Specimen Preparation for Light Microscopy

- Sectioning a sample – must be careful not to significantly alter the microstructure during sectioning
  - Fracturing
  - Shearing – substantial damage which must be subsequently ground off
  - Sawing – HB <350 can typically be cut by sawing. Produces roughness and heat and must be subsequently ground to remove damage.
  - Abrasive Cutting (most common) – thin rotating disk of a suitable abrasive in a supporting media is used.
  - Wire Saws – minimal damage. 3 modes – abrasive, electrolytic, and chemical etching.
Sectioning Artifacts

Figure 2-1 Influence of cutting procedure on deformation and damage to porcelain-enameled steel, 45×, 2% nital, polarized light. (Courtesy of A. O. Benscoter, Bethlehem Steel Corp.)
Sectioning Continued

- Electric Discharge Machining (EDM) – Requires a conductive material. Cutting is accomplished by an electric discharge between an electrode and the sample submerged in a dielectric media.

- Mounting – typically used for small or irregularly shaped samples

- Grinding – removes damage introduced by sectioning
  - Grinding occurs in sequences of finer and finer abrasives
  - Typical grit sequence – 120, 240, 320, 400, 600 mesh. Subsequent finer abrasives might also be employed.
  - Wet grinding is typically used: better to minimize heat generated, minimizes metal entrapment between particles,
Grinding Media – SiC paper

Figure 2-16a Appearance of the surface of silicon carbide grinding paper (Buehler Carbinet), 60x.
Sequential Images Specimen after Grinding and Polishing

Figure 2-15 Appearance of the surface of austenitic stainless steel at each step of the sample preparation sequence, 90×.
Grinding Continued

– Grinding procedure – each step typically 1-2 minutes. Rotate specimen 45-90 degrees after each polishing step. Rinse between steps to remove previous grinding media

– Grinding media
  • Silicon carbide SiC (mohs hardness 9.5) most common (high hardness and low cost)
  • Alumina (Al2O3) (mohs 9.1)
  • Emory (Al2O3 and iron oxide) (mohs 8.0) smoother – only good for dry grinding – not used much because wet grinding is preferred.

– Equipment
  • Polishing wheels – 8-12 inch diameter, moderate pressure moving sample center to edge. Rotation speed ~ 300-600rpm
  • Automatic grinding wheels – more reproducible
Grinding and Polishing Wheel

Figure 2-18 Example of a commercially available polishing table equipped with two 8-in wheels and a sink. (Courtesy of Leco Corp.)
Polishing

• Lapping – preparing surface using a disc surface impregnated with abrasive particles. Rotate sample in circular pattern counter to the wheel rotation

• Polishing
  – Coarse – 30 to 3 micron abrasives
  – Fine – typically < 1 micron
  – Procedure – careful cleaning between polish steps is critical to minimize carryover of larger abrasive particles to smaller abrasives
  – Sample orientation should not be held constant – continuously change moving sample from center to edge in a circular pattern counter to the wheel rotation
  – Rough polish – 150-600rpm ~ 6micron diamond
  – Fine polish – 1 micron diamond then 0.3 and 0.05 micron alumina
Polishing Continued

- Polishing cloths – must hold abrasives and must not contain foreign particles
- Grinding and polishing theory – hard abrasive particles scratch grooves, allow metal removal, and produce a plastically deformed surface region
  - Both grinding and polishing produce these three artifacts, however the extent is different depending on the pressure and particle size
  - The main difference between grinding and polishing is the rigidity of the grinding abrasive for grinding relative to the elasticity of the polishing media. This results in a lower contact pressure for polishing
Polishing Continued

- Electromechanical polishing – add an electrolytic etch to the mechanical polish (requires dc or ac power)
- Attack polishing – add a dilute chemical etchant to the polishing media to facilitate the mechanical abrasive with a chemical etch.
  - Can reduce or eliminate surface damage because it is a much gentler process.
  - Etch chemicals can damage equipment and person must take necessary precautions when handling the solutions
- Chemical polishing – purely chemical etch “a controlled corrosion process”
  - Put sample in a corrosive media and stir rigorously for uniform material removal
Polishing Continued

• Electro polishing – grind to 600 grit or mesh then the sample is made the anode of a electrolytic cell (+)
  – The recipe must have an appropriate (electrolyte, temperature, current, voltage, and time)
  – Advantages – easy to minimize surface damage
  – Disadvantages – dangerous chemicals, some phases in multiphase materials preferentially electrochemically etch so non-uniform polish results
General Sample Preparation

• 1. Sectioning – typically abrasive cutting
• 2. Mounting (optional)
• 3. Coarse grinding
• 4. Fine grinding
  – 3. and 4. Grinding sequence – 120, 240, 320, 400, 600 grit SiC rotating 45 or 90 degrees between steps (center to edge)
• 5. Coarse polishing – 6 micron diamond paste or 6 micron and 1 micron diamond. Rotate sample counter to the wheel rotation. Wash after each step under running water and rinse with alcohol
• 6. Fine polishing – 0.3 and 0.05 micron alumina slurry