



# Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis<sup>1</sup>

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## INTRODUCTION

This practice may be used to produce stereological measurements that describe the amount, number, size, and spacing of the indigenous inclusions (sulfides and oxides) in steels. The method may also be applied to assess inclusions in other metals or to assess any discrete second-phase constituent in any material.

### 1. Scope

1.1 This practice describes a procedure for obtaining stereological measurements that describe basic characteristics of the morphology of indigenous inclusions in steels and other metals using automatic image analysis. The practice can be applied to provide such data for any discrete second phase.

NOTE 1—Stereological measurement methods are used in this practice to assess the average characteristics of inclusions or other second-phase particles on a longitudinal plane-of-polish. This information, by itself, does not produce a three-dimensional description of these constituents in space as deformation processes cause rotation and alignment of these constituents in a preferred manner. Development of such information requires measurements on three orthogonal planes and is beyond the scope of this practice.

1.2 This practice specifically addresses the problem of producing stereological data when the features of the constituents to be measured make attainment of statistically reliable data difficult.

1.3 This practice deals only with the recommended test methods and nothing in it should be construed as defining or establishing limits of acceptability.

1.4 The measured values are stated in SI units, which are to be regarded as standard. Equivalent inch-pound values are in parentheses and may be approximate.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- E 3 Methods of Preparation of Metallographic Specimens<sup>2</sup>
- E 7 Terminology Relating to Metallography<sup>2</sup>
- E 45 Test Methods for Determining the Inclusion Content of Steel<sup>2</sup>
- E 768 Practice for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel<sup>2</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 For definitions of terms used in this practice, see Terminology E 7.

#### 3.2 Symbols: Symbols:

- $\bar{A}$  = the average area of inclusions or particles,  $\mu\text{m}^2$ .
- $A_A$  = the area fraction of the inclusion or constituent.
- $A_i$  = the area of the detected feature.
- $A_T$  = the measurement area (field area,  $\text{mm}^2$ ).
- $H_T$  = the total projected length in the hot-working direction of the inclusion or constituent in the field,  $\mu\text{m}$ .
- $\bar{L}$  = the average length in the hot-working direction of the inclusion or constituent,  $\mu\text{m}$ .
- $L_T$  = the true length of scan lines, pixel lines, or grid lines (number of lines times the length of the lines divided by the magnification), mm.
- $n$  = the number of fields measured.
- $N_A$  = the number of inclusions or constituents of a given type per unit area,  $\text{mm}^2$ .
- $N_i$  = the number of inclusions or constituent particles or the number of feature interceptions, in the field.
- $N_L$  = the number of interceptions of inclusions or constituent particles per unit length (mm) of scan lines, pixel lines, or grid lines.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 03.01.

$PP_i$	= the number of detected picture points.
$PP_T$	= the total number of picture points in the field area.
$s$	= the standard deviation.
$V_V$	= the volume fraction.
$\bar{X}$	= the mean of a measurement.
$X_i$	= an individual measurement.
$\lambda$	= the mean free path ( $\mu\text{m}$ ) of the inclusion or constituent type perpendicular to the hot-working direction.
$\Sigma X$	= the sum of all of a particular measurement over $n$ fields.
$\Sigma X^2$	= the sum of all of the squares of a particular measurement over $n$ fields.
95 % CI	= the 95 % confidence interval.
% RA	= the relative accuracy, %.

#### 4. Summary of Practice

4.1 The indigenous inclusions or second-phase constituents in steels and other metals are viewed with a light microscope or a scanning electron microscope using a suitably prepared metallographic specimen. The image is detected using a television-type scanner tube (solid-state or tube camera) and displayed on a high resolution video monitor. Inclusions are detected and discriminated based on their gray-level intensity differences compared to each other and the unetched matrix. Measurements are made based on the nature of the discriminated picture point elements in the image.<sup>3</sup> These measurements are made on each field of view selected. Statistical evaluation of the measurement data is based on the field-to-field or feature-to-feature variability of the measurements.

#### 5. Significance and Use

5.1 This practice is used to assess the indigenous inclusions or second-phase constituents of metals using basic stereological procedures performed by automatic image analyzers.

5.2 This practice is not suitable for assessing the exogenous inclusions in steels and other metals. Because of the sporadic, unpredictable nature of the distribution of exogenous inclusions, other methods involving complete inspection, for example, ultrasonics, must be used to locate their presence. The exact nature of the exogenous material can then be determined by sectioning into the suspect region followed by serial, step-wise grinding to expose the exogenous matter for identification and individual measurement. Direct size measurement rather than application of stereological methods is employed.

5.3 Because the characteristics of the indigenous inclusion population vary within a given lot of material due to the influence of compositional fluctuations, solidification conditions and processing, the lot must be sampled statistically to assess its inclusion content. The largest lot sampled is the heat lot but smaller lots, for example, the product of an ingot, within the heat may be sampled as a separate lot. The sampling of a given lot must be adequate for the lot size and characteristics.

5.4 The practice is suitable for assessment of the indigenous inclusions in any steel (or other metal) product regardless of its

size or shape as long as enough different fields can be measured to obtain reasonable statistical confidence in the data. Because the specifics of the manufacture of the product do influence the morphological characteristics of the inclusions, the report should state the relevant manufacturing details, that is, data regarding the deformation history of the product.

5.5 To compare the inclusion measurement results from different lots of the same or similar types of steels, or other metals, a standard sampling scheme should be adopted such as described in Practice E 45.

5.6 The test measurement procedures are based on the statistically exact mathematical relationships of stereology<sup>4</sup> for planar surfaces through a three-dimensional object examined using reflected light (see Note 1).

5.7 The orientation of the sectioning plane relative to the hot-working axis of the product will influence test results. In general, a longitudinally oriented test specimen surface is employed in order to assess the degree of elongation of the malleable (that is, deformable) inclusions.

5.8 Oxide inclusion measurements for cast metals, or for wrought sections that are not fully consolidated, may be biased by partial or complete detection of fine porosity or microshrinkage cavities and are not recommended. Sulfides can be discriminated from such voids in most instances and such measurements may be performed.

5.9 Results of such measurements may be used to qualify material for shipment according to agreed upon guidelines between purchaser and manufacturer, for comparison of different manufacturing processes or process variations, or to provide data for structure-property-behavior studies.

#### 6. Interferences

6.1 Voids in the metal due to solidification, limited hot ductility, or improper hot working practices may be detected as oxides because their gray level range is similar to that of oxides.

6.2 Exogenous inclusions, if present on the plane-of-polish, will be detected as oxides and will bias the measurements of the indigenous oxides. Procedures for handling this situation are given in 12.5.9.

6.3 Improper polishing techniques that leave excessively large scratches on the surface, or create voids in or around inclusions, or remove part or all of the inclusions, or dissolve water-soluble inclusions, or create excessive relief will bias the measurement results.

6.4 Dust, pieces of tissue paper, oil or water stains, or other foreign debris on the surface to be examined will bias the measurement results.

6.5 If the programming of the movement of the automatic stage is improper so that the specimen moves out from under the objective causing detection of the mount or air (unmounted specimen), measurements will be biased.

6.6 Vibrations must be eliminated if they cause motion in the image.

6.7 Dust in the microscope or camera system may produce

<sup>3</sup> Vander Voort, G. F., "Image Analysis," Vol 10, 9th ed., *Metals Handbook: Materials Characterization*, ASM, Metals Park, OH, 1986, pp. 309–322.

<sup>4</sup> Underwood, E. E., *Quantitative Stereology*, Addison-Wesley Publishing Co., Reading, MA, 1970.

## E 1245

spurious indications that may be detected as inclusions. Consequently, the imaging system must be kept clean.

### 7. Apparatus

7.1 A reflected light microscope equipped with bright-field objectives of suitable magnifications is used to image the microstructure. The use of upright-type microscope allows for easier stage control when selecting field areas; however, the specimens will require leveling which can create artifacts, such as scratches, dust remnants and staining, on the polished surface (see 12.2.1). The use of inverted microscopes usually result in a more consistent focus between fields, thereby, requiring less focussing between fields and a more rapid completion of the procedure. A scanning electron microscope also may be used to image the structure.

7.2 A programmable automatic stage to control movement in the x and y directions without operator attention is recommended (but not mandatory) to prevent bias in field selection and to minimize operator fatigue.

7.3 An automatic focus device may also be employed if found to be reliable. Such devices may be unreliable when testing steels or metals with very low inclusion contents.

7.4 An automatic image analyzer with a camera of adequate sensitivity is employed to detect the inclusions, perform discrimination, and make measurements.

7.5 A computer is used to store and analyze the measurement data.

7.6 A printer is used to output the data and relevant identification/background information in a convenient format.

7.7 This equipment must be housed in a location relatively free of airborne dust. High humidity must be avoided as staining may occur; very low humidity must also be avoided as static electricity may damage electronic components. Vibrations, if excessive, must be isolated.

### 8. Sampling

8.1 In general, sampling procedures for heat lots or for product lots representing material from a portion of a heat lot are the same as described in Practice E 45 (Microscopical Methods) or as defined by agreements between manufacturers and users.

8.2 Characterization of the inclusions in a given heat lot, or a subunit of the heat lot, improves as the number of specimens tested increases. Testing of billet samples from the extreme top and bottom of the ingots (after discards are taken) will define worst conditions of oxides and sulfides. Specimens taken from interior billet locations will be more representative of the bulk of the material. Additionally, the inclusion content will vary with the ingot pouring sequence and sampling should test at least the first, middle and last ingot teemed. The same trends are observed in continuously cast steels. Sampling schemes must be guided by sound engineering judgment, the specific processing parameters, and producer-purchaser agreements.

### 9. Test Specimens

9.1 In general, test specimen orientation within the test lot is the same as described in Practice E 45 (Microscopical Methods). The plane-of-polish should be parallel to the hot-working axis and, most commonly, taken at the quarter-thickness

location. Other test locations may also be sampled, for example, subsurface and center locations, as desired or required.

9.2 The surface to be polished should be large enough in area to permit measurement of at least 100 fields at the necessary magnification. Larger surface areas are beneficial whenever the product form permits. A minimum polished surface area of 160 mm<sup>2</sup> (0.25 in.<sup>2</sup>) is preferred.

9.3 Thin product forms can be sampled by placing a number of longitudinally oriented pieces in the mount so that the sampling area is sufficient.

### 10. Specimen Preparation

10.1 Metallographic specimen preparation must be carefully controlled to produce acceptable quality surfaces for image analysis. Guidelines and recommended practices are given in Methods E 3, and Practices E 45 and E 768.

10.2 The polishing procedure must not alter the true appearance of the inclusions on the plane-of-polish by producing excessive relief, pitting, cracking or pullout. Minor fine scratches, such as from a 1- $\mu$ m diamond abrasive, do not usually interfere with inclusion detection but heavier scratches are to be avoided. Proper cleaning of the specimen is necessary. Use of automatic grinding and polishing devices is recommended.

10.3 Establishment of polishing practices should be guided by Practice E 768.

10.4 Inclusion retention is generally easier to accomplish if specimens are hardened. If inclusion retention is inadequate with annealed, normalized, or low hardness as-rolled specimens, they should be subjected to a standard heat treatment (hardening) cycle, appropriate for the grade. Because inclusion retention and cracking at carbides may be a problem for certain steels in the as-quenched condition, tempering is recommended; generally, a low tempering temperature, for example, 200–260°C (400–500°F), is adequate.

10.5 Mounting of specimens is not always required depending on their size and shape and the available equipment; or, if hand polishing is utilized for bulk specimens of convenient size and shape.

10.6 The polished surface area for mounted specimens should be somewhat greater than the area required for measurement to avoid edge interferences. Unmounted specimens generally should have a surface area much greater than required for measurement to facilitate leveling using the procedure described in 12.1.1.

10.7 Etching of specimens is not desired for inclusion assessment.

### 11. Calibration and Standardization

11.1 Use a stage micrometer to determine the size of the frame to calibrate the image analyzer and to determine the overall magnification of the system for each objective.

11.2 Follow the manufacturer's recommendations in adjusting the microscope light source and setting the correct level of illumination for the television pick-up camera.

11.3 The flicker method of switching back and forth between the inclusion image and the detected image is recommended to establish the correct setting of the gray-level threshold controls as described in 12.2.1. Inspection of the gray



level histogram of the microstructure can be used to define the gray level range and threshold settings for the inclusion or constituent types (see 12.2.1). These settings are verified by the flicker method.

## 12. Procedure

### 12.1 *Setting Up the Microscope:*

12.1.1 Place the specimen on the microscope stage so that the specimen surface is perpendicular to the optic axis. With an inverted-type microscope, simply place the polished face down on the stage plate and hold it in place with the stage clamps. With an upright-type microscope, place the specimen on a slide and level the surface using clay or plasticene between the specimen and slide. If tissue paper is placed between the specimen surface and the ram of the leveling press, small pieces of tissue paper may adhere to the surface during flattening and produce artifacts that affect measurements. In some cases, adherent tissue paper can be blown off the specimen surface. An alternative leveling procedure to avoid this problem is to place an aluminum or stainless steel ring form of appropriate diameter, that has been flattened slightly in a vise to an oval shape, between the specimen and the ram. If the specimen was mounted, the ring form will rest only on the surface of the mount. If the specimen is unmounted but with a surface area substantially greater than required for measurement, the ring form can rest on the outer edges of the specimen during leveling and thus not affect the measurement area. Some upright-type microscopes can be equipped with an autoleveling stage for mounted specimens.

12.1.2 For an image analyzer that uses the TV-raster lines to make intercept counts, align the specimen on the stage so that the longitudinal direction is parallel to the  $y$  direction of the stage and the inclusions are oriented vertically on the monitor screen. For a software-based system, the longitudinal direction of the specimen may be oriented parallel to either the  $x$  or  $y$  axis of the stage.

12.1.3 The microscope light source should be checked for correct alignment and the illumination intensity should be adjusted to the level required by the television scanner tube.

12.1.4 Adjust the magnification of the system to provide adequate resolution of the inclusions with the largest possible field size. Choice of the optimum magnification is a compromise between resolution and field-to-field measurement variability. Higher magnification objectives have higher numerical aperture ratings and provide improved resolution. However, as magnification increases, the field-to-field measurement variability increases, which increases the standard deviation of the measurement. Also, as the magnification increases, the field area decreases. For example, if the magnification is doubled, four times as many fields must be measured to cover the same test area. In general, the lower the inclusion content, the higher the required magnification, and vice-versa. Intermediate magnification objectives (for example, 32 $\times$ , 40 $\times$ , 50 $\times$ , 60 $\times$ , and 80 $\times$ ) provide the best combination of resolution and field area. Avoid use of lower magnification objectives that will not permit detection of the smaller inclusions. Use the same objective for all measurements of specimens within a lot. It is recommended that the same objective be used for all measurements of specimens with the same level of inclusion content,

for example, 32 $\times$  to 50 $\times$  objectives for grades with large amounts of inclusions, such as free-machining grades, and 50 $\times$  to 80 $\times$  objectives for vacuum degassed, ladle-refined, or double-melt grades.

12.1.5 Select the optimum magnification and adjust the light source for best resolution. If necessary, enable the shading correction adjustment for chosen objective.

### 12.2 *Setting the Densitometer:*

12.2.1 Gray-level threshold settings are selected to permit independent detection of sulfides and oxides, or a specific discrete second phase, using the "flicker method" of switching back and forth between the inclusion image and the thresholded image. The threshold limits are set for the oxides and the sulfides so that the inclusions are detected without enlargement of the larger inclusions. In some instances, the threshold settings may require a minor compromise between detection of the smallest inclusions and overdetection of the largest inclusions. The chosen threshold settings should be tried on inclusions in a number of fields before beginning the analysis. The threshold range for oxides is close to the black end of the reflectance scale while the range for sulfides is somewhat higher. An alternate approach to establish the threshold settings is to develop a gray-level reflectance histogram of the inclusion or constituent types present, as well as the matrix (usually in the as-polished condition). The histogram is used to identify the start (darkest gray) and end (lightest gray) of the gray level range for each inclusion or constituent type and the intersection gray level for two inclusions or constituent types with overlapping gray level ranges. Verify these settings by use of the flicker method for several randomly selected fields.

12.2.2 For steels with very low inclusion contents and very small inclusions, it may not be possible to reliably separate oxides from sulfides. In such cases, detect all inclusions without attempting a separation. For other metals containing inclusions of one or more types, adjust the threshold settings to separate them by type or collectively as required by the particular application.

### 12.3 *Stage Movement:*

12.3.1 The stage controls are set to move the specimen in a square or rectangular pattern without running off the specimen surface. Depending on the area of the plane-of-polish and the number of fields to be measured, the fields can be aligned contiguously or spaced apart somewhat. Field areas should not be overlapped. If the fields are aligned contiguously over a square or rectangular area, after examination of the fields, the detected features in these fields can be added together to minimize edge correction problems.

12.3.2 For a given inclusion content, the statistical precision of the measurements improves as the area measured increases. The measured area depends on the size of each field, which decreases with increasing magnification, and the number of fields measured. The number of fields measured should be based on the relative accuracy of the measurements as defined in 13.4 or by agreement between producer and purchaser.

12.3.3 When the sections are stacked within a mount to produce the desired measurement area, stage movement must be carefully adjusted so that the interface between two adjacent specimens is not included within the measurement field.

#### 12.4 *Setting Up the Computer:*

12.4.1 The computer program developed for data input, image analyzer control, measurement, and data analysis is read into the central processing unit to commence analysis. Input data regarding the specimen identity, data requester, calibration constant, date, number of fields, field spacing, and so forth, are entered.

#### 12.5 *Measurement of Stereological Parameters:*

12.5.1 All the stereological measurements can be performed as field measurements. Two measurements, the inclusion or constituent length and area, also can be performed as feature-specific measurements, if desired, although this may be slower.

12.5.2 For each field, focus the image (manually or automatically) and measure the area fraction of the detected oxides and sulfides by dividing the detected area of each by the area of the measurement field or by dividing the total number of detected picture points by the total number of picture points in each field. The volume fraction is equal to the area fraction as defined by:

$$V_v = A_A = \frac{A_i}{A_T} = \frac{PP_i}{PP_T} \quad (1)$$

The volume fraction may be expressed as a percentage, if desired, by multiplying the area fraction or point fraction by 100. Store in the computer memory the volume fraction and the volume fraction squared of the sulfides and oxides.

12.5.3 For each field, count the number of discrete oxides and sulfides and divide each number by the field area ( $\text{mm}^2$ ) to define the number per unit area in accordance with:

$$N_A = \frac{N_i}{A_T} \quad (2)$$

A counting protocol must be chosen to correct for edge effects so that an inclusion or constituent lying across a field boundary is counted only once. Store in the computer memory the number per unit area and the number per unit area squared of the oxides and sulfides.

12.5.4 With an image analyzer that utilizes the raster lines for intercept counting, for each field, count the number of feature interceptions of the horizontal scan lines with the vertically oriented oxides and sulfides and divide this number by the total length of the horizontal scan lines per field as:

$$N_L = \frac{N_i}{L_T} \quad (3)$$

For software-based systems, where the pixel rows, an artificially created grid, or some other approach is used to produce intercept counts, the longitudinal direction must be perpendicular to the intercept count direction.  $N_L$  is determined in the same manner except that  $L_T$  must be based on the true length of the pixel rows or the artificial grid lines. For either type of measurement, store in the computer memory  $N_L$  and  $N_L^2$  for each type of inclusion (sulfide and oxide) or constituent.

12.5.5 If field measurements are employed to determine the inclusion or particle lengths (in the hot working direction), for each field that contains inclusions or particles, measure the total projected length,  $H_T$ , (in  $\mu\text{m}$ ) of each inclusion or

precipitate type. Store in the computer memory the total projected length per field and the number of inclusions per field,  $N_i$ , for each type of inclusion (sulfide and oxide) or constituent.

12.5.6 If feature-specific measurements are used to determine the size of the inclusions or particles, measure the inclusion length (in the hot working direction) and area for each inclusion in the field. A protocol must be employed to correct for edge effects for inclusions or particles lying on a field boundary to ensure that these inclusions or particles are properly measured only once. Store in the computer memory the length,  $L_i$ , and area,  $A_i$ , of each measured inclusion (sulfide and oxide) or constituent type.

12.5.7 Move the stage to the next field, refocus, and repeat the measurements described in 12.5.1-12.5.6. Add the second set of field measurements ( $A_A, A_A^2, N_A, N_A^2, N_L, N_L^2, H_T, N_i$ ) to the first set, and so forth, as the number of fields increases, to obtain the sum of each measurement and each measurement squared for each inclusion type (oxide and sulfide) or constituent type for  $n$  fields. Alternatively, if computer memory is not a problem, store each field value of  $A_A, N_A$  and  $N_L$ . For the feature-specific measurements  $L_i$  and  $A_i$ , store all of the individual measurements.

12.5.8 For those cases where oxides and sulfides cannot be separated, perform the measurements described in 12.5.1-12.5.6 on all of the inclusions. For other metals, perform the measurements on the constituents present either separately or collectively as required by the application.

12.5.9 If exogenous inclusions are observed on the plane-of-polish of a test specimen, the approximate length and width of the exogenous matter should be measured and reported separately. The depth of the exogenous inclusion can be determined, if desired, by sectioning through the inclusion perpendicular to the original sectioning plane. Because exogenous inclusions are not randomly distributed, stereological measurement methods are not appropriate. A new specimen for stereological measurement of the indigenous inclusions should be prepared from an adjacent section, or after a cut-back, depending on the nature of the application, or as defined by other specifications, or as defined by purchaser-producer agreements.

12.5.10 Ratio parameters, such as the average inclusion area or the mean free path, based on field measurements should be calculated (as described in 13.7, 13.8 and 13.10) only from the mean values of  $\bar{A}_A, \bar{N}_A$  and  $\bar{N}_L$  after all the  $n$  fields have been assessed. Do not determine  $\bar{L}, \bar{A}$  and  $\lambda$  for each field.

### 13. Calculation of Results

13.1 After the desired number of fields,  $n$ , have been measured, calculate the mean value of each field measurement ( $A_A, N_A, N_L$ ) stored in memory by dividing the sum of each measurement for  $n$  fields by  $n$  to determine the average area fraction  $\bar{A}_A$  (as a fraction or as a percentage, as desired), the average number per unit area  $\bar{N}_A$  (per  $\text{mm}^2$ ), and the average number of interceptions per unit length  $\bar{N}_L$  (per mm). Alternatively, if the individual field values of  $A_A, N_A$  and  $N_L$  have been stored, compute the mean of each in the usual way (the sum of the measurements divided by  $n$ ) for each inclusion (oxide and sulfide) or constituent type. The average area  $\bar{A}$  (in  $\mu\text{m}^2$ ) and

## E 1245

the average length  $\bar{L}$  (in  $\mu\text{m}$ ) for oxides and sulfides are calculated by dividing by the number of fields with detectable oxides and sulfides rather than the total number of fields (unless all fields measured have detectable oxides and sulfides).

13.2 If the sum of  $A_A$ ,  $A_A^2$ ,  $N_A$ ,  $N_A^2$ ,  $N_L$ , and  $N_L^2$  have been stored in memory, calculate the standard deviation of the field measures by:

$$s = \left[ \frac{\sum X^2}{n} - \left[ \frac{\sum X}{n} \right]^2 \right]^{1/2} \quad (4)$$

If the individual field measurements of  $A_A$ ,  $N_A$ , and  $N_L$  have been stored in memory, calculate the standard deviation according to:

$$s = \left[ \frac{\sum (X_i - \bar{X})^2}{n - 1} \right]^{1/2} \quad (5)$$

NOTE 2—The dispersion of inclusion field measurement data does not conform to the ideal Gaussian (normal) form and the deviation from normalcy increases as the inclusion content decreases. Thus, the arithmetic standard deviation, calculated by (Eq 4 or Eq 5), does not accurately describe the dispersion of the test values about the arithmetic mean of these measurements. The true data dispersion can be defined by the four-moment method<sup>5</sup> and calculation of the geometric mean, geometric standard deviation, skew, and kurtosis. However, such calculations are beyond the scope of this practice. Although the arithmetic standard deviation does not accurately define the data dispersion for these measurements, it may still be used for comparative purposes as described.

NOTE 3—The standard deviation of the mean free path can only be calculated if the constituent of interest is present in every field (See Note 2 and Note 4).

13.3 Next, calculate the 95 % confidence interval, 95 % CI, for each measurement for both oxides and sulfides by:

$$95 \% \text{ CI} = 2.0s/(n)^{1/2} \quad (6)$$

The value of each measurement is expressed as the mean value plus or minus the 95 % CI.

13.4 Next, calculate the percent relative accuracy, % RA, of each measurement for oxides and sulfides by:

$$\% \text{ RA} = \frac{95 \% \text{ CI}}{\bar{X}} \times 100 \quad (7)$$

The relative accuracy is an estimate of the percent of error of each measurement as influenced by the field-to-field variability of the values.

13.5 If the percent relative accuracy is too high, more fields can be measured. As the area fraction decreases, it will be difficult to obtain percent of RA values below 30 %. For very low area (volume) fractions, percent RA values less than 100 % may be difficult to obtain within a practical measurement time. For most work, the number of fields measured should be in the range of from 200 to 300. A greater number of fields, that is, greater measurement area, may be employed depending on the requirements of the study. However, as the area measured increases, the improvement in the percent of relative accuracy

decreases and, beyond some number of fields (area measured), the improvement in the percent relative accuracy will be small compared to the required effort. For steels with very low inclusion contents where the percent of relative accuracies of the measurements are relatively high, measurement of 200 to 300 fields per specimen on a larger number of specimens is recommended rather than measuring 500 or more fields on a smaller number of specimens. This procedure produces better statistical definition of the inclusions in the lot.

13.6 If the inclusions cannot be separated by type and are analyzed collectively, or if other constituents are analyzed separately or collectively for metals other than steels, apply the analytical treatment as described in 13.1-13.5 as appropriate.

13.7 If field measurements of the total projected length of each inclusion or particle type have been made, calculate the average length of each inclusion or particle type,  $\bar{L}$ , according to:

$$\bar{L} = \frac{\sum H_T}{\sum N_i} \quad (8)$$

where  $H_T$  and  $\bar{L}$  are in  $\mu\text{m}$ .

13.8 If feature-specific measurements of inclusion or constituent areas have not been made, use the average values of  $\bar{A}_A$  and  $\bar{N}_A$  (determined in 13.1) to calculate the average area,  $\bar{A}$ , of each inclusion or particle type by:

$$\bar{A} = \frac{\bar{A}_A}{\bar{N}_A} \quad (9)$$

where  $\bar{A}_A$  must be expressed as a fraction (not as a percentage). Since  $\bar{N}_A$  is the number per  $\text{mm}^2$ , multiply  $\bar{A}$  by  $10^6$  to obtain the mean area in  $\mu\text{m}^2$ .

13.9 If feature-specific measurements of the inclusion or particle length and area have been measured,  $L_i$  and  $A_i$ , respectively, compute the mean,  $\bar{L}$  and  $\bar{A}$ . The individual measurement data may be used to develop frequency histograms of length or area, if desired. The standard deviation of the length or area measurements may be calculated according to Eq 5 (see Note 2). Then, calculate the 95 % confidence interval and the % RA as described in 13.3 and 13.4 for the individual length and area measurements.

13.10 Calculate the mean free path, that is, the mean edge-to-edge distance between inclusions (oxide and sulfide) or particle types, perpendicular to the hot-working axis according to:

$$\lambda = \frac{1 - \bar{A}_A}{\bar{N}_L} \quad (10)$$

where  $\lambda$  is the mean free path perpendicular to the hot working axis in  $\mu\text{m}$  of each inclusion or constituent type,  $\bar{A}_A$  is expressed as a fraction (not as a percentage) and  $\bar{N}_L$  (the mean number of intercepts per mm) must be multiplied by 1000.

## 14. Test Report

14.1 The report should document the identifying information in 12.4.1 regarding the specimen, its origin, data requester, date of analysis, and so forth as needed.

14.2 List the number of fields measured, the area per field and the total measurement area.

14.3 For the measured quantities of each inclusion (sulfides

<sup>5</sup> Vander Voort, G. F., "Inclusion Measurement," *Metallography As A Quality Control Tool*, Phenum Press, NY, 1980, pp. 1-88.



 **E 1245**

and oxides) or constituent particle type, list each measured value, the standard deviation, the 95 % confidence interval, and the percent relative accuracy. For the ratio parameters, list only the calculated mean value. This information may be tabulated in several ways depending on the needs of the application. If individual measurements of length and area have been obtained, these results may be plotted in histogram manner, if desired.

14.4 This format is repeated for each specimen in the lot.

14.5 For all of the specimens in the lot, list the mean values for inclusion (oxide and sulfide) or constituent type per specimen and compute average values of each measurement to produce lot averages.

14.6 For all of the specimens in the lot, compute the standard deviation (in accordance with (Eq 4 or Eq 5)), the 95 % CI (in accordance with (Eq 6)) and the percent RA (in accordance with (Eq 7)) for each measurement type to assess the statistical value of the data. For these calculations,  $n$  is the number of specimens in the lot rather than the number of fields measured.

NOTE 4—The comments in Note 2 (after 13.2) do not apply in this situation because the dispersion of the mean values for each specimen about their mean (heat average) will be Gaussian (normal).

14.7 If the oxides and sulfides cannot be separated and are measured collectively, list the values for all inclusions for each sample and calculate lot averages. If other constituents are analyzed separately or collectively in specimens other than steels, list the measured averages per specimen under the appropriate identifying headings and calculate lot averages. Next, calculate the standard deviation, 95 % CI and percent RA for the lot averages as described in 14.6.

14.8 For the oxides and sulfides, or the total inclusion content if measured collectively, or for other constituents measured, list the lot averages for each measurement type, followed by the standard deviation, the 95 % confidence interval, and the percent relative accuracy.

14.9 Report the location and size of any exogenous inclusions encountered and the action taken to prepare a replacement specimen for stereological measurement of the indigenously inclusions.

## 15. Precision and Bias

15.1 Improper sectioning of the specimen that inclines the plane-of-polish off the longitudinal axis will produce bias in the measurements, particularly the number per unit area, the average area and the average length. Misorientations greater than  $5^\circ$  from the longitudinal axis must be avoided.

15.2 Voids in the microstructure due to solidification, inadequate hot ductility, improper hot working practices, or improper polishing will be detected as oxides and bias the volume fraction and number per unit area to higher values. The average area, average length, and mean free path will be biased towards lower values.

15.3 The presence of dust or other debris on the polished surface or dust in the imaging system will bias results towards higher values.

15.4 Improper stage movement control that permits the objective to measure the mount or free space will bias results.

15.5 Vibrations that affect the detected image will bias results.

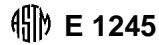
15.6 The choice of the magnification used will influence test results. In general, specimens with large inclusions must be examined at lower magnifications than specimens with a low density of small inclusions. Inclusions that intersect the frame border will be partially detected and a relatively low magnification reduces this problem. For specimens with very small inclusions, higher magnifications must be employed for adequate detection and measurement. However, as the magnification increases, the field area decreases and the field-to-field variability, which influences the standard deviation, increases. As the magnification is decreased, the  $N_A$  value will decrease and the  $\bar{L}$ ,  $\bar{A}$  and  $\bar{\lambda}$  values increase because the smaller inclusions will not be detected. Consequently, results should not be compared if significantly different objective magnifications were used for measurements. The same objective should be used for all measurements of specimens in the same lot. Low magnification objectives should be avoided. Intermediate magnification objectives, e.g.,  $32\times$ ,  $40\times$ ,  $50\times$ ,  $60\times$  and  $80\times$ , provide the best combination of resolution and field size. As the  $A_A$  value decreases, the average inclusion size usually decreases, and higher magnification objectives are required.

15.7 Improper setting of the threshold ranges for detection and discrimination of phases or inclusions will bias results. If the detection scheme appears to be inadequate, the operator should abort the run and reset the threshold levels.

15.8 The precision of the measurements is a direct function of the measurement area, that is, the field size and the number of fields measured. Hence, precision can be improved simply by increasing the number of measurement fields until the percent of relative accuracy reaches the desired level. As the volume fraction of the inclusions decreases, a greater measurement area is required to obtain an acceptable precision. For very low inclusion content specimens, this may require a prohibitive amount of measurement time. A point will be reached where the addition of more fields yields very little improvement in the percent relative accuracy. At this point, it is impractical to continue the analysis. Field areas should be spaced apart or aligned contiguously, never overlapped.

15.9 If the specimen is repolished so that a new parallel plane is measured, reproducibility will not be as good as when the same plane is remeasured.

15.10 Characterization of the inclusion content within a given heat lot, or a subunit of the heat lot, improves as the number of specimens measured increases. For the testing of billets rolled from ingots, the practice of sampling only the extreme tops and bottoms may not be representative of the bulk material. These locations may present extremes in the oxide and sulfide contents. The addition of billet locations within the ingot is recommended to include conditions representative of the bulk of the material. Likewise, for a heat of steel, the inclusion content may vary substantially within the ingot sequence. Hence, as the number of systematically selected test locations increases, the ability to characterize the inclusion content improves. The same rules apply to sampling of continuously cast material.



**16. Keywords**

16.1 exogenous inclusion; feature-specific measurements;  
field measurements; image analysis; inclusions; indigenous

inclusions; light microscopy; oxide; second-phase particles;  
statistical analysis; stereology; sulfide

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