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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.*

## 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 read and understood concerning all of the hazards and safety precautions to be observed. Users should be aware of the type

[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.

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 conform to specifications of the Committee on Analytical

[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.

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<br>Gilding metal<br>Cartridge brass<br>Free-cutting brass | *8b* | general structure |
| 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 | 76, 74a, 77, 78, 79 | general structure |
| and | 74a, 77, 31a, 223 | ferrite grain boundaries |
| Fe + <1C + <4 % additions | 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) | *13b*, 89, 87, 88, *83a*, 80, 94, 95, 91, 101, 212, 221, 226 | general structure |
| | *13a, 102*, 31c, 48c, 213 | carbides and sensitization |
| and | *48, 96, 97*, 98 | stains sigma phase |
| Fe + 16–25 Cr + 3–6 Ni + 5–10 Mn (200 series) | 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 | $\gamma'$ 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 | 118, 119, 74a, 1d, 128, 124, 126, | general structure |
| and | 127, 121, 122 | |
| Mg-Zn-Th-Zr | 120, 121 | phase identification |
| Mg-Th-Zr | 118, 119, 74a, 1d, 124, 127, 121, 122 | general structure |
| and | | |
| Mg-Rare Earth-Zr | 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, | general structure |
| | *48b*, 94, *108*, 34 | |
| 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<br>200 mL water | (a) Swab with cotton for 15 s.<br>(b) Alternately immerse and polish several minutes.<br>(c) Immerse 3–5 s.<br>(d) Immerse 10–120 s. |
| 2 | 3 mL HF<br>100 mL water | (a) Swab 10 s to reveal general structure.<br>(b) Immerse 15 min, wash 10 min in water to form film with hatching which varies with grain orientation. |
| 3 | 2 mL HF<br>3 mL HCl<br>5 mL $HNO_3$<br>190 mL water | (a) Immerse 10–20 s Wash in stream of warm water. Reveals general structure.<br>(b) Dilute with 4 parts water-colors constituents—mix fresh. |
| 4 | 24 mL $H_3$ $PO_4$<br>50 mL Carbitol (diethylene glycol monoethyl ether)<br>4 g boric acid<br>2 g oxalic acid<br>10 mL HF<br>32 mL water | Electrolytic: Use carbon cathode raising d-c voltage from 0–30 V in 30 s. Total etching time 3 min<br>with agitation. Wash and cool. Repeat if necessary. |
| 5 | 5 g $HBF_4$<br>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 $HNO_3$<br>75 mL water | Immerse 40 s at 70°C (160°F). Rinse in cold water. |
| 7 | 10–20 mL $H_2$ $SO_4$<br>80 mL water | Immerse 30 s at 70°C (160°F). Rinse in cold water. |
| 8 | 10 mL $H_3$ $PO_4$<br>90 mL water | (a) Immerse 1–3 min at 50°C (120°F).<br>(b) Electrolytic at 1–8 V for 5–10 s. |
| 9 | 3–4 g sulfamic acid<br>5 drops HF<br>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<br>90 mL methanol (90 %) | Immerse 10–30 s. |
| 11 | 2 mL HF<br>100 mL water | Immerse or swab few seconds to a minute. |
| 12 | 20 mL $HNO_3$<br>60 mL HCl | Use a certified and tested hood. Do not store. Immerse or swab 5–60 s. |
| 13 | 10 g oxalic acid<br>100 mL water | Electrolytic at 6 V:<br>(a) 10–15 s.<br>(b) 1 min.<br>(c) 2–3 s.<br>Use stainless steel cathode and platinum or Nichrome connection to specimen. |
| 14 | 10 mL $HNO_3$<br>90 mL methanol (95 %) | Immerse few seconds to a minute. |
| 15 | 15 mL $HNO_3$<br>15 mL acetic acid<br>60 mL HCl<br>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<br>100 mL water | Electrolytic at 3 V for 2–10 s. |
| 17 | 5 mL HCl<br>10 g $FeCl_3$<br>100 mL water | Electrolytic at 6 V for few seconds. |
| 18 | 2–10 g $CrO_3$<br>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<br>8 g NaOH<br>100 mL water<br>B<br>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 %)<br>100 mL HCl | Use a certified and tested hood. *Mix fresh.* Immerse polished face up for few seconds. |
| 21 | 1 g $CrO_3$<br>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<br>0.5 mL $H_2O_2$ (30 %) | Use a certified and tested hood. Do not store.<br>(a) Immerse or swab ½ –3 min. Add $H_2O_2$ dropwise to maintain action.<br>(b) Electrolytic, 4 V, 3–5 s. |
| 23 | 5 mL HCl<br>95 mL ethanol (95 %) or methanol (95 %) | Electrolytic at 6 V for 10–20 s. |
| 24 | 5 mL $HNO_3$<br>200 mL HCl<br>65 g $FeCl_3$ | Use a certified and tested hood. Immerse few seconds. |
| 25 | 10 g $CuSO_4$<br>50 mL HCl<br>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 $FeCl_3$<br>10 mL HCl<br>50 mL glycerol<br>30 mL water | Swab 16–60 s. Activity may be decreased by substituting glycerol for water. |
| 27 | 1 g KOH<br>20 mL $H_2O_2$ (3 %)<br>50 mL $NH_4OH$<br>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 $FeNO_3$<br>100 mL water | Swab or immerse few seconds to a minute. |
| 29 | 1 g $K_2Cr_2O_7$<br>4 mL $H_2SO_4$<br>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 $NH_4OH$<br>25 mL water<br>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<br>100 mL water | (a) Swab or immerse to 5 s.<br>(b) Immerse to 2 min to darken matrix to reveal carbides and phosphides.<br>(c) Electrolytic at 6 V for few seconds to a minute.<br>(d) Immerse 3–60 s. Can be heated to increase activity. |
| 32 | 60 g $CrO_3$<br>100 mL water | Use a certified and tested hood. Saturated solution.<br>Immerse or swab 5–30 s. |
| 33 | 10 g $CrO_3$<br>2–4 drops HCl<br>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 $FeCl_3$<br>50 mL HCl<br>100 mL water | (a) Immerse or swab few seconds to few minutes. Small additions of $HNO_3$ activate solution and minimize pitting.<br>(b) Immerse or swab few seconds at a time. Repeat as necessary. |
| 35 | 20 g $FeCl_3$<br>5 mL HCl<br>1 g $CrO_3$<br>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 $FeCl_3$<br>25 mL HCl<br>100 mL water | Immerse or swab few seconds at a time until desired results are obtained. |