Designation: E2142 – 08 (Reapproved 2015)

Standard Test Methods for Rating and Classifying Inclusions in Steel Using the Scanning Electron Microscope[1]

This standard is issued under the fixed designation E2142; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers procedures to obtain particle size distribution, chemical classification, and Test Methods E45 ratings of inclusions in steels using an automated scanning electron microscope (SEM) with X-ray analysis and automatic image analysis capabilities.

1.2 There are three discrete methods described. Method 1 is the SEM analog of Test Method E45, which uses image analysis and light microscopy to produce automated Test Methods E45 ratings. Method 2 produces similar ratings based predominantly on sorting inclusions by chemistry into the traditional classes defined in Test Methods E45. Method 3 is recommended when explicit detail is needed on particular inclusion types, not necessarily defined in Test Methods E45, such as to verify the composition of inclusions in inclusion-engineered steel. Method 3 reports stereological parameters such as volume or number fraction, rather than Test Methods E45 type ratings.

1.3 This test method deals only with the recommended test methods and nothing in it should be construed as defining or establishing limits of acceptability for any grade of steel or other alloy where the method is appropriate.

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

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

E45 Test Methods for Determining the Inclusion Content of Steel

E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope

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

E1508 Guide for Quantitative Analysis by Energy-Dispersive Spectroscopy

2.2 *Adjuncts:*

ANSI/IEEE STD 759 IEEE Standard Test Procedure for Semiconductor X-Ray Energy Spectrometers[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Analysis Rules*—

3.2.1.1 *acquisition analysis rules*—include the criteria to terminate X-ray collection (counts or time, or both), the list of elements to be analyzed, the number of fields or particles to be analyzed, morphologies of particles from which spectra will be collected, etc. (see Appendix X1 for a more complete listing of typical Acquisition Rules).

3.2.1.2 *post-acquisition analysis rules*—define ratios of X-ray intensities or elemental compositions required to identify

[1] These test methods are under the jurisdiction of ASTM Committee E04 on Metallography and are the direct responsibility of Subcommittee E04.11 on X-Ray and Electron Metallography.

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

[3] Available from Institute of Electrical and Electronics Engineers, Inc. (IEEE), 445 Hoes Ln., P.O. Box 1331, Piscataway, NJ 08854-1331, http://www.ieee.org.

an inclusion as belonging to a particular chemical classification and, for Methods 1 and 2 herein, define the main inclusion class (A, B, or C) to which each chemical classification belongs.

3.2.2 *chemical classification*—defined compositional categories in which inclusions are placed according to the analysis rules. Categories may be broad (for example, sulfide, aluminate, silicate) or more precise (for example, calcium sulfide, calcium silicate, anorthite, etc.).

3.2.3 *critical aspect ratio*—the aspect ratio of a single inclusion that defines the boundary between "globular" and "elongated".

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

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

4. Summary of Test Method

4.1 A properly prepared as-polished metallographic specimen is mounted in a computer-controlled SEM equipped with image analysis and X-ray analysis subsystems such that inclusions intersecting the plane of polish can be imaged and analyzed.

4.2 During analysis, an inclusion is detected by its gray level in the back-scattered electron signal. Once detected, size parameters of the inclusion are automatically determined and its X-ray spectrum collected. Particle morphology, chemistry, and location are stored in the computer, allowing re-examination of the data or the particle itself. In this manner, a complete or partial size distribution of inclusions by chemical class can be determined.

4.3 There are three methods described (see Fig. 1):

4.3.1 Method 1 is most similar to Test Methods E45 which uses light microscopy, and is intended to be its SEM analog. As such, it uses morphology as the primary basis for sorting particles into classes. As in Test Methods E45, a critical aspect ratio of 2 is defined. Chemistry is used to identify sulfide inclusions and to discriminate among sulfides when more than one type is present, as well as to ensure that exogenous inclusions and surface scratches and debris are not included in the ratings. Inclusions will be classified into four categories, Types A, B, C and D as described in Test Methods E45. Elongated sulfides are Type A. Other inclusions are oxides and are classified as Types B, C or D, depending on their morphology, as follows: discontinuous stringers of three or more inclusions with member particles having low aspect ratio are Type B; discontinuous stringers of two or more inclusions with member particles having high aspect ratio and single elongated oxide inclusions are Type C; remaining isolated inclusions are Type D. The categories will be further subdivided as thin, heavy and oversized, according to their width (tabulated in Table 1). E45-equivalent ratings are determined and recorded, and reported particles are usually limited to those ≥ 2 µm in size.

4.3.2 The inclusion classification strategy in Method 2 is based predominantly on chemistry, but uses morphology when necessary, such as to classify Type D (globular) inclusions and to compute severity ratings for Type B and C inclusions (which require determination of stringer lengths). Method 2 is based on the underlying intention of Test Method E45, namely, that Type A inclusions are deformable sulfides, Type B inclusions are non-deformable oxides (typically alumina), and Type C inclusions are deformable oxides (typically silicates). Each defined chemical class is assigned to one of these categories. Once classifications are made based on chemistry, the globular particles from each classification, or from designated classifications, can be re-classified as Type D inclusions. E45-equivalent ratings are determined and recorded, and reported particles are usually limited to those ≥ 2 µm in size.

4.3.3 Method 3 allows inclusions to be analyzed and reported in a manner individualized to the material and application of interest. It allows complete freedom in formulating chemical classes. Aspect ratio definitions can be chosen appropriately for the application. Termination of the analysis can be based on detecting a certain number of inclusions rather than sample area. Size and morphology distributions of all inclusions by chemical class are determined. Indirect terminology such as "Type A, B, C and D" and "thin" and "heavy" is not used; rather, inclusions are classified directly by chemical class and size range of interest. Particles may be further subdivided by morphology. Method 3, as in Practice E1245, reports basic stereological parameters, such as volume and number fractions of inclusions within each field, as well the maximum Feret's diameter for each inclusion. This method would be used for custom analyses, such to report all non-sulfide particles by thickness, as may be useful in tire cord applications.

5. Significance and Use

5.1 This test method is established to cover automated SEM/EDX-based procedures for:

5.1.1 Rating the inclusion content of steels based on procedures listed in Standards E45 and E1245, with the significant difference that the composition of the individual inclusions, as determined by X-ray analysis, is utilized to sort them into chemical classes.

5.1.2 Determining the number, size and morphological distribution of inclusions in steels sorted by chemical class.

5.2 Methods 1 and 2 of this test method are primarily intended for rating the inclusion content of steels deoxidized with silicon or aluminum, both silicon and aluminum, or vacuum-treated steels without either silicon or aluminum additions. Guidelines are provided to rate inclusions in steel treated with rare earth additions or calcium-bearing compounds (13.4). 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).

5.3 Methods 1 and 2 will provide a quantitative rating of the inclusion content in half-severity number increments from 0 to

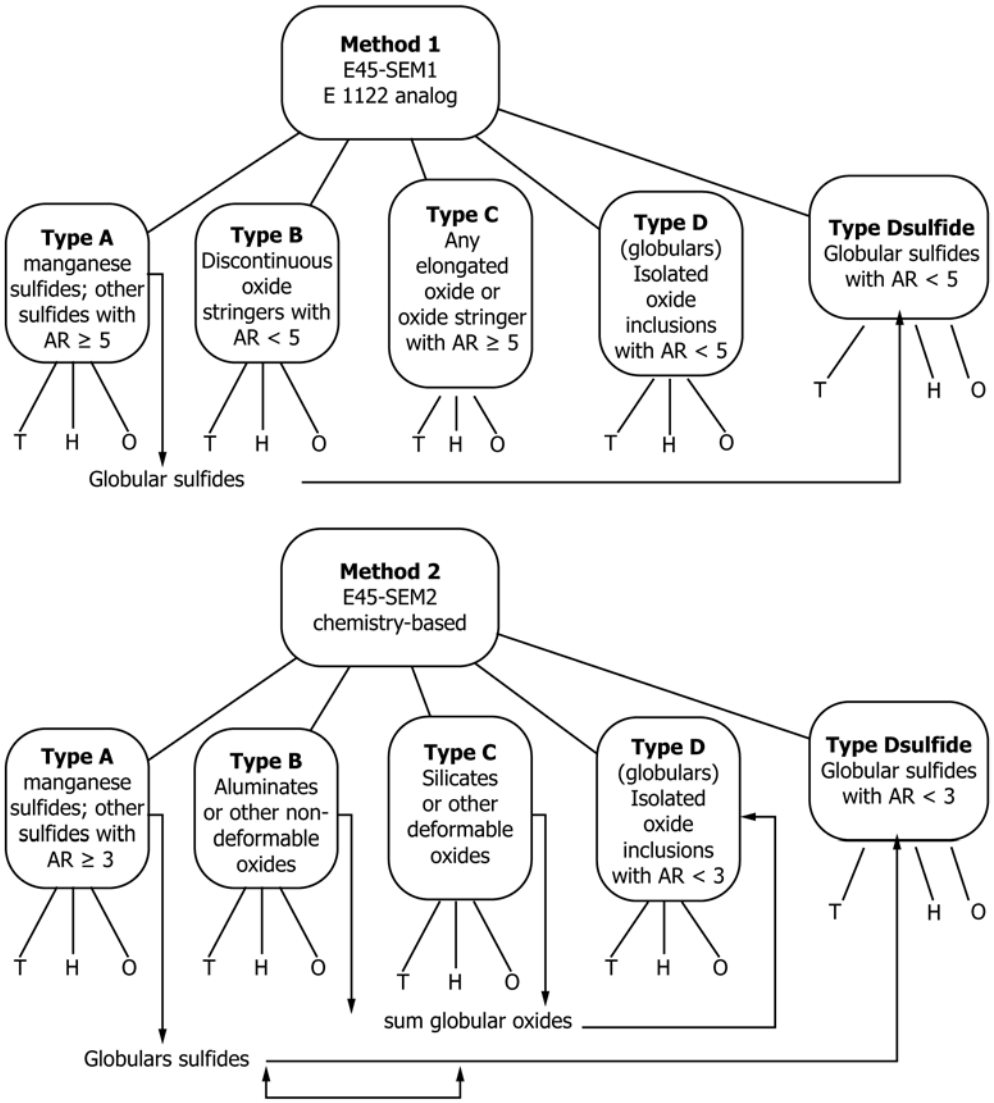

FIG. 1 Illustration of Classification Methods