Designation: E381 – 01(Reapproved 2006)

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

This standard is issued under the fixed designation E381; 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:*[2]

E7 Terminology Relating to Metallography

E340 Test Method for Macroetching Metals and Alloys

E1180 Practice for Preparing Sulfur Prints for Macrostructural Evaluation

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 E7.

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.

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

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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 ASTM Headquarters. Order Adjunct: ADJE038101 (Plate I), ADJE038102 (Plate II), and ADJE038103 (Plate III).

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.

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.

3.2.3.11 *refilled crack*—A defect formed during the solidification of continuously cast steel, either external (bulging) or internal (shrinkage) forces result in the separation of crystallites so as to permit solute rich liquid to refill the gap as it forms.

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 E1180. Examination and rating of specimens shall be in accordance with Sections 10 and 11 of this (E381) 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.