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Advances in Beam-sensitive Sample Preparation and Observation

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This paper describes developments in instrumentation that allow successful beam-sensitive material sample preparation and its controlled atmosphere transfer to the scanning electron microscope (SEM); we also describe the influence of diverse SEM observation parameters when performing beam-sensitive material microstructural characterizations.

To complete accurate and informative SEM analytical studies, the samples must be well-prepared, represent the native microstructure of the study material, and be without oxidation, contamination, or damage induced by preparation. An artifact-free sample preparation technique is critical to better understand and correlate the synthesis, chemistry, and properties of the material. Preparation of beam-sensitive materials, such as lithium or Li-ion batteries, is particularly difficult because these materials are highly sensitive to oxygen, nitrogen, water, and carbon dioxide. They are either a soft material (Li metal) or made from compacted powders (different Li-ion based elements, such as sulfide solid-state electrolyte or Ni-Co-Mn cathode) – both types of materials are easily damaged by mechanical preparation. The material characteristics make sample preparation under ambient atmosphere impossible. However, ion milling-based techniques under controlled environment make Li metal/Li-ion battery sample preparation possible.

Because lithium and some solid-state Li-ion batteries are extremely beam-sensitive, they require cryogenic conditions during ion milling and transfer from ion mill to SEM under protective atmosphere. Figure 1a presents a Li metal sample prepared using broad argon ion beam (BIB) milling [Model 1062 TrionMill, Fischione Instruments] without cryogenic conditions and transferred to the SEM in Ar atmosphere; severe ion beam-induced damage is observed. Figure 2b shows a lithium sample after BIB milling under cryogenic conditions, but transferred to the SEM under ambient atmosphere. No beam-induced damage is evident, but strong surface oxidization is observed. In both cases, no Li microstructure was revealed.

In this work we report on instrument developments that allow the direct transfer of the sample from an ion milling system to SEM without use of a glove box. The sample preparation system's transfer mechanism allows transfer at vacuum, in inert gas, or in a cryogenic environment. All the transfer options and their influence on SEM analytical study are discussed.

Figure 1c shows SEM image of Li metal surface after cryogenic BIB milling and transfer in Ar protective atmosphere to the SEM. Once the sample is transferred to the SEM, observation can be done at ambient or cryogenic conditions. Therefore, different scenarios are considered:

- cryogenic preparation, cryogenic transfer, and cryogenic SEM observation
- cryogenic preparation, ambient temperature transfer, cryogenic SEM observation
- cryogenic preparation, ambient temperature transfer and SEM observation

The SEM observations are done by controlling and reducing electron beam sample interaction, depending on analytical technique that is being used. Our investigations will be supported by numerous SEM images using different detectors, and analytical studies using energy X-ray dispersive spectroscopy and electron backscatter diffraction techniques.

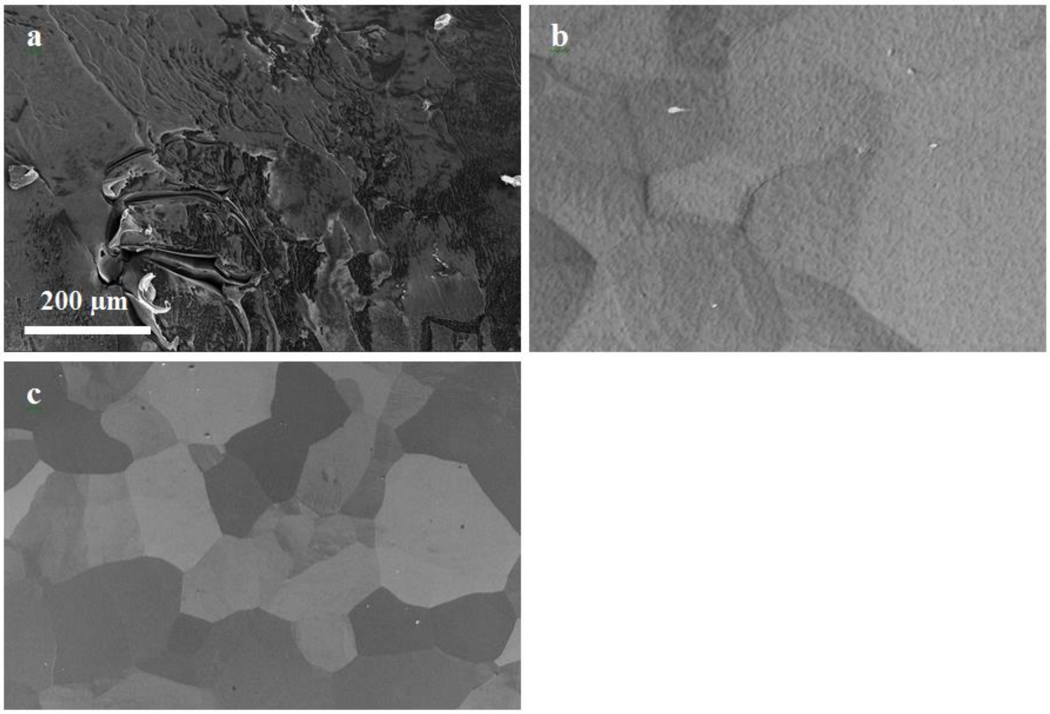

Figure 1. Li metal sample prepared using broad argon ion beam milling technique. A sample ion milled in ambient temperature and transferred to the SEM in Ar atmosphere (a). A sample milled in cryogenic conditions and transferred to the SEM in ambient atmosphere (b). A Li metal surface after cryogenic BIB milling and transfer in Ar atmosphere to the SEM (c).

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