Sample preparation for EBSD

EBSD Image formation

- Typically EBSD patterns come from the top 10-50 nm of material
- EBSD pattern quality is strongly dependent on crystal lattice quality at the specimen surface
- This depth will vary with SEM acceleration voltage and specimen atomic number

Sample Preparation

- Mechanical Polishing
- Electropolishing
- Ion Etching
- Precision Ion Polishing System (PIPS)
- Ion Beam milling (FIB)
- Conductive Coating
Sample Preparation: Mechanical Polishing

- Grinding
  - 240 grit SiC until planar
  - 320 grit SiC 15 sec.
  - 400 grit SiC 15 sec.
  - 600 grit SiC 15 sec.
  - 800 grit SiC 15 sec.
  - 1200 grit SiC 15 sec.

- Polishing
  - 9, 6, 3 micron diamond solution (Optional)
  - 1 micron alpha alumina
  - 0.3 micron alpha alumina
  - 0.05/0.02 micron colloidal silica

SEM micrograph after 1200 grit SiC polish
No EBSD pattern visible
Nickel Sample

SEM micrograph after 3 micron diamond polish
No EBSD pattern visible
Nickel Sample
Sample Preparation: Mechanical Polishing

SEM micrograph after 1 micron alpha alumina
EBSD pattern IQ = 25
Nickel Sample

Sample Preparation: Mechanical Polishing

SEM micrograph after 0.3 micron alpha alumina
EBSD pattern IQ = 166
Nickel Sample

Sample Preparation: Mechanical Polishing

SEM micrograph after 10min colloidal silica
EBSD pattern IQ = 177
Nickel Sample
Sample Preparation: Mechanical Polishing

SEM micrograph after 30min colloidal silica
EBSD pattern IQ = 224
Nickel Sample

Final Stage: Oxide polishing

- Final stage is critical for most metal, ceramic and mineral samples
- Removes all damage layer by both chemical and mechanical means
- Various oxide suspensions with range of particle sizes (30-50nm typical), pH values (7-10) and particle types (alumina or silica). General all purpose polishing media usually have 40nm silica in a slightly alkaline solution, and have various commercial names (e.g. SYTON fluid, OP-5, MASTERMET etc.)
- Polishing time varies from a few minutes (soft metals) to many hours (rock samples)

Electropolishing

- Sometimes necessary after mechanical polishing
- Common approach for preparing Al alloys, some steels, zircalloys etc.
- Every material has a specific recipe, with variations in electrolyte, voltage, flow rate, time etc.
- Many recipes are tried and tested, and same as for TEM sample preparation (many commercial systems have extensive recipe lists)
- Residues from the sample should be completely cleaned before EBSD (e.g. use an ultrasonic bath)
Sample Preparation: Chemical etching

- An alternative to electropolishing technique
- Sample is immersed in an etchant for a few seconds, then rinsed (usually with ethanol) and blow-dried
- Simpler, as there are fewer variables and the choice of etchant is not so critical. One of the most commonly used ones is Nital (5% Nitric acid, 95% Ethanol)
- Ideal for preparing some materials that are difficult using other techniques – e.g. magnesium
- Always need to agitate the sample during etching to prevent bubble formation
- Can cause significant etching of grain boundaries

Ion beam milling – FIB/SEM

- Material removed from a tilted sample by the ion gun
- Ionized inert gas is accelerated towards the sample, and the impact removes sample material.
- Usually a low voltage and a very shallow incident angle (<20°) is necessary – slower is better for EBSD (may take 1-2 hours)
- Ideal for small / awkward samples (nanomaterials, wires, boundary layers...)
- May be limited in sample size (many dedicated ion milling preparation instruments cannot hold samples >10mm diameter)

Charging – a worse case scenario
Sample preparation: Conductive Coatings

Image Quality vs. Coating Thickness

- C on Ni
- Au/Pd on Ni
- C on Si
- Au/Pd on Si

Coating Thickness (Å)

Normalized IQ