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Standard Practice for Microetching Metals and Alloys[1]

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.

ε^1 NOTE—Originally approved date was editorially corrected to 1970 in footnote 1 in January 2016.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific cautionary statements, see 6.1 and Table 2.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E7 Terminology Relating to Metallography

E2014 Guide on Metallographic Laboratory Safety

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 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, TiO_2, 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 nonelectrolytic etchants.

4.2 Table 2 is a numerical listing of all the etchants referenced in Table 1and 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 2corresponding 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 3contains 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 to highlight phases present in most major alloy systems.

6. Safety Precautions

6.1 Before using or mixing any chemicals, all product labels and pertinent Material Safety Data Sheets (MSDS) should be

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[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.

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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.

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 MSDSs 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 odors or toxic 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..

6.2.2 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 MSDS 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 website[3].

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.

6.2.10 Wipe up or flush any and all spills, no matter how minute in nature.

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 material safety data sheets, MSDS) 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 which 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.

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

[3] http://www.epa.gov/ttn/atw/hlthef/chromium.html

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

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 reagent water as defined by Type IV of specification D1193. 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, d-c current is implied unless indicated otherwise.

7.9 A good economical source of d-c current for small scale electrolytic etching is the standard 6-V 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 hydrofluoric 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 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, NH_4 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 delination 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 Metals Handbook (**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; microetch; 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.

NOTE 2—Electrolytic etchants are *italicized*.

Metal	Etchants	Uses
Aluminum Base:		
Pure Al	1a, 2, 3	general structure
	4, 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 identifications
2000 series	3, 2, 1a	general structure
	8a, 6, 7	phase identifications
3000 series	3, 1a	general structure
	4, 5	grain structure under polarized light
	8a, 6, 7	phase identifications
4000 series	3, 1a	general structure
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
	4, 5	grain structure under polarized light
	1a, 2, 7, 6, 8a	phase identifications
7000 series	3, 1a, 2	general structure
	4, 5	grain structure under polarized light
	3b, 6	phase identifications
Beryllium Base:		
Pure Be	9, 10	general structure via polarized light
Be alloys	11	general structure
Chromium Base:	12, *13c*	general structure
Cobalt Base:		
Pure Co	14, 15, 16, 17	general structure
Hard-facing and tool metals	18, 19, 20	general structure
High-temperature alloys	20, 18, 16, 21, 22b, 24, 25	general structure
	19	phase identification
Columbium Base (see niobium base)		
Copper Base:		
Pure Cu	26, 27, 28, 29, 30, 31d, 32, 33, 34b, 35, 36, 37, 38, 39, 40, 41, 42, *8b*, 210, 215	general structure
	43, 28	chemical polish and etch
Cu-Al (aluminum bronze)	44, 31d, 34b, 35, 36, 37, 38, 39, 40, 45, 215	general structure
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
Cu-Sn (tin bronze)	51, 52	general structure
Admiralty metal	*8b*	general structure
Gilding metal		
Cartridge brass		
Free-cutting brass		
Nickel silver	31d, 32, 33, 41, 42, 49	general structure
Cu alloys	26, 27, 28, 29, 30, 44, 41, 31d, 32, 33, 34b, 35, 36, 37, 38, 39, 210, 215	general structure
	53, 43, 28, *49*	chemical polish and etch
	42, *49*, 210	darkens beta in alpha-beta brass
	54	etching of cold worked brass
Dysprosium Base:	55, 56	general structure

TABLE 1 *Continued*

Metal	Etchants	Uses
Erbium Base:	55, 56	general structure
Gadolinium Base:	55, 56, 57	general structure
Germanium Base:	58, 59, 60	general structure
Gold Base:		
Pure Au	61, 62	general structure
	63	chemical polish and etch
Au alloys	64b, 62	general structure
	63	chemical polish and etch
>90 % noble metals	61	general structure
<90 % noble metals	65	general structure
Hafnium base:	66, *67*, *68*, *69*, 70	general structure
	71	grain structure under polarized light
	72	chemical polish and etch
Holmium Base:	55, 56	general structure
Iridium Base:	*73c*	general structure
Iron Base:		
Pure Fe	74a	grain boundaries
	75	substructure
	210	colors ferrite grains
Fe + C and Fe + <1C + <4 % additions	76, 74a, 77, 78, 79	general structure
	74a, 77, 31a, 223	ferrite grain boundaries
	80, 81, 82	prior austenitic grain boundaries in martensitic and bainitic steels
	78, 222a	untempered martensite
	31b, 78	carbides and phosphides (matrix darkened, carbides and phosphides remain bright)
	83	cementite attacked rapidly, sustenite less, ferrite and iron phosphide least
	84	overheating and burning
	85	stains carbides
	86	chemical polish-etch
	210, 211	colors ferrite
	213, 214	colors carbides
	216	colors lath martensite in low-carbon high-alloy grades
	222b	for dual phase steels; reveals pearlite, darkens martensite and outlines austenite
Fe + 4–12 Cr	80, 87, 88, 89, 90, 91, 79, 210	general structure
	86	chemical polish-etch
Fe + 12–30 Cr + <6 Ni (400 Series)	80, 87, 88, 89, 34, 40, 92, *93*, 94, 95, 91, 226	general structure
	96, 97, 98	signs phase
	31c	carbides
	86	chemical polish-etch
	219	grain boundary etch
	220	darkens delta ferrite
Fe + 12–20 Cr + 4–10 Ni + <7 % other elements (controlled transformation, precipitation hardening, stainless maraging alloys)	80, *31c*, 89, 99, 100, 91	general structure
	31c	carbides
	86	chemical polish-etch
	220	darkens delta ferrite
Fe + 15–30 Cr + 6–40 Ni + <5 % other elements (300 Series) and Fe + 16–25 Cr + 3–6 Ni + 5–10 Mn (200 series)	*13b*, 89, 87, 88, *83a*, 80, 94, 95, 91, 101, 212, 221, 226	general structure
	13a, 102, 31c, 48c, 213	carbides and sensitization
	48, 96, 97, 98	stains sigma phase
	103, 104, 98	delineates sigma phase and
	103, 104	welds of dissimilar metals
	86	chemical polish-etch
	219	grain boundary etch (no twins)
	220	darkens delta ferrite
High temperature	89, 25, 105, 106, *97, 212, 221*	general structure
	107, *108*, 213	γ' precipitate
	86	chemical polish-etch
Nonstainless maraging steels	109, 89, 99, 100, 221	general structure
	83b	grain boundaries
	86	chemical polish-etch

TABLE 1 *Continued*

Metal	Etchants	Uses
Tool steels	74a, 80, 14	general structure
	110	grain boundaries in tempered tool steel
	210, 211	colors ferrite, lower alloy grades
	214, 214	colors cementite
	224, 225	carbides attacked and colored
Superalloys	86, 87, 94, 221, 226	general etch
	111	general structure
	111	γ' depletion
Lead Base:		
Pure Pb	57, 112	general structure
	113	for alternate polishing and etching
Pb + <2 Sb	114, 115, 57, 74b	general structure
	113	for alternate polishing and etching
Pb + >2 Sb	114, 57, 74b	general structure
	113	for alternate polishing and etching
Pb + Ca	112	general structure
	113	for alternate polishing and etching
Pb alloys	116, 117b	general structure
Babbitt	74b	general structure
Magnesium Base:		
Pure Mg	118, 119, 74a, 120, 121, 122	general structure
	123	stain-free polish-etch
Mg-Mn	119, 74a, 124, 122	general structure
Mg-Al, Mg-Al-Zn (Al + Zn <5 %)	118, 119, 74a, 125, 124, *123*, 122	general structure
	120, 125, 126, 127	phase identification
	124, 126, 127	grain structure
Mg-Al, Mg-Al-Zn (Al + Zn >5 %)	118, 119, 74a, 125, 124, 121, 122	general structure
	120, 125, 126, 127	phase identification
Mg-Zn-Zr and Mg-Zn-Th-Zr	118, 119, 74a, 1d, 128, 124, 126, 127, 121, 122	general structure
	120, 121	phase identification
Mg-Th-Zr and Mg-Rare Earth-Zr	118, 119, 74a, 1d, 124, 127, 121, 122	general structure
	120, 121	phase identification
Molybdenum Base:	98c, 129, 130, *131*	general structure
As cast	132a	chemical polish prior to etching
Nickel Base:		
Pure Ni and high Ni alloys	133, 134, 47, 135, 136, 25, 108, 31c	general structure
	137	grain boundary sulfidation
Ni-Ag	38, 138, 50, 139	general structure
Ni-Al	*50*, 140, 141, *142*, 89, 143	general structure
Ni-Cr	144, 50, *83, 134, 145*, 98, 146, 147, *13a*	general structure
Ni-Cu	38, 138, 50, 133, 140, 25, *134*, 47, *48b*, 94, *108*, 34	general structure
Ni-Fe	*50*, 140, 141, *83, 134*, 148, 40, 107, 149	general structure
	74e, 25, 150	orientation pitting
Ni-Mn	74e	general structure
Ni-Mo	143	general structure
Ni-Ti	143, 151, 50, 133	general structure
Ni-Zn	152	general structure
Superalloys	94, 105, 138, 153, 12, 87, 89, 212, 226	general structure
	25, 94	grain size
	107, 111, *13a*	reveals microstructural inhomogeneity
	133	grain boundary sulfidation
	154	fine precipitation structure
	19b, 155, 156	differential matrix and nonmetallic staining
	22a	for passive alloys (for example, UNS Alloy N06625)
	157	specific for UNS Alloy N10004
	107	submicroscopic structure in aged super-alloys particularly for electron microscopy. Stains the matrix when γ' precipitates are present
	154	γ' banding
	18	pre-etch activation for passive specimens
	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
	165a	etch-polishing for viewing grains with polarized light
Palladium Base:		
Pure Pd	61, 166, 62, *165a*	general structure
Pd alloys	166, 64a, 62, *165a*	general structure
>90 % noble metals	61	general structure
<90 % noble metals	65	general structure
Platinum Base:		
Pure Pt	64a, *73a*	general structure
	167	electrolytic polish and etch
Pt Alloys	64b, *73a*	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
Plutonium Base:	169	general structure
Rhenium Base:	*13b,* 98c, 132b, 170a	general structure
Rhodium Base:	171	general structure
Ruthenium Base:	73b	general structure
	73b	etch-polishing for viewing grains with polarized light
Silver Base:		
Pure Ag	172, 173, 62	general structure
Ag alloys	65, 61, 174, 175, 62	general structure
Ag-Cu alloys	130	general structure
Ag-Pd alloys	173	general structure
Ag solders	173, 176	general structure
Tantalum Base:		
Pure Ta	177	general structure
Ta alloys	159, 66, 178, 163, 161, 179	general structure
	164	grain boundaries and inclusions
	158	grain boundaries—retains carbide precipitate
Thorium Base:		
Pure Th	185	general structure
Th alloys	185	general structure
Tin Base:		
Pure Sn	74d, 180, 151	general structure
	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	184	general structure
Sn-Sb-Cu	74b	general structure
Titanium Base:		
Pure Ti	186, 187, *67, 68, 69*, 217	general structure
	188	removes stain
	72	chemical polish and etch
Ti-5 Al-2,5 Sn	189	reveals hydrides
Ti-6 Al-6 V-2 Sn	190	Stains alpha and transformed beta, retained beta re mains white
Ti-Al-Zr	191	general structure
Ti-8Mn	192	general structure
Ti-13 V-11 Cr-3 Al (aged)	192	general structure
Ti-Si	193	general structure
Ti alloys	186, 187, 192, 194, 158, 132b, 1c, *67, 68, 69*, 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:		

TABLE 1 *Continued*

Metal	Etchants	Uses
Pure W	98c, *131*	general structure
As cast	132a	chemical polish prior to etching
W-Th	209	general structure
Uranium Base:		
Pure U	67, *69, 195, 196*	general structure
U + Zr	68	general structure
U beryllides	170a	general structure
U alloys	*67, 69, 195, 96*	general structure
	207	carbides
Vanadium Base:		
Pure V	170b, *165b*	general structure
	197, 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, 69, 205	general structure
	206	electrolytic polish and etch
	71	grain structure under polarized light
	72	chemical polish and etch

TABLE 2 Numerical List of Etchants

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

Etchant	Composition	Procedure
1	1 mL HF 200 mL water	(a) Swab with cotton for 15 s. (b) Alternately immerse and polish several minutes. (c) Immerse 3–5 s. (d) Immerse 10–120 s.
2	3 mL HF 100 mL water	(a) Swab 10 s to reveal general structure. (b) Immerse 15 min, wash 10 min in water to form film with hatching which varies with grain orientation.
3	2 mL HF 3 mL HCl 5 mL HNO_3 190 mL water	(a) Immerse 10–20 s Wash in stream of warm water. Reveals general structure. (b) Dilute with 4 parts water-colors constituents—mix fresh.
4	24 mL H_3PO_4 50 mL Carbitol (diethylene glycol monoethyl ether) 4 g boric acid 2 g oxalic acid 10 mL HF 32 mL water	Electrolytic: Use carbon cathode raising d-c voltage from 0–30 V in 30 s. Total etching time 3 min with agitation. Wash and cool. Repeat if necessary.
5	5 g HBF_4 200 mL water	Electrolytic: Use Al, Pb, or stainless steel cathode. Anodize 1–3 min, 20–45 V d-c. 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_2SO_4 80 mL water	Immerse 30 s at 70°C (160°F). Rinse in cold water.
8	10 mL H_3PO_4 90 mL water	(a) Immerse 1–3 min at 50°C (120°F). (b) Electrolytic at 1–8 V for 5–10 s.
9	3–4 g sulfamic acid 5 drops HF 100 mL water	Use just prior to the last polishing operation. It is not intended as a final etchant. The specimen is examined as polished under polarized light.
10	10 mL HF 90 mL methanol (90 %)	Immerse 10–30 s.
11	2 mL HF 100 mL water	Immerse or swab few seconds to a minute.
12	20 mL HNO_3 60 mL HCl	Use a certified and tested hood. Do not store. Immerse or swab 5–60 s.
13	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.
14	10 mL HNO_3 90 mL methanol (95 %)	Immerse few seconds to a minute.
15	15 mL HNO_3 15 mL acetic acid 60 mL HCl 15 mL water	Use a certified and tested hood. Age before use. Immerse 5–30 s. May be used electrolytically.
16	5–10 mL HCl 100 mL water	Electrolytic at 3 V for 2–10 s.
17	5 mL HCl 10 g $FeCl_3$ 100 mL water	Electrolytic at 6 V for few seconds.
18	2–10 g CrO_3 100 mL water	Use a certified and tested hood. Electrolytic at 3 V for 2–10 s.

TABLE 2 *Continued*

Etchant	Composition	Procedure
19	A 8 g NaOH 100 mL water B Saturated aqueous solution of $KMnO_4$	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 agressive staining agent.
20	5 mL H_2O_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 CrO_3. Electrolytic at 3 V for 2–10 s.
22	100 mL HCl 0.5 mL H_2O_2 (30 %)	Use a certified and tested hood. Do not store. (a) Immerse or swab ½ –3 min. Add H_2O_2 dropwise to maintain action. (b) Electrolytic, 4 V, 3–5 s.
23	5 mL HCl 95 mL ethanol (95 %) or methanol (95 %)	Electrolytic at 6 V for 10–20 s.
24	5 mL HNO_3 200 mL HCl 65 g $FeCl_3$	Use a certified and tested hood. Immerse few seconds.
25	10 g $CuSO_4$ 50 mL HCl 50 mL water	Immerse or swab 5–60 s. Made more active by adding few drops of H_2SO_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_2O_2 (3 %) 50 mL NH_4OH 30 mL water	Dissolve KOH in water, then slowly add NH_4OH to solution. Add 3 % H_2O_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 $K_2Cr_2O_7$ 4 mL H_2SO_4 50 mL water	Use a certified and tested hood. Add 2 drops of HCl just before using. Swab few seconds to a minute.
30	25 mL NH_4OH 25 mL water 50 mL H_2O_2 (3 %)	Mix NH_4OH and water before adding H_2O_2. Must be used fresh. Swab 5–45 s.
31	10 g ammonium persulfate 100 mL water	(a) Swab or immerse to 5 s. (b) Immerse 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 2–4 drops HCl 100 mL water	Use a certified and tested hood. Add HCl just before use. Immerse 3–30 s. Phases can be colored by Nos. 35, 36, 37.
34	5 g $FeCl_3$ 50 mL HCl 100 mL water	(a) Immerse or swab few seconds to few minutes. Small additions of 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.