Artifact-free cross-sections

Argon ion beam specimen preparation quickly and easily produces cross sections of a wide variety of materials with minimal artifacts.

N. Erdman and R. Campbell
Jeol USA Inc.
Peabody, Massachusetts
S. Asahina
Jeol Ltd., Japan

The Cross Section Polisher (CP) is a new cross-section sample preparation device that addresses some of the issues involved with preparing very small and relatively soft specimens for SEM analysis. The CP can easily prepare a cross section that is hundreds of micrometers in width and can preserve nanometer-level fine structures.

CP prepares a cross section in the following way: a high-energy argon ion beam irradiates the surface of the specimen masked by a shielding plate, from the direction perpendicular to the surface. Thus, the region that is not masked by the shielding plate is etched. This device provides two advantages:

- It provides a clean cross section that has less distortion or altered layers due to milling than those prepared by conventional polishing methods.
- It prepares a wider-area cross section as compared to FIB.

Utilizing these advantages, CP becomes a powerful cross-section preparation tool for SEM, EPMA, and AES.

The instrument consists of a specimen chamber with a turbo pump vacuum system, an optical microscope for specimen positioning, and controls for the vacuum system and the ion beam. The specimen stage in the chamber features a holder and a masking plate. To cut a cross section, the specimen is placed in the holder, and the region of the sample to be cross sectioned is selected under the optical microscope.

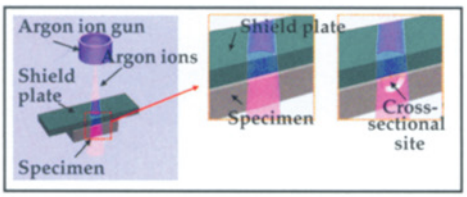

Fig. 1 - In the specimen chamber of the Cross Section Polisher, the selected region is irradiated with a broad argon ion beam.

The masking plate is then placed across the selected region. After evacuating the specimen chamber, the region is irradiated with a broad argon ion beam that has a selectable accelerating voltage range of 2 to 6 kV (Fig. 1).

During milling, the specimen stage can be automatically rocked ± 30 degrees to prevent beam striations and ensure uniform etching of composite materials with different hardnesses, preventing the soft portions from being cut faster than the hard portions. As it is not a mechanical polishing method, abrasives are never embedded in the polished surface, and samples that are sensitive to heat can be prepared with minimal thermal damage. The instrument is set up on a timer, allowing unattended operation during the polishing process.

Device advantages

Advantages of the CP over other preparation techniques include:

- Minimal sample pre-preparation prior to sectioning with the ion beam
- High quality cross sections of composites containing both soft and hard materials
- Wide selection of operating conditions to allow minimum heating of the sample (particularly important for soft materials)
- Minimum strain and distortion of the polished surface
- Large cross section areas are produced compared to FIB methods (a single cut is typically 1.5 mm wide and several hundreds of microns deep)
- No particle embedding in the polished surface
- Ease of operation

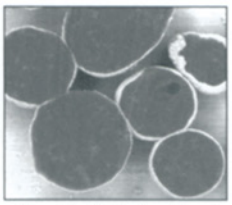

Fig. 2 - This is a back-scattered image of toner particles cross sectioned by the CP.

Application examples

Toner: Figure 2 is a back-scattered image of toner particles cross sectioned by the CP. Toner particles are extremely difficult to prepare via mechanical sample preparation methods, because the particles are very soft and deform during polishing. Sample embedding for mechanical polishing or microtome cutting is problematic due to a likely reaction between the toner particles and the epoxy. The CP preparation allows toner particles to be prepared without any embedding, resulting in clear observation of internal particle structure.

Paper: Preparation of a paper cross section is typically done by cutting the sample with a razor blade or by means of an embedding/staining/mechanical polishing procedure. Both methods can introduce a substantial number of artifacts into the sample. The CP method preserves the integrity of the sample and provides a unique inside view of coated papers, including observation of pigment and carbonate particles (Fig. 3a, b), allowing analysis of the absorbency and ink penetration critical to the paper manufacturing process. The information helps in quality control and optimization of the printing characteristics of the paper.

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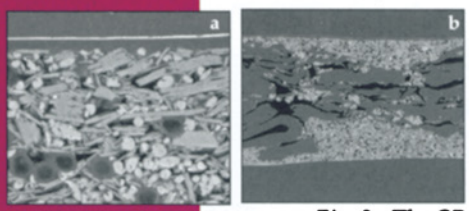


Fig. 3 - The CP method preserves the integrity of the paper sample and provides a unique inside view of coated papers.

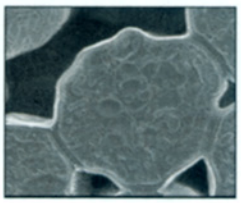
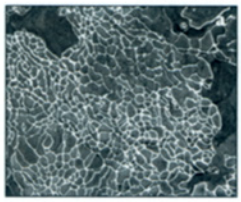


Fig. 4 - This image shows a yeast cell sectioned with the CP with clearly observed vacuoles.



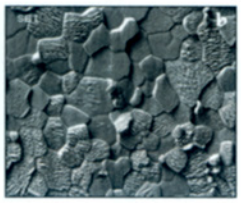


Fig. 5 - The choice of low kV (a) allows mercury to be preserved in the grain boundaries of the amalgam specimen (bright white lines). (b) shows what happens if the operator chooses high kV setting on the same sample: the grain structure is preserved, but the mercury is no longer located in the grain boundaries.

Biomaterials and polymers: Biological materials and polymers can best be mechanically sectioned at cryogenic temperatures. Such samples often present challenges to conventional preparation methods such as chemical fixing, critical point drying, and impression techniques (replica). With the CP method, preparation of a yeast specimen is very similar to preparation of toner samples without an embedding media. Before imaging in the SEM, the sample was etched by an argon ion beam (0.3 kV) for 30 seconds using XPS to enhance the internal structure of the cells. Fig. 4 shows a yeast cell sectioned with the CP with clearly observed vacuoles.

Amalgam: Amalgam samples (Hg-Zn-Sn mixtures) are extremely difficult to prepare mechanically due to possible smearing of mercury. CP allows preparation of amalgam specimens at low kV settings while maintaining structural integrity. Figure 5a shows that the choice of low kV allows mercury to be preserved in the grain boundaries of the specimen (bright white lines). Figure 5b shows what happens if the operator chooses high kV setting on the same sample: the grain structure is preserved, but the mercury is no longer located in the grain boundaries.

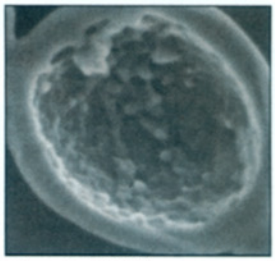


Fig. 6 - This image shows a close-up of a single hollow latex sphere.

Polystyrene latex spheres: PS latex spheres are found in various applications in materials science and biology, including as a template of new structures, calibration standards for electron microscopy, and support materials for catalysis. Polystyrene also represents a class of polymers that are relatively difficult to cross section. The small size of the spheres would require an ion-beam-related sample preparation; however, the ion beam has the potential to cause significant thermal damage to the polymer. The CP enables the operator to make latex-sphere cross-sections in less than ten minutes, significantly minimizing the thermal effects of the beam on the sample. Figure 6 shows a close-up of a single hollow sphere. The CP clearly reveals the internal structure of the latex sphere without any artifacts. MPMD

For more information: Natasha Erdman, Jeol USA Inc., 11 Dearborn Road, Peabody, MA 01960; tel: 978/536-2273; erdman@jeol.com; www.jeol.com.