**Designation: E407 – 07 (Reapproved 2015)$^{\varepsilon 1}$**

# 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.*

$\varepsilon^1$ 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

---

[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.

Current edition approved June 1, 2015. Published September 2015. Originally approved in 1970. Last previous edition approved in 2007 as E407–07$^{\varepsilon 1}$. DOI: 10.1520/E0407-07R15E01.

[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 | 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 % | 80, *31c*, 89, 99, 100, 91 | general structure |
| other elements (controlled trans- | *31c* | carbides |
| formation, precipitation harden- | 86 | chemical polish-etch |
| ing, stainless maraging alloys) | 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 | 103, 104, 98 | delineates sigma phase and |
| Mn (200 series) | 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* | $\gamma$' 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 $\gamma$' precipitates are present |
| | 154 | $\gamma$' banding |
| | *18* | pre-etch activation for passive specimens |
| | 213 | colors carbide and $\gamma$' |

**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 remains 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 $H_3PO_4$<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 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 $H_2SO_4$<br>80 mL water | Immerse 30 s at 70°C (160°F). Rinse in cold water. |
| 8 | 10 mL $H_3PO_4$<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. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 37 | 1 g $FeCl_3$<br>10 mL HCl<br>100 mL water | Immerse or swab few seconds at a time until desired results are obtained |
| 38 | 8 g $FeCl_3$<br>25 mL HCl<br>100 mL water | Swab 5–30 s. |
| 39 | 5 g $FeCl_3$<br>10 mL HCl<br>1 g $CuCl_2$<br>0.1 g $SnCl_2$<br>100 mL water | Immerse or swab few seconds at a time until desired results are obtained. |
| 40 | 5 g $FeCl_3$<br>16 mL HCl<br>60 mL ethanol (95 %) or methanol (95 %) | Immerse or swab few seconds to few minutes. |
| 41 | 2 g $K_2 Cr_2 O_7$<br>8 mL $H_2 SO_4$<br>4 drops HCl<br>100 mL water | Use a certified and tested hood. Add the HCl just before using. Immerse 3–60 s. |
| 42 | 10 g cupric ammonium chloride<br>100 mL water<br>$NH_4$ OH | Add $NH_4$ OH to solution until neutral or slightly alkaline. Immerse 5–60 s. |
| 43 | 20 mL $NH_4$ OH<br>1 g ammonium persulfate<br>60 mL water | Immerse 5–30 s. |
| 44 | 50 mL $NH_4$ OH<br>20–50 mL $H_2 O_2$ (3 %)<br>0–50 mL water | Use fresh. Peroxide content varies directly with copper content of alloy to be etched. Immerse or swab to 1 min. Film on etched aluminum bronze removed by No. 82. |
| 45 | 1 g $CrO_3$<br>100 mL water | Use a certified and tested hood. Electrolytic at 6 V for 3–6 s. Use aluminum cathode. |
| 46 | 15 mL $NH_4$ OH<br>15 mL $H_2 O_2$ (3 %)<br>15 mL water<br>4 pellets NaOH | When mixing, add NaOH pellets last. For best results use before pellets have dissolved. |
| 47 | 5 g NaCN or KCN<br>5 g $(NH_4)_2 S_2 O_2$<br>100 mL water | Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. |
| 48 | 10 g NaCN<br>100 mL water | Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Electrolytic at 6 V:<br>(a) 5 s for sigma.<br>(b) 30 s for ferrite and general structure.<br>(c) to 5 min for carbides. |
| 49 | 3 g $FeSO_4$<br>0.4 g NaOH<br>10 mL $H_2 SO_4$<br>190 mL water | Electrolytic at 8–10 V (0.1 A) for 5–15 s. |
| 50 | 5 mL acetic acid<br>10 mL $HNO_3$<br>85 mL water | Use a certified and tested hood. Do not store. Electrolytic at 1.5 V for 20 to 60 s. Use platinum wires. |
| 51 | 2 g $FeCl_3$<br>5 mL HCl<br>30 mL water<br>60 mL ethanol or methanol | Immerse few minutes. |
| 52 | 1 g sodium dichromate<br>1 g NaCl<br>4 mL $H_2 SO_4$<br>250 mL water | Swab few seconds. |
| 53 | 1–5 mL $NH_4$ OH<br>100 mL water | Immerse 5–60 s. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 54 | 1 g ammonium acetate<br>3 g sodium thiosulfate<br>7 mL $NH_4OH$<br>1300 mL water | Electrolytic at 0.3 A/cm$^2$ for 5–30 s. |
| 55 | 1 mL $H_2SO_4$<br>15 mL $HNO_3$<br>10 mL acetic acid<br>5 mL $H_3PO_4$<br>20 mL lactic acid | Use a certified and tested hood. Swab gently 10–15 s. Rinse with methanol and blow dry. Helps to chemically polish. If final etch is too mild, follow with No. 98. Do not store. |
| 56 | 30 mL $HNO_3$<br>10 mL $H_3PO_4$<br>20 mL acetic acid<br>10 mL lactic acid | Use a certified and tested hood. Swab gently 5–15 s. Rinse with ethanol or methanol and blow dry. Do not store. |
| 57 | 75 mL acetic acid<br>25 mL $H_2O_2$ (30 %) | Use a certified and tested hood. Immerse 6–15 s. Do not store. |
| 58 | 25 mL HF<br>25 mL $HNO_3$<br>5 mL water | Swab 3–20 s. |
| 59 | 2 g $AgNO_3$<br>40 mL water<br>40 mL HF<br>20 mL $HNO_3$ | Mix $AgNO_3$ and water, then add HF and $HNO_3$. Swab ½ –2 min. |
| 60 | 25 mL $HNO_3$<br>15 mL acetic acid<br>15 mL HF<br>5–7 drops bromine | Use a certified and tested hood. Do not store. Let stand ½ h before using. Swab 3–20 s. |
| 61 | 60 mL HCl<br>40 mL $HNO_3$ | Use a certified and tested hood. Immerse few seconds to a minute. |
| 62 | 1–5 g $CrO_3$<br>100 mL HCl | Use a certified and tested hood. Vary composition of reagent and aging of reagent after mixing to suit alloy. Swab or immerse few seconds to a minute. |
| 63 | 0.1 g $CrO_3$<br>10 mL $HNO_3$<br>100 mL HCl | Use a certified and tested hood. Swab few seconds to a minute. |
| 64 | 5 mL $HNO_3$<br>25 mL HCl<br>30 mL water | (a) Immerse 1–5 min.<br>(b) Use hot. Will form chloride film on gold alloys if much silver is present. Ammonia will remove film. |
| 65 | A<br>10 g ammonium persulfate<br>100 mL water<br>B<br>10 g KCN<br>100 mL water | Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Mix 1 + 1 mixture of Solutions A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 in. dia. mount.) Immerse ½ – 2 min. |
| 66 | 30 mL HF<br>15 mL $HNO_3$<br>30 mL HCl | Use a certified and tested hood. Swab 3–10 s or immerse to 2 min. |
| 67 | 10 mL perchloric acid<br>10 mL 2-butoxyethanol<br>70 mL ethanol (95 %)<br>10 mL water | Use in wash down/perchloric rated fume hood. Precaution—Keep cool when mixing and use. Electrolytic at 30–65 V for 10–60 s. |
| 68 | 3 mL perchloric acid<br>35 mL 2-butoxyethanol<br>60 mL methanol (absolute) | Use in wash down/perchloric rated fume hood. Precaution—Keep cool when mixing and use. Electrolytic at 60–150 V for 5–30 s. |
| 69 | 5 mL perchloric acid<br>80 mL acetic acid | Use in wash down/perchloric rated fume hood. Precaution—Keep cool when mixing and use. Electrolytic at 20–60 V for 1–5 min. Do not store. |
| 70 | 5 mL HF<br>2 mL $AgNO_3$ (5 %)<br>200 mL water | Swab for 5–60 s. |
| 71 | 5 mL HF<br>95 mL water | Add 5–10 drops of this solution on the final polishing wheel which has been charged with the polishing solution. The specimen is polished on this wheel until the surface turns black. Distilled water is then slowly added to the wheel and polishing continued until the surface is bright. At this time the specimen should be ready for examination via polarized light. Note—Use inert substance between cloth and wheel to prevent attack of the wheel. Wear appropriate gloves. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 72 | 10 mL HF<br>45 mL $HNO_3$<br>45 mL water | Swab for 5–20 s. |
| 73 | 20 mL HCl<br>25 g NaCl<br>65 mL water | Electrolytic etch—use carbon cathode and platinum wire connection to specimen.<br>(a) 6 V ac for 1 min.<br>(b) 5 V–20 V ac for 1–2 min.<br>(c) 20 V ac for 1–2 min.<br>For etch-polishing, use shorter times. After etching, water rinse, alcohol rinse, and dry. |
| 74 | 1–5 mL $HNO_3$<br>100 mL ethanol (95 %) or methanol (95 %) | Etching rate is increased, sensitivity decreased with increased percentage of $HNO_3$.<br>(a) Immerse few seconds to a minute.<br>(b) Immerse 5–40 s in 5 % $HNO_3$ solution. To remove stain, immerse 25 s in 10 % HCl-methanol solution.<br>(c) For Inconels and Nimonics, use 5 mL $HNO_3$ solution—electrolytic at 5–10 V for 5–20 s.<br>(d) Swab or immerse several minutes.<br>(e) Swab 5–60 s. $HNO_3$ may be increased to 30 mL in methanol only depending on alloy. (Ethanol is unstable with over 5 % $HNO_3$.) Do not store. |
| 75 | 5 g picric acid<br>8 g $CuCl_2$<br>20 mL HCl<br>200 mL ethanol (95 %) or methanol (95 %) | Immerse 1–2 s at a time and immediately rinse with methanol. Repeat as often as necessary. (Long immersion times will result in copper deposition on surface.) |
| 76 | 4 g picric acid<br>100 mL ethanol (95 %) or methanol (95 %) | Composition given will saturate with picric acid. Immerse few seconds to a minute or more. Adding a wetting agent such as zepherin chloride will increase response. |
| 77 | 10 g picric acid<br>5 drops HCl<br>100 mL ethanol (95 %) or methanol (95 %) | Composition given will saturate the solution with picric acid. Immerse few seconds to a minute or more. |
| 78 | 10 g potassium metabisulfite<br>100 mL water | Immerse 1–15 s. Better results are sometimes obtained by first etching lightly with No. 76 or 74. |
| 79 | 40 mL HCl<br>5 g $CuCl_2$<br>30 mL water<br>25 mL ethanol (95 %) or methanol (95 %) | Swab few seconds to a minute. |
| 80 | 5 mL HCl<br>1 g picric acid<br>100 mL ethanol (95 %) or methanol (95 %) | Immerse or swab few seconds to 15 min. Reaction may be accelerated by adding a few drops of 3 % $H_2O_2$. Optional (for prior austenite grain boundaries)—temper specimen at 600–900°F prior to preparation. |
| 81 | 2 g picric acid<br>1 g sodium tridecylbenzene sulfonate.<br>100 mL water | Composition given will saturate the solution with picric acid.<br>(a) Immerse few seconds to a minute.<br>(b) Immerse to 15 min with occasional swabbing for heavy grain boundary attack. |
| 82 | 5 g $FeCl_3$<br>5 drops HCl<br>100 mL water | Immerse 5–10 s. |
| 83 | 10 g $CrO_3$<br>100 mL water | Use a certified and tested hood—(a) Electrolytic at 6 V for 5–60 s. Attacks carbides.<br>(b) Electrolytic at 6 V for 3–5 s. |
| 84 | 10 mL $H_2SO_4$<br>10 mL $HNO_3$<br>80 mL water | Use a certified and tested hood. Precaution—Add $H_2SO_4$ slowly to water and cool, then add $HNO_3$. Immerse 30 s. Swab in running water. Repeat three times and repolish lightly. |
| 85 | 2 g picric acid<br>25 g NaOH<br>100 mL water | Use a certified and tested hood. Immerse in boiling solution for 5 min. Precaution—Do not boil dry—anhydrous picric acid is unstable and highly explosive. Alternative: Electrolytic at 6 V for 40 s (room temperature). Use stainless steel cathode. |
| 86 | 3 g oxalic acid<br>4 mL $H_2O_2$ (30 %)<br>100 mL water | Use a certified and tested hood. Solution should be freshly prepared. Immerse 15–25 min when specimens or parts cannot be given usual metallographic polish. Multiple etching may be required. |
| 87 | 10 mL $HNO_3$<br>20–50 mL HCl<br>30 mL glycerol | Use a certified and tested hood—Can give off nitrogen dioxide gas. Precaution—Mix HCl and glycerol thoroughly before adding $HNO_3$. Do not store. Properly discard before solution attains a dark orange color. Immerse or swab few seconds to few minutes. Higher percentage of HCl minimizes pitting. A hot water rinse just prior to etching may be used to activate the reaction. Sometimes a few passes on the final polishing wheel is also necessary to remove a passive surface. |
| 88 | 10 mL $HNO_3$<br>20 mL HCl<br>30 mL water | Use a certified and tested hood—Can give off nitrogen dioxide gas. Precaution—Properly discard before solution attains a dark orange color. Immerse few seconds to a minute. Much stronger reaction than No. 87. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 89 | 10 mL $HNO_3$<br>10 mL acetic acid<br>15 mL HCl<br>2–5 drops glycerol | Use a certified and tested hood. Do not store. Immerse or swab few seconds to few minutes. |
| 90 | 10 mL $HNO_3$<br>20 mL HF<br>20–40 mL glycerol | Use a certified and tested hood—Immerse 2–10 s. Do not store. Properly discard after use. Solution decomposes on standing. |
| 91 | 5 mL $HNO_3$<br>5 mL HCl<br>1 g picric acid<br>200 mL ethanol (95 %) or methanol (95 %) | This etchant is equivalent to a 1 + 1 mixture of No. 80 and No. 74 (5 % $HNO_3$). Swab for 30 s or longer. |
| 92 | 10 mL HCl<br>100 mL ethanol (95 %) or methanol (95 %) | Immerse 5–30 min or electrolytic at 6 V for 3–5 s. |
| 93 | concentrated $HNO_3$ | Use a certified and tested hood. Electrolytic at 0.2 A/cm$^2$ for few seconds. |
| 94 | 2 g $CuCl_2$<br>40 mL HCl<br>40–80 mL ethanol (95 %) or methanol (95 %) | Submerged swabbing for few seconds to several minutes. Attacks ferrite more readily than austenite. |
| 95 | 2 g $CuCl_2$<br>40 mL HCl<br>40–80 mL ethanol (95 %) or methanol (95 %)<br>40 mL water | Immerse or swab few seconds to few minutes. |
| 96 | 85 g NaOH<br>50 mL water | Electrolytic at 6 V for 5–10 s. |
| 97 | 45 g KOH<br>60 mL water | Composition of solution is approximately 10 N. Electrolytic at 2.5 V for few seconds. Stains sigma and chi yellow to red brown, ferrite gray to blue gray, carbides barely touched, austenite not touched. |
| 98 | 10 g $K_3Fe(CN)_6$ † <br>10 g KOH or NaOH<br>100 mL water | Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Use fresh.<br>(a) Immerse or swab 15–60 s. Stains carbides and sigma. (To differentiate, No. 31 electrolytic at 4 V will attack sigma, but not carbides. If pitting occurs, reduce voltage.)<br>(b) Immerse in fresh, hot solution 2–20 min. Stains carbides dark, ferrite yellow, sigma blue. Austenite turns brown on overetching.<br>(c) Swab 5–60 s. (Immersion will produce a stain etch).<br>Follow with water rinse, alcohol rinse, dry. |
| 99 | 25 mL HCl<br>3 g ammonium bifluoride<br>125 mL water<br>few grains potassium metabisulfite | *Mix fresh*. (For stock solution, mix first three items. Add potassium metabisulfite just before use.) Immerse few seconds to a few minutes. |
| 100 | 10 g $FeCl_3$<br>90 mL water | Immerse few seconds. |
| 101 | 2 g $CrO_3$<br>20 mL HCl<br>80 mL water | Use a certified and tested hood-Immerse 5–60 s. ($CrO_3$ may be increased up to 20 g for difficult alloys. Staining and pitting increase as $CrO_3$ increased.) |
| 102 | concentrated $NH_4OH$ | Use a certified and tested hood. Electrolytic at 6 V for 30–60 s. Attacks carbides only. |
| 103 | 20 mL $HNO_3$<br>4 mL HCl<br>20 mL methanol (99 %) | Use a certified and tested hood. Immerse 10–60 s. |
| 104 | 5 mL $HNO_3$<br>45 mL HCl<br>50 mL water | Use a certified and tested hood. Immerse 10 min or longer. |
| 105 | 5 mL $H_2SO_4$<br>3 mL $HNO_3$<br>90 mL HCl | Use a certified and tested hood. Precaution—add $H_2SO_4$ slowly to HCl with stirring, cool; then add $HNO_3$. Properly discard when dark orange color. Swab 10–30 s. |
| 106 | 7 mL $HNO_3$<br>25 mL HCl<br>10 mL methanol (99 %) | Use a certified and tested hood—Use fresh to avoid pitting. Immerse or swab 10–60 s. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 107 | 10 mL $H_3PO_4$<br>50 mL $H_2SO_4$<br>40 mL $HNO_3$ | Use a certified and tested hood. Precaution—Mix $H_3PO_4$ and $HNO_3$ thoroughly, then add $H_2SO_4$ slowly with stirring. Use fresh, but allow to cool. Electrolytic at 6 V for few seconds. Brown discoloration will form at edges of specimen. To slow reaction, add water (to 100 mL) very carefully with stirring. Attacks bakelite mounts. |
| 108 | 3–10 mL $H_2SO_4$<br>100 mL water | Electrolytic at 6 V for 5–10 s. Tends to pit with longer times. |
| 109 | 50 mL HCl<br>25 mL $HNO_3$<br>1 g $CuCl_2$<br>150 mL water | Make fresh but allow to stand 30 min to avoid plating out copper. Immerse few seconds to a few minutes. |
| 110 | 10 mL HCl<br>5 mL $HNO_3$<br>85 mL ethanol (95 %) or methanol (95 %) | Immerse to several minutes until deeply etched. Follow with light repolish. |
| 111 | 5 mL $H_2SO_4$<br>8 g $CrO_3$<br>85 mL $H_3PO_4$ | Use a certified and tested hood. Electrolytic at 10 V (0.2 A/cm$^2$) for 5–30 s. Reveals Ti- and Cb-rich areas at a faster rate than grain boundaries. |
| 112 | 60 mL acetic acid<br>30 mL $H_2O_2$ (30 %) | Use a certified and tested hood. Immerse 8–15 s. |
| 113 | 15 mL acetic acid<br>15 mL $HNO_3$<br>60 mL glycerol | Use a certified and tested hood. Do not store. Use fresh solution at 80°C (176°F). |
| 114 | 15 mL acetic acid<br>20 mL $HNO_3$<br>80 mL water | Use a certified and tested hood. Use fresh solution at 40–42°C (104–108°F). Immerse 4–30 min depending on depth of worked metal layer. Clean with cotton in running water. Do not store. |
| 115 | 100 mL acetic acid<br>10 mL $H_2O_2$ (30 %) | Use a certified and tested hood. Immerse 10–30 min depending on depth of worked metal layer. Clean in $HNO_3$ if necessary. |
| 116 | 5–10 g $AgNO_3$ 90 mL water | Swab. |
| 117 | 10 mL HCl<br>90 mL water | (a) Immerse for ½ –5 min. Follow with electrolytic etch at low current density in same solution. If specimen has considerable surface flow, immerse in concentrated HCl for a few seconds, then follow above procedure.<br>(b) Immerse for ½ –2 min. |
| 118 | 1 mL $HNO_3$<br>75 mL diethylene glycol<br>25 mL water | Swab 3–5 s for F and T6, 1–2 min for T4 and O temper. |
| 119 | 1 mL $HNO_3$<br>20 mL acetic acid<br>60 mL diethylene glycol<br>20 mL water | Use a certified and tested hood. Swab 1–3 s for F and T6, 10 s for T4 and O temper. Do not store. |
| 120 | 10 mL HF<br>90 mL water | Immerse with gentle agitation 3–30 s. |
| 121 | 0.7 mL $H_3PO_4$<br>4 g picric acid<br>100 mL ethanol (95 %) or methanol (95 %) | Composition critical.<br>(a) Immerse with gentle agitation 10–30 s.<br>(b) To increase staining immerse and withdraw with a meniscus layer. Lightly apply etchant over surface until dark stain develops. |
| 122 | 2 g oxalic acid<br>100 mL water | Swab. |
| 123 | 60 mL $H_3PO_4$<br>100 mL ethanol (95 %) | Electrolytic: Use stainless steel cathode. Space electrodes 2 cm apart. Start at 3 V dc. After 30 s maintain at 1½ V. |
| 124 | 5 mL acetic acid<br>10 mL water<br>6 g picric acid<br>100 mL ethanol (95 %) or methanol (95 %) | Use a certified and tested hood. Immerse with gentle agitation 10–60 s. |
| 125 | 10 mL acetic acid<br>6 g picric acid<br>100 mL ethanol (95 %) or methanol (95 %) | Use a certified and tested hood. Immerse with gentle agitation 15–30 s. |
| 126 | 30 mL acetic acid<br>15 mL water<br>6 g picric acid<br>100 mL ethanol (95 %) or methanol (95 %) | Use a certified and tested hood. Immerse with gentle agitation 1–30 s. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 127 | 20 mL acetic acid<br>20 mL water<br>3 g picric acid<br>50 mL ethanol (95 %) or methanol (95 %) | Use a certified and tested hood. Immerse with gentle agitation 5–30 s. |
| 128 | 8 mL HF<br>5 mL $HNO_3$<br>200 mL water | Use a certified and tested hood. Immerse with gentle agitation 5–15 s. |
| 129 | 10 mL HF<br>30 mL $HNO_3$<br>60 mL lactic acid | Swab 10–20 s. Vary HF to increase or decrease activity. |
| 130 | 25 mL HCl<br>75 mL methanol | Caution—Keep below 24°C (75°F). Electrolytic at 30 V for 30 s. |
| 131 | 5 mL $H_2SO_4$<br>1 mL HF<br>100 mL methanol (95 %) | Use a certified and tested hood. Electrolytic at 50–60 V for 10–20 s. |
| 132 | 5 mL HF<br>10 mL $HNO_3$<br>50 mL lactic acid | Use fresh.<br>(a) Swab with heavy pressure for 5–10 s. Water rinse, alcohol rinse, dry, then etch with No. 98c.<br>(b) Swab for 5–30 s. |
| 133 | 50 mL $HNO_3$<br>50 mL acetic acid | Use a certified and tested hood. Do not store. *Mix fresh.* Immerse or swab 5 to 30 s. Will chemically polish with longer times. Sulfidized grain boundaries etched before normal grain boundaries. Do not store. |
| 134 | 70 mL $H_3PO_4$<br>30 mL water | Electrolytic 5–10 V for 5–60 s. (Polishes at high currents.) |
| 135 | 80 mL $HNO_3$<br>3 mL HF | Use a certified and tested hood. Warm specimen in boiling water prior to immersion for 10 to 120 s. |
| 136 | 20 mL $H_3PO_4$<br>80 mL water | Electrolytic at 10–20 V for 10–15 s. |
| 137 | 10 g $NaNO_3$<br>100 mL water | Electrolytic, 0.2 A/cm$^2$, 1 min. |
| 138 | 5 g $FeCl_3$<br>2 mL HCl<br>100 mL ethanol (95 %) or methanol (95 %) | Swab 10–60 s. |
| 139 | 5 g KCN<br>100 mL water<br>0.5 mL $H_2O_2$ (3 %) | Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Immerse 10–100 s. |
| 140 | 50 mL acetic acid<br>50 mL $HNO_3$<br>50 mL acetone | Use a certified and tested hood. Do not store. Decomposes with possible explosion on standing. Immerse 10–30 s. |
| 141 | 3 g $NH_4Cl$<br>3 g $CrO_3$<br>10 mL $HNO_3$<br>90 mL water | Use a certified and tested hood-Swab 5–30 s. Do not store. |
| 142 | 5 mL HF<br>10 mL glycerol<br>85 mL water | Electrolytic at 2–3 V for 2–10 s. |
| 144 | A<br>10 g sodium thiosulfate<br>100 mL water<br>B<br>10 mL HCl<br>90 mL water | Electrolytic in Solution A: specimen is cathode, 10 V, 5–10 s. Then electrolytic in Solution B: specimen is anode, 10 V, 5–10 s. |
| 145 | 2 mL $H_2SO_4$<br>100 mL water | Electrolytic at 3–10 V for 5–15 s. Use platinum wires. $H_2SO_4$ may be increased to 20 mL for deeper attack. |
| 146 | 10 mL HF<br>100 mL $HNO_3$ | Immerse 30 s–3 min. |
| 147 | 20 mL $HNO_3$<br>80 mL HCl | Immerse 5–30 s. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 148 | 5 mL $HNO_3$<br>100 mL water | Immerse 10–30 s. |
| 149 | 50 mL HCl<br>2 mL $H_2O_2$ (30 %)<br>50 mL water | Immerse 10–30 s. Do not store. |
| 150 | 60 mL HCl<br>20 mL $HNO_3$<br>40 mL glycerol | Use a certified and tested hood. Do not store. Swab few seconds to a minute. Properly discard when solution turns dark yellow. |
| 151 | 10 mL HF<br>25 mL $HNO_3$<br>150 mL water | Swab 5–30 s. |
| 152 | 85 mL $NH_4OH$<br>15 mL $H_2O_2$ (30 %) | Use a certified and tested hood. Immerse 5–15 s. Do not store—Decomposes. |
| 153 | 10 mL $HNO_3$<br>50 mL HCl<br>60 mL glycerol | Use a certified and tested hood. Do not store. Add $HNO_3$ last. Properly discard when dark yellow. Immerse 10–60 s. Preheating specimen in boiling water hastens reaction. |
| 154 | 50 mL HCl<br>50 mL ethanol (95 %) or methanol (95 %) | Immerse 10–100 s. |
| 155 | 3 mL selenic acid<br>10 mL HCl<br>100 mL ethanol (95 %) or methanol (95 %) | Use a certified and tested hood. Immerse 1–15 min. (Up to 30 mL of HCl may be used for more vigorous action.) Stable for 3–90 days, depending on HCl concentrations. Use appropriate gloves. |
| 156 | 1 g thiourea<br>1 mL $H_3PO_4$<br>1000 mL water | Electrolytic, 0.005–0.01 A/cm $^2$, 1–2 min. |
| 157 | 25 g $CrO_3$<br>150 mL HCl<br>50 mL water | Use a certified and tested hood. Immerse 5–20 s. |
| 158 | 10 mL HF<br>10 mL $HNO_3$<br>20 mL glycerol | Swab 5–15 s. Do not store Properly discard after use. Solution decomposes on standing. |
| 159 | 5 mL HF<br>20 mL $HNO_3$<br>50 mL acetic acid | Use a certified and tested hood. Swab 10–30 s. Do not store. |
| 160 | 20 mL HF<br>15 mL $H_2SO_4$<br>5 mL $HNO_3$<br>50 mL water | Immerse to 5 min. |
| 161 | 25 mL $HNO_3$<br>5 mL HF | Immerse 5–120 s. |
| 162 | A<br><br>50 mL lactic acid<br>30 mL $HNO_3$<br>2 mL HF<br>B<br>30 mL lactic acid<br>10 mL $HNO_3$<br>10 mL HF | Swab 1–3 min in Solution A (acts as etch polish). To etch, swab with Solution B for 5 s. Repeat if necessary. The HF may be varied to give more or less etching. Do not store. |
| 163 | 30 mL $H_2SO_4$<br>30 mL HF<br>3–5 drops $H_2O_2$ (30 %)<br>30 mL water | Immerse 5–60 s. Use this solution for alternate etch and polishing. |
| 164 | 50 mL $HNO_3$<br>30 g ammonium bifluoride<br>20 mL water | Use a certified and tested hood. Swab 3–10 s. |
| 165 | 10 mL HCl<br>90 mL ethanol | (a) Electrolytic at 10 V for 30 s. Use carbon cathode and platinum wire connection to specimen. For etch-polishing, use shorter time.<br>(b) Electrolytic at 6 V for 10 s. Use stainless steel cathode and platinum or Nichrome wire contact to specimen. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 166 | A<br>20 g ammonium persulfate<br>90 mL water<br>B<br>20 g KCN<br>90 mL water | Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Mix 1 + 1 ratio of Solution A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 in. dia mount.) Immerse to several minutes. |
| 167 | 5 g NaCN<br>100 mL water | Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Electrolytic at 1–5 V ac for 1–2 min. Use platinum cathode. |
| 168 | 20 mL HCl<br>35 g NaCl<br>80 mL water | Composition given will saturate the solution with NaCl. Electrolytic at 1½ V ac for 1 min. |
| 169 | 5 mL $HNO_3$<br>50 mL ethylene glycol<br>20 mL ethanol (95 %) or methanol (95 %) | Electrolytic at 0.05 A/cm$^2$ for 2 min. Use stainless steel cathode. |
| 170 | 1 mL HF<br>30 mL $HNO_3$<br>30 mL lactic acid | (a) Swab 5–30 s. Follow with water rinse, alcohol rinse, dry.<br>(b) Swab for 10 s intervals. Increase HF to exaggerate grain boundaries. |
| 171 | concentrated HCl | Use a certified and tested hood. Electrolytic at 5 V ac for 1–2 min. For etch-polishing, use shorter times. Follow with water rinse, alcohol rinse, and dry. |
| 172 | A<br>5 g ammonium persulfate<br>100 mL water<br>B<br>5 g KCN<br>100 mL water | Use a certified and tested hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as skin contact. Prepare 1 + 1 mixture of Solutions A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 in. dia mount.) Immerse 1–2 min. |
| 173 | 50 mL $NH_4$ OH<br>10–30 mL $H_2$ $O_2$ (50 %) | Immerse few seconds to a minute. |
| 174 | A<br>25 mL $HNO_3$<br>1 g $K_2$ $Cr_2$ $O_7$<br>1000 mL water<br>B<br>40 g $CrO_3$<br>3 g $Na_2$ $SO_4$<br>200 mL water | Use a certified and tested hood. Prepare 1 + 1 mixture of Solutions A and B. Apply with camel's hair brush. Nonadherent film of silver chromate should form. If film adheres, add more of Solution A, if none forms, add Solution B. |
| 175 | 1 g $CrO_3$<br>1 mL $H_2$ $SO_4$<br>1000 mL water | Use a certified and tested hood. Immerse to 1 min. |
| 176 | 2 g $FeCl_3$<br>100 mL water | Immerse 5–30 s. |
| 177 | 10 g NaOH<br>100 mL water | Swab or immerse 5–15 s. |
| 178 | 20 mL HF<br>20 mL $HNO_3$<br>60 mL lactic acid | Swab for 5–20 s. Do not store. |
| 179 | A<br>10 mL HF<br>10 mL $HNO_3$<br>30 mL lactic acid<br>B<br>10 mL HF<br>90 mL $H_2$ $SO_4$ | Use a certified and tested hood—Mix Solution B very slowly. Solution A is used as a chemical polish, though some etching will occur. Swab 2 or more minutes for desired surface. If surface is insufficiently etched use Solution B electrolytically at ½ –1 A/in.$^2$ of specimen. Use carbon cathode and platinum wire connection to specimen. Properly discard Solution B after 1 hr. Do not store. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 180 | 10 mL $HNO_3$<br>30 mL acetic acid<br>50 mL glycerol | Use a certified and tested hood. Immerse for ½ –10 min at 38 to 42°C (100–108°F). Do not store Properly discard after use. Solution composes on standing. |
| 181 | 2 mL HCl<br>100 mL ethanol (95 %) or methanol (95 %) | Swab for 1–3 min. |
| 182 | 10 mL $HNO_3$<br>10 mL acetic acid<br>80 mL glycerol | Use a certified and tested hood. Immerse for ½ –10 min at 38 to 42°C (100–108°F). Do not store. Properly discard after use. Solution decomposes on standing. |
| 183 | 2 drops HF<br>1 drop $HNO_3$<br>25 mL glycerol | Immerse for 1 min. Do not store. Properly discard after use. Solution decomposes on standing. |
| 184 | 10 g $FeCl_3$<br>2 mL HCl<br>100 mL water | Immerse for ½ –5 min. |
| 185 | 10 mL HF<br>10 mL $HNO_3$ | Swab for few seconds. |
| 186 | 10 mL HF<br>5 mL $HNO_3$<br>85 mL water | Swab 3–20 s. |
| 187 | 10 mL HF<br>30 mL $HNO_3$<br>50 mL water | Swab 3–20 s. |
| 188 | 1 mL HF<br>2 mL $HNO_3$<br>50 mL $H_2O_2$ (30 %)<br>50 mL water | Use a certified and tested hood. Swab until stain is removed. |
| 189 | 10 mL HF<br>25 mL $HNO_3$<br>45 mL glycerol<br>20 mL water | Swab 3–20 s. Do not store Properly discard after use. Solution decomposes on standing. |
| 190 | 8 g KOH<br>10 mL $H_2O_2$ (30 %)<br>60 mL water | Swab 3–20 s. |
| 191 | 25 mL HF<br>18 g benzalkonium chloride<br>35 mL methanol (95 %)<br>40 mL glycerol | Swab 3–20 s. |
| 192 | 1–3 mL HF<br>2–6 mL $HNO_3$<br>100 mL water | Swab 3–10 s or immerse 10–30 s. (HF attacks and $HNO_3$ brightens the surface of titanium. Make concentration changes on this basis.) |
| 193 | 2 drops HF<br>1 drop $HNO_3$<br>3 mL HCl<br>25 mL glycerol | Swab 3–20 s. Do not store Properly discard after use. Solution decomposes on standing. |
| 194 | 20 mL HF<br>20 mL $HNO_3$<br>60 mL glycerol | Immerse 5–30 s.Do not store Properly discard after use. Solution decomposes on standing. |
| 195 | 30 mL $H_3PO_4$<br>30 mL ethylene glycol<br>50 mL ethanol (95 %) | Electrolytic at 18–20 V (0.03 A/cm$^2$) for 5–15 min. |
| 196 | 18 g $CrO_3$<br>75 mL acetic acid<br>20 mL water | Use a certified and tested hood. Dissolve $CrO_3$ in hot water and cool before adding acetic acid. Keep solution below 2°C (35°F) during use. Electrolytic at 80 V for 5–30 min. Do not store. |
| 197 | 5 g oxalic acid<br>100 mL water | Electrolytic at 6 V for 5–20 s. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 198 | 30 mL HF<br>30 mL $HNO_3$<br>30 mL glycerol | Swab for 60 s. Do not store. Properly discard after use. Solution decomposes on standing. |
| 199 | 2 mL HF<br>5 g $AgNO_3$<br>100 mL water | Swab for 5 s. |
| 200 | A<br>40 g $CrO_3$<br>3 g $Na_2 SO_4$<br>200 mL water<br>B<br>40 g $CrO_3$<br>200 mL water | Use a certified and tested hood. Immerse in Solution A with gentle agitation for several seconds. Rinse in Solution B. |
| 201 | A<br>40 g $CrO_3$<br>1.5 g $Na_2 SO_4$<br>200 mL water<br>B<br>40 g $CrO_3$<br>200 mL water | Use a certified and tested hood. Immerse in Solution A with gentle agitation for several seconds. Rinse in Solution B. |
| 202 | A<br>10 g $CrO_3$<br>1 g $Na_2 SO_4$<br>200 mL water<br>B<br>40 g $CrO_3$<br>200 mL water | Use a certified and tested hood. Immerse in Solution A for 2–5 s. Rinse in Solution B. |
| 203 | 20 g $CrO_3$<br>100 mL water | Use a certified and tested hood. Electrolytic at 0.2 A/$cm^2$ for 5 s. |
| 204 | 10 mL perchloric acid<br>10 mL glycerol<br>70 mL ethanol (95 %)<br>10 mL water | Use in a wash down/prechloric rated fume hood. Precaution—Keep cool when mixing and use. Electrolytic at 15–50 V for 15–60 s. |
| 205 | 5 mL HF<br>2 mL $AgNO_3$ (5 %)<br>100 mL water | Swab vigorously for 10–60 s. Wet cotton frequently. |
| 206 | 5 mL HF<br>10 mL $HNO_3$<br>100 mL glycerol | Precaution—Properly discard after use. Solution decomposes on standing. Electrolytic at 9–12 V for 1–10 min. |
| 207 | 30 mL $HNO_3$<br>30 mL acetic acid<br>30 mL water | Swab for 5–30 s. Do not store. |
| 208 | 1 mL $NH_4$ OH<br>3 g ammonium persulfate<br>100 mL water | Immerse or swab few seconds to a minute. |
| 209 | 15 mL $HNO_3$<br>3 mL HF<br>80 mL water | Immerse 5–60 s. |
| 210 | 50 mL water (cold) saturated with sodium thiosulfate<br>1 g potassium metabisulfite | First ingredient in stock solution. Add potassium metabisulfite before use. Solution good for several days, or longer. Immerse face up, gently agitate until coloration begins, allow to settle. Stop etch when surface is red-violet. Etch time varies with material. Colors matrix phases. |
| 211 | 3 g potassium metabisulfite<br>10 g sodium thiosulfate<br>100 mL water | Use fresh solution. Immerse specimen face up, gently agitate solution until coloration begins, allow to settle. Stop etch when surface is red-violet. Etch time varies with material. Colors matrix phases. |

**TABLE 2** *Continued*

| Etchant | Composition | Procedure |
|---|---|---|
| 212 | 10–50 % HCl in water<br>0.5–1.0 g potassium metabisulfite per 100 mL of aqueous HCl solution<br>Optional: 1 g $CuCl_2$<br>1–3 g $FeCl_3$<br>2–10 g ammonium bifluoride | For more corrosion resistant alloys. Increase the HCl and potassium metabisulfite contents. Use optional ingredients to improve coloration, if needed. Colors matrix phases. Use by immersion only. |
| 213 | 2–10 mL HCl<br>0.5–3 mL selenic acid<br>100 mL ethyl alcohol (95 %) | For more corrosion resistant alloys, increase the HCl and selenic acid content. For highly corrosion-resistant alloys, use 20–30 mL HCl. Colors second phase constituents. Use by immersion only. |
| 214 | 1 g sodium molybdate<br>100 mL water | Add nitric acid to lower the pH to 2.5–3. Add 0.1–0.5 g ammonium bifluoride for carbon steels. Use by immersion only. Colors carbides. Immerse about 15 s. |
| 215 | 240 g sodium thiosulfate<br>30 g citric acid<br>24 g lead acetate<br>1000 mL water | Mix in order given. Store in a dark bottle at least 24 h before use at 20°C. Lightly pre-etch specimen before use. Use small portion of stock solution for 4 h max. Pre-etch steel specimens with nital before tinting the MnS (add 0.2 g sodium nitrite to 100 mL of etch) white. Colors phosphides in cast iron. Colors matrix of Cu alloys. |
| 216 | 8–15 g sodium metabisulfite<br>100 mL water | Do not store. Mix fresh. Immerse specimen face up. Agitate solution gently until coloration begins, allow to settle. Stop when surface is dark. Use crossed polarized light and sensitive tint to improve coloration. |
| 217 | 5 g ammonium bifluoride<br>100 mL water | Mix fresh, use plastic coated tongs and polyethylene beaker. Immerse until surface is colored. |
| 218 | 3 g ammonium bifluoride<br>4 mL HCl<br>100 mL water | Mix fresh, use plastic coated tongs and polyethylene beaker. Immerse until surface is colored. Works best with attack-polished specimens. |
| 219 | 60 mL $HNO_3$<br>40 mL water | Electrolytic etch, does not reveal twins in $\gamma$ stainless steel. Excellent grain boundary etch for ferritic stainless steels. Use at 1 V dc, 120 s, with stainless cathode; 0.6 V dc with platinum cathode. |
| 220 | 20 g NaOH<br>100 mL water | Electrolytic etch, colors $\delta$-ferrite in stainless steels. Use at 2–20 V dc, 5–20 s, stainless steel cathode. If $\delta$ is not colored, increase NaOH to 40 g. |
| 221 | 50 mL water<br>50 mL ethyl alcohol<br>50 mL methyl alcohol<br>50 mL HCl<br>1 g $CuCl_2$<br>2.5 g $FeCl_3$<br>2.5 mL $HNO_3$ | Use by immersion. Will not attack sulfides in stainless steels. |
| 222 | 8 g $Na_2 SO_4$<br>100 mL water | (a) Few seconds to 1 minute.<br>(b) Pre-etch 2 s in No. 74, rinse, and etch 20 s. |
| 223 | A<br>8 g oxalic acid<br>5 mL $H_2 SO_4$<br>100 mL water<br>B<br>$H_2 O_2$ (30 %) | Mix equal volumes of Solutions A and B just before use. Etch 2–3 s; 3 s pre-etch in No. 74 may be needed. |
| 224 | 10 mL $H_2 O_2$ (30 %)<br>20 mL 10 % aqueous NaOH | Immerse 10 s at 20°C (68°F). |
| 225 | 4 g NaOH<br>100 mL saturated aqueous $KMnO_4$ | Immerse 10 s at 20°C (68°F). |
| 226 | 15 mL HCl<br>10 mL acetic acid<br>5 mL $HNO_3$<br>2 drops glycerol | **Use a certified and tested hood.**—Can give off nitrogen dioxide gas. **Precaution**—Mix HCl and glycerol thoroughly before adding $HNO_3$. Do not store. Properly discard before solution attains a dark orange color. Use fresh or age up to 1 min. Immerse or swab few seconds to few minutes. Can increase $HNO_3$ to increase strength. Sometimes a few passes on the final polishing wheel is also necessary to remove a passive surface. |

† Editorially corrected in May 2011.

**TABLE 3 Etchant Names**

| Common Name | No. | Common Name | No. |
|---|---|---|---|
| Acetic glyceregia | 89, 226 | Groesbeck's | 19 |
| Alkaline Sodium Picrate | 85 | Hatch | 2 |
| Aqua regia | 12 | Howarth's | 84 |
| Barker's | 5 | Kalling's 1 | 95 |
| Beraha's | 99, 155, 211–215 | Kalling's 2 | 94 |
| Carapella | 138 | Keller's | 3 |
| Chrome regia | 101 | Klemm's | 210 |
| Contrast | 141 | Kroll's | 192, 187 |
| CP 4 | 60 | Marble's | 25 |
| El-1R | 107 | Marshall's | 223 |
| Flat | 133 | Murakami's | 98 |
| Flouregia | 90, 158 | Nital | 74 |
| Frank's | 104 | Palmerton | 200 |
| Fry's | 79 | Phoschromic | 111 |
| G | 107 | Picral | 76 |
| Glyceregia | 87 | Ralph's | 221 |
| Gorsuch | 75 | Super Picral | 77 |
| Grard's No. 1 | 35 | Vilella's | 80 |
| Green contrast | 94 | 92-5-3 | 105 |

## REFERENCES

**(1)** Lewis, R.J., *Sax's Dangerous Properties of Industrial Materials*, 11th ed. John Wiley and Sons., Inc., Hoboken, New Jersey, 2004.

**(2)** *Prudent Practices for Handling Hazardous Chemicals in Laboratories*, National Resource Council, National Academy Press, Washington, DC, 1995.

**(3)** Furr, A.K., *CRC Handbook of Laboratory Safety, 5th ed.* CRC Press, Boca Raton, Florida, 2000.

**(4)** .Wesenberg, G., Proctor, N., *Proctor and Hughes' Chemical Hazards in the Workplace, 5th edition*, edited by Gloria J. Hathaway and Nick H, ProctorWiley-Interscience, Hoboken, New Jersey, 2004.

**(5)** OSHA Lab Safety Standard 29 CFR 1910.1450, Occupational Exposures to Hazardous Chemicals in the Laboratory.

**(6)** *Prudent practices in the laboratory : handling and disposal of chemicals*, Committee on Prudent Practices for Handling, Storage, and Disposal of Chemicals in Laboratories, Board on Chemical Sciences and Technology, Commission on Physical Sciences, Mathematics, and Applications, National Research Council.Washington, D.C. : National Academy Press, 1995 .

**(7)** Lefevre, M. J., and Conibear, S., *First Aid Manual for Chemical Accidents, 2nd ed.*, Van Nostrand Reinhold Co., Inc., New York, 1989.

**(8)** Lewis, R. J., *Rapid Guide to Hazardous Chemicals in the Workplace, 4th ed.*,John Wiley and Sons., Inc., New York, 2000.

**(9)** Anderson, R. L., "Safety in the Metallography Laboratory," Westinghouse Research Lab, Pittsburgh, PA, Scientific Paper 65-1P30-METLL-P2, 1965 .

**(10)** Urben, P. and Bretherick, L., *Bretherick's Handbook of Reactive Chemical Hazards, 6th ed.*, Butterworth-Heinemann, , 1999.

**(11)** NFPA Haz-Mat Quick Guide, 1997.

**(12)** *Woldman's Engineering Alloys, 9th ed.*, J. P. Frick, ed., ASM International, Metals Park, Ohio, 2000.

**(13)** Vander Voort, G. F., *Metallography: Principles and Practice*, ASM International, Materials Park, Ohio, 1999.

**(14)** Beraha, E., and Shpigler, B., *Color Metallography*, ASM, Metals Park, OH, 1977.

**(15)** Vander Voort, G. F., "Tint Etching," Metal Progress, Vol 127, March 1985, pp. 31–33, 36–38, 41.

**(16)** Weck, E., and Leistner, E., *Metallographic Instructions for Colour Etching by Immersion, Parts I, II and II*, Deutscher Verlag, Für Schweisstechnik GmbH, Düsseldorf, West Germany, 1982, 1983, and 1986.

**(17)** Bühler, H. E., and Hougardy, H. P., *Atlas of Interference Layer Metallography* , Deutsche Gesellschaft für Metallkunde, Oberursel 1, West Germany, 1980.

**(18)** *Metals Handbook, Metallography and Microstructures, 10th ed., Vol 9*, ASM International, Metals Park, Ohio, 2004.