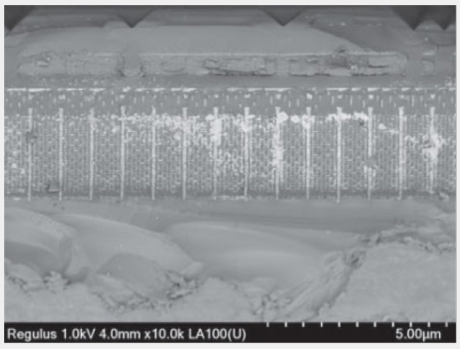
Cross Section Milling



1. Ion gun
2. Broad Ar+ Ion Beam
3. Beam shielding mask, can be positioned with ~15µm accuracy
4. Specimen

Cross-section milling allows for a more detailed analysis of sub-surface structures making it ideal for complex, multi-layered structures consisting of hard, soft, mixed, and heat-sensitive materials.

a)



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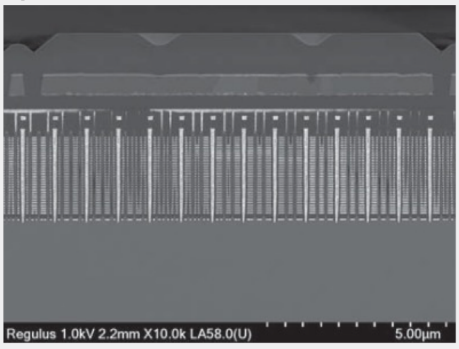
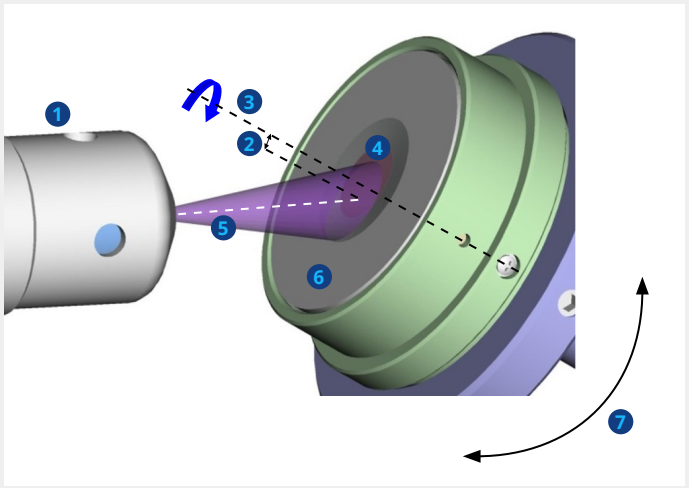


Figure 1. Example of cross-section milling of 2D NAND flash memory. a) Traditional mechanical method (ultrasonic cutting)**. b)** Treatment after ultrasonic cutting using ion milling.

Flat Milling



1. Ion gun
2. Excentricity
3. Rotation/swing axis
4. Irradiated area
5. Ion beam axis
6. Specimen
7. Beam incidence angle

Flat milling, typically following mechanical polishing, achieves defect-free surfaces by fine-tuning ion energy and angle.

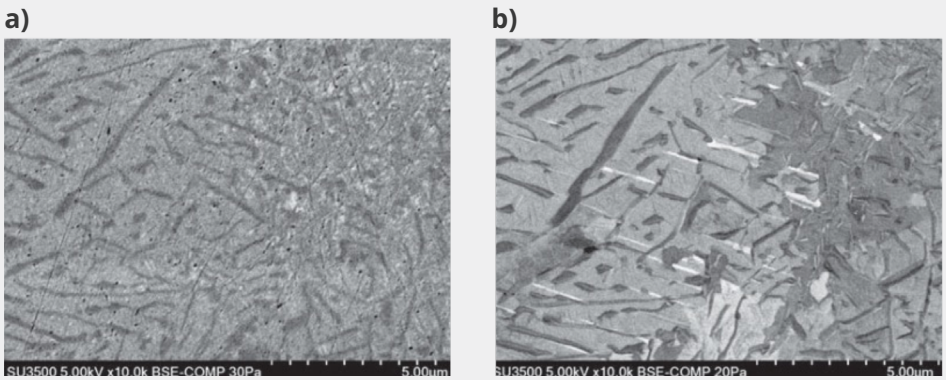


Figure 2. Example of flat milling of gold-silver-copper palladium alloy. **a)** After machine polishing. **b)** After machine polishing and flat milling.

Unlike conventional mechanical polishing, ion milling is adept at handling complex and delicate structures, such as multilayered and heterogeneous materials. BIB milling holds significant utility across diverse fields such as energy storage, semiconductors, and coatings. It enables critical investigations, including the analysis of layer thickness in lithium-ion batteries and the assessment of thin film properties in coatings.

Ion milling plays a crucial role in preparing specimens for advanced imaging modalities like SEM, Transmission Electron Microscopy (TEM), and Atomic Force Microscopy (AFM), where surface integrity is paramount. This guide will provide a comprehensive overview of utilizing the Hitachi IM4000II and ArBlade 5000 Ion Milling Systems for your research.

Meet the Systems

Hitachi ArBlade 5000
Known for its high-speed milling capabilities and superior surface finish, ideal for samples requiring minimal preparation time, this is the most advanced broad ion beam system for producing exceptionally high-quality cross-section or flat-milling samples for electron microscopy.

Hitachi IM4000II
A versatile system for both cross-section and flat milling, featuring an easy-to-use interface and customizable parameters for different materials. This system is optimized for high-resolution imaging applications, featuring automatic end-point detection and a compact user-friendly design.

What you can achieve with these systems

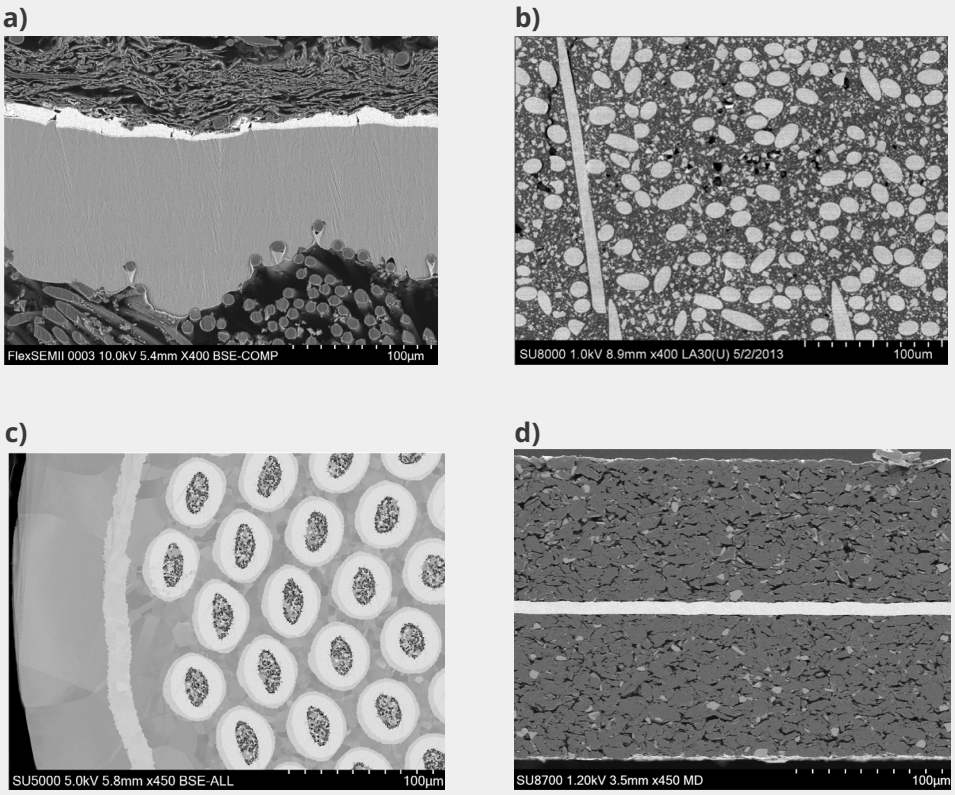


Figure 3. **a)** Polymer fuel cell with carbon fiber mesh enforcement using BIB cross-section milling, with clearly identifiable single fibers and the polymer phase in between as a smooth area with a different composition layer giving you a perfect overview of your polymer fuel cell. **b)** Cross-section milling of Glass-fiber-enhanced polymer with 40% glass fiber and 25% mineral filler demonstrating ease with a soft and hard matter mix. **c)** Non-flat sectioning of superconducting wire with extremely high quality allowing for clear visibility of detail. **d)** Cross section of a Li-Ion battery anode with graphite separated with a copper conductor, cut without active cooling showing the distribution of layers.

Step-by-Step Instructions

1 Preparing your Sample

Select Your Sample Holder: Choose the appropriate sample holder based on the size and type of your specimen. Ensure the sample is sized correctly:

- For the IM4000II holder up to 15mm in diameter.
- For the ArBlade 5000 holder up to 50mm in diameter.

Mount Your Sample: Securely mount the specimen onto the holder, ensuring it is stable and aligned properly. Adjust the tension of the clamp or apply additional adhesive if the sample shifts during mounting.

Ensure specimens are securely attached to specific mounts (e.g., M4 threaded Hitachi or 3.2 mm pin stubs) and avoid loose particles. Conductive carbon tape or paint is recommended for securing samples, especially fragile or non-conductive ones, and allow the paint to dry for 30 minutes to prevent issues.

Pre-Polish the Surface (if necessary): For optimal results, pre-polish the surface of the specimen mechanically to remove any large surface irregularities.

a) b) c) d)

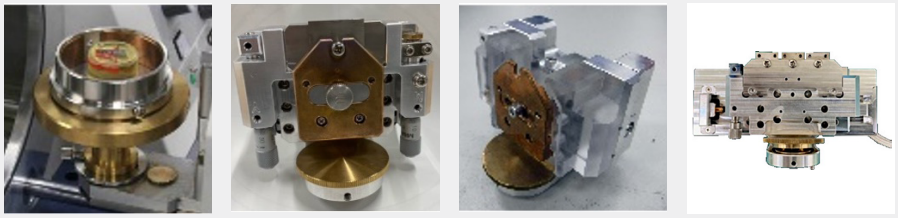


Figure 4. **a)** Flat Milling Holder. **b)** Large Sample Holder. **c)** Dedicated Cryo Holder. **d)** Multi Sample Holder (ArBlade 5000 only).

TROUBLESHOOTING STEPS

Problem: Shifting sample during mounting.

Solution: If the specimen shifts during mounting, check for debris on the holder and clean it with isopropyl alcohol. Ensure the clamps are functioning properly and adjust them to secure the specimen.

TROUBLESHOOTING STEPS

Problem: The sample is too large.

Solution: If the sample is too large, trim it with a precision cutter. Ensure the edges are smooth to prevent uneven milling.

Problem: Uneven surface.

Solution: Ensure you are applying consistent pressure during polishing. Consider using a polishing machine for uniform results.

2 System Setup

Power up: Turn on the Ion Milling system and allow the initialization sequence to complete.

Select Milling Mode: Access the control panel and choose between flat milling or cross-section milling, depending on your analytical needs. Milling conditions can be easily applied via the LCD panel.

Adjust System Parameters: Input the milling parameters dependent on the material properties of your specimen, including beam energy, duration, and angle.

Set the ion beam energy, typically between 1 keV and 6 keV, depending on the material's hardness. ***For example:*** Use a lower energy of 2 keV for delicate polymer samples to minimize damage and adjust the ion beam angle to optimize material removal. A 90-degree angle is often used for flat milling, while 45 degrees might be used for cross-sectioning.

If Applicable: Cooling Temperature Control: If milling heat-sensitive materials, attach the dedicated cryo holder and set the desired cooling temperature using the control panel. The cooling temperature control uses indirect cooling and maintains a temperature during processing to prevent phase transitions. Set the temperature control to prevent thermal damage and ensure the integrity of the specimen.

a) b) c)

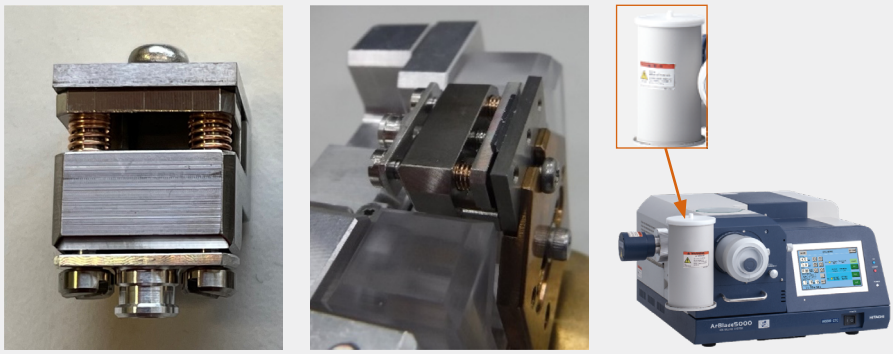


Figure 5. Active specimen cooling by LN_2 : Cooling Temperature Control System. a) The sample is spring-clamped directly against the shielding mask for constant thermal contact. **b)** Loaded into the Dedicated Cryo Holder (see Fig. 2c). **c)** LN_2 Reservoir attached to the Ar Blade 5000 unit.

a) b)

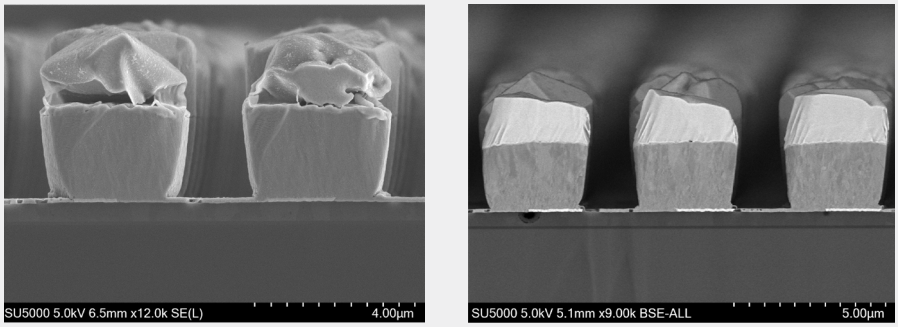


Figure 6. Example of Cryo-application: Indium bumps on Nickel. a) No cooling with accelerating voltage 6 kV. **b)** Cryo cooling at -95°C with accelerating coltage 4.5 kV.

TROUBLESHOOTING STEPS

Problem: The system fails to start or is unresponsive.

Solution: If the system fails to start, check the power cable connections and ensure the emergency stop button is not engaged. If the mode selection is unresponsive, restart the system and ensure all cables are securely connected. Check for software updates that may resolve interface issues. Refer to the user manual for error codes displayed on the screen.

Problem: The system does not recognize input parameters.

Solution: If the system does not accept the parameters, verify that they are within the allowable range. Restart the software if necessary.

TROUBLESHOOTING STEPS

Problem: The holder does not fit correctly in the system.

Solution: If the holder is not fitting correctly, check for obstructions or misalignment. Clean the holder slot and ensure proper alignment and insertion of the holder. Ensure the correct holder type is selected in the software.

Problem: Temperature not stabilizing.

Solution: If the temperature does not stabilize, check for leaks in the cooling system and ensure the liquid nitrogen supply is sufficient. Inspect the temperature sensor for proper placement and functionality.

3 Performing the Ion Milling

Initiate the Milling Process: Start the milling process via the control interface, monitoring progress on the display. Check for error messages if the process does not initiate and ensure all connections are secure. If applicable, use the stereo microscope to monitor the specimen's surface in real time (Fig. 7).

Monitor Milling Progress: Continuously observe the milling through the system's display, ensuring there are no artifacts or irregularities, and adjusting beam parameters as needed to ensure uniform material removal. If uneven milling occurs, verify that the sample is level and adjust the beam angle or energy as necessary. Consider pausing the process to inspect the specimen for any irregularities.

Figure 7. Stereo Microscope Unit for Process Observation: can be outfitted to the unit to observe the specimen during the milling process, with both a binocular and trinocular available. The trinocular microscope allows for viewing on an external monitor when equipped with a CCD camera.

TROUBLESHOOTING STEPS

Problem: Vacuum system failure.

Solution: If the vacuum system fails to reach the desired pressure, inspect the chamber seals for leaks and ensure the vacuum pump is operational. Clean or replace filters if necessary.

Problem: Uneven milling.

Solution: Verify that the sample is level and adjust the beam angle or energy as necessary. Consider pausing the process to inspect the specimen for any irregularities.

Problem: The sample shows signs of overheating.

Solution: Pause the process, reduce beam energy, or increase cooling intervals to prevent thermal damage.

Problem: Failure to start.

Solution: Verify that the vacuum chamber is sealed and at the correct pressure. Ensure the ion source is properly calibrated and free from obstructions. Check for error messages and ensure all connections are secure.

4 Post-Milling Analysis

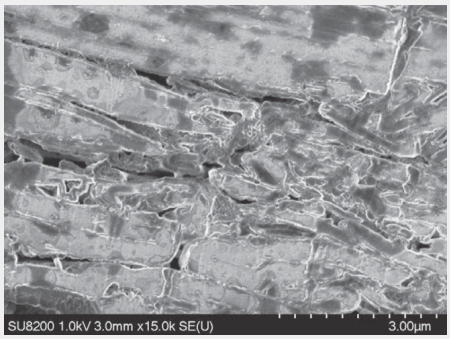
Inspect the Milled Sample: Once the desired surface finish or cross-section is achieved, deactivate the ion beam and allow the system to return to atmospheric pressure. Check for uniformity across the sample surface. Ensure the milling has produced a consistent finish without uneven areas, which could distort SEM results. Use an optical microscope to perform a more detailed examination of the surface. This step helps identify any micro-level imperfections or residual debris that might not be visible to the naked eye.

NOTE: The Air Protection Holder Unit can be utilized to isolate air-sensitive samples from the environment.

- **Transfer Sample to SEM:** Ensure the SEM chamber is clean and ready for sample insertion. Verify that the chamber environment is appropriate for the sample type, considering factors like vacuum level and temperature. Carefully remove the sample and transfer it to an SEM for detailed analysis. Carry the sample carefully, avoiding sudden movements or vibrations that could dislodge it from the stub.
- **Analyze Results:** Evaluate the integrity of the cross-section or surface finish, ensuring clarity and absence of milling artifacts. A well-prepared sample should exhibit minimal surface roughness, which is crucial for obtaining clear and detailed SEM images. Check for the absence of preparation-induced artifacts, such as scratches, smearing, or deformation. These can obscure features and lead to misinterpretation of SEM data.

a)

b)



c)

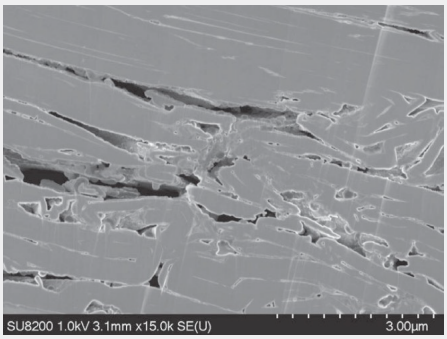


Figure 8. a) The Air Protection Holder, used to keep air-sensitive specimens isolated from the environment allowing a specimen to be loaded into an SEM without environmental exposure. Can be used with the Cooling Temperature Control (The ArBlade 5000 allows for Cross-section milling with the Air Protection Holder). **b)** Lithium-ion battery negative electrode after charge without utilization of the Air Protection Holder. **c)** Lithium-ion battery negative electrode after charge with use of the Air Protection Holder.

TROUBLESHOOTING STEPS

Problem: The sample surface is rough or shows artifacts.

Solution: Verify the cleanliness of the specimen surface and ensure proper SEM calibration. Consider re-milling with lower beam energy or a different milling angle to achieve a smoother finish.

Problem: Failure to vent chamber.

Solution: Check the vent valve for obstructions and ensure the venting system is functioning. If issues persist, consult the user manual for troubleshooting venting issues.

Problem: Damaged sample during transfer.

Solution: Review handling procedures and use tools designed for delicate specimens. Consider using a vacuum tweezer for improved control.

5 Cleaning and Maintenance

Take Safety Precautions: Ensure the system is completely powered down and unplugged before beginning any cleaning or maintenance activities. Wear appropriate personal protective equipment such as gloves and safety goggles to protect against exposure to chemicals and debris, and ensure the area is well-ventilated, especially when using solvents or cleaning agents.

Clean the Chamber: Clean the milling chamber and holders using appropriate tools and solvents to remove debris. Use a lint-free cloth dampened with a suitable solvent (e.g., isopropyl alcohol) to wipe down the interior surfaces of the chamber. Avoid soaking the cloth to prevent excess liquid from entering sensitive areas. Check for any residue or stubborn deposits and gently scrub them with a soft brush or cloth. Repeat the solvent application if necessary.

- **Clean the Holders and Maintain Components:** Carefully remove holders and other detachable components from the system. Place holders and components in a container with a compatible solvent to loosen any adhered materials. Use a soft brush to gently scrub the components, focusing on areas with visible residue or buildup. Rinse the components with clean solvent and allow them to air dry completely or use a lint-free cloth to speed up the drying process.
- **System Inspection:** Check all cables, and connections and inspect seals and gaskets for signs of wear or damage. Apply appropriate lubricants to moving parts as specified in the system manual to ensure smooth operation. Check for any available software or firmware updates for the system and install them to ensure optimal performance and security.

TROUBLESHOOTING STEPS

- **Problem:** Debris is difficult to remove from the chamber.
- **Solution:** Use a vacuum cleaner with a fine nozzle to reach tight spaces and a soft brush to dislodge stubborn particles. If applying a solvent, ensure the cloth is only lightly dampened, and use a high-purity solvent like isopropyl alcohol to minimize residue. Follow with a dry cloth to polish the surface.

- **Problem:** Components are delicate and risk damage during cleaning.
- **Solution:** Handle the components with care, using soft brushes and gentle cleaning techniques. Avoid using excessive force during scrubbing. Test the solvent on a small, inconspicuous area of the component first, or consult the manufacturer for recommended cleaning agents.

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