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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 E768; 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.

ε[1] NOTE—Footnote 1 was editorially corrected in May 2016.

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.

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

E45 Test Methods for Determining the Inclusion Content of Steel

E883 Guide for Reflected–Light Photomicrography

E1122 Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis (Withdrawn 2006)[4]

E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

2.2 *ASTM Adjuncts:*[5]

ADJE0768 Differential Interference Contrast Magnification 100X and 500X (6 micrographs)

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions used in this practice, refer to Terminology E7.

3.1.2 *differential interference contrast microscopy*—a comprehensive definition appears in Guide E883, 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 E45 or automatically using Practice E1122 or E1245 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

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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.

[4] The last approved version of this historical standard is referenced on www.astm.org.

[5] A colored plate, consisting of six micrographs that illustrate the use of DIC in determining a properly prepared sample (at 100x and 500x), is available from ASTM Headquarters. Order Adjuct: ADJE0768.

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.

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 E3, 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).

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.

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

5.2.3 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.

TABLE 2 Preparation Method I Silicon Carbide Abrasive Paper Grinding

Surface	Coolent/ Lubricant	Abrasive Size/Type ANSI[A] [FEPA][B]	Time (seconds)	Force[C] newtons [lbs]	Surface Speed RPM	Relative Rotation
			Planar Grinding			
Paper	Water	80 [P80] grit SIC	60	14 [2]	300	Complementary[D]
			Fine Grinding			
Paper	Water	120 [P120] grit SIC		14 [2]	300	Complementary
Paper	Water	240 [P240] grit SIC	60	14 [2]	300	Complementary
Paper	Water	320 [P500] grit SIC	60	14 [2]	300	Complimentary
Paper	Water	400 [P600] grit SIC	60	14 [2]	300	Complementary
Paper	Water	600 [P1200] grit SIC	60	14 [2]	300	Complimentary
Paper	Water	800[P2400] grit SIC	60	14 [2]	300	Complementary
			Rough Polishing			
NAP free cloth	Extending fluid	3 µm diamond	50	14 [2]	150	Complementary
			Final Polishing			
Low nap cloth	Extending fluid	1 µm diamond	50	14 [2]	150	Complementary
			Optional Polishing (manual)			
Low nap cloth	Extending fluid	1 µm diamond	50	relatively high	NA	NA[E]

[A]American National Standards Institute (ANSI) designation of grit size.
[B]Federation of European Producers of Abrasives (FEPA) designation of grit size.
[C]Force per 3-mm (1.25-in.) diameter specimen.
[D]Complementary rotation, surface and specimens spin in same direction.
[E]Since this optional step is completed manually, these two categories are not applicable.

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 per specimen. (For a specimen holder containing six 32-mm circular mounts, a force of approximately 203 N must be applied (see X1.4.3).

5.3.3 Grind for at least 15 s after achieving a co-planar condition, using a 150-grit alumina grinding stone at 1450 rpm or 80-grit (P80 FEPA) alumina/zirconia grinding paper at 300 rpm.

5.3.3.1 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.

TABLE 3 Preparation Method II - Rigid Grinding Disk Procedure

Surface	Coolant/ Lubricant	Abrasive Size/Type ANSI[A] [FEPA][B]	Time - seconds	Force[C] newtons [lbs]	Surface Speed RPM	Relative Rotation
			Planar Grinding			
Alumina/zirconia paper	Water	80 [P80] grit	>15	34 [8]	300	Complementary[D]
			Or			
Alumina grinding stone	Water	150 [P150] grit	>15	34 [8]	1450	Complementary
			Fine Grinding			
Rigid grinding disk	Alcohol/glycol	9 µm diamond	180	34 [8]	150	Complementary
			Rough Polishing			
Napless or stiff napped cloth	Alcohol/glycol	3 µm diamond	240	34 [8]	150	Complementary
			Final Polishing			
Soft short napped cloth	Alcohol/glycol	1 µm diamond	120	34 [8]	150	Complementary
			Optional Polishing			
Porous Synthetic Cloth	Water	Alkaline Colloidal Silica or Acidic Colloidal Alumina[E]	90	100 [23]	150	Complementary

[A]American National Standards Institute (ANSI) designation of grit size.
[B]Federation of European Producers of Abrasives (FEPA) designation of grit size.
[C]Force per 32-mm (1.25-in.) diameter specimen.
[D]Complementary rotation surface, and specimens spin in same direction.
[E]Water is substituted for the colloidal oxide during the last 20 s.

5.3.3.2 It may be necessary to clean the samples between every grinding step to prevent contamination of the next preparation step.

5.3.4 Finish grind using 9-µm diamond on a rigid grinding disk for 3 m at 150 rpm, using an alcohol/glycol lubricant. Clean the specimens and fixture thoroughly (ultrasonic cleaning is helpful) with alcohol and cotton, then dry with forced air.

5.3.4.1 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.

5.3.5 Rough polish using 3-µm diamond on a napless cloth (silk or non-woven chemotextile) or a low nap cloth (woven wool) for 4 min at 150 rpm using alcohol/glycol lubricant. Clean as in 5.3.4.

5.3.6 Final polish using 1-µm diamond on a high nap cloth for 2 min at 150 rpm, using alcohol/glycol lubricant. Clean as in 5.3.4.

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

5.3.7 An optional oxide-polishing step, using a porous synthetic cloth with either alkaline colloidal silica or acidic colloidal alumina, may be used if necessary to eliminate fine scratches. The conditions are 90 s at 150 rpm, 100 N, with water substituted for the colloidal oxide during the last 20 s. Clean as in 5.3.4.

6. DIC Evaluation Method[5]

6.1 A microscope equipped with DIC illumination at magnifications of about 100x and 500x should be used while developing the preparation procedure to verify the true surface topography of properly prepared specimens. The sensitive tint condition, using a first order gypsum (1λ) plate, is the most useful mode of DIC illumination (plane areas magenta; inclusions dark).

6.2 Observation at 100x will reveal whether residual scratches are present and whether background relief is evident.

6.3 At 500x in the DIC sensitive tint condition, all of the edges of inclusion particles should be sharp and recognizable as sharp edges. Narrow bright lines indicating narrow ditches at some inclusion edges may, however, still be seen. A test may be made to determine whether these ditches will be detected by the image analysis system.

6.3.1 The polarizer is slowly rotated away from the full DIC position and the inclusion edges watched for apparent motion. There could be a critical trough configuration beyond which the bright troughs turn black at some point in the transition to bright field illumination, and the inclusions appear to become larger. This condition indicates insufficient preparation. (The use of a filar eyepiece may be helpful at this point to determine whether the inclusion through has widened)

6.3.2 Narrow troughs can be accepted as harmless if they vanish into the background when the polarizer is opened a small part of its range.

6.4 Some fine scratches from final polishing will usually be seen under DIC at 500x. These normally also vanish on opening the polarizer and, therefore, should not affect quantitative measurement at lower magnification.

NOTE 4—While highly instructive, the DIC examination remains subjective with respect to the question of whether a reproducible and accurate surface preparation has been obtained. Tests by actual measurement are necessary to determine that errors in specimen preparation were not contributory to outlier observations.

7. Keywords

7.1 automatic image analysis; differential interference contrast; inclusions; specimen preparation; steel

APPENDIX

(Nonmandatory Information)

X1. APPLIED LOAD CONVERSIONS

X1.1 Automatic preparation machines commonly display applied force in either pound-force (lbf) or newtons (N). The ability to convert from one unit to another may be necessary when trying to interpret a documented procedure.

X1.1.1 To convert from pound-force to newton, multiply the pound-force value by 4.5.

X1.1.2 To convert from newton to pound-force, multiply the newton value by 0.225.

X1.2 When multiple specimens are held in a holder, the applied force must be divided by the number of specimens in the holder to determine the load per specimen. Some automated machines apply the load individually to each specimen and display the applied load accordingly.

X1.3 Caution should be taken when using automated machines that display pressure in pound-force per square inch (psi). Typically, the machine is displaying the air pressure within the loading cylinder and not the true pressure applied to either the specimen holder or each individual specimen.

X1.4 When converting from a force to a pressure, the surface area of the specimen must be determined. The value of force is then divided by the contact area to determine the required pressure.

X1.4.1 *Example 1*—What is the force in pound-force if 87 N is applied?

$$(87\ N) \times (0.225) = 19.6\ lbf \quad \text{(X1.1)}$$

X1.4.2 *Example 2*—What is the applied load, in newtons, required to apply a pressure of 18 kPa to an individual specimen in a holder containing six 32-mm circular mounts?

Pressure = (Force)/(Area), therefore . . . Force = (Pressure) × (Area)

Area = $\Pi\ r^2 = \Pi\ (16)^2 = 804.25\ mm^2$

Total Area = (6 mounts) × ($804.25\ mm^2$) = $4825.5\ mm^2$

18 kPa = 18 000 Pa = 18 000 N/m^2 = 0.018 N/mm^2

Therefore,

Force = (0.018 N/mm^2) × $4825.5\ mm^2$) = 86.9 N

X1.4.3 *Example 3*—What is the applied load, in newtons, required to apply a pressure of kPa to an individual specimen in a holder containing six 32-mm mounts?

Pressure = (Force)/(Area), therefore . . . Force = (Pressure) × (Area)

Area = $\Pi\ r^2 = \Pi\ (16)^2 = 804.25\ mm^2$

Total Area = (6 mounts) × ($804.25\ mm^2$ = $4825.5\ mm^2$

42 kPa = 42 000 Pa = 42 000 N/m^2 = 0.042 N/mm^2

Therefore,

Force = (0.042 N/mm^2) × $4825.5\ mm^2$) = 203 N