



Designation: E407 – 07

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

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 ( $\epsilon$ ) 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 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:*<sup>2</sup>

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<sub>2</sub>, 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 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 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 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,

<sup>1</sup> This practice 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 May 1, 2007. Published May 2007. Originally approved in 1999. Last previous edition approved in 1999 as E407-99 DOI: 10.1520/E0407-07.

<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.

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 E 2014E2014 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 manufacturer's 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<sup>3</sup>.

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)<sup>4</sup>.

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 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

<sup>3</sup> <http://www.epa.gov/ttn/atw/hlthef/chromium.html>

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

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,  $\text{NH}_4\text{FHF}$ ). 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 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
<b>Aluminum Base:</b>		
Pure Al	1a, 2, 3 4, 5 1b	general structure grain structure under polarized light grain boundaries and slip lines
1000 series	1a, 3, 2 4, 5 6, 7	general structure grain structure under polarized light phase identifications
2000 series	3, 2, 1a 8a, 6, 7	general structure phase identifications
3000 series	3, 1a 4, 5 8a, 6, 7	general structure grain structure under polarized light phase identifications
4000 series	3, 1a	general structure
5000 series	3, 1a, 2, 6, 8a 4, 5	general structure grain structure under polarized light
6000 series	3, 1a, 2, 6, 8a, 222 4, 5 1a, 2, 7, 6, 8a	general structure grain structure under polarized light phase identifications
7000 series	3, 1a, 2 4, 5 3b, 6	general structure grain structure under polarized light phase identifications
<b>Beryllium Base:</b>		
Pure Be	9, 10	general structure via polarized light
Be alloys	11	general structure
<b>Chromium Base:</b>		
	12, 13c	general structure
<b>Cobalt Base:</b>		
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 19	general structure phase identification
<b>Columbium Base (see niobium base)</b>		
<b>Copper Base:</b>		
Pure Cu	26, 27, 28, 29, 30, 31d, 32, 33, 34b, 35, 36, 37, 38, 39, 40, 41, 42, 8b, 210, 215 43, 28	general structure 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 53, 43, 28, 49 42, 49, 210 54	general structure chemical polish and etch darkens beta in alpha-beta brass etching of cold worked brass
<b>Dysprosium Base:</b>		
	55, 56	general structure

**TABLE 1** *Continued*

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

**TABLE 1** *Continued*

Metal	Etchants	Uses
Tool steels	74a, 80, 14 110 210, 211 214, 214 224, 225	general structure grain boundaries in tempered tool steel colors ferrite, lower alloy grades colors cementite carbides attacked and colored
Superalloys	86, 87, 94, 221, 226 111 111	general etch general structure $\gamma'$ depletion
<i>Lead Base:</i> Pure Pb	57, 112 113	general structure for alternate polishing and etching
Pb + <2 Sb	114, 115, 57, 74b 113	general structure for alternate polishing and etching
Pb + >2 Sb	114, 57, 74b 113	general structure for alternate polishing and etching
Pb + Ca	112 113	general structure for alternate polishing and etching
Pb alloys Babbitt	116, 117b 74b	general structure general structure
<i>Magnesium Base:</i> Pure Mg	118, 119, 74a, 120, 121, 122 123	general structure 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, 122 120, 125, 126, 127 124, 126, 127	general structure general structure phase identification grain structure
Mg-Al, Mg-Al-Zn (Al + Zn >5 %)	118, 119, 74a, 125, 124, 121, 122 120, 125, 126, 127	general structure phase identification general structure
Mg-Zn-Zr and Mg-Zn-Th-Zr	118, 119, 74a, 1d, 128, 124, 126, 127, 121, 122 120, 121	phase identification general structure
Mg-Th-Zr and Mg-Rare Earth-Zr	118, 119, 74a, 1d, 124, 127, 121, 122 120, 121	general structure phase identification
<i>Molybdenum Base:</i> As cast	98c, 129, 130, 131 132a	general structure chemical polish prior to etching
<i>Nickel Base:</i> Pure Ni and high Ni alloys	133, 134, 47, 135, 136, 25, 108, 31c 137	general structure 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 74e, 25, 150	general structure 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 25, 94 107, 111, 13a 133 154 19b, 155, 156 22a 157 107  154 18 213	general structure grain size reveals microstructural inhomogeneity grain boundary sulfidation fine precipitation structure differential matrix and nonmetallic staining for passive alloys (for example, UNS Alloy N06625) specific for UNS Alloy N10004 submicroscopic structure in aged super-alloys particu- larly for electron microscopy. Stains the matrix when $\gamma'$ precipitates are present $\gamma'$ banding pre-etch activation for passive specimens colors carbide and $\gamma'$

**TABLE 1** *Continued*

Metal	Etchants	Uses
<i>Niobium (Columbium) Base:</i>	129, 66, 158, 159, 160, 161, 162, 163 164, 129, 160	general structure grain boundaries
<i>Osmium Base:</i>	165a 165a	general structure etch-polishing for viewing grains with polarized light
<i>Palladium Base:</i>		
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
<i>Platinum Base:</i>		
Pure Pt	64a, 73a 167	general structure electrolytic polish and etch
Pt Alloys	64b, 73a 167	general structure electrolytic polish and etch
>90 % noble metals	61	general structure
<90 % noble metals	65	general structure
Pt-10 % Rh	168	general structure
<i>Plutonium Base:</i>	169	general structure
<i>Rhenium Base:</i>	13b,98c, 132b, 170a	general structure
<i>Rhodium Base:</i>	171	general structure
<i>Ruthenium Base:</i>	73b 73b	general structure etch-polishing for viewing grains with polarized light
<i>Silver Base:</i>		
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
<i>Tantalum Base:</i>		
Pure Ta	177	general structure
Ta alloys	159, 66, 178, 163, 161, 179 164 158	general structure grain boundaries and inclusions grain boundaries—retains carbide precipitate
<i>Thorium Base:</i>		
Pure Th	185	general structure
Th alloys	185	general structure
<i>Tin Base:</i>		
Pure Sn	74d, 180, 151 181	general structure grain boundaries
Sn-Cd	74d	general structure
Sn-Fe	74d, 177a	general structure
Sn-Pb	182, 183, 74b 116	general structure darkens Pb in Sn-Pb eutectic
Sn coatings (on steel)	183	general structure
Babbitts	184	general structure
Sn-Sb-Cu	74b	general structure
<i>Titanium Base:</i>		
Pure Ti	186, 187, 67, 68, 69, 217 188 72	general structure removes stain 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 11, 1c 72, 192, 178 170a 188	general structure reveals alpha case chemical polish and etch outlines and darkens hydrides in some alloys removes stain

**TABLE 1** *Continued*

Metal	Etchants	Uses
<i>Tungsten Base:</i>		
Pure W	98c, 131	general structure
As cast	132a	chemical polish prior to etching
W-Th	209	general structure
<i>Uranium Base:</i>		
Pure U	67, 69, 195, 196	general structure
U + Zr	68	general structure
U beryllides	170a	general structure
U alloys	67, 69, 195, 96 207	general structure carbides
<i>Vanadium Base:</i>		
Pure V	170b, 165b 197, 198	general structure grain boundaries
V alloys	199, 198	general structure
<i>Zinc Base:</i>		
Pure Zn	200a	general structure
Zn-Co	177	general structure
Zn-Cu	201 203	general structure distinguishes gamma ( $\gamma$ ) and epsilon ( $\epsilon$ )
Zn-Fe	74a	structure of galvanized sheet
Die castings	202	general structure
<i>Zirconium Base:</i>		
	66, 67, 204, 68, 69, 205 206 71 72	general structure electrolytic polish and etch grain structure under polarized light 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 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 <sub>3</sub> 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 <sub>3</sub> PO <sub>4</sub>  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 <sub>4</sub> 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 <sub>3</sub> 75 mL water	Immerse 40 s at 70°C (160°F). Rinse in cold water.
7	10–20 mL H <sub>2</sub> SO <sub>4</sub> 80 mL water	Immerse 30 s at 70°C (160°F). Rinse in cold water.
8	10 mL H <sub>3</sub> PO <sub>4</sub> 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 <sub>3</sub> 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 <sub>3</sub> 90 mL methanol (95 %)	Immerse few seconds to a minute.
15	15 mL HNO <sub>3</sub> 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 <sub>3</sub> 100 mL water	Electrolytic at 6 V for few seconds.
18	2–10 g CrO <sub>3</sub> 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 $\text{KMnO}_4$	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: $\text{KMnO}_4$ is an aggressive staining agent.
20	5 mL $\text{H}_2\text{O}_2$ (30 %) 100 mL HCl	Use a certified and tested hood. <i>Mix fresh.</i> Immerse polished face up for few seconds.
21	1 g $\text{CrO}_3$ 140 mL HCl	Use a certified and tested hood. To mix, add the HCl to $\text{CrO}_3$ . Electrolytic at 3 V for 2–10 s.
22	100 mL HCl 0.5 mL $\text{H}_2\text{O}_2$ (30 %)	Use a certified and tested hood. Do not store. (a) Immerse or swab $\frac{1}{2}$ –3 min. Add $\text{H}_2\text{O}_2$ 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 $\text{HNO}_3$ 200 mL HCl 65 g $\text{FeCl}_3$	Use a certified and tested hood. Immerse few seconds.
25	10 g $\text{CuSO}_4$ 50 mL HCl 50 mL water	Immerse or swab 5–60 s. Made more active by adding few drops of $\text{H}_2\text{SO}_4$ just before use.
26	5 g $\text{FeCl}_3$ 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 $\text{H}_2\text{O}_2$ (3 %) 50 mL $\text{NH}_4\text{OH}$ 30 mL water	Dissolve KOH in water, then slowly add $\text{NH}_4\text{OH}$ to solution. Add 3 % $\text{H}_2\text{O}_2$ last. Use fresh—immerse few seconds to a minute.
28	1 g $\text{FeNO}_3$ 100 mL water	Swab or immerse few seconds to a minute.
29	1 g $\text{K}_2\text{Cr}_2\text{O}_7$ 4 mL $\text{H}_2\text{SO}_4$ 50 mL water	Use a certified and tested hood. Add 2 drops of HCl just before using. Swab few seconds to a minute.
30	25 mL $\text{NH}_4\text{OH}$ 25 mL water 50 mL $\text{H}_2\text{O}_2$ (3 %)	Mix $\text{NH}_4\text{OH}$ and water before adding $\text{H}_2\text{O}_2$ . 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 $\text{CrO}_3$ 100 mL water	Use a certified and tested hood. Saturated solution. Immerse or swab 5–30 s.
33	10 g $\text{CrO}_3$ 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 $\text{FeCl}_3$ 50 mL HCl 100 mL water	(a) Immerse or swab few seconds to few minutes. Small additions of $\text{HNO}_3$ activate solution and minimize pitting. (b) Immerse or swab few seconds at a time. Repeat as necessary.
35	20 g $\text{FeCl}_3$ 5 mL HCl 1 g $\text{CrO}_3$ 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 $\text{FeCl}_3$ 25 mL HCl 100 mL water	Immerse or swab few seconds at a time until desired results are obtained.