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INTERNATIONAL

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Standard Practice for Determining Average Grain Size Using Electron Backscatter Diffraction (EBSD) in Fully Recrystallized Polycrystalline Materials[1]

This standard is issued under the fixed designation E2627; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is used to determine grain size from measurements of grain areas from automated electron backscatter diffraction (EBSD) scans of polycrystalline materials.

1.2 The intent of this practice is to standardize operation of an automated EBSD instrument to measure ASTM *G* directly from crystal orientation. The guidelines and caveats of E112 apply here, but the focus of this standard is on EBSD practice.

1.3 This practice is only applicable to fully recrystallized materials.

1.4 This practice is applicable to any crystalline material which produces EBSD patterns of sufficient quality that a high percentage of the patterns can be reliably indexed using automated indexing software.

1.5 The practice is applicable to any type of grain structure or grain size distribution.

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

1.7 ~~*The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.*~~ *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.*

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

E7 Terminology Relating to Metallography
E112 Test Methods for Determining Average Grain Size
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope
E1181 Test Methods for Characterizing Duplex Grain Sizes
E1382 Test Methods for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis

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3. Terminology

3.1 *Definitions:*

3.1.1 *cleanup*—Post processing applied to EBSD scan data to reassign extraneous points in the scan grid to neighboring points. The extraneous points are assumed to arise from non-indexed or misindexed EBSD patterns.

3.1.2 *(crystallographic) orientation*—The rotation required to bring the principle axes of a crystal into coincidence with the principle axes assigned to a specimen. For example, in a rolled material with cubic crystal symmetry, it is the set of rotations required to bring the [100], [010] and [001] axes of the crystal into coincidence with the rolling, transverse and normal directions of the specimen. Orientations may be described in terms of various sets of angles, a matrix of direction cosines or a rotation vector.

[1] This practice is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.11 on X-Ray and Electron Metallography.

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[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.

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3.1.3 *electron backscatter diffraction (EBSD).—*A crystalline specimen is placed in a scanning electron microscope (SEM) at a high tilt angle (~70°). When a stationary electron beam is positioned on a grain, the electrons are scattered in a small volume (typically 30nm in the tilt direction, 10nm in the transverse direction and 20 nm in depth for a field emission gun SEM and approximately an order of magnitude larger in the lateral directions for a tungsten filament SEM). Electrons that satisfy Bragg's law are diffracted back out of the specimen. The diffracted electrons strike a phosphor screen (or alternatively a YAG crystal) placed in the chamber. The colliding electrons fluoresce the phosphor and produce a pattern. The pattern is composed of a set of intersecting bands (Kikuchi lines). These bands are indicative of the arrangement of crystal lattice planes in the diffracting crystal volume. Assuming the material is of known crystal structure, the orientation of the crystal within the diffracting volume can be determined.

3.1.4 *EBSD pattern—*An EBSD pattern is composed of a set of intersecting bands. The geometrical arrangement of these bands is indicative of the crystallographic orientation of the crystal lattice within the diffraction volume.

3.1.5 *EBSD scan—*Under computer control, the beam of the SEM is moved to a point on the specimen, an EBSD pattern captured and indexed to determine the crystallographic orientation at the beam location. This process is repeated for a set of points lying on a regular grid.

3.1.6 *grain—*In EBSD, grains have a specific meaning. They are defined as a group of similarly oriented neighboring points on the scan grid. The group is surrounded by a perimeter where misorientation across that perimeter exceeds a specified tolerance value.

3.1.7 *indexing—*The process of identifying the crystallographic orientation of the crystal lattice associated with an EBSD pattern generated by the interaction of the electron beam with that lattice.

3.1.8 *misorientation —*The set of rotations (Euler angles) required to bring one crystal lattice into coincidence with a second crystal lattice.

3.1.9 *misorientation tolerance—*If the angular difference between two neighboring pixels is less than this tolerance value then they are assumed to belong to the same grain.

3.1.10 *orientation Map—*Each point in the scan grid is assigned a color according to its orientation. This forms an image showing the microstructure.

3.1.11 *step size (Δ)* —–The distance between neighboring points on the scan grid.

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4. Summary of Practice

4.1 An EBSD scan is performed on a specimen, post-processing routines are applied to the scan data, and the individual points of the scan are grouped into grains according to their orientation. Average grain size is determined from the field average of grain areas based on the number of points in the EBSD map and the step size.

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5. Significance and Use

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5.1 This practice provides a way to estimate the average grain size of polycrystalline materials. It is based on EBSD measurements of crystallographic orientation which are inherently quantitative in nature. This method has specific advantage over traditional optical grain size measurements in some materials, where it is difficult to find appropriate metallographic preparation procedures to adequately delineate grain boundaries.

6. Apparatus

6.1 An electron backscatter diffraction (EBSD) system mounted on a Scanning Electron Microscope (SEM) is used. The EBSD system is constituted by a low-light sensitive video camera (typically a charge-coupled device or CCD camera). The camera images a medium for detecting the diffracted electrons such as a phosphor screen or YAG crystal. EBSD patterns formed on the detecting medium are imaged using the camera and transmitted to a computer.

6.2 Software capable of reliably indexing an EBSD pattern to determine the crystallographic orientation from the EBSD pattern is needed. The computer and resident software should be capable of rapid collection of orientation data from EBSD patterns.

6.3 Electronics and software to control the beam in the SEM (or the stage, or both) are required to collect orientation data at points on a regular scan grid.

7. Hazards

7.1 There are no hazards specific to this test method. However, SEM operators should be familiar with safe SEM operating procedures to prevent exposure to X rays and coming in contact with the high voltages inherent to SEMs. Care should also be exercised in preparing specimens for EBSD as is the case for specimen preparation for light and electron microscopy.

8. Sampling and Test Specimens

8.1 Specimens should be selected to represent average conditions within a heat lot, treatment lot, or product, or to assess variations anticipated across or along a product or component, depending on the nature of the material being tested and the purpose of the study. Sampling location and frequency can be based upon agreements between the manufacturers and the users.

8.2 Specimens should not be taken from areas affected by shearing, burning, or other processes that will alter the grain structure.

8.3 The surface to be polished should be large enough in area to permit measurement of at least five fields at the desired magnification.

9. Specimen Preparation

9.1 It is important to follow good metallographic preparation procedures for successful EBSD work. EBSD is a surface sensitive technique. The surface should be free from deformation and have minimal topography. Careful mechanical polishing or electropolishing, or both should be performed on specimen surfaces. However, as compared with preparing specimens for grain size measurements based on optical microscopy, the surface should not be etched or treated to produce relief to delineate grain boundaries. This would be disadvantageous for obtaining EBSD patterns that can be indexed. The grain boundaries are delineated by processing of the orientation measurement data.

10. Preparation of Apparatus

10.1 Good practices should be used in the operation of both the SEM and EBSD systems. Particular attention should be given to geometric alignment of the specimen surface with the assumed measurement plane (typically 70° from the horizontal). Misalignments can easily arise from sample preparation or mounting of the sample. It is critical to mitigate such misalignment, as they affect both the accuracy of the orientation measurements as well as the accuracy of the beam position, particularly in the direction of tilt. Accurate measurements of orientation and position are critical for accurate characterization of grain size.

11. Calibration and Standardization

11.1 The EBSD instrumentation should be properly calibrated according to specific manufacturer instructions, including the EBSD imaging system magnification calibration per Practice E766 (SEM magnification calibration).

12. Procedure

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12.1 The first step is to perform an EBSD scan. Some a-priori knowledge of the estimated grain size is helpful in selecting an appropriate scan area and step size. A map should be constructed from the scan data to insure that sensible results are obtained. In particular, the map should be inspected to see if any grains appear speckled. If the automated EBSD software is having difficulty indexing the EBSD patterns, the resulting orientation maps will appear "speckled". Indexing problems arise when the patterns are too weak for the band detection algorithms to accurately detect the bands in the patterns, if the system is poorly calibrated, if the crystal structure data is incorrect, or if the configuration parameters for the indexing algorithms are not optimized. The "speckling" arises because incorrect orientations are obtained from individual patterns. Operators should following good practice as recommended by their EBSD vendors to mitigate such problems.

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12.2 Once the scan data have been collected, a cleanup routine should be applied to the data to assimilate any non-indexed or misindexed points into the surrounding neighborhood grains. The number of points modified by the cleanup process should be monitored. No more than 10% of the points should be modified in the process.

12.3 Points of similar orientation should be grouped together to form grains. A misorientation tolerance value of 5° is recommended to define the orientation similarity, although other misorientation values could be specified in producer/purchaser agreements. After grain grouping, each point in the scan should be associated with a grain. Each grain group is identified as a grain. At least 50 whole grains should be observable in each scan area.

12.4 Set up an EBSD scan so that the average grain contains about 500 points. Count the number of scan points contained within each whole grain, P_i, within the scanned field, for a number of fields (n), until at least 500 grains have been measured. Exclude grains with point counts less than 100. For each grain with P_i exceeding 100 calculate the corresponding area, A_i. For scan data measured on a regular square grid, the area of a grain, A_i, is equal to the number of points in the grain multiplied by the square of the step size:

$$A_i = P_i \Delta^2 \quad (1)$$

For data collected on a regular hexagonal grid, the area of a grain, A_i, is equal to the number of points in the grain multiplied by the square of the step size, Δ^2 multiplied by the square root of three divided by two:

$$A_i = \frac{\sqrt{3}}{2} P_i \Delta^2 \quad (2)$$

Store the areas of each grain in memory or record to a file. Calculate the mean area, $\bar{A}$, for N grains measured in true area units (μm^2 or mm^2):

12.5 A histogram of the frequency of the grain areas can be constructed to determine or illustrate the uniformity of the grain areas and to detect and analyze duplex grain size conditions. The analytical method is described in Test Methods E1181, Appendix X2.

$$\bar{A} = \frac{1}{n} \sum_{i=l}^{N} A_i \quad (3)$$