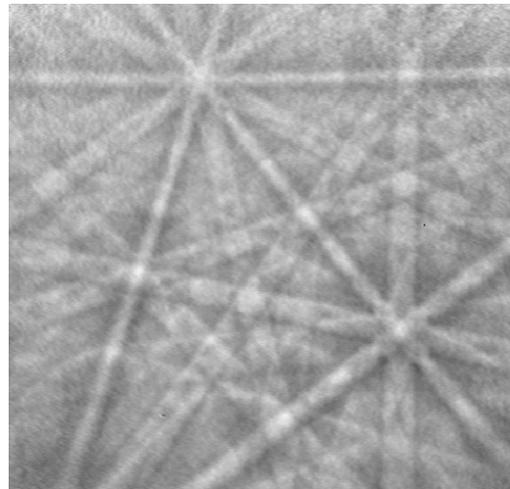


Specimen Preparation for Electron Backscatter Diffraction (EBSD) Analysis

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I. Introduction to Specimen Preparation

The first step in collecting OIM data is preparing the specimen so that high quality electron backscatter diffraction (EBSD) patterns are observed and obtained with the imaging system. The figure here shows a high quality EBSD pattern obtained from a nickel specimen using a SIT camera system.



The near-incident energy backscattered electrons form the EBSD patterns. Backscattered electrons that have experienced inelastic events inside the bulk of the specimen do not contribute to the pattern. This fact, combined with the high tilt angle of the specimen (70 degrees), results in a diffraction region that forms within the top 10-50 nanometers of the specimen. The crystal lattice in this region should be strain-free and clean from contamination or oxide layers for diffraction to occur. If these conditions are not met the resulting EBSD patterns will be of poor quality or will not be visible at all. Proper specimen preparation will address these issues and help in obtaining reliable OIM data.

II. Specimen Preparation Techniques

A. Mechanical Polish

There are many types of samples that are examined with OIM, each with its own preparation technique. The majority of samples require some degree of mechanical polishing. There are six steps to a complete mechanical polish: Sectioning, Coarse Grinding, Mounting, Fine Grinding, Rough Polishing, and Final Polishing.

1. Sectioning

Sectioning is the removal of a representative area from a parent specimen. The final specimen must be small enough to allow for SEM mounting and examination. The limiting size will be SEM dependent.

There are two types of sectioning: high-speed abrasive and low speed, low deformation precision sectioning. Other methods introduce a very large deformation layer at the surface that is difficult and time-consuming to remove. Proper wheel speed and cooling procedure is necessary to preserve the microstructure of the original specimen.

2. Mounting

For a variety of reasons (ease of handling, edge retention) specimens are sometimes mounted in a solid medium for grinding and polishing. There are two types of mounting processes, hot and cold. For each method, there are many different possible mediums that may be used. EBSD examination does not place any specific performance requirements on mounting materials. However, it is often useful to use a conductive medium to help eliminate charging and drift problems. Hot mounting may also provide reasonably close to parallel top and bottom surfaces, which can make it easier for batch or extended scans across large samples.

3. Grinding

Grinding begins to remove the deformation layer introduced during sectioning, and produces a flat surface for examination. Typically, grinding starts with 240 grit SiC paper, either in disk or belt form, and continues through about 800 or 1200 grit SiC. Water is used as a lubricant to flush away removed material and keep fresh abrasive exposed. It is useful to use an optical microscope to examine the surface more closely.

4. Polishing

Polishing is used to remove any deformation introduced earlier during grinding. While the procedure is similar to grinding, polishing has many more variables to consider. Many types of abrasives, suspension mediums, and polishing cloths are available. The choice of consumables is a personal preference. At TSL, we

typically use a general-purpose cloth with alpha and gamma alumina abrasives, along with some diamond solutions for harder materials. Polishing usually consists of five steps, starting at about a 9.0 micron abrasive, and ending with a 0.05 micron abrasive.

5. Colloidal Silica

While alumina works well for a final polishing medium, the best for EBSD pattern quality is colloidal silica. This commercially available solution consists of negatively charged particles of silicon dioxide (SiO_2) with a pH value between 8 and 11. The solution polishes and slightly etches the specimen, removing most of the surface deformation layer. This solution works well with nearly all materials, with particular effectiveness on ceramic and geological samples that are otherwise difficult to prepare.

B. Electropolish

While mechanical polishing can produce good EBSD patterns, it is often necessary to use another step to improve pattern quality. Electropolishing is one method that may be used. Electropolishing is the removal of surface material by electrolytic action. This method removes any remnant deformation layers and surface irregularities. To electropolish, the specimen is made an anode in an electrolytic cell.

The anodic film controls the removal rate during electropolishing. During polishing, the anodic film thickness remains constant, and a planar surface should exist between the film and the electrolyte. Voltage and current should be adjusted to maintain the anodic film. If the electrolyte has free access to the specimen surface, etching will occur.

Unfortunately, there is no universal electrolyte that will work with all materials. The correct solution for a given material is necessary for polishing to occur. Additionally, electrolytes that are used to produce TEM thin foils can be used on bulk specimens. It is important to note that some solutions have a short shelf life.

Variables that affect the anodic film, and consequently the polishing rate are the specimen material, the electrolyte solution used, operating voltage, specimen size, solution temperature, the age of the solution, the flow rate of the solution, and the time of contact.

C. Chemical Etch

Chemical etching is an alternative to electropolishing that does not require as much setup time and experimentation for proper operating conditions. During a chemical etch, the specimen is immersed in or swabbed with a solution that selectively

dissolves the surface material. This technique is effective for removing any remaining surface deformation due to the higher surface energy of the deformed crystal lattice.

Like electropolishing, it is important to select the proper etchant for the best results. An etchant that leaves a film is not desirable. It must not create topographical differences on the surface that would lead to shadowing of the EBSD patterns. A good first choice for many materials is Nital (5% Nitric Acid, 95% Ethanol). Again, many of the solutions use hazardous chemicals.

D. Ion Etch

Ion etching is the process of using an ion beam to remove material from the specimen surface. This technique can be used on almost any material, including metals, semiconductors, ceramics, and geological samples. Ion etching has been used extensively for TEM specimen preparation, but it is relatively new for EBSD/OIM work.

The ion beam removes material at a rate determined by voltage, ion gun current, specimen material, and specimen/gun geometry. If the ion energy is too high, crystal lattice damage can occur, decreasing pattern quality. The best results for EBSD work are obtained using low voltage and low current for longer periods of time at high tilt angles (50-70 degrees).

Ion etching is also used to remove oxide and surface layers from a specimen. This is particularly useful in removing the oxide films that form on semiconductor devices. Ion etching can also be used for cross-sectioning specimen, and doing serial sectioning analysis.

III. Conductive Coating

Observing non-conductive specimens in the SEM is difficult due to charging effects. For OIM work, charging effects includes pattern degradation and beam drifting. The application of a thin, amorphous, conductive coating will eliminate charging effects.

For OIM work, carbon is used as the primary coating material. While it is possible to use other materials (gold, gold-palladium, tungsten), carbon works the best due to its low atomic number. Materials can be either sputtered or evaporated onto a specimen.

As the coating thickness increases, the patterns signal to noise ratio decreases. A carbon coating of approximately 25 angstroms is recommended. This thickness provides adequate conductivity while still keeping the signal to noise ratio high. If this ratio drops to the point that the patterns are difficult to index, it is often useful to increase the acceleration voltage on the SEM, which increases beam penetration through the coating.

Suggested Mechanical Polishing Sequence

- Section and mount the sample
- Grind until planar using 240 grit SiC paper
- 400 grit SiC paper between 100 and 150N Force for about 15-20 seconds
- 600 grit SiC paper between 100 and 150N Force for about 15-20 seconds
- 800 grit SiC paper between 100 and 150N Force for about 15-20 seconds
- 1200 grit SiC paper between 100 and 150N Force for about 15-20 seconds (this step may be repeated 2-3 times)
- 9 micron diamond solution 5-10 minutes
- 3 micron diamond solution 5-10 minutes
- 1 micron alpha alumina solution anywhere from 5-20 minutes
- 0.3 micron alpha alumina solution 5-15 minutes
- 0.05 micron colloidal silica solution anywhere from 1 minute to several hours