

Designation: A604/A604M - 07

Standard Practice for Macroetch Testing of Consumable Electrode Remelted Steel Bars and Billets¹

This standard is issued under the fixed designation A604/A604M; 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 This practice² covers testing and inspection and is applicable to bars, billets, and blooms of carbon, alloy, and stainless steel which have been consumable electrode remelted.

1.2 For the purpose of this practice, the consumable electrode remelting process is defined as a steel refining method wherein single or multiple electrodes are remelted into a crucible producing an ingot which is superior to the original electrode by virtue of improved cleanliness or lower gas content or reduced chemical or nonmetallic segregation. See Appendix X1 and Appendix X2 for descriptions of applicable remelting processes.

1.3 This practice and the accompanying comparison macrographs³ are generally applicable to steel bar and billet sizes up to 225 in.² [1450 cm²] in transverse cross section.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the 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:⁴

E381 Method of Macroetch Testing Steel Bars, Billets, Blooms, and Forgings

2.2 ASTM Adjuncts:

Adjunct to A604/A604MPractice for Macroetch Testing of Consumable Electrode Remelted Steel Bars and Billets³

3. Description of Macroetch Testing

3.1 This practice employs the action of an acid or other corrosive agent to develop the characteristics of a suitably prepared specimen. After etching, the sections are compared visually, or at a very low magnification, if necessary for clarification of conditions, to standard plates describing the various conditions which may be found. Materials react differently to etching reagents because of variations in chemical composition, method of manufacture, heat treatment, and many other variables.

4. Significance and Use

4.1 Macroetch testing, as described herein, is a method for examining and rating transverse sections of bars and billets to describe certain conditions of macro segregation which are often characteristic of consumable electrode remelted materials.

4.2 This practice is not intended to define major defects such as those described by Method E381.

5. Application

5.1 When material is furnished subject to macroetch testing and inspection under this practice, the manufacturer and purchaser should be in agreement concerning the following:

5.1.1 The stage of manufacture at which the test shall be conducted,

5.1.2 The number and location of the sections to be tested,

¹ This practice is under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel and Related Alloys and is the direct responsibility of Subcommittee A01.06 on Steel Forgings and Billets.

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² ASTM Committee A01 gratefully acknowledges the help of the AISI Committee on General Metallurgy in preparing the appendix, assembling the macroetch photographs, and assisting with the text of this practice.

³ A complete set of the 20 macrographs on glossy paper available from ASTM International Headquarters. Order Adjunct No. ADJA0604. Original adjunct produced in 1985.

⁴ 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.

5.1.3 The condition and preparation of the surface to be macroetched,

5.1.4 The etching reagent, temperature and time of etching, or degree of etching including any special techniques which must be used, and

5.1.5 The type and degree of conditions or combinations thereof that shall be considered acceptable or subject to metallurgical review.

6. Sample Preparation

6.1 Unless otherwise specified, the test shall be performed on specimens, usually $\frac{1}{4}$ to 1 in. [6 to 25 mm] thick, cut to reveal a transverse surface.

6.2 Disks for macroetch inspection may be removed from billets by a variety of methods including torch cutting, sawing, machining, or high-speed abrasive wheels. Adequate preparation of the surface for macroetching must completely remove the effects of torch cutting or high-speed abrasive wheels.

6.3 Due to the nature of the conditions to be detected, further surface preparation is usually required.

6.4 When such further preparation is performed, grinding, machining, or sanding should be carried out in such a manner as not to mask the structure.

6.5 The surface of the disk to be etched must be free of dirt, grease, or other foreign material which might impair the result of the test.

7. Etching Reagents

7.1 The etching response and appearance is dependent upon the type and temperature of the etching reagent and the time of immersion. These details must be established by agreement between manufacturer and purchaser.

7.2 For illustrative purposes some of the commonly used etching reagents are as follows:

7.2.1 *Hydrochloric Acid*—A solution of 1 part commercial concentrated hydrochloric acid (HCl, sp gr 1.19) and 1 part water is more generally used than any other macroetching reagent. This solution may be heated without significant change in concentration, and may be reused if it has not become excessively contaminated or weakened. Etching is generally done with the solution at a temperature of approximately 160°F [70°C].

7.2.2 Hydrochloric Acid-Sulfuric Acid Mixture—A mixture containing 50 % water, 38 % commercial concentrated HCl, and 12 % commercial concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) is sometimes used in place of the previously mentioned 50 % HCl solution. The statements in the previous paragraph regarding reuse and temperature of etchant are applicable to this reagent.

7.2.3 Aqua Regia—A solution consisting of 1 part concentrated nitric acid (HNO₃, sp gr 1.42) and 2 parts concentrated HCl is used on corrosion and heat-resistant materials of the 18 % chromium, 8 % nickel type and higher alloy types. This reagent is used at room temperature.

NOTE 1—The reagents in 7.2.1, 7.2.2, and 7.2.3 should be used under ventilating hoods or with some provision to remove the corrosive fumes.

7.2.4 *Nitric Acid*—This etchant consists of 5 % HNO₃ solution in alcohol or water, and is generally used at room temperature. When this reagent is used, the etch disk must have a smooth surface.

8. Etching Containers

8.1 Macroetching must be done in containers that are resistant to attack from the etching reagents. Caution must be exerted to prevent the occurrence of electrolytic couples which can cause uneven attacks and misleading results.

9. Preparation of Etched Surface and Examination

9.1 Upon completion of etching, surfaces of disks should be cleaned by either chemical or mechanical methods that do not affect the macroetch quality. Care should be taken to prevent rusting of the etched surface.

10. Interpretation of Conditions Found by Macroetching

10.1 Four distinct classes of conditions are defined and described under this practice:

10.1.1 *Class 1: Freckles*—Circular or near-circular dark etching areas generally enriched with carbides and carbide-forming elements.

10.1.2 *Class 2: White Spots*—Light etching areas, having no definitive configuration or orientation which are generally reduced in carbide or carbide-forming elements.

10.1.3 *Class 3: Radial Segregation*—Radially or spirally oriented dark etching elongated areas occurring most frequently at mid-radius which are generally carbide enriched. This condition may be easily confused with freckles in some materials.

10.1.4 *Class 4: Ring Pattern*—One or more concentric rings evidenced by a differential in etch texture associated with minor composition gradients and ingot solidification.

10.2 Macroetch photographs show examples of each of the conditions revealed by macroetch testing, with five degrees of severity, identified as A, B, C, D, and E for each condition. Degree A exhibits the minimum occurrence of each condition detectable by visual examination of the etched surface, while degrees B, C, D, and E represent increasing severity of occurrence.

10.3 For each condition, or combination of conditions, ratings shall be obtained by comparing each macroetched section with the standard photographs. Bar or billet sections to 225 in.^2 [1450 cm²] cross-sectional area may be rated against these standards. Larger sizes may be rated by agreement between manufacturer and purchaser, but caution must be exercised in interpretation of such results. Figs. 1-20 have been reduced 44 % in area from the standard photographs.

10.4 If the appearance of a given condition does not exactly match one of the five standard photographs, it shall be assigned the rating of the standard that it most nearly matches.

10.5 No standards for acceptance are stated or implied in these illustrations. The extent to which each condition may be permissible varies with the intended application, and such standards should be stated in the applicable product specification, or may be the subject of negotiation between manufacturer and purchaser.