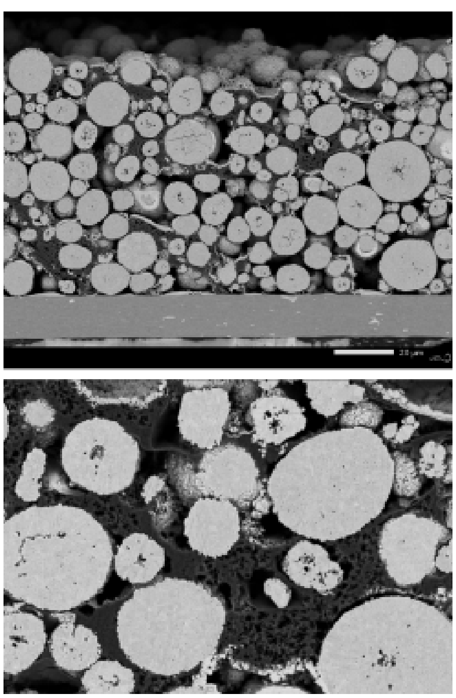
Handle with care – preparing sensitive samples for imaging cross sections in the electron microscope

Scientists use the ultrahigh resolution and magnification range of electron microscopy to observe nanoscale details of both surfaces and internal structures of a wide range of materials. When preparing a cross section of a sample to reveal the internal structure, they need to consider the properties of the samples and be aware of their potential to crumble or melt or change due to exposure to air. Some samples are composites with layers of different materials, both hard and soft, and they can also range in sensitivity to temperature and exposure to air.

Creating a pristine cross section to see internal structuring for such samples is not as simple as cutting with a sharp razor blade, polishing with sandpaper, or using a microtome. None of these options work without smearing or crumbling of coatings and complex internal layers, and also adding artifacts. For this purpose, JEOL developed an ion beam milling system that could create pristine samples of coated papers, powders, and metals, plus a cooled ion beam milling system with special handling for air-sensitive samples like lithium ion batteries.

Here we look at three types of samples that require a more precise cross sectioning technique than traditional methods: Lithium Ion battery, pharmaceutical tablet, and Zn thin film. For each, scientists need to examine a very thin multilayered “sandwich” of different materials.

Temperature and Air-Sensitive Lithium Ion Batteries

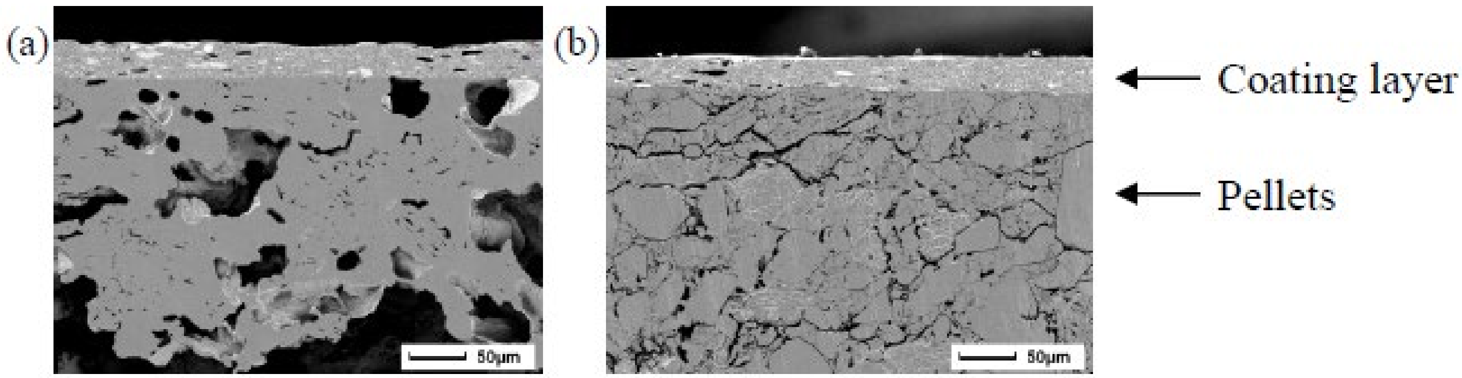


Backscatter SEM image of Lithium Ion Battery Cross Section prepared with JEOL Cross Section Polisher

The basic structure of Lithium Ion batteries (LIB) contains as many as 10 different thin films and at least that many solid−solid interfaces. These interfaces consist of thin layers of cathode material, insulating barriers, anode materials, metal current collectors, and the electrolyte. These various components are in the form of powders, sheets, and fluids and require assessment before and after assembly and after repeated charge/discharge operations. Researchers who are correlating electrochemical behavior to what is physically happening within the cell need to study the 3D microstructure of the battery components as well as the interfaces formed between those layers.

For Lithium ion battery materials that potentially react and degrade upon exposure to air, it is indispensable to have techniques to prevent the exposure of the specimen to the atmosphere during sample preparation, and even in introduction to the electron microscope. For that purpose, JEOL has established a designated workflow that includes a common air-isolated transfer vessel that is used to transfer a specimen that has been prepared in an inert gas environment (such as in a glove box) to the designated specimen preparation equipment (broad ion beam polishing equipment, Cryo Cross-section Polisher), and subsequently into the SEM through a specimen exchange chamber without exposing the specimen to the atmosphere.

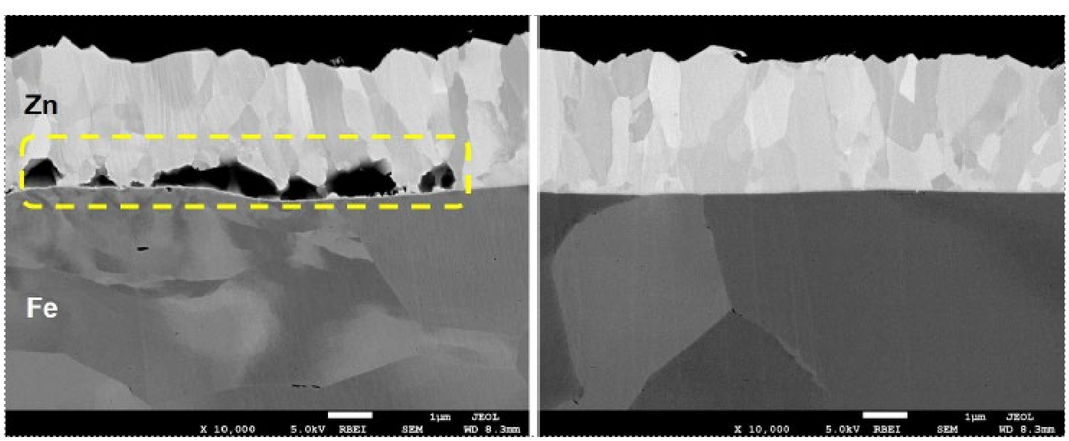
Complex Layers in Pharmaceutical Tablets



Cross section processed (a) at room temperature and (b) -120^{o}C.

To help ensure the proper functions of pharmaceutical tablets, such as sustained or enteric release that influence the effect of the medicine on the body, it may be necessary to observe the tablet's structure and components. Cross sections of tablets are generally very difficult to prepare due to the fragility and softness of the materials. Preparation damage might cause sample deformation such as peel-off of the coating layers after razor blade cutting or mechanical polishing. Using broad argon ion beam milling instead creates high quality cross sections. Since some tablets contain substances with low-melting point such as starch and beeswax, thermal damage in the process might cause sample deformation. In this case utilizing [liquid nitrogen (LN2) cooling during the ion milling is preferred].

Structural Details and Crystallographic Orientation of Thin Films



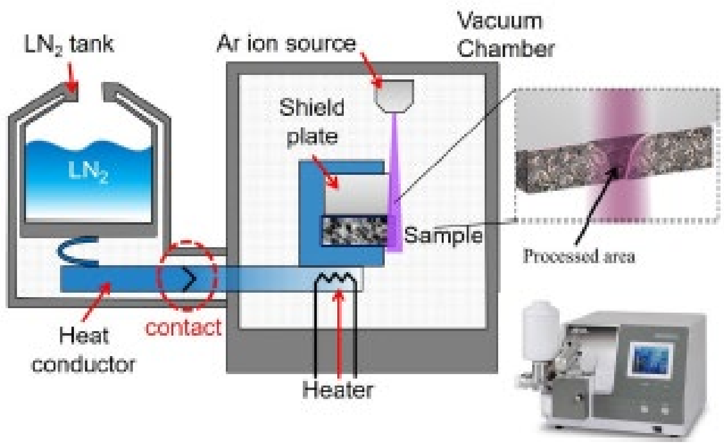
Zn coating on steel prepared at RT (left) and using cooling (right). The image shows preserved thin-film/substrate and crystallographic integrity.

Examination of materials cross sections often provides essential information about the crystal structure, layer or film thicknesses, existence of voids or cracks and other properties that might impact materials performance and reliability. Cross-sectional observation is especially essential in thin film technology to examine layer thickness, deposition integrity (voids/adhesion), as well as film growth and crystallographic orientation. Currently various methods are used to prepare specimen cross sections for scanning electron microscope (SEM) observation. Mechanical methods of cutting and polishing are widely used, particularly for metallographic sample preparation. However, mechanical polishing presents several problems: a) in composite materials with different hardness values, the polished surface becomes uneven as the softer components are cut faster and more easily than the harder components; b) in soft materials, particles of hard abrasive can be buried in the material being polished; c) in materials with voids, the edges of the voids can stretch and deform; e) for metals, due to the strain caused by mechanical polishing on the polished surface, the information about the crystal structure by means of electron back-scatter diffraction (EBSD) becomes difficult or impossible to obtain; f) fine features like hairline cracks and small voids can get smeared shut and will not be recognized as such.

Conclusion

Only broad ion beam milling with consideration for sample properties can produce clean cross sections for electron microscopy. The pristine samples provide microstructural information, and, when analyzed with SEM-EDS, produce data with high spatial resolution with phase and chemical mapping.

Ion milling is one of the only reliable techniques to get a clear sense of different layers as well as interfaces between layers – all without artifacts.



Schematic of cooling broad ion beam polisher (JEOL IB-19520CCP Cross Section Polisher)