# ELEMENTARY METALLOGRAPHY

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## INTRODUCTION

Materials and Processes I (MET 141) with two one-hour lectures and one two-hour laboratory is offered to freshmen by the Mechanical Engineering Department at Purdue University. This elementary course, required for MET/CIMT majors, is an elective for other majors (IT and AT) and is also a prerequisite for other process courses (casting, welding and metallurgy). The goal of MET 141 is to broaden the technical background of students who have not yet had any college science courses. Hence, applied physics, chemistry and mathematics are included, and quantitative problem-solving is involved. The course outline consists of (1) General Inspection; (2) Properties - Physical, Chemical and Mechanical; (3) Processes and Selection - Metallic, Ceramics and Plastics. The goal of the laboratory is to reinforce and supplement the lecture material. There are eight experiments: (1) General Materials Inspection; (2) Hardness; (3) Toughness; (4) Polymers; (5) Steel Heat Treatments; (6) Aluminum Alloy Heat Treatments; (7) Metallography; (8) Recycling. Each lab section has up to 24 students. Depending on the type of the experiment and the available equipment, some experiments may split the students into subgroups (A and B). In the metallography experiment, there should be a maximum of 12 students in each group for alternate weeks.[1]

In an elementary metallography experiment, the objectives are: (1) Introduce the vocabulary and establish outlook; (2) Make qualitative observations and quantitative measurements; (3) Demonstrate the proper use of equipment; (4) Review basic mathematics and science.

## BACKGROUND

Metallography, a branch of physical metallurgy, is the study of the constitution and structure of metals and alloys. When a molten metal starts to solidify, small nuclei or seeds at various locations and orientations are formed. On each seed the neighboring atoms attach themselves in an orderly manner and grow continuously at different orientations, until confronted by neighbors. Each dendritic growth is roughly spherical, separated from its neighbors by definite boundaries, and is called a "grain" or "crystal." The average diameter, called "grain size," is a function of nucleation rate, growth, later heat-treatment, etc. The grains of a pure metal have the same lattice but different orientation of their crystal axes. When two grains of two different orientations come in contact, the "grain boundary" is really a zone of mismatch[2]. Since these border atoms (along grain boundary) are packed less densely (have fewer neighbors) they possess more energy than those others within the grain. Also, other atoms diffuse more easily along the boundaries than in the grain proper. This indicates that atoms along the boundaries are less tightly held, more reactive and energetic[3]. Exposed to corrosives or etchants, the grain boundary dissolves more than does the grain proper. The etched boundary then appears as a groove (in an otherwise flat surface) and the microstructure reveals black lines.[4]

It has been shown that most of the complex "polycrystallines" have intersections of any three grain boundaries at angles of 120° (see Fig. 1). Ideally, the grain structure of pure metals approximates the tri-corners (shown in Fig. 2.)[4]. Grains can be described as macroscopic or microscopic.

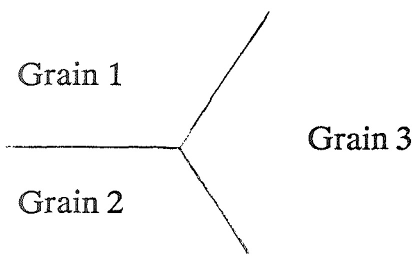



Figure 1 Three grains at angles of 120° to each other.

Figure 2 Micrograph of vacuum-deposited aluminum film, 0.5 $\mu m$ at 26,000 x.

## MACROSCOPIC AND MICROSCOPIC SAMPLES

### A. Qualitative Examination[1,5,6]

Macrophotography is appropriate when the grains of a specimen are visible to the naked eye or with a diametral magnification up to 50x. Examples of macroexamination include: (1) surface grains (mottling) on a cast-brass doorknob polished by normal use; (2) large size crystals (spangles) on galvanized steel-sheets; (3) aluminum grains on light-poles visible from the road; (4) columnar crystals at broken edges of cast-iron; (5) ice crystals at fractured ends of Popsicles™.

Preparation of samples for macroexamination is usually simple, involving less polishing and larger areas, for gross problems. Specimens may be burned off by acetylene torch, trimmed by hacksaw, then sectioned by cutter (water-cooled). After burrs are removed, the sample surface is planed by belt-sanders and smoothed by table grinders (water-cooled). Polishing is done by coarse 40-150 grits of emery paper. Frictional heating and structural distortions should be avoided.

After surface cleaning by water, the sample is etched. Use a 10% solution of nitric acid ($HNO_3$) for steel structure; a 50% water solution of hydrochloric acid (HCl) or 25% solution of nitric acid may reveal the flow-lines in forged steel and quench-cracks from heat treating. After deep etching is complete, the sample is rinsed in hot water, dipped in benzene or acetone, and dried in warm air-blast. When a dry surface is coated with printer's ink, an impression may be transferred to art paper. Simple macrographs may even be produced by pencil rubbings or photographs.

Unprocessed specimens may be examined. For example, a tensile or bending specimen may show ductile necking and cup-cone fracture, whereas a brittle piece may have glassy-smooth fracture without necking.

B. Quantitative Examination

Specimens are examined and evaluated by metallurgical microscopes and reflected light (50-2000x). Specimen preparation is critical, since small areas are inspected and measured, with statistical interpretation of data. Samples are cut and smoothed as before; however, flame cutting and frictional heating are risky. A thin section is cut off (water-cooled) and mounted in "buttons" of thermoset plastic (phenolic Bakelite™, or acrylic Lucite™) about one-inch diameter and half-inch thickness. The sample surface is carefully flattened by a wet-belt sander, which leaves parallel scratches. The sample is always washed (in soapy water) and then rotated ($90^{o}$) before each subsequent grind or polish. Exactly 2.54 cm equals an inch.

Polished samples are examined by microscopes for defects which require repolishing. Ideally, a mirror-surface (scratch-free at 1000x) is achieved before etching. The polishers are usually fine emery (or diamond paste). The etchant is a weaker acid or base solution; conservative practice is to under-etch, then to re-etch as desired. This flushes away the last layer of "distorted atoms".

C. Grain Size, Grain Size Number and Grain Boundary Area[7,8]

Since grain size and grain boundary area of a metal are important, the American Society for Testing and Materials (ASTM) has established reference nets with grain size numbers for comparison. When observed at 100x, the ASTM grain size number (n) can be determined from the ASTM grain size equation $N = 2^{n-1}$, where N is the number of grains per $in^2$. If N' is the number of grains per $in^2$ for other than 100x, say (50x), then multiply N' by $(100/50)^2$ to obtain N; then use the ASTM grain size equation.

There are three methods to estimate grain size and determine the ASTM grain size number: (1) Planimeter (Jeffries) method; (2) Intercept Methods; (3) Comparison Method.

(1) Planimeter Method: A circle or rectangle of known area (usually 5000 $mm^2$) is inscribed on a micrograph, or ground-glass screen of the metallograph, of known magnification. The magnification should be such to give at least 50 grains in the inscribed area. The number of grains counted within the area is added to one-half of those grains intersected by the boundary. The number of grains per $mm^2$ (N) for the given magnification is calculated readily. For a reasonable average a minimum of three representative fields should be tried. From known magnification, the ASTM grain size number (n) can be determined.

This method works well with equiaxed grains; otherwise, count should be made on three mutually perpendicular planes determined by the longitudinal, transverse and normal conditions.

(2) Intercept Methods: The intercepts, more convenient than the planimeter method, are recommended for structures which depart from uniform equiaxed structures. The procedure may be done by hand or by machines. There are two common methods: a) Lineal and b) Circular.

a) Lineal Intercept (Heyn) Method: The grain size can be estimated by counting on a photomicrograph (or ground-glass screen or specimen itself) the number of grains intercepted by one or more straight lines. Grains intersected by the end of the line count as half grains. For a reasonable average, counts must be made on at least three different lines. The length of the line, divided by the average number of grains intersected by it, gives the average intercept length (or grain diameter).

b) Circular Intercept Methods: These are favored[7], because circular arrays automatically compensate for non-equiaxed shapes of grains. This procedure avoids overweighting local portions of the sample's field and eliminates ambiguous intersections at straight-line ends. The manual routine is effective for grain estimation in quality control. A circular method is slightly biased, overvaluing the mean-intercept distance for samples with few intersections, but bias fades fast as intersections increase. There are two common procedures: i) Monocircle, and ii) Tri-circle.

The single-circle procedure is recommended when grain-size variation is large, and high precision is not required. Draw a circle of known circumference (100, 200, or 250mm) and count the intersected grain-boundaries. Different circles can be drawn at different locations. Select the magnification to yield no less than 35 counts per circle.

The three-circle procedure draws concentric and equally spaced circles, whose total circumference is 500 mm. Subsequent steps are the same as above.

The grain boundary area[4,5]: In the circular intercept method, after the circle is drawn on a photo-micrograph, the number of points of intersections per unit length of test line $P_L$ can be determined. If $S_V$ is the boundary area per unit volume (say in$^2$/in$^3$), and $L_A$ is the sum of lengths of linear features per unit test area (say in/in$^2$) then $S_V = 2P_L$ and $L_A = \frac{\pi}{2} P_L$.

(3) Comparison Method: After the sample is prepared, the image of the microstructure projected at 100x (or its photograph at 100x) is compared with a series of graded standards (grain size charts) indexed in four categories. Plates I, II, III, IV are available from ASTM Headquarters, 1916 Race Street, Philadelphia, PA 19103. By direct comparison of the specimen's microstructure with the appropriate photomicrograph of a standard grain size, one can select the standard photomicrograph (or interpolate between two) which most nearly matches the specimen. If a specimen has two different grain-sizes, it is reported in terms of two numbers, designating the approximate percent of each size.

## EQUIPMENT AND SUPPLIES

A. Units in the Materials Laboratory of the MET Dept.

1. <u>Equipment</u>:

Cut-off wheel, deburring wheel, mounting press, belt sanders, grinding wheels, polishing wheels, metallographic microscopes, ventilation hood, desiccator, planimeter ruler, compass.

2. <u>Supplies</u>:

Metal samples (raw, polished), polishing powders (aluminum 0.3 and 0.05 micron), ethanol, acetone, coating oil, distilled water, water glass, swab (Q-tip™); etchants.

B. <u>Notes</u>

1) The equipment and supplies are well maintained.
2) Students operate equipment and handle supplies properly and cautiously (under instructor's supervision).
3) The space for the above equipment and supplies is about 300-400 ft.$^2$ (30-40m$^2$).

C. <u>Alternatives</u>:

1) Using smaller portable equipment may occupy one-quarter of normal space.

2) Visiting the metallography laboratory of a metallurgy or materials department may involve a brief walk-through or an extensive show-and-tell.

3) The last resort is obtaining a film on metallography, to elucidate proper techniques.

## PROCEDURE

A. Observation

1. The instructor gives an overview of metallography terms, realms, and uses. Then, students get a show-and-tell session, which may be followed by practice from sample-cutting to etching. Different students perform the tasks, while others watch. Under the supervision of the instructor, a student may be asked to cut the sample, another to debur, and a third to mount it; other students observe, while the instructor comments on their safety and technique. All students take notes.
2. Students observe commercial samples having microscopic grain, such as brass door-knobs and galvanized steel-plates. Full-size (1x) photographs, impressions, or prints of these metals are also exhibited. Students estimate grain size and grains/area using standard charts (Hexagonal or McQuaid-Ehn).
3. Microphotographs of some alloys, with large or small magnifications, are exhibited. Students then calculate actual grain-size number from counts of grains (number per square inch).
4. Micrographs of non-metallic ceramics and polymers are also exhibited. Some 2-D (Optical) and 3-D (Scanning Electron Microscope) views are compared and contrasted. Metal powders (P/M) are identified by size and shape, with 3-D photos.

B. Measurements

1. During observation and experimentation, each student makes free-hand sketches of equipment. Then, students sketch samples they polished, as observed under the microscope. They are asked to record magnification, identify different regions, and label phases.
2. Photographs of different samples and various magnifications are handed to the students to determine grain concentration (grains/$in^2$) or ASTM grain-size number by: a) line intercepts, and b) planimeter.
3. Commercial samples (brass doorknob and galvanized plate) are given to students to identify grain/$in^2$ and to practice solving the formula for grain-size number (at 1x).

C. Report

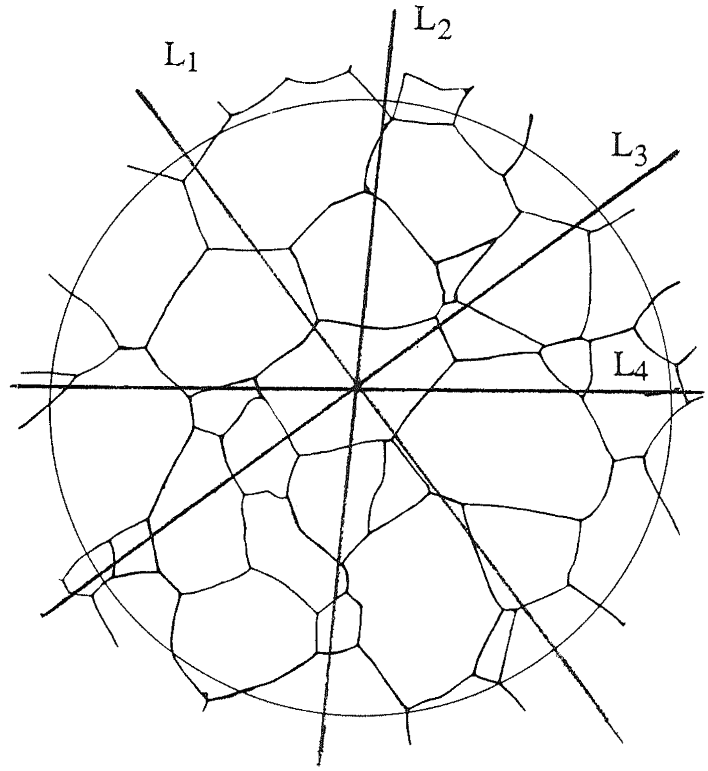
Each student prepares a report which consists of

1. A brief theory of metallography.
2. Basic procedures of sample preparation and sketch of a sample observed under microscope.
3. Sample calculations for grain size and ASTM grain size number.
4. Answers to questions.
5. Conclusions.

## Sample Calculations

Consider the photo (100x).

1. Using a) lineal and b) circular intercept methods, determine i) number of grains/area at 100x (grains/in², grains/cm²) ; ii) ASTM grain size number; (iii) grain size (average diameter); and c) Check the above values, by planimeter method.
2. Assuming d) magnification of 50x, and e) magnification of 200x, calculate "ASTM" grain size number in each case (at 100x).
3. Using the circular method determine f) $P_L$, g) $S_V$, and h) $L_A$ for the photo shown.




Solution:

1. a. Draw lines $L_1$ to $L_4$ at random. Measure lengths, and count the boundary-intercepts, $b_1$ to $b_4$.

i) $N = \frac{\text{Grains}}{\text{in}^2}$ at 100x

$L_1 = L_2 = L_3 = L_4 = 3.13$ in $= 7.95$ cm

$b_1 = 7$, $b_2 = 8$, $b_4 = 7$ boundaries

$$N = \frac{1}{4}\Sigma\left(\frac{b-1}{L}\right)^2 = 4.01\frac{\text{Grains}}{\text{in}^2} = 0.621\frac{\text{Grains}}{\text{cm}^2}$$

ii) From $N = 2^{n-1}$, where n = ASTM Grain size number,

$$n = \frac{\ell nN}{\ell n2} + 1 = \frac{\ell n4.01}{\ell n2} + 1 = 3.0 \text{ at } 100x$$

iii) $\dfrac{\text{Area}}{\text{Grain}} = \dfrac{1}{N} = 0.23\dfrac{in^2}{grain} = 1.49\dfrac{cm^2}{grain}$, at 100x

$$\frac{\text{Actual area}}{\text{grain}} = \frac{1}{N}\left(\frac{1}{100}\right)^2 = 2.3 \times 10^{-5}\frac{in^2}{grain} = 1.48 \times 10^{-4}\frac{cm^2}{grain}$$

Average size $= 0.54 \times 10^{-2}$ in $= 1.37 \times 10^{-2}$ cm

b. Circular Intercept Method: Determine the circumference C and the boundary intercepts b.

C = 9.84 in; b = 21 at 100x

$$N = \left(\frac{b-1}{c}\right)^2 = \left(\frac{21-1}{9.84}\right)^2 \frac{\text{Grains}}{in^2} = 4.48\frac{\text{Grains}}{in^2} = 0.694\frac{\text{grains}}{cm^2}$$

Similarly, n = 3.15; and $d = 5.33 \times 10^{-3}$ in $= 1.35 \times 10^{-2}$ cm

c. Planimeter Method:

A = Area = 7.694 $in^2$; Grains = 27

i) $N = \dfrac{\text{Grains}}{\text{Area}} = \dfrac{27 \text{ Grains}}{7.694 \text{ in}^2} = 3.51\dfrac{\text{Grains}}{in^2} = 0.544\dfrac{\text{grains}}{cm^2}$

ii) n = 2.81; (iii) $d = 5.34 \times 10^{-3}$ in $= 1.35 \times 10^{-2}$ cm

2. n' = n-2 = 3.11-2 = 1.11 at 50x
n'' = n+2 = 3.11 + 2 = 5.11 at 200x

3. f. $P_L = \dfrac{\text{Intercepts x } 100}{C} = \dfrac{21 \times 100}{9.843 \text{ in}} = 213 / \text{in} = 84 / \text{cm}$

g. $S_v = 2P_L = 426 \text{ in}^2 / \text{in}^3 = 168 \text{ cm}^2 / \text{cm}^3$

h. $L_A = \dfrac{\pi}{2} P_L = \dfrac{\pi}{2}(213)\dfrac{in}{in^3} = 135 \text{ cm} / \text{cm}^2$

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