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Standard Practice for Macroetch Testing of Tool Steel Bars[1]

This standard is issued under the fixed designation A561; 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 U.S. Department of Defense.

1. Scope

1.1 This practice for macroetch testing has been found to be a useful and reliable method for evaluating the quality of tool steel bars. It is used as a quality control and inspection test to reveal by deep acid etching the macrostructure in specimens cut from bars and to show the presence of such conditions as pipe, cracks, porosity, segregation, or foreign material. The etched surface is generally examined visually, but magnification up to about 10× is occasionally employed.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

2. Referenced Documents

2.1 *ASTM Adjuncts:*[2]
Six Photomacrographs

3. Apparatus

3.1 *Etching Containers*—Macroetching may be performed in a vessel of borosilicate glass, porcelain, corrosion-resistant metals, or some other acid-resisting material.

4. Reagent

4.1 *Etching Reagent*—A solution of equal volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) and water is commonly used for macroetching tool steels. This solution must be used under a ventilating hood because HCl is volatile and the fumes are corrosive and irritating, although nontoxic. The solution may be reused within limits. With use, the concentration of dissolved iron and other metals increases and the acidity of the solution decreases retarding the etching action. Spent solution shall be replaced with fresh solution, not replenished with concentrated acid.

5. Sampling

5.1 The selection of specimens for macroetch testing must be done with care for interpretations to be of value.

5.1.1 Specimens are usually cut from hot-rolled annealed bars, but may be cut from machined or ground bars if the bars are to be finish machined or ground.

5.1.2 The specimen should be located at a sufficient distance from the end of the bar to avoid end effects.

5.1.3 For ease in handling, use specimens ¼ to ½ in. (6.35 to 12.7 mm) thick.

5.1.4 Cut specimens to expose a transverse section of the bar; however, the test is occasionally performed on a longitudinal section.

5.1.5 Specimens may be taken from one or both ends of a bar. Each bar may be sampled, or a few typical specimens may be tested as representative of a large number of bars.

6. Specimen Preparation

6.1 In all cutting and grinding operations on the specimen, care must be exercised to avoid heating the surface to an excessively high temperature. Specimens are cut from bars by sawing, machining, abrasive wheel cutting, or other means. Cutting should be controlled to prevent smearing the cut face and masking the structure.

6.1.1 The "as-cut" surface of a specimen may be sufficiently smooth to reveal the defects for which the examination is conducted. No additional surface preparation may then be necessary.

6.1.2 Additional surface preparation may be required to remove cutting marks and to allow details to be revealed by etching. In such circumstances, machining, grinding, or polishing may be necessary. Generally, the degree of surface smoothness required is greater the finer the detail that must be resolved. When the action of the etchant is drastic, a coarser surface finish may be used.

6.1.3 The surface must be free of adhering grease and oil. There should be no scale or oxide on the surface which will be examined after etching.

7. Procedure

7.1 *Temperature for Macroetching*—Etching characteristics are influenced markedly by the temperature of the etchant.

[1] This practice is under the jurisdiction of ASTM Committee A01 on Steel, Stainless Steel and Related Alloysand is the direct responsibility of Subcommittee A01.29 on Tool Steels.

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[2] Available from ASTM International Headquarters. Order Adjunct No. ADJA0561. Original adjunct produced in 1984.

Thus, the reagent temperature should be controlled for macroetching if comparative results are desired. Tool steels are generally macroetched at about 160°F (71°C). At this temperature, the etching reaction is vigorous and solution losses through evaporation are not excessive. The solution may be heated on a gas or electric hot plate, by an acid-proof immersion heater, or by steam.

7.2 *Etching Time*—The etching time should be sufficiently long to completely reveal the structure in the specimen, yet should not be so long as to develop artifacts such as etch pits or to obscure or obliterate the structure. Generally, etching times under 10 min or over 40 min are avoided with tool steels. The time of etching is best adjusted by trial to develop the degree of etching desired. Where recovery of temperature of the solution is slow after immersion of cold specimens, reproducibility is enhanced by preheating the specimens in hot water prior to immersing in the etchant.

7.3 *Macroetching Technique:*

7.3.1 After preparation as described above, clean the specimen of dirt and grease with a solvent, if necessary. Then place directly into the etching solution already heated to the proper temperature, or preheat in water as in 7.2 and then transfer to the hot etching bath. The purpose of preheating the specimen is to obtain better control of the etching conditions where close reproducibility of degree of development is desired. The specimen must be completely immersed.

7.3.2 Moderate agitation (stirring) of the solution during macroetching promotes uniform development by eliminating temperature and composition gradients. Provision for agitating the etchant may be desirable where the volume of solution is relatively small.

7.3.3 At the end of the etching period, remove the specimen from the hot acid and rinse immediately in a stream of water. Flush off the sludge which forms on the surfaces of the specimen during etching with the aid of a stiff brush. Then dry the specimen with alcohol, a clean air blast, or live steam.

7.3.4 After drying, protect the etched surface from rusting by an application of oil, grease, glycerine, or transparent lacquer.

8. Interpretation of Results

8.1 Examine the etched surface visually or at up to 10× magnification to determine its structure. Note the presence, absence, and severity of the following conditions:

8.1.1 *Internal:*

8.1.1.1 Pipe,

8.1.1.2 Bursts,

8.1.1.3 Carbide (see Note 1) or alloy segregation,

8.1.1.4 Concentrations of nonmetallic inclusions,

8.1.1.5 Porosity, and

8.1.1.6 Internal cracks or thermal flakes.

NOTE 1—Carbide segregation is better determined by examination of a polished surface after etching in 4 % nital solution.

8.1.2 *Surface and Subsurface:*

8.1.2.1 Seams, laps, cracks, etc.,

8.1.2.2 Ingot corner segregation or cracks, and

8.1.2.3 Pinholes.

8.1.3 *Miscellaneous:*

8.1.3.1 Entrapped metallic or nonmetallic material, and

8.1.3.2 Ingot pattern (dendritic segregation, columnar grain structure, etc.).

8.2 Six Photomacrographs provide a numerical rating of severity levels of porosity (8.1.1.5) and ingot pattern (8.1.3.2) that may be present in tool steel bars.

8.2.1 These conditions identified in general as porosity and ingot pattern are described as follows:

8.2.1.1 *Center Porosity*—A center concentration of minute voids related to etched out carbides, or nonmetallic inclusions, etc.

8.2.1.2 *Ring Ingot Pattern*—One or more concentric rings characterized by differential etching associated with a minor gradient in chemical composition or ingot solidification.

8.2.2 Ratings for the conditions can be determined by comparing macroetched disks with these photographs.

8.2.3 The photographs are not intended as standards for acceptance or rejection. The numerical identity listed with each figure only refers to a degree of severity. The extent that each condition is permissible for a given application should be stated by the tool steel specification covering the application or as negotiated between the supplier and purchaser.

9. Keywords

9.1 ingot structure; macroetch; macrostructure; porosity; segregation