2009 spring

# *Microstructural Characterization of Materials*

**04. 01. 2009**

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# Contents for previous class

- Presentation for various optical microscopes
  - Phase contrast microscope
  - Differential interference contrast microscope
  - Fluorescence Microscopy
  - Multi-photon laser scanning microscope
  - 벡터장 나노 현미경
  - Digital Holographic Microscope

# Sample Preparation

## What is Metallography??

- **Metallography is the science and art of preparing a surface for analysis by grinding,**

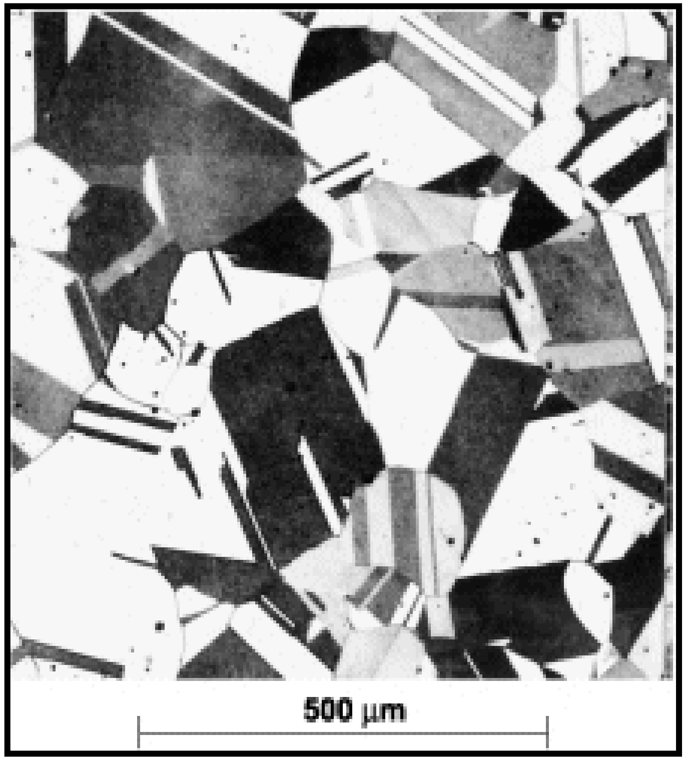
  **polishing, and**

  **etching**

  **to show microstructual component.**

  **(mainly metallic materials)**

- **Metallography does not only apply to metallic materials, such technology can also be used examine ceramics, polymers and semiconductors.**



# 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

## (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.

**Sectioning a sample** – must be careful not to significantly alter the microstructure during sectioning.

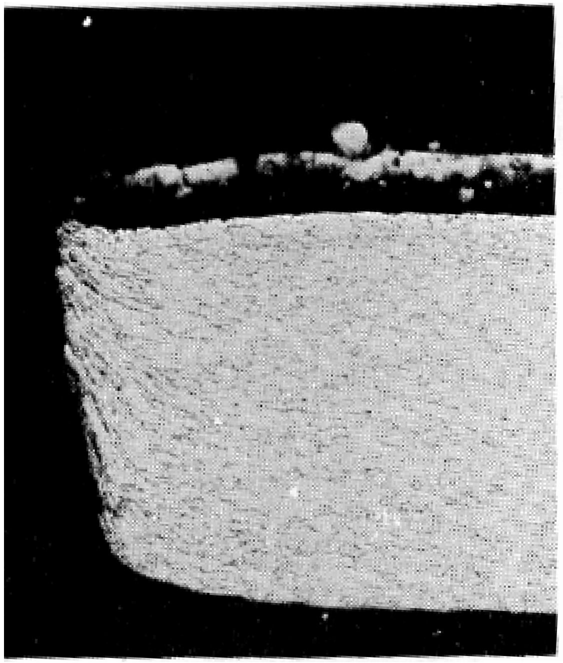
- **Fracturing**
- **Shearing**: substantial damage which must be subsequently ground off
- **Sawing**: HB<350 can typically be cut by sawing. Produces roughness and heat and must be subsequently ground to remove damage.
- **Abrasive Cutting**: (most common) thin rotating disk of a suitable abrasive in a supporting media is used.

# Sample Preparation

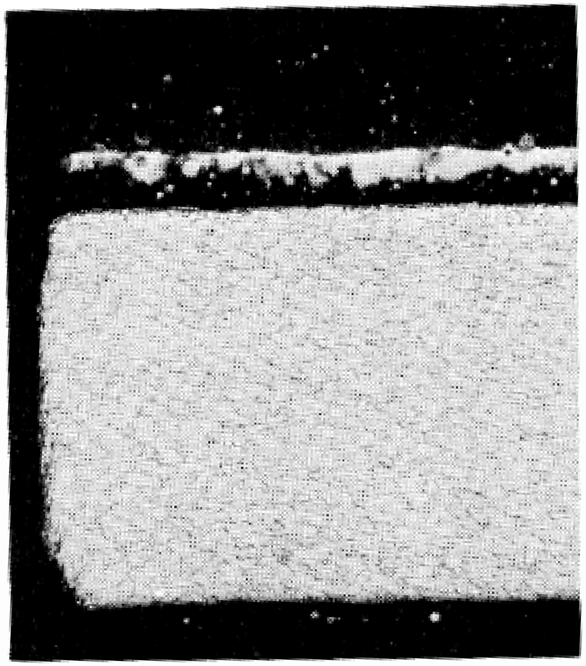
- Abrasive Cutting is the most common sectioning method.

**Band saw** **Abrasive cutter**

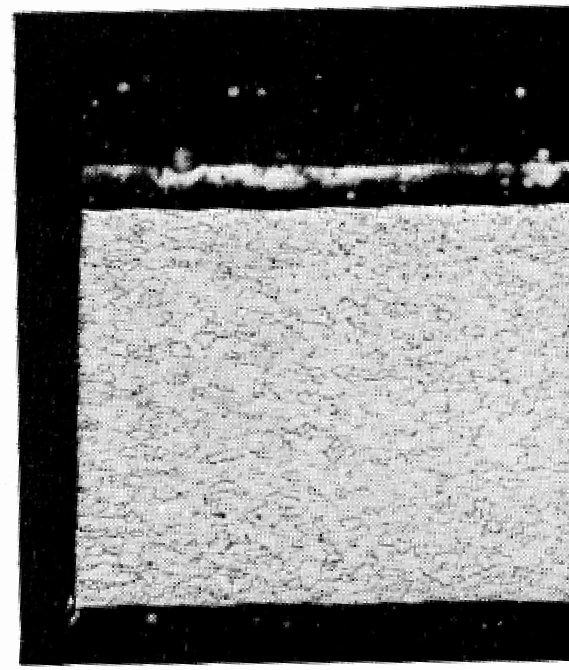
# Sectioning Artifacts



**shear**



**Band saw**



**Abrasive cutoff**

**Influence of cutting procedure on deformation and damage to porcelain-enameled Steel, 45x, 2% nital, polarized light. (*courtesy of A. O. Benscoter, Bethlehem Steel Corp*.)**

# Sample Preparation

**Electric Discharge Machining (EDM)**

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

**http://drm.kist.re.kr/cerapedia/process/jgl007.html**

# Sample Preparation

**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

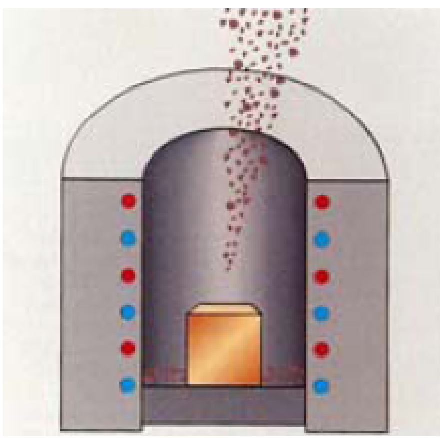
# Sample Preparation

**(1) 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 plastics at high temperatures and pressure



# Sample Preparation Techniques

*Cold Mounting*:

The sample is encased in epoxy type materials

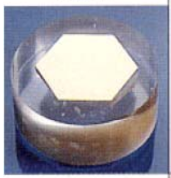
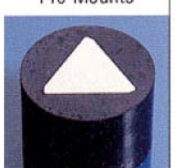







# Sample Preparation Techniques

## Hot Mounting Materials

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

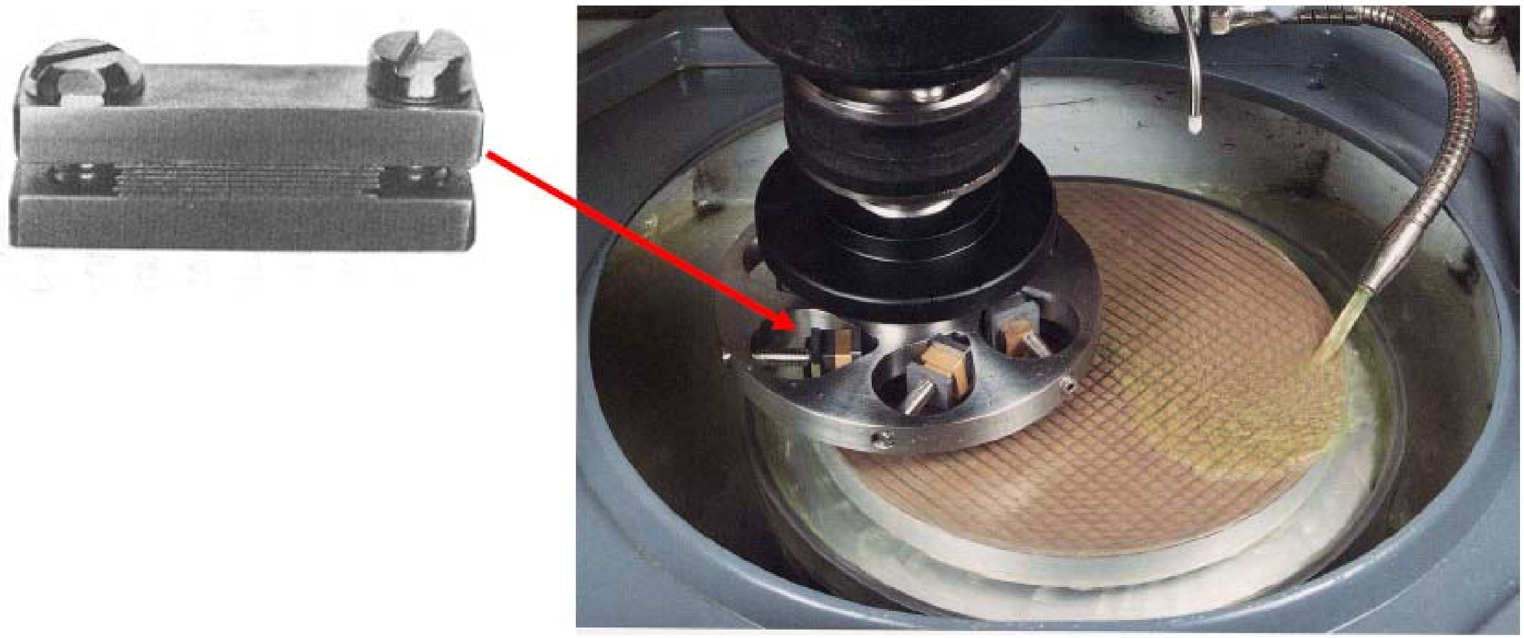
## Cold Mounting Materials

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

# Sample Preparation Techniques

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

*Clamps:* The sample is fixed in mechanical clamping devices



# Sample Preparation

**(3) Grinding**

1. Grinding removes the damage from the surface produced by sectioning.
2. Grinding also produced damage which must be minimized so that 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.

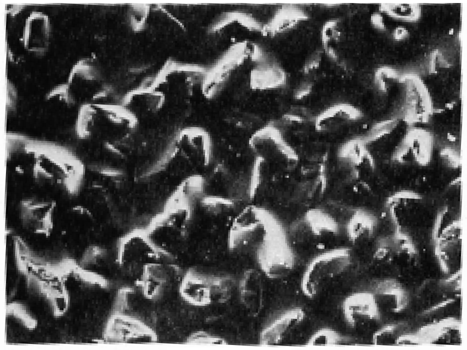
Grinding Materials: Abrasive paper (covered with silicon carbide grit). Commonly a series of abrasive papers are used from coarse to fine.

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

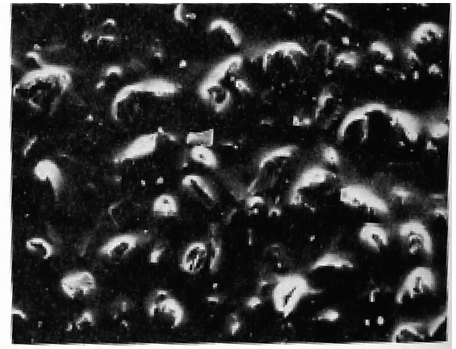
# What is grit?

- When talking about sandpaper, "**grit**" is a reference to the **number of abrasive particles per inch** of sandpaper.
- The **lower** the grit the rougher the sandpaper and conversely, the **higher** the grit number the smoother the sandpaper.
- This make sense if you imagine how small the particles on an 800-grit sandpaper would need to be to fit into a 1" square.
- Sandpaper is referred to by the **size of its grit** (i.e. 150-grit sandpaper).

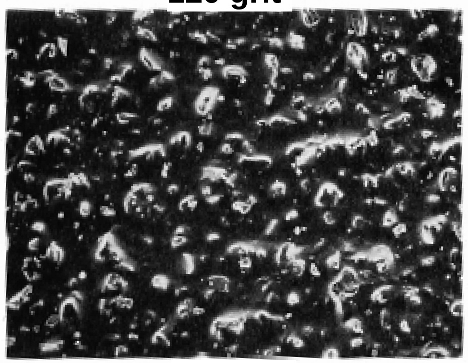
# Grinding Media – SiC paper



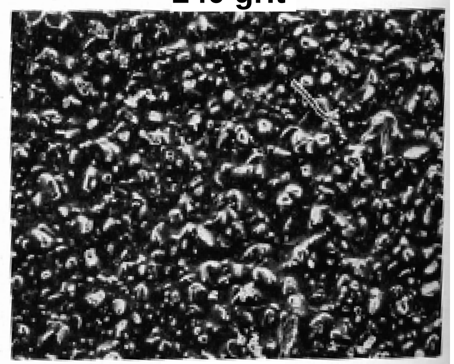
120 grit



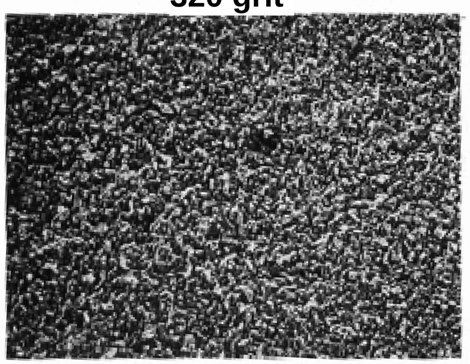
240 grit



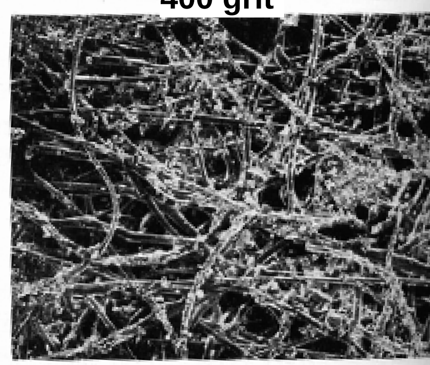
320 grit



400 grit



600 grit



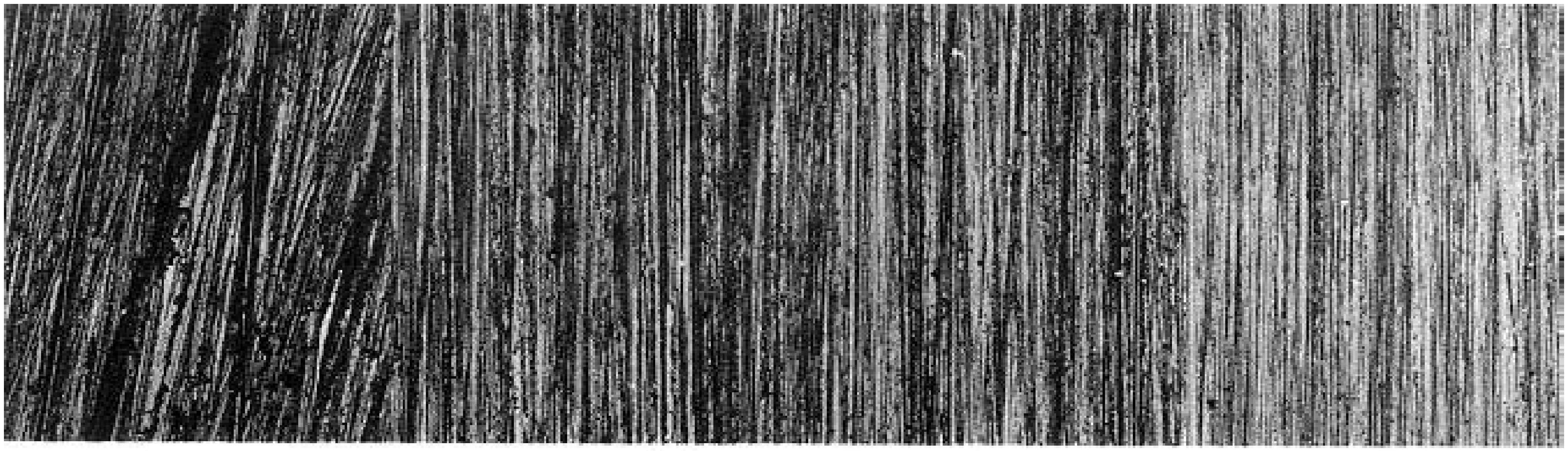
600 soft

**Appearance of the surface of silicon carbide grinding paper (Buchler Carbiment). 60 x**

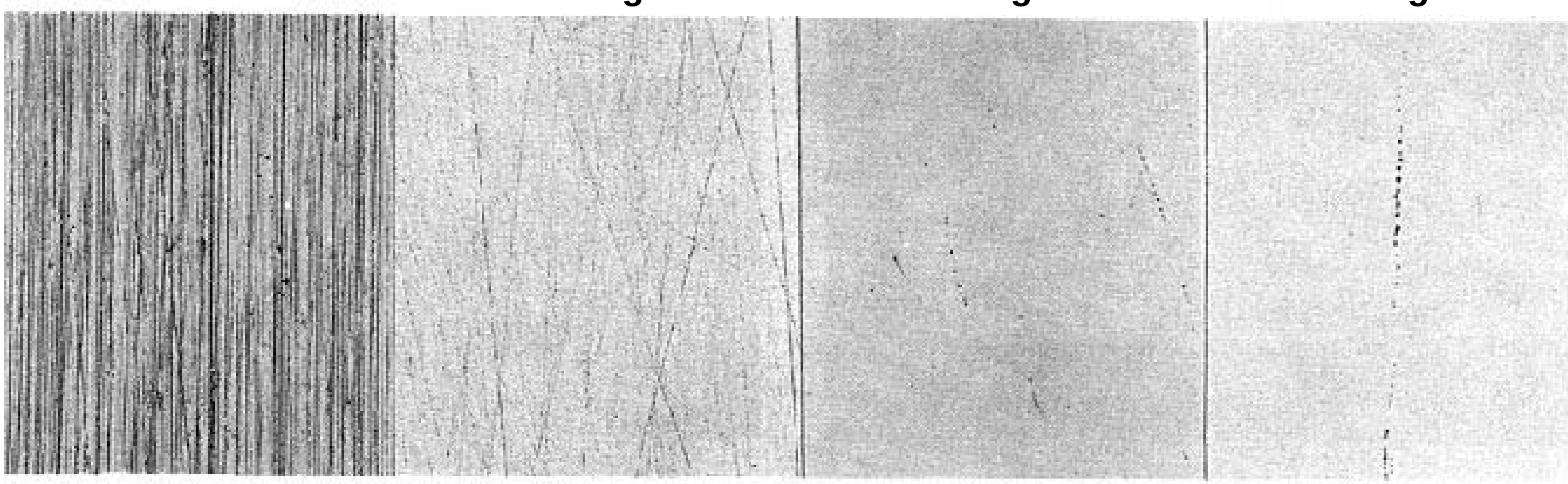
# Sample Preparation Techniques

- 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


# Sample Preparation Techniques



120 grit | 240 grit | 320 grit | 400 grit



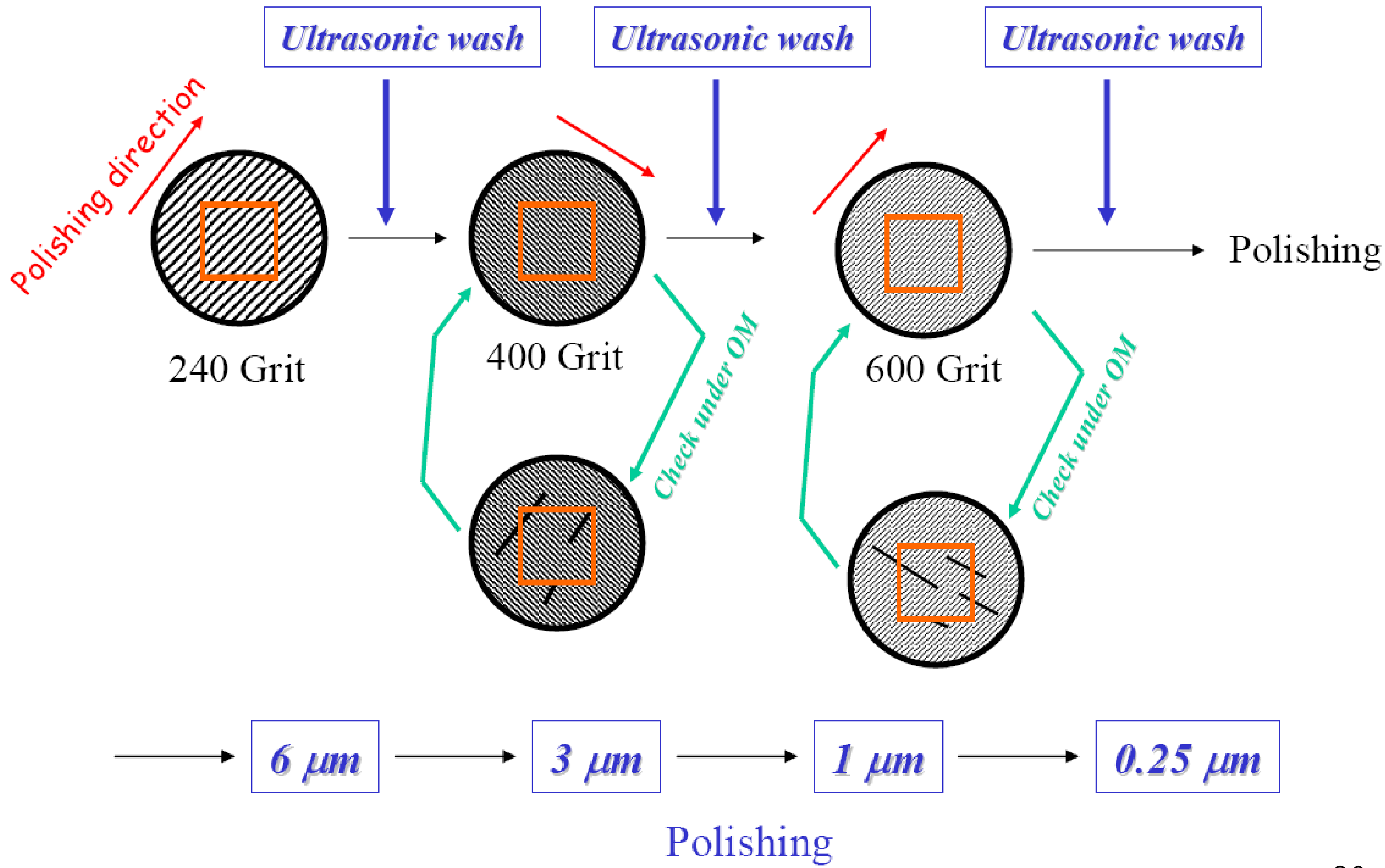
600 grit | 6-μm diamond | 1-μm diamond | colloidal silica

# Sample Preparation Techniques

## (4) Polishing

- Lapping – preparing sruface 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 sampple from center to edge in a circular pattern counter to the wheel rotation
  - Rough polish – 150-600rpm ~ 6micron diamond
  - Finle polish – 1 micron diamond then 0.3 and 0.05 micron alumina

# Sample Preparation Techniques



# Sample Preparation Techniques

## Polishing Continued

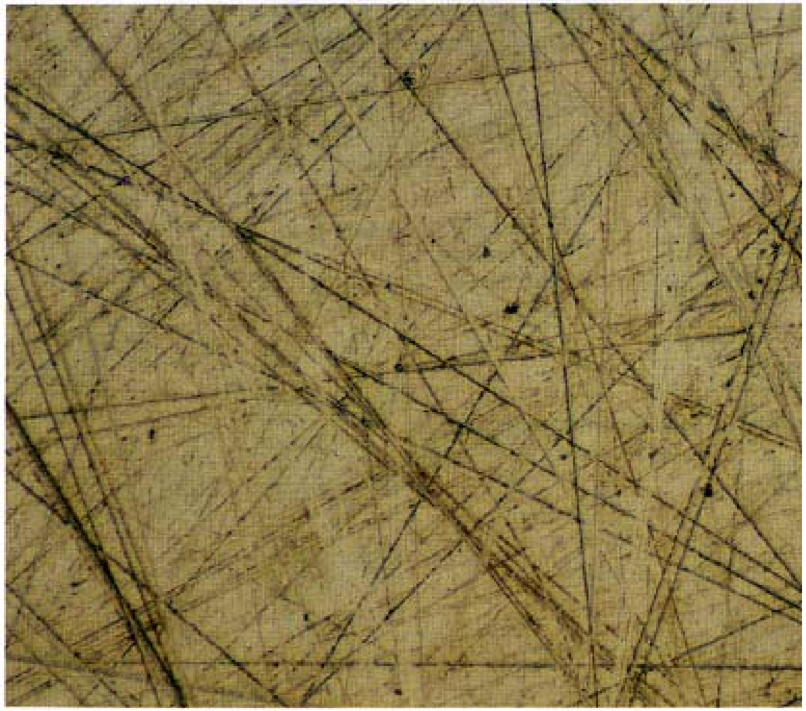
- Polishing cloths – must hold abrasives and must not contain foreign particles
- Grinding and polishing theory – hard abrasive particles scratch grooves, allow metal removal, and produce a plastically deformed surface region
  - Both grinding and polishing produce these three artifacts, however the 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

# Sample Preparation Techniques

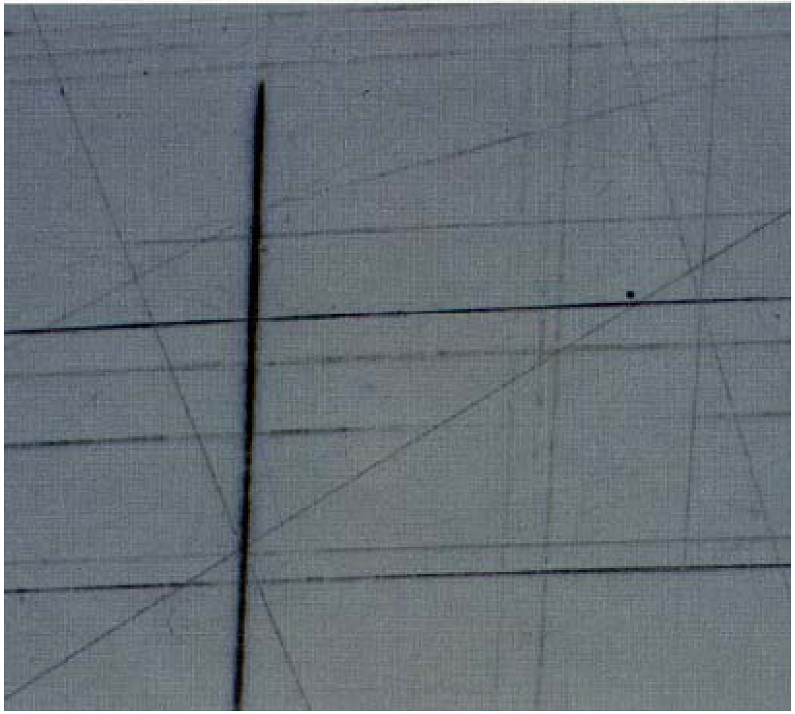
# Sample Preparation Techniques

**Artifact structure from improper grinding**

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



*After FG, scratches from PG are still visible.*
**Mag: 200x**



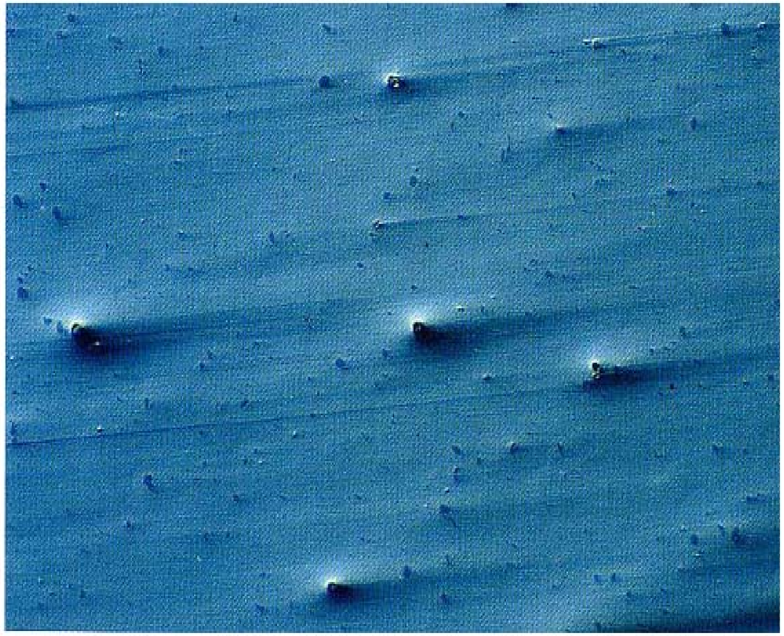
*After diamond polishing, scratches from FG are still remain.*
*The very deep vertical scratch might even be left over from PG.*
**Mag: 200x**

# Sample Preparation Techniques

**Artifact structure from improper polishing**

Polishing should produce a scratch-free surface. Excessive pressure may cause artifact of second phase particles as shown below.

**20 x**



**200 x**

# 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

# Sample Preparation Techniques

- 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
  - Disadvantage – dangerous chemicals, some phases in multiphase materials preferentially electrochemically etch so non-uniform polish results.

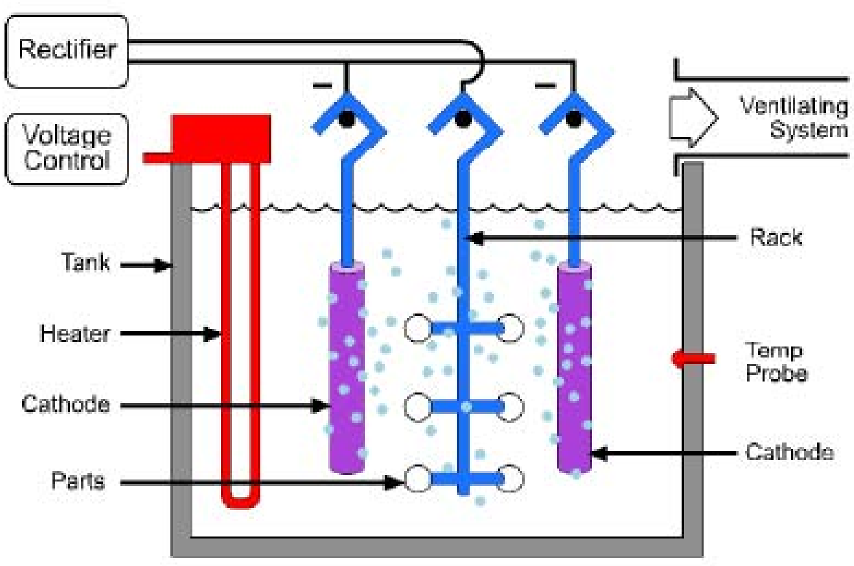



Figure 1: Typical Electropolishing Installation

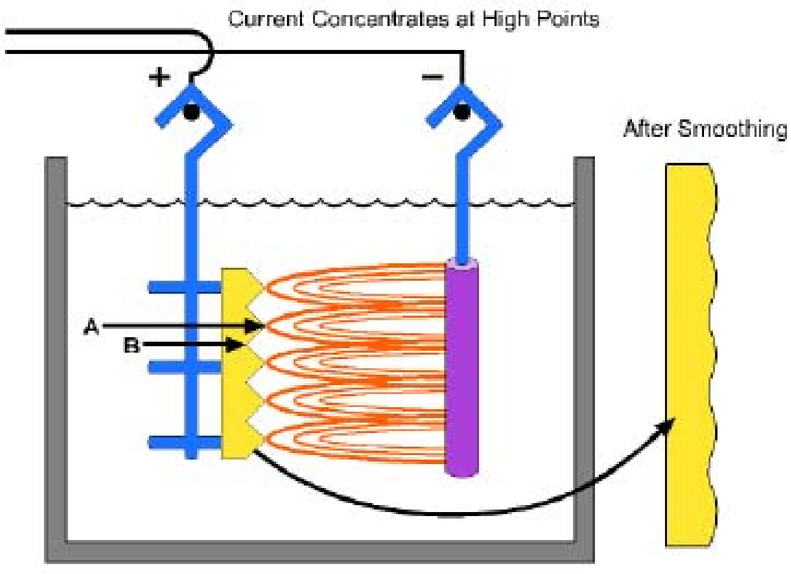


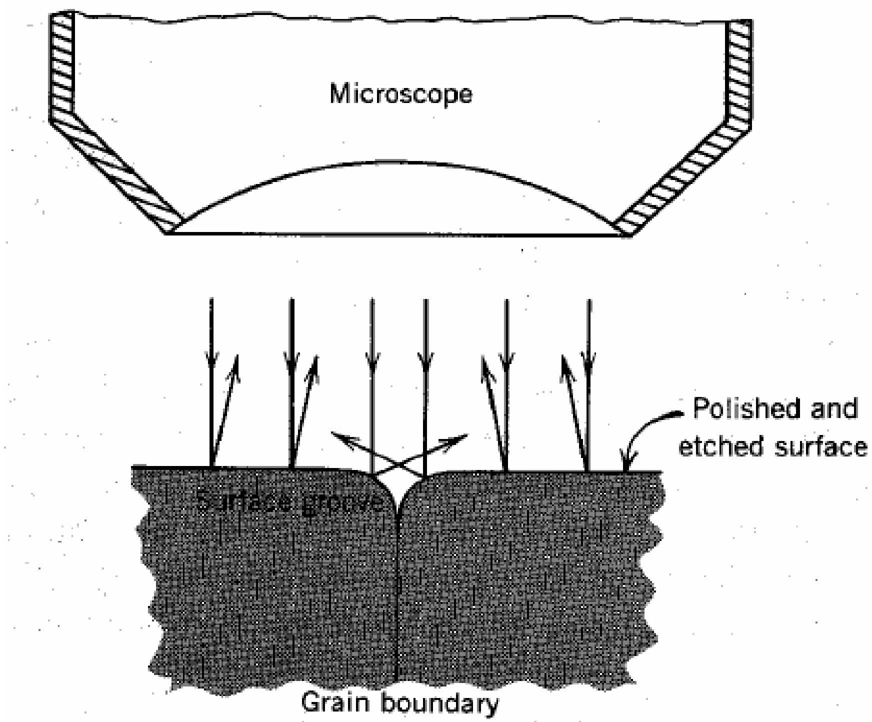

Figure 2: Smoothing by Electropolishing

# Sample Preparation Techniques

**(5) Etching**

**Using chemical to dissolve selectively the surface of materials in order to reveal the inhomogeneous nature in microscopic scale.**

***For example the grain boundaries of polycrystalline metal***



# Sample Preparation Techniques

etching

Etching is basically a controlled corrosion process resulting from electrolytic action between surface area 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, $Fe^{3+}$, $Cu^{2+}$…)

# Sample Preparation Techniques

***Common etchants for metals (for example)***

***Keller's reagent***
**2.5 ml $HNO_3$/1.5 ml HCl**
**1.0 ml HF, 95 ml water** **for Al and alloys**

***Nital***
**1-10 ml $HNO_3$ in 90-99 ml methanol** **for Fe and steel**

***Picral***
**4 -10 g picric acid, 100 ml ethanol** **for Fe and steel**

**10 ml HF/5 ml $HNO_3$ 85 ml water** **for Ti and alloys**

**$NH_4OH$/3% $H_2O_2$** **for Cu and alloys**

# Sample Preparation Techniques

***Etchants for polymers (for example)***

| | |
|---|---|
| **Aqueous solution of $CrO_3$** | **for bulk polypropylene (PP)** |
| **Aqueous solution of $H_2SO_4/H_3PO_4/CrO_3$** | **for ABS, HIPS and PPO** |

***Etchants for minerals (for example)***

| | |
|---|---|
| **Concentrated HCl** | **for CaO, or MgO.** |
| **Aqueous solution of $H_3PO_4$** | **for $Al_2O_3$** |
| **Aqueous solution of $HNO_3$/HF** | **for $CeO_2$, $SrTiO_3$, $Al_2O_3$, and ZrO-ZrC.** |

# Sample Preparation Techniques

## Safety Instructions

- Optical Metallography

  involves the use of etchants (standard solutions containi ng a variety of chemicals su ch as strong acids and solv ents) which can be very cor rosive and poisonous.

# Sample Preparation Techniques

## **Safety**

- You must wear gloves and goggles and handle the chemicals with extreme care.
- **You must not have direct skin or eye contact with the etchants. Etchants must be used in well ventilated area.**
- **Do not try to smell the etchants.**