The metric grain size number, G_M , is approximately 4.5% higher than the ASTM International grain size number, G, for the same structure, or:

$$G = G_M - 0.045$$
 (Eq 14)

This very small difference usually can be ignored (unless the value is near a specification limit).

Planimetric Method

The oldest procedure for measuring the grain size of metals is the planimetric method introduced by Zay Jeffries (Ref 19), a founding member of ASTM Committee E-4 on Metallography, in 1916 based on earlier work by his Ph.D. advisor, Albert Sauveur. A circle of known size (generally 79.8 mm, or 3.14 in., diameter; 5000 mm², or 7.75 in.², area) is drawn on a micrograph or used as a template on a projection screen or a photograph. 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 $1 \times$, N_A , is determined by:

$$N_A = f(n_1 + n_2/2)$$
 (Eq 15)

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

$$A = 1/N_A \tag{Eq 16}$$

and the average grain diameter, d, in millimeters, is:

$$d = (A)^{1/2} = 1/N_A^{1/2}$$
 (Eq 17)

The ASTM International grain size, G, can be found by using the tables in ASTM E 112 or by the equation:

$$G = [3.322 (log N_A) - 2.95]$$
(Eq 18)

Figure 12 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 in shape, which they are not.

In theory, a straight test line will, on average, bisect grains intercepting a straight line. If the test line is curved, however, bias can be introduced (Ref 2, 29, 30). Saltykov (Ref 2) suggested that such bias could occur, but he did not do any tests to evaluate this potential problem. Two other studies (Ref 29, 30) claimed that if such bias occurred, it was not evaluated properly. Theoretically, bias should decrease as the number of grains within the circle increases. If only a few grains are within the circle, the error could be large, for example, a 10% error if only ten grains are within the circle. ASTM E 112

recommends adjusting the magnification so that at least 50 grains are within the field to be counted. Under this condition, the possible error is reduced to approximately 2%. This degree of error is not excessive. If the magnification is decreased or the circle is enlarged to encompass more grains, for example, 100 or more, obtaining an accurate count of the grains inside the test circle becomes very difficult. An extensive test was performed where a very wide range of test circle diameters and grain size image magnifications were utilized to make grain size measurements using the planimetric method. This test revealed that bias did not result. At very small numbers of grains within the circle and intercepting the circle (Eq 15), data scatter increased, but no bias was observed (Ref 31). In this experiment using



Fig. 12 Microstructure of an austenitic manganese steel, solution annealed and aged to precipitate a pearlitic phase on the grain boundaries (at 100×). There are 43 grains within the circle (n_1) , and there are 25 grains intersecting the circle (n_2) . The area of the test circle is 0.5 mm² (0.0008 in.²) at 1×. N_A is 111 grains/mm², and the ASTM International grain size is 3.8.

the planimetric method with test circles, data scatter was observed when the value of $(n_1 + 0.5n_2)$ was below ~30. For the Saltykov rectangle, data scatter was observed when $(n_1 + 0.5n_2)$ was below ~11. For the triple-point-count method, data scatter was observed when $(n_1 + 0.5n_2)$ was below ~25. Bias was not observed in any of these methods (and not with the intercept method, either).

A simple alternative to the data-scatter problem exists and is amenable to image analysis. If the test pattern is a square or rectangle, rather than a circle, data scatter occurs with low counts per placement, and the extensive test results (Ref 31) revealed the approach exhibited the least data scatter at the lowest grain count. However, counting grains intersecting the test line, n_2 , is slightly different using a square or rectangular grid. In this method, grains intercept the four corners of the square or rectangle. Statistically, the portions intercepting the four corners would be in parts of four such contiguous test patterns. Thus, when counting n_2 , the grains intercepting the four corners are not counted but are weighted as one intercepted grain. A count of all other grains intercepting the test square or rectangle (of known size) is weighted as half inside. Equation 15 is modified as follows:

$$N_A = f(n_1 + (n_2/2) + 1)$$

where n_1 is still the number of grains completely within the test figure (square or rectangle), n_2 is the number of grains intercepting the sides of the square or rectangle but not the four corners, 1 is for the four grains that intercept the four corners, and *f* is the magnification divided by the area of the square or rectangle grid. Figure 13 demonstrates such a counting procedure.



Fig. 13 Grain structure of Monit, a ferritic stainless steel at $400 \times$ magnification (the magnification bar is 25 µm). There are 15 grains completely inside the 102×114 mm (4 × 4.5 in.) rectangle and 24 grains that intersect the rectangle, ignoring the four grains at the corners (coded C1 to C4), which adds 1 to Eq 15. The area of the rectangle is 0.07258 mm² (0.00011 in.²) at 1×. N_A is 385.7785 mm² (0.60 in.²), and the ASTM International grain size is 5.64.

Intercept Method

The intercept method, developed by Emil Heyn (Ref 20) in 1903, 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 (Ref 22) consisting of three concentric circles with a total circumferential line length of 500 mm (20 in.) (template available from ASTM International). 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 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 1/2 of an intersection. If a triple-point line junction is intersected, it is counted as 11/2 or 2. The latter is preferred because the small diameter of the inner circle could introduce a slight bias to the measurement, which is offset by weighing a triple-point test line intersection as two hits.

The mean lineal intercept length, l, determined as shown in Eq 4, is a measure of ASTM International grain size. This length is smaller than the maximum grain diameter because the test lines do not intersect each grain at their maximum breadth. The ASTM International grain size, G, can be determined by using the tables in ASTM E 112 or can be calculated from:

$$G = [-6.644 (\log l) - 3.288]$$
(Eq 19)

where l is in millimeters. Figure 14 illustrates the intercept method for a single-phase alloy.

Nonequiaxed Grains

Nonequiaxed grain structures require measurements on the three principal planes, that is, the longitudinal, planar, and transverse planes. (In practice, measurements on any two of the three are adequate.) 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, oriented as described subsequently. 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 graininterception counts:

- *N*_{LII}: parallel to the grain elongation, longitudinal or planar surface
- N_{L⊥}: perpendicular to the grain elongation (through-thickness direction), longitudinal or transverse surface
- *N_{LP}*: perpendicular to the grain elongation (across width), planar or transverse surface

The average N_L value is obtained from the cube root of the product of the three

directional N_L values. Grain size number, G, is determined from the tables in ASTM E 112 or by using Eq 19 (*l* is the reciprocal of N_L , Eq 4).

Two-Phase Grain Structures

The grain size of a particular phase in a twophase structure requires determination of the volume fraction of the phase of interest, for example, 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_{α} , is counted. The mean lineal intercept length of the alpha grains, l_{α} , is determined by:

$$l_{\alpha} = (V_V)(L/M)/N_{\alpha} \tag{Eq 20}$$

where V_V is a fraction (not a percent), *L* is the line length, and *M* is the magnification. The ASTM International grain size number can be determined from the tables in ASTM E 112 or by using Eq 19. The method is illustrated in Fig. 15. Again, a circular test grid could introduce some data scatter in counting (as described previously for the planimetric method) if the number of interceptions per grid placement is quite low. This can be eliminated by using a square or rectangular test grid and counting as described earlier. (The grains intercepted by the four corners are counted as 1, while all of the grains intercepting the sides are weighted as one each.)

Inclusion Content

Assessment of inclusion types and amounts (Ref 34-50) is commonly performed on high-

Fig. 14 100× micrograph of 304 stainless steel etched electrolytically with 60% HNO₃ (0.6 V direct current, 120 s, platinum cathode) to suppress etching of the twin boundaries. The three circles have a total circumference of 500 mm (20 in.). A count of the grain-boundary intersections yielded 75 (P = 75). The mean lineal intercept length is 0.067 mm (0.003 in.), and the ASTM International grain size number, G, is 4.5.

parison chart methods such as those described in ASTM E 45, "Standard Test Methods for Determining the Inclusion Content of Steel.' SAE J422a, "Microscopic Determination of Inclusions in Steels," ISO 4967, "Steels-Determination of Content of Non-Metallic Inclusions—Micrographic Method Using Standard Diagrams," or the German standard SEP 1570 (DIN 50602), "Metallographic Examination: Microscopic Examination of Special Steels Using Standard Diagrams to Assess the Content of Non-Metallic Inclusions." In these chart methods, the inclusion images are defined by type and graded by severity (amount, width, and length). Either qualitative procedures (worst rating of each type observed) or quantitative procedures (all fields in a given area rated) are used. Only the Japanese standard JIS-G-0555, "Microscopic Testing Method for Non-Metallic Inclusions in Steel," uses actual volume fraction measurements for the rating of inclusion content (although the statistical significance of the data is questionable due to the limited number of counts required). Qualitative chart ratings of inclusions using methods such as ASTM E 45 vield poor evaluations of inclusions by type and by severity in interlaboratory roundrobins conducted by a number of mill metallographers doing such work regularly (Ref 50). Raters often misidentified A- and C-type inclusions, and the severity ratings for the same specimens often covered the full severity range.

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 (Ref 39, 42). When the volume fraction is below 0.02 (2%), which is the case for inclusions, even in free-machining steels, acceptable relative accuracies (Eq 22) cannot

Fig. 15 $_{\rm forged}^{500\times}$ micrograph of Ti-6242 alpha/beta forged and alpha/beta annealed, then etched with Kroll's reagent. The circumference of the three circles is 500 mm (20 in.). Point counting revealed an alpha-phase volume fraction of 0.485 (48.5%). The three circles intercepted 76 alpha grains. The mean lineal intercept length of the α -Ti phase is 0.00638 mm (0.00025 in.), and the ASTM International grain size, G, is 11.3.



quality steels. Production evaluations use com-

be obtained using manual point counting without a large amount of counting time (Ref 39). The use of image analyzers has 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 autofocusing, enough field measurements can be made in a relatively short time to obtain reasonable statistical precision. Image analysis is also used to measure the length of inclusions and to determine stringer lengths.

Two image-analysis-based standards have been developed: ASTM E 1122, "Standard Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis" (Ref 41), and ASTM E 1245, "Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis" (Ref 42, 44, 47, 48). ASTM E 1122 produces JK ratings by image analysis that overcome most of the weaknesses of manual JK ratings (in 2006, ASTM E 1122 information was merged into ASTM E 45). ASTM E 1245 is a stereological approach defining, for oxides and sulfides, the volume fraction (V_V) , number per unit area (N_A) , average length, average area, and the mean free path (mean edge-toedge spacing). These data are easily stored in a database and are statistical. This enables developing means and standard deviations to compare data from different tests to determine if the differences between the measurements are valid at a particular confidence level.

At an ASTM E-4 meeting, George A. Moore of the former National Bureau of Standards (now the National Institute of Standards and Technology) recommended measuring the area fraction using eight to twelve sets of 30 to 100 sequential measurements of the area fraction on each specimen. The mean value of the area fraction would be calculated based on the eight to twelve set means. This would enable obtaining a valid arithmetic standard deviation. Calculating the mean and standard deviation of all individual measurements would result in a log-normal rather than Gaussian distribution. The mean of the eight to twelve sets will differ slightly from the mean of all the individual values.

To test the approach, an experiment was conducted using nine specimens with sulfur contents from 0.020 to 0.34 wt% and using image analysis with 16, 32, and $80 \times$ objectives (Ref 26, 39). The area fraction of sulfides was measured using three rows of 30 contiguous fields and 12 such sets (1080 fields). The total areas examined were 165.8, 42.69, and 6.63 mm² (0.26, 0.067, and 0.010 in.²) for the 16, 32, and $80 \times$ objectives. This was repeated using manual point counting at $500 \times$ and using a 100-point grid and 10 sets of 10 contiguous fields (1 h/specimen). Lineal analysis used a Hurlbut counter at $1000 \times$ and using eight to ten linear measurements on each specimen, with 15 min duration for each run. Results are shown in Fig. 16 for the manual point-count method, Fig. 17 for the lineal



Fig. 16 Manganese sulfides in each specimen were manually point counted with a 100-point grid using 10 sets of 10 contiguous fields (~1 h/specimen) to obtain the data shown. The correlation between the wt% S and the manual point fraction was quite good, although the time required was impractical. $P_P = 4.7278$ (%S) + 0.2561. Correlation coefficient, r = 0.9555



Fig. 17 Lineal analysis was performed using a Hurlbut counter at $1000 \times$ with 8 to 10 linear measurements on each specimen, each lasting 15 min. The correlation between the wt% S and the lineal fraction, L_L , was slightly better than the point fraction data, but the amount of effort was more than double per specimen. $L_L = 4.9808 (\%S) + 0.1904$. Correlation coefficient, r = 0.972

analysis data, and Fig. 18 for the image analysis data. As would be expected, the results were best, and took the least time and effort, for the image analysis experiment.

Measurement Statistics

In performing stereological measurements, it is necessary to make the measurements on several fields and average the results. Measurements on a single field are unlikely to 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 simply by using Microsoft Excel or an inexpensive pocket calculator.

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

$$95\%$$
 CL = $ts/(n-1)^{1/2}$

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 regardless of n. Table 5 lists the student's t-values for calculating the 95% CL as a function of the number of measurements, n, and the degrees of freedom, n - 1. The measurement value is expressed as the average, X, plus and minus the 95% CL value. This means that if the test was 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 by:

$$%$$
RA = (95% CL) · 100/X (Eq 22)

Usually, a 10% relative accuracy is adequate. DeHoff (Ref 51) 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% CL:

(Eq 21)
$$N = \left(\frac{200}{\sqrt[6]{RA}} - \frac{s}{X}\right)^2$$
(Eq 23)



Fig. 18 Image analysis measurements were performed with three objectives using three rows of 30 contiguous fields and a total of 12 sets per specimen (1080 fields/specimen). The area evaluated was 165.8, 42.69, and 6.63 mm² (0.26, 0.067, and 0.010 in.²) for the 16, 32, and 80× objectives, respectively. Image analysis gave the best correlation and was fastest. For the 16, 32, and 80× objective data, r-values were 0.9764, 0.9884, and 0.9878. $A_A = 5.3285$ (%5) + 0.1909. Correlation coefficient, r = 0.9866 (all data)

Table 5	Student's <i>t</i> -values for a 95%	
confiden	ce limit, where <i>n</i> is the number o	0
measuren	nents, and <i>n</i> – 1 is the degrees o	f
freedom		

12.706	1.0	
	18	2.101
4.303	19	2.093
3.182	20	2.086
2.776	21	2.080
2.571	22	2.074
2.447	23	2.069
2.365	24	2.064
2.306	25	2.060
2.262	26	2.056
2.228	27	2.052
2.201	28	2.048
2.179	28	2.045
2.160	30	2.042
2.145	40	2.021
2.131	60	2.000
2.120	120	1.980
2.110	∞	1.960
	4.303 3.182 2.776 2.571 2.447 2.365 2.306 2.262 2.228 2.201 2.179 2.160 2.145 2.131 2.120 2.110	$\begin{array}{cccccc} 4.303 & 19 \\ 3.182 & 20 \\ 2.776 & 21 \\ 2.571 & 22 \\ 2.447 & 23 \\ 2.365 & 24 \\ 2.306 & 25 \\ 2.262 & 26 \\ 2.228 & 27 \\ 2.201 & 28 \\ 2.179 & 28 \\ 2.160 & 30 \\ 2.145 & 40 \\ 2.131 & 60 \\ 2.120 & 120 \\ 2.110 & \infty \end{array}$

Image Analysis

The measurements described in this brief review, and other measurements not discussed, can be made by use of automatic image analyzers. These devices rely primarily on the gray level of the image on the display monitor to detect the desired features. In some instances, complex image editing can be used to aid separation. Use of image analysis to perform these measurements is discussed elsewhere in this Volume and in Ref 52 to 58.

Summary

This article reviewed many commonly used stereological counting measurements and the relationships based on these parameters. The measurements described are easy to learn and use. They enable metallographers to discuss microstructures in a more quantitative manner, and they reveal relationships between the structure and properties of the material. The measurements also enable determination of whether one batch of metal products is better, worse, or the same quality as another, or if one manufacturing process produced better quality, equal quality, or poorer quality than another. Qualitative analysis work using chart methods cannot meet the statistical quality needed to make such evaluations unless the results are vastly different, which is possible but not likely.

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Scanning Electron Microscopy

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Overview

General Uses

- Imaging of surface features from 10 to 100,000× magnification. Resolution is typically 1 to 10 nm, although <1 nm is possible.
- Depending on available detectors, the microscope enables differentiation of polycrystalline grains for unetched samples, microstructural components with and without surface etching, topographical features, crystallographic structure and material phase, extended defects, and local variations in material properties.

Examples of Applications

- Examination of metallographically prepared samples at magnifications well above the capabilities of optical microscopy
- Examination of fracture surfaces and deeply etched surfaces requiring depth of field well beyond that possible with the optical microscope
- Evaluation of phase and crystallographic orientation for individual grains, precipitates, microstructural constituents, and nanoscale particles
- Identification of the chemistry for micrometer and submicrometer features, such as inclusions, precipitate phases, and wear debris
- Mapping of elemental composition across micrometer to millimeter length scales
- Examination of semiconductor devices for failure analysis, critical length determination, and design verification

Samples

 Form: Any low-vapor-pressure (0.1 Pa, or ≤10⁻³ torr), conductive, solid material including particles, thin films, and bulk samples. Nonconductive solids can be imaged using a conductive coating. Uncoated nonconductive solids can be imaged using low-beam energy or using a variable-pressure/ environmental scanning electron microscope (SEM). Wet and fixed biological specimens can also be imaged using variablepressure/environmental SEM.

- *Size:* Limited by the SEM specimen chamber size and stage travel. Generally, samples as large as ~20 cm (8 in.) can be placed in the microscope, but regions on such samples that can be examined without repositioning are limited to ~5 cm (2 in.).
- *Preparation:* Standard metallographic polishing and etching techniques are adequate for electrically conductive materials. Nonconducting materials are generally coated with a thin layer of carbon or metal. Samples must be electrically grounded to the holder, and fine samples, such as powders, can be dispersed on an electrically conducting adhesive. Samples must be free of high-vapor-pressure liquids, such as water, organic cleaning solutions, and remnant oil-based films, unless imaged inside a variable-pressure/environmental SEM.

Limitations

- Image quality on relatively flat samples, such as metallographically polished and etched samples, is generally inferior to the optical microscope below 300 to 400× magnification.
- Point resolution, although much better than the optical microscope, is inferior to the transmission electron microscope and the scanning transmission electron microscope.

Capabilities of Related Techniques

- X-ray diffraction: provides bulk crystallographic information
- Optical microscopy: faster, less expensive, and provides superior image quality on relatively flat samples at less than 300 to 400× magnification
- *Transmission electron microscopy:* provides similar information with superior resolution but is more expensive and requires thinned specimens

Introduction

A scanning electron microscope (SEM) is a type of instrument that magnifies and images sample surfaces through controlled rastering

of a highly focused electron beam across the area of interest. A variety of signals are produced, particularly backscattered and secondary electrons, as the electron beam interacts with the sample surface; these signals provide local topographic and compositional information regarding the specimen. The SEM was invented in 1937 (Ref 1) and was first commercialized in 1965 (Ref 2). There have been continual improvements in SEM resolution, dependability, ease of operation, and reduction in instrument size. Scanning electron microscopes are regularly used in materials research, forensics, failure analysis, geological studies, biological imaging, metallurgy, nanomaterials development, microelectronics, and fractography. Scanning electron microscopes are a common instrument in most materials characterization laboratories and are increasingly used for immediate, on-site metrology and quality control at the manufacturing floor.

The primary use of an SEM is to produce high-resolution images at magnifications unattainable by optical microscopy, while also providing direct topographic and compositional information. Magnifications up to $100,000 \times$ or more are possible by modern SEMs. Scanning electron microscopes produce nanoscale-resolution imaging and mapping because the electron beam is focused to an ~1 to 10 nm sized probe. However, issues consider regarding electron probe size and depth sensitivity are the influence of accelerating voltage, beam current, sample composition, and the specific signal being detected. Signals are most commonly secondary electrons, backscattered electrons, and x-rays. Other possible signals that can be generated and detected in the SEM include visible light (cathodoluminescence), beam induced current, and Auger electrons.

This article provides detailed information on the instrumentation and principles of the SEM, including:

- Description of the primary components of a conventional SEM instrument
- Advantages and disadvantages of the SEM compared with other common microscopy and microanalysis techniques
- Critical issues regarding sample preparation
- Details on the physical principles regarding electron beam-sample interaction
- Mechanisms for many types of image contrast
- · Details of SEM-based techniques
- Specialized SEM instruments
- Example applications using various SEM modes

The Microscope

Figure 1 illustrates the basic components of a typical SEM. The components can be categorized as the electron-optics column, the specimen chamber, the support system, and the control and imaging system. The electron beam is generated at an electron gun and accelerated toward the sample housed inside a specimen chamber, typically below the electron-optics column. Electromagnetic lenses below the electron gun focus the electron beam to a small probe at the sample surface. Scanning coils deflect the electron probe across the sample surface, and detectors housed either in the specimen chamber or in the electronoptics column collect and report resultant signal intensity as a function of beam position. The SEM operator uses a computer to control the electron-optics, detectors, and a motorized stage for sample positioning. During SEM operation, a support system provides cooling water for the electro-magnetic lenses and maintains a low pressure within the electronoptics column and the specimen chamber using vacuum pump.

Electron-Optics Column

The electron-optics column produces a narrowly divergent beam of electrons along the centerline of the column and steers the beam onto the sample surface. The major components of the electron-optics column are the electron gun, condenser lens, objective lens, and scanning coils.

Electron Gun

The electron gun contains the source of electrons and components that accelerate electrons to high energies. Two example guns are shown in Fig. 2. The electron gun produces a beam of electrons and focuses them to a spot of diameter, d_0 , and divergence half-angle, α_0 . The electron source is typically a tungsten hairpin, tungsten needle (Fig. 2), or a sharp-tipped crystal composed of a rare earth metal hexaboride (LaB₆ or CeB₆). Electron sources are generally classified as thermionic sources or field-emission sources. Thermionic

sources use heat to energize and release electrons from the source material; field-emission sources primarily rely on an electrostatic field for electron beam generation.

Thermionic emission sources heat a tungsten filament or La/CeB6 crystal to temperatures of ~2500 or 1600 °C (4500 or 2900 °F), respectively. Current applied to the thermionic source resistively heats the material and energizes electrons from the sharpest radius-of-curvature (ROC) point on the source. The thermal energy imparted to the electrons at the surface must be sufficient to overcome the work function of the source material surface. Most metals reach or exceed their melting temperature before the electrons can overcome the work function. Tungsten has the highest melting temperature, lowest vapor pressure, lowest thermal expansion coefficient, and a very high tensile strength. Thus, tungsten is an ideal metal for an electron source. Tungsten filaments are bent into a "hairpin" shape with an ROC of ~100 µm (Ref 4). The LaB₆ is preferable to tungsten, because it has a lower work function and higher electron emissivity, thus requiring a lower operating temperature while emitting more electrons. The CeB₆ is less susceptible to contamination than LaB₆ and thus is seeing increased use. Stable hexaboride crystals have 90° cone tips with ~15 µm ROC.

Field-emission sources use a high electric field in the vicinity of the source. Field enhancement at the sharpest point in the source tunnels electrons off the source surface while also greatly localizing electron emission. Therefore, field-emission sources produce



Fig. 1 Schematic of the basic components of a conventional scanning electron microscope. BE, backscattered electron; SE, secondary electron

much higher electron current densities (1000 to $10,000 \text{ A/cm}^2$) than thermionic sources (1 to 10 A/cm²). Field-emission sources, or emitters, are typically etched tungsten single-crystal needles with nanometer-sized ROCs. A ZrO coating on sharp tungsten needles (tens of nanometers ROC) creates a local Schottky barrier within the electric field, which lowers the work function of the tungsten (100) facet, thus requiring a much lower operating temperature (1500 °C, or 2700 °F) than a thermionic tungsten source. The ZrO-tungsten sources are called Schottky field-emission sources (Fig. 2b). Single-crystal (310) tungsten can be etched to achieve even sharper needle tips (few nanometers ROC), so the local electric field alone is enough to tunnel electrons off the tungsten (310) facet surface; these sources are called cold field-emission sources, since they operate at room temperature.

Within the electron gun, electron extraction from the electron source requires a voltage potential between the source (cathode) and another electrode in proximity (anode). The anode accelerates the electrons to a specific kinetic energy and directs the initial electron beam trajectory down the electron-optics column. The cathode is negatively biased, and the accelerating anode is positively biased. For thermionic sources, the cathode is considered "hot," and another electrode with an aperture is positioned near the tungsten filament or hexaboride crystal. The aperture electrode, the Wehnelt cylinder, is negatively biased to a few hundred volts and functions as an electrostatic lens, which helps control electron extraction from the thermionic source. Farther away, the accelerating anode is strongly biased positive. For field-emission guns (FEGs), the appreciable voltage gradient present between the anode and the cathode provides a sufficiently strong electrostatic field, so the much sharper tip of the field-emission sources produces adequate field enhancement for electron extraction. A negatively biased electrode, the suppressor, helps localize the electron-extraction region at the source, while a positively biased extracting anode farther away pulls the electrons toward the accelerating anode.

The most important parameter distinguishing these four types of electron guns is the brightness, β :

$$\beta = \frac{I}{\left(\frac{\left(\pi d_0^2\right)}{4}\right)\left(\pi \alpha_0^2\right)}$$
(Eq 1)

where β equals the amount of current, *I*, focused on an area, $\pi d_0^2/4$, entering and exiting this area through a solid angle, $\pi \alpha_0^2$ (Ref 5). Increasing β improves the overall performance of the SEM. The value of β is primarily a function of the electron source material and the type of electron gun. Cold FEG sources provide the highest β values but require ultrahigh-vacuum conditions, because the room-temperature emitter is more susceptible to

contamination. Contaminants that attach to the cold FEG source are removed by flashing the emitter with bursts of current. Cold FEG sources greatly increase SEM instrumentation costs but are highly stable and long-lasting sources. Schottky FEG sources produce lower β values than cold FEG sources, but they are also less expensive and require less stringent vacuum demands. Thermionic sources have much lower β values than FEG sources but are also much cheaper. They do require occasional source replacement, particularly the less expensive tungsten filaments as opposed to the more expensive but higher-brightness hexaboride crystals. Table 1 compares important properties for the four types of electron sources.

Lenses

Lenses within electron microscopes use electromagnetism to focus electron beams. Electromagnetic lenses (Fig. 3a) consist of copper wire windings inside an iron fixture, which is carefully machined to specific dimensions with pole pieces designed to localize magnetic fields. Current passed through the windings of copper wire magnetizes the iron and produces a magnetic field, which is radially symmetric about the lens axis. As an electron moves through the magnetic field, it experiences a radial force inward, which is proportional to the Lorentz force, $v \times \mathbf{B}$, where v is the electron velocity, and **B** is the magnetic flux density. The function of the pole pieces is to produce progressively higher density magnetic flux lines farther away from the lens axis. Electrons traveling parallel, but at different distances to the lens axis, experience different inward Lorentz forces, so that they converge at the focal plane. The resulting lensing action is comparable to the function of an optical lens, in which a ray parallel to the axis of the lens is bent toward the lens axis. Eventually, the ray meets the lens axis at a distance below the lens principal plane that corresponds to the focal length, f, of the lens (Fig. 3b).

For an optical lens, f is determined by the lens surface curvature and refractive index of the lens medium; the focal length is fixed. The focal length of an electromagnetic lens depends on two factors: the acceleration voltage (which determines the electron velocity, v) and the amount of current passing through the copper winding (which determines the flux density, **B**). Therefore, the operator can control and tune the electromagnetic lens focal length by adjusting the currents supplied to them; an



Fig. 2 Schematics of conventional (a) thermionic tungsten hairpin filament gun and (b) Schottky field-emission gun. Adapted from Ref 3

Table 1 Comparison of electron source properti
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Property	Thermionic tungsten filament	Thermionic LaB ₆ /CeB ₆	Schottky field-emission ZrO/W (100)	Cold field-emission tungsten (310)
Brightness (β), A/cm ² · sr	10 ⁶	107	5×10^8	10 ⁹
Crossover or virtual source diameter (d_0) , nm	>10 ⁴	>10 ³	15–25	3–5
Work function, eV	4.5	2.6/2.4	2.8	4.3
Energy spread, eV	1-3	>1	0.3-1	0.2-0.3
Operating temperature, °C (°F)	2500 (4500)	1600 (2900)	1500 (2700)	25 (75)
Typical service lifetime, h	40-100	1000/1500	>2000	>2000
Operating vacuum, Pa (torr)	$10^{-4} (7.5 \times 10^{-7})$	$10^{-6} (7.5 \times 10^{-9})$	10^{-8} to 10^{-9} (7.5 × 10^{-11} to 7.5 × 10^{-12})	10^{-9} to 10^{-11} (7.5 × 10^{-12} to 7.5 × 10^{-14})
Source: Ref 6				





Fig. 3 (a) Schematic cross section of conventional electromagnetic objective lens. (b) Ray diagram of a standard lens. Magnification, M = L'/L

increase in current increases the overall radial force experienced by all electrons within the beam and thus reduces f.

Rather than achieving magnification, the purposes of lenses in an SEM is to reduce the initial electron beam diameter, d_0 , to a much small diameter at the sample surface. Therefore, SEM lenses demagnify the electron beam diameter, as illustrated in Fig. 3(b). The arrow in the object plane is reproduced upside down in the image plane, and the arrow-tip image can be located by following rays (1) and (2). The magnification is M = L'/L; as focal length f is reduced, the value of L' is reduced, reducing magnification, M. Therefore, if the length of the arrow is taken as the electron beam diameter, d_0 , produced by the gun, then the beam diameter, d_1 , after passing through the first condenser lens is $d_1 = M_1 d_0$, where M_1 is the magnification of the first condenser lens.

Figure 4 shows the coupling of two condenser lenses and one objective lens; the object plane for a given lens is the image plane from the lens above it. The net result is that the diameter of the electron beam at the sample surface, d_3 , is:

$$d_3 = d_0 \cdot M_1 \cdot M_2 \cdot M_3 \tag{Eq 2}$$

where M_1 , M_2 , and M_3 are the demagnification factors for each lens. Spot size, or actual beam size, on the sample surface, d_s , is somewhat



Fig. 4 Ray diagram of beam demagnification by three lenses in a scanning electron microscope. WD, working distance

larger than d_3 due to lens aberrations. Considering a typical value of $d_0 = 15 \ \mu m$ for a thermionic tungsten filament gun, and the value of d_3 may reach 15 nm, this corresponds to $M_1 \cdot M_2 \cdot M_3 = 1000$. Note that the $1000 \times$ demagnification has no direct relationship to the actual magnification achieved by the SEM using scanning coils, as described later. Also, apertures located near each lens serve to limit the angular spread of the electron beam as it progresses along the electron-optics column axis. Apertures are metal discs with machined holes of various diameters ranging from 5 to 1000 µm. The user can often select aperture sizes to adjust beam convergence angle, depth of field, and beam current (number of electrons) arriving at the specimen.

The sample position relative to the objective lens focal plane, L_3' in Fig. 4, can also be independently adjusted by the user via stage positioning inside the specimen chamber. The distance of the focal plane below the bottom of the objective lens, and/or final aperture, is termed the working distance (WD). The center axis of the electro-optics column is typically considered the z-axis direction with respect to sample orientation. If the sample surface position is changed vertically along the z-axis, the objective lens current must be adjusted to have L_3' fall on the sample surface. The objective lens current adjustment is the focusing procedure performed by the operator to obtain the sharpest image possible. Because f_3 and therefore L_{3}' are functions of current applied to the objective lens, a unique value of WD corresponds to objective lens current; WD is often displayed on the control system. Therefore, once the operator ensures the sample is at focus, the associated WD value can often serve as a useful z-axis value for the stage position.

Stronger lens focusing produces a smaller beam diameter. Therefore, image resolution improves as WD is decreased. In the past, special high-resolution SEMs used an immersion lens that greatly reduced WD by positioning the sample inside the objective lens. Modern SEMs might incorporate a snorkel lens, which extends the electromagnetic field below the objective lens pole piece, effectively immersing the sample within the focusing lens field. These modes greatly reduce f_3 and further reduce d_3 to enhance resolution.

As mentioned previously, the actual beam spot size at the sample, d_s , is larger than d_3 due to lens aberrations, including spherical aberration, chromatic aberration, and astigmatism. Of these, only astigmatism is regularly corrected in a typical SEM and primarily for the objective lens, since it is the probe-forming lens of the SEM. Astigmatism arises from imperfections in the machined soft iron pole piece of the electromagnetic lenses, which diminish the radial symmetry of the electromagnetic field produced by the lens. This leads to a larger beam diameter at the lens focal plane. To improve the field radial symmetry, an array of independently controlled stigmator coils are arranged around the inside circumference of a lens. Each coil produces additional magnetic field lines, enabling the user to adjust and balance the overall electromagnetic lens action of the entire assembly. Figure 5 illustrates the effect of astigmatism on SEM images of gold nanoparticles. When the sample is slightly out of focus, features exhibit a stretched appearance because of the elongated beam diameter overlapping adjacent image pixels. The directionality of the stretching is orthogonal when going from an underfocus to overfocus condition or vice versa. Adjusting the stigmators to ensure the beam diameter is circular in underfocus and overfocus conditions serves to reduce the beam size when at focus. For this reason, Fig. 5(d) exhibits sharper image quality than Fig. 5(a), even though both images are technically in focus. Astigmatism correction for the objective lens is a common task during SEM operation, particularly for higher magnifications where it becomes crucial to obtain the smallest possible spot size, d_s .

It may be necessary to increase the number of electrons arriving at the sample, even at the expense of producing a larger spot size. More electrons impinging the sample lead to greater overall signal, which is particularly desirable for x-ray analysis and backscattered electron imaging. The condenser lens immediately above the objective lens enables controlling both spot size and number of arriving electrons, as shown in Fig. 6. The L_2' and d_2 decrease with increasing condenser lens strength. For the same objective lens strength, a smaller d_2 beam diameter produces a smaller d_3 beam diameter at the sample surface. However, as the cross-over point for the condenser lens moves farther away from the objective lens and objective aperture, fewer electrons can pass through the objective aperture. Therefore, any reduction in the spot size accomplished by increasing condenser lens strength also results in fewer electrons arriving at the sample surface. Sacrificing beam current for smaller spot size is a standard trade-off in the SEM.

Scan Coils

The main function of scan coils is scanning the focused electron beam across the sample surface to produce an image. Two sets of scan coils located in the bore of the objective lens cage shown in Fig. 1 perform the scanning function. The coils, further detailed in Fig. 7, deflect the beam to scan over a square area of size $r \times r$ on the sample surface. The scanned area is generally termed the raster. Although the beam is shown in Fig. 7 as a line, it is diverging as it passes through the scan coils. However, because the divergence half-angle is in the milliradian range, representation as a line is reasonable. For simplicity, Fig. 7 depicts the scanning process at approximately midway during the formation of a single frame.

Scanning electron microscopes achieve magnification using this double-deflection system, with the beam deflected by the Lorentz force produced by low-impedance coil windings driven by a low-voltage power supply. The scan generator produces a voltage across each coil pair. The upper scan coil pair, X₁, produces a magnetic field at time 1, which provides a Lorentz force that deflects the beam to the right through angle θ_{max} . The lower scan coil pairs, X2, deflect the beam back to the left through angle $2\theta_{max}$ so it strikes the sample at the left edge of the raster. The X1 - X2 voltage signal decreases stepwise with time, as shown at the upper left of Fig. 7. For each short time interval, termed dwell time, the beam sits at a single position on the sample surface. From time 1 to time 5, the beam scans along the line of length ron the sample surface.

At time 5, the $X_1 - X_2$ scan voltage "flies back" quickly to 1′, causing the beam to return rapidly to the left side of the raster.



Fig. 5 Scanning electron microscope images of gold nanoparticles obtained before astigmatism correction. (a) At focus. (b) Underfocus. (c) Overfocus. (d) At focus after astigmatism correction. Inset depicts electron beam spot size relative to pixel size.







Fig. 7 Diagram of double-deflection system showing progressive line scanning to produce a frame of size $r \times r$

Concurrently, Y1 and Y2 coil pairs now have a small voltage, causing a small deflection so a new line begins at position 1' in detail A of Fig. 7. During the fly-back time from 5 to 1', the beam moves along the dashed line shown in detail A in Fig. 7. The next line is scanned, and the process is repeated until a full raster is accomplished and a single frame is produced. The scan generator controls the frame size. number of lines within each frame, number of positions on each line, and the dwell time for each position on each line. Each position corresponds to an individual pixel in the resultant digital image. The pixel value in terms of grayscale depends on the relative signal intensity collected during the dwell time interval.

The magnification, M, achieved by the SEM is simply the ratio of the displayed size to the raster size, r. For example, consider a 50 cm (20 in.) screen digital display that is 44.3 cm wide by 24.9 cm high (17.5 by 10 in.) and projects in 16:9 aspect ratio with 1600 × 900 pixels. If the raster size matches the aspect ratio, but over a much smaller region corresponding to 32.0 µm by 18.0 µm of the sample surface, then the magnification is M = 44.3 cm/0.0032 cm or M = 24.9 cm/0.0018 cm, which corresponds to a magnification of ~13,800×.

The SEM magnification is determined by the user-controlled size r of the raster via the scanning coils. Higher magnification simply requires reducing r through smaller variations in the scan generator voltage, thus decreasing the deflection angles θ_{max} and $2\theta_{max}.$ The double-deflection system ensures the electron beam passes through the objective lens plane consistently at the same point on the electrooptics axis. Therefore, the objective lens current can be maintained, and the sample generally remains in focus as the magnification is altered. The SEMs typically enable controlling and tuning the directionality of the raster, enabling scan rotation that effectively rotates the apparent image on the digital screen. Sometimes reduced areas within the frame can be selected, often so microscope parameters can be tuned based on a single feature of interest within the field of view. If the working distance is increased with no change to scanning conditions, then the raster size increases and M correspondingly decreases; the lowest available magnification depends on the WD used.

Because the objective lens current, is a known function of WD for a focused sample, magnification is reported based, in part, on this lens current; SEM instruments show the magnification directly on the digital display. More importantly, a scale bar is also provided. The scale bar provides a direct correlation between the pixel units of the digital image to the actual physical size of the raster and associated surface features being imaged in the SEM. Commercially available specimens with patterned features of well-established physical sizes are used to calibrate any SEM. The calibration step is crucial to ensure the accuracy of both the displayed magnification and associated scale bar.

Specimen Chamber

The specimen chamber of an SEM contains the sample and the primary detectors for imaging and analysis. Specimen chambers range in size from small enclosures used for tabletop SEMs housing centimeter-sized samples, to medium-range sizes typical for research-grade SEMs capable of handling up to ~ 20 cm (8 in.) samples, to large enclosures designed to handle specimens up to meters in size. The chambers are vacuum capable enclosures usually constructed of nonmagnetic stainless steel. The inside chamber walls must be maintained dry and free of residue to facilitate obtaining the necessary low operating pressures during imaging and analysis, while also minimizing sample surface contamination. Plasma cleaners can be attached to SEM chambers to enable removal of residual contamination during operation. Specimen chambers can be vented to atmosphere and opened for sample loading. For SEMs that regularly operate at high-vacuum conditions $<1.33 \times 10^{-5}$ Pa ($<10^{-8}$ torr), a load-lock apparatus is attached to the specimen chamber. This enables the operator to load the sample into a secondary chamber that is pumped to low pressures before sample insertion to the main specimen chamber, thus avoiding the need to vent the entire chamber to atmosphere.

Stage

When the sample is loaded into the SEM specimen chamber, it sits on a motorized, computer-controlled stage. Some SEMs with manual micrometer control and no motorization are still in use, but they are becoming increasingly obsolete. The SEM operator positions the sample in the electron beam path and selects regions of interest for imaging and/or analysis. Simple SEM systems may only have a two-axis stage, which enables x- and y-axis control of the sample position. More advanced SEMs typically have a five-axis stage for x-, y-, and z-axis positioning plus sample rotation and tilt control. As mentioned previously, the z-axis is conventionally defined parallel to the center axis of the electro-optics column. The y-axis is often oriented relative to the direction of the secondary electron detector. In an analytical SEM, the y-axis may be oriented with respect to the energy-dispersive spectrometer.