

TECH NOTES

Introduction to Quantitative Metallography

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Quantitative Metallography- 30 Years Ago

For most of its history, metallographic observations have been largely qualitative in nature. The structure might be described as being relatively coarse or fine, or layered, or uniform. Particles might be referred to as being globular or spheroidal, lamellar, acicular, or blocky. Microstructures were single-phase or duplex, and so forth.

Thirty years ago when I entered industry, chart ratings and visual examinations were the main approach toward quantification. I can well remember the mill metallographers looking at spheroidized carbide tool steel structures and stating that it was, for example, 95%

spheroidized (many raters would never say 100%, just as some teachers would never grade an essay at 100%!) or that it was 60% spheroidized and 40% lamellar tending to spheroidize. Or, without looking at the chart (a seasoned rater never did), they would pronounce that the grain size was, for example, 100% 6 to 8 or perhaps 70% 8 and 30% 3 to 5 if it was duplex in appearance.

As a novice metallurgist, I was quite impressed by these pronouncements and tried to repeat the practice on my own. But, I found myself unable to repeat such ratings, if done several weeks apart, unless I made my estimates very broad. Later, I tried submitting the same specimens to different metallographers or to the same person at different times. I quickly learned that their repeatability/reproducibility wasn't that much better than mine. (Don't ever let a mill metallographer know that you are checking him/her!)

The greatest mystery to me, however, was inclusion chart ratings. I was very impressed that they could scan the 160 square mm area in a few minutes and, without taking any notes, jot down worst-field ratings. Absolute black magic! I never could do this. I had to use a scale and measure stringer lengths or count inclusions when I saw a field that looked like it had a high inclusion content. And I had to keep notes. But, I was a young, "hot-shot" metallurgical engineer – why couldn't I do as well? Was I overly dense? Did I go to too many parties and too few classes?

Naturally, I tried testing their ability to reproduce test results, and I found out that maybe I wasn't as slow as I thought. They didn't do that well reproducing their own results. Later, when I became active with ASTM Committee E-4, I found that inclusion ratings on round-robins, which had been tried numerous times, were notoriously non-reproducible. Well, that was good for soothing my shattered ego, but it did not solve my problems of describing structures.

I Discover Stereology

In the early 1970's, I came across some articles and a book, Quantitative Stereology, written by the late Ervin E. Underwood (Addison-Wesley Publishing Co., 1970). This was the beginning of a lifelong interest in quantifying microstructures and a long time friendship with a fascinating gentleman, Erv Underwood.



The late Erv Underwood at his home in Big Canoe, Georgia

Stereology is used to quantify matrix microstructures, as opposed to standard metrology techniques for measuring case depth, plating thickness, or particle size. Microstructural measurements are made on a two-dimensional plane-of-polish through a three-dimensional opaque metal. Stereology converts these 2-D measurements into 3-D estimates of microstructural parameters. Most procedures are very simple to use, but there are special considerations for their validity.

Metallographers commonly perform metrology type measurements; e.g., when measuring case depths, decarburization or plating/coating thickness. A scale is placed over the structure, and the depth or thickness perpendicular to the surface is measured. For example, ASTM E 1077 describes the measurement of decarburization of steel specimens using such measurements.

Nomenclature

Application of stereology has been hampered by confusion due to the use of different mathematical symbols for the same parameters. To minimize this problem, the International Society for Stereology has promoted a standard nomenclature which is constantly evolving as new approaches are developed. The most basic symbols are:

P = Point
 L = Line
 A = Area
 S = Surface
 V = Volume
 N = Number

These symbols can be combined in a number of ways to generate different symbols. For example, PP represents the point fraction; that is, the fraction of grid points lying in a phase of interest. While A and S seem to be the same, A is for a flat surface while S is for a curved surface. Thus, SV represents the grain boundary surface area per unit volume. NA is the number of particles per unit area while NV is the number per unit volume.

Phase Proportions

One of the most common measurements, determination of the amount of phases present, can be done using three different methods. Areal analysis, developed by Delesse in 1848, says that the area percent of a phase on a 2-D plane is equal to its volumetric percent, that is, AA = VV. However, measuring the area of second phases is very tedious unless they are quite coarse. Lineal analysis, developed by Rosiwal in 1898, says that the lineal fraction of test lines in a phase on the 2-D plane is equal to its volumetric percentage, that is, LL = VV. This is easier to determine but still rather tedious.

Starting around 1930, several workers in different fields and countries showed that the percentage of points on a test grid lying in the phase of interest was equal to the volumetric percentage, that is, PP = VV. Of the three methods, this is the most efficient technique; that is, it produces the best precision for the least effort when done manually. The point counting technique is described fully in ASTM E 562 (also ISO 9042). Image analyzers use essentially the same procedure; that is, the amount of a phase (usually called the area fraction or volume fraction even if it actually is a point fraction) is determined by the number of picture elements or "pixels" in the phase of interest divided by the total number of pixels; i.e., PP, expressed usually as a percentage.

Point Counting Example

ASTM E 562 describes the point counting procedure for determining the amount of second-phase constituents. A grid with systematically spaced points (e.g., 10 rows of 10 equally spaced points) is superimposed over the structure, either on an eyepiece reticle or a plastic sheet placed over or behind a ground glass projection screen or on a TV monitor screen. The points are usually drawn as fine perpendicular crossing lines and the "point" is the intersection of the two lines. This is done because actual points would be very difficult to see. The optimum point density for manual point counting is usually determined from $3/VV$ where the volume fraction is a fraction (not a percent). If the volume fraction is 0.5 (50%), then the optimum grid point density is 6. On the other hand, if the volume fraction is 0.01 (1%), the optimum point density is 300. The point fraction is the ratio of the points in the phase of interest to the number of grid points. Some people like to use a 100 point grid for all work since the division is unnecessary. Points falling on the interface are counted as $\frac{1}{2}$ a hit. For best manual results we need to sample more fields and do as little work as possible on each field measurement (the adage, "do more, less well"). The field-to-field variability has a greater influence on measurement precision than the counting precision on a given field.

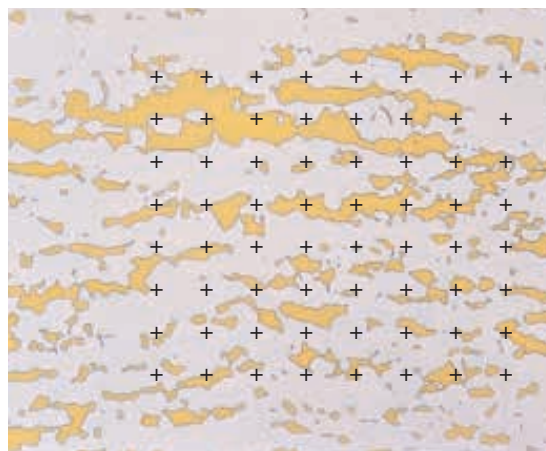


Figure 2. "Super-pure" aluminum electrolytically etched with Barker's reagent and viewed with polarized light plus a sensitive tint filter revealing an equiaxed, recrystallized grain structure. There are some fine impurity precipitates in this specimen, however, that cannot be seen with this etch and magnification (50x).

The microstructure above shows the beta phase in Muntz metal (Cu-40% Zn) preferentially colored by Klemm's I reagent while the alpha matrix is unaffected - ideal conditions for point counting. Since there is less β than α , we will count the number of times the points fall in the colored β grains. The amount of α is simply $100 - \% \beta$. As you can see, we have superimposed a 64-point test grid (8 rows of 8 points) over the structure and we have 15 hits and 4 tangent hits. The point fraction (volume fraction) is $17/64 = 0.266$ or 26.6%.

The point counting grid would be placed randomly over the structure a number of times so that the point fraction is determined for a number of fields. The necessary number of fields to yield a 10% relative accuracy varies inversely with the volume fraction (the lower the volume fraction, the greater the number of fields, i.e., the greater the total number of applied grid points).

Grain Size

Grain size is perhaps the most commonly performed microstructural measurement, although chart ratings are more commonly done than actual measurements (this is changing). A recent ASTM inter-laboratory "round-robin" showed that chart ratings of grain size are biased; that is, the ASTM grain size number is 0.5 to 1 unit too low (See Appendix X1 of E 112 -96). No bias existed when planimetric measurements were compared to intercept measurements by the same raters.

The ASTM grain size number, G , is defined as:

$$n = 2^{G-1}$$

where n is the number of grains per square inch at 100X. To convert n to NA (the number of grains per square mm at 1X), multiply n by 15.5. The four ASTM grain size charts show graded series of grain structures of different types.

Grain size can be measured by the planimetric method (developed by Zay Jeffries in 1916) or by the intercept method (developed by Emil Heyn in 1904). In the planimetric method, a count is made of the number of grains completely within a circle of known area and half of the number of grains intersected by the circle to obtain NA. Then, NA is related to G . This method is slow when done manually because the grains must be marked when counted to obtain an accurate count.

In the intercept method, either straight lines, curved lines, or circles are placed over the structure and a count is made of either the number of grain boundary intersections, P , or the number of grains intercepted, N , by the line. P or N is divided by the true line length, LT , to determine PL or NL , the number of intersections or interceptions per unit length (for a single phased structure). The reciprocal of PL or NL gives the mean lineal intercept length, l ,

$$l = 1/N_L = 1/P_L$$

a measure of grain size that can be converted to a G value. The intercept method is more efficient than the planimetric method yielding acceptable measurement precision (<10% relative accuracy) in much less time. ASTM E 112 contains a complete description of these methods. A major revision of E 112 was approved in 1995.

Grain Size Example

ASTM E 112 describes the manual measurement of grain size for structures with a single grain size distribution while ASTM E 1382 covers image analysis measurements. Note that E 112 was heavily revised in 1995 (additional minor changes in 1996), so it is best to read the latest version. Grain size can be measured using either the planimetric or the intercept methods. In the examples, we will simplify the approach slightly to illustrate the methods. Additional field sampling should be done to obtain good statistical data.

In the planimetric method, ASTM recommends using a test circle with a diameter of 79.8 mm (5000 sq. mm area) placed randomly over the grain structure. To obtain an accurate count of the number of grains inside the circle and the number intercepted by the circle, we must mark the grains on the template as we count which makes this method slow (although this is not a problem by image analysis). We must know the magnification of the image as well.

The image below shows the grain structure at 200X of a lowcarbon sheet steel after color etching. A circle of known size (64.4mm diameter) has been placed over the image to illustrate the method. There are 44 grains within the circle (ninside) and 34 grains intercepted by the circle (nintercepted). The number of grains per sq. mm, N_A , is calculated from:

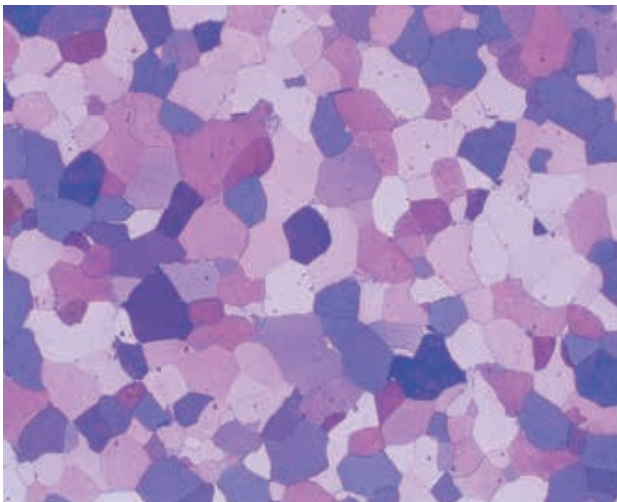
$$N_A = f \{n_{\text{inside}} + \frac{1}{2}(n_{\text{intercepted}})\}$$

The multiplier f is calculated from ($M^2/\text{circle area}$), where M is the linear magnification of the image. For this example,

$$N_A = 12.28 \{44 + \frac{1}{2}(34)\} = 749.1 \text{ grains /sq. mm}$$

From N_A , we can calculate the ASTM grain size number, G , using the following formula from E 112-96:

$$G = \{3.322 (\log_{10} N_A) - 2.954\} = 6.6$$



The ASTM grain size can also be determined using the intercept method counting either the number of grains intercepted, N , or the number of grain boundaries intersected, P , with a test line. ASTM recommends using a grid with three concentric circles with a 500mm total line length. To illustrate the principle of the method, we will use the same image with a single circle (shown on previous page). The count of the number of grains intercepted by the circle is N . To calculate the number of interceptions per mm, N_L , we divide N by the true length (circumference) of the circle. Since the diameter of the circle is 64.4mm, its circumference is πD , that is, 202.3mm. The true length is 202.3mm divided by the magnification, M , that is, 1.01mm. Hence, $N_L = N/LT = 34/ 1.01 = 33.6$ interceptions per mm.

To calculate the grain size, we first determine the mean lineal intercept length, l , which is the reciprocal of N_L (or of P_L , the number of grain boundary intersections per unit length). Thus, $l = 1/ 33.6 = 0.0298\text{mm}$. G is calculated from an equation from E 112-96:

$$G = \{-6.644 (\log_{10} l) - 3.288\}$$

where l is in mm. In this example, $G = 6.85$. Since the two methods are sensing different geometric aspects of the three-dimensional grain structure, they will not give exactly the same value, but they will be close, generally within the experimental limitations of the measurements. In practice, we would repeat these measurements on a number of fields in order to obtain a good estimate of the grain size.

Spacings

The spacing between second-phase particles, such as carbides or inclusions in steels or between intermetallic particles in aluminum alloys, can affect mechanical properties and formability. A special case is the interlamellar spacing of pearlite in high carbon steels (like rail steel) where refinement of the spacing improves both strength and toughness.

Spacings are easily assessed using a simple NL (number of particles intercepted per unit length of test line) measurement. The mean center-to-center spacing, sometimes called σ , is simply:

$$\sigma = 1/N_L$$

This is not a nearest-neighbor spacing, but the mean spacing between particles in the test line direction (either placed randomly or in some preferred direction, such as the through-thickness direction).

If the amount of the second phase is determined, for example, by point counting, the mean edge-to-edge spacing, called λ (or the mean free path, MFP), can be calculated by:

$$\lambda = (1-PP)/N_L$$

where PP is a fraction rather than a percentage. This is a very good structure-sensitive parameter.

By a simple subtraction of $(\sigma - \lambda)$, we can obtain the mean intercept length of the second phase particles – without measuring any particles! Furthermore, if we count the number of particles within a known area to obtain N_A (including only half of the particles intersected by the field edges), we can determine the average cross sectional area of the particles, A , by:

$$A = P_P / N_A$$

where PP is the point fraction (as a fraction, not a %) of the second phase. Thus, the average size of particles can be determined manually without actually measuring the particles. With modern image analyzers, individual measurements of particles are fast and simple. Besides generating average particle dimensions, the distribution of particle sizes can be obtained by feature-specific image analysis.

To determine the interlamellar spacing of pearlite (or of any eutectic or eutectoid), it is common practice to count the number of carbide interceptions with a straight test line perpendicular to the lamellae.

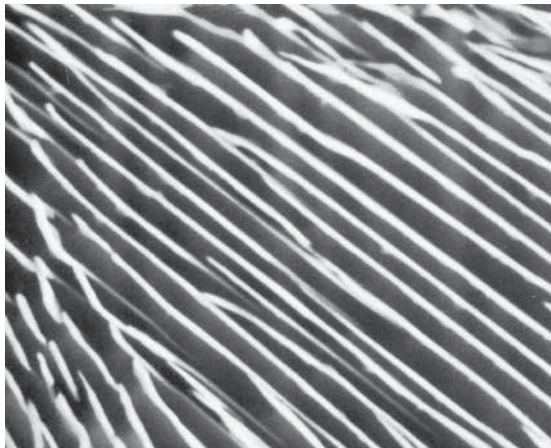
However, because the lamellae intersect the surface at different angles, it is better to determine a mean random spacing, σ_r , than a mean directed spacing, σ_d . A mean random spacing is obtained by determining NL using randomly oriented test lines (or curved or circular lines). The mean random spacing is easily used to calculate the mean true spacing, σ_t , by:

$$\sigma_t = \sigma_r / 2$$

In the past, the mean directed spacing, σ_d , was determined for the pearlite colony with the finest observed spacing, and this was assumed to be the true spacing. This is a better technique for isothermally-formed pearlite than for pearlite formed during continuous cooling. However, the longer you search for the finest colony, the finer the measured colony size! That is, the σ_d value obtained depends upon the amount of time spent looking for the finest colony, even in isothermally-formed pearlite. Any effort spent looking for a “best” or “worst” field condition, of any type, is strongly influenced by the amount of search time, and the results are neither reproducible nor precise.

Interlamellar Spacing Example

Traditionally, the metallographer has searched for the finest appearing interlamellar colony and made a measurement of its spacing using a test line perpendicular to the lamellae. This spacing is claimed to be the true interlamellar spacing. However, this method is not reproducible as the longer you search, the finer the measured



spacing. A better approach is to measure a mean random spacing and divide that by two to get the mean true spacing. This method was verified and proven to be correct (see Metallography, Vol. 17, No. 1, February 1984, pp. 1-17). The micrograph shown above is that of an as-rolled carbon steel of about 0.45% C, etched with 4% picral and photographed with the SEM (specimen perpendicular to the beam) using secondary electrons at 17,800x magnification. A circle with a diameter of 49.7mm was placed over the lamellae and the number of carbide lamellae intercepted by the test line, N, was counted. NL was again determined as N (23) divided by the true line length, $\pi D/M$, where the circle diameter, D, is 49.7mm and M is 17,800x. Thus NL is 2622 interceptions per mm. The mean random spacing, σ_r , is given by:

$$\sigma_r = 1/NL = 0.381\mu\text{m} = 381\text{nm}$$

The mean true spacing, σ_t , is $\sigma_r / 2 = 190.7\text{nm}$.

Statistics

Other measurements are possible, but the ones described above represent some of the simplest and most useful. Each can be repeated on a number of fields on the plane-of-polish so that a mean and standard deviation can be obtained. The number of fields measured influences the precision of the measurement. Manual measurements are tedious and time-consuming so sampling statistics may be less than desired. Image analysis removes most of the barriers to inadequate sampling.

A good measure of statistical precision is the 95% confidence interval (or confidence limit). This defines a range around the mean value where, 95 times out of 100, a subsequently determined mean will fall. For example, a mean volume fraction of $10\% \pm 2\%$ implies that for 95 of 100 measurements, the mean value will be between 8 and 12%. The 95% confidence interval is determined by:

$$95\% \text{ CI} = ts/n^{1/2}$$

where t is the Student's t factor (t is a function of the confidence level desired and the number of measurements, n, and can be found in standard textbooks and in some ASTM standards, e.g., E 562 and E 1382) and s is the standard deviation.

The relative accuracy, RA, of a measurement is determined by:

$$\%RA = 100 \cdot (95\% \text{ CI})/X$$

where X is the mean value. In general, a relative accuracy of 10% or less is considered to be satisfactory for most work.

Sampling

So far, we have discussed measurements on a single plane-of-polish on one specimen. Because we are usually dealing with large quantities of material (such as an entire “heat” of metal or alloy, a large heat treatment lot of bars, forgings, etc., or a large forging or casting), a single specimen may not be representative of the whole quantity. Ideally, random sampling of a large batch would be best, but practical considerations usually rule this out.

In most cases, sampling is done at predetermined convenient locations, such as the extreme ends of a coil, bar, plate, etc., or at locations which will be subjected to extensive machining. In some cases, excess metal is added to a forging or casting to provide test material as similar as possible to that of the component. Sampling is often a compromise and is rarely excessive due to cost considerations. However, inadequate sampling or sampling of nonrepresentative material or locations may degrade the value of the measurements.

Stereological measurements are best employed on sectioning planes that sample the microstructure randomly. This means that any oriented plane will produce the same data within the limits of statistical precision. However, for certain materials, the microstructure varies with the test plane. A classic example is that of inclusions in wrought steels which are preferentially elongated in the deformation direction. Sampled perpendicularly to the deformation axis (transverse plane), the inclusions look like spherical particles while, when sampled on a plane parallel to the deformation axis (longitudinal plane), they appear as long, thin rods or as broken “stringers”. If measurements are made on these planes, we obtain different values for NA, their length (or diameter), their spacing, and even the volume fraction. Thus, if we want to characterize their 3-D characteristics, measurements must be made on the three principal planes and averaged (or an alternate technique such as the trisector with vertical sectioning must be employed). In practice, the true 3-D characteristics may not be needed, and measurements are made using one standard test plane orientation which yields data suitable for quality control and material comparisons. This is the procedure employed in ASTM E 1245.

Specimen Preparation

If we are to obtain precise, unbiased measurements, we must prepare the specimens to reveal the true structure. In general, it is best to prepare specimens so that only the desired aspect of the structure is visible. In this way, the eye sees only what is needed, and other information is not present to obscure what is of interest. To study inclusions, intermetallics, or other constituents with a reflectivity or color different than the matrix, etching is not required. To measure matrix phases, use a selective etch that reveals only the constituent of interest. For example, for a carbon steel, etch with picral and point count the amount of ferrite or pearlite (always point count the constituent with the lower concentration). Etching

with nital will bring up the ferrite grain boundaries and produce less uniform etching of the pearlite making point counting more difficult and less precise.

The laws of stereology for measuring microstructure are based on the assumption that the etched surface is planar. There are rules for non-planar (curved) surfaces and for measurements with transmitted light or electrons, but these are much more difficult to employ. For microstructural measurements, the surface must be flat and etch depth should be minimal (do not over-etch!). Deep etching can dramatically change the apparent amount of a second phase and its size and spacing. Remember, good specimen preparation procedures are a necessary requirement for obtaining good data.

Conclusions

Although all of the most important stereological rules for measuring matrix microstructures were well known and described in Dr. Underwood's 1970 book (and elsewhere), their application was chiefly limited to research studies. Production metallographers did not adopt these methods because of the time required to make measurements manually.

The incredible growth in microcomputer power and in video technology over the past fifteen years has permitted development of very capable, reasonably priced user-friendly image analyzers, such as BUEHLER's OMNIMET® Advantage System. Image analysis permits collection of microstructural data (some of which are difficult to obtain manually) rapidly, painlessly, and with sufficient sampling to provide good statistical data. Future copies of Tech-Notes will be devoted to the subject of image analysis and its applications.

Tech-Tips

Question: We have been making delta ferrite measurements using the AMS 2301 procedures for many years, starting with the occupied squares method and then with the point counting methods added in versions B and C of the method. Why have these methods been changed, which should I use?

Answer: When AMS 2301 was developed, it contained a single method for estimating the amount of delta ferrite in a specimen (many specifications require less than 5% δ), called the "occupied squares" method. In this method, a grid consisting of 500 small squares (made by intersecting horizontal and vertical lines) was placed over the microstructure and a count was made of the number of "boxes" fully occupied by delta ferrite, the number 3/4 occupied, 1/2 occupied, and 1/4 occupied. The results were tabulated and added up to determine the fraction of the grid area covered, or "occupied," by the delta ferrite. This is basically an areal analysis method and it gives a rather precise measurement for that particular field, but only a few fields can be evaluated because it is so tedious. Measurement precision for the specimen is influenced much more by the field-to-field variability of the delta ferrite than by the precision of measuring each field. Thus, it is necessary to measure the delta on more fields. This method is too tedious for evaluating 20 or more fields, unless there is virtually no delta present. But, if that is the case, it may not be necessary to make a measurement at all. However, when the amount of delta is close to the typical 5% limit, the occupied squares method is too slow for practical use.

AMS 2301 next added a point counting method using a 500 point grid, which was later changed to 100 points. The optimum point

grid density is a function of the volume fraction and is found using the equation $3/\sqrt{V}$. Thus, for 5, 3 or 1% volume fractions (0.05, 0.03 and 0.01, respectively), the optimum point densities are 60, 100 and 300 points, respectively. A grid with 500 points takes more time to count than a lower point grid. For example, a 500 point grid takes five times as long to count as a 100 point grid. To optimize the method, that is, to make it most efficient, we want to measure as many fields as possible in the least amount of time (in agreement with the adage, "do more, less well"). To obtain a 10% relative accuracy with a 5% volume fraction and a 100 point grid, we do need to analyze at least 50 fields, which is often viewed as prohibitive when done manually. Hence, the interest in image analysis.

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