Designation: E45 – 18a

Standard Test Methods for Determining the Inclusion Content of Steel[1]

This standard is issued under the fixed designation E45; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

1. Scope

1.1 These test methods cover a number of recognized procedures for determining the nonmetallic inclusion content of wrought steel. Macroscopic methods include macroetch, fracture, step-down, and magnetic particle tests. Microscopic methods include five generally accepted systems of examination. In these microscopic methods, inclusions are assigned to a category based on similarities in morphology, and not necessarily on their chemical identity. Metallographic techniques that allow simple differentiation between morphologically similar inclusions are briefly discussed. While the methods are primarily intended for rating inclusions, constituents such as carbides, nitrides, carbonitrides, borides, and intermetallic phases may be rated using some of the microscopic methods. In some cases, alloys other than steels may be rated using one or more of these methods; the methods will be described in terms of their use on steels.

1.2 These test methods cover procedures to perform JK-type inclusion ratings using automatic image analysis in accordance with microscopic methods A and D.

1.3 Depending on the type of steel and the properties required, either a macroscopic or a microscopic method for determining the inclusion content, or combinations of the two methods, may be found most satisfactory.

1.4 These test methods deal only with recommended test methods and nothing in them should be construed as defining or establishing limits of acceptability for any grade of steel.

1.5 The values stated in SI units are to be regarded as the standard. Values in parentheses are conversions and are approximate.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens
E7 Terminology Relating to Metallography
E381 Method of Macroetch Testing Steel Bars, Billets, Blooms, and Forgings
E709 Guide for Magnetic Particle Testing
E768 Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel
E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
E1444 Practice for Magnetic Particle Testing
E1951 Guide for Calibrating Reticles and Light Microscope Magnifications

2.2 *SAE Standards:*[3]

J422, Recommended Practice for Determination of Inclusions in Steel

2.3 *Aerospace Material Specifications:*[3]

AMS 2300, Premium Aircraft-Quality Steel Cleanliness: Magnetic Particle Inspection Procedure
AMS 2301, Aircraft Quality Steel Cleanliness: Magnetic Particle Inspection Procedure
AMS 2303, Aircraft Quality Steel Cleanliness: Martensitic Corrosion-Resistant Steels Magnetic Particle Inspection Procedure
AMS 2304, Special Aircraft-Quality Steel Cleanliness: Magnetic Particle Inspection Procedure

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[1] These test methods are under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.09 on Inclusions.

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

[3] Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

2.4 *ISO Standards:*[4]

ISO 3763, Wrought Steels—Macroscopic Methods for Assessing the Content of Nonmetallic Inclusions

ISO 4967, Steel—Determination of Content of Nonmetallic Inclusions—Micrographic Methods Using Standard Diagrams

2.5 *ASTM Adjuncts:*

Inclusions in Steel Plates I-A and II[5]

Four Photomicrographs of Low Carbon Steel[6]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in these test methods, see Terminology E7.

3.1.2 Terminology E7 includes the term *inclusion count*; since some methods of these test methods involve length measurements or conversions to numerical representations of lengths or counts, or both, the term *inclusion rating* is preferred.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the length-to-width ratio of a microstructural feature.

3.2.2 *discontinuous stringer*—three or more Type B or C inclusions aligned in a plane parallel to the hot working axis and offset by no more than 15 µm, with a separation of less than 40 µm (0.0016 in.) between any two nearest neighbor inclusions.

3.2.3 *inclusion types*—for definitions of sulfide-, alumina-, and silicate-type inclusions, see Terminology E7. Globular oxide, in some methods refers to isolated, relatively nondeformed inclusions with an aspect ratio not in excess of 2:1. In other methods, oxides are divided into deformable and nondeformable types.

3.2.4 *JK inclusion rating*—a method of measuring nonmetallic inclusions based on the Swedish Jernkontoret procedures; Methods A and D of these test methods are the principal JK rating methods, and Method E also uses the JK rating charts.

3.2.5 *stringer*—an individual inclusion that is highly elongated in the deformation direction or three or more Type B or C inclusions aligned in a plane parallel to the hot working axis and offset by no more than 15 µm, with a separation of less than 40 µm (0.0016 in.) between any two nearest neighbor inclusions.

3.2.6 *threshold setting*—isolation of a range of gray level values exhibited by one constituent in the microscope field.

3.2.7 *worst-field rating*—a rating in which the specimen is rated for each type of inclusion by assigning the value for the highest severity rating observed of that inclusion type anywhere on the specimen surface.

4. Significance and Use

4.1 These test methods cover four macroscopic and five microscopic test methods (manual and image analysis) for describing the inclusion content of steel and procedures for expressing test results.

4.2 Inclusions are characterized by size, shape, concentration, and distribution rather than chemical composition. Although compositions are not identified, Microscopic methods place inclusions into one of several composition-related categories (sulfides, oxides, and silicates—the last as a type of oxide). Paragraph 11.1.1 describes a metallographic technique to facilitate inclusion discrimination. Only those inclusions present at the test surface can be detected.

4.3 The macroscopic test methods evaluate larger surface areas than microscopic test methods and because examination is visual or at low magnifications, these methods are best suited for detecting larger inclusions. Macroscopic methods are not suitable for detecting inclusions smaller than about 0.40 mm (1⁄64 in.) in length and the methods do not discriminate inclusions by type.

4.4 The microscopic test methods are employed to characterize inclusions that form as a result of deoxidation or due to limited solubility in solid steel (indigenous inclusions). As stated in 1.1, these microscopic test methods rate inclusion severities and types based on morphological type, that is, by size, shape, concentration, and distribution, but not specifically by composition. These inclusions are characterized by morphological type, that is, by size, shape, concentration, and distribution, but not specifically by composition. The microscopic methods are not intended for assessing the content of exogenous inclusions (those from entrapped slag or refractories). In case of a dispute whether an inclusion is indigenous or exogenous, microanalytical techniques such as energy dispersive X-ray spectroscopy (EDS) may be used to aid in determining the nature of the inclusion. However, experience and knowledge of the casting process and production materials, such as deoxidation, desulfurization, and inclusion shape control additives as well as refractory and furnace liner compositions must be employed with the microanalytical results to determine if an inclusion is indigenous or exogenous

4.5 Because the inclusion population within a given lot of steel varies with position, the lot must be statistically sampled in order to assess its inclusion content. The degree of sampling must be adequate for the lot size and its specific characteristics. Materials with very low inclusion contents may be more accurately rated by automatic image analysis, which permits more precise microscopic ratings.

4.6 Results of macroscopic and microscopic test methods may be used to qualify material for shipment, but these test methods do not provide guidelines for acceptance or rejection purposes. Qualification criteria for assessing the data developed by these methods can be found in ASTM product standards or may be described by purchaser-producer agreements. By agreements between producer and purchaser, these test methods may be modified to count only certain inclusion types and thicknesses, or only those inclusions above a certain

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[5] Available from ASTM International Headquarters. Order Adjunct No. ADJE004502A. Original adjunct produced in 1983. Adjunct revised in 2011.

[6] Available from ASTM International Headquarters. Order Adjunct No. ADJE004501. Original adjunct produced in 1983.

severity level, or both. Also, by agreement, qualitative practices may be used where only the highest severity ratings for each inclusion type and thickness are defined or the number of fields containing these highest severity ratings are tabulated.

4.7 These test methods are intended for use on wrought metallic structures. While a minimum level of deformation is not specified, the test methods are not suitable for use on cast structures or on lightly worked structures.

4.8 Guidelines are provided to rate inclusions in steels treated with rare earth additions or calcium-bearing compounds. When such steels are evaluated, the test report should describe the nature of the inclusions rated according to each inclusion category (A, B, C, D).

4.9 In addition to the Test Methods E45 JK ratings, basic (such as used in Practice E1245) stereological measurements (for example, the volume fraction of sulfides and oxides, the number of sulfides or oxides per square millimeter, the spacing between inclusions, and so forth) may be separately determined and added to the test report, if desired for additional information. This practice, however, does not address the measurement of such parameters.

MACROSCOPIC METHODS

5. Macroscopical Test Methods Overview

5.1 *Summary:*

5.1.1 *Macro-etch Test*—The macro-etch test is used to indicate inclusion content and distribution, usually in the cross section or transverse to the direction of rolling or forging. In some instances, longitudinal sections are also examined. Tests are prepared by cutting and machining a section through the desired area and etching with a suitable reagent. A solution of one part hydrochloric acid and one part water at a temperature of 71 to 82°C (160 to 180°F) is widely used. As the name of this test implies, the etched surface is examined visually or at low magnification for inclusions. Details of this test are included in Method E381. The nature of questionable indications should be verified by microscopic examination or other means of inspection.

5.1.1.1 Sulfides are revealed as pits when the standard etchant described in 5.1.1 is used.

5.1.1.2 Only large oxides are revealed by this test method.

5.1.2 *Fracture Test*—The fracture test is used to determine the presence and location of inclusions as shown on the fracture of hardened slices approximately 9 to 13 mm (3⁄8 to 1⁄2 in.) thick. This test is used mostly for steels where it is possible to obtain a hardness of approximately 60 HRC and a fracture grain size of 7 or finer. Test specimens should not have excessive external indentations or notches that guide the fracture. It is desirable that fracture be in the longitudinal direction approximately across the center of the slice. The fractured surfaces are examined visually and at magnifications up to approximately ten diameters, and the length and distribution of inclusions is noted. Heat tinting, or blueing, will increase visibility of oxide stringers. ISO 3763 provides a chart method for fracture surface inclusion ratings. In some instances, indications as small as 0.40 mm (1⁄64 in.) in length are recorded.

5.1.3 *Step-Down Method*—The step-down test method is used to determine the presence of inclusions on machined surfaces of rolled or forged steel. The test sample is machined to specified diameters below the surface and surveyed for inclusions under good illumination with the unaided eye or with low magnification. In some instances, test samples are machined to smaller diameters for further examination after the original diameters are inspected. This test is essentially used to determine the presence of inclusions 3 mm (1⁄8 in.) in length and longer.

5.1.4 *Magnetic Particle Method*—The magnetic particle method is a variation of the step-down method for ferromagnetic materials in which the test sample is machined, magnetized, and magnetic powder is applied. Discontinuities as small as 0.40 mm (1⁄64 in.) in length create magnetic leakage fields that attract the magnetic powder, thereby outlining the inclusion. See Practice E1444 and Guide E709 on magnetic particle examinations for more details of the procedure. Refer to Aerospace Materials Specifications AMS 2300, AMS 2301, AMS 2303, and AMS 2304.

5.2 *Advantages:*

5.2.1 These test methods facilitate the examination of specimens with large surface areas. The larger inclusions in steel, which are the main concern in most cases, are not uniformly distributed and the spaces between them are relatively large, so that the chances of revealing them are better when larger specimens are examined.

5.2.2 Specimens for macroscopic examination may be quickly prepared by machining and grinding. A highly polished surface is not necessary. The macroscopic methods are sufficiently sensitive to reveal the larger inclusions.

5.3 *Disadvantages:*

5.3.1 These test methods do not distinguish among the different inclusion shapes.

5.3.2 They are not suitable for the detection of small globular inclusions or of chains of very fine elongated inclusions.

5.3.3 The magnetic particle method can lead to incorrect interpretation of microstructural features such as streaks of retained austenite, microsegregation, or carbides in certain alloys; this is particularly likely if high magnetization currents are employed.

MICROSCOPIC METHODS

6. Microscopic Test Methods Overview

6.1 Microscopic methods are used to characterize the size, distribution, number, and type of inclusions on a polished specimen surface. This may be done by examining the specimen with a light microscope and reporting the types of inclusions encountered, accompanied by a few representative photomicrographs. This method, however, does not lend itself to a uniform reporting style. Therefore, standard reference charts depicting a series of typical inclusion configurations (size, type, and number) were created for direct comparison with the microscopic field of view. A method using image analysis to make these comparisons has also been developed.

6.2 Various reference charts of this nature have been devised such as the JK chart[7] and the SAE chart found in SAE Recommended Practice J422 of the SAE Handbook. The microscopic methods in Test Methods E45 use refined comparison charts based on these charts. Method A (Worst Fields), Method D (Low Inclusion Content) and Method E (SAM Rating) use charts based on the JK chart while Method C (Oxides and Silicates) uses the SAE chart. ISO Standard 4967 also uses the JK chart.

6.3 No chart can represent all of the various types and forms of inclusions. The use of any chart is thus limited to determining the content of the most common types of inclusions, and it must be kept in mind that such a determination is not a complete metallographic study of inclusions.

6.4 An alternate to comparison (chart) methods such as Methods A, C and D[8] may be found in Method B. Method B (Length) is used to determine inclusion content based on length. Only inclusions 0.127 mm (0.005 in.) or longer are recorded regardless of their type. From this method one may obtain data such as length of the longest inclusion and average inclusion length. In addition, photomicrographs may also be taken to characterize the *background inclusions* that were not long enough to measure.

6.5 The advantages of the microscopic methods are:

6.5.1 Inclusions can be characterized as to their size, type, and number.

6.5.2 Extremely small inclusions can be revealed.

6.6 A disadvantage of the microscopic methods is that individual rating fields are very small (0.50 mm^2). This limits the practical size of the specimen, as it would simply take a prohibitive number of fields to characterize a large specimen. The result obtained by a microscopic characterization of the inclusions in a large section is governed by chance if local variations in the inclusion distribution are substantial. The end use of the product determines the importance of the microscopic results. Experience in interpreting these results is necessary in order not to exaggerate the importance of small inclusions in some applications.

6.7 In determining the inclusion content, it is important to realize that, whatever method is used, the result actually applies only to the areas of the specimens that were examined. For practical reasons, such specimens are relatively small compared with the total amount of steel represented by them. For the inclusion determination to have any value, adequate sampling is just as necessary as a proper method of testing.

6.8 Steel often differs in inclusion content not only from heat to heat, but also from ingot to ingot in the same heat and even in different portions of the same ingot. It is essential that the unit lot of steel, the inclusion content of which is to be determined, shall not be larger than one heat. Sufficient samples should be selected to represent the lot adequately. The exact sampling procedure should be incorporated in the individual product requirements or specifications. For semifinished products, the specimens should be selected after the material has been sufficiently cropped and suitable discards made. If the locations of the different ingots and portions of ingots in the heat cannot be identified in the lot being tested, random sampling should involve a greater number of test specimens for an equivalent weight of steel. A value for the inclusion content of an isolated piece of steel, even if accurately determined, should not be expected to represent the inclusion content of the whole heat.

6.9 The size and shape of the wrought steel product tested has a marked influence on the size and shape of the inclusions. During reduction from the cast shape by rolling or forging, the inclusions are elongated and broken up according to the degree of reduction of the steel cross section. In reporting results of inclusion determinations, therefore, the size, shape, and method of manufacture of the steel from which the specimens were cut must be stated. In comparing the inclusion content of different steels, they must all be rolled or forged as nearly as possible to the same size and shape, and from cast sections of about the same size. Specimens cut lengthwise or parallel to the direction of rolling or forging shall be used.

6.10 It may be convenient, in order to obtain more readily comparable results, to forge coupons from larger billets. These forged sections may then be sampled in the same way as rolled sections. Exercise care, however, to crop specimens of sufficient length from the billets for forging; otherwise, there is danger of the shear-dragged ends being incorporated in the specimens. Such distorted material will give a false result in the inclusion determination. To avoid this, it is helpful to saw the ends of the billet length for forging and to take the specimen from the middle of the forged length.

6.11 Several of the methods described in these test methods require that a specific area of the prepared surface of the specimen is surveyed, and all the significant inclusions observed be recorded and expressed in the results. The reported result for each specimen examined is, therefore, a more accurate representation of the inclusion content than a photomicrograph or diagram. A disadvantage of the Worst Field approach is that no such distribution of inclusion ratings is obtained.

6.12 To make comparisons possible between different heats and different parts of heats, the results shall be expressed in such a manner that an average for the inclusion content of the different specimens in the heat can be obtained. When the lengths of the inclusions are measured, the simplest number is that for the aggregate length of all the inclusions per area examined; however, it may be desirable not merely to add the lengths but also to weight the inclusions according to their individual lengths. The length of the largest inclusion found and the total number of inclusions may also be expressed.

[7] The JK chart derives its name from its sponsors Jernkontoret, the Swedish Ironmasters Association.

[8] Note that while these methods are called comparison chart methods, the procedure used may also consist of length measurements or counts of inclusions, or both.