



# 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 values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.14 on Quantitative Metallography.

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### 2. Referenced Documents

2.1 *ASTM Standards*:<sup>2</sup>

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

E45 Test Methods for Determining the Inclusion Content of Steel

E768 Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel

### 3. Terminology

3.1 *Definitions*:

3.1.1 For definitions of terms used in this practice, see Terminology E7.

3.2 *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, $\text{mm}^2$ .

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

$N_i$	= 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.
$PP_i$	= the number of detected picture points.
$PP_T$	= the total number of picture points in the field area.
$s$	= the standard deviation.
$t$	= a multiplier related to the number of fields examined and used in conjunction with the standard deviation of the measurements to determine the 95 % CI
$V_V$	= 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.
$\sum X$	= the sum of all of a particular measurement over $n$ fields.
$\sum X^2$	= 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 E45.

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 micro-shrinkage 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.

<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.

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 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 E45 (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 E45 (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> 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.

9.4 Practice E768 lists two accepted methods for preparing steel samples for the examination of inclusion content using image analysis. The standard also lists a procedure to test the quality of the preparation using differential interference contrast (DIC).

## 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 E3, and Practices E45 and E768.

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 E768.

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