



Designation: **E340—15 E340 – 23**

## Standard Practice for Macroetching Metals and Alloys<sup>1</sup>

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 ( $\epsilon$ ) 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—the International System (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, safety, health, and environmental 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.* 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. It is further recommended to review the guidance in Guide E2014.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

- 2.1 *ASTM Standards:*<sup>2</sup>
- E3 Guide for Preparation of Metallographic Specimens
  - E381 Method of Macroetch Testing Steel Bars, Billets, Blooms, and Forgings
  - E2014 Guide on Metallographic Laboratory Safety

### 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

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<sup>2</sup> 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.

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~~, carburization, 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 and 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.

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 the desired 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~~method; saws and abrasive cutoff wheels are particularly effective. ~~The use of torch~~ 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 44 in. to 5 in. (102(102 mm 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,

**TABLE 1 Macroetchants for Aluminum and Aluminum Alloys**

Alloy	Composition		Procedure	Comments
All	NaOH H <sub>2</sub> 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 <sub>3</sub> solution. Rinse and repeat etching if necessary.	Good general-purpose etchant, can be used on almost all aluminum alloys. Does not require fine grinding.
All	NaOH H <sub>2</sub> O	10 g 100 mL	Immerse sample 5 min to 15 min in solution heated to 140 °F to 160 °F (60°C to 70 °C). Rinse in water, and remove smut in strong HNO <sub>3</sub> 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 <sub>3</sub> (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 <sub>3</sub> (concentrated) HF (48 %) H <sub>2</sub> 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 <sub>3</sub> (concentrated) HF (48 %) H <sub>2</sub> 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 <sub>2</sub> 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 <sub>3</sub> . Rinse in warm water and dry.	Flick's reagent. Best results are obtained with a ground surface. 180 grit will suffice.

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 [Tables 1-14](#) the tabulation of procedures.

5.2 Care should be taken to examine the surface before and after etching to ensure induced damage from surface preparation does not interfere with analysis. Specific guidance is included in the following sections.

5.3 After surface preparation, the sample is cleaned carefully with suitable solvents. Any grease, oil, or other residue will produce uneven attack, an uneven etch response. 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 laboratory grade of

**TABLE 2 Macroetchants for Beryllium and Beryllium Alloys**

Metal	Composition	Procedure	Comments
Be	HCl	10 mL	Either swab or immerse at room temperature for a few minutes, rinse in water and dry.
	NH <sub>4</sub> Cl	4 g	
	H <sub>2</sub> O	90 mL	
Be	HCl	10 mL	As above.
	NH <sub>4</sub> Cl	2 g	
	Picric acid	2 g	
	H <sub>2</sub> O	90 mL	

**TABLE 3 Macroetchants for Cobalt and Cobalt Alloys**

Alloy	Composition	Procedure	Comments
49Co-49Fe-V Some Co-Cr alloys	HCl	50 mL	Immerse specimen in hot solution (140 to 180°F) for 30 to 60 min. Rinse in hot water and dry.
	H <sub>2</sub> O	50 mL	
25Cr-10Ni-8W 21Cr-20Ni- 3W-3Mo-1Cb	HCl	50 mL	Swab until desired contrast is obtained then rinse in warm water and dry.
	HNO <sub>3</sub>	10 mL	
	FeCl <sub>3</sub>	10 g	
	H <sub>2</sub> O	100 mL	
18Cr-10Ni-14W	CuCl <sub>2</sub> · 2NH <sub>4</sub> Cl · 2H <sub>2</sub> O	2 g	As above.
	FeCl <sub>3</sub>	5 g	
	HNO <sub>3</sub>	5 mL	
	HCl	50 mL	
	H <sub>2</sub> O	80 mL	

**TABLE 4 Macroetchants for Copper and Copper Alloys**

Alloys	Composition	Procedure	Comments
Cu and all brasses	HNO <sub>3</sub>	10 mL	Immerse specimen in solution at room temperature for a few minutes. Rinse in water and dry.
	H <sub>2</sub> O	90 mL	
Cu and all brasses	HNO <sub>3</sub>	50 mL	As above.
	H <sub>2</sub> O	50 mL	
Cu and all brasses	HCl	30 mL	As above.
	FeCl <sub>3</sub>	10 g	
	H <sub>2</sub> O or ethanol	120 mL	
Cu, high Cu alloys, phosphorus, tin bronzes	K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> sat soln of NaCl	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.
	H <sub>2</sub> SO <sub>3</sub>		
	H <sub>2</sub> O		
All	HNO <sub>3</sub>	50 mL	Immerse specimen in solution at room temperature. Rinse in warm water and dry.
	AgNO <sub>3</sub>	5 g	
	H <sub>2</sub> O	50 mL	
Brass	20% acetic acid	20 mL	As above.
	5% chromic acid	10 mL	
	10% FeCl <sub>3</sub> in H <sub>2</sub> O	5 mL	
Brass	20% CH <sub>3</sub> COOH	20 mL	As above.
	5% H <sub>2</sub> CrO <sub>4</sub>	10 mL	
	10% FeCl <sub>3</sub> in H <sub>2</sub> O	5 mL	
Silicon brass or bronze	CrO <sub>3</sub>	40 g	Immerse specimen in solution at room temperature, rinse in warm water and dry.
	NH <sub>4</sub> Cl	7.5 g	
	HNO <sub>3</sub> (concentrated)	50 mL	
	H <sub>2</sub> SO <sub>4</sub> (concentrated)	8 mL	
	H <sub>2</sub> O	100 mL	

reagent should be used but need not be chemically pure or of analytical quality. The so-called technical grades are usually satisfactory. Technical grades may provide satisfactory results. The solution should be clean and clear, free of suspended particles, scum, and so forth. Solutions may lose effectiveness through age or reuse, and should be replaced if not performing consistently.

6.2 Caution must be observed in mixing. Many of the etchants are strong acids. In all cases, the various chemicals should be added

**TABLE 5 Macroetchants for Iron and Steel**

Alloys	Composition		Procedure	Comments
Plain and alloy steels, —high-speed and tool steels, —cutlery (12–14 % Cr) and —stainless steels	HCl (concentrated)	50 mL	Immerse specimen in solution heated to 160 to 180°F for 15 to 30 min. Desmut by vigorous scrubbing with vegetable fiber brush under running water. Stainless steels may be desmuted by dipping in a warm 20 % HNO <sub>3</sub> to give a bright finish.	General-purpose.
	H <sub>2</sub> O	50 mL		
Plain and alloy steels, high-speed and tool steels, cutlery (12 %–14 % Cr) and stainless steels	HCl (concentrated)	50 mL	Immerse specimen in solution heated to 160 °F to 180 °F for 15 min to 30 min. Desmut by vigorous scrubbing with vegetable fiber brush under running water. Stainless steels may be desmuted by dipping in a warm 20 % HNO <sub>3</sub> to give a bright finish.	General purpose.
	H <sub>2</sub> O	50 mL		
High-alloy steels	HCl (concentrated) HNO <sub>3</sub> (concentrated) H <sub>2</sub> O	50 mL 25 mL 25 mL	Immerse specimen for 10 to 15 min in solution at room temperature. Rinse in warm water and dry.	Ratio HCl:HNO <sub>3</sub> runs 2:1 to 3:1.
High-alloy steels	HCl (concentrated) HNO <sub>3</sub> (concentrated) H <sub>2</sub> O	50 mL 25 mL 25 mL	Immerse specimen for 10 min to 15 min in solution at room temperature. Rinse in warm water and dry.	Ratio HCl:HNO <sub>3</sub> runs 2:1 to 3:1.
Plain and alloy steels, cutlery —steels	HCl (concentrated) H <sub>2</sub> SO <sub>4</sub> (concentrated) H <sub>2</sub> O	38 mL 12 mL 50 mL	Immerse specimen for 15 to 45 min in solution heated to 160 to 180°F. Rinse in warm water and dry.	Works well on 12 % Cr steel.
Plain and alloy steels, cutlery —steels	HCl (concentrated) H <sub>2</sub> SO <sub>4</sub> (concentrated) H <sub>2</sub> O	38 mL 12 mL 50 mL	Immerse specimen for 15 min to 45 min in solution heated to 160 °F to 180 °F. Rinse in warm water and dry.	Works well on 12 % Cr steel.
High-alloy steels	HNO <sub>3</sub> (concentrated) HF (48 %) H <sub>2</sub> O	10 mL 4 mL 87 mL	Immerse specimen in solution heated to 160 to 180°F until desired etch is obtained and rinse in warm water and dry.	Ratio HNO <sub>3</sub> -HF varies.
High-alloy steels	HNO <sub>3</sub> (concentrated) HF (48 %) H <sub>2</sub> O to HNO <sub>3</sub> (concentrated) HF (48 %) H <sub>2</sub> O	10 mL 4 mL 87 mL  40 mL 10 mL 50 mL	Immerse specimen in solution heated to 160 °F to 180 °F until desired etch is obtained and rinse in warm water and dry.	Ratio HNO <sub>3</sub> -HF varies.
Stainless steels, high-alloy —steels	HCl (concentrated) H <sub>2</sub> O H <sub>2</sub> O <sub>2</sub> (30 %)	50 mL 50 mL 20 mL	Mix HCl and water then heat to 160 to 170°F. Immerse specimen and add H <sub>2</sub> O <sub>2</sub> in several parts. Do not mix. Make each subsequent addition after foaming from previous addition has stopped.	Produces bright finish.
Stainless steels, high-alloy —steels	HCl (concentrated) H <sub>2</sub> O H <sub>2</sub> O <sub>2</sub> (30 %)	50 mL 50 mL 20 mL	Mix HCl and water then heat to 160 °F to 170 °F. Immerse specimen and add H <sub>2</sub> O <sub>2</sub> in several parts. Do not mix. Make each subsequent addition after foaming from previous addition has stopped.	Produces bright finish.
Austenitic stainless steels	HCl (concentrated) sat —soln CuSO <sub>4</sub> in H <sub>2</sub> O	50 mL — 25 mL	Immerse specimen in solution which may be heated or not depending upon alloy. Time also depends on alloy. Rinse in warm water and dry.	Marble's reagent. Light etch, good for structure.
Austenitic stainless steels	HCl (concentrated) saturated solution of CuSO <sub>4</sub> in H <sub>2</sub> O	50 mL — 25 mL	Immerse specimen in solution which may be heated or not depending upon alloy. Time also depends on alloy. Rinse in warm water and dry.	Marble's reagent. Light etch, good for structure.
Plain and low-alloy steels	(NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub> (ammo- nium persulfate) H <sub>2</sub> O	10 g 100 mL	Swab solution at room temperature over specimen. Rinse and dry.	Grain size, weldments.
Plain and alloy steels	CuCl <sub>2</sub> MgCl <sub>2</sub> HCl (concentrated) Alcohol—up to	2.5 g 10 g 5 mL 250 mL	Immerse in solution at room temperature until a coppery sheen appears. Rinse thoroughly and dry.	Stead's reagent. Salts dissolved in HCl with minimum of hot water. To bring out P-rich areas and P banding.
Mild steel, Bessemer and high —N <sub>2</sub> steel	CuCl <sub>2</sub> HCl (concentrated) H <sub>2</sub> O	90 g 120 mL 100 mL	The surface should be rubbed with cloth soaked in etching solution. Wash in alcohol or rinse in HCl (1 + 1) after etching to prevent deposition of copper.	Fry's reagent. Before etching, sample should be heated to 200 to 250°C (302 to 482°F) for 5 to 30 min depending on condition of steel. To show strain lines due to cold work.

Alloys	Composition	Procedure	Comments	
Mild steel, Bessemer and high N <sub>2</sub> steel	CuCl <sub>2</sub> HCl (concentrated) H <sub>2</sub> O	90 g 120 mL 100 mL	The surface should be rubbed with cloth soaked in etching solution. Wash in alcohol or rinse in HCl (1 + 1) after etching to prevent deposition of copper.	Fry's reagent. Before etching, sample should be heated to 200 °C to 250 °C (302 °F to 482 °F) for 5 min to 30 min depending on condition of steel. To show strain lines due to cold work.
Plain and alloy steels	CuCl <sub>2</sub> HCl (concentrated) H <sub>2</sub> O	45 g 180 mL 100 mL	As above.	Modified Fry's reagent. Same as for reagent No. 9 but modified by Wazau, may give more contrast specimen can be washed in water without depositing copper.
Plain and alloy steels	CuCl <sub>2</sub> HCl (concentrated) H <sub>2</sub> O	45 g 180 mL 100 mL	As above.	Modified Fry's reagent. Same as Fry's Reagent but modified by Wazau, may give more contrast. Specimen can be washed in water without depositing copper.
Stainless and high-Cr steels	HCl Alcohol Picric acid	10 mL 100 mL 1 g	Immerse specimen in solution at room temperature until desired contrast is obtained. Rinse and dry.	Vilella's reagent.
Plain and alloy steels	HCl (concentrated) H <sub>2</sub> O	6 to 12 mL 100 mL	Electrolytic, 5 to 10 A per square inch, specimen vertical, rinse, brush, and dry.	Small specimens, <20 in. <sup>2</sup> area.
Plain and alloy steels	HCl (concentrated) H <sub>2</sub> O HBO <sub>3</sub>	6 mL 100 mL 1 g	Electrolytic, specimen moves past a cathode bar, 30 to 40 A per inch of specimen width, rinse, brush, and dry.	For specimens over 20 in. <sup>2</sup> area, blooms, billets, and slabs.
Plain and alloy steels	H <sub>2</sub> O HCl (concentrated) H <sub>2</sub> O <sub>2</sub> (30 %)	50 mL 33 mL 17 mL	Immerse in solution at room temperature until reaction stops. Rinse in warm water, brush, dry immediately.	"3-2-1 etch." Specimens can be completely immersed. Refresh for reuse with small additions of 30 % H <sub>2</sub> O <sub>2</sub>

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