Precise SEM Cross Section Polishing via Argon Beam Milling

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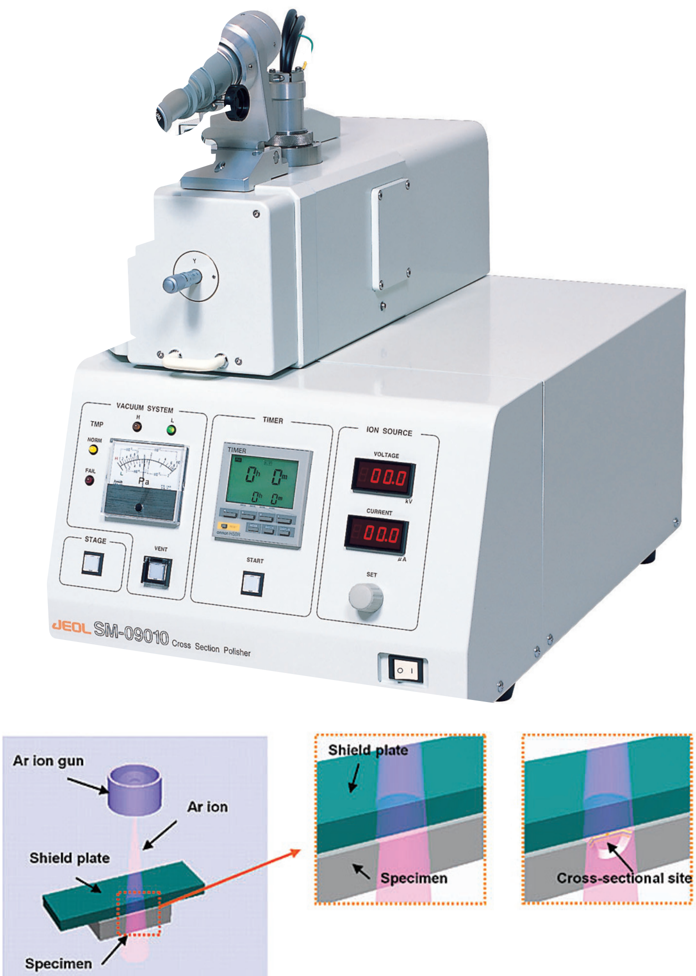
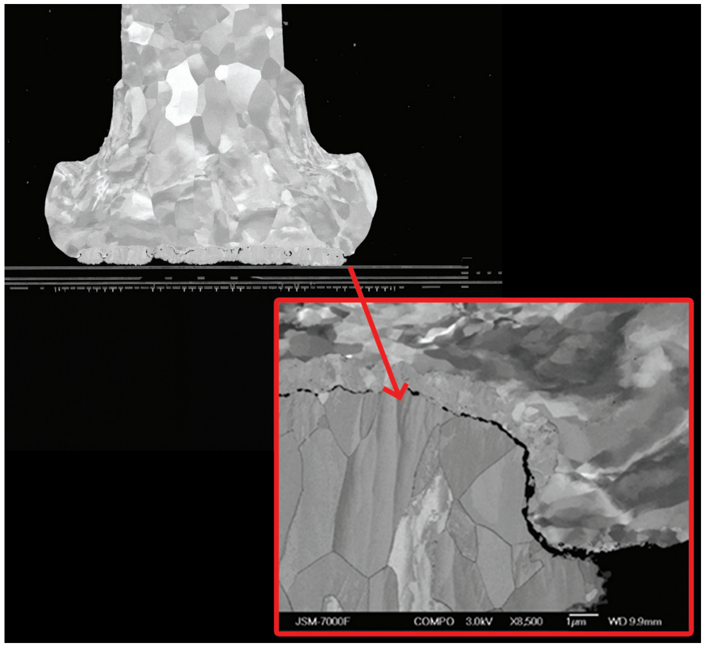
SEM observation of a specimen cross section can provide important information for research and development as well as failure analysis. In most cases, surface observation alone cannot provide information concerning the cross sectional structure of granular materials, layered materials, fibrous materials, and powders. Preparing highly-polished cross sections of these materials is both a science and an art.

Typically, a cross section is prepared using mechanical means like conventional mechanical polishing methods or a microtome. The sample is first embedded in a holder or device, and then polished to achieve a flat cross section. In some cases, a staining procedure is used to highlight a specific component of the sample. Such methods can be lengthy procedures that require a great deal of skill, and can introduce artifacts into soft materials, deform the material around voids, or compress layers of soft and hard materials in composite samples. Mechanical polishing can miss fine details such as the presence of hairline cracks, and present a challenge to water-soluble phases.



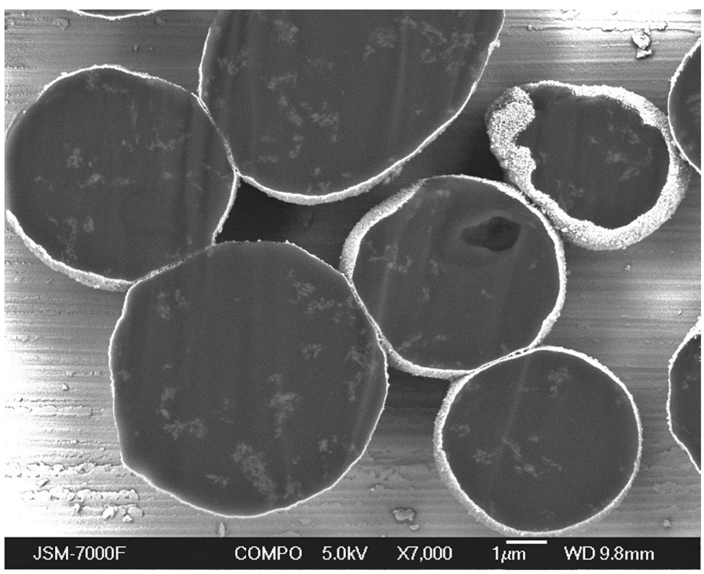
1. (a) JEOL Cross section polisher; (b) Schematic of general instrument operation.



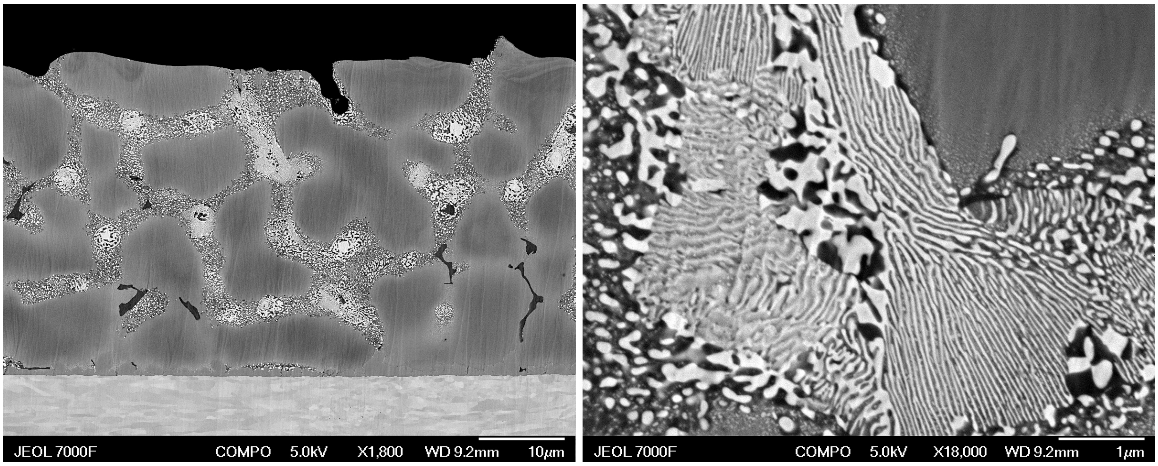
2. Gold wirebond cross section prepared using the CP. The inset shows magnified view of grain structure and voids.

In other cases, a Focused Ion Beam (FIB) System is used when precise positioning of the cross section is required, such as in the case of stacks of thin films or micro area specimen preparation. However, the size of the resulting cross section is limited, and the heavy gallium ions in the beam can damage the sample surface.

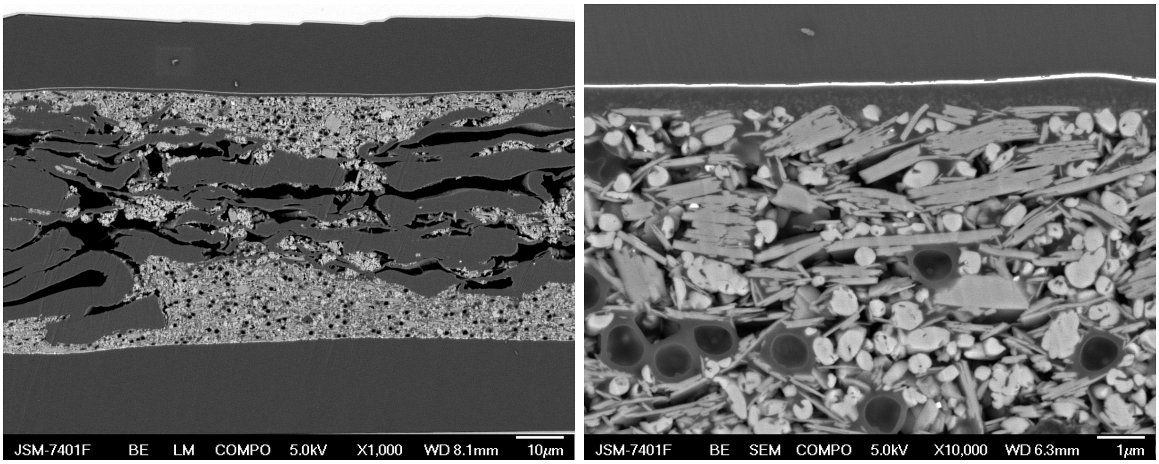
A new precision argon ion beam cross section polisher simplifies the preparation of samples and makes it possible to prepare truly representative cross sections of samples free of artifacts and distortion. Use of the broad argon ion beam eliminates the problems associated with conventional polishing and allows for larger specimens to be prepared with precision. (1, 2)



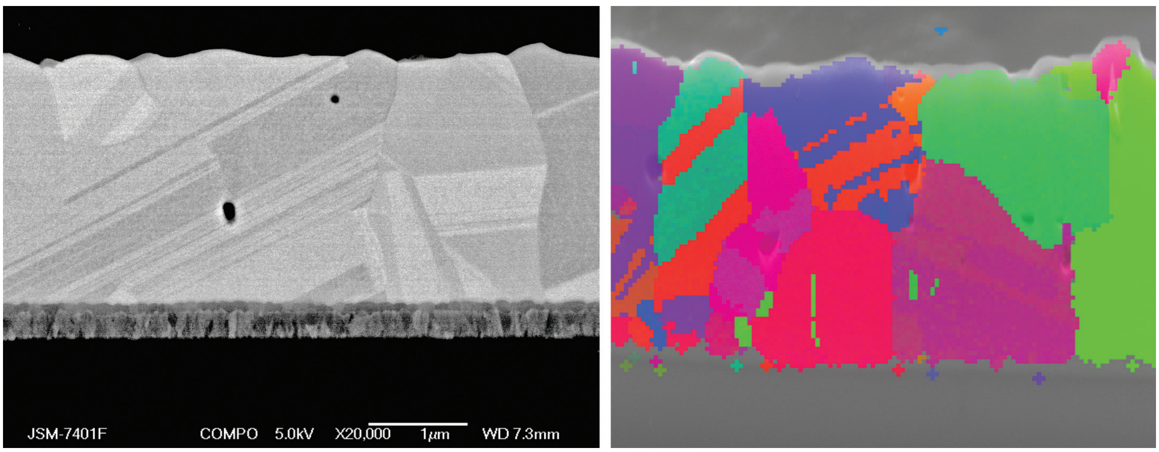
3. Toner particles cross section prepared using the CP.



4. *(a) Back-scattered image of galvanized coating sample sectioned with the CP; (b) Close up of the eutectic phase.*



5. *(a) Cross section of a paper sample, (b) shows the structure of the paper coating.*



6. *(a) Back-scattered image of a thin metal coating on glass; (b) EBSD map of the sample.*

The JEOL Cross Section Polisher (CP) consists of a specimen chamber with a turbo pump vacuum system, an optical microscope for specimen positioning, and controls for the vacuum system and stationary ion beam (Fig. 1). The specimen stage in the chamber features a holder and masking plate.

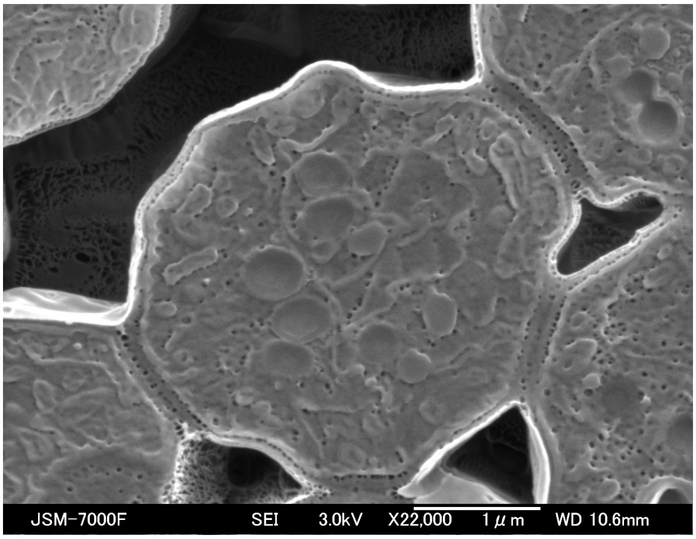
To produce a cross section using the CP, the specimen is placed in the holder, and the region of the sample to be cross sectioned is selected under the optical microscope. The masking plate is placed across the selected region. After evacuating the specimen chamber, the region is irradiated with a broad argon ion beam with a selectable accelerating voltage range of 2 to 6kV.

During milling, the specimen stage can be automatically rocked ± 30° to prevent beam striations and insure 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 without distortion.

The instrument is set up on a timer, allowing unattended operation during the polishing process.

Advantages of the CP over other preparation techniques include:

- High quality cross sections of composites of soft and hard materials
- Minimum strain and distortion of the polished surface, enabling one to observe grain contrast (channeling contrast) clearly and easily
- Large cross section areas are obtained 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 as compared to mechanical polishing
- Ease of operation



7. Image of yeast cell cross section.

Applications

Gold wire bond. Fig. 2 is a backscattered electron image of a cross section of gold wire bonding on a silicon IC. Despite differences in hardness between all the materials in the sample, including silicon, aluminum and gold, the cross section prepared with the CP clearly reveals narrow cracks and small voids in the bonding layers. The ability to examine such features is typically crucial for failure analysis and quality control. Channeling contrast in the gold can also be observed clearly.

Toner. Fig. 3 is a back-scattered image of toner particles cross sectioned using the CP. Toner particles are extremely difficult to prepare using 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.

Crystal structure of galvanized coating. Fig. 4a shows an overview of a specimen of galvanized coating on steel prepared using the CP. The processing conditions typically affect the adhesion properties of such a coating, therefore it is crucial to preserve the integrity of the interface layer. The CP preparation provides a unique insight into coating technology while preserving the structural integrity of the metal interface. Fig. 4b shows the Zn-Al-Si eutectic in the coating.

Paper. Preparation of a paper cross section is typically done by cutting the sample with a razor blade or using 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. 5a, 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.

Thin film analysis using EBSD. Cross-sectional observation is critical for thin film manufacturing for determination of adhesion properties of thin film; however, typical mechanical sample preparation often smear the interface layers and makes such observations somewhat ambiguous. In Fig. 6a and b we show a backscattered image and corresponding EBSD pattern from metal thin film on glass. The image clearly shows differences in the layer structure of the film, the CP preparation accentuates the channeling contrast from the different grains. The CP preparation facilitates easy and straightforward EBSD pattern acquisition due to minimal surface strain and distortion.

Yeast. The use of cryo-temperatures is the preferred method for obtaining information from biological materials and polymers. Such samples often present challenges to conventional preparation methods for biological materials such as chemical fixing, critical point drying and impression techniques (replica). Using the CP method, preparation of a yeast specimen is very similar to preparation of toner samples without using an embedding media. Fig. 7 shows a yeast cell sectioned with the CP with clearly observed vacuoles.

In conclusion, the cross section specimen preparation method using an argon ion beam enables one to easily create cross sections of a wide variety of materials including polymers, metals, ceramics, and composites with minimal artifacts. It is suitable for not only image observation but also for microanalysis and determination of crystal structure. ■

References

(1) M. Shibata, S. Asahina, T. Negishi, *Proc. 8 APEM*, (Kanazawa, 2004) p. 258.

(2) W. Hauffe, *Electron Microscopy*, Vol. 2, EUREM 92, Granada, Spain, 1992.