Analytical Methods for Materials

Lesson 4

Metallography

Suggested Reading

•Y. Leng, *Materials Characterization, 2nd Edition*, (2013), Wiley, Hoboken, NJ – Chapter 1.

Reference

•Goodhew, Humphreys and Beanland, Chapter 1

•Brandon and Kaplan, Chapter 3, pp. 123-177

•K. Geels, D.B. Fowler, W-U. Kopp, and M. Rückert, *Metallographic and Materialographic Specimen Preparation, Light Microscopy, Image Analysis and Hardness Testing*, (2007) ASTM International, West Conshohocken, PA.

•G.F. Vander Voort, Metallography Principles and Practice, (1999) ASM International, Materials Park, OH.

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/).

Sample Preparation

sectioning

Electric Discharge Machining (EDM)

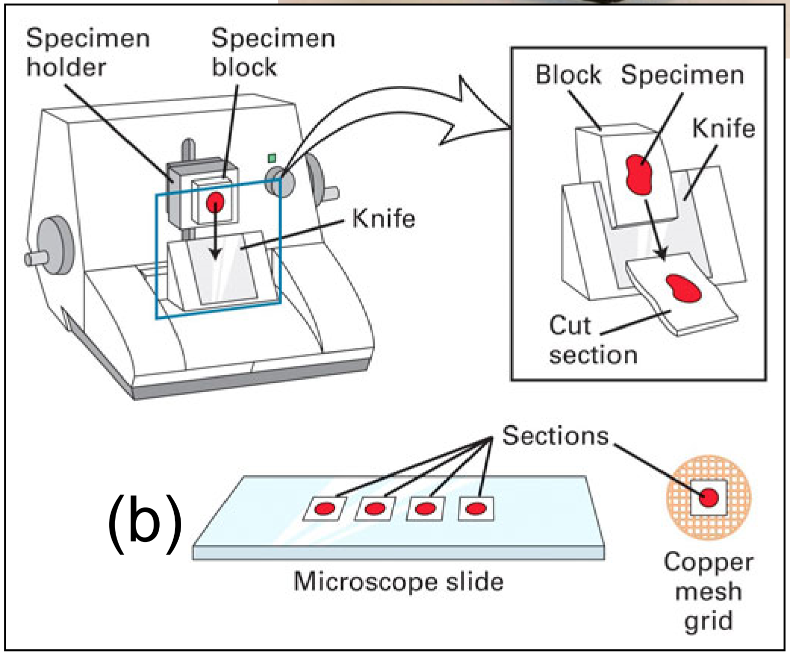
- Electrically conductive materials can be produced via EDM.
- Cutting is accomplished by an electric discharge between an electrode and the sample submerged in a dielectric fluid.

http://en.bhmachinery.com/login/images/upload/20092141553480249.jpg

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.



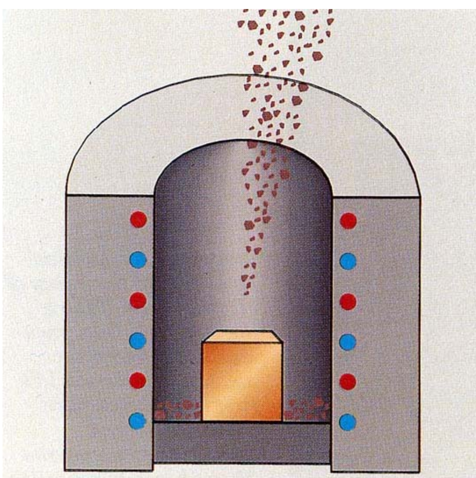
(a) Adapted from http://www.me.ust.hk/~mejswu/.
(b) From http://blass.com.au/definitions/microtome

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

<u>*Thermal Mounting*</u>:
The sample is encased in thermosetting or thermoplastic polymers at high temperature and pressure



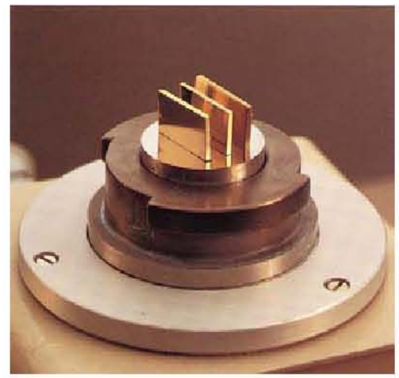
Adapted from MECH 4430 Lecture notes by Dr. Jingshen Wu and Dr. Yang Leng, Department of Mechanical Engineering, Hong Kong University of Science and Technology (http://www.me.ust.hk/~mejswu/).

Sample Preparation Techniques

Cold Mounting:

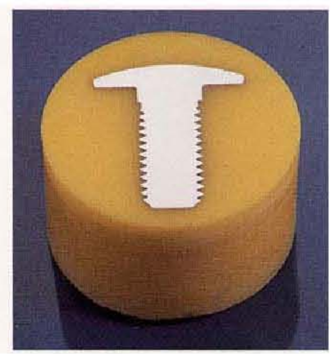
The sample is encased in epoxy type materials. Type of epoxy depends upon material being analyzed.











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Sample Preparation Techniques

Hot Mounting Materials

Name	Features: Application	Features: Specific property	Material
Resin 1 	Electropolishing	Electrically conductive Low shrinkage	Acrylic Thermoplastic
Resin 3 	Transparent mounts Porous material	Transparent Low shrinkage	Acrylic Thermoplastic
Resin 5 	Edge retention Planeness For highest requirements	Hard Good adhesion Wear resistant No shrinkage	Epoxy Thermosetting
Resin 6 	Serial mounting	Medium shrinkage	Bakelite Thermosetting
Pre-Mounts 	Serial mounting of un-complicated shapes	Easy to handle Medium shrinkage	Bakelite Thermosetting

Cold Mounting Materials

Name	Features: Application	Features: Specific property	Material/filler / Curing time	Moulds
Citofix 	Serial mounting Irregularly shaped specimens	Low viscosity Good adhesion Translucent Low shrinkage	Acrylic 7 - 10 min	Epoform Flexiform Seriform Monoform
Durofix 	Serial mounting Edge retention Irregularly shaped specimens	Low viscosity Hard Wear resistant Low shrinkage	Acrylic Mineral fillers 15 min	Epoform Flexiform Seriform Monoform
Triofix-2 	Edge retention Planeness	Good adhesion Very hard Wear resistant Very low shrinkage	Polyester/ Acrylic/ Mineral filler 15 - 18 min	Epoform Flexiform Monoform
Epofix 	Vacuum impregnation Porous samples Mineralogical samples	Low vapour-pressure Transparent Good adhesion Low viscosity No shrinkage	Epoxy 8 h	Epoform
Caldofix 	Vacuum impregnation Porous samples Mineralogical samples	Low vapour-pressure Transparent Good adhesion Low viscosity Very low shrinkage	Epoxy 1 h at 80°C	Epoform

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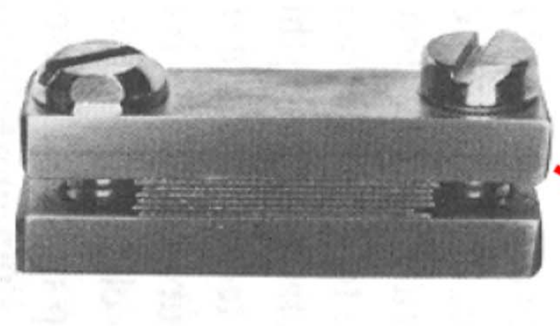
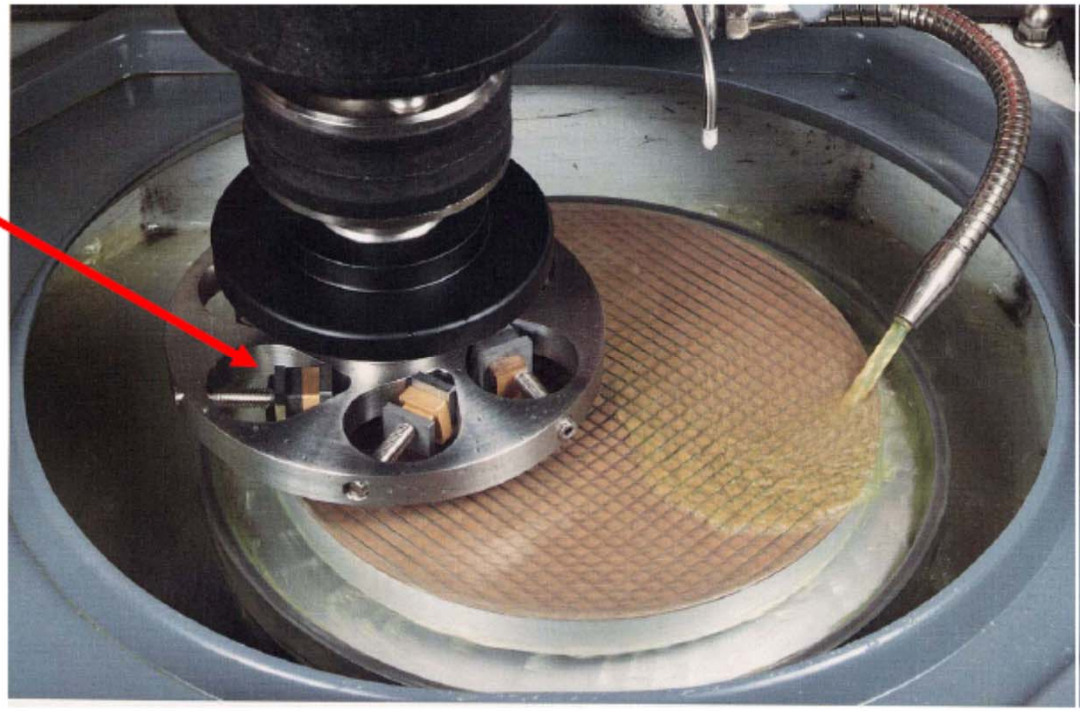
Sample Preparation Techniques

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

Clamp Mounting: The sample is fixed in mechanical clamping devices





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

(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.

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

- 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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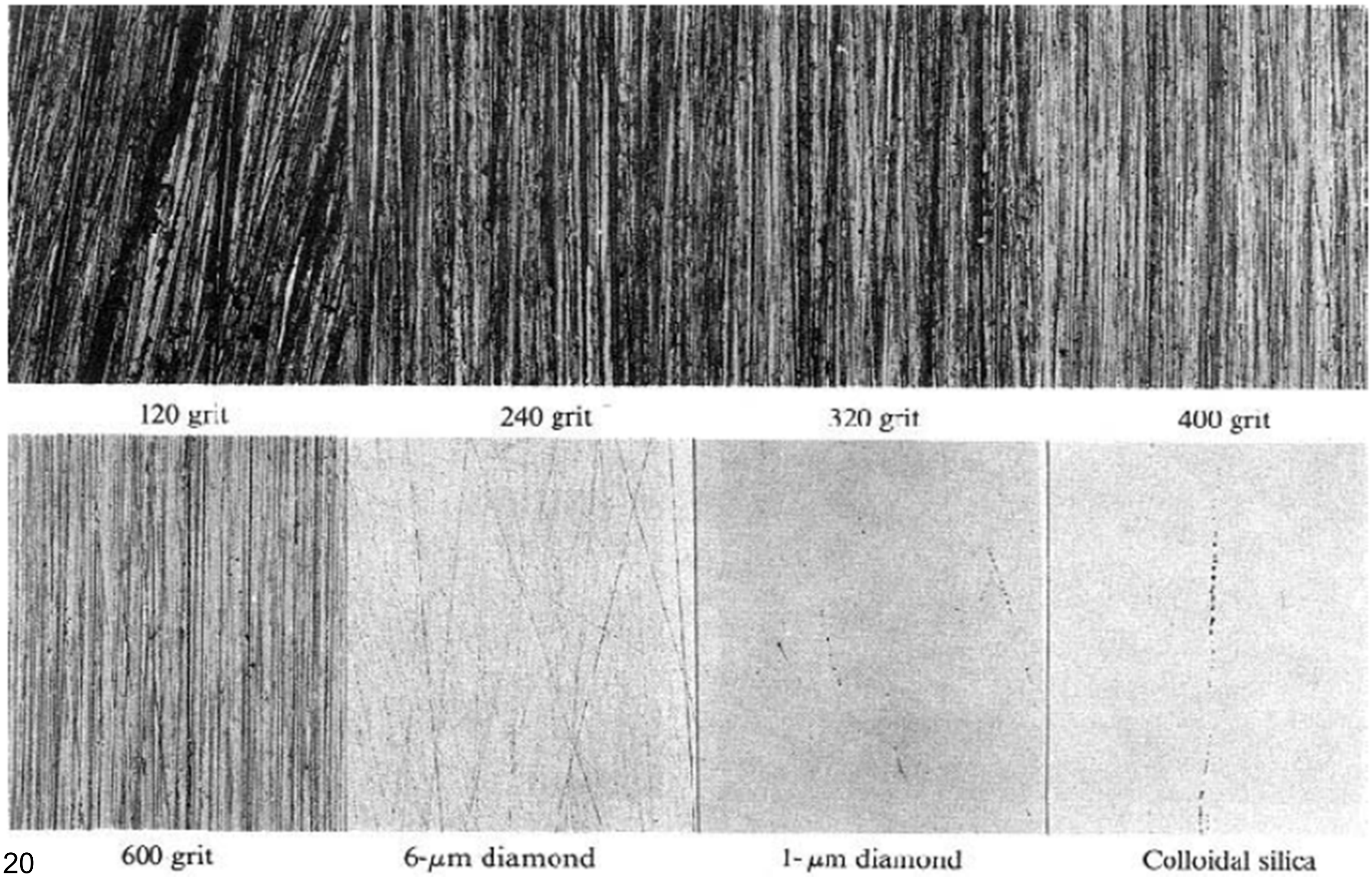
Sample Preparation Techniques



Leng, p. 20

Specimen surfaces after grinding and polishing.

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 μm to ~3 μm using diamond grits of the appropriate size.

Fine polishing: abrasives in the range of 1μm or less using diamond grits of the appropriate size.

Final polishing: 0.25-0.05 μm diamond, silica, or alumina slurries.

Automatic Polishing Machines

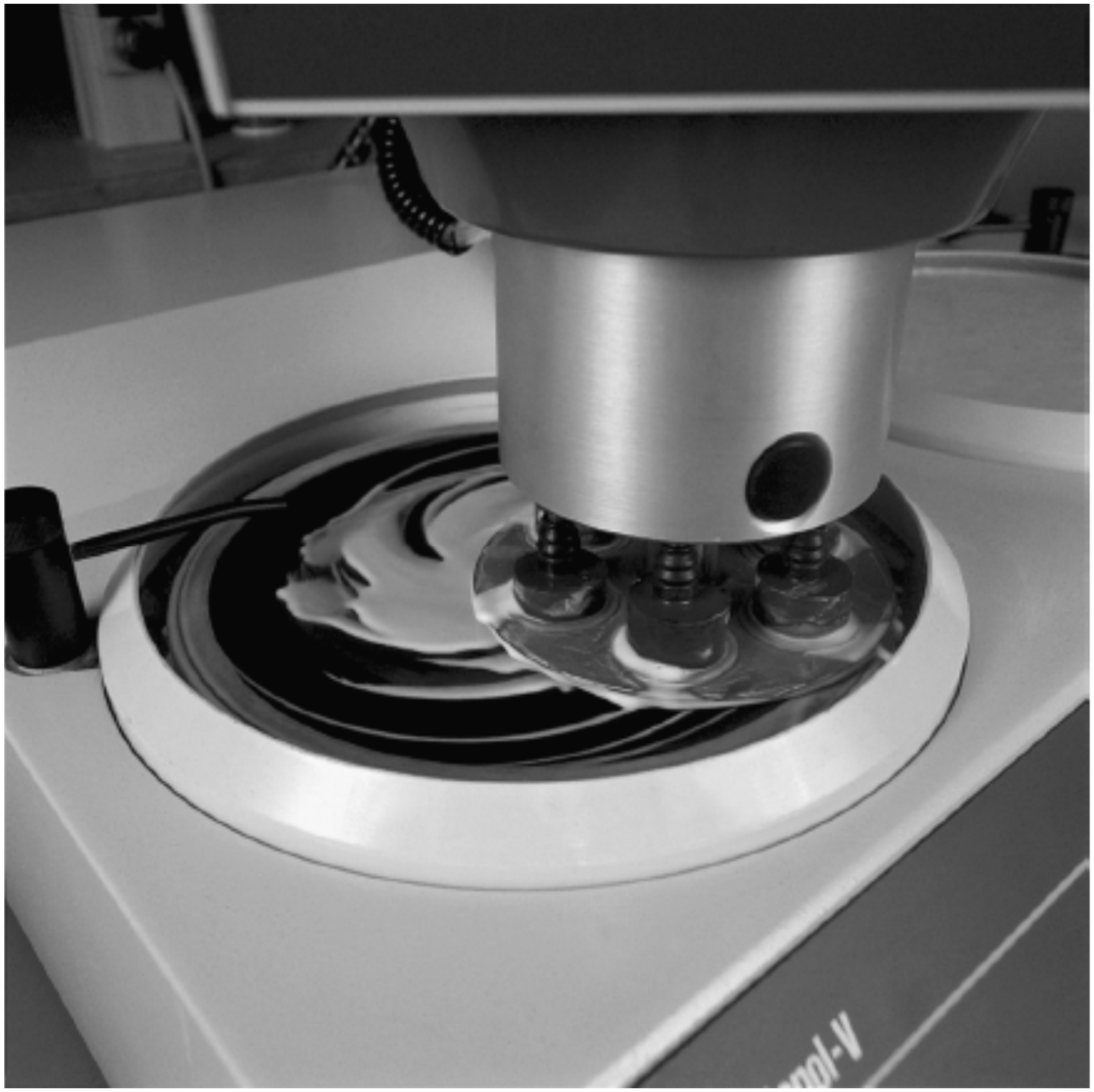
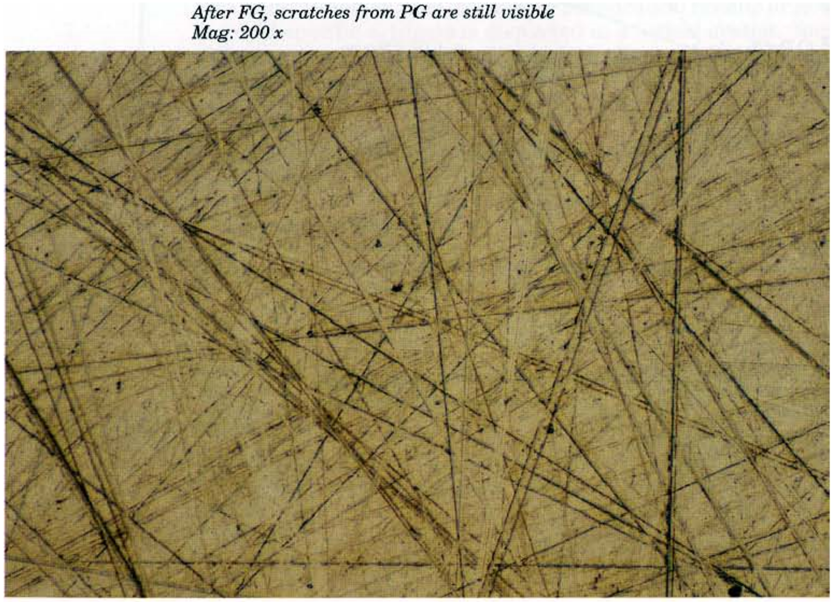


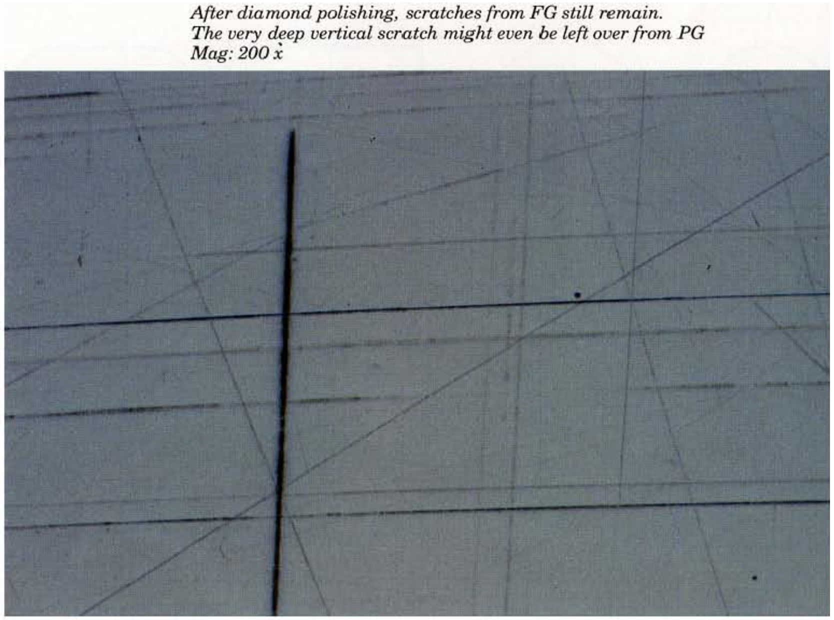
Figure 1.24 Polishing on a rotating wheel with a mechanical sample holder.
From Leng, p. 21.

Sample Preparation

Artifact structure from improper grinding

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





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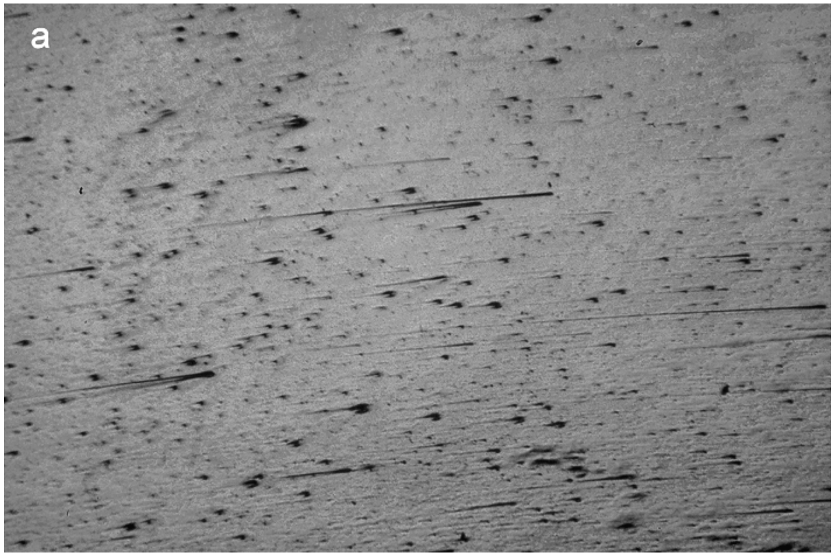
Sample Preparation

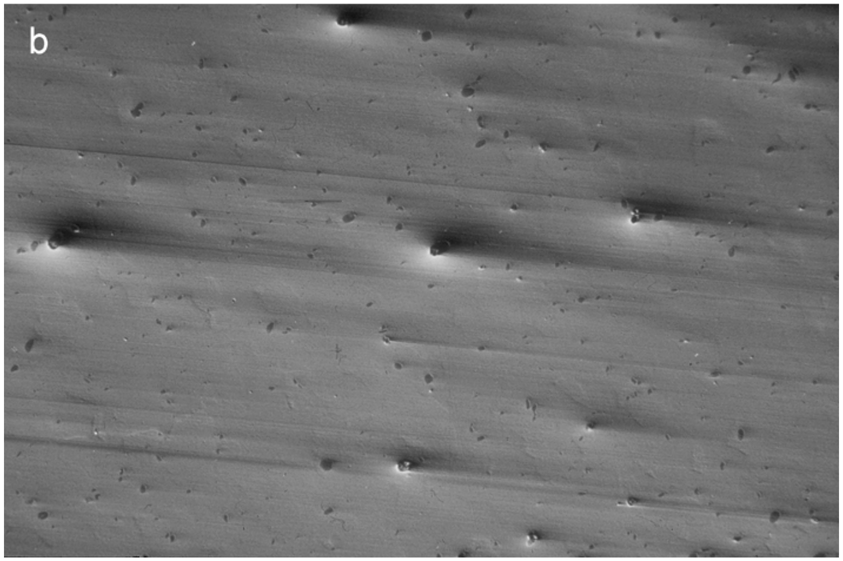
Artifacts from improper polishing

Polishing should produce a scratch-free surface.

Too much pressure can cause artifacts such as the comet tail artifacts shown below.



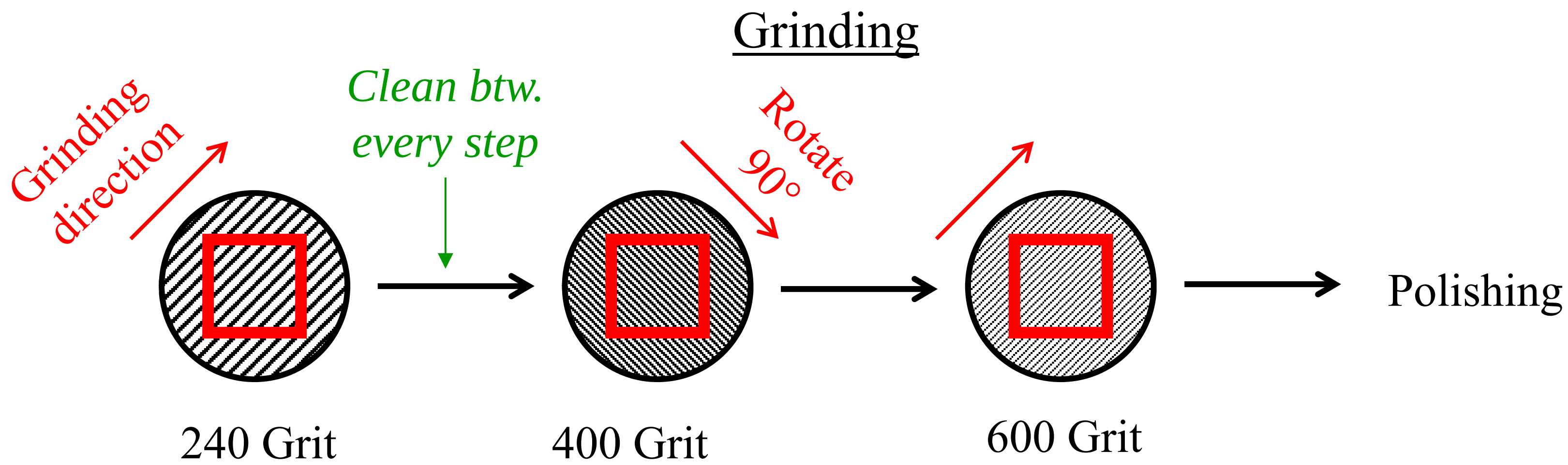
Bright-field image



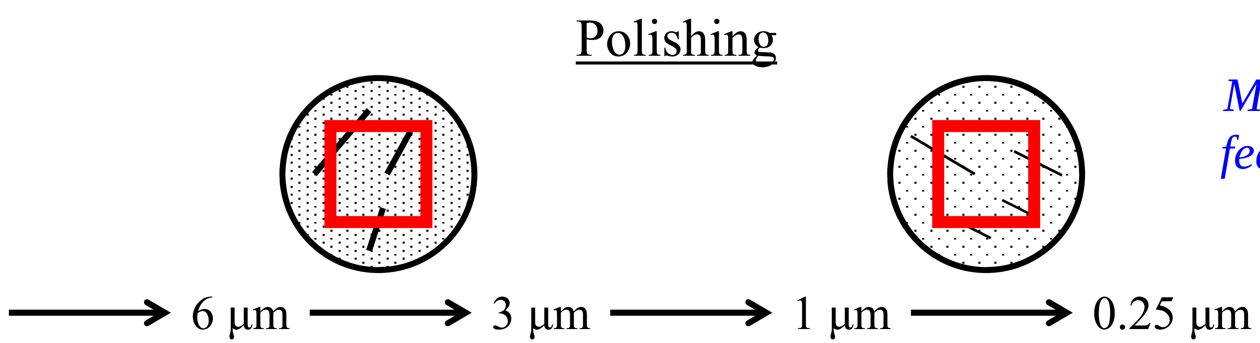
DIC image

From page 22 in Leng

Sample Preparation



Check under LOM after each step



Microstructural features become more visible

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/).

Optical Microscopy

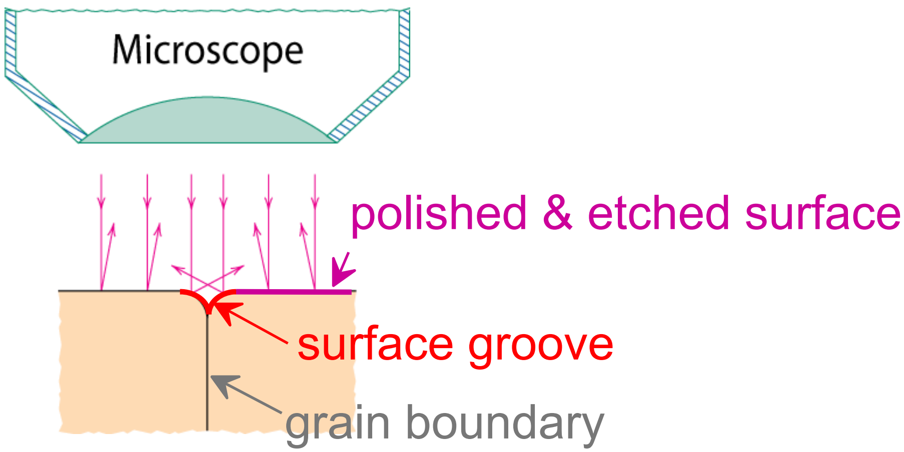
(5) Etching

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

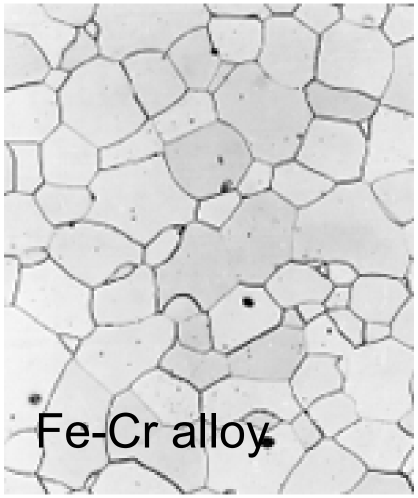
- Grain boundaries are more susceptible to etching.

- May be revealed as dark lines.

- Due to change in crystal orientation across GB.



(a)



(b)

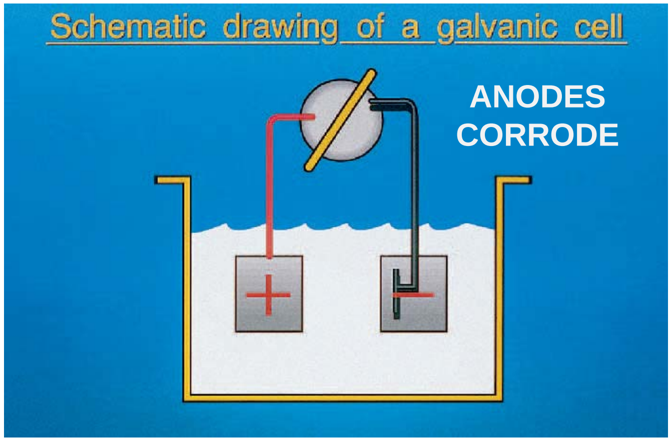
Adapted from Fig. 4.14(a) and (b), *Callister & Rethwisch 8e.* (Fig. 4.14(b) is courtesy of L.C. Smith and C. Brady, the National Bureau of Standards, Washington, DC [now the National Institute of Standards and Technology, Gaithersburg, MD].)

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.



From page 156 in Geels

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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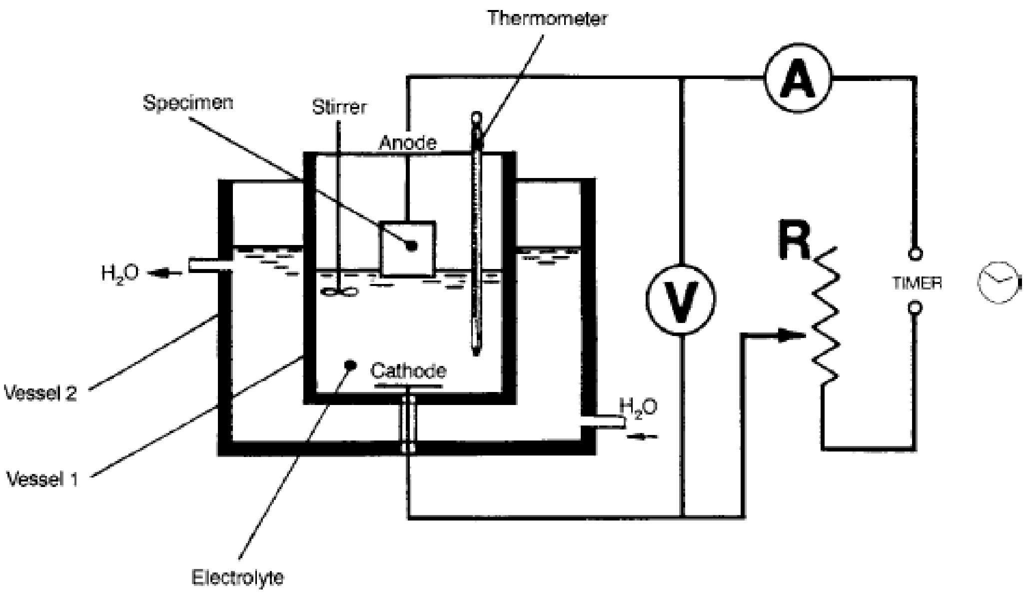
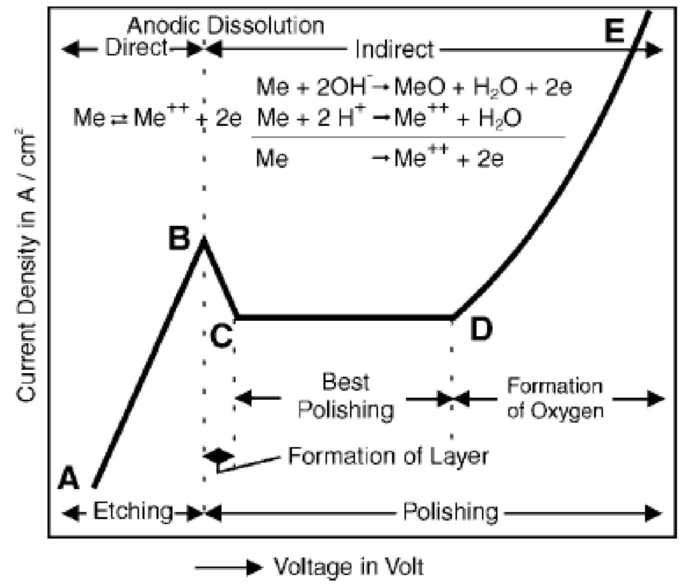
Sample Preparation

electrolytic polishing/etching



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.

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