INTERNATIONAL

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Standard Test Methods for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis[1]

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INTRODUCTION

These test methods may be used to determine the mean grain size, or the distribution of grain intercept lengths or areas, in metallic and nonmetallic polycrystalline materials. The test methods may be applied to specimens with equiaxed or elongated grain structures with either uniform or duplex grain size distributions. Either semiautomatic or automatic image analysis devices may be utilized to perform the measurements.

1. Scope

1.1 These test methods are used to determine grain size from measurements of grain intercept lengths, intercept counts, intersection counts, grain boundary length, and grain areas.

1.2 These measurements are made with a semiautomatic digitizing tablet or by automatic image analysis using an image of the grain structure produced by a microscope.

1.3 These test methods are applicable to any type of grain structure or grain size distribution as long as the grain boundaries can be clearly delineated by etching and subsequent image processing, if necessary.

1.4 These test methods are applicable to measurement of other grain-like microstructures, such as cell structures.

1.5 This standard deals only with the recommended test methods and nothing in it should be construed as defining or establishing limits of acceptability or fitness for purpose of the materials tested.

1.6 The sections appear in the following order:

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

[1] These test methods are under the jurisdiction of ASTM Committee E04 on Metallography and are the direct responsibility of Subcommittee E04.14 on Quantitative Metallography.

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1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens
E7 Terminology Relating to Metallography
E112 Test Methods for Determining Average Grain Size
E407 Practice for Microetching Metals and Alloys
E562 Test Method for Determining Volume Fraction by Systematic Manual Point Count
E883 Guide for Reflected–Light Photomicrography
E930 Test Methods for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size)
E1181 Test Methods for Characterizing Duplex Grain Sizes
E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, (*feature-specific measurement, field measurement, flicker method, grain size, gray level,* and *threshold setting*), see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *chord (intercept) length*—the distance between two opposed, adjacent grain boundary intersection points on a straight test line segment that crosses the grain at any location due to random placement of the test line.

3.2.2 *grain intercept count*—determination of the number of times a test line cuts through individual grains on the plane of polish (tangent hits are considered as one half an interception).

3.2.3 *grain boundary intersection count*—determination of the number of times a test line cuts across, or is tangent to, grain boundaries (triple point intersections are considered as 1½ intersections).

3.2.4 *image processing*—a generic term covering a variety of video techniques that are used to enhance or modify contrast, find and enhance edges, clean images, and so forth, prior to measurement.

3.2.5 *skeletonization*—an iterative image amendment procedure in which pixels are removed from the periphery of the grain boundaries (“thinning”), or other features, unless removal would produce a loss of connectivity, until each pixel has no more than two nearest neighbors (except at a junction); this is followed by extension of line ends until they meet other line ends, to connect missing or poorly delineated grain boundaries.

3.2.6 *watershed segmentation*—an iterative image amendment procedure in which each grain, or other features, is eroded to a single pixel, without loosing that pixel ("ultimate erosion”); this is followed by dilation without touching to rebuild the grain structure with a very thin line (grain boundaries) separating each grain.

3.3 *Symbols:*

α = the phase of interest for grain size measurement in a two-phase (constituent) microstructure.

$\bar{A}_\alpha$ = average area of α grains in a two-phase (constituent) microstructure.

$\bar{A}_{A\alpha}$ = area fraction of α grains in a two-phase microstructure.

A_{gi} = total area of grains in the i^{th} field.

A_i = true area of the i^{th} grain; or, the test area of the i^{th} field.

$\bar{A}_i$ = mean grain area for the i^{th} field.

A_{max} = area of the largest observed grain.

A_{ti} = true test area for the i^{th} field.

d = diameter of test circle.

G = ASTM grain size number.

$\bar{l}$ = mean lineal intercept length.

$\bar{l}_\alpha$ = mean lineal intercept length of the α phase in a two-phase microstructure for n fields measured.

$\bar{l}_{\alpha i}$ = mean lineal intercept length of the α phase in a two-phase microstructure for the i^{th} field.

L = test line or scan line length.

$\bar{L}_A$ = mean grain boundary length per unit test area.

L_{Ai} = grain boundary length per unit test area for the i^{th} field.

l_i = intercept length for the i^{th} grain.

$\bar{l}_i$ = mean intercept length for the i^{th} field.

L_i = length of grain boundaries in the i^{th} field.

L_{ti} = true test line or scan line length for the i^{th} field.

L_v = length of grain edges per unit volume.

M = magnification.

n = number of fields measured or the number of grid placements (or the number of any measurements).

N = number of grains measured or the number of grain intercepts counted.

$\bar{N}_A$ = mean number of grains per unit test area for $n$fields measured.

N_{Ai} = number of grains per unit area for the i^{th} field.

$\bar{N}_\alpha$ = mean number of α grains in a two-phase microstructure intercepted by the test lines or scan lines

$N_{\alpha i}$ = number of α grains in a two-phase microstructure intercepted by the test lines or scan lines for the i^{th} field.

N_i = number of grains intercepted by the test lines or scan lines for the i^{th} field; or, the number of grains counted in the i^{th} field.

$\bar{N}_L$ = mean number of grain intercepts per unit length of test lines or scan lines for n fields measured.

N_{Li} = number of grains intercepted per unit length of test lines or scan lines for the i^{th} field.

P_i = number of grain boundaries intersected by the test lines or scan lines for the i^{th} field.

$\bar{P}_L$ = mean number of grain boundary intersections per unit length of test lines or scan lines for $n$fields measured.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

P_{Li} = number of grain boundary intersections per unit length of test lines or scan lines for the i^{th} field.

$\bar{P}_{P\alpha}$ = point fraction of the α grains in a two-phase microstructure.

s_v = grain boundary surface area per unit volume.

s = standard deviation = $[(1/(n-1) \sum (X_i - \bar{X})^2]^{1/2}$.

$\bar{X}$ = any mean value = $\sum X_i / n$.

X_i = any individual measurement.

95 % CI = 95 % confidence interval.

% RA = percent relative accuracy.

4. Summary of Test Methods

4.1 Determination of the mean grain size is based on measurement of the number of grains per unit area, the length of grain boundaries in unit area, grain areas, the number of grain intercepts or grain boundary intersections per unit length, or grain intercept lengths. These measurements are made for a large number of grains, or all of the grains in a given area, within a microscopical field and then repeated on additional fields to obtain an adequate number of measurements to achieve the desired degree of statistical precision.

4.2 The distribution of grain intercept lengths or areas is accomplished by measuring intercept lengths or areas for a large number of grains and grouping the results in histogram fashion; that is, frequency of occurrence versus class limit ranges. A large number of measurements over several fields are required to obtain an adequate description of the distribution.

5. Significance and Use

5.1 These test methods cover procedures for determining the mean grain size, and the distribution of grain intercept lengths or grain areas, for polycrystalline metals and nonmetallic materials with equiaxed or deformed grain shapes, with uniform or duplex grain size distributions, and for single phase or multiphase grain structures.

5.2 The measurements are performed using semiautomatic digitizing tablet image analyzers or automatic image analyzers. These devices relieve much of the tedium associated with manual measurements, thus permitting collection of a larger amount of data and more extensive sampling which will produce better statistical definition of the grain size than by manual methods.

5.3 The precision and relative accuracy of the test results depend on the representativeness of the specimen or specimens, quality of specimen preparation, clarity of the grain boundaries (etch technique and etchant used), the number of grains measured or the measurement area, errors in detecting grain boundaries or grain interiors, errors due to detecting other features (carbides, inclusions, twin boundaries, and so forth), the representativeness of the fields measured, and programming errors.

5.4 Results from these test methods may be used to qualify material for shipment in accordance with guidelines agreed upon between purchaser and manufacturer, to compare different manufacturing processes or process variations, or to provide data for structure-property-behavior studies.

6. Interferences

6.1 Improper polishing techniques that leave excessively large scratches on the surface, or produce excessive deformation or smearing of the microstructure, or produce pull-outs and other defects, will lead to measurement errors, particularly when automatic image analyzers are employed.

6.2 Etching techniques or etchants that produce only partial delineation of the grain boundaries will bias test results and must be avoided.

6.3 Etching techniques or etchants that reveal annealing twins in certain face-centered cubic metals and alloys usually should be avoided if the grain size is to be measured by automatic image analyzers. The presence of twin boundaries can be tolerated when semiautomatic digitizing tablets are utilized but measurement errors are more likely to occur. Etching techniques and etchants that do not delineate twin boundaries are preferred for these specimens. Discrimination of grain boundaries but not twin boundaries using image amendment techniques may be possible with some automatic image analyzers. Such techniques may be employed if the operator can demonstrate their reliability. Each field evaluated using these methods should be carefully examined before (or after) measurements are made and manually edited, if necessary.

6.4 Image processing techniques employed to complete missing or incompletely developed grain boundaries, or to create grain boundaries in grain-contrast/color etched specimens, must be used with caution as false boundaries may be created in the former case, and grain boundaries may not be produced between adjacent grains with similar contrast or color in the latter case.

6.5 Inclusions, carbides, nitrides, and other similar constituents within grains may be detected as grain boundaries when automatic image analyzers are utilized. These features should be removed from the field before measurements are made.

6.6 Orientation-sensitive etchants should be avoided as some boundaries are deeply etched, others are properly etched, while some are barely revealed or not revealed at all. Excessively deep etching with such etchants to bring out the fainter boundaries should not be done because deep etching creates excessive relief (deviation from planar conditions) and will bias certain measurements, particularly grain intercept lengths and grain areas, performed by automatic image analysis and also measurements made with a digitizing tablet.

6.7 Detection of proeutectoid α grains in steels containing ferrite and pearlite (and other alloys with similar structures) by automatic image analyzers can result in detection of ferrite within the pearlitic constituent when the interlamellar spacing is coarse. Use of high magnifications accentuates this problem. For such structures, use the lowest possible magnification, or use semiautomatic devices.

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