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Standard Guide for Electrolytic Polishing of Metallographic Specimens¹

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1. Scope

1.1 This guide deals with electrolytic polishing as a means of preparation of specimens for metallographic purposes. Procedures are described for polishing a variety of metals.

NOTE 1—References (1-133)² on electrolytic polishing will provide the reader with specific information beyond the scope of this guide.

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.* Specific safety precautions are described in Section 5 and 6.3.1.

2. Referenced Documents

2.1 *ASTM Standards:*³

E 7 Terminology Relating to Metallography

E 407 Test Methods for Microetching Metals and Alloys

3. Terminology

3.1 *Definitions*—All terms used in this guide are either defined in Terminology E 7 or are discussed in 3.2.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *electrolytic polish (electropolish)*—A method of polishing metals and alloys in which material is removed from the surface by making the metal the anode in an electrolytic bath.

4. Significance and Use

4.1 *Advantages of Electrolytic Polishing:*

4.1.1 For some metals, a high quality surface finish can be produced that is equivalent to, or better than, that which can be obtained by mechanical methods.

4.1.2 Once procedures have been established, satisfactory results can be obtained rapidly with reproducibility.

4.1.3 There can be a marked saving of time if many specimens of the same material are polished sequentially.

4.1.4 Electropolishing a selected area on the surface of a relatively large metal part can be accomplished nondestructively, that is, without the need for sectioning to remove a piece.

4.1.5 Soft, single-phase metals, which may be difficult to polish by mechanical methods, may be successfully electropolished.

4.1.6 The true microstructure of a specimen can be obtained because artifacts (such as disturbed metal, scratches, and mechanical twins) produced on the surface, even by careful grinding and mechanical polishing operations, can be removed. These features are important in low-load hardness testing, X-ray diffraction studies, and in electron microscopy, where higher resolution puts a premium on undistorted metal surfaces.

4.1.7 After electropolishing is completed, etching can often be accomplished by reducing the voltage (generally to about one-tenth that required for polishing) for a short time before it is turned off.

NOTE 2—Not all electropolishing solutions produce good etching results.

4.2 *Disadvantages of Electrolytic Polishing:*

4.2.1 Many of the chemical mixtures used in electropolishing are poisonous or dangerous if not properly handled (see Section 5). These hazards are similar to those involved in the mixing and handling of etchants, see Test Methods E 407.

4.2.2 In multi-phase alloys, the polishing rate of each phase may be different. The result may be a non-planar surface.

4.2.3 Electropolished surfaces may be slightly undulated rather than perfectly planar and, therefore, may not be suitable for examination at all magnifications.

4.2.4 The rate of polishing in areas adjacent to various inhomogeneities, such as nonmetallic inclusions and voids, is usually greater than that in the surrounding matrix and tends to exaggerate the size of the inclusions and voids.

4.2.5 Dimples, pits, and waviness limit applications involving surface phenomena, coatings, interfaces, and cracks. Edges tend to be attacked preferentially, resulting in edge rounding.

4.2.6 Artifacts may be produced by electropolishing.

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² The **boldface** numbers in parentheses refer to the references at the end of this standard.

³ 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.

4.2.7 Specimen mounting materials may react with the electrolyte.

4.2.8 The electropolished surfaces of certain materials may be passive and difficult to etch.

4.2.9 Metal removal rates by electropolishing are usually quite low, typically about 1 $\mu\text{m}/\text{min}$, and all of the prior induced damage from cutting and grinding may not be removed if preparation is stopped after a 600-grit SiC grind and electropolishing times are short.

4.2.10 A large number of electrolytes may be needed to polish the variety of metals encountered by a given laboratory. Considerable time may be required to develop a procedure for a new alloy.

5. General Safety Precautions

5.1 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.

5.1.1 Consult the product labels and MSDS for recommendations concerning proper protective clothing.

5.1.2 All chemicals are potentially dangerous. All persons using any electrolyte 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.

5.1.3 Table 1 includes specific safety precautions for the mixing or use of some electrolytes. The user should take care to observe each of these specific precautions.

TABLE 1 Electrolytes for Electropolishing

Class	Use	Formula	Cell Voltage	Time	Remarks
Group I (Electrolytes Composed of Perchloric Acid and Alcohol With or Without Organic Additions)					
I-1	Al and Al alloys with less than 2 percent Si	ethanol (95 %) distilled water perchloric acid (60 %)	800 mL 140 mL 60 mL	30 to 80	15 to 60 s
	steels—carbon, alloy, stainless Pb, Pb-Sn, Pb-Sn-Cd, Pb-Sn-Sb Zn, Zn-Sn-Fe, Zn-Al-Cu Mg and high Mg alloys			35 to 65 12 to 35 20 to 60 ...	15 to 60 s 15 to 60 s ... nickel cathode
I-2	stainless steel and aluminum	ethanol (95 %) perchloric acid (60 %)	800 mL 200 mL	35 to 80	15 to 60 s
I-3	stainless steel	ethanol (95 %) perchloric acid (65 %)	940 mL 60 mL	30 to 45	15 to 60 s
I-4	steel, cast iron, Al, Al alloys, Ni, Sn, Ag, Be, Ti, Zr, U, heat-resisting alloys	ethanol (95 %) 2-butoxy ethanol perchloric acid (30 %)	700 mL 100 mL 200 mL	30 to 65	15 to 60 s
I-5	steels—stainless, alloy, high-speed; Fe, Al, Zr, Pb	ethanol (95 %) glycerin perchloric acid (30 %)	700 mL 100 mL 200 mL	15 to 50	15 to 60 s
I-6	Al, Al-Si alloys	ethanol (95 %) diethyl ether perchloric acid (30 %)	760 mL 190 mL 50 mL	35 to 60	15 to 60 s
I-7	Mo, Ti, Zr, U-Zr alloy	methanol (absolute) 2-butoxy ethanol perchloric acid (60 %)	600 mL 370 mL 30 mL	60 to 150	5 to 30 s
I-8	Al-Si alloys	methanol (absolute) glycerin perchloric acid (65 %)	840 mL 125 mL 35 mL	50 to 100	5 to 60 s
I-9	vanadium	methanol (absolute) 2-butoxy ethanol perchloric acid (65 %)	590 mL 350 mL 60 mL	30	3 s
	germanium titanium zirconium			25 to 35 58 to 66 70 to 75	30 to 60 s 45 s 15 s
I-10	aluminum	methanol (absolute) nitric acid perchloric acid (60 %)	950 mL 15 mL 50 mL	30 to 60	15 to 60 s
I-11	steels—carbon, alloy, stainless Ti, high-temperature alloys, Pb, Mo	methanol (absolute) butylcellosolve perchloric acid	600 mL 360 mL 60 mL	30–40	5–60 s
I-12	Al and Al alloys	ethanol (95 %) perchloric acid	1000 mL 200 mL	10	2 min
I-13	steel, Al, Ni, Sn, Ti, Be stainless steel Al ₃ Ni	ethanol (95 %) butylcellosolve water perchloric acid	700 mL 100 mL 137 mL 62 mL	20	20 s
I-14	Ni, Ag, or Cu alloys Cd	ethanol (95 %) butylcellosolve perchloric acid	700 mL 100 mL 200 mL	70–80	15 s

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
I-15	Mo and Mo alloys	methanol (absolute) water butylcellosolve perchloric acid	600 mL 13 mL 360 mL 47 mL		20 s	Mix methanol and water, add perchloric acid carefully. Add butylcellosolve before use.
Group II (Electrolytes Composed of Perchloric Acid and Glacial Acetic Acid in Varying Proportions)						
II-1	Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless	acetic acid (glacial) perchloric acid (60 %)	940 mL 60 mL	20 to 60	1 to 5 min	good general-purpose electrolyte
II-2	Zr, Ti, U, steel—carbon and alloy	acetic acid (glacial) perchloric acid (60 %)	900 mL 100 mL	12 to 70	0.5 to 2 min	
II-3	U, Zr, Ti, Al, steel—carbon and alloy	acetic acid (glacial) perchloric acid (60 %)	800 mL 200 mL	40 to 100	1 to 15 min	
II-4	Ni, Pb, Pb-Sb alloys	acetic acid (glacial) perchloric acid (60 %)	700 mL 300 mL	40 to 100	1 to 5 min	
II-5	3 percent Si-Fe	acetic acid (glacial) perchloric acid (60 %)	650 mL 350 mL	...	5 min	0.06 A/cm ²
II-6	Cr	acetic acid (glacial) perchloric acid	1000 mL 5 mL	30–50	2–3 min	can lower voltage to 25 V by adding 5–15 % water.
II-7	Hf, steel—carbon and alloy	acetic acid (glacial) perchloric acid	1000 mL 50 mL	...	...	Used to polish Hf wires.
Group III (Electrolytes Composed of Phosphoric Acid in Water or Organic Solvent)						
III-1	cobalt	phosphoric acid (85 %)	1000 mL	1.2	3 to 5 min	
III-2	pure copper	distilled water phosphoric acid (85 %)	175 mL 825 mL	1.0 to 1.6	10 to 40 min	copper cathode
III-3	stainless, brass, Cu and Cu alloys except Sn bronze	water phosphoric acid (85 %)	300 mL 700 mL	1.5 to 1.8	5 to 15 min	copper cathode
III-4	alpha or alpha plus beta brass, Cu-Fe, Cu-Co, Co, Cd	water phosphoric acid (85 %)	600 mL 400 mL	1 to 2	1 to 15 min	copper or stainless steel cathode
III-5	Cu, Cu-Zn	water pyrophosphoric acid	1000 mL 580 g	1 to 2	10 min	copper cathode
III-6	steel	diethylene glycol monoethyl ether phosphoric acid (85 %)	500 mL 500 mL	5 to 20	5 to 15 min	120°F
III-7	Al, Ag, Mg	water ethanol (95 %) phosphoric acid (85 %)	200 mL 380 mL 400 mL	25 to 30	4 to 6 min	aluminum cathode, 100 to 110°F
III-8	uranium	ethanol (absolute) glycerin (cp) phosphoric acid (85 %)	300 mL 300 mL 300 mL	...	...	
III-9	Mn, Mn-Cu alloys	ethanol (95 %) glycerin phosphoric acid (85 %)	500 mL 250 mL 250 mL	18	...	
III-10	Cu and Cu-base alloys	distilled water ethanol (95 %) phosphoric acid (85 %)	500 mL 250 mL 250 mL	...	1 to 5 min	
III-11	stainless steel	ethanol (absolute), to pyrophosphoric acid	1 L 400 g	...	10 min	good for all austenitic heat resistant alloys, 100°F plus
III-12	Mg-Zn	ethanol (95 %) phosphoric acid (85 %)	625 mL 375 mL	1.5 to 2.5	3 to 30 min	
III-13	uranium	ethanol (95 %) ethylene glycol phosphoric acid (85 %)	445 mL 275 mL 275 mL	18 to 20	5 to 15 min	0.03 A/cm ²
III-14	Al-Mg alloys	water ethanol (95 %) phosphoric acid (85 %)	250 mL 380 mL 400 mL	50–60	2 min	
III-15	Cu-Pb alloys	ethanol (95 %) phosphoric acid (85 %)	620 mL 380 mL			good up to 30 % Pb
III-16	Neptunium	ethanol (95 %) glycerol phosphoric acid (85 %)	400 mL 400 mL 800 mL			after 600-grit SiC, use 6-μm diamond on nylon before electropolishing.
Group IV (Electrolytes Composed of Sulfuric Acid in Water or Organic Solvent)						
IV-1	stainless steel	water sulfuric acid	250 mL 750 mL	1.5 to 6	1 to 2 min	
IV-2	stainless steel, Fe, Ni	water sulfuric acid	400 mL 600 mL	1.5 to 6	2 to 6 min	
IV-3	stainless steel, Fe, Ni, Mo	water sulfuric acid	750 mL 250 mL	1.5 to 6	2 to 10 min Mo— 0.3 to 1 min	particularly good for sintered Mo—32 to 80°F
IV-4	molybdenum	water sulfuric acid	900 mL 100 mL	1.5 to 6	0.3 to 2 min	particularly good for sintered Mo—32 to 80°F

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
IV-5	stainless steel	water glycerin sulfuric acid	70 mL 200 mL 720 mL	1.5 to 6	0.5 to 5 min	
IV-6	stainless steel, aluminum	water glycerin sulfuric acid	220 mL 200 mL 580 mL	1.5 to 12	1 to 20 min	
IV-7	molybdenum	methanol (absolute) sulfuric acid	875 mL 125 mL	6 to 18	0.5 to 1.5 min	32 to 80°F
IV-8	Ni-base superalloys	methanol (absolute) sulfuric acid	800 mL 200 mL	30	20 s	for alloy 625
Group V (Electrolytes Composed of Chromic Acid in Water)						
V-1	stainless steel	water chromic acid	830 mL 620 g	1.5 to 9	2 to 10 min	
V-2	Zn, brass	water chromic acid	830 mL 170 g	1.5 to 12	10 to 60 s	
Group VI (Mixed Acids or Salts in Water or Organic Solvent)						
VI-1	stainless steel	phosphoric acid (85 %) sulfuric acid	600 mL 400 mL	...	...	
VI-2	stainless steel	water phosphoric acid (85 %) sulfuric acid	150 mL 300 mL 550 mL	...	2 min	0.3 A/cm ²
VI-3	stainless and alloy steel	water phosphoric acid (85 %) sulfuric acid	240 mL 420 mL 340 mL	...	2 to 10 min	0.1 to 0.2 A/cm ²
VI-4	stainless steel	water phosphoric acid (85 %) sulfuric acid	330 mL 550 mL 120 mL	...	1 min	0.05 A/cm ²
VI-5	bronze (to 9 % Sn)	water phosphoric acid (85 %) sulfuric acid	450 mL 390 mL 160 mL	...	1 to 5 min	0.1 A/cm ²
VI-6	bronze (to 6 % Sn)	water phosphoric acid (85 %) sulfuric acid	330 mL 580 mL 90 mL	...	1 to 5 min	0.1 A/cm ²
VI-7	steel	water glycerin phosphoric acid (85 %) sulfuric acid	140 mL 100 mL 430 mL 330 mL	...	1 to 5 min	1 to 5 A/cm ² , 100°F plus
VI-8	stainless steel	water glycerin phosphoric acid (85 %) sulfuric acid	200 mL 590 mL 100 mL 110 mL	...	5 min	1 A/cm ² , 80 to 120°F
VI-9	stainless steel	water chromic acid phosphoric acid (85 %) sulfuric acid	260 mL 175 g 175 mL 580 mL	...	30 min	0.6 A/cm ² , 80 to 120°F
VI-10	stainless steel	water chromic acid phosphoric acid (85 %) sulfuric acid	175 mL 105 g 460 mL 390 mL	...	60 min	0.5 A/cm ² , 80 to 120°F
VI-11	stainless and alloy steel	water chromic acid phosphoric acid (85 %) sulfuric acid	240 mL 80 g 650 mL 130 mL	...	5 to 60 min	0.5 to A/cm ² , 100 to 130°F
VI-12	tantalum	hydrofluoric acid sulfuric acid	100 mL 900 mL	...	9 min	graphite cathode, 0.1 A/cm ² , 90 to 100°F
VI-13	stainless steel	water hydrofluoric acid sulfuric acid	210 mL 180 mL 610 mL	...	5 min	0.5 A/cm ² , 70 to 120°F
VI-14	zinc	water chromic acid sulfuric acid sodium dichromate acetic acid (glacial)	800 mL 100 g 46 mL 310 g 96 mL	...	...	0.002 A/cm ² , 70 to 100°F
VI-15	stainless steel	hydrogen peroxide (30 %) (Caution) hydrofluoric acid sulfuric acid	260 mL 240 mL 500 mL	...	5 min	0.5 A/cm ² (Caution) Dangerous
VI-16	stainless steel	water hydrofluoric acid sulfuric acid	520 mL 80 mL 400 mL	...	½ to 4 min	0.08 to 0.3 A/cm ²