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Standard Practice for Macroetching Metals and Alloys[1]

This standard is issued under the fixed designation E340; 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 These procedures describe the methods of macroetching metals and alloys to reveal their macrostructure.

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.

1.3 *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.* For specific warning statements, see 6.2, 7.1, 8.1.3, 8.2.1, 8.8.3, 8.10.1.1, and 8.13.2.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens

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

3. Significance and Use

3.1 *Applications of Macroetching:*

3.1.1 Macroetching is used to reveal the heterogeneity of metals and alloys. Metallographic specimens and chemical analyses will provide the necessary detailed information about specific localities but they cannot give data about variation from one place to another unless an inordinate number of specimens are taken.

3.1.2 Macroetching, on the other hand, will provide information on variations in (*1*) structure, such as grain size, flow lines, columnar structure, dendrites, and so forth; (*2*) variations in chemical composition as evidenced by segregation, carbide and ferrite banding, coring, inclusions, and depth of carburization or decarburization. The information provided about variations in chemical composition is strictly qualitative but the location of extremes in segregation will be shown. Chemical analyses or other means of determining the chemical composition would have to be performed to determine the extent of variation. Macroetching will also show the presence of discontinuities and voids, such as seams, laps, porosity, flakes, bursts, extrusion rupture, cracks, and so forth.

3.1.3 Other applications of macroetching in the fabrication of metals are the study of weld structure, definition of weld penetration, dilution of filler metal by base metals, entrapment of flux, porosity, and cracks in weld and heat affected zones, and so forth. It is also used in the heat-treating shop to determine location of hard or soft spots, tong marks, quenching cracks, case depth in shallow-hardening steels, case depth in carburization of dies, effectiveness of stop-off coatings in carburization, and so forth. In the machine shop, it can be used for the determination of grinding cracks in tools and dies.

3.1.4 Macroetching is used extensively for quality control in the steel industry, to determine the *tone* of a heat in billets with respect to inclusions, segregation, and structure. Forge shops, in addition, use macroetching to reveal flow lines in setting up the best forging practice, die design, and metal flow. For an example of the use of macroetching in the steel forging industry see Method E381. Forging shops and foundries also use macroetching to determine the presence of internal faults and surface defects. The copper industry uses macroetching for control of surface porosity in wire bar. In the aluminum industry, macroetching is used to evaluate extrusions as well as the other products such as forgings, sheets, and so forth. Defects such as coring, cracks, and porthole die welds are identified.

4. Sampling

4.1 As in any method of examination, sampling is very important. When macroetching is used to solve a problem, the problem itself largely dictates the source of the sample as to the location on the work piece and the stage of manufacture; for example, when looking for pipe, the sample should represent the top of the ingot, or when looking for bursts or flakes, the sample should be taken as soon after hot working as possible.

[1] This test 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.

4.2 When macroetching is used as an inspection procedure, sampling ought to be done in an early stage of manufacturing so that if the material proves faulty, no wasteful unnecessary work is done. However, the sample should not be taken so early that further working can introduce serious defects. In the steel industry, for example, the sample is usually taken after ingot breakdown and after most chances of bursts or flakes occurring have passed. Billets or blooms going into small sizes are sampled after initial breakdown. Material going into forging billets or die blocks is sampled near finish size. Sampling may be done systematically or on a random basis.

4.3 Samples may be cold cut from the source by any convenient fashion; saws and abrasive cutoff wheels are particularly effective. The use of torch cutting or hot cutting should be used only when necessary to cut a sample from a large piece. The sample then is sectioned well away from the hot-cut surface. An example of permissible use of torch cutting is the excising of a piece from a large plate and then cutting a sample for macroetching 4 to 5 in. (102 to 127 mm) away from the torch-cut edge.

4.4 Some common methods of sampling, listed by source, are as follows:

4.5 *Billets, Blooms, and Hot-Rolled Products*—Disks are usually cut from these products near the end. Samples cut too close to the end, however, may have false structures because of fish-tailing. Disks from large blooms are sometimes cut into smaller pieces for ease in handling.

4.5.1 *Forgings and Extrusions*—Disks cut transverse to the long dimension will show flakes, bursts, and so forth. Forgings may also be cut parallel to the long dimension to show flow lines. In complicated forgings, some thought will have to be given to the proper method of cutting so as to show flow lines. Macroetching of an unprepared specimen will show surface defects such as shuts, flats, seams, and so forth. In extrusions, coring and coarse grain are more commonly found in the back end of the extrusion.

4.5.2 *Sheets and Plates*—A sufficiently large sample should be taken when looking for surface defects. An ideal length would be the circumference of the last roll, but this may be inconveniently long. Several samples totaling some given fraction of the circumference can be used; however, there is always a chance then that a defect arising from faulty rolls would not be detected. When seeking information on laminations, a transverse section is used. In many cases, however, to reduce the size of the specimen, only a section out of the center of the plate may be taken.

4.5.3 *Weldments*—A disk cut perpendicular to the direction of welding will show weld penetration, heat affected zone, structure, and so forth. Careful preparation is usually rewarded with highly detailed structures giving a large amount of information. Welds involving dissimilar metals will produce problems in etching. The best method is to etch the least corrosion-resistant portion first and the more resistant portion afterwards. Occasionally an intermediary etchant may be required. The boundaries between etched and unetched portion will give an idea of weld penetration and dilution.

4.5.4 *Castings*—Cut the specimen to display the defect or feature being sought.

4.5.5 *Machined and Ground Parts*—When looking for grinding cracks, and so forth, the surface itself is used as a sample. Because the machined or ground part is often the finished part, it may be undesirable to immerse the part in acid. In this case, other methods such as dye penetrant methods may be more desirable.

5. Preparation

5.1 Sample preparation need not be elaborate. Any method of presenting a smooth surface with a minimum amount of cold work will be satisfactory. Disks may be faced on a lathe or a shaper. The usual procedure is to take a roughing cut, then a finish cut. This will generate a smooth surface and remove cold work from prior operations. Sharp tools are necessary to produce a good specimen. Grinding is usually conducted in the same manner, using free-cutting wheels and light finishing cuts. When fine detail is required, the specimen should be ground down through the series of metallographic papers (see Guide E3). Where necessary, details are given in the tabulation of procedures.

5.2 After surface preparation, the sample is cleaned carefully with suitable solvents. Any grease, oil, or other residue will produce uneven attack. Once cleaned, care should be taken not to touch the sample surface or contaminate it in any way.

6. Solutions

6.1 The solutions used for macroetching are given in the tables listed under each alloy. In most cases a good grade of reagent should be used but need not be chemically pure or of analytical quality. The so-called technical grades are usually satisfactory. The solution should be clean and clear, free of suspended particles, scum, and so forth.

6.2 Caution must be observed in mixing. Many of the etchants are strong acids. In all cases, the various chemicals should be added slowly to the water or solvent while stirring. In the cases where hydrofluoric acid is used, the solution should be mixed and used in polyethylene vessels. (**Warning—**Hydrofluoric acid must not be allowed to contact the skin since it can cause painful serious ulcers if not washed off immediately.)

7. Procedure

7.1 Many of the solutions are aggressive and may give off irritating and corrosive fumes. Etching should be done in a well-ventilated room, preferably under a fume hood. The solution should be mixed and placed in a corrosion resistant tray or dish and brought to the operating temperature. The specimen or specimens should be placed in a tray of stainless steel screen or on some non-reactive support. Glass rods often are placed on the bottom of the acid container and the specimens laid directly on the rods. When etching is completed, remove the specimens from the dish taking great care not to touch the etched surface. When desmutting is required, dip the specimen into a second solution. After rinsing the specimen with hot water, blow dry with clean compressed air.

7.2 In the case of large specimens, such as ingot sections, swabbing may be the only practical method of macroetching.

TABLE 1 Macroetchants for Aluminum and Aluminum Alloys

Alloy	Composition		Procedure	Comments
All	NaOH H_2O	10 g 100 mL	Immerse sample 5 to 15 min in solution heated to 140 to 160°F (60 to 70°C). Rinse in water, and remove smut in strong HNO_3 solution. Rinse and repeat etching if necessary.	Good general-purpose etchant, can be used on almost all aluminum alloys. Does not require fine grinding.
3XXX 4XXX 5XXX 6XXX High Si castings	HCl (concentrated) HNO_3 (concentrated) HF (48 %)	75 mL 25 mL 5 mL	Mix fresh before using. Use at room temperature. May be used as immersion etch or swabbed over specimen surface. Rinse specimen in warm water and dry.	Used to develop grain structure. May be diluted with 25 % water to slow down etching. Does not require fine grinding.
High purity A1 1XXX 3XXX 4XXX 5XXX 6XXX	HCl (concentrated) HNO_3 (concentrated) HF (48 %) H_2O	45 mL 15 mL 15 mL 25 mL	Immerse specimen at room temperature until desired contrast is developed. Rinse in warm water and dry.	Tucker's etch. General purpose etch for revealing microstructure of both cast and wrought aluminum. Does not require fine grinding.
All except high Si castings	HCl (concentrated) HNO_3 (concentrated) HF (48 %) H_2O	15 mL 5 mL 5 mL 75 mL	Same as above.	1 + 2 Tucker's. Same as above, but slower acting.
2XXX High Cu alloys	HCl (concentrated) HF (48 %) H_2O	15 mL 10 mL 90 mL	May be used as an immersion etch or swabbed over the specimen surface. When desired contrast is obtained, rinse in water and remove deposits with concentrated HNO_3. Rinse in warm water and dry.	Flick's reagent. Best results are obtained with a ground surface. 180 grit will suffice.

Saturate a large wad of cotton held in stainless steel or nickel tongs with the etchant and sweep over the surface of the specimen. An effort should be made to wet the entire surface as soon as possible. After the initial wetting, keep the swab saturated with solution and frequently sweep over the surface of the specimen to renew the solution. When the structure has been suitably developed, rinse the specimen, either with a swab saturated with water, or better still, by pouring water over the specimen. After rinsing with hot water, blow the specimen dry with compressed air. Details of the procedure not discussed here are covered in the sections for the various metals and their alloys.

7.3 The times and temperatures given in individual tabulations are only intended as guides. In fact, the progress of etching should be closely watched and etching stopped when the preferred structural details have been revealed. Specimens should be etched to develop structure. Generally, a light etch is better than a heavy etch; overetching can often lead to misinterpretation. The actual time to develop a structure properly may be quite different from the one suggested.

8. Specific Preparation Procedures and Recommended Solutions

8.1 *Aluminum:*

8.1.1 The specimens can be cut using common cutting tools, hack saws, band saws, shears, abrasive cutoff wheels, and so forth. All these methods will cause cold work at the surface and will generate heat. The temperature rise can be enough to cause changes in structure. For these reasons sharp tools and generous lubrication are necessary for sectioning.

8.1.2 The cold-worked surface should be removed by machining the surface. Again sharp tools and copious lubrication are required. If fine detail is required, the machined surface should be ground using silicon carbide paper lubricated with water or kerosine.

8.1.3 Several of the solutions used in macroetching react vigorously with the metal and can overheat the specimen. In these cases the specimen is periodically removed from the solution, cooled in running water, and reimmersed in the etchant. This procedure is repeated until the desired degree of etching is obtained.

8.1.4 *Macroetchants for Aluminum and Aluminum Alloys* (Table 1).

8.2 *Beryllium:*

8.2.1 While beryllium in the massive form is not dangerous, beryllium and its compounds in the finely divided state are extremely poisonous. (**Warning—**Before starting any work involving beryllium, a review of hazards and plans for handling should be made. A number of references on beryllium are available. Particular mention may be made of "Toxicity of Beryllium" ASD-TR-62-7-667, prepared by the Kettering Laboratory for the Air Force.)

8.2.1.1 Generally speaking, beryllium and its alloys have given difficulty in obtaining good macroetched specimens. First, beryllium is a rather brittle metal and sectioning can be difficult. Cut-off wheels with the designation C46FR70 have been the most successful. Secondly, beryllium does not grind easily; hence, specimens should be as small as possible to minimize grinding time. Grinding has been most successful with the entire sequence of wet silicon carbide papers.

8.2.1.2 The etching of fine grained metal may not always be entirely successful, and further preparation will be required. Rough polishing with 15 μm Al_2O_3 suspended in water is performed on a low-nap cloth. Light pressure and frequent