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Standard Test Method for Macroetching Metals and Alloys¹

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 Department of Defense.

1. Scope

1.1 These test 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 the standard. ~~The SI equivalents of inch-pound units may be approximate 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, etc.; (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, etc.

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, etc. 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, etc. 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, etc. Defects such as coring, cracks, and porthole die welds are identified.

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. Current edition approved Oct 1, 2006 June 1, 2013. Published October 2006 October 2013. Originally approved in 1968. Last previous edition approved in 2000 2006 as E340 – 00¹-(2006). DOI: 10.1520/E0340-00R06-10.1520/E0340-13.

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. 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.

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, etc. 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, etc. 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, etc. 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, etc., 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 Methods 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, etc.

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 should 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. 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 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, etc. 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 kerosene.

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 change of cutting direction produce the best results. If further polishing is required, 1- μm green Cr_2O_3 in tap water on synthetic suede works best.

8.2.2 Macroetchants for Beryllium and Beryllium Alloys—(Table 2).

8.3 Cobalt and Cobalt Alloys:

8.3.1 Many of the cobalt-base high-temperature alloys can be etched using the same procedures as those for iron- and nickel-base high-temperature alloys. Other cobalt alloys, such as the stellites used as machine tools, require special treatment.

8.3.1.1 The cobalt-base alloys, as a group, are not easily machined. The specimens should be sectioned with abrasive cutoff wheels and ground on wet silicon carbide papers. Because of the rapid work-hardening characteristics of these alloys, fresh paper and copious cooling should be used.

8.3.2 Macroetchants for Cobalt and Cobalt Alloys (Table 3).

8.4 Copper and Copper Alloys:

8.4.1 These metals are usually macroetched to bring out the general structure of wire bar and billets as well as variations in grain size in extrusions and forgings.

8.4.1.1 Specimens may be sectioned using common cutting tools. To minimize cold working the tools should be kept sharp.

TABLE 1 Macroetchants for Aluminum and Aluminum Alloys

Alloy	Composition		Procedure	Comments
All	NaOH H ₂ O	10 g 100 mL	Immerse sample 5 to 15 min in solution heated to 60 to 70°C (140 to 160°F). 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 Al 1XXX 3XXX 4XXX 5XXX 6XXX	HCl (concentrated) HNO ₃ (concentrated) HF (48%) H ₂ O	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 ₂ O	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 ₂ O	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.

TABLE 1 Macroetchants for Aluminum and Aluminum Alloys

Alloy	Composition		Procedure	Comments
All	NaOH H ₂ O	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 Al 1XXX 3XXX 4XXX 5XXX 6XXX	HCl (concentrated) HNO ₃ (concentrated) HF (48%) H ₂ O	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 ₂ O	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 ₂ O	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.

8.4.1.2 Good results can be obtained by machining a smooth surface in two stages, the first being a heavy cut to remove the cold work from sectioning and the second a fine cut with a V-shaped tool to remove the remaining cold work. Grinding through the series of metallographic papers will give more detailed results. The degree of grinding depends upon the amount of detail required. The etching solutions listed in Table 4 are simple to prepare and their use requires no special technique.

NOTE 1—It should be pointed out that heavy etching often will remove the effect of cold work but at the expense of producing a rough surface. If the specimen is then given light regrinding to remove the rough etched surface, the second etch will provide good results.

8.4.2 Macroetchants for Copper and Copper Alloys—(Table 4).

8.5 Iron and Steel:

8.5.1 Macroetching has been most highly developed and is used extensively in the iron and steel industries. In hot-mill products such as bars, billets, sheet, and plate, the disk cut with a parting tool is prepared by facing on a lathe or by grinding. In facing, the first cut is moderately heavy with a sharp tool. The second facing is a light cut with a V-shaped tool run at high speed.

TABLE 2 Macroetchants for Beryllium and Beryllium Alloys

Metal	Composition	Procedure	Comments	
Be	HCl NH ₄ Cl H ₂ O	10 mL 4 g 90 mL	Either swab or immerse at room temperature for a few minutes, rinse in water and dry.	Works best on coarse grained Be.
Be	HCl NH ₄ Cl Picric acid H ₂ O	10 mL 2 g 2 g 90 mL	As above.	An alternative when No. 1 does not work. Fine-grained metal may not give good results in either case.

TABLE 2 Macroetchants for Beryllium and Beryllium Alloys

Metal	Composition	Procedure	Comments	
Be	HCl NH ₄ Cl H ₂ O	10 mL 4 g 90 mL	Either swab or immerse at room temperature for a few minutes, rinse in water and dry.	Works best on coarse grained Be.
Be	HCl NH ₄ Cl Picric acid H ₂ O	10 mL 2 g 2 g 90 mL	As above.	An alternative when No. 1 does not work. Fine-grained metal may not give good results in either case.

Specimens produced in this manner are adequate for general inspection. A better though slower method is to grind the specimen. For inspection purposes, finishing on a 120-grit wheel will be sufficient.

8.5.1.1 When the maximum amount of detail is required, as in weldments, polishing the specimen with the series of metallographic papers gives the best results. When examining for surface defects, the surface itself should be etched directly without much preparation. The only preparation that is advisable is to brush off the loose scale and then to give the specimen a light grinding pass with very coarse abrasive to break through the adherent scale. When etching in 1 + 1 HCl for example, this scale will be removed, exposing the surface underneath. If care has been exercised in the grinding operations, the grinding scratches will not interfere with examination.

8.5.1.2 The most commonly used solutions for macroetching iron and steel are Solution Nos. 1 and 3 in [Table 5](#).

8.5.1.3 Electrolytic etching using a solution of 3 to 6 % concentrated HCl in water at room temperature may be used. The specimen is immersed in the solution, placed in a carriage, and passed over a cathodic bar at a current of 40 A/in. of specimen width. After etching, the specimen is cleaned by using a vegetable fiber brush and a 10 % sodium citrate solution. The specimen finally is air dried.

8.5.2 *Forgings*—In addition to the examination for internal structure, surface defects, and structure, closed-die forgings are often sectioned to show flow lines. Etching for flow lines requires extremely careful preparation to provide a smooth surface with a minimum of cold work. Long pieces such as crank shafts are awkward to handle and are best prepared on a grinding machine using successively finer grinding wheels. Sectioning into shorter lengths may be advisable. The specimen should be heavily etched in 1 + 1 HCl or 20 % H₂SO₄. Contrast can often be increased by wiping the surface lightly with very fine metallographic paper after etching. Examination for structure, defects, etc. is carried out in the same fashion as hotmill products.

8.5.3 *Special Tests for Segregation*—There are a number of etchants containing copper salts which will reveal segregation. Careful specimen preparation through the metallographic papers is required. Very careful cleaning after grinding is extremely important. When a specimen is immersed in this type of solution, copper plates out onto the specimen by a replacement reaction. The rate of deposition depends on the composition of the steel and the copper plating will cover the segregated regions. Sometimes the specimen can be left in a little longer than recommended and then rubbed lightly with metallographic papers to increase contrast.

8.5.4 *Macroetchants for Iron and Steel* ([Table 5](#)).

8.6 *Stainless Steels and High-Temperature Alloys:*

8.6.1 These alloys are generally more susceptible to cold working of the surface than are the lower alloy grades of steel. The best method of preparation is to grind the specimens as described for iron and steel. A smut tends to form on the surface of the steel when immersed in 1 + 1 HCl. This can be prevented by adding a small quantity of HNO₃ to the etching bath. It can also be removed by scrubbing the specimen with a vegetable fiber brush under running warm water or by immersion in warm 20 % HNO₃. Scrubbing will provide a higher contrast for detection of segregation and inclusions. The desmutting either by the addition of HNO₃ to the etching bath or by the secondary rinse in HNO₃ will provide a brighter surface which is suitable for determination of grain size and structure. High-alloy stainless steels and austenitic high-temperature alloys because of their extreme corrosion resistance often will give trouble in etching. Aqua regia, HCl-H₂O₂, and Marble's reagent are the recommended etchants. All three of these require very careful specimen preparation.

8.6.2 *Macroetchants for Stainless Steels and High-Temperature Alloys* ([Table 6](#)).

8.7 *Lead and Lead Alloys:*

TABLE 4 Macroetchants for Copper and Copper Alloys

Alloys	Composition		Procedure	Comments
Cu and all brasses	HNO ₃ H ₂ O	10 mL 90 mL	Immerse specimen in solution at room temperature for a few minutes. Rinse in water and dry.	Emphasize grains and cracks.
Cu and all brasses	HNO ₃ H ₂ O	10 mL 90 mL	Immerse specimen in solution at room temperature for a few minutes. Rinse in water and dry.	Emphasize grains and cracks.
Cu and all brasses	HNO ₃ H ₂ O	50 mL 50 mL	As above.	Brings out grain contrast, pits result unless agitated. Aluminum bronzes may form smut which can be removed by brief immersion in concentrated HNO ₃ .
Cu and all brasses	HNO ₃ H ₂ O	50 mL 50 mL	As above.	Brings out grain contrast, pits result unless agitated. Aluminum bronzes may form smut which can be removed by brief immersion in concentrated HNO ₃ .
Cu and all brasses	HCl FeCl ₃ H ₂ O or ethanol	30 mL 10 g 120 mL	As above.	Good grain contrast.
Cu and all brasses	HCl FeCl ₃ H ₂ O or ethanol	30 mL 10 g 120 mL	As above.	Good grain contrast.
Cu, high-Cu alloys, —phosphorus, tin —bronzes	K ₂ Cr ₂ O ₇ sat —soln of NaCl H ₂ SO ₃ H ₂ O	2 g	Immerse specimen in solution at room temperature for 15 to 30 min then swab with fresh solution. Rinse in warm water and dry.	Emphasizes grain boundaries and oxide inclusions.
Cu, high Cu alloys, —phosphorus, tin —bronzes	K ₂ Cr ₂ O ₇ sat —soln of NaCl H ₂ SO ₃ H ₂ O	2 g	Immerse specimen in solution at room temperature for 15 to 30 min then swab with fresh solution. Rinse in warm water and dry.	Emphasizes grain boundaries and oxide inclusions.
All	HNO ₃ AgNO ₃ H ₂ O	50 mL 5 g 50 mL	Immerse specimen in solution at room temperature. Rinse in warm water and dry.	Brilliant deep etch.
All	HNO ₃ AgNO ₃ H ₂ O	50 mL 5 g 50 mL	Immerse specimen in solution at room temperature. Rinse in warm water and dry.	Brilliant deep etch.
Brass	20 % acetic acid 5 % chromic acid 10 % FeCl ₃ in H ₂ O	20 mL 10 mL 5 mL	As above.	Strain lines.
Brass	20 % acetic acid 5 % chromic acid 10 % FeCl ₃ in H ₂ O	20 mL 10 mL 5 mL	As above.	Strain lines.
Silicon brass or bronze	CrO ₃ NH ₄ Cl HNO ₃ (concentrated) H ₂ SO ₄ (concentrated) H ₂ O	40 g 7.5 g 50 mL 8 mL 100 mL	Immerse specimen in solution at room temperature, rinse in warm water and dry.	
Silicon brass or bronze	CrO ₃ NH ₄ Cl HNO ₃ (concentrated) H ₂ SO ₄ (concentrated) H ₂ O	40 g 7.5 g 50 mL 8 mL 100 mL	Immerse specimen in solution at room temperature, rinse in warm water and dry.	

8.7.1 Lead and its alloys are among the most difficult metals to prepare for macroetching. They are not only very soft and cold work easily, but they (pure lead especially) recrystallize readily at temperatures which can be easily achieved in careless preparation.

8.7.2 For best results in the macroetching of lead, all surfaces other than that to be examined must be masked from the macroetch by the use of several coats of a plastic spray. The surface to be examined should be filed prior to etching. Three 14-in. (360-mm) files, (see **Note 2**), are usually required and used in the following order: (1) aluminum, Type A, (2) hand smooth, and (3) hand-finishing smooth.

NOTE 2—Nicholson designations. Be sure to preserve the distinction between hand smooth and hand-finishing smooth.