



Standard Practice for Microetching Metals and Alloys¹

This standard is issued under the fixed designation E 407; 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 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:

D 1193 Specification for Reagent Water²

E 7 Terminology Relating to Metallography³

3. Terminology

3.1 Definitions:

3.1.1 For definition of terms used in this standard, see Terminology E 7.

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, TiO₂, ZnS or ZnO) with a known index of refraction, generally due to light interference effects (also known as the "Pepperhoff method").

4. Summary of Practice

4.1 Table 1 is an alphabetical listing of the metals (including rare earths) and their alloys for which etching information is available. For each metal and alloy, one or more etchant numbers and their corresponding use is indicated. Alloys are listed as a group or series when one or more etchants are common to the group or series. Specific alloys are listed only when necessary. When more than one etchant number is given for a particular use, they are usually given in order of preference. The numbers of electrolytic etchants are *italicized* to differentiate them from nonelectrolytic etchants.

4.2 Table 2 is a numerical listing of all the etchants referenced in Table 1 and includes the composition and general procedure to be followed for each etchant.

4.3 To use the tables, look up the metal or alloy of interest in Table 1 and note the etchant numbers corresponding to the results desired. The etchant composition and procedure is then located in Table 2 corresponding to the etchant number.

4.4 If the common name of an etchant is known (Marble's, Vilella's, etc.), and it is desired to know the composition, Table 3 contains an alphabetical listing of etchant names, each coded with a number corresponding to the etchant composition given in Table 2.

5. Significance and Use

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

6. Safety Precautions

6.1 Before using or mixing any chemicals, all product labels and pertinent Material Safety Data Sheets (MSDS) should be read and understood concerning all of the hazards and safety precautions to be observed. Users should be aware of the type of hazards involved in the use of all chemicals used, including those hazards that are immediate, long-term, visible, invisible, and with or without odors.

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,

¹ This practice is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.01 on Sampling, Specimen Preparation, and Photography.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 03.01.

mixing, and disposing of each chemical, as well as any combinations of those chemicals.

6.1.3 Table 2 includes specific safety precautions for the mixing or use of some etchants. The user should observe each of these specific precautions.

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.).

6.2.2 Use proper devices (glass or plastic) for weighing, mixing, containing, and storage of solutions.

6.2.3 When mixing etchants, always add reagents to the

solvent unless specific instructions indicate otherwise.

6.2.4 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).

6.2.5 In general, it is good practice to work under a properly designed chemical fume hood, and it is imperative with those etchants that give off noxious odors or toxic vapors.

6.2.6 Methanol is a cumulative poison hazard. Where ethanol or methanol are listed as alternates, ethanol is the preferred solvent. Methanol should be used in a properly designed chemical fume hood.

TABLE 1 Etchants for Metals

NOTE 1—Electrolytic etchants are *italicized*.

| Metal | Etchants | Uses |
|--|---|---|
| <i>Aluminum Base:</i> | | |
| Pure Al | 1a, 2, 3 4, 5 1b | general structure grain structure under polarized light grain boundaries and slip lines |
| 1000 series | 1a, 3, 2 4, 5 6, 7 | general structure grain structure under polarized light phase identifications |
| 2000 series | 3, 2, 1a 8a, 6, 7 | general structure phase identifications |
| 3000 series | 3, 1a 4, 5 8a, 6, 7 | general structure grain structure under polarized light phase identifications |
| 4000 series | 3, 1a | general structure |
| 5000 series | 3, 1a, 2, 6, 8a 4, 5 | general structure grain structure under polarized light |
| 6000 series | 3, 1a, 2, 6, 8a, 222 4, 5 1a, 2, 7, 6, 8a | general structure grain structure under polarized light phase identifications |
| 7000 series | 3, 1a, 2 4, 5 3b, 6 | general structure grain structure under polarized light phase identifications |
| <i>Beryllium Base:</i> | | |
| Pure Be | 9, 10 | general structure via polarized light |
| Be alloys | 11 | general structure |
| <i>Chromium Base:</i> | | |
| | 12, 13c | general structure |
| <i>Cobalt Base:</i> | | |
| Pure Co | 14, 15, 16, 17 | general structure |
| Hard-facing and tool metals | 18, 19, 20 | general structure |
| High-temperature alloys | 20, 18, 16, 21, 22b, 24, 25 19 | general structure phase identification |
| <i>Columbium Base (see niobium base)</i> | | |
| <i>Copper Base:</i> | | |
| Pure Cu | 26, 27, 28, 29, 30, 31d, 32, 33, 34b, 35, 36, 37, 38, 39, 40, 41, 42, 8b, 210, 215 43, 28 | general structure chemical polish and etch |
| Cu-Al (aluminum bronze) | 44, 31d, 34b, 35, 36, 37, 38, 39, 40, 45, 215 | general structure |
| Cu-Be | 46, 41, 45 | general structure |
| Cu-Cr | 41 | general structure |
| Cu-Mn | 41 | general structure |
| Cu-Ni | 34, 47, 48, 40, 49, 50 | general structure |

TABLE 1 *Continued*

| Metal | Etchants | Uses |
|--|--|--|
| Cu-Si | 41 | general structure |
| Cu-Sn (tin bronze) | 51, 52 | general structure |
| Admiralty metal Gilding metal Cartridge brass Free-cutting brass Nickel silver | 8b 31d, 32, 33, 41, 42, 49 | general structure general structure |
| Cu alloys | 26, 27, 28, 29, 30, 44, 41, 31d, 32, 33, 34b, 35, 36, 37, 38, 39, 210, 215 53, 43, 28, 49 42, 49, 210 54 | general structure chemical polish and etch darkens beta in alpha-beta brass etching of cold worked brass |
| <i>Dysprosium Base:</i> | 55, 56 | general structure |
| <i>Erbium Base:</i> | 55, 56 | general structure |
| <i>Gadolinium Base:</i> | 55, 56, 57 | general structure |
| <i>Germanium Base:</i> | 58, 59, 60 | general structure |
| <i>Gold Base:</i> | | |
| Pure Au | 61, 62 63 | general structure chemical polish and etch |
| Au alloys | 64b, 62 63 | general structure chemical polish and etch |
| >90 % noble metals | 61 | general structure |
| <90 % noble metals | 65 | general structure |
| <i>Hafnium base:</i> | 66, 67, 68, 69, 70 71 72 | general structure grain structure under polarized light chemical polish and etch |
| <i>Holmium Base:</i> | 55, 56 | general structure |
| <i>Iridium Base:</i> | 73c | general structure |
| <i>Iron Base:</i> | | |
| Pure Fe | 74a 75 210 | grain boundaries substructure colors ferrite grains |
| Fe + C and Fe + <1C + <4 % additions | 76, 74a, 77, 78, 79 74a, 77, 31a, 223 80, 81, 82 78, 222a 31b, 78 83 84 85 86 210, 211 213, 214 216 222b | general structure ferrite grain boundaries prior austenitic grain boundaries in martensitic and bainitic steels untempered martensite carbides and phosphides (matrix darkened, carbides and phosphides remain bright) cementite attacked rapidly, susenite less, ferrite and iron phosphide least overheating and burning stains carbides chemical polish-etch colors ferrite colors carbides colors lath martensite in low-carbon high-alloy grades for dual phase steels; reveals pearlite, darkens martensite and outlines austenite |
| Fe + 4–12 Cr | 80, 87, 88, 89, 90, 91, 79, 210 86 | general structure chemical polish-etch |
| Fe + 12–30 Cr + <6 Ni (400 Series) | 80, 87, 88, 89, 34, 40, 92, 93, 94, 95, 91, 226 96, 97, 98 31c 86 219 220 | general structure signs phase carbides chemical polish-etch grain boundary etch darkens delta ferrite |
| Fe + 12–20 Cr + 4–10 Ni + <7 % other elements (controlled trans- formation, precipitation harden- ing, stainless maraging alloys) | 80, 31c, 89, 99, 100, 91 31c 86 220 | general structure carbides chemical polish-etch darkens delta ferrite |

TABLE 1 *Continued*

| Metal | Etchants | Uses |
|---|--|---|
| Fe + 15–30 Cr + 6–40 Ni + <5 % other elements (300 Series) | 13b, 89, 87, 88, 83a, 80, 94, 95, 91, 101, 212, 221, 226 13a, 102, 31c, 48c, 213 | general structure carbides and sensitization |
| and Fe + 16–25 Cr + 3–6 Ni + 5–10 Mn (200 series) | 48, 96, 97, 98 103, 104, 98 103, 104 86 219 220 | stains sigma phase delineates sigma phase and welds of dissimilar metals chemical polish-etch grain boundary etch (no twins) darkens delta ferrite |
| High temperature | 89, 25, 105, 106, 97, 212, 221 107, 108, 213 86 | general structure γ' precipitate chemical polish-etch |
| Nonstainless maraging steels | 109, 89, 99, 100, 221 83b 86 | general structure grain boundaries chemical polish-etch |
| Tool steels | 74a, 80, 14 110 210, 211 214, 214 224, 225 | general structure grain boundaries in tempered tool steel colors ferrite, lower alloy grades colors cementite |
| Superalloys | 86, 87, 94, 221, 226 111 111 | carbides attacked and colored general etch general structure γ' depletion |
| <i>Lead Base:</i> Pure Pb | 57, 112 113 | general structure for alternate polishing and etching |
| Pb + <2 Sb | 114, 115, 57, 74b 113 | general structure for alternate polishing and etching |
| Pb + >2 Sb | 114, 57, 74b 113 | general structure for alternate polishing and etching |
| Pb + Ca | 112 113 | general structure for alternate polishing and etching |
| Pb alloys Babbitt | 116, 117b 74b | general structure general structure |
| <i>Magnesium Base:</i> Pure Mg | 118, 119, 74a, 120, 121, 122 123 | general structure stain-free polish-etch |
| Mg-Mn | 119, 74a, 124, 122 | general structure |
| Mg-Al, Mg-Al-Zn (Al + Zn <5 %) | 118, 119, 74a, 125, 124, 123, 122 120, 125, 126, 127 124, 126, 127 | general structure general structure phase identification grain structure |
| Mg-Al, Mg-Al-Zn (Al + Zn >5 %) | 118, 119, 74a, 125, 124, 121, 122 120, 125, 126, 127 | general structure phase identification |
| Mg-Zn-Zr and Mg-Zn-Th-Zr | 118, 119, 74a, 1d, 128, 124, 126, 127, 121, 122 120, 121 | general structure phase identification |
| Mg-Th-Zr and Mg-Rare Earth-Zr | 118, 119, 74a, 1d, 124, 127, 121, 122 120, 121 | general structure phase identification |
| <i>Molybdenum Base:</i> As cast | 98c, 129, 130, 131 132a | general structure chemical polish prior to etching |
| <i>Nickel Base:</i> Pure Ni and high Ni alloys | 133, 134, 47, 135, 136, 25, 108, 31c 137 | general structure grain boundary sulfidation |
| Ni-Ag | 38, 138, 50, 139 | general structure |
| Ni-Al | 50, 140, 141, 142, 89, 143 | general structure |
| Ni-Cr | 144, 50, 83, 134, 145, 98, 146, 147, 13a | general structure |
| Ni-Cu | 38, 138, 50, 133, 140, 25, 134, 47, 48b, 94, 108, 34 | general structure |
| Ni-Fe | 50, 140, 141, 83, 134, 148, 40, 107, 149 74e, 25, 150 | general structure orientation pitting |
| Ni-Mn | 74e | general structure |
| Ni-Mo | 143 | general structure |
| Ni-Ti | 143, 151, 50, 133 | general structure |
| Ni-Zn | 152 | general structure |

TABLE 1 *Continued*

| Metal | Etchants | Uses |
|---------------------------|--|--|
| Superalloys | 94, 105, 138, 153, 12, 87, 89, 212, 226 25, 94 107, 111, 13a 133 154 19b, 155, 156 22a 157 107 154 18 213 | general structure grain size reveals microstructural inhomogeneity grain boundary sulfidation fine precipitation structure differential matrix and nonmetallic staining for passive alloys (for example, UNS Alloy N06625) specific for UNS Alloy N10004 submicroscopic structure in aged super-alloys particularly for electron microscopy. Stains the matrix when γ' precipitates are present γ' banding pre-etch activation for passive specimens colors carbide and γ' |
| Niobium (Columbium) Base: | 129, 66, 158, 159, 160, 161, 162, 163 164, 129, 160 | general structure grain boundaries |
| Osmium Base: | 165a 165a | general structure 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 167 | general structure electrolytic polish and etch |
| Pt Alloys | 64b, 73a 167 | general structure electrolytic polish and etch |
| >90 % noble metals | 61 | general structure |
| <90 % noble metals | 65 | general structure |
| Pt-10 % Rh | 168 | general structure |
| Plutonium Base: | 169 | general structure |
| Rhenium Base: | 13b, 98c, 132b, 170a | general structure |
| Rhodium Base: | 171 | general structure |
| Ruthenium Base: | 73b 73b | general structure 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, 179 164 158 | general structure grain boundaries and inclusions grain boundaries—retains carbide precipitate |
| Thorium Base: | | |
| Pure Th | 185 | general structure |
| Th alloys | 185 | general structure |
| Tin Base: | | |
| Pure Sn | 74d, 180, 151 181 | general structure grain boundaries |
| Sn-Cd | 74d | general structure |
| Sn-Fe | 74d, 177a | general structure |
| Sn-Pb | 182, 183, 74b 116 | general structure darkens Pb in Sn-Pb eutectic |
| Sn coatings (on steel) | 183 | general structure |
| Babbitts | 184 | general structure |
| Sn-Sb-Cu | 74b | general structure |
| Titanium Base: | | |
| Pure Ti | 186, 187, 67, 68, 69, 217 | general structure |

TABLE 1 *Continued*

| Metal | Etchants | Uses |
|---------------------------|--|--|
| | 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 |
| <i>Tungsten Base:</i> | | |
| Pure W | 98c, 131 | general structure |
| As cast | 132a | chemical polish prior to etching |
| W-Th | 209 | general structure |
| <i>Uranium Base:</i> | | |
| Pure U | 67, 69, 195, 196 | general structure |
| U + Zr | 68 | general structure |
| U beryllides | 170a | general structure |
| U alloys | 67, 69, 195, 96 | general structure |
| | 207 | carbides |
| <i>Vanadium Base:</i> | | |
| Pure V | 170b, 165b | general structure |
| | 197, 198 | grain boundaries |
| V alloys | 199, 198 | general structure |
| <i>Zinc Base:</i> | | |
| Pure Zn | 200a | general structure |
| Zn-Co | 177 | general structure |
| Zn-Cu | 201 | general structure |
| | 203 | distinguishes gamma (γ) and epsilon (ϵ) |
| Zn-Fe | 74a | structure of galvanized sheet |
| Die castings | 202 | general structure |
| <i>Zirconium Base:</i> | | |
| | 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

| Etchant | Composition | Procedure |
|---------|--|---|
| 1 | 1 mL HF 200 mL water | (a) Swab with cotton for 15 s. (b) Alternately immerse and polish several minutes. (c) Immerse 3–5 s. (d) Immerse 10–120 s. |
| 2 | 3 mL HF 100 mL water | (a) Swab 10 s to reveal general structure. (b) Immerse 15 min, wash 10 min in water to form film with hatching which varies with grain orientation. |
| 3 | 2 mL HF 3 mL HCl 5 mL HNO ₃ 190 mL water | (a) Immerse 10–20 s Wash in stream of warm water. Reveals general structure. (b) Dilute with 4 parts water-colors constituents—mix fresh. |
| 4 | 24 mL H ₃ PO ₄ 50 mL Carbitol (diethylene glycol monoethyl ether) 4 g boric acid 2 g oxalic acid 10 mL HF 32 mL water | Electrolytic: Use carbon cathode raising d-c voltage from 0–30 V in 30 s. Total etching time 3 min with agitation. Wash and cool. Repeat if necessary. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|---|---|
| 5 | 5 g HBF ₄ 200 mL water | Electrolytic: Use Al, Pb, or stainless steel cathode. Anodize 1–3 min, 20–45 V d-c. At 30 V, etch for 1 min. |
| 6 | 25 mL HNO ₃ 75 mL water | Immerse 40 s at 70°C (160°F). Rinse in cold water. |
| 7 | 10–20 mL H ₂ SO ₄ 80 mL water | Immerse 30 s at 70°C (160°F). Rinse in cold water. |
| 8 | 10 mL H ₃ PO ₄ 90 mL water | (a) Immerse 1–3 min at 50°C (120°F). (b) Electrolytic at 1–8 V for 5–10 s. |
| 9 | 3–4 9 sulfamic acid 5 drops HF 100 mL water | Use just prior to the last polishing operation. It is not intended as a final etchant. The specimen is examined as polished under polarized light. |
| 10 | 10 mL HF 90 mL methanol (90 %) | Immerse 10–30 s. |
| 11 | 2 mL HF 100 mL water | Immerse or swab few seconds to a minute. |
| 12 | 20 mL HNO ₃ 60 mL HCl | Use hood. Do not store. Immerse or swab 5–60 s. |
| 13 | 10 g oxalic acid 100 mL water | Electrolytic at 6 V: (a) 10–15 s. (b) 1 min. (c) 2–3 s. Use stainless steel cathode and platinum or Nichrome connection to specimen. |
| 14 | 10 mL HNO ₃ 90 mL methanol (95 %) | Immerse few seconds to a minute. |
| 15 | 15 mL HNO ₃ 15 mL acetic acid 60 mL HCl 15 mL water | Age before use. Immerse 5–30 s. May be used electrolytically. |
| 16 | 5–10 mL HCl 100 mL water | Electrolytic at 3 V for 2–10 s. |
| 17 | 5 mL HCl 10 g FeCl ₃ 100 mL water | Electrolytic at 6 V for few seconds. |
| 18 | 2–10 g CrO ₃ 100 mL water | Electrolytic at 3 V for 2–10 s. |
| 19 | A 8 g NaOH 100 mL water B Saturated aqueous solution of KMnO ₄ | Immerse in freshly mixed Solutions A + B (1:1) for 5–10 s. If surface activation is necessary, first use Etch #18, then rinse in water. While still wet, immerse in Solutions A + B (1:1). Mixture of solutions A + B has 15-min useful life. |
| 20 | 5 mL H ₂ O ₂ (30 %) 100 mL HCl | Use hood. <i>Mix fresh</i> . Immerse polished face up for few seconds. |
| 21 | 1 g CrO ₃ 140 mL HCl | Use hood. To mix, add the HCl to CrO ₃ . Electrolytic at 3 V for 2–10 s. |
| 22 | 100 mL HCl 0.5 mL H ₂ O ₂ (30 %) | Use hood. Do not store. (a) Immerse or swab ½–3 min. Add H ₂ O ₂ dropwise to maintain action. (b) Electrolytic, 4 V, 3–5 s. |
| 23 | 5 mL HCl 95 mL ethanol (95 %) or methanol (95 %) | Electrolytic at 6 V for 10–20 s. |
| 24 | 5 mL HNO ₃ 200 mL HCl 65 g FeCl ₃ | Use hood. Immerse few seconds. |
| 25 | 10 g CuSO ₄ 50 mL HCl 50 mL water | Immerse or swab 5–60 s. Made more active by adding few drops of H ₂ SO ₄ just before use. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|---|--|
| 26 | 5 g FeCl ₃ 10 mL HCl 50 mL glycerol 30 mL water | Swab 16–60 s. Activity may be decreased by substituting glycerol for water. |
| 27 | 1 g KOH 20 mL H ₂ O ₂ (3 %) 50 mL NH ₄ OH 30 mL water | Dissolve KOH in water, then slowly add NH ₄ OH to solution. Add 3 % H ₂ O ₂ last. Use fresh—immerse few seconds to a minute. |
| 28 | 1 g FeNO ₃ 100 mL water | Swab or immerse few seconds to a minute. |
| 29 | 1 g K ₂ Cr ₂ O ₇ 4 mL H ₂ SO ₄ 50 mL water | Add 2 drops of HCl just before using. Swab few seconds to a minute. |
| 30 | 25 mL NH ₄ OH 25 mL water 50 mL H ₂ O ₂ (3 %) | Mix NH ₄ OH and water before adding H ₂ O ₂ . Must be used fresh. Swab 5–45 s. |
| 31 | 10 g ammonium persulfate 100 mL water | (a) Swab or immerse to 5 s. (b) Immerse to 2 min to darken matrix to reveal carbides and phosphides. (c) Electrolytic at 6 V for few seconds to a minute. (d) Immerse 3–60 s. Can be heated to increase activity. |
| 32 | 60 g CrO ₃ 100 mL water | Saturated solution. Immerse or swab 5–30 s. |
| 33 | 10 g CrO ₃ 2–4 drops HCl 100 mL water | Add HCl just before use. Immerse 3–30 s. Phases can be colored by Nos. 35, 36, 37. |
| 34 | 5 g FeCl ₃ 50 mL HCl 100 mL water | (a) Immerse or swab few seconds to few minutes. Small additions of HNO ₃ activate solution and minimize pitting. (b) Immerse or swab few seconds at a time. Repeat as necessary. |
| 35 | 20 g FeCl ₃ 5 mL HCl 1 g CrO ₃ 100 mL water | Immerse or swab few seconds at a time until desired results are obtained. |
| 36 | 25 g FeCl ₃ 25 mL HCl 100 mL water | Immerse or swab few seconds at a time until desired results are obtained. |
| 37 | 1 g FeCl ₃ 10 mL HCl 100 mL water | Immerse or swab few seconds at a time until desired results are obtained |
| 38 | 8 g FeCl ₃ 25 mL HCl 100 mL water | Swab 5–30 s. |
| 39 | 5 g FeCl ₃ 10 mL HCl 1 g CuCl ₂ 0.1 g SnCl ₂ 100 mL water | Immerse or swab few seconds at a time until desired results are obtained. |
| 40 | 5 g FeCl ₃ 16 mL HCl 60 mL ethanol (95 %) or methanol (95 %) | Immerse or swab few seconds to few minutes. |
| 41 | 2 g K ₂ Cr ₂ O ₇ 8 mL H ₂ SO ₄ 4 drops HCl 100 mL water | Add the HCl just before using. Immerse 3–60 s. |
| 42 | 10 g cupric ammonium chloride 100 mL water NH ₄ OH | Add NH ₄ OH to solution until neutral or slightly alkaline. Immerse 5–60 s. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|--|--|
| 43 | 20 mL NH ₄ OH 1 g ammonium persulfate 60 mL water | Immerse 5–30 s. |
| 44 | 50 mL NH ₄ OH 20–50 mL H ₂ O ₂ (3 %) 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 ₃ 100 mL water | Electrolytic at 6 V for 3–6 s. Use aluminum cathode. |
| 46 | 15 mL NH ₄ OH 15 mL H ₂ O ₂ (3 %) 15 mL water 4 pellets NaOH | When mixing, add NaOH pellets last. For best results use before pellets have dissolved. |
| 47 | 5 g NaCN or KCN 5 g (NH ₄) ₂ S ₂ O ₂ 100 mL water | Use hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as contact. |
| 48 | 10 g NaCN 100 mL water | Use hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as contact. Electrolytic at 6 V: (a) 5 s for sigma. (b) 30 s for ferrite and general structure. (c) to 5 min for carbides. |
| 49 | 3 g FeSO ₄ 0.4 g NaOH 10 mL H ₂ SO ₄ 190 mL water | Electrolytic at 8–10 V (0.1 A) for 5–15 s. |
| 50 | 5 mL acetic acid 10 mL HNO ₃ 85 mL water | Use hood. Do not store. Electrolytic at 1.5 V for 20 to 60 s. Use platinum wires. |
| 51 | 2 g FeCl ₃ 5 mL HCl 30 mL water 60 mL ethanol or methanol | Immerse few minutes. |
| 52 | 1 g sodium dichromate 1 g NaCl 4 mL H ₂ SO ₄ 250 mL water | Swab few seconds. |
| 53 | 1–5 mL NH ₄ OH 100 mL water | Immerse 5–60 s. |
| 54 | 1 g ammonium acetate 3 g sodium thiosulfate 7 mL NH ₄ OH 1300 mL water | Electrolytic at 0.3 A/cm ² for 5–30 s. |
| 55 | 1 mL H ₂ SO ₄ 15 mL HNO ₃ 10 mL acetic acid 5 mL H ₃ PO ₄ 20 mL lactic acid | 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. |
| 56 | 30 mL HNO ₃ 10 mL H ₃ PO ₄ 20 mL acetic acid 10 mL lactic acid | Swab gently 5–15 s. Rinse with ethanol or methanol and blow dry. |
| 57 | 75 mL acetic acid 25 mL H ₂ O ₂ (30 %) | Immerse 6–15 s. |
| 58 | 25 mL HF 25 mL HNO ₃ 5 mL water | Swab 3–20 s. |
| 59 | 2 g AgNO ₃ 40 mL water 40 mL HF 20 mL HNO ₃ | Mix AgNO ₃ and water, then add HF and HNO ₃ . Swab ½ –2 min. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|---|--|
| 60 | 25 mL HNO ₃ 15 mL acetic acid 15 mL HF 5–7 drops bromine | Use hood. Let stand ½ h before using. Swab 3–20 s. |
| 61 | 60 mL HCl 40 mL HNO ₃ | Use hood. Immerse few seconds to a minute. |
| 62 | 1–5 g CrO ₃ 100 mL HCl | 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 10 mL HNO ₃ 100 mL HCl | Swab few seconds to a minute. |
| 64 | 5 mL HNO ₃ 25 mL HCl 30 mL water | (a) Immerse 1–5 min. (b) Use hot. Will form chloride film on gold alloys if much silver is present. Ammonia will remove film. |
| 65 | A 10 g ammonium persulfate 100 mL water B 10 g KCN 100 mL water | Use hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as 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 15 mL HNO ₃ 30 mL HCl | Swab 3–10 s or immerse to 2 min. |
| 67 | 10 mL perchloric acid 10 mL 2-butoxyethanol 70 mL ethanol (95 %) 10 mL water | Precaution—Keep cool when mixing and use. Electrolytic at 30–65 V for 10–60 s. |
| 68 | 3 mL perchloric acid 35 mL 2-butoxyethanol 60 mL methanol (absolute) | Precaution—Keep cool when mixing and use. Electrolytic at 60–150 V for 5–30 s. |
| 69 | 5 mL perchloric acid 80 mL acetic acid | Precaution—Keep cool when mixing and use. Electrolytic at 20–60 V for 1–5 min. |
| 70 | 5 mL HF 2 mL AgNO ₃ (5 %) 200 mL water | Swab for 5–60 s. |
| 71 | 5 mL HF 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 gloves. |
| 72 | 10 mL HF 45 mL HNO ₃ 45 mL water | Swab for 5–20 s. |
| 73 | 20 mL HCl 25 g NaCl 65 mL water | Electrolytic etch—use carbon cathode and platinum wire connection to specimen. (a) 6 V ac for 1 min. (b) 5 V–20 V ac for 1–2 min. (c) 20 V ac for 1–2 min. For etch-polishing, use shorter times. After etching, water rinse, alcohol rinse, and dry. |
| 74 | 1–5 mL HNO ₃ 100 mL ethanol (95 %) or methanol (95 %) | Etching rate is increased, sensitivity decreased with increased percentage of HNO ₃ . (a) Immerse few seconds to a minute. (b) Immerse 5–40 s in 5 % HNO ₃ solution. To remove stain, immerse 25 s in 10 % HCl-methanol solution. (c) For Inconels and Nimonics, use 5 mL HNO ₃ solution—electrolytic at 5–10 V for 5–20 s. (d) Swab or immerse several minutes. (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 8 g CuCl ₂ 20 mL HCl 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.) |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|--|---|
| 76 | 4 g picric acid 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 5 drops HCl 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 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 5 g CuCl ₂ 30 mL water 25 mL ethanol (95 %) or methanol (95 %) | Swab few seconds to a minute. |
| 80 | 5 mL HCl 1 g picric acid 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 ₂ O ₂ . Optional (for prior austenite grain boundaries)—temper specimen at 600–900°F prior to preparation. |
| 81 | 2 g picric acid 1 g sodium tridecylbenzene sulfonate. 100 mL water | Composition given will saturate the solution with picric acid. (a) Immerse few seconds to a minute. (b) Immerse to 15 min with occasional swabbing for heavy grain boundary attack. |
| 82 | 5 g FeCl ₃ 5 drops HCl 100 mL water | Immerse 5–10 s. |
| 83 | 10 g CrO ₃ 100 mL water | (a) Electrolytic at 6 V for 5–60 s. Attacks carbides. (b) Electrolytic at 6 V for 3–5 s. |
| 84 | 10 mL H ₂ SO ₄ 10 mL HNO ₃ 80 mL water | Precaution—Add H ₂ SO 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 25 g NaOH 100 mL water | 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 4 mL H ₂ O ₂ (30 %) 100 mL water | Solution solution 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 20–50 mL HCl 30 mL glycerol | Use hood—Can give off nitrogen dioxide gas. Precaution—Mix HCl and glycerol thoroughly before adding HNO ₃ . Do not store. 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 20 mL HCl 30 mL water | Use hood—Can give off nitrogen dioxide gas. Precaution—Discard before solution attains a dark orange color. Immerse few seconds to a minute. Much stronger reaction than No. 87. |
| 89 | 10 mL HNO ₃ 10 mL acetic acid 15 mL HCl 2–5 drops glycerol | Use hood. Do not store. Immerse or swab few seconds to few minutes. |
| 90 | 10 mL HNO ₃ 20 mL HF 20–40 mL glycerol | Immerse 2–10 s. |
| 91 | 5 mL HNO ₃ 5 mL HCl 1 g picric acid 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 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 hood. Electrolytic at 0.2 A/cm ² for few seconds. |
| 94 | 2 g CuCl ₂ 40 mL HCl 40–80 mL ethanol (95 %) or methanol (95 %) | Submerged swabbing for few seconds to several minutes. Attacks ferrite more readily than austenite. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|--|--|
| 95 | 2 g CuCl ₂ 40 mL HCl 40–80 mL ethanol (95 %) or methanol (95 %) 40 mL water | Immerse or swab few seconds to few minutes. |
| 96 | 85 g NaOH 50 mL water | Electrolytic at 6 V for 5–10 s. |
| 97 | 45 g KOH 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 ₃ Fe(CN) ₄ 10 g KOH or NaOH 100 mL water | Use hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as contact. Use fresh. (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.) (b) Immerse in fresh, hot solution 2–20 min. Stains carbides dark, ferrite yellow, sigma blue. Austenite turns brown on overetching. (c) Swab 5–60 s. (Immersion will produce a stain etch). Follow with water rinse, alcohol rinse, dry. |
| 99 | 25 mL HCl 3 g ammonium bifluoride 125 mL water few grains potassium metabisulfite | <i>Mix fresh.</i> (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 90 mL water | Immerse few seconds. |
| 101 | 2 g CrO ₃ 20 mL HCl 80 mL water | 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 ₄ OH | Use hood. Electrolytic at 6 V for 30–60 s. Attacks carbides only. |
| 103 | 20 mL HNO ₃ 4 mL HCl 20 mL methanol (99 %) | Immerse 10–60 s. |
| 104 | 5 mL HNO ₃ 45 mL HCl 50 mL water | Immerse 10 min or longer. |
| 105 | 5 mL H ₂ SO ₄ 3 mL HNO ₃ 90 mL HCl | Use hood. Precaution—add H ₂ SO ₄ slowly to HCl with stirring, cool; then add HNO ₃ . Discard when dark orange color. Swab 10–30 s. |
| 106 | 7 mL HNO ₃ 25 mL HCl 10 mL methanol (99 %) | Use fresh to avoid pitting. Immerse or swab 10–60 s. |
| 107 | 10 mL H ₃ PO ₄ 50 mL H ₂ SO ₄ 40 mL HNO ₃ | Use hood. Precaution—Mix H ₃ PO ₄ and HNO ₃ thoroughly, then add H ₂ SO 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 ₂ SO ₄ 100 mL water | Electrolytic at 6 V for 5–10 s. Tends to pit with longer times. |
| 109 | 50 mL HCl 25 mL HNO ₃ 1 g CuCl ₂ 150 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 5 mL HNO ₃ 85 mL ethanol (95 %) or methanol (95 %) | Immerse to several minutes until deeply etched. Follow with light repolish. |
| 111 | 5 mL H ₂ SO ₄ 8 g CrO ₃ 85 mL H ₃ PO ₄ | 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. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|--|--|
| 112 | 60 mL acetic acid 30 mL H ₂ O ₂ (30 %) | Immerse 8–15 s. |
| 113 | 15 mL acetic acid 15 mL HNO ₃ 60 mL glycerol | Do not store. Use fresh solution at 80°C (176°F). |
| 114 | 15 mL acetic acid 20 mL HNO ₃ 80 mL water | 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. |
| 115 | 100 mL acetic acid 10 mL H ₂ O ₂ (30 %) | 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 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. (b) Immerse for ½–2 min. |
| 118 | 1 mL HNO ₃ 75 mL diethylene glycol 25 mL water | Swab 3–5 s for F and T6, 1–2 min for T4 and O temper. |
| 119 | 1 mL HNO ₃ 20 mL acetic acid 60 mL diethylene glycol 20 mL water | Swab 1–3 s for F and T6, 10 s for T4 and O temper. |
| 120 | 10 mL HF 90 mL water | Immerse with gentle agitation 3–30 s. |
| 121 | 0.7 mL H ₃ PO ₄ 4 g picric acid 100 mL ethanol (95 %) or methanol (95 %) | Composition critical. (a) Immerse with gentle agitation 10–30 s. (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 100 mL water | Swab. |
| 123 | 60 mL H ₃ PO ₄ 100 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 10 mL water 6 g picric acid 100 mL ethanol (95 %) or methanol (95 %) | Immerse with gentle agitation 10–60 s. |
| 125 | 10 mL acetic acid 6 g picric acid 100 mL ethanol (95 %) or methanol (95 %) | Immerse with gentle agitation 15–30 s. |
| 126 | 30 mL acetic acid 15 mL water 6 g picric acid 100 mL ethanol (95 %) or methanol (95 %) | Immerse with gentle agitation 1–30 s. |
| 127 | 20 mL acetic acid 20 mL water 3 g picric acid 50 mL ethanol (95 %) or methanol (95 %) | Immerse with gentle agitation 5–30 s. |
| 128 | 8 mL HF 5 mL HNO ₃ 200 mL water | Immerse with gentle agitation 5–15 s. |
| 129 | 10 mL HF 30 mL HNO ₃ 60 mL lactic acid | Swab 10–20 s. Vary HF to increase or decrease activity. |
| 130 | 25 mL HCl 75 mL methanol | Caution—Keep below 24°C (75°F). Electrolytic at 30 V for 30 s. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|---|---|
| 131 | 5 mL H ₂ SO ₄ 1 mL HF 100 mL methanol (95 %) | Electrolytic at 50–60 V for 10–20 s. |
| 132 | 5 mL HF 10 mL HNO ₃ 50 mL lactic acid | Use fresh. (a) Swab with heavy pressure for 5–10 s. Water rinse, alcohol rinse, dry, then etch with No. 98c. (b) Swab for 5–30 s. |
| 133 | 50 mL HNO ₃ 50 mL acetic acid | Use hood. Do not store. <i>Mix fresh.</i> Immerse or swab 5 to 30 s. Will chemically polish with longer times. Sulfidized grain boundaries etched before normal grain boundaries. |
| 134 | 70 mL H ₃ PO ₄ 30 mL water | Electrolytic 5–10 V for 5–60 s. (Polishes at high currents.) |
| 135 | 80 mL HNO ₃ 3 mL HF | Use hood. Warm specimen in boiling water prior to immersion for 10 to 120 s. |
| 136 | 20 mL H ₃ PO ₄ 80 mL water | Electrolytic at 10–20 V for 10–15 s. |
| 137 | 10 g NaNO ₃ 100 mL water | Electrolytic, 0.2 A/cm ² , 1 min. |
| 138 | 5 g FeCl ₃ 2 mL HCl 100 mL ethanol (95 %) or methanol (95 %) | Swab 10–60 s. |
| 139 | 5 g KCN 100 mL water 0.5 mL H ₂ O ₂ (3 %) | Use hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as contact. Immerse 10–100 s. |
| 140 | 50 mL acetic acid 50 mL HNO ₃ 50 mL acetone | Use hood. Do not store. Decomposes with possible explosion on standing. Immerse 10–30 s. |
| 141 | 3 g NH ₄ Cl 3 g CrO ₃ 10 mL HNO ₃ 90 mL water | Swab 5–30 s. Do not store. |
| 142 | 5 mL HF 10 mL glycerol 85 mL water | Electrolytic at 2–3 V for 2–10 s. |
| 144 | A 10 g sodium thiosulfate 100 mL water B 10 mL HCl 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 ₂ SO ₄ 100 mL water | Electrolytic at 3–10 V for 5–15 s. Use platinum wires. H ₂ SO ₄ may be increased to 20 mL for deeper attack. |
| 146 | 10 mL HF 100 mL HNO ₃ | Immerse 30 s–3 min. |
| 147 | 20 mL HNO ₃ 80 mL HCl | Immerse 5–30 s. |
| 148 | 5 mL HNO ₃ 100 mL water | Immerse 10–30 s. |
| 149 | 50 mL HCl 2 mL H ₂ O ₂ (30 %) 50 mL water | Immerse 10–30 s. Do not store. |
| 150 | 60 mL HCl 20 mL HNO ₃ 40 mL glycerol | Use hood. Do not store. Swab few seconds to a minute. Discard when solution turns dark yellow. |
| 151 | 10 mL HF 25 mL HNO ₃ 150 mL water | Swab 5–30 s. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|---|---|
| 152 | 85 mL NH ₄ OH 15 mL H ₂ O ₂ (30 %) | Immerse 5–15 s. Do not store—Decomposes. |
| 153 | 10 mL HNO ₃ 50 mL HCl 60 mL glycerol | Use hood. Do not store. Add HNO ₃ last. Discard when dark yellow. Immerse 10–60 s. Preheating specimen in boiling water hastens reaction. |
| 154 | 50 mL HCl 50 mL ethanol (95 %) or methanol (95 %) | Immerse 10–100 s. |
| 155 | 3 mL selenic acid 10 mL HCl 100 mL ethanol (95 %) or methanol (95 %) | 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. |
| 156 | 1 g thiourea 1 mL H ₃ PO ₄ 1000 mL water | Electrolytic, 0.005–0.01 A/cm ² , 1–2 min. |
| 157 | 25 g CrO ₃ 150 mL HCl 50 mL water | Immerse 5–20 s. |
| 158 | 10 mL HF 10 mL HNO ₃ 20 mL glycerol | Swab 5–15 s. |
| 159 | 5 mL HF 20 mL HNO ₃ 50 mL acetic acid | Swab 10–30 s. |
| 160 | 20 mL HF 15 mL H ₂ SO ₄ 5 mL HNO ₃ 50 mL water | Immerse to 5 min. |
| 161 | 25 mL HNO ₃ 5 mL HF | Immerse 5–120 s. |
| 162 | A 50 mL lactic acid 30 mL HNO ₃ 2 mL HF B 30 mL lactic acid 10 mL HNO ₃ 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. |
| 163 | 30 mL H ₂ SO ₄ 30 mL HF 3–5 drops H ₂ O ₂ (30 %) 30 mL water | Immerse 5–60 s. Use this solution for alternate etch and polishing. |
| 164 | 50 mL HNO ₃ 30 g ammonium bifluoride 20 mL water | Use hood. Swab 3–10 s. |
| 165 | 10 mL HCl 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. (b) Electrolytic at 6 V for 10 s. Use stainless steel cathode and platinum or Nichrome wire contact to specimen. |
| 166 | A 20 g ammonium persulfate 90 mL water B 20 g KCN 90 mL water | Use hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as 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 100 mL water | Use hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as contact. Electrolytic at 1–5 V ac for 1–2 min. Use platinum cathode. |
| 168 | 20 mL HCl 35 g NaCl 80 mL water | Composition given will saturate the solution with NaCl. Electrolytic at 1½ V ac for 1 min. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|--|--|
| 169 | 5 mL HNO ₃ 50 mL ethylene glycol 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 30 mL HNO ₃ 30 mL lactic acid | (a) Swab 5–30 s. Follow with water rinse, alcohol rinse, dry. (b) Swab for 10 s intervals. Increase HF to exaggerate grain boundaries. |
| 171 | concentrated HCl | Use 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 5 g ammonium persulfate 100 mL water B 5 g KCN 100 mL water | Use hood—Can give off extremely poisonous hydrogen cyanide. Precaution—Also poisonous by ingestion as well as 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 10–30 mL H ₂ O ₂ (50 %) | Immerse few seconds to a minute. |
| 174 | A 25 mL HNO ₃ 1 g K ₂ Cr ₂ O ₇ 1000 mL water B 40 g CrO ₃ 3 g Na ₂ SO ₄ 200 mL water | 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 1 mL H ₂ SO ₄ 1000 mL water | Immerse to 1 min. |
| 176 | 2 g FeCl ₃ 100 mL water | Immerse 5–30 s. |
| 177 | 10 g NaOH 100 mL water | Swab or immerse 5–15 s. |
| 178 | 20 mL HF 20 mL HNO ₃ 60 mL lactic acid | Swab for 5–20 s. |
| 179 | A 10 mL HF 10 mL HNO ₃ 30 mL lactic acid B 10 mL HF 90 mL H ₂ SO ₄ | Use 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 V/in. ² of specimen. Use carbon cathode and platinum wire connection to specimen. Discard Solution B after 1 hr. |
| 180 | 10 mL HNO ₃ 30 mL acetic acid 50 mL glycerol | Immerse for ½–10 min at 38 to 42°C (100–108°F). |
| 181 | 2 mL HCl 100 mL ethanol (95 %) or methanol (95 %) | Swab for 1–3 min. |
| 182 | 10 mL HNO ₃ 10 mL acetic acid 80 mL glycerol | Immerse for ½–10 min at 38 to 42°C (100–108°F). |
| 183 | 2 drops HF 1 drop HNO ₃ 25 mL glycerol | Immerse for 1 min. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|---|--|
| 184 | 10 g FeCl ₃ 2 mL HCl 100 mL water | Immerse for ½ –5 min. |
| 185 | 10 mL HF 10 mL HNO ₃ | Swab for few seconds. |
| 186 | 10 mL HF 5 mL HNO ₃ 85 mL water | Swab 3–20 s. |
| 187 | 10 mL HF 30 mL HNO ₃ 50 mL water | Swab 3–20 s. |
| 188 | 1 mL HF 2 mL HNO ₃ 50 mL H ₂ O ₂ (30 %) 50 mL water | Swab until stain is removed. |
| 189 | 10 mL HF 25 mL HNO ₃ 45 mL glycerol 20 mL water | Swab 3–20 s. |
| 190 | 8 g KOH 10 mL H ₂ O ₂ (30 %) 60 mL water | Swab 3–20 s. |
| 191 | 25 mL HF 18 g benzalkonium chloride 35 mL methanol (95 %) 40 mL glycerol | Swab 3–20 s. |
| 192 | 1–3 mL HF 2–6 mL HNO ₃ 100 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 1 drop HNO ₃ 3 mL HCl 25 mL glycerol | Swab 3–20 s. |
| 194 | 20 mL HF 20 mL HNO ₃ 60 mL glycerol | Immerse 5–30 s. |
| 195 | 30 mL H ₃ PO ₄ 30 mL ethylene glycol 50 mL ethanol (95 %) | Electrolytic at 18–20 V (0.03 A/cm ²) for 5–15 min. |
| 196 | 18 g CrO ₃ 75 mL acetic acid 20 mL water | 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 100 mL water | Electrolytic at 6 V for 5–20 s. |
| 198 | 30 mL HF 30 mL HNO ₃ 30 mL glycerol | Swab for 60 s. |
| 199 | 2 mL HF 5 g AgNO ₃ 100 mL water | Swab for 5 s. |
| 200 | A 40 g CrO ₃ 3 g Na ₂ SO ₄ 200 mL water B 40 g CrO ₃ 200 mL water | Immerse in Solution A with gentle agitation for several seconds. Rinse in Solution B. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|--|--|
| 201 | A 40 g CrO ₃ 1.5 g Na ₂ SO ₄ 200 mL water B 40 g CrO ₃ 200 mL water | Immerse in Solution A with gentle agitation for several seconds. Rinse in Solution B. |
| 202 | A 10 g CrO ₃ 1 g Na ₂ SO ₄ 200 mL water B 40 g CrO ₃ 200 mL water | Immerse in Solution A for 2–5 s. Rinse in Solution B. |
| 203 | 20 g CrO ₃ 100 mL water | Electrolytic at 0.2 A/cm ² for 5 s. |
| 204 | 10 mL perchloric acid 10 mL glycerol 70 mL ethanol (95 %) 10 mL water | Precaution—Keep cool when mixing and use. Electrolytic at 15–50 V for 15–60 s. |
| 205 | 5 mL HF 2 mL AgNO ₃ (5 %) 100 mL water | Swab vigorously for 10–60 s. Wet cotton frequently. |
| 206 | 5 mL HF 10 mL HNO ₃ 100 mL glycerol | Precaution—Discard after use. Solution decomposes on standing. Electrolytic at 9–12 V for 1–10 min. |
| 207 | 30 mL HNO ₃ 30 mL acetic acid 30 mL water | Swab for 5–30 s. |
| 208 | 1 mL NH ₄ OH 3 g ammonium persulfate 100 mL water | Immerse or swab few seconds to a minute. |
| 209 | 15 mL HNO ₃ 3 mL HF 80 mL water | Immerse 5–60 s. |
| 210 | 50 mL water (cold) saturated with sodium thiosulfate 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 10 g sodium thiosulfate 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. |
| 212 | 10–50 % HCl in water 0.5–1.0 g potassium metabisulfite per 100 mL of aqueous HCl solution Optional: 1 g CuCl ₂ 1–3 g FeCl ₃ 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 0.5–3 mL selenic acid 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 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 30 g citric acid 24 g lead acetate 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 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. |

TABLE 2 *Continued*

| Etchant | Composition | Procedure |
|---------|--|---|
| 217 | 5 g ammonium bifluoride 100 mL water | Mix fresh, use plastic coated tongs and polyethylene beaker. Immerse until surface is colored. |
| 218 | 3 g ammonium bifluoride 4 mL HCl 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 40 mL water | Electrolytic etch, does not reveal twins in γ 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 100 mL water | Electrolytic etch, colors δ -ferrite in stainless steels. Use at 2–20 V dc, 5–20 s, stainless steel cathode. If δ is not colored, increase NaOH to 40 g. |
| 221 | 50 mL water 50 mL ethyl alcohol 50 mL methyl alcohol 50 mL HCl 1 g CuCl ₂ 2.5 g FeCl ₃ 2.5 mL HNO ₃ | Use by immersion. Will not attack sulfides in stainless steels. |
| 222 | 8 g Na ₂ SO ₄ 100 mL water | (a) Few seconds to 1 minute. (b) Pre-etch 2 s in No. 74, rinse, and etch 20 s. |
| 223 | A 8 g oxalic acid 5 mL H ₂ SO ₄ 100 mL water B 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 %) 20 mL 10 % aqueous NaOH | Immerse 10 s at 20°C (68°F). |
| 225 | 4 g NaOH 100 mL saturated aqueous KMnO ₄ | Immerse 10 s at 20°C (68°F). |
| 226 | 15 mL HCl 10 mL acetic acid 5 mL HNO ₃ 2 drops glycerol | Use hood —Can give off nitrogen dioxide gas. Precaution —Mix HCl and glycerol thoroughly before adding HNO ₃ . Do not store. 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. |

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

TABLE 3 Etchant Names

| Common Name | No. | Common Name | No. |
|-------------------------|------------------|--------------|----------|
| Acetic glycergia | 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 |
| Glycergia | 87 | Ralph's | 221 |
| Gorsuch | 75 | Super Picral | 77 |
| Grard's No. | 135 | Vilella's | 80 |
| Green contrast | 94 | 92-5-3 | 105 |

6.2.8 Properly dispose of all solutions that are not identified

by composition and concentration.

6.2.9 Store, handle and dispose of chemicals according to the manufacturer's recommendations. Observe printed cautions on reagent bottles.

6.2.10 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 (1-9).⁴

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 (10).

7.2 Reagent grade chemicals shall be used for all etchants. Unless otherwise indicated, it is intended that all reagents conform to specifications of the Committee on Analytical Reagents of the American Chemical Society where such

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

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 D 1193. 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 (**9, 11-13**) 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_4FHF). 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 (**9, 11-14**). A light pre-etch, or a slight amount of polishing relief, may lead to sharper delineation of the constituents after vapor deposition. The deposition is conducted inside a vacuum evaporator of the type used to prepare replicas for electron microscopy. One or several small lumps of a suitable dielectric compound with the desired index of refraction is heated under a vacuum until it evaporates. A vacuum level of 1.3 to 0.013 Pa (10^{-3} to 10^{-5} mm Hg) is adequate and the polished surface should be about 10–15 cm beneath the device that holds the dielectric compound. Slowly evaporate the lumps and observe the surface of the specimen. It may be helpful to place the specimen on a small piece of white paper. As the film thickness increases, the surface (and the paper) will become colored with the color sequence changing in the order yellow, green, red, purple, violet, blue, silvery blue. Stop the evaporation when the color is purple to violet, although in some cases, thinner films with green or red colors have produced good results.

7.15 Metals Handbook (**15**) 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

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