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Standard Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel[1]

This standard is issued under the fixed designation E 768; 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 guide[2] covers two preparation methods for steel metallographic specimens that will be analyzed for nonmetallic inclusions with automatic image analysis (AIA) equipment. The two methods of preparation are offered as accepted methods used to retain nonmetallic inclusions in steel. This guide does not limit the user to these methods.

1.2 A procedure to test the suitability of the prepared specimen for AIA inclusion work, using differential interference contrast (DIC), is presented.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

E 3 Practice for Preparation of Metallographic Specimens

E 7 Terminology Relating to Metallography

E 45 Test Methods for Determining the Inclusion Content of Steel

E 883 Guide for Reflected-Light Photomicrography

E 1122 Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis

E 1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions used in this practice, refer to Terminology E 7.

3.1.2 *differential interference contrast microscopy*—a comprehensive definition appears in Guide E 883, paragraph 11.8.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *rigid grinding disk*—a non-fabric support surface, such as a composite of metal/ceramic or metal/polymer, charged with an abrasive (usually 6 to 15-µm diamond particles), and used as the fine grinding operation in a metallographic preparation procedure.

4. Significance and Use

4.1 Inclusion ratings done either manually using Test Methods E 45 or automatically using Practice E 1122 or E 1245 are influenced by the quality of specimen preparation. This guide provides examples of proven specimen preparation methods that retain inclusions in polished steel specimens.

4.2 This guide provides a procedure to determine if the prepared specimens are of suitable quality for subsequent rating of inclusions. None of these methods should be construed as defining or establishing specific procedures or limits of acceptability for any steel grade.

5. Preparation Methods

5.1 *Background*:

5.1.1 The inclusions in the plane of polish must be fully preserved and clearly visible. Preparation should not produce excessive relief around the perimeter of the inclusions that would exaggerate the size and number of inclusions on the plane of polish. In many cases, the preparation of specimens for inclusion rating is more readily performed after the specimens have been hardened by a suitable heat treatment procedure (austenize, quench to fully martensitic structure, temper at a relatively low temperature).

5.1.2 Cleanliness is an important consideration in all stages of specimen preparation.

5.1.2.1 Heat-treated specimens should be wire brushed or shot blasted or have the surface ground to remove adherent scale.

5.1.2.2 After completing the grinding steps and before performing the polishing steps, the specimens and specimen holders must be cleaned to prevent contamination of the next preparation step. Cleaning the specimens and specimen holders between each grinding step can eliminate contamination of coarse abrasives to the following finer preparation step.

5.1.2.3 After the preparation is complete, swab the surface carefully with cotton and a water/soap solution containing a corrosion inhibitor such as a machine coolant or ethyl alcohol solution to remove any films or other debris that would interfere with the inclusion rating.

[1] This test method is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Sampling Specimen Preparation, and Photography.

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[2] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: E04–1002.

[3] 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.

5.1.2.4 It is advisable to perform the inclusion analysis as soon as possible after preparation to minimize staining or other problems that can affect the analysis.

5.1.3 The two methods that follow have been found to be reliable procedures for retaining inclusions in steel and achieving the desired results when evaluated by DIC. There are other methods that will result in a quality specimen as revealed by DIC. Each laboratory should develop preparation procedures for their materials so that the prepared surfaces meet the requirements presented in 6 of this guide.

5.1.4 As described in Prractice E 3, the specimens may be sectioned and mounted to ease handling during preparation. It is advisable to use a mounting medium that is hard enough to preserve edges and maintain flatness.

5.1.5 Abrasive grit size designations in this guide are expressed in the American National Standards Institute (ANSI) or Coated Abrasives Manufacturers Institute (CAMI) system units with the corresponding Federation of European Abrasive Procedure (FEPA) numbers in parentheses. Table 1 provides a correlation between these two systems and the approximate median particle diameter for a given grit size in micrometres.

5.1.6 Most preparation systems apply pressure on the specimens being processed. The best pressure to be used for each preparation step should be determined experimentally. Conversions between applied force and pressure are discussed in the Appendix X1.

NOTE 1—Care must be taken to protect the polished specimen surface from scratches or contaminants when using a specimen leveling device.

5.2 *Silicon Carbide Procedure*:

5.2.1 A summary of the silicon carbide procedure can be found in Table 2.

5.2.2 When using a semi-automatic polishing equipment, grinding and polishing should be performed using approximately 18 kPa pressure per specimen. (For a specimen holder containing six 32-mm mounts, a force of approximately 87 N must be applied (see X1.4.2).

TABLE 1 Comparison of ANSI (CAMI) versus FEPA versus Median Diameter of Grit Size in Micrometers

ANSI[A] (CAMI)[B]	FEPA[C]	Approximate Median Diameter (μm)
60	P60	250
80	P80	180
100	P100	150
120	P120	125
150	P150	90
180	P180	75
220	P220	63
240	P240	58.5
	P280	52.2
280	P320	46.2
320	P360	40.5
	P400	35.0
360	P500	30.2
400	P600	25.8
	P800	21.8
500	P1000	18.3
600	P1200	15.3
800	P2400	8.4
1200	P4000[D]	3.0

[A] ANSI - American National Standards Institute
[B] CAMI - Coated Abrasives Manufacturers Institute
[C] FEPA - Federation of European Abrasive Producers
[D] Not a FEPA designation

5.2.2.1 Low pressures are recommended to ensure the retention of an assortment of inclusion types found in a variety of steel grades. The relatively low pressures suggested in this procedure will not necessarily result in a satisfactory polish for etching and the further evaluation of the specimen's general microstructure. The pressures used in the following rigid disk procedure are more likely to result in a surface more satisfactory for revealing general microstructures.

5.2.3 Grind the specimens on ANSI 80 grit (P80 FEPA) silicon carbide paper to ensure all sectioning artifacts and deformation damage have been removed and the entire specimen surface is co-planar to the grinding surface.

NOTE 2—If the sectioning method resulted in a smooth face and little deformation damage, and if after securing the specimens in a fixture for polishing, the entire surface of interest is co-planar to the grinding surface, then finer grit papers, such as ANSI 180 to 240 (P180 to P240 FEPA) can be used for the initial grinding step.

5.2.3.1 An adequate flow of water should remove all loosened abrasive and grinding debris from the paper during the grinding procedure. The flow of water should ensure the specimen is kept cool during grinding.

5.2.4 Continue grinding through the sequence of silicon carbide papers listed in Table 2. It may be necessary to clean the samples between every grinding step to prevent contamination of the next preparation step.

5.2.5 After completing the entire grinding operation, clean the specimens thoroughly, using ethyl alcohol and cotton, then rinse and dry. Ultrasonic cleaning can be used.

5.2.5.1 The use of a soap and water solution in an ultrasonic cleaner can attack non-metallic inclusions in some carbon and low alloy steels, leading to an exaggeration in the inclusion's apparent size. Adding an inhibitor (such as that used as a machining coolant) may reduce this size exaggeration.

5.2.6 Polish the specimens using 3-μm diamond abrasive on a low nap cloth, such as woven wool, for 50 s. Clean and dry the specimens as described in 5.2.5.

5.2.7 Polish the specimens using a 1-μm diamond abrasive on a high nap cloth for 50 s. Clean and dry the specimens as described in 5.2.5.

5.2.8 A final manual polishing step may be added, using 0.25-μm diamond abrasive on a low nap cloth for 10 to 20 s, using relatively high pressure. Clean and dry the specimens as described in 5.2.5.

5.2.8.1 Ultrasonic cleaning is not recommended after the final manual or automatic polishing step. Ultrasonic cleaning can cause cavitation damage, ultimately distorting the inclusion sizes.

NOTE 3—Depending on the material, it may be advisable to avoid water after polishing.

5.3 *Rigid Grinding Disk Procedure*:

5.3.1 A summary of the rigid grinding disk procedure can be found in Table 3.

5.3.2 Semi-automatic grinding equipment is required, with specimens contained in a circular fixture for the entire procedure. For 5.3.2-5.3.5, a pressure of 42 kPa should be applied