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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 *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.*

1.7 The sections appear in the following order:

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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.

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.

[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.

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; i.e., frequency of occurrence vs. 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.

6.9 If photographic images are measured using a digitizing tablet, uncertainties in the magnification (particularly when enlargements are used) will bias the test results.

6.10 Vibrations, if present, can blur the image and bias test results and must be minimized or eliminated when using automatic image analysis.

6.11 Dust in the microscope or camera system may produce spurious detail in the image that may be detected as a grain boundary, particularly on automatic image analyzers, and will bias the test results. Consequently, the imaging system must be kept clean.

6.12 Nonuniform illumination can influence feature detection and thresholding using automatic image analyzers. Prior to analysis, center the light source (as described in the operating instructions for the microscope) and adjust the field and aperture diaphragms for best image clarity. Digital correction methods for nonuniform illumination may be used subsequently; however, these methods should not be used in lieu of proper microscope alignment and adjustment.

7. Apparatus

7.1 A high quality, research-type reflected light microscope is most commonly used to image the microstructure (images from electron metallographic instruments may also be used). If a digitizing tablet is utilized for the measurements, illumination modes other than bright field may be useful for certain specimens. For example, for optically anisotropic materials that are difficult to etch, crossed polarized light may be required to observe the grain structure. Such images exhibit grain contrast or color differences between grains rather than grain boundary delineation. These images, which usually exhibit low light intensities, can be measured using a digitizing tablet but may be more difficult to measure with automatic image analyzers.

7.1.1 If an automatic image analyzer is employed to perform the measurements, an upright-type metallurgical microscope is preferred over an inverted microscope due to the greater ease in observing the specimen surface with automatic stage movement.

7.2 A semiautomatic digitizing tablet with a measurement resolution of at least 0.1 mm can be used to measure the grain size. A variety of approaches can be employed. The simplest is to fix a photograph (usually an enlargement) to the tablet surface and place a suitable grid over the photograph (placement done without bias), tape down the corners of the grid, and use the cursor, fitted with fine cross hairs, to measure the appropriate features. Alternatively, the grid can be placed on an eyepiece reticle. The cursor is moved over the tablet surface and the microscopist can see the illuminated cross hairs in the cursor through the eyepieces over the field of view and grid pattern. A third approach is to transfer the microstructural image, test grid image and cursor image to a television monitor. The microscopist moves the cursor across the tablet surface while watching the monitor to make the appropriate measurements.

7.2.1 A variety of test grids, in the form of transparent sheets or as eyepiece reticles, may be utilized with a semiautomatic digitizing tablet. For counting grain boundary intersections or grains intercepted, a circular test grid, such as described in Test Methods E112, may be used. For measuring intercept lengths, a test grid with a number of equally spaced straight, parallel lines is used.

7.3 An automatic image analyzer with a camera of adequate sensitivity can be used to detect the grain boundaries, or grain interiors, and make the appropriate measurements.

7.3.1 A programmable automatic stage to control movement in the *x* and *y* directions without operator attention may be used, but is not mandatory. Use of a programmable stage prevents bias in field selection.

7.4 A computer, of suitable capability, is used with either a digitizing tablet or automatic image analyzer to store and analyze the measurement data. For automatic image analysis, the computer also controls all of the operations except, perhaps, focusing (automatic focusing is optional).

7.5 A printer is used to output the data and relevant identification/background information in a convenient format. Graphical data may be produced with either a printer or plotter, as desired.

7.6 This equipment must be housed in a location relatively free of airborne dust, particularly for automatic image analyzers. High levels of humidity must be avoided as staining of specimen surfaces may occur during, or before, analysis. Very low levels of humidity must also be avoided as static electricity may damage electronic components. Vibrations, if excessive, must be isolated, particularly for automatic image analysis.

8. Sampling

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 aims of the investigation. Sampling location and frequency should be based upon agreements between manufacturers and users.

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

9. Test Specimens

9.1 In general, if the grain structure is equiaxed, any specimen orientation is acceptable. However, the presence of an equiaxed grain structure in a wrought specimen can only be determined by examination of a plane of polish parallel to the deformation axis. Consequently, preparation of longitudinally oriented specimens, where the plane-of-polish is parallel to the deformation axis or grain elongation direction, is recommended.

9.2 If the grain structure of a longitudinally oriented specimen is equiaxed, then grain size measurements on this plane, or any other, will be equivalent within the statistical precision of the test method. If the grain structure is not equiaxed but elongated, then grain size measurements on specimens with different orientations will vary. In this case, the grain size must be determined on longitudinal, transverse, and planar surfaces, or radial and transverse surfaces, depending on the product shape, and averaged, as described in Annex A1, to obtain the mean grain size. If directed test lines (rather than random) are used for intercept counts on non-equiaxed grains in plate or sheet type specimens, the required measurements can be made using only two principle test planes, rather than all three, due to the equivalence of test directions, as described in A1.4.3 and A1.4.2.2.

9.3 The surface to be polished should be large enough in area to permit measurement of at least five fields, preferably more, at the necessary magnification. In most cases (except for

thin sheet or wire specimens), a minimum polished surface area of 160 mm^2 (0.25 $in.^2$) is adequate.

9.4 Thin product forms can be sampled by placing one or more longitudinally oriented (or transverse, if required for non-equiaxed grain structures) pieces in the mount so that the sampling area is sufficient. Adjust the stage movement so that the interface between adjacent specimens is avoided, that is, is not in the field of measurement.

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 Practice E3.

10.2 The polishing procedure must remove all deformation and damage induced by the cutting and grinding procedure. All scratches and smearing must be removed, although very fine scratches from the final polishing step can usually be tolerated. Scratches from grinding, or from polishing with abrasives larger than about 1-μm in diameter, must be removed. Excessive relief, pitting or pullout must be avoided. Specimens must be carefully cleaned and dried after polishing.

10.3 Specimens to be rated for grain size should be in the desired heat treated condition representative of the product, for example, solution annealed, annealed, as-quenched, or quenched and tempered. Other treatment conditions, such as as-hot rolled, as-hot forged, or as-cold drawn, may be tested as required but it must be recognized that the grain structure for these conditions may not be equiaxed.

10.4 Mounting of specimens is not always required depending on their size and shape and the available preparation equipment; or, if hand polishing is utilized for bulk specimens of convenient size and shape.

10.5 The polished surface area for mounted specimens should be somewhat greater than the area required for measurement to avoid edge interferences. Unmounted specimens generally should have a surface area much larger than required for measurement to facilitate leveling, if automatic image analysis is to be utilized, as described in 12.2.

10.6 Etching of specimens is a critical step in the preparation sequence. The choice of the proper etchant depends on the composition and heat treatment condition of the specimen. For automatic image analysis, a flat etch condition, that is, where the grain boundaries appear dark against a light matrix, is normally required. Test Methods E407 and Ref (**1**)[3] list many suitable etchants. A very high degree of grain boundary delineation is required.

10.7 A greater range of grain structure etchant types may be used for grain size measurement with a semiautomatic digitizing tablet. Grain contrast (**1**) and tint etchants (**1,2**) are very effective because they generally provide full delineation of the grain structure.

10.8 For certain specimens, for example, austenitic stainless steels, grain boundary delineation can be improved if the specimen is subjected to a sensitization treatment which precipitates carbides at the grain boundaries.

10.9 Specimens that contain annealing twins are difficult to measure for grain size because the twin boundaries are detected as well as the grain boundaries. For such specimens, semiautomatic digitizing tablet measurements are preferred. Certain electrolytic etching techniques, (**3,4**) as summarized in Ref (**1**) will delineate the grain boundaries but not the twin boundaries thus permitting use of automatic image analysis.

10.10 Specimens that have been carburized for grain size measurement according to the McQuaid-Ehn technique, as described in Test Methods E112, should be etched using a reagent that darkens the cementite preferentially, such as alkaline sodium picrate (see Test Methods E407, or Ref (**1**)) or Beraha's sodium molybdate tint etchant. (**1, 2**) Any cementite not present at a grain boundary is ignored, if a digitizing tablet is used, or deleted from the image prior to measurement, if automatic image analysis is used.

10.11 Delineation of prior-austenite grain boundaries in a hardened alloy steel specimen is quite difficult and usually requires considerable experimentation. The nature of the heat treatment is usually important, particularly the tempering temperature, if used. Subjecting the specimen to a temper embrittlement cycle may enhance the etch response, but this treatment is not helpful if the amounts of P, Sn, As, and Sb are very low. In general, coarse-grained specimens are more easily etched for prior-austenite grain size. Reference (**1**) provides guidance for development of prior-austenite grain boundaries. In general, it is difficult to reveal the prior-austenite grain boundaries to the level required for automatic image analysis, unless the image can be edited successfully prior to measurement, and measurements with a digitizing tablet may be preferable.

10.12 Heat treatments that precipitate a second-phase constituent along the prior-austenite grain boundaries in steel specimens may be useful. Again, this technique works best with relatively coarse-grained steels.

10.13 Image signal processing techniques, such as skeletonization (**5,6**) or watershed segmentation, (**6-8**) may be used to complete missing grain boundaries or produce grain boundaries in grain contrast etched specimens. However, these techniques must be used with caution because skeletonization can produce false grain boundaries and watershed segmentation may not produce grain boundaries between two adjacent grains with similar color or gray level. Light pen, mouse, or trackball editing of images to complete missing grain boundaries before measurement is an acceptable technique, although slow.

10.14 Photomicrographs may be prepared, as described in Guide E883, for measurement with a digitizing tablet. If enlargements are used, the magnification must be determined to a precision of ±1 % maximum before measurements are made. A sufficient number of fields, selected blindly without bias, must be photographed at the required magnification to ensure adequate statistical precision.

[3] The boldface numbers in parentheses refer to the list of references at the end of this standard.

10.15 Annex A2 shows micrographs of a variety of metals and alloys exhibiting properly and improperly etched grain structures with comments concerning their suitability for subsequent analysis using either a semiautomatic digitizing tablet or an automatic image analyzer.

10.16 Annex A1 describes methods for determining the grain size of specimens with nonequiaxed grain shapes, as well as procedures for defining the grain anisotropy index (degree of grain elongation).

11. Calibration and Standardization

11.1 Use a stage micrometer to determine the true linear magnification for each objective and eyepiece combination to be used.

11.2 Determine the magnification of photomicrographs by photographing the stage micrometer image and dividing the magnified length of the micrometer scale by the true length.

11.3 If enlargements are made from photographic negatives, set up the enlarger using the negative of the micrometer scale and determine the magnification of the enlarged micrograph in the same manner as described in paragraph 11.2. Then, make enlargements of the grain structure images using the same enlarger setting. Alternatively, determine the degree of enlargement by comparing the size of features on the enlargement to their size on the contact print. Repeat this process for a number of features in the image. Determine the average enlargement factor of the measured features and multiply this value by the magnification of the contact print.

11.4 If a video monitor is used with a semiautomatic digitizing tablet, determine the video monitor magnification for each objective and projection eyepiece/camera multiplying factor combination using a stage micrometer scale.

11.5 If an automatic image analyzer is used, determine the size of the test area or magnification bar using a stage micrometer scale for each objective and projection eyepiece/camera multiplying factor combination (consult the manufacturer's instruction book for the calibration procedure specific to the instrument used).

11.6 Use a ruler with a millimetre scale to determine the actual length of straight test lines or the diameter of test circles used as grids.

11.7 Use a stage micrometer to measure the length of straight test lines or the diameter of test circles on eyepiece reticles.

11.8 When a video camera is employed, follow the manufacturer's recommendation in adjusting the microscope light source and setting the correct level of illumination for the particular camera used.

11.9 For automatic image analysis measurements, use the flicker method of switching back-and-forth between the live video image and the detected image of either the grain boundaries or grain interiors to establish the correct setting of the gray-level threshold controls, as described in 13.3.

12. Procedure: Semiautomatic Digitizing Tablet

12.1 When photomicrographs are used for measurements, choose the magnification so that at least fifty grains, preferably more, are present, unless the grain structure is extremely coarse. Avoid an excessively high number of grains per photograph as counting accuracy may be impaired. To minimize operator fatigue, and to ensure measurement accuracy, the smallest grain on the photomicrograph should be about 5 mm in diameter. Take the micrographs at random, that is, without bias in the field selection, and prepare a sufficient number, at least five, to obtain adequate statistical precision. Fix each micrograph to the tablet surface, for example using masking tape, to prevent movement during analysis. Drop the measurement grid onto the photograph to prevent placement bias. Tape the grid corners to the micrograph or tablet surface to prevent movement during measurement.

12.2 When a microscope is used to produce the image of the grain structure for subsequent measurement, using either a semiautomatic or automatic image analyzer, place the specimen on the microscope stage so that its surface is perpendicular to the optic axis. With an inverted-type microscope, simply place the specimen face down on the stage plate and hold it in place with the stage clamps. With an upright-type microscope, place the specimen on a slide and level the surface using clay or plasticene between the specimen and slide. To avoid problems with adherent tissue paper, follow the alternate leveling procedure described in Practice E1245 (Procedure section).

12.2.1 The microscope light source should be checked for correct alignment and the illumination intensity should be adjusted to the level required by the television camera.

12.2.2 When a live microscopical image is used, either with a digitizing tablet, or an automatic image analyzer, field selection should be done blindly without bias. Never attempt to choose" typical" or "worst" fields (except for the ALA method, see 12.6.1) as bias will be introduced. For manual stage movement, move the x- and y-stage controls without looking at the image. If a programmable stage is available, set the stage controls to sample the image in a systematic manner. Measurement fields should not be overlapped.

12.2.3 To obtain a reasonable degree of measurement precision, it is not necessary to sample a large number of fields. Generally, from five to twenty fields are adequate (see the comments about the number of fields or measurements required for each type of measurement described in the following sections).

12.2.4 Adjust the magnification of the system so that at least 50 grains, preferably more (unless the grain structure is extremely coarse), are observed through the eyepieces or on the television monitor. If an excessively high number of grains are present in the image, measurement precision will be impaired. For accurate measurement of intercept lengths or grain areas, the smallest grains should be at least 5 mm in diameter on the television monitor (**9**) (for a typical 305–330 mm (12–13 in.) diameter monitor).

NOTE 1—For automatic image analyzers with a pixel density substantially greater than the commonly used 512 × 512 array, grains somewhat smaller than 5 mm in diameter (on the monitor screen) may be measured

with reasonable precision. The operator must determine the minimum apparent size grain that can be measured with a deviation of no more than 10 % of the diameter or 20 % of the area using test circles or squares of known size (see Ref. (**9**) for an example of this procedure).

12.2.5 When a semiautomatic digitizing tablet is used with a live microscope image and an eyepiece test grid reticle, select the appropriate reticle for measurement and adjust the magnification so that about 50 grains are visible, unless the grain size is extremely coarse. Counting accuracy will be impaired if the number of grains visible is excessively high (smaller apparent size in the field of view).

12.2.6 The grain size measurement methods described in the following paragraphs are those known to produce accurate results with reasonable precision and minimal bias. There may be other possible methods, or other equivalent procedures, that can be used to measure grain size. The operator should evaluate the precision and accuracy of such methods on specimens carefully evaluated by one or more of the recommended methods before utilizing an alternate method or procedure. It should be recognized that slight differences in grain size ratings may be obtained using different methods because different aspects of the grain structure are being assessed. Also, minor deviations from equiaxed conditions may accentuate these differences. Methods based on the average grain area or the number of grains per unit area are directly related to the total length of grain edges per unit volume, L_V. Methods based on the mean intercept length or the number of grain boundary intersections per unit length are directly related to the grain boundary surface area per unit volume, S_V. Hence, because these methods are based upon two different geometrical characteristics of the grain structure, minor grain size differences may result when the planar grain size is determined using methods based on L_V vs. S_V .

12.3 *Intercept Length Method:*

12.3.1 When a digitizing tablet is used to measure grain intercept lengths using a template consisting of parallel straight test lines, as described in 12.3.2, measure only the lengths of the test lines that intersect grains (that is, measure the chord distances between successively intersected grain boundaries). Generally, each test line will begin and end within a grain and these partial chords are not measured (see Table 1).

12.3.2 The test grid, consisting of a number of parallel, straight test lines with a spacing greater than the apparent mean grain diameter, should be randomly superimposed over the photographic or live image, without bias, at two or more orientations to average any anisotropy that may be present. If the grain structure is clearly elongated, four different orientations with respect to the longitudinal direction, for example, 0°, 45°, 90° and 135°, should be used as described in the Appendix in Test Methods E1181. This procedure should be repeated on each of at least five photomicrographs or live microscope images, each randomly selected, until at least 500 grain intercept lengths (chords) are measured. If the degree of grain elongation (grain anisotropy) is of interest, use grid line orientations of 0° and 90° with respect to the deformation direction of the specimen. The degree of grain elongation, or anisotropy, is the ratio of the average intercept lengths parallel to the deformation direction (0°) to the average intercept length perpendicular to the deformation direction (90°). Annex A1 provides information concerning the measurement of grain size and grain anisotropy for non-equiaxed grain structures.

12.3.3 The average intercept length, $\bar{l}$, is calculated from the number of measured values, N, of l_i using true length units (μm or mm) by dividing the apparent length on the photomicrograph or microscope image by the magnification, M.

12.3.4 A histogram of the intercept lengths may be constructed to determine or illustrate the uniformity of the grain intercept lengths and to detect and analyze duplex grain size conditions. The analytical method is described in Test Methods E1181, Appendix X2.

12.3.5 Calculate the standard deviation, s, of the individual intercept measurements. Most digitizing tables have software programs established for such computations. If the histogram reveals a duplex condition, calculate s for the intercepts within each region of the distribution curve. To do this, sort the intercept lengths in ascending order, separate the data into the

TABLE 1 Summary of Counting/Measuring Restrictions for Semiautomatic and Automatic Image Analysis Methods

Method	Paragraph	Measurement	Restrictions
		Semiautomatic Image Analysis Methods	
Intercept Lengths	12.3	l_i	Measure only whole intercept lengths, ignore intercepts that end within a grain.
Intercept & Intersection Counts	12.4	P_{Li}, N_{Li}	No restrictions except for the diameter of a circular test grid; the number of grains per field.
Grain Count	12.5	N_{Ai}	Count only whole grains within a known test area.
Grain Area	12.6.1	Ai	Measure areas of whole grains only.
ALA method	12.6.2	A_{max}	Measure entire area of the largest observed grain section.
Two-Phase Methods	12.7.1	$l_{\alpha i}$	Measure only whole intercept lengths, ignore intercepts that end within a grain.
Two-Phase Methods	12.7.2	$A_{A\alpha}$, $P_{P\alpha}$ N_α	No restrictions.
		Automatic Image Analysis Methods	
Grain Boundary Length/ Area	13.5.1	L_{Ai}	No restrictions as long as the field contains a large number of grains.
Intersection Counts	13.6.1	P_{Li}	No restrictions as long as the field contains a large number of grains.
Intercept Lengths	13.7.1	$\bar{l}_i$, l_i	Measure only whole intercept (chord) lengths, delete grains intersecting the test area border.
Grain Count	13.8.1	N_{Ai}	Count only whole grains within a known test area.
Grain Areas	13.9.1	$\bar{A}_i$, A_i	Only whole grains should be in the test area.
ALA Method	13.9.9	A_{max}	Measure the entire area of the largest observed grain section.
Two-Phase Methods	13.10	$\bar{l}_\alpha$,$\bar{A}_\alpha$	Measure only whole intercept (chord) lengths or whole grain areas.

two individual distributions, and compute $\bar{l}$ and s for the intercept lengths in each distribution. Such a computation is easy to perform if the data can be read into a spreadsheet type computer program.

12.4 *Intercept and Intersection Count Methods:*

12.4.1 A digitizing table can be used to count the number of grain boundary intersections, P_i, or the number of grains intercepted, N_i, (the former is preferred) by a circular test grid in the same manner as described for manual measurements of P and N in Test Methods E112.

12.4.2 The test grid or reticle should be a circle, or three circles as described in Test Methods E112. Although any size circle can be used, as long as the circle is larger than the largest grain in the field, relatively small circles are not recommended as the efficiency of the analysis is impaired. In general, the same recommendations as in Test Methods E112 apply, that is, use a test circle or three concentric circles with a total line length of 500-mm. The average number of intercepts or intersections should be about 100, with a minimum of 70 and a maximum of 150 (unless the grain size is too coarse). This ideal range may not always be achievable depending upon the available magnification steps, and values outside this range may be used in such cases (the number of fields measured should be changed to achieve the counting total described in 12.4.3). When using an eyepiece reticle, use of a single test circle, of diameter significantly larger than the largest grain, is recommended to minimize operator fatigue. In this case, the average number of intercepts or intersections should be at least 25 per circle.

12.4.3 Repeat the analysis until at least 500 grain boundary intersections or grain interceptions have been counted on five or more randomly selected fields or photomicrographs. If five photomicrographs are used and one placement per photograph is insufficient to produce at least 500 counts, repeat the measurements using different regions of the same photomicrographs. For example, if the number of counts for the first grid placement on micrograph one is significantly below 100, drop the grid on a second region of the micrograph and repeat the measurement until about 100 counts are obtained per micrograph. This is easily performed, without producing bias, if enlargements are used. Alternatively, a greater number of micrographs can be made and analyzed. When using a live microscope image and an eyepiece reticle, simply select more fields, at random, until at least 500 total counts are made.

12.4.4 Grain boundary intersections and grains intercepted can also be counted using a test grid composed of straight test lines. However, because of the problems associated with counts at the ends of the test lines, this practice is not recommended unless half intercepts or intersections can be tallied separately. For such work, follow the counting rules described in Test Methods E112.

12.4.5 With a circular test grid, end counting problems are eliminated. When counting grain boundary intersections, which is usually easier, a tangential intersection with a grain boundary is counted as one intersection. Each grain boundary cut by the test line is also counted as one intersection. Count an intersection of the junction of three grains, a“ triple point”, as 1½ intersections. If the cursor can be programmed to record each triple point intersection as 1½, count these separately. If this cannot be done, count every other triple point intersection twice. If the test line should intersect a junction between four grains, which occurs rarely, count this intersection twice, that is, as 2 intersections.

12.4.6 For each test circle (or concentric circles) placement, determine P_{Li} or N_{Li} by dividing the number of intersections, P_i, or the number of intercepts, N_i, by the true test line length, that is, the true circle circumference, L_{ti}:

$$P_{Li} = \frac{P_i}{L_{ti}} \tag{1}$$

or,

$$N_{Li} = \frac{N_i}{L_{ti}} \tag{2}$$

where:

$$L_{ti} = \frac{\pi d}{M} \tag{3}$$

for a single circle of diameter d; or,

$$L_{ti} = \frac{(\pi d_1) + (\pi d_2) + (\pi d_3)}{M} \tag{4}$$

for three concentric circles of diameter d_1, d_2, and d_3 and magnification M.

12.4.7 Next, calculate the mean values $\bar{P}_L$ or $\bar{N}_L$, for N fields measured, or n grid placements.

12.4.8 Then, determine the mean lineal intercept length, $\bar{l}$:

$$\bar{l} = \frac{1}{\bar{P}_L} = \frac{1}{\bar{N}_L} \tag{5}$$

where $\bar{l}$ is in µm or mm.

12.4.9 Calculate the standard deviation, s, of the individual measurements of P_{Li} or N_{Li}.

12.5 *Grain Count (Planimetric) Method:*

12.5.1 Grain size may also be determined by the Jeffries planimetric procedure using a digitizing tablet and several procedures may be used. However, as with manual application of the Jeffries method, the tablet method also requires marking off of the grains in order to obtain an accurate count. Hence, the method is less efficient than the intercept procedures. Because of the need to mark off the grains as they are counted, this method is best utilized with photomicrographs.

12.5.2 Prepare at least five photomicrographs, taken randomly. Enlargements are easiest to use. Each micrograph should contain at least fifty whole grains, unless the grain size is extremely coarse. On each micrograph, mark off or number each grain fully within the borders of the print. The number of whole grains counted per micrograph is N_i.

12.5.3 Place each micrograph on the digitizing tablet and trace the region that encloses the counted grains to determine the area, A_i, containing N_i grains, that is, the outer grain boundary traces between the whole grains counted and the partial grains not counted. Divide each area, A_i, by the magnification squared, M^2, to obtain the true area of each group of counted grains per micrograph, A_{ti}.

12.5.4 Determine the number of grains per unit area, N_{Ai} (µm^2 or mm^2), for each test area in accordance with:

$$N_{Ai} = \frac{N_i}{A_{ti}} \quad (6)$$

12.5.5 Calculate the mean value, $\bar{N}_A$, for n measured fields with number per mm^2 true units.

12.5.6 Calculate the standard deviation, s, of the N_{Ai} measurements.

12.5.7 The mean grain area, $\bar{A}$, can be determined in accordance with:

$$\bar{A} = \frac{1}{\bar{N}_A} \quad (7)$$

12.6 *Grain Area Method:*

12.6.1 Grain areas can also be measured using a digitizing tablet. However, because of the tedious nature of this analysis, for a sufficiently large number of grains to achieve adequate statistical precision, this method is not recommended.

12.6.2 The area of the largest grain observed on a metallographic section, the ALA grain size as described in Test Methods E930, can be measured using a digitizing tablet.

12.6.3 The polished and etched specimen is examined and the largest grain is photographed, and its area, A_{max}, is measured with the tablet, or the area of this grain is measured directly if the cursor image is superimposed upon the image viewed through the eyepieces or on a video monitor.

12.7 *Methods for Two-Phase Structures:*

12.7.1 The grain size of a particular phase, α, in a two-phase microstructure can also be determined using a digitizing tablet. The easiest method is the measurement of intercept lengths of straight test lines in the phase of interest, as described in paragraph 12.3.2. Only the length of the test lines intersecting the grains of interest are measured and the average intercept length and standard deviation are determined as described in paragraphs 12.3.3 and 12.3.5.

12.7.2 An alternative, more difficult procedure for determining the grain size of a particular phase in a two-phase microstructure is to first determine the area fraction or point fraction (such as by Practice E562) of the phase of interest, $\bar{A}_{A\alpha}$ or $\bar{P}_{P\alpha}$, and then to apply a circular test grid over the structure and count the number of grains of the phase of interest, N_α, intercepted by the circular test line or lines of known length. The mean lineal intercept length of the phase of interest, $\bar{l}_\alpha$, is calculated using the following equation:

$$\bar{l}_\alpha = \frac{\left(\bar{A}_{A\alpha}\right)(L_{ti})}{\bar{N}_\alpha} = \frac{\left(\bar{P}_{P\alpha}\right)(L_{ti})}{\bar{N}_\alpha} \quad (8)$$

where:

$\bar{A}_{A\alpha}$ and $\bar{P}_{P\alpha}$ = the average area fraction and point fraction of the phase of interest, α

a, L_{ti} = the true test line length of the grid (circumference of test circle or circles used divided by the magnification M), and

$\bar{N}_\alpha$ = the average value of the number of α grains intercepted by the test circle or circles for n fields measured.

12.7.3 It is recommended that the standard deviation be determined for the measured values rather than a value calculated from the measured values. For this measurement, the standard deviation of the area fraction or point fraction of the α phase and the $N_{\alpha i}$ values should be determined. However, because it is difficult to combine these standard deviations, the simplest procedure would be to calculate $l_{\alpha i}$ for each field, based on the $A_{A\alpha}$ or $P_{P\alpha}$ and $N_{\alpha i}$ for each field, and determine the standard deviation of $l_{\alpha i}$ for n fields measured.

13. Procedure: Automatic Image Analysis

13.1 The precision and bias of grain size measurements using automatic image analysis is highly dependent on the quality of the etch delineation of the grain boundaries. The grain boundaries should be fully and uniformly delineated. When working with a new alloy composition or a new etchant, it may be helpful to measure the grain size as a function of etch time, or other experimental conditions, to develop a reliable practice **(10)** that agrees with manual determination of the grain size in accordance with Test Methods E112. The grain size measurement methods described in the following paragraphs are those known to produce results with acceptable precision and minimal bias. There may be other methods or alternate procedures that can produce acceptable results but they must be carefully evaluated before use (see 12.2.6).

13.2 Place the etched specimen on the microscope stage in the same manner as described in 12.2.

13.2.1 Align the light source and set the illumination level as described in 12.2.1.

13.2.2 Do field selection blindly as described in 12.2.2.

13.2.3 Measure and select the number of fields in the same manner as described in 12.2.3, although a greater number of fields can be easily measured if greater precision is required.

13.2.4 Choose the magnification in the same manner as described in 12.2.4.

13.3 Adjust the gray-level threshold settings to detect either the grain boundaries or the grain interiors, depending on the nature of the analysis technique. The “flicker method” of alternately switching between the live video image and the detected image is used to obtain the correct settings.

13.4 Store the detected image within the measurement area in memory and delete those grains that intersect the test area border to eliminate edge effects when grain intercept lengths or grain areas are measured. Particles, such as carbides, nitrides, or inclusions, within the grains should be removed from the image by filling in “holes” within the detected grain interiors. This image can be inverted (reverse detected and non-detected pixels) to produce the grain boundaries within the measurement field after the grains intersecting the test area border have been deleted. The grain boundary image can be thinned by erosion to delete any particles at the boundary and then this image can be inverted to produce an image of the grain interiors. The measurement area, A_{ti}, is then determined by combining the grain boundary and grain interior images, if the number of grains per unit area is to be determined. The final image of the grain boundaries should be thinned to a 1–2 pixel width, if possible, so that the perceived width of the grain boundaries does not significantly influence the measurement of grain intercept lengths or grain areas.

13.4.1 When grain intercept (chord) lengths or grain areas are measured, failure to delete those grains that intersect the

test area border will produce measurement bias, which will increase as the magnification is increased, that is, as a greater proportion of the grains in the image intersect the test area border. For accurate measurement of grain intercept (chord) lengths or grain areas, the smallest grains should be at least 5-mm in diameter on the television monitor (see 12.2.4 and Note 1 and Note 2).

13.4.2 If the number of grain boundary intersections per unit length of scan line length (P_L), or the total length of the grain boundaries per unit area ($\bar{L}_A$), is to be determined, it is not necessary to delete the grains that intersect the test area border, so long as the field contains a large number of grains, see Table 1.

NOTE 2—There are other procedures for dealing with grains that intersect the test area border. These methods are based on certain rules to decide which grains that intersect the test area border are fully sized or not sized at all. However, no corrections are made to the size of the original test area and its relationship to the area of the grains included in the measurement is unknown. It is assumed that when a number of fields are measured, the differences between the original measurement test area and the detected feature area (plus the grain boundary area) balance out. Because of the uncertainties introduced by such procedures, they should be used with caution, or avoided, until their influence on the measurements has been determined.

13.5 *Grain Boundary Length/Area Method:*

13.5.1 The simplest procedure to determine the grain size of a single phase microstructure is to detect only the grain boundaries, as described in 13.3 and 13.4, in each field, without deleting grains that intersect the test area border, and measuring the total length of the grain boundaries in the field, L_i. Next, divide L_i by the true field measurement area, A_{ti} , to obtain the grain boundary length per unit area, L_{Ai}, preferably in units of mm/mm^2, that is:

$$L_{Ai} = \frac{L_i}{A_{ti}} \tag{9}$$

13.5.2 Then, calculate P_{Li} for each field by dividing L_{Ai} by $\pi/2$, that is:

$$P_{Li} = \frac{L_{Ai}}{\pi/2} \tag{10}$$

13.5.3 Next, calculate the average value, $\bar{P}_L$, for n field measurements of P_{Li} , using Eq 7.

13.5.4 Then, calculate the mean lineal intercept length, $\bar{l}$, in accordance with Eq 9, and the standard deviation in accordance with Eq 10.

13.6 *Intersection Count Method:*

13.6.1 Grain size can also be determined by field intersection counts. The grain boundaries of a single phase microstructure are detected in the manner described in 13.3 and 13.4. Grains intersecting the test area border do not need to be deleted. The number of intersections of the grain boundaries by the scan lines is determined. However, to eliminate grain anisotropy effects (non-equiaxed grains), either the image should be rotated using a prism to rotate the live image, or the digitized image can be rotated in memory, or scan lines of several orientations may be used, depending upon the capabilities of the image analyzer used.

13.6.2 The number of grain boundary intersections with the scan lines, P_i, is divided by the true length of the scan lines, L_{ti}, to obtain P_{Li}, that is:

$$P_{Li} = \frac{P_i}{L_{ti}} \tag{11}$$

13.6.3 This procedure is repeated for n fields and the average value, $\bar{P}_L$, is determined using Eq 7.

13.6.4 Then, calculate the mean lineal intercept length, $\bar{l}$, in accordance with Eq 9 and the standard deviation in accordance with Eq 10.

13.7 *Intercept (Chord) Length Method:*

13.7.1 Grain size can also be determined by field or feature-specific chord length measurements. Detect the grain interiors in the manner described in 13.3 and 13.4. Delete all grains intersecting the test area border from the image so that partial chord lengths within these grains are not measured. If the grains are equiaxed, measurements using any orientation for the chords is acceptable. However, if the grains exhibit anisotropy, that is, they are not equiaxed, the image must be either rotated using a prism to rotate the live video image, or the digitized image can be rotated in memory, or scan lines of several orientations may be used, depending upon the capabilities of the image analyzer used. If the degree of grain elongation is of interest, measure the average chord length parallel and perpendicular to the deformation direction in the same manner as described in 12.3.2. The ratio of the average chord lengths parallel and perpendicular to the deformation direction defines the degree of grain elongation (anisotropy). Annex A1 describes methods for measuring the grain size of non-equiaxed structures and for evaluating the degree of grain shape anisotropy.

13.7.2 For field measurements, divide the total length of all the chords within the grains by the number of chords to obtain an average chord length for the field. This value is equivalent to the mean lineal intercept length, $\bar{l}_i$, for the field.

13.7.3 This measurement is repeated for at least five fields, preferably more. Then calculate the mean lineal intercept, $\bar{l}$, for n measurement fields with true length units (μm or mm).

13.7.4 Calculate the standard deviation, s, of the n field measurements of the mean lineal intercept values, $\bar{l}_i$.

13.7.5 An alternate approach is to measure the chord lengths individually and store all of the chord lengths for n fields in memory. The mean lineal intercept length, $\bar{l}$, is determined from the N number of chord (intercept) lengths in accordance with Eq 1. A histogram of the chord (intercept) lengths can also be constructed as described in 12.3.4 and the standard deviation of the chord lengths is determined in accordance with Eq 2. If the grain size distribution is duplex, the grain size within each portion of the distribution, and the amount of each type, is determined as described in 12.3.5 and Appendix X2 of Test Methods E1181.

13.8 *Grain Count Method:*

13.8.1 Grain size may also be determined by a count of the number of grains within a known test area. The grain interiors are detected as described in 13.4 and 13.5. All grains intersecting the test area border should be deleted from the image. The

measurement area is the sum of the grain interior and grain boundaries between these grains, A_{ti}.

13.8.2 The grains completely within the measurement area, N_i, are counted and divided by the test area to obtain the number of grains per unit area for each field, N_{Ai}, in accordance with Eq 11.

13.8.3 This process is repeated for n fields, at least five, but preferably more, and the average value of the number of grains per unit area, $\bar{N}_A$, is determined in accordance with Eq 12.

13.8.4 Calculate the standard deviation, s, of the N_{Ai} measurements for n fields in accordance with Eq 13.

13.9 *Average Grain Area Method:*

13.9.1 Grain size can also be determined by measuring the total area of all of the grains within a field and then dividing by the number of grains in the field to obtain the average grain area, A_i. The grain interiors are detected as described in 13.3 and 13.4. Grains intersecting the test area border must be deleted (see Table 1).

13.9.2 The total area of the grains in the field, A_{gi}, is determined and divided by the number of grains, N_i, to determine the average area of the grains in the field, $\bar{A}_i$, in accordance with:

$$\bar{A}_i = \frac{A_{gi}}{N_i} \tag{12}$$

13.9.3 Repeat this measurement for n fields, at least five, but preferably more, and calculate the mean grain area, $\bar{A}$, with true area units (μm^2 or mm^2).

13.9.4 Calculate the standard deviation, s, for n field measurements of the mean grain area per field, A_i.

13.9.5 *Individual Grain Area Method*—Grain size can also be determined by individual determination of the area of each grain completely within the test area border. The grain interiors are detected as described in 13.3 and 13.4. All grains intersecting the test area border must be deleted from the image (see Table 1).

13.9.6 Measure the area of each grain interior, A_i, in the field, for n fields, until at least 500 grains have been measured. Store the areas of each grain in memory. Calculate the mean grain area, $\bar{A}$, for N grains measured in true area units (μm^2 or mm^2).

13.9.7 A histogram of the frequency of grain areas can be constructed in a manner analogous to that for intercept lengths, as described in 12.3.4.

13.9.8 Calculate the standard deviation, s, of the measured grain areas, A_i.

13.9.9 The area of the largest grain observed on a metallographic section, the ALA grain size as described in Test Methods E930, can be measured using an automatic image analyzer.

13.9.10 Examine the polished and etched specimen and place the largest observed grain in the field of view. Measure the area of this grain, A_{max}, by selecting it with a light pen, mouse, or track ball.

13.10 *Methods for Two-Phase Structures:*

13.10.1 The grain size of a particular phase, α, in a two-phase microstructure can be determined using an automatic image analyzer. The grains of interest are detected as described in 13.3 and 13.4. Grains intersecting the test area border must be deleted.

13.10.2 Chord length measurements, as described in 13.7, can be made in the detected phase of interest and be used to determine the mean lineal intercept length of the α phase, $\bar{l}_\alpha$. This measurement can be performed using field averages, as described in 13.7.2 – 13.7.5, or individual chord lengths in the phase of interest can be stored in memory as described in 13.7.5 to determine the α phase mean lineal intercept length, $\bar{l}_\alpha$.

13.10.3 Grain area measurements, as described in 13.9, can be made for the detected phase of interest and be used to determine the average grain area of the α phase, $\bar{A}_\alpha$. This measurement can be performed using field averages, as described in paragraphs 13.9.2 – 13.9.4, or individual grain areas in the phase of interest can be stored in memory, as described in 13.9.5 – 13.9.8, to determine the α phase mean grain area, $\bar{A}_\alpha$.

14. Calculation of Results

14.1 After the desired number of fields, n, or grains, N, have been measured, calculate the mean value of the measurement and its standard deviation as described in 12 and 13 for each method. Depending on the method used, a mean value of the particular microstructural feature is determined which can be used to calculate, or estimate, the ASTM grain size number, G.

14.2 Table 2 lists values of $\bar{N}_A$, $\bar{A}$, $\bar{N}_L$, or $\bar{P}_L$, and $\bar{l}$ as a function of G in half grain size units (except for 00 vs. 0 grain size). This table may be used to estimate the ASTM grain size based upon the particular mean test value obtained in the analysis.

14.2.1 For the ALA grain size (Test Methods E930), the average area column in Table 2 is the same data as in Table 1of Test Methods E930. The ASTM grain size number of the largest observed grain, A_{max}, is determined using these data.

14.3 Table 3 lists equations that mathematically describe the relationships between the ASTM grain size number, G, and the measured parameters: $\bar{N}_A$, $\bar{N}_L$ or $\bar{P}_L$, $\bar{A}$ and $\bar{l}$. These equations can be entered into the computer program, or used with a hand calculator, to determine G. Round off the value of G to the nearest tenth unit. The equation relating $\bar{A}$ and G can be used to compute the corresponding ALA grain size number.

14.4 Determine the 95 % confidence intervals, 95 % CI, of each measurement in accordance with:

$$95\,\%\ \text{CI} = \pm\frac{t\cdot s}{\sqrt{n}} \tag{13}$$

or,

$$95\,\%\ \text{CI} = \pm\frac{t\cdot s}{\sqrt{N}} \tag{14}$$

where:

n = the number of fields measured (for field measurements),

N = the number of grain areas or intercept lengths (for individual measurements) and

(· indicates a multiplication operation.)

Table 4 lists the values of t as a function of n or N.

TABLE 2 Grain Size Relationships Computed for Uniform, Randomly Oriented, Equiaxed Grains

Grain Size No. (G)	$\bar{N}_A$ (No./in^2 @ 100 ×)	$\bar{N}_A$ (No./mm^2 @ 1 ×)	$\bar{A}$ (mm^2)	$\bar{A}$ (μm^2)	$\bar{N}_L$ or $\bar{P}_L$ (mm^{-1})	$\bar{l}$ (mm)	$\bar{l}$ (μm)
00	0.25	3.88	0.2581	258100	2.210	0.4525	452.5
0	0.50	7.75	0.1290	129000	3.125	0.3200	320.0
0.5	0.71	10.96	0.0912	91200	3.716	0.2691	269.1
1.0	1.00	15.50	0.0645	64500	4.419	0.2263	226.3
1.5	1.41	21.92	0.0456	45600	5.256	0.1903	190.3
2.0	2.00	31.00	0.0323	32300	6.250	0.1600	160.0
2.5	2.83	43.84	0.0228	22800	7.433	0.1345	134.5
3.0	4.00	62.00	0.0161	16100	8.839	0.1131	113.1
3.5	5.66	87.68	0.0114	11400	10.511	0.09514	95.1
4.0	8.00	124.00	0.00806	8060	12.500	0.08000	80.0
4.5	11.31	175.36	0.00570	5700	14.865	0.06727	67.3
5.0	16.00	248.00	0.00403	4030	17.678	0.05657	56.6
5.5	22.63	350.73	0.00285	2850	21.023	0.04757	47.6
6.0	32.00	496.00	0.00202	2020	25.000	0.04000	40.0
6.5	45.25	701.45	0.00143	1430	29.730	0.03364	33.6
7.0	64.00	992.00	0.00101	1010	35.356	0.02828	28.3
7.5	90.51	1402.90	0.000713	713	42.045	0.02378	23.8
8.0	128.00	1984.00	0.000504	504	50.000	0.02000	20.0
8.5	181.02	2805.81	0.000356	356	59.461	0.01682	16.8
9.0	256.00	3968.01	0.000252	252	70.711	0.01414	14.1
9.5	362.04	5611.61	0.000178	178	84.090	0.01189	11.9
10.0	512.00	7936.02	0.000126	126	100.001	0.01000	10.0
10.5	724.08	11223.22	0.0000891	89.1	118.922	0.008409	8.4
11.0	1024.00	15872.03	0.0000630	63.0	141.423	0.007071	7.1
11.5	1448.15	22446.44	0.0000446	44.6	168.181	0.005946	5.9
12.0	2048.00	31744.06	0.0000315	31.5	200.002	0.005000	5.0
12.5	2896.31	44892.89	0.0000223	22.3	237.844	0.004204	4.2
13.0	4096.00	63488.13	0.0000158	15.8	282.845	0.003536	3.5
13.5	5792.62	89785.77	0.0000111	11.1	336.362	0.002973	3.0
14.0	8192.00	126976.25	0.0000079	7.9	400.004	0.002500	2.5

NOTE 1—$\bar{N}_A$ is the number of grains per unit area.
NOTE 2—$\bar{A}$ is the average grain area.
NOTE 3—$\bar{N}_L$ is the number of grains intercepted per unit length.
NOTE 4—$\bar{P}_L$ is the number of grain boundary intersections per unit length.
NOTE 5—$\bar{l}$ is the mean lineal intercept distance.
NOTE 6—$\bar{N}_L = \bar{P}_L$ for a single phase grain structure.
NOTE 7—The above table was calculated based upon the grain size definitions in Test Methods E112.

TABLE 3 Grain Size Equations Relating Measured Parameters to the ASTM Grain Size, *G*

Determine the ASTM Grain Size, G, using the following equations:	
Equation	Units
1. $G = (3.321928 \text{ Log } \bar{N}_A) - 2.954$	$\bar{N}_A$ in mm^{-2}
2. $G = (6.643856 \text{ Log } \bar{N}_L) - 3.288$	$\bar{N}_L$ in mm^{-1}
3. $G = (6.643856 \text{ Log } \bar{P}_L) - 3.288$	$\bar{P}_L$ in mm^{-1}
4. $G = (-6.643856 \text{ Log } \bar{l}) - 3.288$	$\bar{l}$ in mm
5. $G = (-3.3223 \text{ Log } \bar{A}) - 2.955$	$\bar{A}$ in mm^2

NOTE 1—Equations 2 and 3 are for single phase grain structures.
NOTE 2—To convert micrometres to millimetres, divide by 1000.
NOTE 3—To convert square micrometres to square millimetres, divide by 10^6.
NOTE 4—A calculated G value of – 1 corresponds to ASTM $G = 00$.

14.5 Determine the percent relative accuracy, % RA, of the measurement by dividing the 95 % CI value by the mean value and multiplying by 100, that is:

$$\% \text{ RA} = \frac{95\% \text{ CI}}{\bar{X}} \cdot 100 \qquad (15)$$

where:
$\bar{X}$ = the mean value measured ($\bar{N}_A$, $\bar{N}_L$ or $\bar{P}_L$, $\bar{A}$ or $\bar{l}$).

TABLE 4 95 % Confidence Interval Multipliers, t (Eq 13 and Eq 14)

No. of Fields, n^A	t multiplier	No. of Fields, n^A	t Multiplier
5	2.776	19	2.101
6	2.571	20	2.093
7	2.447	21	2.086
8	2.365	22	2.080
9	2.306	23	2.074
10	2.262	24	2.069
11	2.228	25	2.064
12	2.201	26	2.060
13	2.179	27	2.056
14	2.160	28	2.052
15	2.145	29	2.048
16	2.131	30	2.045
17	2.120	40	2.020
18	2.110	60	2.000
		∞	1.960

[A] or, number of grains, *N*.

14.6 For specimens with non-equiaxed grain structures (see Annex A1), determine the mean value of the grain count, grain area, or random intercept measurements made on longitudinal,

transverse and planar oriented surfaces. If measurements were made using only the longitudinal plane, determine the mean value of the measurements. If directed test lines were used on one, two or three planes, determine the mean of the measurements and any desired anisotropy ratios. Calculate the grain size number based on the mean of the measurements using Table 2 or the equations in Table 3. Calculate the pooled standard deviation when more than one measurement is involved (see Annex A1) and then determine the 95 % CI and the % RA, based on the pooled standard deviation and the mean values of the three measurements and Eq 13 or Eq 14 and Eq 15.

14.7 For a duplex grain size distribution, the analysis is conducted as described in Appendix X2 of Test Methods E1181.

15. Test Report

15.1 The report should document the identifying information regarding the specimen, its composition, specification designation or trade name, customer or data requester, date of analysis, heat treatment or processing history, specimen location and orientation, etchant and etch method, analysis method, and so forth, as required.

15.2 List the number of fields measured, or the number of individual intercepts or grains measured, the magnification, field area and total measurement area.

15.3 A photomicrograph illustrating the typical appearance of the grain structure may be provided, if required or desired.

15.4 If image preprocessing techniques were used to improve the image, state the method or methods used.

15.5 List the mean measurement value, its standard deviation, 95 % confidence interval and percent relative accuracy.

15.6 List the computed, or estimated, ASTM grain size number.

15.7 For a two-phase microstructure, describe the nature of the phases or constituents present and the test results, as described in paragraphs 15.5 and 15.6, for the phase or constituent measured.

15.8 For a duplex microstructure, describe the nature of the duplex condition and the test results for each portion of the distribution as described in paragraph X2.10 of Appendix X2, Test Methods E1181.

15.9 For a specimen with non-equiaxed, elongated/flattened grains, report the specific test plane or planes measured, the nature of the measurement (random—grain count, grain area or intercepts, or directed test lines), the specific measurements for each test plane or direction on the plane(s), and the mean test measurement and resulting mean grain size number for the specimen, as described in Annex A1. Report the pooled standard deviation (if calculated when multiple mean values are involved), the 95 % CI, and the % relative accuracy. If measurements were made on only a single plane, for example, the longitudinal plane, report that this was done and the nature of the test method, plus the statistical evaluation of the data. If the anisotropy index, or other descriptions of the grain shape, was determined (see Annex A1), report the value(s) for the plane or planes, or directions evaluated.

16. Precision and Bias

16.1 The precision and bias of grain sizes determined by the use of semiautomatic and automatic image analyzers are dependent on the same conditions and problems as described in Test Methods E112, E930 and E1181 plus other factors discussed in this section.

16.2 The precision and bias of grain size measurements depend on the representativeness of the specimens selected and the areas on the plane-of-polish chosen for measurement. If the grain size varies within the product, specimen and field selection must adequately sample the variation.

16.2.1 The relative accuracy of the grain size measurement of the product improves as the number of specimens is increased while the relative accuracy of the grain size measurement of each specimen improves as the number of fields sampled, or grains measured, increases.

16.3 Bias in measuring the grain size will result if specimen preparation is inadequate. Excessively deep scratches, excessive relief, preparation-induced deformation, pull-out and other artifacts will produce false detail and promote inaccurate measurements. Automatic image analyzers are affected by these problems to a greater degree than are semiautomatic digitizing tablet measurements.

16.3.1 The grain boundaries must be clearly delineated for best results. The best possible sequence of specimen preparation and etching should be used before relying upon video techniques to further improve the image prior to detection.

16.3.2 Detection of microstructural features other than grain boundaries, for example, inclusions, carbides, nitrides, annealing twin boundaries, slip, or deformation twins, will bias the test results. These features must be eliminated from the image before automatic image analysis is conducted.

16.3.3 Dust in the system or on the specimen, adherent tissue paper, stains, and other interferences must be eliminated or reduced to a harmless level. Detection of these features, particularly during measurement with an automatic image analyzer, will bias test results.

16.3.4 Inaccurate determination of microscope magnification, of photographs, or on the video monitor, will bias test results.

16.3.5 Image processing techniques must be used with caution as they can create false grain boundaries, may not connect all broken grain boundaries, or poorly etched grain boundaries, or may not detect boundaries between grains of the same gray level or color.

16.4 If the grain structure is deformed, analysis of the grain size on only one sectioning plane is inadequate to define the grain size. In such cases, the grain size must be measured on the three principal planes and averaged as described in Annex A1.

16.5 For a specimen with a duplex grain size distribution, the grain size of each portion of the distribution, and the

amounts of each, must be determined, as described in Test Methods E1181, rather than assigning a single mean value to the test results.

16.6 When properly performed, the ASTM grain size can be defined to an accuracy of one-tenth a unit with a 95 % confidence and a relative accuracy of less than 10 %.

16.7 While several different microstructural parameters can be measured from which the ASTM grain size number can be computed, the ASTM grain size numbers determined by these methods, for the same specimen, will be in close agreement, generally within two-tenths of an ASTM grain size number, for a properly prepared specimen with a uniform, equiaxed grain structure.

17. Keywords

17.1 ALA grain size; anisotropy index; area fraction; ASTM grain size number; austenite grains; automatic image analysis; calibration; chord length; confidence level; digitizing tablet; duplex grain structures; equiaxed grains; etchant; ferrite grains; grain boundary; grains; grain size; intercept length; intersection count; magnification; non-equiaxed grains; polycrystalline; prior-austenite grain boundaries; relative accuracy; semiautomatic image analysis; skeletonization; standard deviation; twin boundary; watershed segmentation.

ANNEXES

(Mandatory Information)

A1. GRAIN SIZE OF NON-EQUIAXED GRAIN STRUCTURE SPECIMENS

A1.1 This annex provides instructions for the measurement and calculation of grain size for non-equiaxed, single phase grain structure specimens.

A1.2 If the grain shape has been altered by processing so that the grains are no longer equiaxed in shape, grain size measurements should be made on longitudinal (*l*), transverse (*t*) and planar (*p*) oriented surfaces for plate or sheet type material. In the case of round bars, radial longitudinal and transverse surfaces are employed. A reasonable estimate of the grain size may be obtained by measurements made on the longitudinal plane only, depending upon the manner in which the grain shape has been distorted. If directed test lines are used for the analysis, measurements in the three principle directions can be made using only two of the three principle test planes.

A1.3 *Grain Count or Area Method:*

A1.3.1 For grain count (planimetric or Jeffries method) or grain area methods, determine the mean number of grains per unit area at 1 × on each of the three principle planes, that is, $\bar{N}_{Al}$, $\bar{N}_{At}$, and $\bar{N}_{Ap}$; or, determine the average grain area on each of the three principle planes, that is, $\bar{A}_l$, $\bar{A}_t$, and $\bar{A}_p$. Note that the average grain area is the reciprocal of $\bar{N}_A$.

A1.3.2 The mean number of grains per unit area for the specimen, $\bar{N}_A$, is obtained from the three $\bar{N}_A$ values for the three principle planes:

$$\bar{N}_A = \left(\bar{N}_{Al} \cdot \bar{N}_{At} \cdot \bar{N}_{Ap}\right)^{1/3} \quad \text{(A1.1)}$$

where:

indicates a multiplication operation and the bar above the quantities indicates an average value.

The average grain area, $\bar{A}$, for the specimen can be determined in the same manner, that is:

$$\bar{A} = \left(\bar{A}_l \cdot \bar{A}_t \cdot \bar{A}_p\right)^{1/3} \quad \text{(A1.2)}$$

and $\bar{A}$ is the reciprocal of $\bar{N}_A$.

A1.3.3 A reasonable approximation of the grain size of a non-equiaxed specimen can be made from $\bar{N}_{Al}$ or $\bar{A}_l$, that is, from grain count or grain area measurements on the longitudinal plane alone, or the plane parallel to the grain elongation axis.

A1.3.4 The ASTM grain size number can be determined using Table 2 or the equations in Table 3. Round off the grain size number to the nearest tenth unit.

A1.4 *Intersection Count or Intercept Length Methods:*

A1.4.1 The grain size of non-equiaxed grain structures can be determined from measurements of the mean number of grain boundary intersections per unit length, $\bar{P}_L$, or the mean lineal intercept length, $\bar{l}$, determined using at least four different orientations per plane, or using preferred test directions (that is, parallel and perpendicular to the deformation axis) on longitudinal, transverse and planar oriented specimens. ($\bar{N}_L$ values are handled in the same way as $\bar{P}_L$ values, but equations for $\bar{P}_L$ only will be presented. In practice, it is generally easier to make P counts (intersections of test lines with grain boundaries) than N counts (interceptions of test lines with grains)).

A1.4.2 For the case of randomly determined values of $\bar{P}_L$ *or* $\bar{l}$ on the three principal planes, compute the average value in accordance with:

$$\bar{P}_L = \left(\bar{P}_{Ll} \cdot \bar{P}_{Lt} \cdot \bar{P}_{Lp}\right)^{1/3} \quad \text{(A1.3)}$$

or,

$$\bar{l} = \left(\bar{l}_l \cdot \bar{l}_t \cdot \bar{l}_p\right)^{1/3} \quad \text{(A1.4)}$$

Then, determine the mean grain size of the specimen using Table 2 or the equations in Table 3. Round off the grain size number to the nearest tenth unit.

A1.4.3 A reasonable approximation of the grain size of a non-equiaxed specimen can be made from $\bar{P}_{Ll}$ or $\bar{l}_l$

measurements, that is, from the intersection counts or intercept length measurements on the longitudinal plane alone, or the plane parallel to the grain elongation axis.

A1.4.4 For the case of directional measurements of $\bar{P}_L$ or $\bar{l}$, determine the following values on the three principal planes:

$\bar{P}_{Ll(0°)}$ = Mean number of grain boundary intersections per unit length parallel to the elongation direction on the longitudinal plane.

$\bar{P}_{Lt(90°)}$ = Mean number of grain boundary intersections per unit length perpendicular to the spread (i.e., the through-thickness direction) on the transverse plane.

$\bar{P}_{Lp(90°)}$ = Mean number of grain boundary intersections per unit length perpendicular to the elongation axis (that is, across the width) on the planar (rolling plane) surface.

$\bar{l}_{l(0°)}$,$\bar{l}_{t(90°)}$, and $\bar{l}_{p(90°)}$, are the equivalent mean lineal intercept lengths measured in the same directions on the same planes.

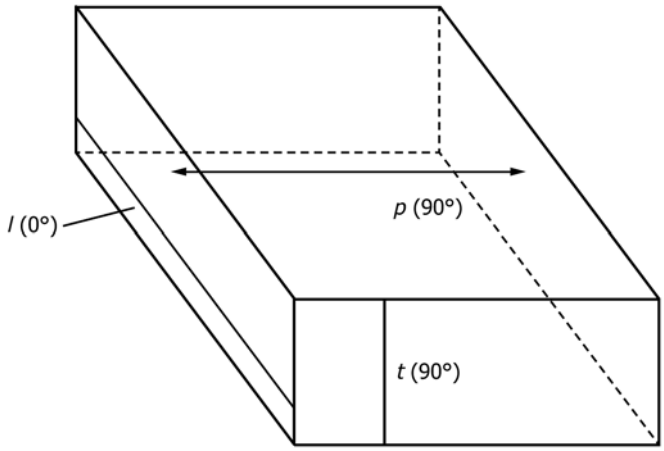

FIG. A1.1 Principal Test Line Orientations Plate or Sheet Products

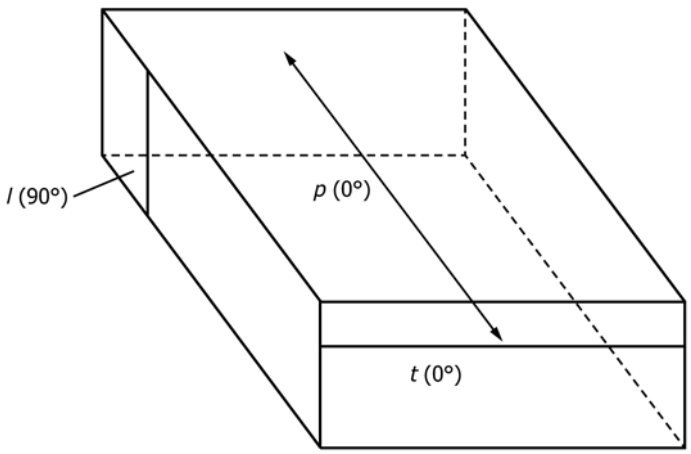

FIG. A1.2 Alternate Test Line Orientations Plate or Sheet Products

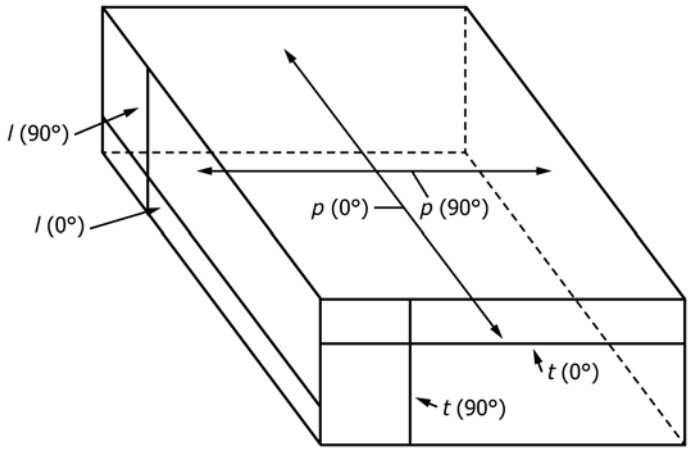

FIG. A1.3 Oriented Test Line Directions Plate or Sheet Products

A1.4.4.1 Fig. A1.1 shows the three principle test planes and the test line orientations for directed intersection measurements of plate or sheet type specimens. For each of the three principle test planes, three directions perpendicular to those shown in Fig. A1.1 can be defined to produce an alternate set of oriented test lines, as shown in Fig. A1.2. Fig. A1.3 shows all six of these oriented test lines. Hence, there are three pairs of statistically identical oriented test lines for intersection or intercept counts:

$$\bar{P}_{Ll(0°)} \equiv \bar{P}_{Lp(0°)} \text{ and } \bar{l}_{l(0°)} \equiv \bar{l}_{p(0°)} \quad \text{(A1.5)}$$

$$\bar{P}_{Lt(90°)} \equiv \bar{P}_{Ll(90°)} \text{ and } \bar{l}_{t(90°)} \equiv \bar{l}_{l(90°)}$$

$$\bar{P}_{Lp(90°)} \equiv \bar{P}_{Lt(0°)} \text{ and } \bar{l}_{p(90°)} \equiv \bar{l}_{t(0°)}$$

A1.4.4.2 Because of these equivalent test line orientations, valid determinations of $\bar{P}_L$ or $\bar{l}$ can be made using only two of the three principle test planes, for example, the longitudinal and transverse planes only for thin sheet specimens where $t(0°)$ test line (Fig. A1.1) measurements on the transverse plane can be substituted for $p(90°)$ test line (Fig. A1.1) measurements on the planar surface in Eq A1.6 or Eq A1.7.

A1.4.4.3 Calculate the mean number of grain boundary intersections per unit length, $\bar{P}_L$, or the mean lineal intercept length, $\bar{l}$, for the specimen in accordance with:

$$\bar{P}_L = \left(\bar{P}_{Ll(0°)} \cdot \bar{P}_{Lt(90°)} \cdot \bar{P}_{Lp(90°)}\right)^{1/3} \quad \text{(A1.6)}$$

or,

$$\bar{l} = \left(\bar{l}_{t(0°)} \cdot \bar{l}_{t(90°)} \cdot \bar{l}_{p(90°)}\right)^{1/3} \quad \text{(A1.7)}$$

Then, determine the mean grain size of the specimen using Table 2 or the equations in Table 3. Round off the grain size number to the nearest tenth unit.

A1.5 Standard deviations for the measurements made on each specimen, for each test plane (longitudinal, transverse and planar) or test line orientation, are calculated in the same way as described in Sections 12 and 13. The standard deviations for the measurements on each specific plane can be pooled and used as an estimate of the data dispersion for the mean of the

three oriented test values (longitudinal, transverse and planar). The pooled standard deviation, s_{pooled}, is computed from the pooled variance (s^2_{pooled}) for the three oriented measurements:

$$s^2_{pooled} = \frac{(n_l - 1)s_l^2 + (n_t - 1)s_t^2 + (n_p - 1)s_p^2}{(n_l - 1) + (n_t - 1) + (n_p - 1)} \quad (A1.8)$$

where:

n is the number of fields measured (or the number of individual measurements, N) on each plane,
s is the standard deviation, and
l, t, and p subscripts indicate the test plane. The square root of the pooled variance is the pooled standard deviation.

A1.6 *Anisotropy Index (Grain Elongation Ratio):*

A1.6.1 The usual approach for measuring the degree of grain elongation, or anisotropy index, is to determine $\bar{P}_L$ or $\bar{l}$ parallel (0°) and perpendicular (90°) to the elongation axis on a longitudinally oriented specimen. The grain elongation ratio, or anisotropy index, AI, is defined by:

$$AI = \bar{P}_{L l(90°)} / \bar{P}_{L l(0°)} \quad (A1.9)$$

or,

$$AI = \bar{l}_{l(0°)} / \bar{l}_{l(90°)} \quad (A1.10)$$

A1.6.2 The three-dimensional mean grain size and shape may also be defined by the directed mean lineal intercept values for the longitudinal, transverse, and planar surfaces. These values could be written in the form $\bar{l}_{l(0°)} / \bar{l}_{t(90°)} / \bar{l}_{p(90°)}$, *as demonstrated by the example below. Alternatively, the three mean lineal intercept lengths can be normalized (divide each by the value of the smallest) and the results expressed as ratios, as shown below. This procedure is useful for showing the overall mean shape of the grains but does not give size information.*

A1.7 *Example of Grain Size and Anisotropy Index Calculation:*

A1.7.1 A sheet steel specimen with an initial grain size of ASTM ~10 was cold rolled to a 30 % reduction in thickness and longitudinal, transverse and planar oriented specimens were prepared. The grain structure was measured on each surface using the planimetric method to determine $\bar{N}_A$, and by the intercept method to determine $\bar{l}$, using randomly oriented test lines and with straight test lines parallel and perpendicular to the deformation direction as defined in A1.4.4. Table A1.1 lists the measurement data.

TABLE A1.1 Summary of Test Data for the Example

Test Plane	$\bar{N}_A$ (mm^{-2})	Random $\bar{P}_L$ (mm^{-1})
l	7056.25	123.6
t	11625.0	141.44
p	3493.75	68.31
	Directed Test Line Data	
Principle Test Line Orientations	$\bar{P}_L$ (mm^{-1})	Alternate Test Line Orientations	$\bar{P}_L$ (mm^{-1})
l (0°)	60.6	*p* (0°)	56.1
t (90°)	167.71	*l* (90°)	167.71
p (90°)	74.5	*t* (0°)	93.56

A1.7.2 The grain size of the specimen was determined using the $\bar{N}_A$ data and Eq A1.1:

$$\bar{N}_A = (7056.25 \cdot 11625 \cdot 3493.75)^{1/3} = 6593.05 \text{ mm}^{-2} \quad (A1.11)$$

Using the equation relating $\bar{N}_A$ and G listed in Table 3, the ASTM grain size was 9.73 (in the example, we give G to two decimal places only to show the degree of agreement between the measurement methods; normally G is rounded to the nearest tenth unit). If each $\bar{N}_A$ value is converted to an average grain area value, $\bar{A}$, using Eq 7, and the mean $\bar{A}$ value is calculated according to Eq A1.2, the same value is obtained for the ASTM grain size using the equation relating $\bar{A}$ and G in Table 3.

A1.7.3 Next, the grain size of the specimen was determined using the random $\bar{P}_L$ data and Eq A1.3:

$$\bar{P}_L = (123.6 \cdot 141.44 \cdot 68.31)^{1/3} = 106.094 \text{ mm}^{-1} \quad (A1.12)$$

Using the equation relating $\bar{P}_L$ and G in Table 3, the ASTM grain size was found to be 10.17. If each $\bar{P}_L$ value is converted to a mean lineal intercept length using Eq 5, and the mean $\bar{l}$ value is obtained using Eq. A2.4, the same ASTM grain size is obtained using the equation relating $\bar{l}$ and G in Table 3.

A1.7.4 Then, the grain size of the specimen was determined from the three principle directed values according to Eq A1.6:

$$\bar{P}_L = (60.6 \cdot 167.71 \cdot 74.5)^{1/3} = 91.144 \text{ mm}^{-1} \quad (A1.13)$$

where $\bar{l}$ = 0.01097 mm. Using the equation relating either $\bar{P}_L$ and G or $\bar{l}$ and G from Table 3, we obtain a mean grain size of 9.73. Again, if each directed $\bar{P}_L$ value is converted to a lineal intercept value and the mean value, $\bar{l}$, is calculated using Eq A1.7, the same grain size number is obtained.

A1.7.5 The grain size of this specimen was also determined from the alternate directed test line orientation data (Table A1.1) in accordance with Eq A1.6:

$$\bar{P}_L = (56.1 \cdot 167.71 \cdot 93.56)^{1/3} = 95.838 \text{mm}^{-1} \quad (A1.14)$$

where:

$\bar{l}$ = 0.01043 mm,
G = 9.88.

A1.7.6 The directed test line data in Table A1.1 was used to compute the grain size by using data from only two test planes rather than the three principle test planes and directions, that is, by substituting data from the alternate test direction (Fig. A1.2). The computed ASTM grain sizes are 9.95 (no planar specimen), 9.66 (no longitudinal specimen), and 9.73 (no transverse specimen).

A1.7.7 The anisotropy index, using directed measurements on the longitudinal plane, was calculated in accordance with Eq A1.9:

$$AI = 167.71/60.6 = 2.77 \quad (A1.15)$$

The same anisotropy index is obtained if the directed $\bar{P}_L$ values are converted to mean lineal intercept values and Eq A1.10 is used. (Anisotropy indexes for the two perpendicular

test orientations on the transverse and planar surfaces revealed values of 0.56 and 1.33, respectively).

A1.7.8 The three-dimensional grain size and shape can be expressed in terms of the three directed measurements of the mean lineal intercept lengths, as described in paragraph A1.6.1, as 16.5/5.96/13.4 (note that the units are in μm, for simplicity of expression, rather than mm). The normalizing approach (dividing each of these values by the smallest) can be used as a simple approach for describing the mean shape (but not size). For this example, we obtain 2.77:1:2.25 (expressed as a ratio). To avoid possible confusion, it may be advisable to list the test planes as subscripts, for example, $16.5_l/5.96_t/13.4_p$ or 2.77_l: l_t: 2.25_p.

A2. EXAMPLES OF PROPER AND IMPROPER GRAIN BOUNDARY DELINEATION

A2.1 This annex provides photomicrographs that illustrate correctly etched and improperly etched grain structures of a variety of metals and alloys with comments pertaining to the problems that may result when grain size measurements are performed using either a semiautomatic digitizing tablet or an automatic image analyzer.

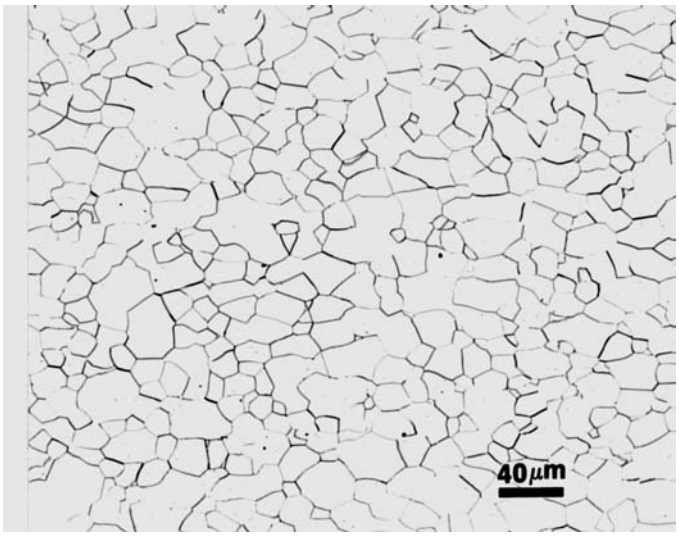

FIG. A2.1 Ferrite grain boundaries in a low-carbon sheet steel specimen (longitudinal plane) are partially revealed using 2 % nital. Grain boundary delineation is inadequate for automatic image analysis without extensive editing.

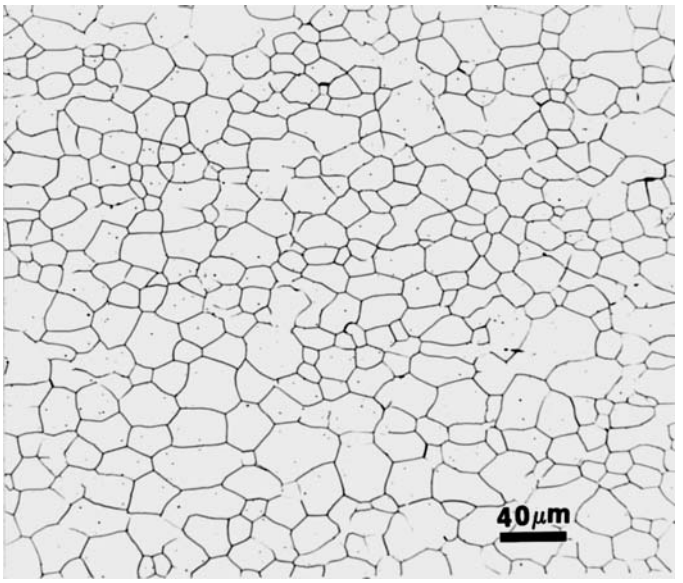

FIG. A2.2 Ferrite grain boundaries in the low-carbon sheet steel specimen shown in Fig. A2.1 but revealed using a 3 s immersion in 2 % nital followed by a 3 s immersion in Marshall's reagent (1 part 5 mL H_2 SO_4, 8 g oxalic acid and 100 mL water plus 1 part H_2 O_2 (30 %)).

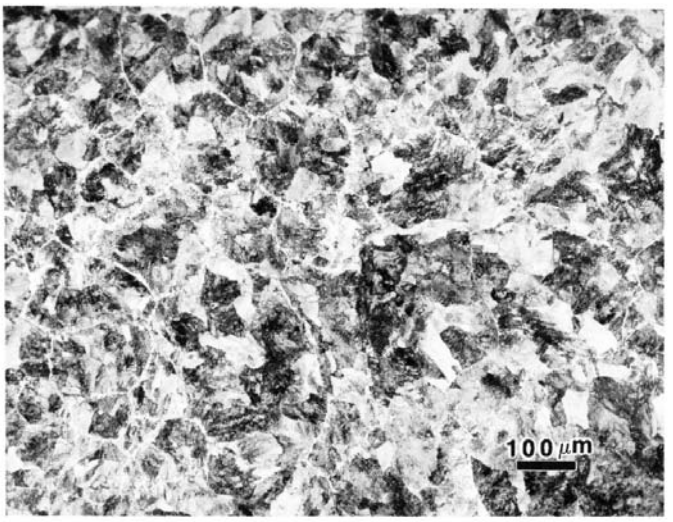

FIG. A2.3 McQuaid-Ehn carburized specimen of a coarse-grained carbon steel etched with 2 % nital. The cementite grain boundary films are very difficult to see and cannot be preferentially detected.

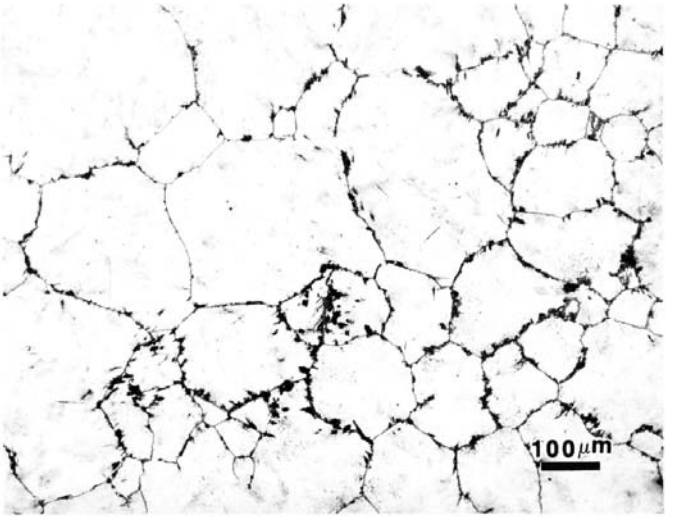

FIG. A2.4 The McQuaid-Ehn specimen shown in Fig. A2.3 etched with Beraha's sodium molybdate tint etc. (1,2). Similar results can be obtained using alkaline sodium picrate, boiling or electrolytically. The grain boundaries are reasonably well delineated. This specimen can be easily measured with a digitizing tablet but substantial image editing would be required for automatic image analysis.

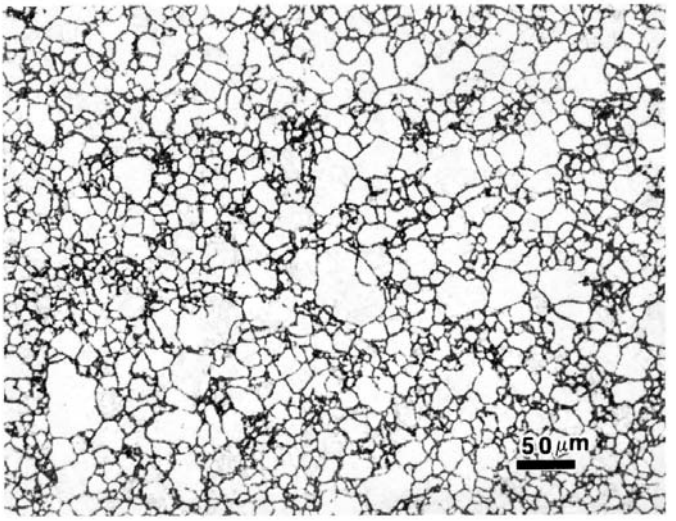

FIG. A2.5 Oxidation grain size specimen of AISI 9310 alloy steel etched with 2 % nital to enhance grain boundary delineation. The grain boundaries are reasonably well delineated. Easily measured with a digitizing tablet; some image editing would be required for automatic image analysis.

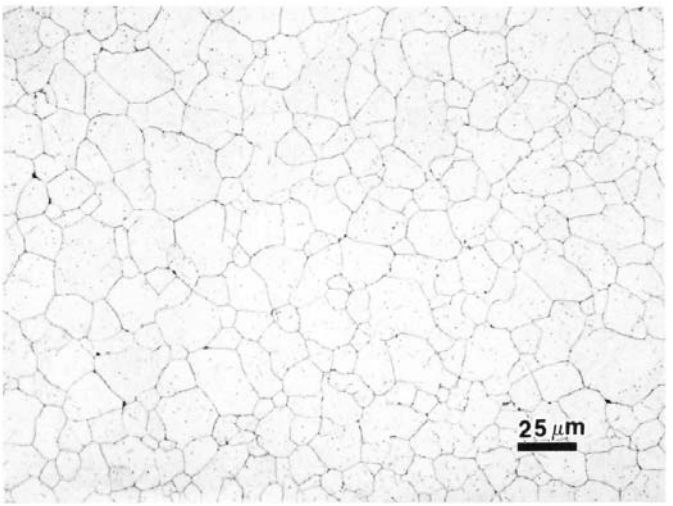

FIG. A2.6 Prior-austenite grain boundaries in this hot-worked tool steel specimen were decorated with cementite/pearlite by an isothermal hold at 1300°F (704°C) after austenitization (glyceregia etch). The boundaries are reasonably well developed and the specimen can be analyzed with a digitizing tablet but some editing would be required for automatic image analysis.

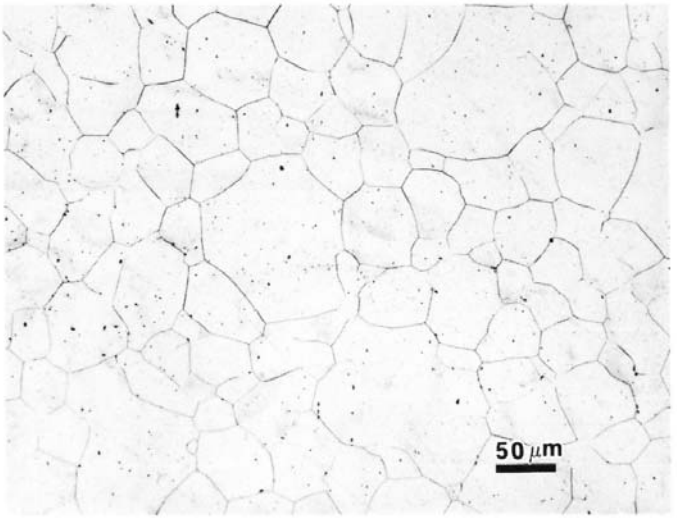

FIG. A2.7 Prior-austenite grain boundaries in a solution annealed 18Ni250 maraging steel revealed by electrolytic etching with aqueous 10 % CrO_3 (9 A/cm^2, 5 min). There are a number of missing grain boundaries that must be added before measurement.

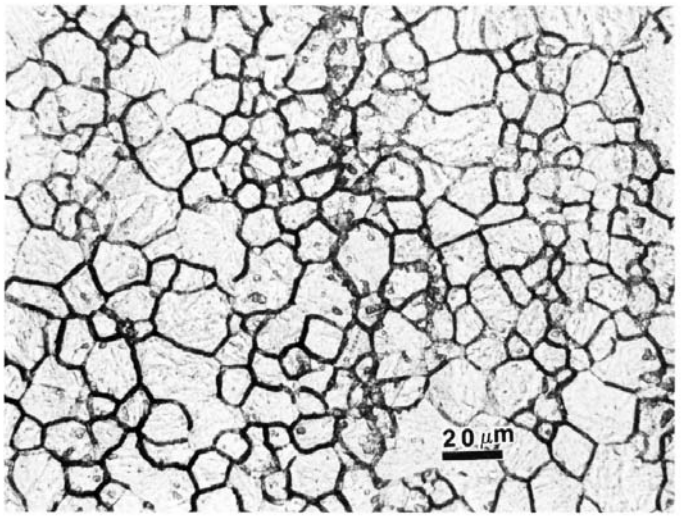

FIG. A2.8 Prior-austenite grain boundaries in a tempered, isothermally transformed lower bainite microstructure of AISI 4340 alloy steel revealed by etching with saturated aqueous picric acid plus a wetting agent (sodium tridecylbenzene sulfonate). Some boundaries are light while others are missing. Moderate image editing is required. Prior to automatic image analysis, the grain boundaries should be thinned and the etch pitting must be removed.

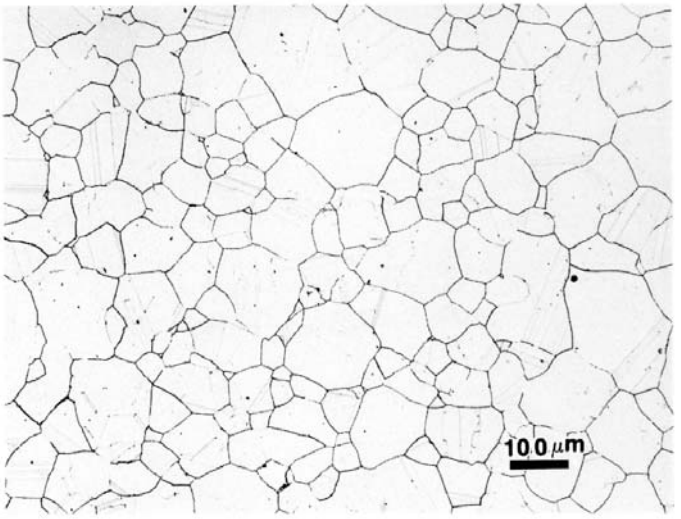

FIG. A2.9 Austenitic grain structure of solution annealed and double aged Waspaloy revealed using glyceregia. Some grain boundaries are missing. The faint annealing twins would interfere with automatic image analysis but not measurement with a digitizing tablet.

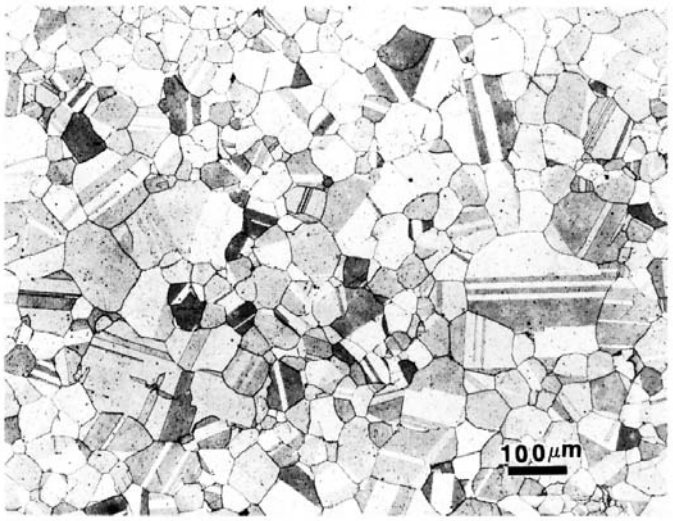

FIG. A2.10 Austenitic grain structure of solution annealed and double aged Waspaloy revealed using Beraha's tint etch (50 % HCl in water plus 1 g potassium metabisulfite (per 100 mL) and 2 g ammonium bifluorite (per 100 mL)). The grain structure is well revealed but the annealing twins would prohibit use of an automatic image analyzer, but not a digitizing tablet.

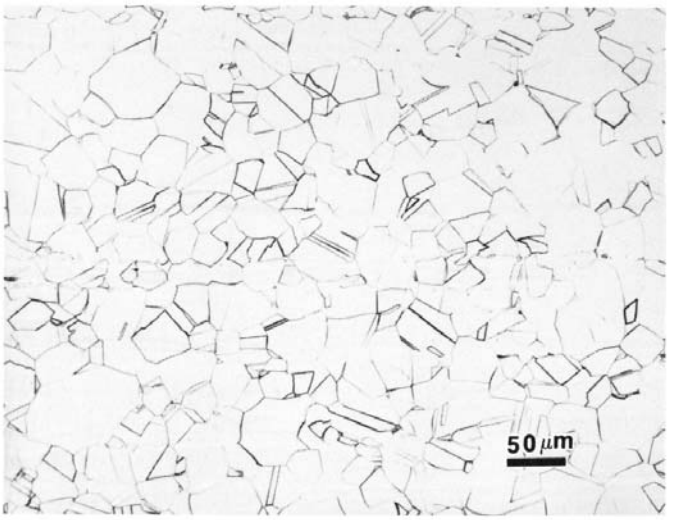

FIG. A2.11 Austenitic grain structure of AISI 316L stainless steel revealed using waterless Kalling's reagent. A substantial percentage of the grain boundaries are not visible and the annealing twins are revealed. The grain structure is too poorly revealed for successful image editing.

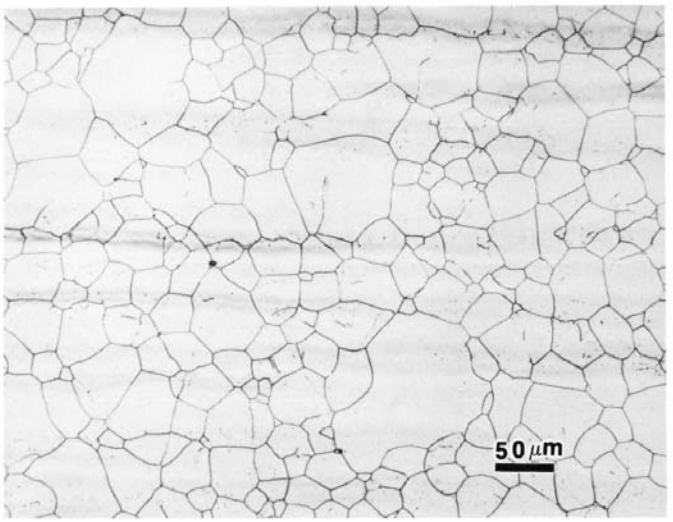

FIG. A2.12 Austenitic structure of the AISI 316L specimen shown in Fig. A2.11 but after electrolytic etching with aqueous 60 % HNO_3 (Pt cathode, 1 V dc, 20 s). The grain structure is nearly completely revealed without the twin boundaries. Minor image editing would be required before measurement.

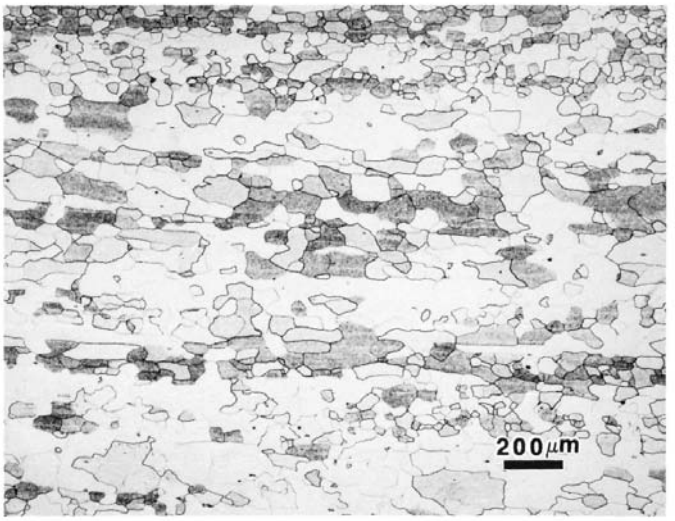

FIG. A2.13 Grain structure of a ferritic stainless steel (26 % Cr—1 % Mo) revealed using acetic glyceregia (longitudinal plane). The etch delineation is too poor for accurate measurement. Image editing cannot be performed reliably.

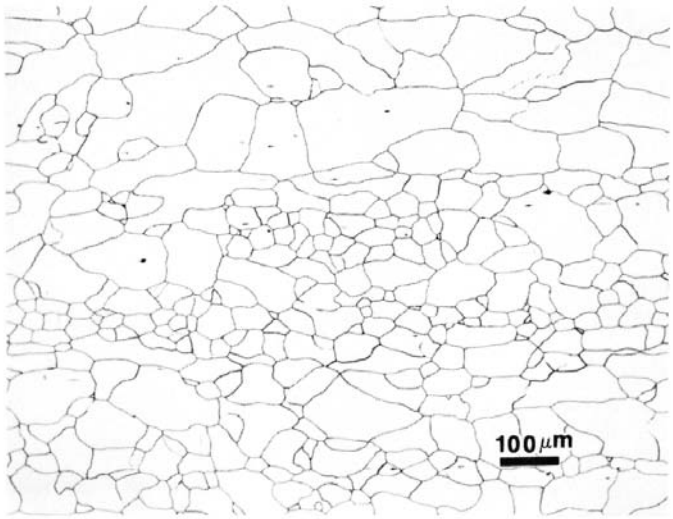

FIG. A2.14 Grain structure of the ferritic stainless steel specimen shown in Fig. A2.13 but after electrolytic etching with aqueous 60 % HNO_3 (Pt cathode, 1.2 V dc15 s). Easily measured using a digitizing tablet and by automatic image analysis (after a minor amount of image editing).

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