Designation: E 381 – 98

Standard Method of Macroetch Testing Steel Bars, Billets, Blooms, and Forgings[1]

This standard is issued under the fixed designation E 381; 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 Department of Defense.

1. Scope

1.1 Macroetching, which is the etching of specimens for macrostructural examination at low magnifications, is a frequently used technique for evaluating steel products such as bars, billets, blooms, and forgings.

1.2 Included in this method is a procedure for rating steel specimens by a graded series of photographs showing the incidence of certain conditions. The method is limited in application to bars, billets, blooms, and forgings of carbon and low alloy steels.

1.3 A number of different etching reagents may be used depending upon the type of examination to be made. Steels react differently to etching reagents because of variations in chemical composition, method of manufacture, heat treatment and many other variables. Establishment of general standards for acceptance or rejection for all conditions is impractical as some conditions must be considered relative to the part in which it occurs.

1.4 *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.* See the specific precautionary statement in 5.3.

2. Referenced Documents

2.1 *ASTM Standards:*

E 7 Terminology Relating to Metallography[2]

E 340 Test Method for Macroetching Metals and Alloys[2]

E 1180 Practice for Preparing Sulfur Prints for Macrostructural Examination[2]

2.2 ASTM Adjuncts:

Photographs for Rating Macroetched Steel (3 plates)[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this method, see Terminology E 7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Terminology Applicable Only to Ingot Cast Product*:

3.2.1.1 *splash*—a nonuniform etch pattern where irregularly-shaped areas exhibit a different etch contrast than surrounding areas. Splash is normally associated with molten steel which solidifies and oxidizes during initial pouring and which is not completely redissolved by the remaining molten steel.

3.2.1.2 *butt tears*—subsurface cracks normally parallel to the surface of the ingot mold wall.

3.2.1.3 *flute cracks*—cracks perpendicular to the surface of the ingot mold wall which may, or may not, extend to the surface of the product.

3.2.1.4 *burst*—a single or multi-rayed crack normally located at the center of the wrought product.

3.2.2 *Definitions Applicable Only to Continuously Cast Products*:

3.2.2.1 *chill zone*—rapidly cooled metal with a fine structure at the surface of the product which is normally continuous around that surface.

3.2.2.2 *chill zone crack*—any crack which is located partially or completely in the chill zone and may extend to the surface of the product.

3.2.2.3 *diagonal crack*—a crack which lies completely or partially in the diagonal regions of a non-round product where adjacent columnar or dendritic growth patterns intersect.

3.2.2.4 *subsurface crack*—a crack perpendicular to and just beneath the chill zone.

3.2.2.5 *mid-radius crack*—a crack perpendicular to the surface of the product located approximately halfway between the surface and center of the product.

3.2.2.6 *center crack*—a crack with an aspect ratio (length/width) of approximately 3 or greater located at, or near, the center of the product.

3.2.2.7 *star crack*—a star-shaped or multi-rayed crack at the center of the product.

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[1] This method is under the jurisdiction of ASTM Committee E-4 on Metallography, and is the direct responsibility of Subcommittee E04.01 on Sampling, Specimen Preparation, and Photography.

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[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] Available from ASTM Headquarters. Order Adjunct: ADJE038101 (Plate I), ADJE038102 (Plate II), and ADJE038103 (Plate III).

3.2.2.8 *scattered porosity*—multiple round or irregularly-shaped pores uniformly distributed about the central portion of the product.

3.2.2.9 *white band*—a light etching continuous band(s) parallel to the surface of the product usually located between the one-quarter and three-quarter radius position, normally associated with electromagnetic stirring.

3.2.2.10 *columnar grains*—a coarse structure of parallel, elongated grains formed by unidirectional growth during solidification.

3.2.3 *Conditions Applicable to Both Ingot and Continuously Cast Product*:

3.2.3.1 *nonmetallic inclusions*—nonmetallic particles trapped in the steel or the voids resulting when inclusions are dissolved by the macroetchant.

3.2.3.2 *pattern*—a dark etching band, usually rectangular or square, enclosing the central portion of the cross section, normally visible only in wrought product. In ingot cast product, it is sometimes called ingotism or ingot pattern.

3.2.3.3 *pipe or center void*—a single large cavity located at, or near, the center of the product.

3.2.3.4 *center unsoundness*—multiple round or irregularly-shaped voids concentrated at the center of the product.

3.2.3.5 *dark center*—a dark etching area at the center of the product. Dark center is solid material and should not be confused with center unsoundness.

3.2.3.6 *pinholes*—small pores which lie at, or just beneath, the surface of the product.

3.2.3.7 *mold slag*—inclusions which are normally associated with entrapped fused mold powder and are normally located at, or just beneath, the surface of the product. They are usually found in continuously cast or bottom poured products.

3.2.3.8 *flakes*—short discontinuous internal cracks attributed to stresses produced by localized transformation and hydrogen solubility effects during cooling after hot working. In an etched transverse section, they appear as short, tight discontinuities which are usually located in the midway to center location of the section. They are also known as shatter cracks or hairline cracks.

3.2.3.9 *gassy*—irregularly-shaped voids which may, or may not, be uniformly distributed throughout the cross section. These may be located anywhere from the near surface region of the product to the center of the product, depending on the source and severity of the condition.

3.2.3.10 *dendritic*—a "tree-like" pattern with branches (primary, secondary, and tertiary arms) due to compositional differences that arise during solidification. For a specific composition, a weak dendritic structure is associated with a low superheat while a strong dendritic structure is associated with a high superheat during casting. Compositional differences also influence the clarity of the dendrites.

4. Significance and Use

4.1 Macroetching is used in the steel industry because it is a simple test that will provide information about the relative homogeneity of the sample. The method employs the action of an acid or other corrosive agent to develop the macrostructural characteristics of a suitably prepared specimen. The name implies that the etched surface is examined visually, or at low magnifications (usually <10×).

4.2 Macroetching will show: (*1*) variations in structure such as grain size, dendrites, and columnar structure; (*2*) variations in chemical composition such as segregation, coring, and banding; and, (*3*) the presence of discontinuities such as laps, seams, cracks, porosity, bursts, pipe. and flakes.

4.3 When, in accordance with the requirements of the inquiry, contract, order or specifications, forgings, billets, blooms, etc., are to be produced subject to macroetch testing and inspection, the manufacturer and the purchaser should be in agreement concerning the following: (*1*) the stage of manufacture at which the test shall be conducted; (*2*) the number and locations of the sections to be examined; (*3*) the necessary surface preparation prior to etching of the specimen; (*4*) the etching reagent, temperature, and time of etching; and, (*5*) the type, size, number, location, and orientation of conditions that are to be considered injurious.

4.4 When not specified, the procedures of the test may be selected by the manufacturer to satisfy the requirements of the governing specification.

4.5 When agreed upon by purchaser and producer, sulfur printing of as cast-sections, if continuously cast, is an acceptable alternative to macroetching. Sulfur printing shall be performed in accordance with Practice E 1180. Examination and rating of specimens shall be in accordance with Sections 10 and 11 of this (E 381) standard.

4.6 Steel from ingots shall be examined according to procedures described in Section 9. Continuously cast steel blooms and billets, in the as cast condition, shall be examined according to the procedures described in Sections 10 and 11. With reductions over a 3:1 area ratio, wrought product from continuously cast steel may be examined according to Section 9.

5. Reagents

5.1 The most common reagent for macroetching iron and steel is a 1:1 mixture, by volume, of concentrated hydrochloric acid (HCl) and water. The hydrochloric acid need not be reagent grade. Commercial quality hydrochloric acid (also known as muriatic acid) is satisfactory. The etching solution should be clear and free from scum. It should be hot, 70 to 80°C (160 to 180°F). The reagent should be used under a fume hood, or some other means of carrying off the corrosive fumes must be provided. The solution may be heated without serious change in concentration. The etching solution may be reused if it has not become excessively contaminated or weakened.

NOTE 1—The addition of hydrogen peroxide (H_2O_2) may be necessary in order to provide sufficient reaction to properly etch some types of product. This should be added to an etching bath that is operating at room temperature.

5.2 A second macroetching solution, favored by some as producing a clearer structure, is composed of concentrated HCl (38 volume %), sulfuric acid, H_2SO_4 (12 volume %) and water, H_2O (50 volume %). (See 5.1 with respect to acid quality, heating, and ventilation.)

5.3 Observe caution in mixing macroetch solutions. The acids are strong and they can cause serious chemical burns. Add acid slowly to water with stirring. This is especially true

for sulfuric acid. Mix solutions and macroetch under a fume hood.

5.4 An ammonium persulfate solution, a 10 to 20 % aqueous solution, is used primarily on longitudinal sections to detect certain types of ghost lines, segregation, flow lines, etc. A freshly made solution is necessary for best results. The solution should be swabbed on the finished surface at room temperature. Inspection is most effective when done while the piece is still wet.

5.5 A nitric acid solution, 5 % or 10 % nitric acid in alcohol or water, is used to detect local overstraining, grinding cracks, overheated areas, and depth of carburized or decarburized surface zones. The use of this reagent necessitates a smooth surface. The reagent is used at room temperature by immersion or swabbing.

5.6 Many other reagents have been used for special applications. When the use of a reagent other than those described in 5.2-5.5 is desired, it should be by agreement between the purchaser and the manufacturer. (See Test Method E 340 for other etching solutions.)

6. Sampling

6.1 When macroetching is used as an inspection procedure, sampling should be done at an early stage of manufacture so that, if the material is inadequate, the minimum amount of unnecessary processing is done (or the processing can be modified to salvage the material). For ingot cast product, the specimen is usually taken after ingot breakdown. Billets or blooms going into small sizes are sampled after the initial breakdown. Sampling of continuously cast product is usually done in the as-cast condition, or after intermediate or final processing, depending on size and preference. Random sampling of the finished product may be performed if the locations within the cast are not known.

6.2 Normally, the specimens are disks cut from the ends of bars, billets or blooms. Enough material should be discarded before taking the specimen to eliminate any extraneous effects of rolling such as “fish tails.” Specimens may be cut cold by any convenient means; saws and abrasive cut-off wheels are particularly effective. Torch cutting or other hot cutting will materially affect the structure of the specimen and may be used only when necessary to remove a larger piece prior to cutting to size by cold methods. Sufficient torch cut material should be removed by cold cutting to eliminate the thermal effects of torch cutting.

6.3 The macroetch test, as applied to the inspection of steel products of this specification, is carried out on slices, usually 13 to 25 mm (½ to 1 in.) in thickness. Disks or specimens are usually cut to reveal a transverse surface, but the requirements of the specification, contract, or order may include the preparation and examination of a longitudinal surface.

6.4 When the test is conducted on single pieces (bar, billet, bloom, etc.), the purchaser may specify that the specimen disks be cut to represent both ends, or only one end, of the piece.

6.5 When the test is conducted on a number of pieces made from a heat of steel, the purchaser may require that each piece be individually tested; or, a representative method of sampling may be agreed upon by the manufacturer and the purchaser.

6.6 For the indication of certain internal types of discontinuities, such as thermal cracks or flakes, the purchaser may specify that disks for macroetch inspection be taken a certain minimum distance from the ends of the specimen. In the case of forgings, depending upon prior agreement, this may be accomplished by adding excess metal for discard on the ends or ends of the forging; or, by forging in multiple lengths and removing the test disk between individual pieces when cutting up the multiple forging.

7. Preparation

7.1 Specimen preparation need not be elaborate. Any method of preparing smooth surfaces with a minimum amount of cold work should be satisfactory. Disks may be faced on a lathe or a shaper. The usual procedure is to take a roughing cut, then a finishing cut. This will generate a smooth surface and remove cold work from prior operations. Sharp tools are necessary to produce a good specimen. Grinding, which also may be used, is usually conducted in the same manner, using free-cutting wheels and light feeds. When fine detail must be revealed, the specimen should be prepared with metallographic grinding papers, or even with a metallographic polish.

7.2 After specimen preparation, the surface to be etched should be cleaned. Any grease, oil, or other residue will produce an uneven attack and must be removed. It may be necessary to use solvents to clean the surface. Once cleaned, care should be taken to avoid touching or otherwise contaminating the surface.

7.3 Large cross sections may be cut into smaller pieces to facilitate handling and to comply with safety requirements. The sectioning of the large specimens should be done so as not to disturb the central portion of the section.

8. Procedure

8.1 Macroetching is carried out in containers which must be resistant to the attack of the etching reagent. Small pieces may be etched in glass or porcelain vessels of the types commonly available in laboratories. Larger disks are etched in corrosion-resistant alloy vessels, various types of ceramic pots, rubber-lined kettles or wooden tanks. If metal tanks are used, lead is most common for containing sulfuric acid solutions while high-nickel iron or high-silicon iron are preferred to contain hydrochloric acid solutions. A nickel-molybdenum alloy can be used for containing solutions of sulfuric or hydrochloric acid, or both. If the tank is metallic, the disks being etched should not be in contact with each other or with the vessel. Such contact will set up electrolytic (galvanic) couples and will produce an uneven and misleading etch attack. Resins in wood used to make tanks sometimes act as uncertain and uneven inhibitors resulting in unsatisfactorily etched disks.

8.2 Mix the solution and place it in a suitable tray, dish or tank and bring the solution to the desired operating temperature before commencing etching. Do not place specimens in a cold solution and then heat it to the desired temperature. The specimens may be placed directly into the solution, but the best practice is to place the specimen in corrosion-resistant baskets or on corrosion-resistant supports, such as glass rods underneath the specimen to raise it off the bottom of the dish. Maintain a sufficient volume of solution in the vessel to cover the specimen with a layer of at least 25 mm (1 in.) of liquid.

When etching is completed, remove the specimen from the solution, taking care not to mar the specimen surface. Remove the smut that forms on the specimen surface by scrubbing the surface with a stiff brush under hot running water. This brush may have natural vegetable or synthetic fibers but not metal fibers. After the smut is removed, rinse the specimen under hot running water and blow it dry with compressed air. The specimen should not be blotted dry. The best time for examination is immediately after drying. Dried surfaces may be protected with oil or a transparent lacquer.

8.3 Etching time will vary depending on composition, size, whether preheated, or not, etc. Etch the specimens to reveal the structure clearly and then remove it from the etch bath. Overetching can lead to misinterpretation. In most cases, 15 to 30 min will be sufficient for hot-acid etching. Etching times with cold solutions (see 5.4 and 5.5) are shorter.

9. Examination of Ingot Cast Product Specimens

9.1 After drying (except when using the ammonium persulfate etchant described in 5.4), compare the appearance of the specimen to the photographs in Plate I and report the photograph most nearly representing the appearance of the specimen in each of the series. Also report the presence of any of the conditions in Plate II.

9.2 Several series of photographs of etched specimens are presented in two groups.

9.2.1 *Plate I*—Graded series for three conditions: (1) subsurface conditions; (2) random conditions; and, (3) center segregation.

9.2.2 *Plate II*—Ungraded series showing various other conditions.

10. Examination of Continuously Cast Product Specimens

10.1 Specimens are examined after etching and drying (except when using the ammonium persulfate etchant described in 5.4). The type of condition present may be identified by comparison with Plate III. Rating of each condition is accomplished by measurement. Some conditions related to the cast structure (for example, chill zone and columnar structure) may not be discernible in product which has been reduced by rolling and forging while pattern is only discernible in a wrought specimen.

10.2 Plate III consists of photographs of macroetched specimens showing the typical location and general appearance of conditions found in continuously cast product. Some of the conditions are represented by sketches drawn on the photomacrographs. Plate III is used to identify the type of condition observed on the macroetch specimens.

11. Rating Methods for Continuously Cast Product Conditions

11.1 This section describes methods for rating conditions in continuously cast product. Conditions to be rated and their level may be established by agreement between the purchaser and producer.

11.1.1 Linear conditions shown on Plate III are rated as the length of the maximum individual condition present and are expressed as a percentage of the square root of the transverse cross-sectional area of the specimen.

11.1.2 Circular conditions shown on Plate III are rated as the diameter of a circle which circumscribes the maximum individual condition and are expressed as a percentage of the square root of the transverse cross-sectional area of the specimen. In highly rectangular product, circular conditions may not be compatible with this rating method. In those cases, circular conditions shall be rated as the average of the maximum length and maximum width of the condition and expressed as a percentage of the square root of the transverse area of the specimen.

11.1.3 Pinholes are to be rated by counting the number of pinholes present and expressing the frequency either as pinholes per unit length or as the total number.

11.1.4 White band is to be rated as either present or not present.

11.1.5 Chill zone is to be rated as either present or not present in a continuous layer around the circumference of the product.

11.1.6 The equiaxed zone is to be rated as the minimum dimension of the equiaxed zone divided by the minimum section dimension.

11.1.7 If nonmetallic inclusions are observed during macroetch inspection, their size, location and quantity may be noted on the inspection report. Other rating methods may be established per agreement between the user and producer.

11.1.8 Mold slag is rated as either present or not present.

11.1.9 Flakes are rated as either present or not present.

12. Report

12.1 Report the following information:

12.1.1 Date of the test,

12.1.2 Name(s) of the personnel performing the test,

12.1.3 Identity of the material (heat number, grade, order number, etc.),

12.1.4 Location, orientation and identifying numbers of the test specimens,

12.1.5 Nature of the disk surface preparation,

12.1.6 Etching reagent used, and its temperature,

12.1.7 Duration of etching, and

12.1.8 Any specifications governing the testing.

12.2 Document the type, number, location and orientation of the observed conditions (see Sections 9 and 10) and the ratings (see Section 11) of the measured macrostructural conditions in continuously cast specimens.

12.3 List any specific purchaser-producer agreements regarding either the test method or the rating.

12.4 If sulfur printing was employed, list the details of the test procedure, as defined in Practice E 1180.

13. Precision and Bias

13.1 No statement is made about either the precision or the bias of this method for macroetch testing of steel bars, billets, blooms, and forgings since the result merely states whether there is conformance to the criteria for success specified in the procedure.

14. Keywords

14.1 bars; billets; blooms; bottom-poured ingots; continuous casting; forgings; homogeneity; ingots; macroetch; macrostructure; quality control; steel; sulfur printing