Designation: E407 - 23

Standard Practice for Microetching Metals and Alloys¹

This standard is issued under the fixed designation E407; 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 covers chemical solutions and procedures to be used in etching metals and alloys for microscopic examination. Safety precautions and miscellaneous information are also included.
- 1.2 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific cautionary statements, see 6.1 and Table 2.
- 1.3 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:²

E7 Terminology Relating to Metallography
E2014 Guide on Metallographic Laboratory Safety 6b616

3. Terminology

- 3.1 Definitions:
- 3.1.1 For definition of terms used in this standard, see Terminology E7.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *tint etch*—an immersion etchant that produces color contrast, often selective to a particular constituent in the microstructure, due to a thin oxide, sulfide, molybdate, chromate or elemental selenium film on the polished surface that

¹ This practice is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Specimen Preparation.

reveals the structure due to variations in light interference effects as a function of the film thickness (also called a "stain etch").

3.2.2 vapor-deposition interference layer method— a technique for producing enhanced contrast between microstructural constituents, usually in color, by thin films formed by vacuum deposition of a dielectric compound (such as ZnTe, ZnSe, TiO₂, ZnS or ZnO) with a known index of refraction, generally due to light interference effects (also known as the "Pepperhoff method").

4. Summary of Practice

- 4.1 Table 1 is an alphabetical listing of the metals (including rare earths) and their alloys for which etching information is available. For each metal and alloy, one or more etchant numbers and their corresponding use is indicated. Alloys are listed as a group or series when one or more etchants are common to the group or series. Specific alloys are listed only when necessary. When more than one etchant number is given for a particular use, they are usually given in order of preference. The numbers of electrolytic etchants are *italicized* to differentiate them from non-electrolytic etchants.
- 4.2 Table 2 is a numerical listing of all the etchants referenced in Table 1 and includes the composition and general procedure to be followed for each etchant.
- 4.3 To use the tables, look up the metal or alloy of interest in Table 1 and note the etchant numbers corresponding to the results desired. The etchant composition and procedure is then located in Table 2, corresponding to the etchant number.
- 4.4 If the common name of an etchant is known (Marble's, Vilella's, etc.), and it is desired to know the composition, Table 3 contains an alphabetical listing of etchant names, each coded with a number corresponding to the etchant composition given in Table 2.

5. Significance and Use

5.1 This practice lists recommended methods and solutions for the etching of specimens for metallographic examination. Solutions are listed that highlight the phases and constituents present in most major alloy systems.

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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.

6. Safety Precautions

- 6.1 Before using or mixing any chemicals, all product labels and pertinent Safety Data Sheets (SDS) should be read and understood concerning all of the hazards and safety precautions to be observed. Users should be aware of the type of hazards involved in the use of all chemicals used, including those hazards that are immediate, long-term, visible, invisible, and with or without odors. See Guide E2014 on Metallographic Laboratory Safety for additional information on; Chemical Safety, Electrolytic Polishing/Etching and Laboratory Ventilation/Fume Hoods.
- 6.1.1 Consult the product labels and SDSs for recommendations concerning proper protective clothing.
- 6.1.2 All chemicals are potentially dangerous. All persons using any etchants should be thoroughly familiar with all of the chemicals involved and the proper procedure for handling, mixing, and disposing of each chemical, as well as any combinations of those chemicals. This includes being familiar with the federal, state, and local regulations governing the handling, storage, and disposal of these chemical etchants.
- 6.2 Some basic suggestions for the handling and disposing of etchants and their ingredients are as follows:
- 6.2.1 When pouring, mixing, or etching, always use the proper protective equipment, (glasses, gloves, apron, etc.) and it is strongly recommended to always work under a certified and tested fume hood. This is imperative with etchants that give off noxious fumes or vapors that may accumulate or become explosive. In particular, note that solutions containing perchloric acid must be used in an exclusive hood equipped with a wash down feature to avoid accumulation of explosive perchlorates. See Guide E2014 on Metallographic Laboratory Safety for additional information on safety precautions for electrolytes containing perchloric acid.
- 16.2.2 No single type of glove will protect against all possible hazards. Therefore, a glove must be carefully selected and used to ensure that it will provide the needed protection for the specific etchant being used. In some instances it may be necessary to wear more than one pair of gloves to provide proper protection. Information describing the appropriate glove may be obtained by consulting the SDS for the chemical being used. If that does not provide enough detailed information, contact the chemical manufacturer directly. Additionally, one can contact the glove manufacturer or, if available, consult the manufacturers glove chart. If the chemical is not listed or if chemical mixtures are being used, contact the glove manufacturer for a recommendation.
- 6.2.3 Use proper devices (glass or plastic) for weighing, mixing, containing, and storage of solutions. A number of etchants generate fumes or vapors and should only be stored in properly vented containers. Storage of fuming etchants in sealed or non-vented containers may create an explosion hazard.
- 6.2.4 When mixing etchants, always add reagents to the solvent unless specific instructions indicate otherwise.
- 6.2.5 When etching, always avoid direct physical contact with the etchant and specimen; use devices such as tongs to hold the specimen (and tufts of cotton, if used).

- 6.2.6 Methanol is a cumulative poison hazard. Where ethanol or methanol, or both are listed as alternates, ethanol is the preferred solvent. Methanol should be used in a properly designed chemical fume hood.
- 6.2.7 When working with HF always be sure to wear the appropriate gloves, eye protection and apron. Buying HF at the lowest useable concentration will significantly reduce risk. Additionally, it is recommended that a calcium gluconate cream or other appropriate HF neutralizing agent be available for use if direct skin contact of the etchant occurs.
- 6.2.8 The EPA states that human studies have clearly established that inhaled chromium (VI) is a human carcinogen, resulting in an increased risk of lung cancer. Animal studies have shown chromium (VI) to cause lung tumors via inhalation exposure. Therefore, when working with Cr(VI) compounds such as $K_2Cr_2O_7$ and CrO_3 always use a certified and tested fume hood. Additional information can be obtained at the EPA website³.
- 6.2.9 For safety in transportation, picric acid is distributed by the manufacturer wet with greater than 30% water. Care must be taken to keep it moist because dry picric acid is shock sensitive and highly explosive especially when it is combined with metals such as copper, lead, zinc, and iron. It will also react with alkaline materials including plaster and concrete to form explosive compounds. It should be purchased in small quantities suitable for use in six to twelve months and checked periodically for lack of hydration. Distilled water may be added to maintain hydration, It must only be stored in plastic or glass bottles with nonmetallic lids. If dried particles are noted on or near the lid, submerge the bottle in water to re-hydrate them before opening. It is recommended that any bottle of picric acid that appears dry or is of unknown vintage not be opened and that proper emergency personnel be notified.
- 7-26.2.10 Wipe up or flush any and all spills, no matter how minute in nature. 94-a0268e2c22af/astm-e407-23
- 6.2.11 Properly dispose of all solutions that are not identified by composition and concentration.
- 6.2.12 Store, handle and dispose of chemicals according to the manufacturer's recommendations. Observe printed cautions on reagent bottles.
- 6.2.13 Information pertaining to the toxicity, hazards, and working precautions of the chemicals, solvents, acids, bases, etc. being used (such as safety data sheets, SDS) should be available for rapid consultation. A selection of useful books on this subject is given in Refs. (1-11)⁴.
- 6.2.14 Facilities that routinely use chemical etchants should have an employee safety training program to insure the employees have the knowledge to properly handle chemical etchants.
- 6.2.15 When working with etchants always know where the nearest safety shower, eye-wash station, and emergency telephone are located. These facilities should be close enough to the chemical working area to be effective.

³ https://iris.epa.gov/ChemicalLanding/&substance_nmbr=144

⁴ The **boldface** numbers in parentheses refer to the list of references at the end of this standard.

7. Miscellaneous Information

- 7.1 If you know the trade name of an alloy and need to know the composition to facilitate the use of Table 1, refer to a compilation such as Ref (12).
- 7.2 Reagent grade chemicals shall be used for all etchants. Unless otherwise indicated, it is intended that all reagents conform to specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades, such as United States Pharmacopeia (USP), may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without detrimental effect.
- 7.2.1 Unless otherwise indicated, references to water shall be understood to mean distilled water. Experience has shown that the quality of tap water varies significantly and can adversely affect some etchants.
- 7.3 Methanol is usually available only as absolute methanol. When using this alcohol it is imperative that approximately 5 volume % of water is added whenever an etchant composition calls for 95 % methanol. Some of these etchants will not work at all if water is not present.
- 7.4 For conversion of small liquid measurements, there are approximately 20 drops/mL.
- 7.5 Etching should be carried out on a freshly polished specimen.
- 7.6 Gentle agitation of the specimen or solution during immersion etching will result in a more uniform etch.
- 7.7 The etching times given are only suggested starting ranges and not absolute limits.
- 7.8 In electrolytic etching, direct current (DC) is implied unless indicated otherwise. AC for alternating current.
- 7.9 A good economical source of direct current for small scale electrolytic etching is the standard 6V lantern battery.
- 7.10 In electrolytic etching, the specimen is the anode unless indicated otherwise.
- 7.11 Do not overlook the possibility of multiple etching with more than one solution in order to fully develop the structure of the specimen.
- 7.12 Microscope objectives can be ruined by exposure to acid fumes from etchant residue inadvertently left on the specimen. This problem is very common when the specimen or mounting media contain porosity and when the mounting material (such as Bakelite) does not bond tightly to the specimen resulting in seepage along the edges of the specimen. In all cases, extreme care should be taken to remove all traces

of the etchant by thorough washing and complete drying of the specimen before placing it on the microscope stage.

- 7.13 Tint etchants (13, 14-16) are always used by immersion, never by swabbing, as this would inhibit film formation. An extremely high quality of polish is required as tint etchants will reveal remaining polishing damage even if it is not visible with bright field illumination. After polishing, the surface must be carefully cleaned. Use a polyethylene beaker to contain the etchant if it contains fluorine ions (for example, etchants containing ammonium bifluoride, NH₄ FHF). The specimen is placed in the solution using tongs, polished face up. Gently agitate the solution while observing the polished surface. After coloration begins, allow the solution to settle and remain motionless. Remove the specimen from the etchant when the surface is colored violet, rinse and dry. A light pre-etch with a general-purpose chemical etchant may lead to sharper delineation of the structure after tint etching.
- 7.14 Specimens should be carefully cleaned before use of a vapor-deposition interference film ("Pepperhoff") method (13, **14-17**). A light pre-etch, or a slight amount of polishing relief, may lead to sharper delineation of the constituents after vapor deposition. The deposition is conducted inside a vacuum evaporator of the type used to prepare replicas for electron microscopy. One or several small lumps of a suitable dielectric compound with the desired index of refraction is heated under a vacuum until it evaporates. A vacuum level of 1.3 to 0.013 Pa (10⁻³ to 10⁻⁵ mm Hg) is adequate and the polished surface should be about 10-15 cm beneath the device that holds the dielectric compound. Slowly evaporate the lumps and observe the surface of the specimen. It may be helpful to place the specimen on a small piece of white paper. As the film thickness increases, the surface (and the paper) will become colored with the color sequence changing in the order yellow, green, red, purple, violet, blue, silvery blue. Stop the evaporation when the color is purple to violet, although in some cases, thinner films with green or red colors have produced good results.
- 7.15 The ASM Handbook Metallography and Microstructure (18) provides additional advice on etching solutions and techniques for various alloys.

8. Precision and Bias

8.1 It is not possible to specify the precision or bias of this practice since quantitative measurements are not made.

9. Keywords

9.1 etch; etchant; interference method; metallography; metals; micro-etch; microscope; microstructure; Pepperhoff method; tint etch



TABLE 1 Etchants for Metals

Note 1—It is strongly recommended to always mix and use etchants under a certified and tested fume hood.

	Metal	Etchants	Uses
Numinum Base:			
	Pure Al	1a, 2, 3	general structure
		<i>4</i> , 5	grain structure under polarized light
		1b	grain boundaries and slip lines
	1000 series	1a, 3, 2	general structure
		4, 5	grain structure under polarized light
		6, 7	phase identification
	2000 parios	3, 2, 1a	general etructure
	2000 series	8a, 6, 7	general structure phase identification
		oa, o, r	priase identification
	3000 series	3, 1a	general structure
		4, 5	grain structure under polarized light
		8a, 6, 7	phase identification
	4000 series	3, 1a	general structure
	5000	0.4.000	
	5000 series	3, 1a, 2, 6, 8a	general structure
		4, 5	grain structure under polarized light
	6000 series	3, 1a, 2, 6, 8a, 222	general structure
	0000 001100	4, 5	grain structure under polarized light
		1a, 2, 7, 6, 8a	phase identification
			F
	7000 series	3, 1a, 2	general structure
		Teh St 3b, 6 0 210 S	grain structure under polarized light
		3b, 6	phase identification
5			
eryllium Base:	Duro Po	(https://standards.itah.ai)	general structure via polarized light
	Pure Be Be alloys	(https://stanc ^{9, 10} rds.iteh.ai)	general structure via polarized light general structure
	be alloys		general structure
hromium Base:		Documer ^{12, 13c} review	general structure
oa Daco.		Ducument I eview	gonoral ciractare
obalt Base:			
	Pure Co	14, 15, 16, 17	general structure
Hard-	facing and tool meta		general structure
	h-temperature alloys		general structure
		atalog/standards/sist/6b616 19 31-2767-4496-a094-a02	68e2c_phase identification /-23
aloualaioua Dana (/:-b: b)		
olumbium Base ((see niobium base)		
opper Base:			
оррег вазе.	Pure Cu	26, 27, 28, 29, 30, 31d, 32, 33, 34b, 35,	general structure
		36, 37, 38, 39, 40, 41, 42, <i>8b</i> , 210, 215	gonoral chactare
		43, 28	chemical polish and etch
			•
Cu-Al	I (aluminum bronze)	44, 31d, 34b, 35, 36, 37, 38, 39, 40,	general structure
	_	45, 215	
	Cu-Be	46, 41, 45	general structure
	Cu-Cr	41	general structure
	Cu-Mn	41	general structure
	Cu-Ni	34, 47, 48, 40, 49, 50	general structure
	Cu-Si	41	general structure
Cı	u-Sn (tin bronze)	51, 52	general structure
	Admiralty matal	Oh	gonoral etructura
Admiralty metal Gilding metal Cartridge brass		8b	general structure
(ree-cutting brass		
	Nickel silver	31d, 32, 33, 41, 42, 49	general structure
	Nickei Silvei	, - ,,,,	9
	Nickei Silvei		
	Cu alloys	26, 27, 28, 29, 30, 44, 41, 31d, 32, 33,	general structure
		26, 27, 28, 29, 30, 44, 41, 31d, 32, 33, 34b, 35, 36, 37, 38, 39, 210, 215	general structure
			chemical polish and etch
		34b, 35, 36, 37, 38, 39, 210, 215	
		34b, 35, 36, 37, 38, 39, 210, 215 53, 43, 28, <i>49</i>	chemical polish and etch
Fr		34b, 35, 36, 37, 38, 39, 210, 215 53, 43, 28, <i>49</i> 42, <i>49</i> , 210	chemical polish and etch darkens beta in alpha-beta brass

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Iron Base: 73e	Indium Base: Pure Fe			·
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Mg-Al, Mg-Al-Zn (Al + Zn <5 %) 118, 119, 74a, 125, 124, 123, 122 120, 125, 126, 127 Mg-Al, Mg-Al-Zn (Al + Zn >5 %) 118, 119, 74a, 125, 124, 121, 122 120, 125, 126, 127 Mg-Al, Mg-Al-Zn (Al + Zn >5 %) 118, 119, 74a, 125, 124, 121, 122 120, 125, 126, 127 Mg-Zn-Tn -Zr 118, 119, 74a, 14, 128, 124, 126, 127 Mg-Zn-Tn -Zr 118, 119, 74a, 14, 128, 124, 126, 127 Mg-Tn-Th-Zr Mg-Tn-Tr -Zn Mg-Rare Earth-Zr Mg-Rare Earth-Zr Molybdenum Base: As cast 132a Nickel Base: 133a Ni-Ag Ni-Al Ni-Ag Ni-Al Ni-Cr 144, 50, 83, 134, 145, 98, 146, 147, 13a Ni-Cr 144, 50, 83, 134, 145, 98, 146, 147, 13a general structure Ni-Cu 38, 138, 50, 139 general structure 38, 138, 50, 139 general structure Ni-Cu 38, 138, 50, 139, 146, 147, 13a general structure Ni-Cu 38, 138, 50, 133, 140, 25, 134, 47, general structure 74e general structure 148b, 94, 108, 34 Ni-Fe 50, 140, 141, 183, 184, 148, 40, 107, 149 general structure				
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ASTM E407-23 Nickel Base: ASTM E407-23			<u> </u>	
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74e, 25, 150 Orientation pitting	Ni-Fe	<i>50</i> , 140, 141, <i>83</i> , <i>134</i> , 148, 40, 107, 149	general structure	
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154 fine precipitation structure		107, 111, <i>13a</i>	reveals microstructural inhomogeneity	
154 fine precipitation structure		133	grain boundary sulfidation	
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700, 100, 100 dillorential matrix and nonlinetallic stallilli			·	
22a for passive alloys (for example, UNS Alloy NO			for passive alloys (for example, UNS Alloy N06625)
				,
157 specific for UNS Alloy N10004				
		10/	submicroscopic structure in aged superalloys particular	
			larly for electron microscopy. Stains the matrix when	γ'
precipitates are present			· · ·	
154 γ' banding				
18 pre-etch activation for passive specimen		18	pre-etch activation for passive specimens	
213 colors carbide and v'		213	colors carbide and γ'	



	TABLE 1 Continued	
Metal	Etchants	Uses
Niobium (Columbium) Base:	129, 66, 158, 159, 160, 161, 162, 163	general structure
	164, 129, 160	grain boundaries
Osmium Base:	165a	general structure
Osimiani Base.	165a	etch-polishing for viewing grains with polarized light
		F
Palladium Base:		
Pure Pd	61, 166, 62, <i>165a</i>	general structure
Pd alloys >90 % noble metals	166, 64a, 62, <i>165a</i>	general structure
<90 % noble metals	61 65	general structure general structure
200 /6 Hobic Hictars	00	general structure
Platinum Base:		
Pure Pt	64a, <i>73a</i>	general structure
	167	electrolytic polish and etch
Pt Alloys	64b, <i>73a</i>	general structure
	167	electrolytic polish and etch
>90 % noble metals	61	general structure
<90 % noble metals	65	general structure
Pt-10 % Rh	168	general structure
Distanting B	400	
Plutonium Base: Rhenium Base:	169 13h 98c 132h 170a	general structure
Rhenium Base: Rhodium Base:	<i>13b</i> , 98c, 132b, 170a 171	general structure general structure
Ruthenium Base:	73b	general structure
	73b	etch-polishing for viewing grains with polarized light
Silver Base:	470 470 00	and and about the
Pure Ag Ag alloys	172, 173, 62 65, 61, 174, 175, 62	general structure general structure
Ag-Cu alloys	130	general structure
Ag-Pd alloys	173	general structure
Ag solders	173, 176	general structure
Tantalum Base:	ups://stangards.item.	al)
Pure Ta	177	general structure
Ta alloys	159, 66, 178, 163, 161, 179 164	general structure grain boundaries and inclusions
	158 164 164 N	grain boundaries—retains carbide precipitate
Thorium Base:		
Pure Th	ASTM F185 7_23	general structure
Th alloys	185 1767 1406 a00	general structure
https://standards.iteh.ai/catalog		
<i>Tin Base:</i> Pure Sn	74d, 180, 151	general structure
i die on	181	grain boundaries
Sn-Cd	74d	general structure
Sn-Fe	74d, 177a	general structure
Sn-Pb	182, 183, 74b	general structure
	116	darkens Pb in Sn-Pb eutectic
Sn coatings (on steel)	183	general structure
Babbitts Sn-Sb-Cu	184 74b	general structure general structure
S/I-Ob-Ou	7-75	gonoral structure
Titanium Base:		
Pure Ti	186, 187, <i>67, 68, 69</i> , 217	general structure
	188	removes stain
TI F ALO F Co	72	chemical polish and etch
Ti-5 Al-2,5 Sn Ti-6 Al-6 V-2 Sn	189 190	reveals hydrides Stains alpha and transformed beta, retained beta
11 0 711 0 7 2 011	100	remains white
Ti-Al-Zr	191	general structure
Ti-8Mn	192	general structure
Ti-13 V-11 Cr-3 AI (aged)	192	general structure
Ti-Si Ti allovs	193 196 197 102 104 159 1225 10 67	general structure
Ti alloys	186, 187, 192, 194, 158, 132b, 1c, <i>67, 68, 69,</i> 3a, 218	general structure
	11, 1c	reveals alpha case
	72, 192, 178	chemical polish and etch
	170a	outlines and darkens hydrides in some alloys
	188	removes stain
Tungsten Base:		

Metal	Etchants	Uses
Б. Ж	00 404	
Pure W	98c, <i>131</i>	general structure
As cast	132a	chemical polish prior to etching
W-Th	209	general structure
Uranium Base:		
Pure U	67, <i>69, 195, 196</i>	general structure
U + Zr	68	general structure
U beryllides	170a	general structure
U alloys	67, 69, 195, 96	general structure
2 3, 5	207	carbides
Vanadium Base:		
Pure V	170b, <i>165b</i>	general structure
	<i>197</i> , 198	grain boundaries
V alloys	199, 198	general structure
Zinc Base:		
Pure Zn	200a	general structure
Zn-Co	177	general structure
Zn-Cu	201	general structure
	203	distinguishes gamma (γ) and epsilon (ϵ)
Zn-Fe	74a	structure of galvanized sheet
Die castings	202	general structure
Zirconium Base:	66 67 204 68 60 205	general structure
Ziiconium base.	66, <i>67</i> , 204, 68, 69, 205 206	electrolytic polish and etch
	71	grain structure under polarized light
	72	chemical polish and etch

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TABLE 2 Numerical List of Etchants

Note 1—It is strongly recommended to always mix and use etchants under a certified and tested fume hood.

	Composition	Procedure
1	1 mL HF	(a) Swab with cotton for 15 s.
	200 mL water	(b) Alternately immerse and polish several minutes.(c) Immerse 3–5 s.(d) Immerse 10–120 s.
2	3 mL HF	(a) Swab 10 s to reveal general structure.
_	100 mL water	(b) Immerse 15 min, wash 10 min in water to form film with hatching that varies with grain orientation.
3	2 mL HF	(a) Immerse 10-20 s Wash in stream of warm water. Reveals general structure.
	3 mL HCl	(b) Dilute with 4 parts water. Colors constituents—mix fresh.
	5 mL HNO ₃	
	190 mL water	
4	24 mL $\rm H_3PO_4$	Electrolytic: Use carbon cathode raising DC voltage from 0–30 V in 30 s. Total etching time 3 min with agitation.
	50 mL Carbitol (diethylene glycol monoethy	
	ether)	
	4 g boric acid	
	2 g oxalic acid	
	10 mL HF	
	32 mL water	
5	5 g HBF₄ 200 mL water	Electrolytic: Use Al, Pb, or stainless steel cathode. Anodize 1–3 min, 20–45 V DC. At 30 V, etch for 1 min.
6	25 mL HNO ₃ 75 mL water	Immerse 40 s at 70°C (160°F). Rinse in cold water.
7	10-20 mL H ₂ SO ₄	Immerse 30 s at 70°C (160°F). Rinse in cold water.
	80 mL water	
8	10 mL H ₃ PO ₄	(a) Immerse 1–3 min at 50°C (120°F).
	90 mL water	(b) Electrolytic at 1–8 V for 5–10 s.
9	3-4 g sulfamic acid	Use just prior to the last polishing operation. It is not intended as a final etchant. The
	5 drops HF	specimen is examined as polished under polarized light.
	100 mL water	
10	10 mL HF 90 mL methanol (90 %)	Immerse 10–30 s.
	AST	
11	2 mL HF	Immerse or swab few seconds to a minute.
	s.iteh.ai/catal(100 mL water ds/sist/6)	
	00 LUNO	Here a servicio de servicio de la Companya de la Co
12	20 mL HNO ₃	Use a certified and tested hood. Do not store. Immerse or swab 5-60 s.
12	$20~\mathrm{mL}~\mathrm{HNO_3}$ $60~\mathrm{mL}~\mathrm{HCI}$	Use a certified and tested hood. Do not store. Immerse or swab 5-60 s.
	60 mL HCl	
13	60 mL HCl 10 g oxalic acid	Electrolytic at 6 V:
	60 mL HCl	Electrolytic at 6 V: (a) 10–15 s.
	60 mL HCl 10 g oxalic acid	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min.
	60 mL HCl 10 g oxalic acid	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s.
	60 mL HCl 10 g oxalic acid	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min.
	60 mL HCl 10 g oxalic acid	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s.
13	60 mL HCI 10 g oxalic acid 100 mL water	Electrolytic at 6 V: (a) 10-15 s. (b) 1 min. (c) 2-3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen.
13	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute.
13	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute.
13	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %)	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute.
13	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolyti-
13	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolyti-
13 14 15	10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCl 15 mL water	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically.
13	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCI 15 mL water 5–10 mL HCI	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolyti-
13 14 15	10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCl 15 mL water	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically.
13 14 15	10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCl 15 mL water 5–10 mL HCl 100 mL water	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically. Electrolytic at 3 V for 2–10 s.
13 14 15	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCI 15 mL water 5–10 mL HCI 100 mL water 5 mL HCI	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically.
13 14 15	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCI 15 mL water 5–10 mL HCI 100 mL water 5 mL HCI 100 g FeCI ₃	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically. Electrolytic at 3 V for 2–10 s.
13 14 15	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCI 15 mL water 5–10 mL HCI 100 mL water 5 mL HCI	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically. Electrolytic at 3 V for 2–10 s.
13 14 15 16 17	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCI 15 mL water 5–10 mL HCI 100 mL water 5 mL HCI 100 mL water	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically. Electrolytic at 3 V for 2–10 s. Electrolytic at 6 V for few seconds.
13 14 15	60 mL HCI 10 g oxalic acid 100 mL water 10 mL HNO ₃ 90 mL methanol (95 %) 15 mL HNO ₃ 15 mL acetic acid 60 mL HCI 15 mL water 5–10 mL HCI 100 mL water 5 mL HCI 100 g FeCI ₃	Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute. Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically. Electrolytic at 3 V for 2–10 s.



Etchant	Composition	Procedure Procedure
19	A 8 g NaOH 100 mL water B	Immerse in freshly mixed Solutions A + B (1:1) for 5–10 s. If surface activation is necessary, first use Etch #18, then rinse in water. While still wet, immerse in Solutions A + B (1:1). Mixture of solutions A + B has 15-min useful life. Note: KMnO ₄ is an aggressive staining agent.
	Saturated aqueous solution of KMnO	sive staining agent.
20	5 mL H $_2$ O $_2$ (30 %) 100 mL HCl	Use a certified and tested hood. Mix fresh. Immerse polished face up for few seconds.
21	1 g CrO ₃ 140 mL HCl	Use a certified and tested hood. To mix, add the HCl to ${\rm CrO_3}$. Electrolytic at 3 V for 2–10 s.
22	100 mL HCl 0.5 mL H $_2$ O $_2$ (30 %)	Use a certified and tested hood. Do not store. (a) Immerse or swab $\frac{1}{2}$ –3 min. Add H ₂ O ₂ dropwise to maintain action. (b) Electrolytic, 4 V, 3–5 s.
23	5 mL HCI	Electrolytic at 6 V for 10–20 s.
24	95 mL ethanol (95 %) or methanol (95 %) 5 mL HNO ₃ 200 mL HCl 65 g FeCl ₃	Use a certified and tested hood. Immerse few seconds.
25	$10~{\rm g~CuSO_4}$ $50~{\rm mL~HCl}$ $50~{\rm mL~water}$	Immerse or swab 5–60 s. Made more active by adding few drops of $\rm H_2~SO_4$ just before use.
26	5 g FeCl ₃ 10 mL HCl 50 mL glycerol 30 mL water	Swab 16-60 s. Activity may be decreased by substituting glycerol for water.
27	1 g KOH 20 mL H ₂ O ₂ (3 %) 50 mL NH ₄ OH 30 mL water	Dissolve KOH in water, then slowly add NH ₄ OH to solution. Add 3 % H ₂ O ₂ last. Use fresh—immerse few seconds to a minute.
28	1 g FeNO ₃ 100 mL water	Swab or immerse few seconds to a minute.
29	1 g K_2 Cr_2 O_7 4 mL H_2 SO_4 50 mL water	Use a certified and tested hood. Add 2 drops of HCl just before using. Swab few seconds to a minute.
https://standards	$\begin{array}{c} 25 \text{ mL NH}_4 \text{ OH} \\ 25 \text{ mL water rds/sist/} \\ 50 \text{ mL H}_2 \text{ O}_2 \text{ (3 \%)} \end{array}$	Mix NH $_4$ OH and water before adding H $_2$ O $_2$. Must be used fresh. Swab 5–45 s. 6b616d31-2767-44496-a094-a0268e2c22a1/astm-e407-23
31	10 g ammonium persulfate 100 mL water	 (a) Swab or immerse up to 5 s. (b) Immerse up to 2 min to darken matrix to reveal carbides and phosphides. (c) Electrolytic at 6 V for few seconds to a minute. (d) Immerse 3–60 s. Can be heated to increase activity.
32	60 g CrO ₃ 100 mL water	Use a certified and tested hood. Saturated solution. Immerse or swab 5–30 s.
33	10 g CrO ₃	Use a certified and tested hood. Add HCl just before use. Immerse 3–30 s. Phases can be colored by Nos. 35, 36, 37.
	2–4 drops HCl 100 mL water	Colored by 1403. 33, 30, 37.
34	5 g FeCl ₃ 50 mL HCl 100 mL water	(a) Immerse or swab few seconds to few minutes. Small additions of ${\rm HNO_3}$ activate solution and minimize pitting.
		(b) Immerse or swab few seconds at a time. Repeat as necessary.
35	20 g FeCl ₃ 5 mL HCl 1 g CrO ₃ 100 mL water	Use a certified and tested hood. Immerse or swab few seconds at a time until desired results are obtained.
36	25 g FeCl ₃ 25 mL HCl 100 mL water	Immerse or swab few seconds at a time until desired results are obtained.