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.

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Standard Test Methods for Characterizing Duplex Grain Sizes[1]

This standard is issued under the fixed designation E1181; 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.

INTRODUCTION

Test methods are well established for the determination of average grain size, and estimation of largest grain size, in products assumed to contain a single log-normal distribution of grain sizes. The test methods in this standard are set forth to characterize grain size in products with any other distributions of grain size.

The term "duplex grain size" is chosen to describe any of these other distributions of grain size, because of its common usage and familiarity. However, the use of that term does not imply that only two grain size distributions exist.

These test methods are equally aimed at describing the nature of the deviation from a single log-normal distribution of grain sizes, and at describing with reasonable accuracy the distributions of sizes that actually exist.

1. Scope

1.1 These test methods provide simple guidelines for deciding whether a duplex grain size exists. The test methods separate duplex grain sizes into one of two distinct classes, then into specific types within those classes, and provide systems for grain size characterization of each type.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. 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.*

1.4 *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
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)

2.2 *ASTM Adjuncts:*

Comparison Chart for Estimation of Area Fractions[3]

3. Terminology

3.1 *Definitions:*

3.1.1 All terms used in these test methods are either defined in Terminology E7, or are discussed in 3.2.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bands or banding*— in grain size, alternating areas of significantly different grain sizes. These areas are usually elongated in a direction parallel to the direction of working.

3.2.2 *grain size*—equivalent in meaning to the average of a distribution of grain sizes.

[1] These test methods are under the jurisdiction of ASTM Committee E04 on Metallography and are the direct responsibility of Subcommittee E04.08 on Grain Size.

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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.

[3] This comparison chart shows different area percentages of light grains among dark grains. Available from ASTM Headquarters. Order Adjunct: ADJE1181.

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3.2.3 *necklace or necklace structure*—individual coarse grains surrounded by rings of significantly finer grains.

3.2.4 *topologically varying*—varying nonrandomly, in some definable pattern; that pattern may be related to the shape of the specimen or product being examined.

4. Summary of Test Method

4.1 These test methods provide means for recognizing the presence of duplex grain size. The test methods separate duplex grain sizes into two classes (randomly varying, and topologically varying), and define specific types of duplex grain sizes within these classes. The test methods provide means for estimating area fractions occupied by distinct grain sizes, and offer existing standard methods (Test Methods E112, Test Methods E930) for determining grain size in specific identified areas. The test methods provide for reporting of specific, distinctive information for each type of duplex grain size. And, as an alternative, the test methods offer a procedure for statistically determining the distribution of all the grain sizes present in a duplex grain size specimen.

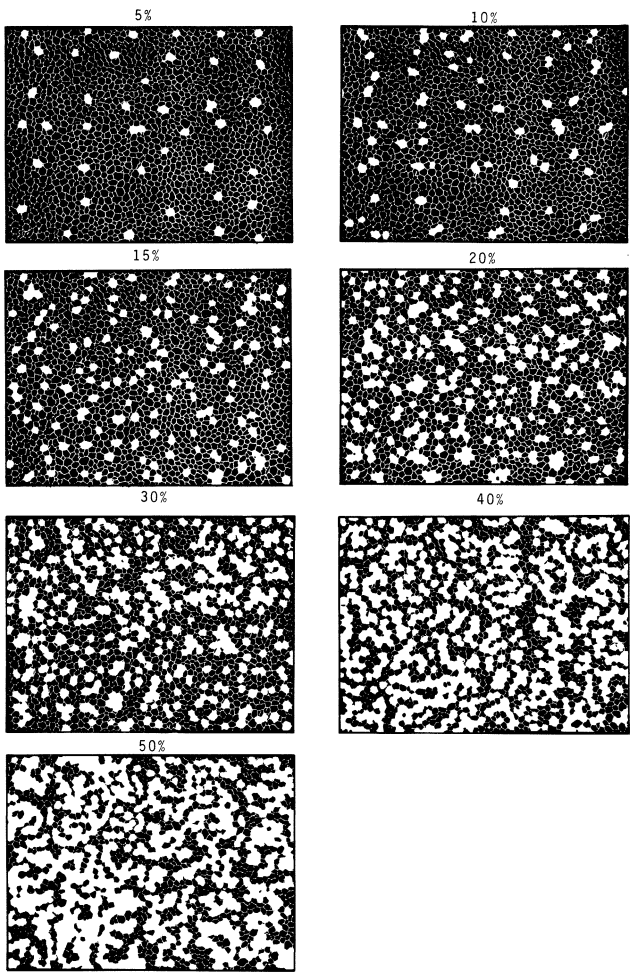

FIG. 1 Comparison Chart for Estimation of Area Fractions[1]
(Showing area percentages of light grains among dark grains)

5. Significance and Use

5.1 Duplex grain size may occur in some metals and alloys as a result of their thermomechanical processing history. For comparison of mechanical properties with metallurgical features, or for specification purposes, it may be important to be able to characterize grain size in such materials. Assigning an average grain size value to a duplex grain size specimen does not adequately characterize the appearance of that specimen, and may even misrepresent its appearance. For example, averaging two distinctly different grain sizes may result in reporting a size that does not actually exist anywhere in the specimen.

5.2 These test methods may be applied to specimens or products containing randomly intermingled grains of two or more significantly different sizes (henceforth referred to as random duplex grain size). Examples of random duplex grain sizes include: isolated coarse grains in a matrix of much finer grains, extremely wide distributions of grain sizes, and bimodal distributions of grain size.

5.3 These test methods may also be applied to specimens or products containing grains of two or more significantly different sizes, but distributed in topologically varying patterns (henceforth referred to as topological duplex grain sizes). Examples of topological duplex grain sizes include: systematic variation of grain size across the section of a product, necklace structures, banded structures, and germinative grain growth in selected areas of critical strain.

5.4 These test methods may be applied to specimens or products regardless of their state of recrystallization.

5.5 Because these test methods describe deviations from a single, log-normal distribution of grain sizes, and characterize *patterns* of variation in grain size, the total specimen cross-section must be evaluated.

5.6 These test methods are limited to duplex grain sizes as identifiable within a single polished and etched metallurgical specimen. If duplex grain size is suspected in a product too large to be polished and etched as a single specimen, macroetching should be considered as a first step in evaluation. The entire macroetched cross-section should be used as a basis for estimating area fractions occupied by distinct grain sizes, if possible. If microscopic examination is subsequently necessary, individual specimens must be taken to allow estimation of area fractions for the entire product cross-section, and to allow determination of grain sizes representing the entire cross-section as well.

5.7 These test methods are intended to be applied to duplex grain *sizes*. Duplex grain *structures* (for example, multiphase alloys) are not necessarily duplex in grain size, and as such are not the subject of these methods. However, the test methods described here for area fraction estimation may be of use in describing duplex grain structures.

6. Apparatus

6.1 Certain items may be helpful or necessary in applying the various procedures of these test methods. These items are

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briefly described below, under the headings of the specific procedures to which they apply.

6.1.1 *Comparison Procedure for Estimation of Area Fractions*—This procedure requires the use of a comparison chart to improve the accuracy of visual estimates of area fractions occupied by distinct grain sizes. This comparison chart is shown in Fig. 1[4]. The chart shows different area percentages of light grains among dark grains.

6.1.2 *Point Count Procedure for Estimation of Area Fractions*—This procedure requires the use of a test grid on a transparent overlay, or in a reticle, that can be superimposed on the specimen image. The grid should consist of equally spaced points formed by the intersection of fine lines. Practice E562 gives examples of such grids, as well as details on recommended grid spacing, and use of the grid.

6.1.3 *Planimetric Procedure for Estimation of Area Fractions*—This procedure requires the use of a planimeter, a device for measuring the areas of irregular polygons. The regions occupied by a distinct grain size are manually outlined on a photomicrograph or transparent overlay. The area of each of those regions is then measured by tracing its outline with the planimeter.[5]

6.1.4 Test Methods E930, Comparison Procedure for Estimation of Largest Grain Size Observed.

6.1.4.1 This procedure requires the use of a visual aid for estimation of the size of the largest grain found in a given metallographic section. That visual aid is shown in Test Methods E930, and is available as an ASTM Adjunct (see Test Methods E930 for details).

6.1.5 Test Methods E930, Measuring Procedure for Estimation of Largest Grain Size Observed.

6.1.5.1 This procedure may require the use of a measuring microscope eyepiece or measuring microscope reticle. These are available from microscope manufacturers.

6.1.6 Test Methods E930, Referee Procedure for Estimation of Largest Grain Size Observed.

6.1.6.1 This procedure requires the use of a test grid on a transparent overlay that can be superimposed on the specimen image. The test grid consists of a square network of grid lines, with a recommended interline spacing of 5 mm. Use of the grid is described in Test Methods E930.

6.1.7 Test Methods E112, Comparison Procedure for Determination of Average Grain Size.

6.1.7.1 This procedure requires the use of grain size comparison charts or overlay transparencies, or grain size comparison reticles fitted into microscopes. Various comparison charts and overlay transparencies are available as ASTM adjuncts (see Test Methods E112 for details).

6.1.7.2 Grain size comparison reticles are available from various manufacturers of microscopes.

6.1.8 Test Methods E112, Intercept Procedures for Determination of Average Grain Size,

6.1.8.1 The Intercept Procedures of Test Methods E112 require the use of patterns of test lines, usually on transparent overlays. The use of these is described in detail in Test Methods E112. A transparency of one such pattern is available as an ASTM adjunct (see Test Methods E112 for details).

6.1.9 *Statistical Determination of Grain Size Distribution:*

6.1.9.1 This procedure requires the use of a test grid on a transparent overlay that can be superimposed on the specimen image. The test grid consists of a series of fine, parallel lines, with an interline spacing of 5 mm. Use of the grid is described in 8.7.

6.1.9.2 This procedure may be carried out using manual measuring and counting techniques, but as such, will be very laborious and time-consuming. This procedure can be carried out much more efficiently through the use of an automated image analysis system with an electronic pencil or cursor, or through the use of a semi-automated image analysis system with a digitizing tablet and electronic pencil or cursor.[6] The use of this equipment is also described in 8.7.

7. Sampling and Test Specimens

7.1 *Sampling:*

7.1.1 These test methods are intended to characterize *patterns* of variation in grain size, when they occur in a given specimen or product. To characterize these patterns accurately, the entire cross-section of the specimen or product must be evaluated.

7.1.2 If variations in grain size occur in a product too large to be polished and etched as a single specimen, individual specimens must be taken to allow estimation of area fractions for the entire product cross-section, and to allow determination of grain sizes representing the entire cross-section as well.

7.2 *Specimen Orientation:*

7.2.1 All of the types of duplex grain size described in this test method (see 3.2 and 8.3) can be detected in a longitudinal specimen orientation (that is, in a plane parallel to the direction of maximum product deformation, during manufacture). Accordingly, the longitudinal orientation is recommended, with one exception. If the specimen being examined is the full cross-section of a round bar, the longitudinal section should not be used to estimate the area fraction occupied by different grain sizes. That estimate can be made most accurately only on a transverse section. For a tubular product, estimates of area fractions made on longitudinal sections are reasonable approximations of the same estimates made on transverse sections. For all other products, area fraction estimates should be equally accurate with either specimen orientation.

7.2.2 Other specimen orientations may be used, provided that their limitations are recognized. For instance, banding present in a given specimen may not be easily recognizable in a transverse orientation.

7.2.3 The specimen orientation used should be reported along with the duplex grain size characterization.

[4] Leidheiser, H., Jr.. and Kim, D. K., "A Chemical Test for Identifying the Fraction of Grains in the Surface of Galvanized Steel Sheet That Have Orientations Approximating (0001)—Importance to Paint Adherence,"*Metallurgical Transactions "B,"* American Society for Metals, Metals Park, OH 44073, December, 1978, p. 590.

[5] A Keuffel & Esser Compensating Polar Planimeter available from drafting equipment suppliers, or equivalent, has been found to be satisfactory for this purpose.

[6] A Zeiss Videoplan System, or its equivalent, has been found satisfactory for this purpose.

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8. Procedure

8.1 *Specimen Preparation*—Prepare specimens according to Methods E3, and etch specimens in accordance with Practice E407. Etch specimens so that all grain boundaries are distinct and easily visible.

8.2 *Preparation of Photomicrographs*—If photomicrographs are required for characterizing duplex grain size, prepare them in accordance with Guide E883.

8.3 *Recognizing and Classifying Duplex Grain Size:*

8.3.1 A random duplex grain size is defined as any of the following:

8.3.1.1 The presence of randomly distributed individual coarse grains, differing in size by three or more ASTM grain size numbers from the average size of the balance of the grains (henceforth referred to as the ALA (As Large As) condition). These individual coarse grains should comprise 5 % or less of the area of the specimen. If they comprise more than 5 % of the area, treat the specimen as described in 8.3.1.3. An example photomicrograph of the ALA condition appears in Fig. A1.1. (See Annex A1.)

8.3.1.2 The presence of an unusually wide range of grain sizes, randomly distributed, with the largest size differing from the smallest size by five or more ASTM grain size numbers (henceforth referred to as the wide-range condition). An example photomicrograph of the wide-range condition appears in Fig. A1.2.

8.3.1.3 The presence of two distinct grain sizes, randomly distributed such that the sizes differ by more than four ASTM grain size numbers, *and* such that the two sizes together comprise 75 % more of the total area of the specimen (henceforth referred to as the bimodal condition). An example photomicrograph of the bimodal condition appears in Fig. A1.3. A further example appears in Fig. X1.1 in Appendix X1.

8.3.2 A topological duplex grain size is defined as any of the following:

8.3.2.1 The presence of a systematic variation in grain size across the section of the product, such that the average grain size differs from one area to another by three or more ASTM grain size numbers; or, the presence of different grain sizes in specific areas of a product cross-section (for example, coarse grains resulting from germinative grain growth at areas of critical strain), such that the grain size in those specific areas differs from the grain size in the bulk of the cross-section by three or more ASTM grain size numbers (both conditions henceforth referred to as the cross-section condition). An example photomicrograph of a cross-section condition appears in Fig. A1.4.

8.3.2.2 The presence of individual coarse grains, each surrounded by rings of finer grains, the coarse and fine grains differing in size by three or more ASTM grain size numbers (henceforth referred to as the necklace condition). An example photomicrograph of the necklace condition appears in Fig. A1.5.

8.3.2.3 The presence of bands of distinct grain sizes, such that the sizes differ by three or more ASTM grain size numbers (henceforth referred to as the banding condition). An example photomicrograph of the banding condition appears in Fig. A1.6.

8.4 *Estimation of Area Fractions:*

8.4.1 Estimation of the area fractions (expressed as percentages of the total area) occupied by the distinct grain sizes in a given specimen is the most subjective part of characterizing duplex grain sizes, and is the part most prone to error. Four procedures for estimating area fraction are described, the simplest to apply resulting in the least precision, and the most complicated resulting in the greatest precision. One, the Direct Measurement Procedure (see 8.4.6), is applicable only in specific cases.

8.4.2 Because this test method is aimed at characterizing variations in grain size within a specimen, the following precautions should be taken in applying any of the area fraction estimation procedures to the specimen:

8.4.2.1 For the random class of duplex grain sizes, apply the chosen estimation procedure to a minimum of five randomly selected areas of the specimen.

8.4.2.2 For the topological class of duplex grain sizes, apply the chosen estimation procedure to all of the specimen area, if possible. If this is not practical, apply the estimation procedure to as much of the specimen as is reasonable, but recognize that, by not sampling all of the specimen area, some undefined bias may be introduced into the estimate.

(a) For example, if banding is present in a full-thickness specimen taken from a plate product, that banding may vary in severity from surface to center of the specimen. The most precise estimate of the area fractions occupied by each grain size will be obtained by evaluating the entire surface of that specimen. Selecting only part of the specimen surface may inadvertently eliminate areas that have high (or low) concentrations of one of the grain sizes, thus biasing the estimate.

(b) In this example, the entire specimen surface could be examined either by using a single, low-magnification image, or by using sufficient multiple images at higher magnification. If neither of these is practical, then, as an absolute minimum, the area of the specimen to be examined must include one complete traverse from surface to surface of the original plate section. If additional traverses can be made, they will improve the precision of the final area fraction estimate.

8.4.3 *Comparison Procedure:*

8.4.3.1 Do not estimate area fractions occupied by distinct grain sizes based solely on unaided visual scanning of a specimen. Such a method is subject to considerable error. Reduce that error by using graded area fraction comparison figures (see Fig. 1 for an example). Compare such figures with eyepiece or projected images from a microscope, or with photomicrographs. Use the lowest magnification that allows visual resolution of the coarse- and fine-grained *areas* as distinct regions. Note that it is not necessary to resolve the individual fine grains in the image. The *area occupied by* the fine grains is what must be determined.

8.4.3.2 Apply the comparison figures to as many areas of the specimen as recommended in 8.4.2, and calculate the average estimated area fractions (expressed as percentages of the total area) occupied by the distinct grain sizes, over the number of areas examined.

8.4.4 *Point Count Procedure:*

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8.4.4.1 Practice E562 may be used to estimate the area fractions occupied by distinct grain sizes. Apply this practice to projected images from a microscope, or to photomicrographs. Begin by outlining the distinct grain size regions in a given image, either on a transparent overlay placed over the projected image, or directly on a photomicrograph. Such outlining will simplify decisions during point counting, and thus speed the counting process. On the overlay, also mark the outline of the total field of view (the limits of the image). Next apply a regular two-dimensional grid to the outlined image. The grid spacing should be matched to the image magnification to fulfill the recommendations of Practice E562. Count the number of grid points falling within the particular grain size region being evaluated. Count any points falling on the outline of the region as one half. If there is any doubt whether a point falls inside or outside the outlined region, count the point as one half. Estimate the area fraction for the grain size region being evaluated as the number of grid points falling within the region, divided by the total number of grid points that lie within the total image outline (the grid must be large enough to completely cover the total image).

8.4.4.2 Repeat these steps for as many areas of the specimen as recommended in 8.4.2, and calculate the average estimated area fractions occupied by the distinct grain sizes, over the number of areas examined.

8.4.5 *Planimetric Procedure:*

8.4.5.1 A planimeter (a device for measuring areas of irregular polygons) may be used to measure area fractions occupied by distinct grain sizes on specific images of a specimen. Apply this procedure to projected images from a microscope, or to photomicrographs. Begin by manually outlining all of the areas occupied by one of the distinct grain sizes in a given image, either on a transparent overlay placed over the projected image, or directly on the photomicrograph. On the overlay, also mark the outline of the total field of view (the limits of the image). Then use the planimeter to measure the area enclosed within each outlined region. Also use the planimeter to measure the total area of the image. Estimate the area fraction for the grain size being evaluated as the total of the areas of the outlined regions, divided by the total image area.

8.4.5.2 Repeat these steps for as many areas of the specimen as recommended in 8.4.2, and calculate the average estimated area fractions occupied by the distinct grain sizes, over the number of areas examined.

8.4.5.3 This same procedure may also be followed using a digitizing tablet or other types of image analysis equipment.

8.4.6 *Direct Measurement Procedure:*

8.4.6.1 This procedure may be applied to topological duplex specimens that show surface layers of differing grain size. Make at least 10 measurements (at different locations) of the depth of a given surface layer, and calculate the average depth from those measurements. Use this average and the overall product dimensions to calculate an estimated area fraction for that surface layer.

8.4.6.2 As an example, suppose a plate product 19.0 mm thick contains a layer of grains of ASTM No. 0 at each surface, with the grains in the balance of the cross-section being ASTM No. 4. Suppose that measurements indicate that the surface layers each average 3.2 mm in depth. The layer depth of 3.2 mm divided by the total product thickness of 19.0 mm, would give an estimate of 16.8% for the area fraction represented by *each* surface layer.

8.4.6.3 For another example, suppose a tubular product with an outside diameter of 115.0 mm and a wall thickness of 7.2 mm showed a surface layer at the outside diameter of grains of ASTM No. 1, with the balance of the wall thickness showing grains of ASTM No. 6. Further suppose that measurements indicate an average depth of that surface layer of 1.0 mm. A tube whose wall thickness is small compared to its outside diameter may be treated as equivalent to a product of rectangular cross-section, in that a surface layer of a given depth at the outside diameter covers essentially the same area as a layer of the same depth at the inside diameter. Accordingly, the layer depth of 1.0 mm, divided by the total wall thickness of 7.2 mm, would give an estimate of 13.9 % as the area fraction represented by the coarse-grained surface layer.

8.5 *Determination of Grain Sizes:*

8.5.1 Determine the size of individual coarse grains according to Test Methods E930. The Comparison Procedure is the simplest, but offers the least accuracy. The Measuring and Referee Procedures are more difficult to apply, but offer greater accuracy.

8.5.2 Determine average grain sizes, when required, according to the procedures set forth in Test Methods E112. Of these, the Comparison Procedure is the simplest, but offers the least precision. The Planimetric Procedure of E112 does not lend itself to determination of grain size in distinct small areas, and so should not be used. Any of the Intercept Procedures of E112 may be used, within the regions of distinct grain size. These procedures will be more difficult to apply than the Comparison Procedure of E112, but will offer greater precision.

8.5.3 Different applications of Test Methods E930 and Test Methods E112 are used, depending on the class and type of duplex grain size identified. These different applications are described in 8.6.

8.6 *Characterizing and Reporting Duplex Grain Sizes:*

8.6.1 Begin a duplex grain size report with the specimen orientation used for the evaluation. Use the abbreviation "Long." for longitudinal, and the abbreviation "Trans." for transverse.

8.6.2 For the random—ALA condition, use Test Methods E930 to estimate the size of the largest individual grain present in the specimen. Use Test Methods E112 to determine the average size of the balance of the grains in the specimen.

8.6.2.1 Report these results as "Duplex, ALA, ASTM No. ____, ALA ASTM No. ____", with the proper values inserted in the blanks. An example of a complete report might read:

"Long. Duplex, ALA, ASTM No. 6,
ALA ASTM No. 1"

8.6.3 For the random—wide-range condition, use Test Methods E112 to determine the average grain size of the specimen. Use Test Methods E112 to determine the size of the smallest grains found in the specimen, and Test Methods E930 to estimate the size of the largest grain found in the specimen.

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