Analytical Methods for Materials

Lesson 4

Metallography

Suggested Reading


Reference

• Goodhew, Humphreys and Beanland, Chapter 1
• Brandon and Kaplan, Chapter 3, pp. 123-177
Sample Preparation

Metallography:
- A systematic method to examine microstructure of materials (mainly metallic materials).
- Can also be used to examine ceramics, polymers and semiconductors.

(1) Sectioning
Why sectioning?
1. Size limitation of specimen to be examined under optical microscope.
2. Locate area needs to be selected from a large sample.
Sample Preparation

sectioning

• Abrasive Cutting is the most common sectioning method.

Adapted from MECH 4430 Lecture notes by Dr. Jingshen Wu and Dr. Yang Leng, Department of Mechanical Engineering, Hong Kong University of Science and Technology (http://www.me.ust.hk/~mejswu/).
Electric Discharge Machining (EDM)

- Electrically conductive materials can be produced via EDM.
- Cutting is accomplished by an electric discharge between an electrode and the sample submerged in a dielectric fluid.
Sample Preparation

sectioning

Microtomy:

- Useful for preparing soft materials such as polymer samples.
- Steel, glass or diamond knives in a microtome can cut samples into very thin sections.

(a) Adapted from http://www.me.ust.hk/~mejswu/.
(b) From http://blass.com.au/definitions/microtome
Sample Preparation

(2) Mounting

Required when (1) the sample is small or too oddly shaped to be handled. (2) The sample edge area needs to be examined

Thermal Mounting:
The sample is encased in thermosetting or thermoplastic polymers at high temperature and pressure

Adapted from MECH 4430 Lecture notes by Dr. Jingshen Wu and Dr. Yang Leng, Department of Mechanical Engineering, Hong Kong University of Science and Technology (http://www.me.ust.hk/~mejswu/).
Sample Preparation Techniques

**Cold Mounting:**
The sample is encased in epoxy type materials. Type of epoxy depends upon material being analyzed.

Adapted from MECH 4430 Lecture notes by Dr. Jingshen Wu and Dr. Yang Leng, Department of Mechanical Engineering, Hong Kong University of Science and Technology (http://www.me.ust.hk/~mejswu/).
### Sample Preparation Techniques

#### Hot Mounting Materials

<table>
<thead>
<tr>
<th>Name</th>
<th>Features</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin 1</td>
<td>Electropolishing</td>
<td>Electrically conductive Low shrinkage</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resin 3</td>
<td>Transparent mounts Porous material</td>
<td>Transparent Low shrinkage</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resin 5</td>
<td>Edge retention Planeness For highest requirements</td>
<td>Hard Good adhesion Wear resistant No shrinkage</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resin 6</td>
<td>Serial mounting</td>
<td>Medium shrinkage</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pre-Mounts</td>
<td>Serial mounting of uncomplicated shapes</td>
<td>Easy to handle Medium shrinkage</td>
</tr>
</tbody>
</table>

#### Cold Mounting Materials

<table>
<thead>
<tr>
<th>Name</th>
<th>Features</th>
<th>Material/filler</th>
<th>Curing time</th>
<th>Moulds</th>
</tr>
</thead>
<tbody>
<tr>
<td>Citofix</td>
<td>Serial mounting Irregularly shaped specimens</td>
<td>Low viscosity Good adhesion Translucent Low shrinkage</td>
<td>Acrylic 7 - 10 min</td>
<td>Epoform Flexform Senform Monoform</td>
</tr>
<tr>
<td>Durofix</td>
<td>Serial mounting Edge retention Irregularly shaped specimens</td>
<td>Low viscosity Hard Wear resistant Low shrinkage</td>
<td>Acrylic Mineral fillers 15 min</td>
<td>Epoform Flexform Senform Monoform</td>
</tr>
<tr>
<td>Triotol</td>
<td>Edge retention Planeness</td>
<td>Good adhesion Very hard Wear resistant Very low shrinkage</td>
<td>Polyester/ Acrylic/ Mineral filler 15 - 18 min</td>
<td>Epoform Flexform Monoform</td>
</tr>
<tr>
<td>Epofix</td>
<td>Vacuum impregnation Porous samples Mineralogical samples</td>
<td>Low vapour-pressure Transparent Good adhesion Low viscosity No shrinkage</td>
<td>Epoxy 8 h</td>
<td>Epoform</td>
</tr>
<tr>
<td>Calotol</td>
<td>Vacuum impregnation Porous samples Mineralogical samples</td>
<td>Low vapour-pressure Transparent Good adhesion Low viscosity Very low shrinkage</td>
<td>Epoxy 1 h at 80°C</td>
<td>Epoform</td>
</tr>
</tbody>
</table>

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Sample Preparation Techniques

*Adhesive Mounting:* The sample is glued to a piece of a large holder

*Clamp Mounting:* The sample is fixed in mechanical clamping devices

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Sample Preparation

(3) Grinding

1. Removes the damage from the surface produced by sectioning.
2. Grinding also produces damage which must be minimized by subsequent grinding with finer abrasives.
3. At the end of grinding phase, the only grinding damage present must be from the last grinding step.
4. Such damage will be removed by polishing.

Grinding Materials: Abrasive paper (covered with silicon carbide grit). Commonly a series of abrasive papers are used from coarse to fine.

Typical Grit Sequence: 120-, 240-, 320-, 400-, 600-, 1200-, 2400-, etc.

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Sample Preparation Techniques

- The initial grit size depends on the surface roughness and depth of damage from sectioning.

- Surfaces cut with abrasive cutoff saws generally start with 120- to 240- grit surface finishes.

- Surfaces cut by EDM or diamond saws generally start with 320- to 400- grit surface finishes.

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Sample Preparation Techniques

Specimen surfaces after grinding and polishing.

Leng, p. 20
Sample Preparation Techniques

(4) Polishing

After being ground to a 600-grit finish (or better), the sample is polished to produce a flat and scratch-free surface with high reflectivity.

Coarse polishing: abrasives in the range of 30 $\mu$m to $\sim$3 $\mu$m using diamond grits of the appropriate size.

Fine polishing: abrasives in the range of 1$\mu$m or less using diamond grits of the appropriate size.

Final polishing: 0.25-0.05 $\mu$m diamond, silica, or alumina slurries.
Automatic Polishing Machines

Figure 1.24  Polishing on a rotating wheel with a mechanical sample holder. From Leng, p. 21.
Sample Preparation

Artifact structure from improper grinding

Surface deformation from improper grinding should be avoided, otherwise the microstructure may be obscured.

Adapted from MECH 4430 Lecture notes by Dr. Jingshen Wu and Dr. Yang Leng, Department of Mechanical Engineering, Hong Kong University of Science and Technology (http://www.me.ust.hk/~mejswu/).
Sample Preparation

**Artifacts from improper polishing**

Polishing should produce a scratch-free surface. Too much pressure can cause artifacts such as the comet tail artifacts shown below.

From page 22 in Leng
Sample Preparation

Grinding

- 240 Grit
- 400 Grit
- 600 Grit

Polishing

- Clean btw. every step
- Rotate 90°

Check under LOM after each step

Grinding direction

Microstructural features become more visible

6 μm → 3 μm → 1 μm → 0.25 μm

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(5) Etching

- Using chemicals to selectively dissolve the surface of a material in order to reveal microstructural details.

- Grain boundaries are more susceptible to etching.

- May be revealed as dark lines.

- Due to change in crystal orientation across GB.

Adapted from Fig. 4.14(a) and (b), Callister & Rethwisch 8e.
(Fig. 4.14(b) is courtesy of L.C. Smith and C. Brady, the National Bureau of Standards, Washington, DC [now the National Institute of Standards and Technology, Gaithersburg, MD].)
Sample Preparation

electrolytic polishing/etching

Etching is basically a controlled corrosion process. Results from electrolytic action between surface areas of different potential.

Electrolytic activity results from local physical or chemical heterogeneities which render some features anodic and others cathodic under the specific etching conditions.

Chemical Etchants produce contrast by

- Crystal faceting
- Selective phase dissolution.

Common chemical etchants have three components:

- A corrosive agent (acids)
- A modifier (alcohol, glycerin…)
- An oxidizer (hydrogen peroxide, Fe$^{3+}$, Cu$^{2+}$…)

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Sample Preparation

electrolytic polishing/etching

- Specific conditions are needed to ensure best polishing.
- Same procedures can be used to anodize materials.

Schematic drawing of an electropolishing apparatus. From page 159 in Geels.

Theoretical current density versus voltage curve for electrolytic polishing/etching. From page 159 in Geels.