Designation: ~~E930 – 99 (Reapproved 2015)~~ E930 – 18

Standard Test Methods for
Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size)[1]

This standard is issued under the fixed designation E930; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

INTRODUCTION

Commercial material specifications sometimes include, in size limits for grain structures, the need for identification of the largest grain observed in a sample, often expressed as ALA (as large as) grain size. The methods presented here are for use when the number of large grains is too few for measurement by Test Methods E112. It shall be understood that larger (but unobserved) grains may exist in the local volume sampled.

1. Scope

1.1 These test methods describe simple manual procedures for measuring the size of the largest grain cross-section observed on a metallographically prepared plane section.

1.2 These test methods shall only be valid for microstructures containing outlier coarse grains, where their population is too sparse for grain size determination by Test Methods E112.

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

1.4 *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
E1181 Test Methods for Characterizing Duplex Grain Sizes
2.2 *ASTM Adjuncts:*
ALA Grain Size Visual Aid for Comparison Procedure (One Opaque Print and One Transparency)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 ~~All~~ For definitions of terms used in these test ~~methods are either defined in~~ methods, see Terminology E7~~, or are discussed in~~ ~~3.2~~.

3.2 ~~*Definitions of Terms Specific to This Standard:*~~ Definitions:

3.2.1 *ALA grain, n*—the largest grain observed in a random scatter of individual coarse grains comprising 5 % or less of the specimen area, where the apparent grain size of ~~these~~the coarse ~~grains~~grain(s) differs by 3 or more ASTM grain size numbers from the balance of the microstructure.

3.2.2 *outlier grain, n*—a grain substantially different in size from the predominant grain size in a microstructure; for example, an ALA grain.

4. Significance and Use

4.1 The presence of large grains has been correlated with anomalous mechanical behavior in, for example, crack initiation, crack propagation, and fatigue. Thus there is engineering justification for reporting the ALA grain size.

4.2 These methods shall only be used with the presence of outlier coarse grains, 3 or more ASTM grain size numbers larger than the rest of the microstructure and comprising 5 % or less of the specimen area. A typical example is shown in Annex A1 as Fig. A1.1.

4.3 These methods shall not be used for the determination of average grain size, which is treated in Test Methods E112. Examples of microstructures that do not qualify for ALA treatment are shown in Annex A1 as Fig. A1.2, Fig. A1.3, and Fig. A1.4.

4.4 These methods may be applied in the characterization of duplex grain sizes, as instructed in the procedures for Test Methods E1181.

5. Sampling

5.1 Sampling shall have been performed according to sampling procedures in Test Method E112.

5.2 The generally intended plane of polish is a plane passing through the center of the thickness and exhibiting maximum grain aspect ratio.

5.3 Other polishing planes which may be more useful or predictive in specific products or applications are allowed.

5.4 An unambiguous description of the plane of polish or a reference to a description or drawing of the plane of polish shall be a part of the test report.

5.5 Specimens shall be prepared in accordance with Methods E3 and Practice E407.

6. Procedures

6.1 In 6.2 a comparison procedure is presented with accuracy near to ±1 ASTM grain size number, for the apparent size of the largest grain. For greater accuracy, a measuring procedure is described in 6.3. A manual quantitative method, to serve as referee procedure, is described in 6.4. (The measuring procedure is especially recommended over the comparison procedure when the ALA grain section's shape is substantially different from those shown in Annex A2.)

6.2 *Comparison Procedure:*

6.2.1 Scan the entire microsection at a convenient magnification to locate the larger grains.

6.2.2 Position the largest grain in the middle of the microscope viewing screen, eyepiece, or on a photomicrograph.

6.2.3 Estimate the grain size by comparing the ALA grain to a visual aid that is based on the relationship of area to grain size expressed in Table 1. Examples of visual aids are shown in Annex A2, with their specifications in Annex A3. Fig. A2.1 may be used only at the magnification specified on the aid.

NOTE 1—The use of Test Methods E112 comparison plates is not allowed, since few of the grain sections illustrated correspond to the average area for that grain size number.

6.3 *Measuring Procedure* (for greater accuracy than with comparison procedure):

6.3.1 Locate and position the largest grain in a microscope image or in a photomicrograph, as in 6.2.1 and 6.2.2.

6.3.2 Using a measuring eyepiece, internal reticle, or external scale, as appropriate, measure the maximum caliper diameter and the caliper diameter perpendicular to the maximum caliper diameter.

6.3.3 Multiply the product of these two measurements by 0.785, to obtain the area of an ellipse with axes equal to the caliper diameters at the magnification used.

6.3.4 Divide this area by the square of the magnification used, to obtain the true grain area at 1X.

6.3.5 Compare this area with the grain areas in Table 1. Use the nearest area in the table to obtain the ALA grain size number, unless the next smaller or the next larger area is agreed upon between the interested parties.

NOTE 2—~~Any automatic or semiautomatic measuring device which provides the area of a grain section can also be used within the framework of this manual method.~~

6.4 *Referee Procedure (Image Analysis):*

6.4.1 Photograph the largest grain, using the largest magnification that shows the entire grain in the image area. (In case of uncertainty about which of several grain sections is the largest, photograph them all and carry out the following steps for all of the photographs.)

TABLE 1 Relationship of ALA Grain Area to ALA Micro-Grain Size Number[A]

Area, mm²	Size
2.06	00000 or – 4.0
1.46	−3.5
1.03	0000 or – 3.0
0.703	−2.5
0.516	000 or – 2.0
0.365	−1.5
0.258	00 or – 1.0
0.182	−0.5
0.129	0
0.0912	0.5
0.0645	1.0
0.0456	1.5
0.0323	2.0
0.0228	2.5
0.0161	3.0
0.0114	3.5
0.00807	4.0
0.00570	4.5
0.00403	5.0
0.00285	5.5
0.00202	6.0
0.00143	6.5
0.00101	7.0

[A]Adapted from Test Methods E112, Table 2.

6.4.1.1 Verify that the magnification on the microscope aligns with the magnification selected in the image analysis software.

6.4.1.2 Using the image analysis software, trace the perimeter of the grain. This may be accomplished manually or by automatic detection provided the software has the capability.

6.4.1.3 Verify that the perimeter traced best represents the geometry of the grain and record the area (mm²).

6.4.1.4 Compare the area determined with the area column in Table 1. Use the nearest area in the table to report the ALA grain size number, unless the next smaller or the next larger area is agreed upon between the interested parties.

6.4.1.5 If necessary, retain the photomicrograph that includes a reference scale.

6.5 *Referee Procedure:* *Alternate Referee Procedure (Manual Overlay):*

6.5.1 Photograph the largest grain, using the largest magnification that shows the entire grain in the image area. (In case of uncertainty about which of several grain sections is the largest, photograph them all and carry out the following steps for all of the photographs.)

6.5.2 Apply a transparent overlay containing a square network of grid lines to the photograph, so that the large grain is completely covered by the grid. An interline grid spacing of 5 mm is recommended. Count the number of grid intersections (points) that fall within the large grain being estimated. Points falling on the grain boundary are counted as one half.

6.5.3 Reapply the overlay to the photo at least four more times at different angular placements, each time tallying the grid intersections as in 6.4.2 6.5.2.

6.5.4 The estimated area of the grain section is determined by the following equation:

$$A = \frac{\overline{P(\theta)} \cdot d^2}{M^2}$$

where:

$\overline{P(\theta)}$ = number of points falling within the grain averaged over several angles,
d^2 = area of each small square of the grid with spacing d,
M = magnification of the photomicrograph, and
A = estimated area of the grain section.

6.5.5 Compare the area determined with the area column in Table 1. Use the nearest area in the table to report the ALA grain size number, unless the next smaller or the next larger area is agreed upon between the interested parties.

6.5.6 Retain the photomicrograph, and record the following: grid points counted for each grid placement, $P_i(\theta)$; total grid points counted, $\sum P_i(\theta)$; average number of grid points counted, $\overline{P(\theta)}$; inter-point spacing in grid, d; magnification used, M; measured area of ALA grain, A; and the ALA grain size number.

7. Precision and Bias

7.1 The precision and bias of these methods have not yet been determined.

8. Keywords

8.1 ALA grain; caliper diameter; comparison procedure; ellipse measurement; grain size; largest grain; measuring procedure; metallography; microstructure; outlier grain; point-count procedure

ANNEXES

(Mandatory Information)

A1. MICROSTRUCTURAL EXAMPLES

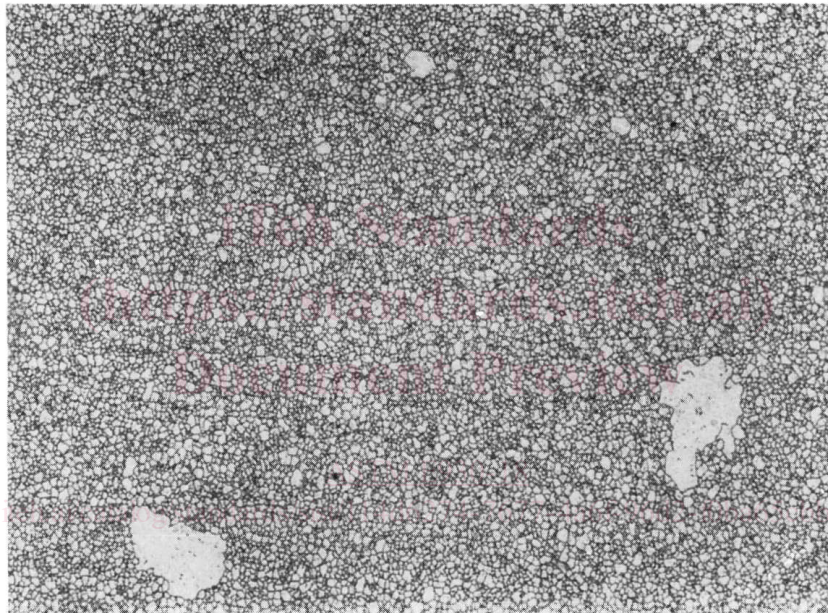NOTE A1.1—These figures are taken from Test Methods E1181, Annex A1.

FIG. A1.1 125×, ALA Condition Rateable to E930