MSE 3922 Metallography

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Lecture 3. Metallographic Sample Preparation-2

Introduction to Metallography

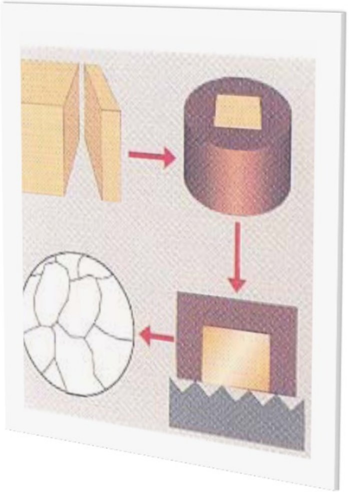
- Metallography is basically the study of the structures and constitution of metals and alloys, using metallurgical microscopes and magnifications, so that the physical and mechanical properties of an alloy can be related to its observed microstructure.
- • Can also be used to examine ceramics, polymers and semiconductors.
  - the size and shape of the grains (crystallites) and phases,
  - the presence of micro defects (such as segregation, hair cracks, and nonmetallic inclusions), and
  - the nature and distribution of secondary phases.

Metallographic Sample Preparation

- Proper preparation of metallographic specimens to determine microstructure and a rigid step-by-step process be followed. In sequence, the steps include course. Specimens must be kept clean and preparation procedure carefully followed in order to reveal accurate microstructures

1- sectioning (cutting),

2- mounting,



4- etching and microscopic examination

3- grinding, fine grinding, and polishing,

Metallographic Sample Preparation

- It consists of following operations.

1. **SECTIONING**: Cut a small piece from parent material by fracturing, hacksawing or abrasive cutting.
2. **MOUNTING**: If sample is small, first it is mounted before further operation. This can be done by Mechanical Mount, Compression Mount, or Cold Mount. In lab we will use Compression Mount.
3. **GRINDING/ROUGH POLISHING**: Grinding is perform to make surface smooth.
4. **POLISHING**: Polishing produces mirror like surface.
5. **ETCHING**: A dilute acid is react with the surface of the sample. This operation is called etching. After etching, the grain boundaries are visible in microscope.

Content

- **Metallography**
- **Metallographic Sample Preparation**
  - Sample Sectioning
  - Sample Cutting
  - Sample Mounting
  - **Sample Grinding/Rough Polishing**
  - Sample Polishing and Electro polishing
  - Etching
  - Observing
  - Quantitative analysis

Grinding

- Grinding is a most important operation in specimen preparation.
- During grinding the operator has the opportunity of minimizing mechanical surface damage that must be removed by subsequent polishing operations.
- Even if sectioning is done in a careless manner, resulting is severe surface damge, the damage can be elimenated by prolonged grinding.
- However, prolonged polishing will do little toward eliminating severe surface damage introduced by grinding.

Grinding

- Grinding is accomplished by abrading the specimen surface through a sequence of operations using progressively finer abrasive grit.

Table VI. Particle Size vs. Common

coarse abrasives / fine abrasives

GRIT NUMBER		
European (P-grade)	Standard grit	Median Diameter, (microns)
60	60	250
80	80	180
100	100	150
120	120	106
150	150	90
180	180	75
220	220	63
P240	240	58.5
P280		52.2
P320	280	46.2
P360	320	40.5
P400		35
P500	360	30.2
P600	400	25.75
P800		21.8
P1000	500	18.3
P1200	600	15.3
P2400	800	6.5
P4000	1200	2.5

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.

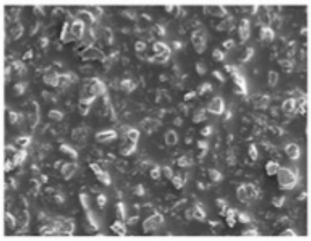
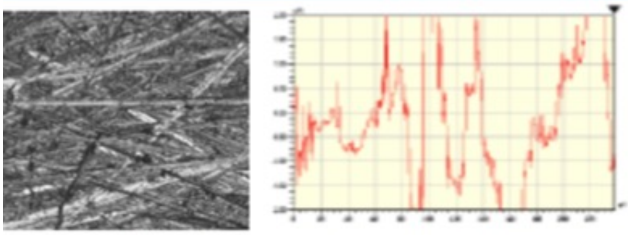
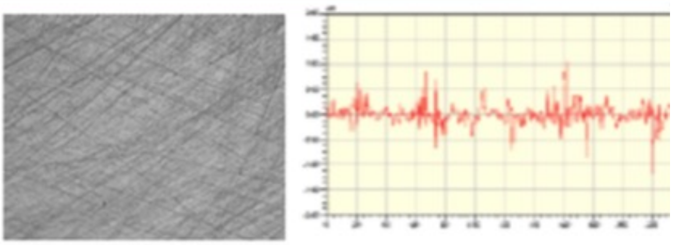


Figure 1. SEM micrograph of 600 grit SiC Abrasive Paper (original mag. 150x)

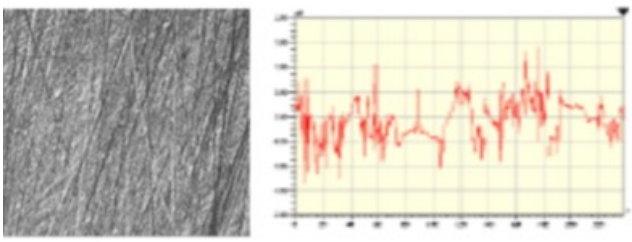
Grinding



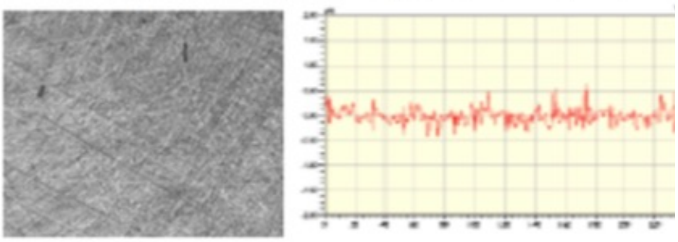
60 grit surface roughness micrograph and 2D line profile, 100X.



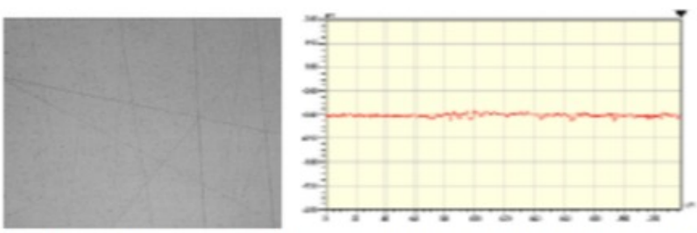
400 (P800) grit surface roughness micrograph and 2D line profile, 100X



240 (P220) grit surface roughness micrograph and 2D line profile, 100X.



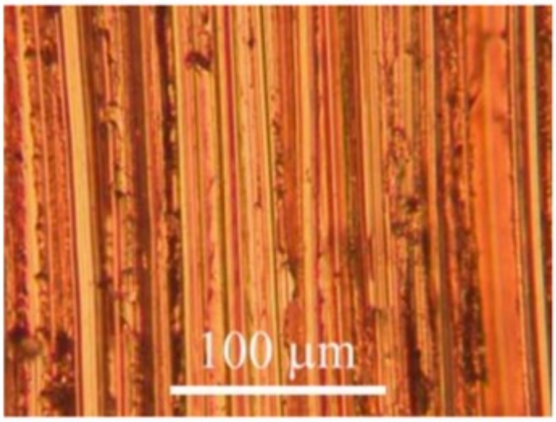
600 (P1200) grit surface roughness micrograph and 2D line profile, 100X



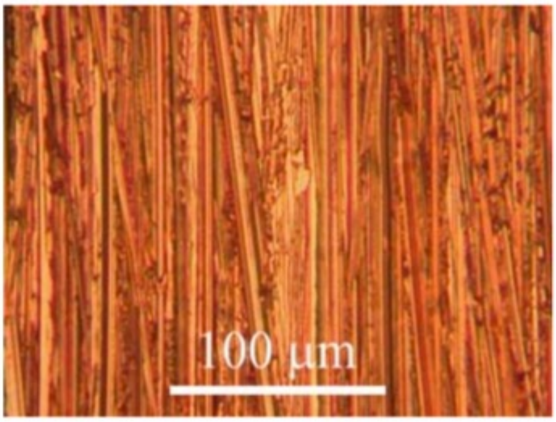
1200 (P4000) grit surface roughness micrograph and 2D line profile, 100X.

Grinding

Rough Grinding



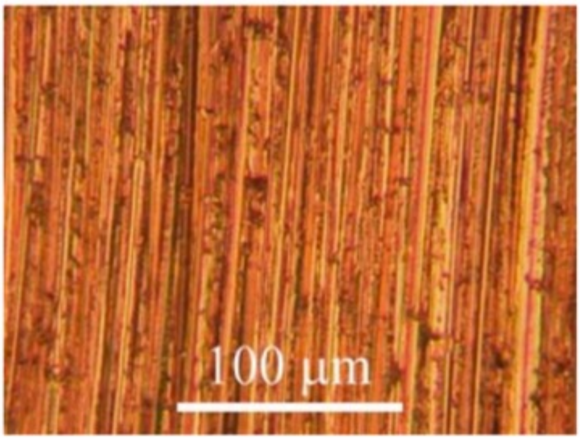
Copper specimen ground with 180 grit paper



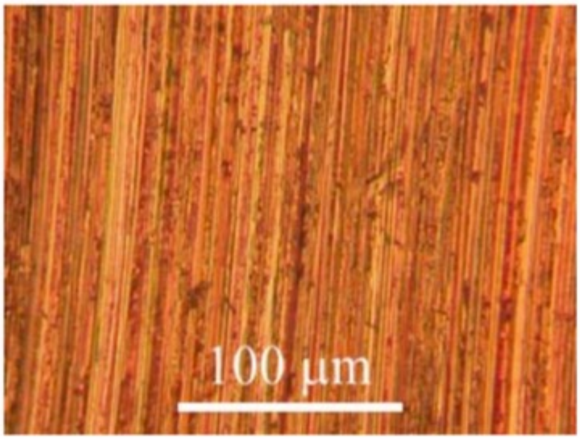
Copper specimen ground with 400 grit paper

Grinding

Rough Grinding



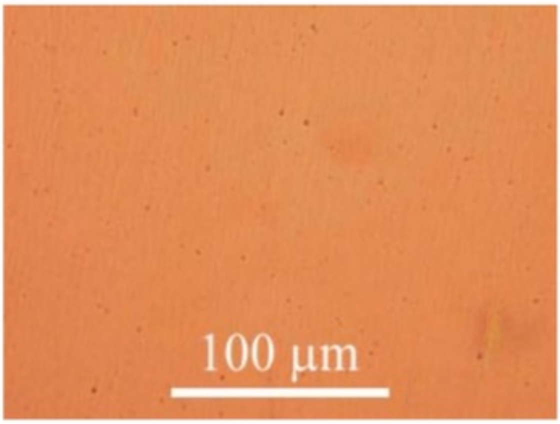
Copper specimen ground with 800 grit paper



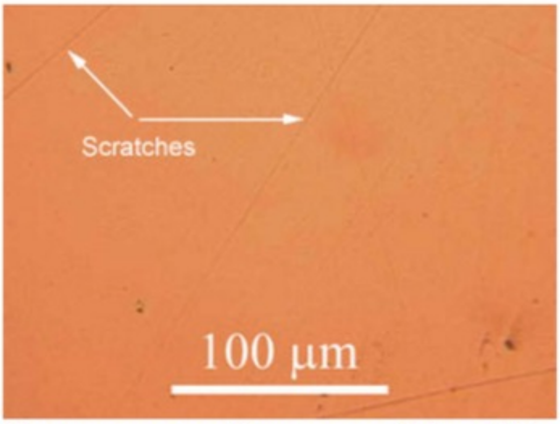
Copper specimen ground with 1200 grit paper

Grinding

Polishing



Copper specimen polished to 6 micron level



Copper specimen polished to 1 micron level. Ideally there should be no scatches after polishing, but it is often hard to completely remove them all.

Coarse Grinding

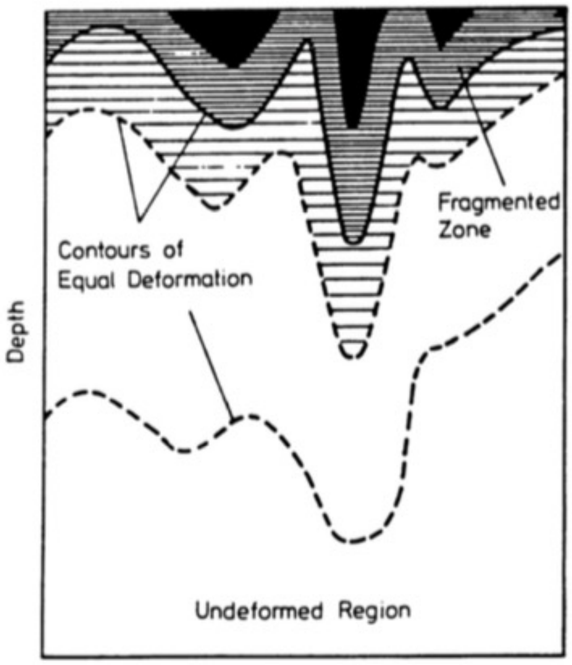
- Grinding can be achieved in a variety of ways, using a variety of abrasives. Fixed abrasive surfaces are available using diamond or cubic boron nitride (CBN) abrasives. The method used **to bind the abrasives to the wheel** affects the grinding characteristics, **the harder or more rigid the bonding medium, the more aggressive the grinding action of the surface. Therefore metal bonded fixed abrasive wheels are the most aggressive grinding surfaces, whereas resin bonded wheels are less aggressive.**
- Coarse grit Silicon Carbide or Alumina abrasives may be used, but the durability or characteristics of such materials may be inappropriate for certain materials. **Generally, in order to maintain sharp abrasive particles, grinding papers need frequent changing**.

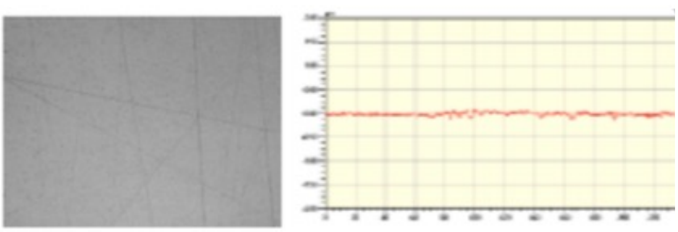
Grinding

- Grinding should commence with coarse grit size that will establish an initial flat surface and remove the effects of sectioning within a few minutes.
- An abrasive grit size 150 or 180 mesh is coarse enough to use on specimen surfaces sectioned by an abrasive cutoff wheels.
- Hacksawed, band sawed or other rough surfaces usually require abrasive grit sizes in the range 80 to 150 mesh.
- The abrasive used for each succeeding grinding operation should be one or two grit size smaller than that used in the proceeding operation.
- A satisfactory grinding sequence might involve grit sizes of 180, 240, 400 and 600 mesh.

Fine Grinding

- Grinding is accomplished by abrading the specimen surface through a sequence of operations using progressively finer abrasive grit.





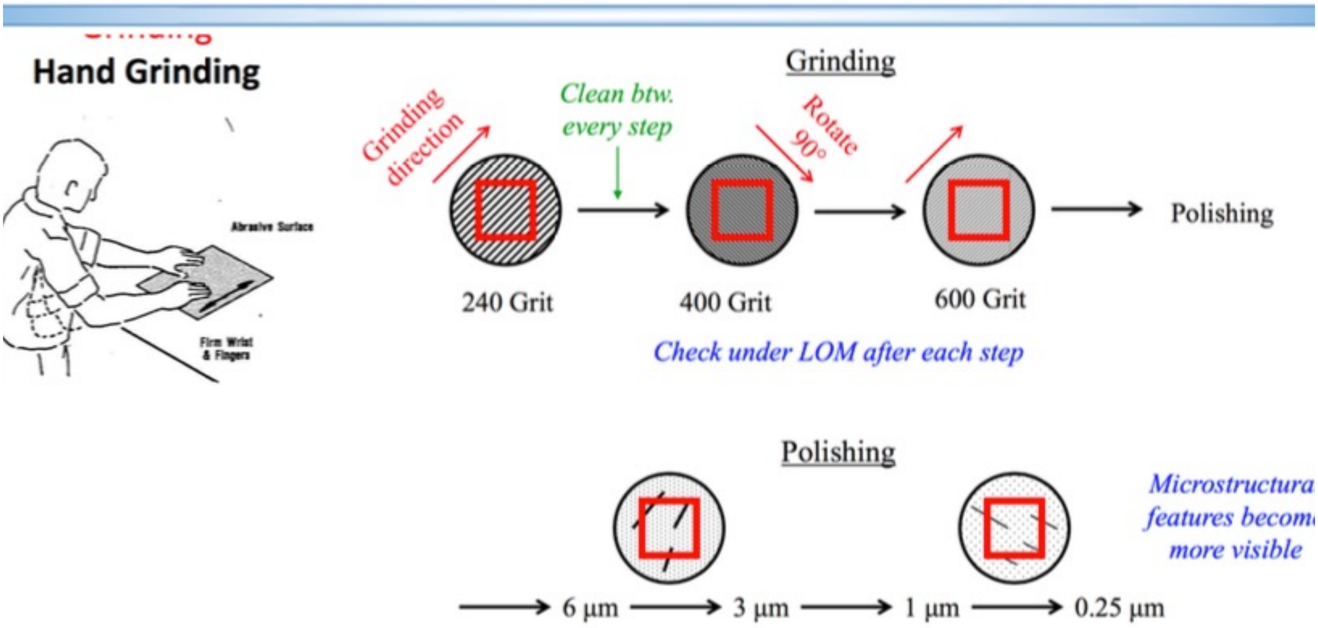
1200 (P4000) grit surface roughness micrograph and 2D line profile, 100

Grinding: Wet grinding

- Another advantage of the **wet** grinding is the cooling effect of the water.
- Considerable frictional heat can develop at the surface of a specimen during grinding and can cause alterations of the true microstructure.
- For example, tempering of martensite in steel - that cannot be removed during polishing.

- **Wet** grinding provides effective control of overheating. The abraded surface of a specimen may become embedded with loose abrasive particles during grinding. These particles may persist in the surface and appear to be nonmetallic inclusions in the polished specimen.
- The flushing action of the water removes many of loose particles that might otherwise become embedded.

Grinding-Hand Grinding



LOM: light optical microscopy

Grinding

Hand Grinding

A simple setup for hand grinding can be provided by a piece of **plate glass, or other material with hard, flat surface, on which an abrasive sheet rests.**

The specimen is held by hand against the abrasive sheet as the operator moves specimen in rhythmic style away from and toward him **in a straight line**.

Enough pressure should be applied on the forward stroke than on the return stroke to hold flat and on the paper.
The grinding can be done wet by sloping the plate glass surface toward the operator and providing a copious flow of water over the abrasive sheet.

Grinding-Planar Grinding

Hand Grinding

Planar Grinding - or course grinding is required to planarize the specimen and to reduce the damage created by sectioning.

The planar grinding step is accomplished by decreasing the abrasive grit/ particle size sequentially to obtain surface finishes that are ready for polishing.

Care must be taken to avoid being too abrasive in this step, and actually creating greater specimen damage than produced during cutting (this is especially true for very brittle materials such as silicon).

Grinding

Hand Grinding

Grinding Pressure - Grinding/polishing pressure is dependent upon the applied force (pounds or Newtons) and the area of the specimen and mounting material.

For specimens significantly harder than the mounting compound, pressure is better defined as the force divided by the specimen surface area.

Thus, for larger hard specimens higher grinding/polishing pressures increase stock removal rates, however higher pressure also increases the amount of surface and subsurface damage. Note for SiC grinding papers, as the abrasive grains dull and cut rates decrease, increasing grinding pressures can extend the life of the SiC paper.

Automatic Grinding Machine

Machines for grinding, land polishing for 200 or 230 mm discs.

Automatic

Automatic Grinding Machine

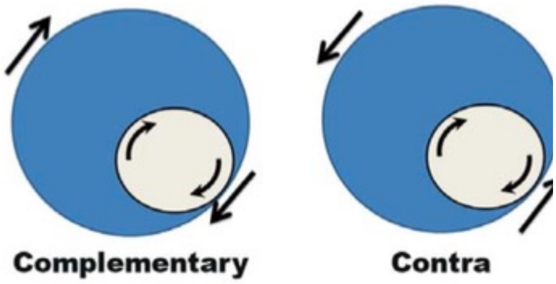
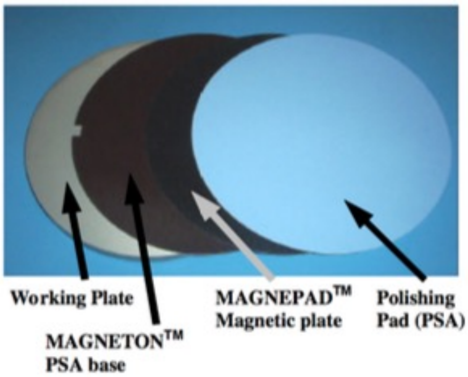
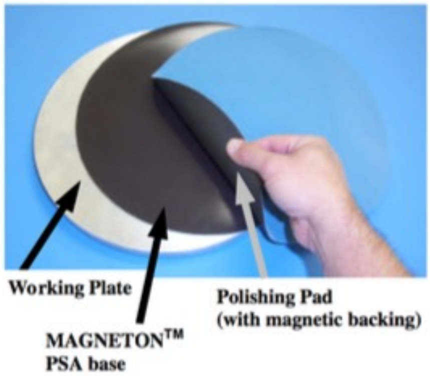


Figure 5-18 Complementary and contra disk on disk rotation.





Automatic Grinding Machine

The machine parameters which effect the preparation of metallographic specimens includes: grinding/polishing pressure, relative velocity distribution, and the direction of grinding/polishing.

Higher grinding/polishing pressures can also generate additional frictional heat which may actually be beneficial for the chemical mechanical polishing (CMP) of ceramics, minerals and composites. Likewise for extremely friable specimens such as nodular cast iron, higher pressures and lower relative velocity distributions can aid in retaining inclusions and secondary phases.

Relative Velocity - Current grinding/polishing machines are designed with the specimens mounted in a disk holder and machined on a disk platen surface. This disk on disk rotation allows for a variable velocity distribution depending upon the head speed relative to the base speed.

Automatic Grinding Machine

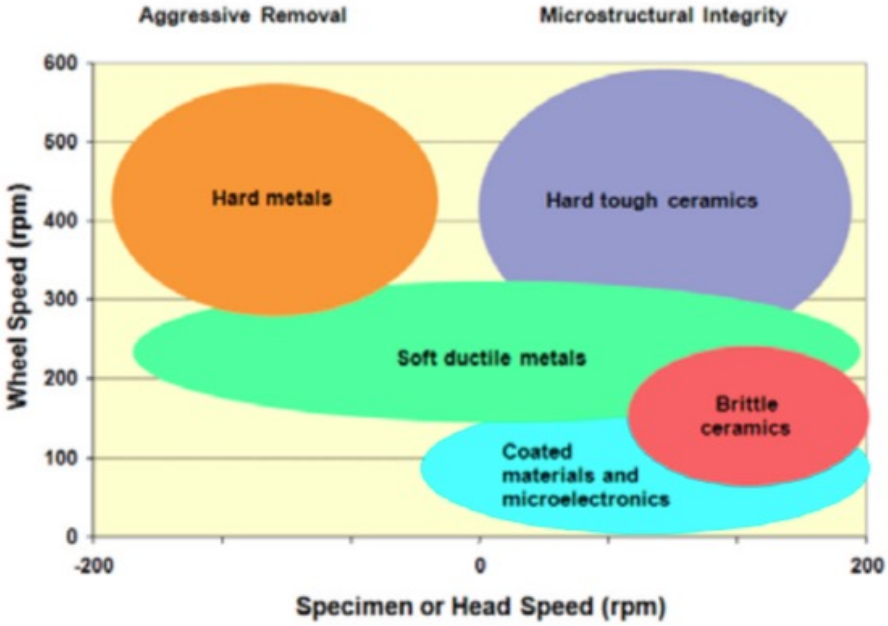


Figure 5-21a Relative velocity distribution guidelines for planar grinding of various materials using Central Pressure Machines.

Automatic Grinding Machine

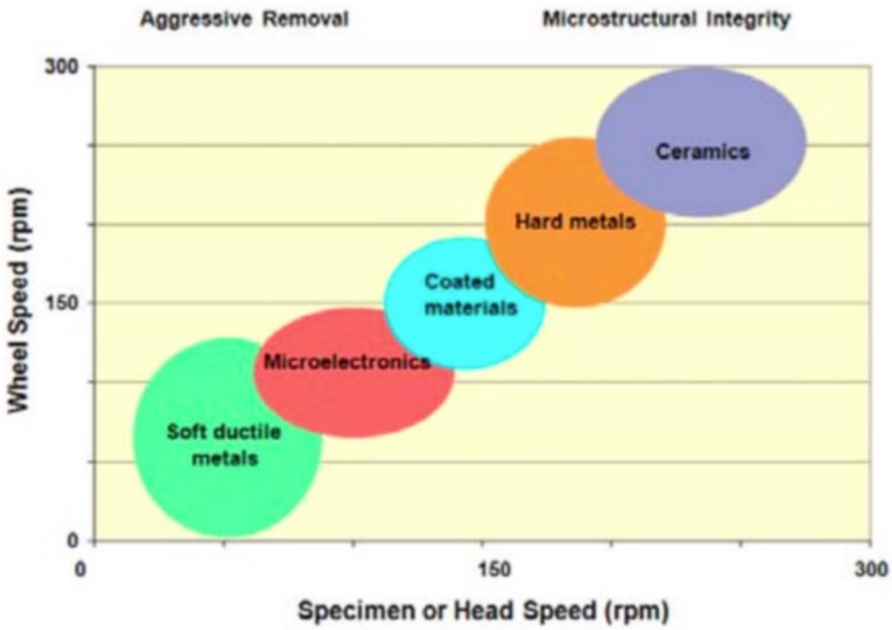


Figure 5-21b Relative velocity distribution guidelines for planar grinding of various materials using Individual Pressure Machines.

Grinding

Head Speed (rpm)	Base Speed (rpm)	Relative Velocity Distribution	Characteristic	Application
150	300 to 600	High	• Aggressive stock removal • Differential grinding across the specimen surface	• Useful for gross removal on hard specimens
150	150	Minimal	• Matching head and base speed in the same direction eliminates relative velocity distributions • Uniform stock removal • Low stock removal • Produces minimal damage	• Provides superior flatness over the specimen • Useful for retaining inclusions and brittle phases

Polishing

- **Polishing is the final step in production a surface that is flat, scratch free, and mirror like in appearance.**
- Such a surface is necessary for subsequent accurate metallographic interpretation, both qualitative and quantitative.
- The **polishing technique used should not introduce extraneous structure such as disturbed metal, pitting, dragging out of inclusions, comet tails and staining**.
- Before final polishing is started, the surface condition should be at least as good that obtained by grinding with a 600-grit abrasive.

Polishing

- Colloidal Silica is a chemo-mechanical polish, i.e., it combines the effect of **mechanical polishing with etching.**
- This type of stock removal is ideal in many cases, as a damage free surface can be obtained with little effort. **Typical abrasive size is 0.05 micron**.

Note: Colloidal Silica crystallizes readily and will ruin polishing cloths if left to dry.

Further, a film can form on the polished surface of the sample which must be removed.

A convenient method to achieve this is to flush the polishing wheel with water during the last few seconds of polishing to clean the sample surface.

Polishing

- Remove and dry the sample in the usual manner, using a solvent with low water content and not so volatile as to cause water condensation on the surface.
- Alcohol is **ideal**, whereas acetone is **not**. Flush the polishing wheel with water until all traces of colloidal silica is washed away, spin to drain and store in a suitable container such that contamination of the wheel cannot occur.
- Meticulous attention to avoiding contamination of wheels is an important aspect to achieve the best results

Sample Preparation: Mechanical Polishing

- Hand Polishing
- Specimen Movement
- Polishing Pressure
- Washing and Drying
- Cleanness
- Automatic Polishing
- Polishing Cloths
- Polishing Abrasives

Sample Preparation: Mechanical Polishing

- The term mechanical polishing is frequently used to describe the various final polishing procedures involving the use of cloth-covered laps and suitable polishing abrasives.

- The laps have either **a rotating** or **a vibrating motion, and the specimen are held by hand, held mechanically, or merely confined within the polishing area.**

Sample Preparation: Mechanical Polishing

Polishing be done in a relatively dust-free area, preferably removed from the area for sectioning, mounting and rough grinding.

Any contamination of a polishing lap by abrasive particles carried over from a preceding operation or by dust, dirt or other foreign matter in the air cannot be tolerated.

Carryover as a result of improper cleaning between final polishing steps is another prime source of contamination. It is just as important for the operator to wash his hand meticulously as is for him to remove all traces of polishing abrasive from the specimen before proceeding to the next polishing operation.

Sample Preparation: Mechanical Polishing

The specimen can be cleaned ultrasonically or by careful washing under running water and swabbing with cotton. In automatic equipment in which holding fixtures for the specimens are also transferred through successive polishing steps, proper cleaning of the assembly can be accomplished only by using an ultrasonic cleaner.

If a polishing lap becomes contaminated, it is virtually impossible to remove all of the contaminant by washing the polishing cloth.

Instead, the operator should replace the cloth and use fresh polishing solution. Cleanness cannot be overemphasized. **It takes only one particle of grit on a final polishing lap to ruin all prior preparation.**

Sample Preparation: Hand Polishing

• Aside from the use of improved polishing cloths and abrasives, hand polishing techniques still follow the basic practice established years ago:

1- Specimen Movement

The specimen is held with one or both hands, depending on the operator's preference, and is **rotated in a direction counter to the rotation of the polishing wheel.**

In addition, the specimen is continually **moved back and forth between the center and the edge of the wheel, thereby ensuring even distribution of the abrasive and uniform wear of the polishing cloth.**

The main reason for rotating the specimen is to prevent the formation **of "comet tails".**

Sample Preparation: Hand Polishing

• Aside from the use of improved polishing cloths and abrasives, hand polishing techniques still follow the basic practice established years ago:

2-Polishing Pressure

The correct amount of applied pressure must be determined by experience; in general, **firm hand pressure is applied to the specimen in the initial polishing step and is proportionally decreased with successively finer polishing steps.**

For very soft materials, pressure other than **that from the weight of the specimen** itself may be eliminated entirely in the last polishing operation.

Sample Preparation: HandPolishing

• Aside from the use of improved polishing cloths and abrasives, hand polishing techniques still follow the basic practice established years ago:

3- Washing and Drying

The specimen is preferably **washed and swabbed** in worm running water, rinsed with **methanol or any other alcohol** that **does not leave a residue, and dried in a stream of warm air.**

Alcohol can usually be employed for washing when **the abrasive carrier is not soluble in water.**

4 Cleanness

The precautions for cleanness, as previously mentioned, must be strickly followed.

Sample Preparation: Automatic Polishing

- High quality preparation of most metallographic specimens often can be expedited by the use of automatic polishers.
- Automatic polishing equipment usually allows the preparation of several specimens simultaneously.
- Some methods of specimen preparation can be **done only with automatic polishers**, such as remote polishing of radioactive materials, chemical-mechanical polishing, and polishing in special atmospheres.
- There is no ideal automatic polisher; each has its merits and shortcomings and each metallographer must determine which is best for his particular requirements.

Sample Preparation: Polishing Cloths

The requirements of any good polishing cloth include **the ability to hold an abrasive, long life, absence of any foreign material** that may cause scratches, and **absence of any processing chemicals** that may react with the specimen

A cloth without nap or with a very low nap is preferred for the preliminary or rough polishing operation. The absence of nap ensures maximum contact with the polishing abrasive, and results in fast cutting with minimum of relief.

The cloths most frequently used are **canvas, low-nap, cotton, nylon, silk and Pelon**. These cloths are stretched tight on the laps and fastened securely, usually by a band-type clamp. Some cloths are available with a contact adhesive on the back, which greatly simplifies installation on the wheel.

Sample Preparation: Polishing Cloths

- After installation, the cloths are charged with the appropriate abrasive (**usually in sizes from 15 microns down to 1 microns**) and carrier.
- Rough polishing is usually done with the laps rotating at **500 to 600 rpm.**
- Cloths with a medium or high nap are ordinarily used on slow rotating laps (less than 300 rpm) for intermediate and final polishing.
- **Felt or billiard cloths** (100% virgin wool), used with 0.3 micron aluminum oxide or other comparable abrasive, are excellent for **intermediate polishing of soft metals (most nonferrous alloys and low carbon steels)** and **final polishing of hard materials (such as hardened alloy steels).**

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Sample Preparation: Polishing Cloths

- **One of the most popular cloths for final polishing of most metals** is **composed of densely packed, vertically aligned, synthetic fibers bonded to a suitable backing.**
- For some metals or for particular types of polishing, other cloths, such as velvets, satins, cashmeres or cottons, may be required.

- **The ability to select the proper combination of**
  - **cloth,**
  - **abrasive,**
  - **carrier,.**
  - **polishing speed (rotational speed of the polishing wheel),**
  - **pressure applied**

Sample Preparation: Polishing Abrasives

- **Polishing usually involves** the use one or more of **five types of abrasive:**
  - **aluminum oxide (Al_2O_3),**
  - **magnesium oxide (MgO),**
  - **chromic oxide (Cr_2O_3),**
  - **iron oxide (Fe_2O_3), and**
  - **diamond compound**.
- With the exception of diamond compound these abrasives are normally used in **a distilled water suspension**
- if the metal to be polished is **not compatible with water,** other suspensions, such as **ethylene glycol, alcohol, kerosine or glycerin**, may be required.
- The diamond compounds should be extended only with the carrier recommended by the manufacturer.

Sample Preparation: Polishing Abresives

Diamond polishing compounds are becoming increasingly popular for preparing metallographic specimen.

Diamond is the only substance **hard enough and with good enough cutting qualities** to be used for mechanical polishing of materials such as boron carbide and sintered tungsten.

Specimens that have both hard and soft constituents, such as graphite in cast iron and silicon in aluminium, can be polished without causing relief, with diamond compounds on an appropriate lap.

These polishing compounds are available either in water soluble and oi soluble carriers or in the form of dry diamond powder in particle size down to 0.25 microns.

Sample Preparation: Rough Polishing

The purpose of the rough polishing step is to remove the damage produced during cutting and planar grinding.

Proper rough polishing will maintain specimen flatness and retain all inclusions or secondary phases.

By eliminating the previous damage and maintaining the microstructural integrity of the specimen at this step, a minimal amount of time should be required to remove the cosmetic damage at the final polishing step.

Rough polishing is accomplished primarily with diamond abrasives ranging from 9 micron down to 1 micron diamond.

Polycrystalline diamond because of its multiple and small cutting edges, produces high cut rates with minimal surface damage,

Sample Preparation: Final Polishing

The purpose of final polishing is to remove only surface damage. It should not be used to remove any damage remaining from cutting and planar grinding. If the damage from these steps is not complete, the rough polishing step should be repeated or continued.

Thank you for attention