

Objectives

1. To learn and to gain experience in the preparation of metallographic specimens.
2. To examine and analyze the microstructures of metals and metallic alloys.

Abstract

Proper preparation of metallographic specimens to determine microstructure and content requires that a rigid step-by-step process be followed. In sequence, the steps include sectioning, mounting, course grinding, fine grinding, polishing, etching and microscopic examination. Specimens must be kept clean and preparation procedure carefully followed in order to reveal accurate microstructures. Each student will prepare and examine a brass, steel or aluminium sample for metallographic examination. Additionally, a brass, steel or aluminium sample with different composition, a eutectoid steel sample and a eutectoid Pb-Sn sample will be examined under the metallographic microscope. Photographs will be taken of the samples and the ASTM grain size number determined for each of the specimens if applicable.

Background

Metallography consists of the study of the constitution and structure of metals and alloys. Much can be learned through specimen examination with the naked eye, but more refined techniques require magnification and preparation of the material's surface. Optical microscopy is sufficient for general purpose examination; advanced examination and research laboratories often contain electron microscopes (SEM and TEM), x-ray and electron diffractometers and possibly other scanning devices.

Incorrect techniques in preparing a sample may result in altering the true microstructure and will most likely lead to erroneous conclusions. It necessarily follows that the microstructure should not be altered. Hot or cold working can occur during the specimen preparation process if the metallurgist is not careful. Expertise at the methods employed to produce high-quality metallographic samples requires training and practice. The basic techniques can be learned through patient persistence in a matter of hours. This module takes the student through the metallographic sample preparation process step-by-step with demonstrations and explanations of sectioning, mounting, course & fine grinding, polishing, etching and microscopic examination.

Sectioning

Operations such as shearing produce severe cold work, which can alter the microstructure of a sample. Abrasive cutting (sectioning) offers the best solution to eliminate these undesirable features; the resultant surface is smooth, and the sectioning task is quickly accomplished. Low-speed cut-off wheels are utilized in cases where the heat created by standard abrasive cutters must be avoided. Ample coolant and proper speed control are essential in all sectioning operations.

Abrasive Cut-Off Wheels consist of abrasive grains (such as Aluminum Oxide or Silicon Carbide), bonded together with rubber or other materials in the form of a thin wheel. Rubber bonded wheels are most extensively used for wet operation and resin bonds for dry cutting. When sectioning a piece of metal, the abrasive grains on the cutoff wheel become dull, therefore, the bond must be destroyed at the correct rate so that new grains are always at the edge and are still held firmly by the bonding material. The rate at which the bond breaks down is affected by several different factors, including:

- 1) The hardness of the bond.
- 2) The hardness and workability of the metal sample.
- 3) The size and speed of the wheel.
- 4) The power of the driving motor.
- 5) The type and amount of coolant and its method of application.
- 6) The amount of pressure by which the wheel is applied to the sample.
- 7) The amount of vibration in the machine.

Cutoff wheels are indexed from hard to soft and are referred to by "grade". As a rule of thumb, a hard bonded wheel should be selected when sectioning soft stock, and a soft wheel for sectioning harder material. A reasonable amount of wheel pressure must be applied so the abrasive grains will "bite" and remove the metal

in fine chips. An experienced operator can tell whether the wheel is cutting or merely "dragging" by the feel of the control arm and monitoring of the power consumption of the cutting equipment.

Proper cutting procedures must be followed; abrasive cut-off wheels will crack and become flying projectiles when used improperly.

Lab Procedure - Sectioning

1. Place the sample on the slotted Table so that it is at the center of the swing of the head.
2. Place the Locking Lever in the forward position to allow the Control Rod to be moved to the furthest position. Move the Rear Vise section to contact the rear face of the sample. Move the Front Section to a position near the front face of the sample.
3. Tighten the Nuts that secure the Vise sections to the Table.
4. Push the Control Rod in to make firm contact with the sample and pull back the Locking Level to secure the sample.
5. Turn on the coolant; it should flow readily to both cool and clean the cutting area.
6. Apply steady moderate pressure until the specimen is sectioned; avoid jerking motions that may cause the blade to splinter.

Mounting

Small samples are generally mounted in plastic for convenience in handling and to protect the edges of the specimen being prepared. Compression-type molding is commonly applied to encase specimens in 1 to 1.5 inch diameter plugs of a hard polymer. Compression molding materials are classified as either thermosetting or thermoplastic:

- 1) Bakelite is a low cost, relatively hard thermosetting polymer that is commonly utilized.
- 2) Expensive Transoptic Thermoplastics are utilized when transparency is required.

Transoptic materials remain molten at maximum temperature and become transparent with an increase in pressure and a decrease in temperature. By definition, thermosetting materials require heat and pressure during the molding cycle. During mounting, the pressure and molding temperature are generally held constant thus time is the only variable. The metal sample is placed in the mounting cylinder which is then encased in a pre-measured amount of powdered polymers. The mounting cylinder is then sealed, pressurized and heated to complete the polymerization process necessary to solidly encase the metal sample.

Although inferior in quality to compression-type molding, cold molding (room temperature) is often used with epoxy to mount samples by simply mixing the epoxy and pouring it over a sample that is positioned face-down in a cold-mounting ring. When the epoxy cures the specimen can be prepared. Caution must be exercised when cold mounting due to relatively poor adhesion between the specimen edges and the epoxy plug; gaps often form which can degrade the quality of the specimen.

Lab Procedure Mounting Procedure

(Hot-Mounting Equipment Currently Unavailable @ LSU!)

Supplies for Cold-Mounting:

- KoldMound Kit (Vernon-Benshoff Co.): Mix 2 parts powder with 1 part liquid
- Plastic Mounting Ring with Removable Bottom
- 8 Ounce Paper Cup with Stirrer
- Weight Scale
- Liquid Soap

1. Apply liquid soap to the inner walls and base of the mounting ring.
2. Place the specimen in the center/bottom of the plastic mounting ring base with the examination surface face-down.
3. Attach the mounting ring cylinder to the base by simple pressing firmly in place.

4. Mix the appropriate amount of KoldMount (2/1 Powder/Liquid - 15 grams of Powder/7.5 grams of Liquid per Sample) in a paper cup.
5. Simply pour the epoxy mixture into the mounting ring; sample should be approximately 0.75" thick.
6. After one hour, remove the sample from the mounting ring and proceed with course grinding.

Coarse Grinding

In view of the perfection required in an ideally prepared metallographic sample, it is essential that each preparation stage be carefully performed. The specimen must:

1. Be free from scratches, stains and others imperfections which tend to mark the surface.
2. Retain non-metallic inclusions.
3. Reveal no evidence of chipping due to brittle intermetallic compounds and phases.
4. Be free from all traces of disturbed metal.

The purpose of the coarse grinding stage is to generate the initial flat surface necessary for the subsequent grinding and polishing steps. As a result of sectioning and grinding, the material may get cold worked to a considerable depth with a resultant transition zone of deformed material between the surface and the undistorted metal. Course grinding can be accomplished either wet or dry using 80 to 180 grit electrically powered disks or belts, but care must be taken to avoid significant heating of the sample. The final objective is to obtain a flat surface free from all previous tool marks and cold working due to specimen cutting.

An important factor throughout the Coarse Grinding and Fine Grinding Stages is that the scratches be uniform in size and parallel to each other in any one grinding stage. Proper grinding involves rotation of the sample by 90° between stages while the grinding angle must be held constant during the grinding at any one stage.

The sample MUST be washed thoroughly before proceeding from one grinding stage to the next !!!

Failure to follow this basic rule will result in transferring abrasive particles between stages and will cause time-consuming, frustrating problems in removing unwanted scratches.

Lab Procedure-Coarse Grinding:

***Note: Safety Glasses must be worn when operating the Course Grinding Equipment !!!**

1. Label the specimen with your name so that it can be easily identified.
2. Turn the Motor On, then the water, adjust the flow to obtain a good film of water. Too much water will cause a spray when it contacts the sample.
3. Beginning with the 120 grit belt and using both hands to hold the specimen, carefully place the sample face onto the exposed area of the belt being careful not to contact the rotating surface with a sharp edge of the specimen or your hand.
4. Applying moderate pressure evenly, move the sample left-and-right across the belt surface to obtain uniform grinding. Use both hands to hold the specimen; unsecured specimens can "Catch an Edge and FLY"!
5. Lift the sample from the wheel periodically to determine the progress of grinding but do not rotate the sample. The 120 grit stage is complete when all the lines scratched in by the grinder are parallel on the specimen surface. If any line or scratch is not in the same direction as the other lines, continue grinding until all of the lines are parallel.
6. When all of the sample's scratches are parallel, carefully wash all of the debris from sample using tap water and dry the specimen immediately using a paper towel or pressurized air to avoid corrosion.

7. Proceed to the 180 grit stage with the scratches oriented approximately perpendicular to the intended grinding direction and repeat steps 3 through 6.
8. When the 180 grit stage is complete, you're ready to move on to the medium/fine grinding station.

Medium and Fine Grinding

Medium and Fine Grinding of metallurgical samples are closely allied with the Coarse Grinding which precedes them. Each stage of metallographic sample preparation must be carefully performed; the entire process is designed to produce a scratch free surface by employing a series of successively finer abrasives. Failure to be careful in any stage will result in an unsatisfactory sample.

The idea is to carefully move from one stage to the next where the abrasives become finer at each successive stage. Movement from one stage to the next should only proceed when all of the scratches from the preceding stage are completely removed. In general, successive steps are 240, 320, 400 and 600 grit SiC and the grinding rate should steadily decrease from one stage to the next. Proper grinding involves the rotation of the sample between stages while the grinding angle must be held constant during the grinding at any on stage. Recall that

"The sample MUST be washed thoroughly before proceeding from one grinding operation to the next!"

Wet grinding is generally applied in medium and fine grinding to avoid possible side effects due to heating such as tempering, transformation, aging, incipient melting, etc. Wet grinding also provides a flushing action for loose particles and keeps sharp edges of the grinding medium exposed at all times.

Repeated for Emphasis: ***Proper grinding involves the rotation of the sample between stages while the grinding angle must be held constant during the grinding at any on stage.***

Lab Procedure-Fine Grinding

"The sample MUST be washed thoroughly before proceeding from one fine grinding stage to the next!!!"

1. Manual Fine Grinding is performed by drawing the specimens in one direction across the surface of the water lubricated abrasive paper. (Back to front is recommended) Use of backward and forward motion is less desirable because there is a tendency to rock the sample, producing a curved rather than a flat surface.
2. Begin with the lowest grade abrasive paper (240 grit) and proceed to the highest (600 grit).
3. To monitor progress, each fine grinding step should be performed in a direction off-angle with respect to the previous step.
4. Fine Grinding should be continued until the previous stage's scratches are gone, using a few extra strokes to assure complete scratch removal.
5. Sufficient water must be applied to provide lubrication and flush away the removal products. Too much water will result in a hydroplaning action where the sample rides on a film of water, thereby reducing the effectiveness of the abrasive. Use the valve at the top of the roll-grinder to increase and decrease the quantity of water needed or desired.
6. The specimen should be carefully rinsed after each step of Fine Grinding; debris from one step must not contaminate the next step! Rinse the specimen very thoroughly before proceeding to polishing.

Mechanical Polishing

Polishing involves the use of abrasives, suspended in a water solution, on a cloth-covered electrically powered wheel. Diamond abrasives provide the best, and most expensive, compounds utilized in polishing; standard sized aluminum oxide powders are applied for general use purposes. Following the final 600 grit fine-grinding stage, the sample MUST be washed and carefully dried before proceeding to the first polishing stage! At the polishing stages, even hard dust particles in the air which settles on the polishing cloth can cause unwanted scratching of the specimen! Careful washing of the specimen and the operator's hands must be carried out prior to each stage of polishing! Beginning with 25-micron suspended aluminum oxide particles (suspended in water) on a Nylon-cloth, the final fine-grinding surface layer resulting from the previous grinding procedure should be completely removed with a rotation rate of 150-200 rpm.

The specimen is initially held at one position on the wheel, without rotation, until most of the previous grinding marks are removed. It can be rotated slowly, counter to the wheel rotation, until only scratches from the 25-micron aluminum oxide are visible. During the initial polishing stage, moderate pressure can be applied to the specimen and the entire stage should generally take 1 or 2 minutes!

After carefully washing the specimen, proceed to the 5-micron stage where a separate polishing wheel with 5-micron particles; repeat the procedure outlined above using lighter pressure and a gradual rotating motion across the polishing cloth.

The final polishing stage with 1-micron suspended aluminum oxide particles should be carried out on a separate polishing wheel following "Careful washing of the specimen and operator's hands" at a slower speed of 100 - 150 rpm using a napped cloth. After 1 or 2 minutes a properly polished specimen should have a mirror-like surface free of scratches! During final polishing, minimal pressure should be applied and time should be kept to a minimum since the napped material will conform to the specimen shape under pressure. The wetness of the cloth used for Final Polishing has a great influence on the end result. If the cloth is too wet the sample will show pits; if too dry, buffing and/or smearing will result. To determine the proper wetness, the sample should be removed from the wheel and the time required for the polishing film to dry (five to eight seconds) should be checked. A thin opaque film indicates that sufficient abrasive is present.

For precision work, extremely fine grades of diamond abrasives may often be used for the final polishing sequence. Cerium Oxide is especially excellent for the final polishing of aluminum and other soft metals and alloys. The best abrasive types and sizes for various metals and alloys are listed in the literature; references should be consulted for recommended practice.

Generally, polishing can also be accomplished electrochemically, using an electrolytic cell which is similar to the ones used for electroplating, except that the polishing cell operates in reverse. The metal atoms are thus stripped from the specimen surface. Careful control is required for electropolishing; it is best suited to finishing a large number of identical specimens.

The following general flow sheet is included for the various stages of steel sample preparation.

Stage	Abrasive	Lubricant	Cloth
Fine Grinding	240, 320, 400 then 600	Water	None
Rough Polishing	5 to 25-Micron Aluminum Oxide	Distilled Water	Nylon
Final Polishing	1-Micron Aluminum Oxide	Distilled Water	Napped Microcloth

The ability to prepare a specimen is an art and the required skill is acquired only after significant hands-on experience. Using automated equipment, an inexperienced technician can be taught to prepare well polished specimens in a few hours; he/she could not, however, hand polish well without further training and experience.

Lab Procedure - Mechanical Polishing

1. Safety goggles must be worn when using the Polishers!
2. Make sure your specimen and hands have been thoroughly cleaned before Polishing!
3. Begin with the 25-micron, nylon cloth polishing station!
4. Turn the water on adjusting to less than one drop per second!
5. Apply a small amount of the aluminum oxide abrasive solution to the polishing cloth.
6. When polishing the specimen, hold it with both hands, apply a moderate amount of pressure, and don't let it go. The Rough Polishing Stages (5-25 microns) should take between 1 and 2 minutes each! If you let go of the specimen it may fly, harm you or others in the laboratory and become damaged forcing you to start over again with coarse grinding - hold it tight and be careful!
7. Do not contaminate the polishing wheel; cover the wheel when it's not in use!
8. Before proceeding to the next polishing stage, wash and dry both the specimen and your hands thoroughly then rinse the specimen.
9. Repeat steps 4 through 8 for the 5-micron stage.
10. Proceed to the Final Polishing Station (1 micron) when all of the 25 micron marks are removed at the 5 micron stage.
11. Hold the specimen with both hands and apply a SMALL amount of pressure, and Don't Let Go!
12. The Final Polishing Stage (1-micron) should take between 1/2 and 1 minute!
13. Be very careful that you do not contaminate the polishing wheel! Cover the wheel when it's not in use!!!
14. Before proceeding to Etching, wash and dry both the specimen and your hands thoroughly then rinse the specimen with distilled water. DO NOT TOUCH THE SPECIMEN SURFACE!!!

Etching

Microscopic examination of a properly polished, unetched specimen will reveal only a few structural features such as inclusions and cracks or other physical imperfections. Etching is used to highlight, and sometimes identify, microstructural features or phases present. Even in a carefully prepared sample, a surface layer of disturbed metal, resulting from the final polishing stage, is always present and must be removed. Etchants are usually dilute acid or dilute alkalis in a water, alcohol or some other solvent. Etching occurs when the acid or base is placed on the specimen surface because of the difference in rate of attack of the various phases present and their orientation. The etching process is usually accomplished by merely applying the appropriate solution to the specimen surface for several seconds to several minutes.

Nital, a Nitric Acid - Alcohol mixture, is the etchant commonly utilized with common irons and steels. Nital is dripped onto the specimen using an eye-dropper or cotton swab. Ten seconds to one minute is usually sufficient for proper etching depending on sample and nital concentration. The sample is immediately washed under running water, rinsed with alcohol and dried in an air blast. Do not touch, wipe or swab the specimen following etching; dry off the rinsing alcohol on the specimen with the air blast and then move on to the microscopic examination stage!

Etchants

Materials	Composition	Application Procedure
Iron & Steel	1-5 Parts Nitric Acid 100 Parts Alcohol	Immerse/Swab
Copper & Brass	1 Part Ammonium Hydroxide 1 Part 3% Hydrogen Peroxide 1 Part Water	Swab
	5 g Ferric Chloride, 10 ml Hydrochloric Acid 100 ml Water	Immerse
Aluminum	5-10 g Ammonium Persulphate 1 ml Hydrofluoric Acid 99 ml Water	Immerse
	10 g Sodium Hydroxide, 100 ml Water	Immerse
Stainless Steels	10 g Oxalic Acid 100 ml Water	Use Electrolytically
	5 ml Sulfuric Acid 100 ml Water	Use Electrolytically

Lab Procedure- Etching Steel Specimens

Note: Always wear Acid Resistant gloves when Etching!

1. Place the specimen on the table under the Fume Hood with the polished surface up.
2. Turn on the Fume Hood.
3. Without touching the specimen surface, clean the surface with alcohol and let it dry using the hot air gun. Do not let anything but the alcohol touch the specimen surface!
4. Using the Eye-Dropper, apply a few drops of Etchant to the specimen surface covering the entire metallic surface of the specimen.
5. After about 20 to 30 seconds, rinse the Etchant into the sink with water and quickly rinse the specimen with alcohol, but do not touch the surface!
6. Use the Hot Air Gun to dry the sample.
7. Proceed to Microscopic Examination; if further etching is required you may return and proceed through steps 1 through 6 varying the time in step 5 depending on the results.
8. If the specimen has many scratches and marks or the microstructure cannot be seen after several etches, return to fine grinding and go back through the necessary steps.

Microscopic Examination

Initial microscopic viewing should be done utilizing a stereomicroscope, which reveals a three-dimensional scanning of the specimen surface. The specimen is placed on the stage of the microscope so that its surface is perpendicular to the optical axis.

Detailed viewing is done with a Metallurgical Microscope. A metallurgical microscope has a system of lenses (objectives and eyepiece) so that different magnifications (25X to 1000X) can be achieved. The important characteristics of the microscope are: (1) magnification, (2) resolution and (3) flatness of field. The resultant magnification is the product of the magnifying power of the objective and that of the ocular.

Scanning Electron Microscopes (SEMs) are capable of magnifications up to 20,000X and Transmission Electron Microscopes (TEMs) are utilized to view at magnifications up to 100,000X for highly detailed microstructural study.

Grain Size Calculation

In single phase specimens, the ASTM grain size of the metal can be estimated by comparing the image at 100X with standard microstructure examples corresponding to standard grain sizes from 1 to 10. Also, suitable eyepieces etched with a square 0.01" x 0.01" in size can be used to calculate the number of grains per square inch, N.

The ASTM grain size number, n , can be calculated using the following relationship:

$$N (M/100)^2 = 2^{(n - 1)}$$

N = number of grains per square inch at 100X

n = ASTM grain size number

M = Magnification

For single phase materials, ASTM grain size number is given to denote the grain sizes. These are not the actual grain size values, but the latter can be derived from the ASTM grain size number, n ; the larger the grain size number, the smaller the grains.

If there are N grains per square inch at a magnification M then there are $(N)^{1/2}$ grains along a 1 inch length. The size of each grain at magnification M is then $1/(N)^{1/2}$ inches.

The actual size of the grain is given by Actual **Grain Size** = $1/(N M)^{1/2}$

Lab Procedure- Microscopic Examination

1. Each student will examine the following four samples in the microscope:
 - A metallic specimen provided by the instructor and prepared by the student;
 - A metallic specimen provided by the instructor and prepared by another student;
 - A Eutectoid Steel Specimen (provided by instructor); and
 - A Eutectic Pb-Sn Specimen (provided by instructor).

Initially examine the specimens through the stereomicroscope, which gives a three-dimensional impression of the specimen surface. With a properly etched specimen, grains can be differentiated and impurity particles can be clearly seen.

2. Use a Metallurgical Microscope to view the specimen at various magnifications and note the microstructural aspects of the material.
3. Take a photograph of each specimen at the optimal magnification. (if the camera is not working then sketch the microstructure of each specimen.)
4. Make sure that the specimen that you prepared is clearly labeled and the specimen surface is in its final condition; it must be submitted with your lab write-up to the laboratory instructor.

Lab Requirements

1. Prepare a metallographic brass, steel or aluminum specimen going through the course grinding, fine grinding, polishing and etching stages of specimen preparation. Clearly label your specimen and submit it with the lab write up; the quality of your specimen will be graded.
2. Examine the four requisite samples and photograph each at the optimal magnification (if a camera is not available then sketch each of the microstructures).
3. Determine the ASTM grain size number and the average grain size for the single-phase brass specimen.
4. Briefly describe the salient features of the eutectoid specimens that you examined. How many phases and constituents are present in each?
5. Based on the observed microstructure of the steel sample, can you determine the carbon content of the steel? If yes, then briefly explain how you know the carbon content.
6. Based on the observed microstructures, which of the two brass specimens would you expect to be:

- Strongest? Why?
- Hardest? Why?
- More Ductile? Why?
- Stiffer (Elastic Modulus)? Why?

Homework

1. Briefly (one concise sentence each) describe the steps involved in the preparation of a metallographic sample:

Sectioning
Mounting
Course Grinding
Fine Grinding
Polishing
Etching

2. Why must metallographic samples be washed and carefully dried before proceeding from one grinding or polishing operation to the next?
3. What is the purpose of etching metallographic samples?
4. Why are metallographic samples sometimes mounted in plastic?
5. What is the difference between a Eutectoid steel and a Hypoeutectoid steel?
6. At room temperature :
 - What are the phases of a eutectoid steel?
 - What are the constituents of a eutectoid steel?
 - What are the phases of a hypoeutectoid steel?
 - What are the constituents of a hypoeutectoid steel?
7. If the ASTM grain size number of a single phase monel alloy (nickel solvent) is given as 5, approximate the average grain size for this alloy?(M = 100X)
8. If the grain size of a piece of brass is 0.07 mm, what is the ASTM grain size number? (M = 100X) For this piece of brass, how many grains will be there per square inch at a magnification of 250X?
9. Briefly explain the effect of grain size on the strength of a metal.