Measurement of Microstructure

QUANTIFYING MICROSTRUCTURAL PARAMETERS has received considerable attention in recent years and success in developing procedures and using such data to develop structure/property relationships has been achieved. Chart methods for rating microstructures have been used for many years to evaluate microstructures, chiefly for conformance to specifications. At this time, true quantitative procedures have not replaced chart methods for such purposes, but they have gained wide usage in quality control and research studies.

Basically, two types of measurements of microstructures are made. The first group includes measurements of depths (i.e., depth of decarburization, depth of surface hardening, or coating thicknesses). These measurements are made at a specific location (the surface) and may be subject to considerable variation. To obtain reproducible data, these surface conditions must be measured at a number of positions on a given specimen, and on several specimens if the material being sampled is rather large.

The second group of measurements belongs to the field referred to as stereology. This body of measurements describes the relationship between measurements made on the two-dimensional plane of polish and the three-dimensional microstructural features sampled. To facilitate communications, the International Society for Stereology (ISS) has proposed a standard system of notation, as shown in Table 1.

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Units</th>
<th>Description</th>
<th>Common name</th>
</tr>
</thead>
<tbody>
<tr>
<td>$P$</td>
<td>. . .</td>
<td>Number of point elements or test points</td>
<td>. . .</td>
</tr>
<tr>
<td>$P_p$</td>
<td>. . .</td>
<td>Point fraction (number of point elements per total number of test points)</td>
<td>Point count</td>
</tr>
<tr>
<td>$L$</td>
<td>mm</td>
<td>Length of linear elements or test-line length</td>
<td>. . .</td>
</tr>
<tr>
<td>$P_L$</td>
<td>mm$^{-1}$</td>
<td>Number of point intersections per unit length of test line</td>
<td>. . .</td>
</tr>
<tr>
<td>$L_L$</td>
<td>mm/mm</td>
<td>Sum of linear intercept lengths divided by total test-line length</td>
<td>Lineal fraction</td>
</tr>
<tr>
<td>$A$</td>
<td>mm$^2$</td>
<td>Planar area of intercepted features or test area</td>
<td>. . .</td>
</tr>
<tr>
<td>$S$</td>
<td>mm$^2$</td>
<td>Surface area or interface area, generally reserved for curved surfaces</td>
<td>. . .</td>
</tr>
<tr>
<td>$V$</td>
<td>mm$^3$</td>
<td>Volume of three-dimensional structural elements or test volume</td>
<td>. . .</td>
</tr>
<tr>
<td>$A_A$</td>
<td>mm$^2$/mm$^2$</td>
<td>Sum of areas of intercepted features divided by total test area</td>
<td>Areal fraction</td>
</tr>
<tr>
<td>$S_V$</td>
<td>mm$^2$/mm$^3$</td>
<td>Surface or interface area divided by total test volume</td>
<td>. . .</td>
</tr>
<tr>
<td>Symbol</td>
<td>Unit</td>
<td>Description</td>
<td>Notes</td>
</tr>
<tr>
<td>--------</td>
<td>--------</td>
<td>-----------------------------------------------------------------------------</td>
<td>--------------------------------------------</td>
</tr>
<tr>
<td>$V_Y$</td>
<td>mm$^3$/mm$^3$</td>
<td>Sum of volumes of structural features divided by total test volume</td>
<td>Volume fraction</td>
</tr>
<tr>
<td>$N$</td>
<td>. . .</td>
<td>Number of features</td>
<td></td>
</tr>
<tr>
<td>$N_L$</td>
<td>mm$^{-1}$</td>
<td>Number of interceptions of features divided by total test-line length</td>
<td>Lineal density</td>
</tr>
<tr>
<td>$P_A$</td>
<td>mm$^{-2}$</td>
<td>Number of point features divided by total test area</td>
<td></td>
</tr>
<tr>
<td>$L_A$</td>
<td>mm/mm$^2$</td>
<td>Sum of lengths of linear features divided by total test area</td>
<td>Perimeter (total)</td>
</tr>
<tr>
<td>$N_A$</td>
<td>mm$^{-2}$</td>
<td>Number of interceptions of features divided by total test area</td>
<td>Areal density</td>
</tr>
<tr>
<td>$P_V$</td>
<td>mm$^{-3}$</td>
<td>Number of points per test volume</td>
<td></td>
</tr>
<tr>
<td>$L_Y$</td>
<td>mm/mm$^3$</td>
<td>Length of features per test volume</td>
<td></td>
</tr>
<tr>
<td>$N_Y$</td>
<td>mm$^{-3}$</td>
<td>Number of features per test volume</td>
<td>Volumetric density</td>
</tr>
<tr>
<td>$L$</td>
<td>mm</td>
<td>Mean linear interception distance, $L_L/N_L$</td>
<td></td>
</tr>
<tr>
<td>$A$</td>
<td>mm$^2$</td>
<td>Mean area intercept, $A_A/N_A$</td>
<td></td>
</tr>
<tr>
<td>$S$</td>
<td>mm$^2$</td>
<td>Mean particle surface area, $S_Y/N_Y$</td>
<td></td>
</tr>
<tr>
<td>$V$</td>
<td>mm$^3$</td>
<td>Mean particle volume, $V_Y/N_Y$</td>
<td></td>
</tr>
</tbody>
</table>

Note: Fractional parameters are expressed per unit length, area or volume.

These measurements are made manually with the aid of templates outlining a fixed field area, systems of straight or curved lines of known length, or a number of systematically spaced points. The simple counting measurements, $P_P$, $P_L$, $N_L$, $P_A$, and $N_A$, are most important and are easily made. These measurements are useful by themselves and can be utilized to derive other important relationships. These measurements can also be made using semiautomatic or automatic image analyzers.

**Volume Fraction**

One of the simplest and most useful measurements is the point count (described in ASTM E 562) used to estimate volume fractions of microstructural constituents. While other manual procedures can be employed, the point count is most efficient—it gives the best accuracy with minimum effort. To perform this test, a clear plastic grid with a number of systematically spaced points (usually crosses are employed, where the "point" is the intersection of the arms), typically 9, 16, 25, 49, 64, or 100, is placed on a micrograph, on a projection screen, or inserted as an eyepiece reticle. The number of points lying on the phase or constituent of interest is counted and divided by the total number of grid points. Points lying on a boundary are counted as half-points. This procedure is repeated on a number of fields selected without bias (without looking at the image).

The point fraction, $P_P$, is given by:
\[ P_P = \frac{P_\alpha}{P_T} \]  

(Eq 1)

where \( P_\alpha \) is the number of grid points lying inside the feature of interest, \( \alpha \), plus one-half the number of grid points lying on particle boundaries, and \( P_T \) is the total number of grid points. Studies have shown that the point fraction is equal to the area fraction, \( A_A \), and volume fraction, \( V_V \), of the second-phase particles:

\[ P_P = A_A = V_V \]  

(Eq 2)

The volume fraction can also be estimated by dividing the total length of linear elements of a test pattern lying within the phase by the total length of the test pattern. The lineal fraction, \( L_L \), is also equal to the parameters in Eq 2. Point counting is always performed on the minor phases—where \( V_V = 0.5 \). The amount of the major (matrix) phase can be determined by difference.

The fields measured should be selected at locations over the entire polished surface, not confined to a small portion of the specimen surface. The field measurements should be averaged, and the standard deviation can be used to assess the relative accuracy of the measurement, as described in ASTM E 562.

In general, the number of points on the grid should be increased as the volume fraction of the feature of interest decreases. One study suggested that the optimum number of grid test points is \( 3/V_V \). Hence, for volume fractions of 0.5 (50%) and 0.01 (1%), the optimum numbers of grid points are 6 and 300, respectively. If the structure is heterogeneous, measurement accuracy is improved by using a low-point-density grid and increasing the number of fields measured.

Figure 1 demonstrates the point counting procedure and shows a synthetic microstructure consisting of 24 circular particles, each 6 mm in diameter, within a field area of 12,100 mm\(^2\). The total area of the circular particles is 678.6 mm\(^2\), which is an area fraction of 0.056 (5.6%). A square grid pattern has been drawn over this field, producing 100 intersection points. Four of these intersections are completely within the particles and four lie on the particle interface. The number of "hits" is, therefore, \( 4 + \frac{1}{2} (4) = 6 \). Thus, \( P_P \) is \( \frac{6}{100} \) or 0.06 (6%), which agrees very closely with the theoretically calculated area fraction. The area fraction, \( A_A \), is equal to the volume fraction, \( V_V \), as long as the sectioning plane intersects the structural features at random.

![Fig. 1 Synthetic microstructure of uniformly shaped, identical size spherical particles in a matrix phase. Test area is 12,100 mm\(^2\). Test lines, ten horizontal and ten vertical, are 110 mm long](image)

**Number per Unit Area**

The count of the number of particles within a given measurement area, \( N_A \), is a useful microstructural parameter and is used in other calculations. Referring again to **Fig. 1**, there are 24 particles in the
measurement area (12,100 mm$^2$). Hence, the number of particles per unit area, $N_A$, is 0.00198 mm$^{-2}$. The average area of the particles can be calculated by dividing the volume fraction, $V_r$, by $N_A$:

$$A = \frac{V_r}{N_A} \quad \text{(Eq 3)}$$

This yields an average area, $A$, of 28.23 mm$^2$, which agrees well with the calculated area of a 6 mm diameter particle of 28.27 mm$^2$.

**$P_L$ and $N_L$**

Counting of the number of intersections of a line of known length with particle or grain features, $P_L$, or the number of interceptions of particles or grains by a line of known length, $N_L$, provides two very useful microstructural parameters. For space-filling grain structures (single phase), $P_L = N_L$, while for two-phase structures, $P_L = 2N_L$ (this may differ by one count in actual cases).

**Grain-Structure Measurements.** For single-phase grain structures, it is usually easier to count the grain-boundary intersections with a line of known length. This is the basis of the Heyn intercept grain-size procedure described in ASTM E 112. For most work, a circular test grid composed of three concentric circles with a total line length of 500 mm is preferred. Grain size is defined by the mean lineal intercept length, $l$:

$$l = \frac{1}{P_L} = \frac{1}{N_L} \quad \text{(Eq 4)}$$

This equation must be modified, as described later, for two-phase structures. To calculate the ASTM grain size number, $l$ can be used.

$P_L$ measurements can be utilized to define the surface area per unit volume, $S_V$, and the length per unit area, $L_A$, of grain boundaries:

$$S_V = 2P_L \quad \text{(Eq 5)}$$

and

$$L_A = (\pi/2)(P_L) \quad \text{(Eq 6)}$$

For single-phase structures, $P_L$ and $N_L$ are equal, and either measurement can be used. For two-phase structures, it is best to measure $P_L$ to determine the phase-boundary surface area per unit volume, or phase-boundary length per unit area.

**Grain Size**

Perhaps the most common quantitative microstructural measurement is that of the grain size of metals and alloys. Numerous procedures have been developed to estimate grain size; these procedures are summarized in detail in ASTM E 112 and illustrated in. Several types of grain sizes can be measured:
ferrite grain size, austenite grain size, and prior-austenite grain size. Each type presents particular problems associated with revealing these boundaries so that an accurate rating can be obtained. To complicate matters, a variety of parameters are utilized to define grain size:

- Average grain diameter, \( d \)
- Average grain area, \( A \)
- Number of grains per unit area, \( N_A \)
- Average intercept length, \( l \)
- Number of grains intercepted by a line of fixed length
- Number of grains per unit volume, \( N_V \)
- Average grain volume, \( V \)

These parameters can be related to the ASTM grain size number, \( G \).

The ASTM grain-size scale was established using the Imperial system of units, but no difficulty is introduced by metric measurements. The ASTM grain size equation is:

\[
n = 2^{G-1} \quad \text{(Eq 12)}
\]

where \( n \) is the number of grains per square inch at 100×. Multiplication of \( n \) by 15.5 gives the number of grains per square millimeter at 1×, \( N_A \).

**Planimetric Method.** The oldest procedure for measuring the grain size of metals is the planimetric method. A circle of known size (generally 79.8 mm diameter, 5000 mm\(^2\) area) is drawn on a photomicrograph or used as a template on a projection screen. The number of grains completely within the circle, \( n_1 \), and the number of grains intersecting the circle, \( n_2 \), are counted. For accurate counts, the grains must be marked off as they are counted, which makes this method slow. The number of grains per square millimeter at 1×, \( N_A \), is determined:

\[
N_A = f(n_1 + n_2/2) \quad \text{(Eq 13)}
\]

where \( f \) is the magnification squared divided by 5000 (the circle area). The average grain area, \( A \), in square millimeters, is:

\[
A = 1/N_A \quad \text{(Eq 14)}
\]

and the average grain diameter, \( d \), in millimeters, is:

\[
d = (A)^{1/2} = 1/(N_A)^{1/2} \quad \text{(Eq 15)}
\]

The ASTM grain size, \( G \), can be found by using the tables in ASTM E 112 or by the following equation:
Figure 2 illustrates the planimetric method. Expressing grain size in terms of \( d \) is being discouraged by ASTM Committee E-4 on Metallography, because the calculation implies that grain cross sections are square, which they are not.

**Eq 16**

\[
G = 3.322(\log N_A) - 2.95
\]

The intercept method is faster than the planimetric method because the micrograph or template does not require marking to obtain an accurate count. ASTM E 112 recommends use of a template consisting of three concentric circles with a total line length of 500 mm (template available from ASTM). The template is placed over the grain structure without bias, and the number of grain-boundary intersections, \( P \), or the number of grains intercepted, \( N \), is counted. Dividing \( P \) or \( N \) by the true line length, \( L \), gives \( P_L \) or \( N_L \), which are identical (\( N \) or \( P \) can differ slightly due to tangent hits) for a single-phase grain structure. It is usually easier to count grain-boundary intersections for single-phase structures. If a grain boundary is tangent to the line, it is counted as \( \frac{1}{2} \) of an intersection. If a triple-point line junction is intersected, it is counted as \( \frac{1}{2} \) or 2. The latter is preferred because the small diameter of the inner circle introduces a slight bias to the measurement that is offset by weighing triple-line intersections as 2 hits.

The mean lineal intercept length, \( l \), determined as shown in **Eq 4**, is a measure of ASTM grain size. It is smaller than \( d \), because the test lines do not intersect each grain at its maximum breath. The ASTM grain size, \( G \), can be determined by use of the tables in ASTM E 112 or can be calculated:

**Eq 17**

\[
G = -6.644 \log l - 3.288
\]

where \( l \) is in millimeters. **Figure 3** shows the intercept method for a single-phase alloy.
Nonequiaxed grain structures require measurements on the three principle planes: the longitudinal, planar, and transverse. For such structures, the intercept method is preferred, but the test grid should consist of a number of straight, parallel test lines of known length rather than circles. Because the ends of the straight lines generally end within grains, these interceptions are counted as half-hits. Three mutually perpendicular orientations are evaluated using grain-interception counts:

- $N_{L_1}$--parallel to the grain elongation, longitudinal plane
- $N_{L_2}$--perpendicular to the grain elongation (through-thickness direction), transverse plane
- $N_{L_3}$--perpendicular to the grain elongation (across width), planar surface

The average $N_L$ value is obtained from the cube root of the product of the three directional $N_L$ values. $G$ is determined by reference to the tables in ASTM E 112 or by use of Eq 17 ($l$ is the reciprocal of $N_L$, Eq 4).

Two-Phase Grain Structures. The grain size of a particular phase in a two-phase structure requires determination of the volume fraction of the phase of interest, such as by point counting. The minor phase (second phase) is point-counted and the volume fraction of the major phase (matrix phase) is determined by difference.

Next, a circular test grid is applied to the microstructure without bias and the number of grains of the phase of interest intercepted by the test line, $N_{\alpha}$, is counted. The mean lineal intercept length of the alpha grains, $l_{\alpha}$, is determined by:

$$l_{\alpha} = \left[\frac{(V_r)(L/M)}{N_{\alpha}}\right]$$  \hspace{1cm} (Eq 18)

where $L$ is the line length and $M$ is the magnification. The ASTM grain size number can be determined from the tables in ASTM E 112 or by use of Eq 17. The method is shown in Fig. 4.
**Inclusion Content**

Assessment of inclusion types and contents is commonly performed on high-quality steels. Production evaluations utilize comparison chart methods such as those described in ASTM E 45, SAE J422a, or the German standard SEP 1570 (DIN 50602). In these chart methods, the inclusion pictures are defined by type and graded by severity (amount). Either qualitative procedures (worst rating of each type observed) or quantitative procedures (all fields in a given area rated) are employed. Only the Japanese standard JIS-G-0555 uses actual volume fraction measurements for rating of inclusion content (although the statistical significance of the data is questionable).

Manual measurement of the volume fraction of inclusions requires substantial effort to obtain acceptable measurement accuracy due to the rather low volume fractions usually encountered. Consequently, extensive use of image analyzers has been made to overcome this problem. Image analyzers separate the oxide and sulfide inclusions on the basis of their gray-level differences. By using automated stage movement and autofocus, enough field measurements can be made in a relatively short time to obtain good statistical accuracy. Image analysis is also employed to measure the length of inclusions or to determine stringer lengths.

**Measurement Statistics**

In performing stereological measurements, it is necessary to make these measurements on a number of fields and average the results. Measurements on a single field may not be representative of the bulk conditions, because few (if any) materials are sufficiently homogeneous. Calculation of the standard deviation of the field measurements gives a good indication of measurement variability. Calculation of the standard deviation can be done quite simply with an inexpensive pocket calculator.

A further refinement of statistical analysis is calculation of the 95% confidence limit based on the standard deviation, $s$, of the field measurements. The 95% confidence limit is calculated:

$$95\%\ CL = ts/N^{1/2} \quad \text{(Eq 19)}$$
where \( t \) is the student's \( t \) value that varies with \( N \), the number of measurements. Many users standardize on a single value of \( t \), 2, for calculations irrespective of \( N \). The measurement value is expressed as the average \( X \pm \) the 95% CL value. This means that if the test were conducted 100 times, the average values would be between plus and minus the average, \( X \), in 95 of the measurements. Next, one can calculate the relative accuracy, \( % \ RA \), of the measurement:

\[
% \ RA = (95\% \ CL)X
\]

(Eq 20)

Usually, a 10% relative accuracy is considered to be adequate. DeHoff (Ref 7) has developed a simple formula to determine how many fields, \( N \), must be measured to obtain a specific desired degree of relative accuracy at the 95% confidence limit:

\[
N = [(200/%RA) - (S/X)]^2
\]

(Eq 21)