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Standard Guide for
Electrolytic Polishing of Metallographic Specimens[1]

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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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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]
E7 Terminology Relating to Metallography
E407 Practice for Microetching Metals and Alloys

3. Terminology

3.1 *Definitions*—All terms used in this guide are either defined in Terminology E7 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 E407.

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.

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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.

4.2.5 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.

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 µm/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 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. In particular, note that solutions containing perchloric acid must be mixed and used in an exclusive hood equipped with a wash down feature to avoid accumulation of explosive perchlorates.

5.1.4 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.

5.2 Some basic suggestions for the handling and disposal of electrolytes and their ingredients are as follows:

5.2.1 As previously stated, it is good practice to always work under a certified fume hood when mixing and utilizing any electrolyte and it is imperative with those electrolytes that give off noxious odors or toxic vapor. Additionally, the electrolytes in Groups I and II must be treated with extra caution because dried perchlorates can accumulate in hood ductwork and on work surfaces creating the potential for a powerful accidental explosion. Therefore, these electrolytes must only be used in an exclusive hood equipped with a wash down feature. To avoid the accumulation of explosive, dry perchlorates, the hood should undergo a wash down cycle following each use.

5.2.2 When pouring, mixing, or using electrolytes, always use the proper protective equipment (eyewear, gloves, apron, and so forth).

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

5.2.4 When mixing electrolytes, always add reagents to the solvent unless specific instructions indicate otherwise.

5.2.5 When using an electrolyte, always avoid direct physical contact with the electrolyte and the specimen. Use tongs or some other indirect method of handling specimens.

5.2.6 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.

5.2.7 All spills should be cleaned up and disposed of properly, no matter how small the spill.

5.2.8 Properly dispose of all solutions that are not identified by composition and concentration.

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

5.2.10 Information pertaining to the toxicity hazards and working precautions of chemicals, solvents, acids, bases, and so forth, being used (such as MSDS) should be available for rapid consultation.

5.3 Many of the electrolytes in the following listing can be exceedingly dangerous if carelessly handled. The pertinent safety precautions for each class of electrolyte should be read before any electrolyte is mixed or used.

5.4 Electrolytes containing perchloric acid and acetic anhydride are very dangerous to mix and may be unpredictable in use. Many industrial firms and research laboratories forbid the use of such mixtures. Certain cities also have ordinances prohibiting the use of such potentially explosive mixtures. These facts are considered sufficient reason for recommending against their use.

5.5 Mixtures of oxidizable organic compounds and powerful oxidizing agents are always potentially dangerous. After some use, any electrolyte will become heavily laden with ions of the metals polished. These ions may interfere with further polishing or catalyze the decomposition of the electrolyte. The electrolyte then must be discarded in accordance with appropriate regulations.

5.6 Most electrolytes (with few exceptions) should be mixed and stored in clean glass containers and never be in contact with foreign materials or organic compounds. The exceptions are those electrolytes containing fluorides and strong alkaline solutions that should be mixed and stored in polyethylene or other appropriate material containers. Electrolytes must never be allowed to become concentrated by evaporation. All electrolytes should be discarded appropriately as soon as they have exceeded their immediate usefulness.

5.7 Specimens mounted in bismuth or bismuth-containing metals must not be electropolished in perchloric acid solutions because this mounting medium may react explosively with the electrolyte. Likewise, bismuth or bismuth-containing alloys must not be electropolished in solutions containing perchloric

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) Use in a washdown/perchloric rated fume hood.						
I-1	Al and Al alloys with less than 2 percent Si	ethanol (95 %)	800 mL	30 to 80	15 to 60 s	
		distilled water	140 mL			
		perchloric acid (60 %)	60 mL			
	steels—carbon, alloy, stainless			35 to 65	15 to 60 s	
	Pb, Pb-Sn, Pb-Sn-Cd, Pb-Sn-Sb			12 to 35	15 to 60 s	
	Zn, Zn-Sn-Fe, Zn-Al-Cu			20 to 60	...	
	Mg and high Mg alloys			...	...	nickel cathode
I-2	stainless steel and aluminum	ethanol (95 %)	800 mL	35 to 80	15 to 60 s	
		perchloric acid (60 %)	200 mL			
I-3	stainless steel	ethanol (95 %)	940 mL	30 to 45	15 to 60 s	
		perchloric acid (65 %)	60 mL			
I-4	steel, cast iron, Al, Al alloys, Ni, Sn, Ag, Be, Ti, Zr, U, heat-resisting alloys	ethanol (95 %)	700 mL	30 to 65	15 to 60 s	one of the best formulas for universal use
		2-butoxy ethanol	100 mL			
		perchloric acid (30 %)	200 mL			
I-5	steels—stainless, alloy, high-speed; Fe, Al, Zr, Pb	ethanol (95 %)	700 mL	15 to 50	15 to 60 s	universal electrolyte comparable to I-4
		glycerin	100 mL			
		perchloric acid (30 %)	200 mL			
I-6	Al, Al-Si alloys	ethanol (95 %)	760 mL	35 to 60	15 to 60 s	particularly good with Al-Si alloys
		diethyl ether	190 mL			
		perchloric acid (30 %)	50 mL			
I-7	Mo, Ti, Zr, U-Zr alloy	methanol (absolute)	600 mL	60 to 150	5 to 30 s	
		2-butoxy ethanol	370 mL			
		perchloric acid (60 %)	30 mL			
I-8	Al-Si alloys	methanol (absolute)	840 mL	50 to 100	5 to 60 s	
		glycerin	125 mL			
		perchloric acid (65 %)	35 mL			
I-9	vanadium	methanol (absolute)	590 mL	30	3 s	three-second cycles repeated at least seven times to prevent heating
		2-butoxy ethanol	350 mL			
		perchloric acid (65 %)	60 mL			
	germanium			25 to 35	30 to 60 s	
	titanium			58 to 66	45 s	polish only
	zirconium			70 to 75	15 s	polish and etch simultaneously
I-10	aluminum	methanol (absolute)	950 mL	30 to 60	15 to 60 s	
		nitric acid	15 mL			
		perchloric acid (60 %)	50 mL			
I-11	steels—carbon, alloy, stainless Ti, high-temperature alloys, Pb, Mo	methanol (absolute)	600 mL	30–40	5–60 s	good all purpose electropolish
		butylcellosolve	360 mL			
		perchloric acid	60 mL			
I-12	Al and Al alloys	ethanol (95 %)	1000 mL	10	2 min	not good for Al-Cu and Al-Si alloys. Black film forms. Peel off after 1–1.5 min and polish 1 min more.
		perchloric acid	200 mL			
I-13	steel, Al, Ni, Sn, Ti, Be stainless steel	ethanol (95 %)	700 mL	20	20 s	Mix ethanol and water, add perchloric acid carefully. Then, add butylcellosolve before use.
		butylcellosolve	100 mL			
	Al_3 Ni	water	137 mL			
		perchloric acid	62 mL			
I-14	Ni, Ag, or Cu alloys	ethanol (95 %)	700 mL	70–80	15 s	
	Cd	butylcellosolve	100 mL			
		perchloric acid	200 mL			
I-15	Mo and Mo alloys	methanol (absolute)	600 mL		20 s	Mix methanol and water, add perchloric acid carefully. Add butylcellosolve before use.
		water	13 mL			
		butylcellosolve	360 mL			
		perchloric acid	47 mL			
Group II (Electrolytes Composed of Perchloric Acid and Glacial Acetic Acid in Varying Proportions) Use in a washdown/perchloric rated fume hood.						
II-1	Cr, Ti, Zr, U, Fe, steel—carbon, alloy, stainless	acetic acid (glacial)	940 mL	20 to 60	1 to 5 min	good general-purpose electrolyte
		perchloric acid (60 %)	60 mL			
II-2	Zr, Ti, U, steel—carbon and alloy	acetic acid (glacial)	900 mL	12 to 70	0.5 to 2 min	
		perchloric acid (60 %)	100 mL			
II-3	U, Zr, Ti, Al, steel—carbon and alloy	acetic acid (glacial)	800 mL	40 to 100	1 to 15 min	
		perchloric acid (60 %)	200 mL			
II-4	Ni, Pb, Pb-Sb alloys	acetic acid (glacial)	700 mL	40 to 100	1 to 5 min	
		perchloric acid (60 %)	300 mL			
II-5	3 percent Si-Fe	acetic acid (glacial)	650 mL	...	5 min	0.06 A/cm^2
		perchloric acid (60 %)	350 mL			
II-6	Cr	acetic acid (glacial)	1000 mL	30–50	2–3 min	can lower voltage to 25 V by adding 5–15 % water.
		perchloric acid	5 mL			
II-7	Hf, steel—carbon and alloy	acetic acid (glacial)	1000 mL	...	...	Used to polish Hf wires.
		perchloric acid	50 mL			
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	175 mL	1.0 to 1.6	10 to 40 min	copper cathode
		phosphoric acid (85 %)	825 mL			
III-3	stainless, brass, Cu and Cu alloys except Sn bronze	water	300 mL	1.5 to 1.8	5 to 15 min	copper cathode
		phosphoric acid (85 %)	700 mL			

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
III-4	alpha or alpha plus beta brass, Cu-Fe, Cu-Co, Co, Cd	water	600 mL	1 to 2	1 to 15 min	copper or stainless steel cathode
		phosphoric acid (85 %)	400 mL			
III-5	Cu, Cu-Zn	water	1000 mL	1 to 2	10 min	copper cathode
		pyrophosphoric acid	580 g			
III-6	steel	diethylene glycol monoethyl ether	500 mL	5 to 20	5 to 15 min	49°C
		phosphoric acid (85 %)	500 mL			
III-7	Al, Ag, Mg	water	200 mL	25 to 30	4 to 6 min	aluminum cathode, 38 to 43°C
		ethanol (95 %)	380 mL			
		phosphoric acid (85 %)	400 mL			
III-8	uranium	ethanol (absolute)	300 mL	...	...	
		glycerin (cp)	300 mL			
		phosphoric acid (85 %)	300 mL			
III-9	Mn, Mn-Cu alloys	ethanol (95 %)	500 mL	18	...	
		glycerin	250 mL			
		phosphoric acid (85 %)	250 mL			
III-10	Cu and Cu-base alloys	distilled water	500 mL	...	1 to 5 min	
		ethanol (95 %)	250 mL			
		phosphoric acid (85 %)	250 mL			
III-11	stainless steel	ethanol (absolute), to	1 L	...	10 min	good for all austenitic heat resistant alloys, 38°C plus
		pyrophosphoric acid	400 g			
III-12	Mg-Zn	ethanol (95 %)	625 mL	1.5 to 2.5	3 to 30 min	
		phosphoric acid (85 %)	375 mL			
III-13	uranium	ethanol (95 %)	445 mL	18 to 20	5 to 15 min	0.03 A/cm^2
		ethylene glycol	275 mL			
		phosphoric acid (85 %)	275 mL			
III-14	Al-Mg alloys	water	250 mL	50–60	2 min	
		ethanol (95 %)	380 mL			
		phosphoric acid (85 %)	400 mL			
III-15	Cu-Pb alloys	ethanol (95 %)	620 mL			good up to 30 % Pb
		phosphoric acid (85 %)	380 mL			
III-16	Neptunium	ethanol (95 %)	400 mL			after P1200–grit SiC, use 6-µm diamond on nylon before electropolishing.
		glycerol	400 mL			
		phosphoric acid (85 %)	800 mL			
Group IV (Electrolytes Composed of Sulfuric Acid in Water or Organic Solvent)						
IV-1	stainless steel	water	250 mL	1.5 to 6	1 to 2 min	
		sulfuric acid	750 mL			
IV-2	stainless steel, Fe, Ni	water	400 mL	1.5 to 6	2 to 6 min	
		sulfuric acid	600 mL			
IV-3	stainless steel, Fe, Ni, Mo	water	750 mL	1.5 to 6	2 to 10 min Mo—0.3 to 1 min	particularly good for sintered Mo—0 to 27°C
		sulfuric acid	250 mL			
IV-4	molybdenum	water	900 mL	1.5 to 6	0.3 to 2 min	particularly good for sintered Mo—0 to 27°C
		sulfuric acid	100 mL			
IV-5	stainless steel	water	70 mL	1.5 to 6	0.5 to 5 min	
		glycerin	200 mL			
		sulfuric acid	720 mL			
IV-6	stainless steel, aluminum	water	220 mL	1.5 to 12	1 to 20 min	
		glycerin	200 mL			
		sulfuric acid	580 mL			
IV-7	molybdenum	methanol (absolute)	875 mL	6 to 18	0.5 to 1.5 min	0 to 27°C
		sulfuric acid	125 mL			
IV-8	Ni-base superalloys	methanol (absolute)	800 mL	30	20 s	for alloy 625
		sulfuric acid	200 mL			
Group V (Electrolytes Composed of Chromic Acid in Water)						
V-1	stainless steel	water	830 mL	1.5 to 9	2 to 10 min	
		chromic acid	620 g			
V-2	Zn, brass	water	830 mL	1.5 to 12	10 to 60 s	
		chromic acid	170 g			
Group VI (Mixed Acids or Salts in Water or Organic Solvent)						
VI-1	stainless steel	phosphoric acid (85 %)	600 mL	...	...	
		sulfuric acid	400 mL			
VI-2	stainless steel	water	150 mL	...	2 min	0.3 A/cm^2
		phosphoric acid (85 %)	300 mL			
		sulfuric acid	550 mL			
VI-3	stainless and alloy steel	water	240 mL	...	2 to 10 min	0.1 to 0.2 A/cm^2
		phosphoric acid (85 %)	420 mL			
		sulfuric acid	340 mL			
VI-4	stainless steel	water	330 mL	...	1 min	0.05 A/cm^2
		phosphoric acid (85 %)	550 mL			
		sulfuric acid	120 mL			
VI-5	bronze (to 9 % Sn)	water	450 mL	...	1 to 5 min	0.1 A/cm^2
		phosphoric acid (85 %)	390 mL			
		sulfuric acid	160 mL			

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
VI-6	bronze (to 6 % Sn)	water	330 mL	...	1 to 5 min	0.1 A/cm^2
		phosphoric acid (85 %)	580 mL			
		sulfuric acid	90 mL			
VI-7	steel	water	140 mL	...	1 to 5 min	1 to 5 A/cm^2, 38°C plus
		glycerin	100 mL			
		phosphoric acid (85 %)	430 mL			
		sulfuric acid	330 mL			
VI-8	stainless steel	water	200 mL	...	5 min	1 A/cm^2, 27 to 49°C
		glycerin	590 mL			
		phosphoric acid (85 %)	100 mL			
		sulfuric acid	110 mL			
VI-9	stainless steel	water	260 mL	...	30 min	0.6 A/cm^2, 27 to 49°C
		chromic acid	175 g			
		phosphoric acid (85 %)	175 mL			
		sulfuric acid	580 mL			
VI-10	stainless steel	water	175 mL	...	60 min	0.5 A/cm^2, 27 to 49°C
		chromic acid	105 g			
		phosphoric acid (85 %)	460 mL			
		sulfuric acid	390 mL			
VI-11	stainless and alloy steel	water	240 mL	...	5 to 60 min	0.5 to A/cm^2, 38 to 54°C
		chromic acid	80 g			
		phosphoric acid (85 %)	650 mL			
		sulfuric acid	130 mL			
VI-12	tantalum	hydrofluoric acid	100 mL	...	9 min	graphite cathode, 0.1 A/cm^2, 32 to 38°C
		sulfuric acid	900 mL			
VI-13	stainless steel	water	210 mL	...	5 min	0.5 A/cm^2, 21 to 49°C
		hydrofluoric acid	180 mL			
		sulfuric acid	610 mL			
VI-14	zinc	water	800 mL	...	...	0.002 A/cm^2, 21 to 49°C
		chromic acid	100 g			
		sulfuric acid	46 mL			
		sodium dichromate	310 g			
		acetic acid (glacial)	96 mL			
VI-15	stainless steel	hydrogen peroxide (30 %) **(Caution)**	260 mL	...	5 min	0.5 A/cm^2 **(Caution) Dangerous**
		hydrofluoric acid	240 mL			
		sulfuric acid	500 mL			
VI-16	stainless steel	water	520 mL	...	½ to 4 min	0.08 to 0.3 A/cm^2
		hydrofluoric acid	80 mL			
		sulfuric acid	400 mL			
VI-17	stainless steel	water	600 mL	...	...	
		chromic acid	180 g			
		nitric acid	60 mL			
		hydrochloric acid	3 mL			
		sulfuric acid	240 mL			
VI-18	bismuth	glycerin	750 mL	12	1 to 5 min	0.5 ± A/cm^2 **(Caution)** This mixture will decompose vigorously after a short time. Do not try to keep.
		acetic acid (glacial)	125 mL			
		nitric acid	125 mL			
VI-19	magnesium	ethylene-glycol-monoethyl ether	900 mL	50 to 60	10 to 30 s	Bath should be stirred. Cool with cracked ice below 2°C
		hydrochloric acid	100 mL			
VI-20	molybdenum, sintered and cast	methanol (absolute)	685 mL	19 to 35	20 to 35 s	Mix slowly. Heat is developed. Avoid contamination with water. Use below 2°C.
		hydrochloric acid	225 mL			
		sulfuric acid	90 mL			
Group VI (Mixed Acids or Salts in Water or Organic Solvent)— *Continued*						
VI-21	titanium	ethanol (95 %)	900 mL	30 to 60	1 to 6 min	**(Caution)** Anhydrous aluminum chloride is extremely dangerous to handle.
		n-butyl alcohol	100 mL			
		aluminum chloride (anhydrous) (add very slowly) **(Caution)**	60 g			
		zinc chloride (anhydrous)	250 g			
VI-22	uranium	acetic acid (glacial)	750 mL	80	5 to 30 min	The chromic acid is dissolved in the water before adding to the acetic acid. Use below 2°C.
		distilled water	210 mL			
		chromic acid	180 g			
VI-23	pure zinc	ethanol (95 %)	720 mL	25 to 40	0.5 to 3 min	**(Caution)** Anhydrous aluminum chloride is extremely dangerous to handle. Use below 16°C.
		aluminum chloride (anhydrous) **(Caution)**	50 g			
		zinc chloride (anhydrous)	225 g			
		distilled water	160 mL			
		n-butyl alcohol	80 mL			
VI-24	zirconium. Polish and etch simultaneously	glycerin **(Caution)**	870 mL	9 to 12	1 to 10 min	**(Caution)** will decompose on standing, dangerous if kept too long
		hydrofluoric acid	43 mL			
		nitric acid	87 mL			
VI-25	bismuth	saturated solution KI in distilled water	980 mL	7	30 s	polish 30 s but allow to remain in electrolyte until brown film is dissolved
		hydrochloric acid	20 mL			

TABLE 1 *Continued*

Class	Use	Formula		Cell Voltage	Time	Remarks
VI-26	Sb	methanol (absolute) sulfuric acid hydrochloric acid	300 mL 50 mL 30 mL	6–10	2–4 min	pure Sb. Use Pt cathode and anode lead wires. Agitate bath. Do not touch polished surface with cotton.
VI-27	Sb	ethanol (95 %) glycerol phosphoric acid sulfuric acid	30 mL 30 mL 100 mL 30 mL			good for polarized light work
VI-28	Bi	water phosphoric acid sulfuric acid	200 mL 100 mL 200 mL			good for polarized light work
VI-29	Cr	water phosphoric acid sulfuric acid	210 mL 640 mL 150 mL	18		stir bath or specimen
VI-30	Ge	methanol (absolute) hydrochloric acid	1000 mL 10 mL			
VI-31	Nb	water sulfuric acid hydrofluoric acid	300 mL 100 mL 100 mL	40		polish to α-alumina before electropolishing
VI-32	Nb	methanol (absolute) sulfuric acid hydrofluoric acid	940 mL 50 mL 15 mL	50–60	10 s	
VI-33	Ni-base superalloy	methanol (absolute) hydrochloric acid	170 mL 30 mL	30	20 s	for Waspaloy and IN-100 mod. Etch at 5 V for 4 s.
Group VII (Alkaline Electrolytes)						
VII-1	gold	water to potassium cyanide potassium carbonate gold chloride	1000 mL 80 g 40 g 50 g	7.5	2 to 4 min	graphite cathode
VII-2	silver	water to sodium cyanide potassium ferrocyanide	1000 mL 100 g 100 g	2.5	To 1 min	graphite cathode
VII-3	silver	water to potassium cyanide silver cyanide potassium dichromate	1000 mL 400 g 280 g 280 g	...	To 9 min	graphite cathode, 0.003 to 0.009 A/cm^2
VII-4	tungsten	water to trisodium phosphate	1000 mL 160 g	...	10 min	graphite cathode, 0.09 A/cm^2, 38 to 49°C
VII-5	tungsten, lead	water to sodium hydroxide	1000 mL 100 g	...	8 to 10 min	graphite cathode, 0.03 to 0.06 A/cm^2
VII-6	zinc, tin	water to potassium hydroxide	1000 mL 200 g	2 to 6	15 min	copper cathode, 0.1 to 0.2 A/cm^2
VII-7	W	water sodium hydroxide	1000 mL 20 g		5 min	
Group VIII (Mixture of Methyl Alcohol and Nitric Acid)						
VIII-1	Ni, Cu, Zn, Monel, brass, Ni-chrome, stainless steel	methanol (absolute) nitric acid	660 mL 330 mL	40 to 70	10 to 60 s	very useful but dangerous

acid. Specimens mounted in organic mounting compounds, such as Bakelite, must not be electropolished in electrolytes containing perchloric acid as they may also react explosively.

5.8 *Specific Safety Precautions for Each Group of Electrolytes:*

5.8.1 The electrolytes recommended for use are classified into eight groups. Their chemical components are listed in the order of mixing. This ordering has been done to prevent possibly dangerous reactions. Unless other instructions are specifically given, the electrolytes are intended to be used in the temperature range from about 18 to 27°C. Cooling may be necessary to maintain this range during use.

5.8.2 *Group I—(Electrolytes Composed of Perchloric Acid and Alcohol (Methanol or Ethanol) With or Without Organic Additions):*

5.8.2.1 These electrolytes are believed to be safe to mix and use provided the following safety precautions are followed. Use these electrolytes in an exclusive hood equipped with a wash down feature. The hood should undergo a wash down cycle following each use to avoid accumulation of explosive, dry perchlorates. Only small quantities should be mixed and stored in glass-stoppered bottles filled to capacity. Any evaporated solvents should be replaced to keep the bottle filled. Spent or exhausted polishing baths are to be promptly discarded in a manner consistent with prevailing regulations. The electrolytes are always to be protected from heat or fire.

NOTE 3—In this, and all the following formulations, the term 95 % ethanol refers to a specifically denatured alcohol which is composed of 95 parts by volume absolute ethanol and 5 parts by volume absolute methanol. In case this formulation is not available, the use of 100 % absolute ethanol is advised. Alcohol formulations containing benzene, gasoline, or other denaturing substances are likely to cause difficulties and their use is not recommended.

5.8.3 *Group II—(Electrolytes Composed of Perchloric Acid and Glacial Acetic Acid):*

5.8.3.1 Use these electrolytes in an exclusive hood equipped with a wash down feature. The hood should undergo a wash down cycle following each use to avoid accumulation of explosive, dry perchlorates. Very little heat is developed when perchloric acid is mixed with glacial acetic acid. In mixing, the perchloric acid should be added to the acetic with stirring. These mixtures are normally perfectly safe to mix and use but, nonetheless, great care should be exercised with them. Temperatures must never be allowed to exceed 29°C. They are flammable and must be guarded against fire or the evaporation of the acetic acid. Plastic parts are likely to be quickly damaged by exposure to such mixtures.

5.8.4 *Group III—(Electrolytes Composed of Phosphoric Acid in Water or Organic Solvents):*

5.8.4.1 These mixtures are generally quite easy to prepare. In mixing, they are handled exactly as a mineral acid; namely, the acid must be slowly poured into the water or solvent with constant stirring to prevent the formation of a heavy layer of acid at the bottom of the vessel. Some solid phosphoric acids are quite energetic in their combination with water, requiring extra care in their mixing.

5.8.5 *Group IV—(Electrolytes Composed of Sulfuric Acid in Water or Organic Solvents):*

5.8.5.1 The addition of sulfuric acid to water produces an extremely exothermic reaction. The acid must always be poured into the water slowly and with constant stirring. Cooling is necessary. Great care should be taken to prevent spattering. Even dilute solutions of sulfuric acid strongly attack the skin or clothing. Such solutions are also very hygroscopic. They vigorously attack most plastics. The mixtures of sulfuric acid with other inorganic acids are generally more useful as electrolytes.

5.8.6 *Group V—(Electrolytes Composed of Chromium Trioxide in Water):*

5.8.6.1 The addition of crystalline chromium trioxide (CrO_3) to water is simple, since very little heat is developed. The resulting chromic acid is a powerful oxidant. Under certain conditions it will liberate considerable quantities of free oxygen. It is generally dangerous, and possibly incendiary, in the presence of oxidizable materials. It cannot be safely mixed with most organic liquids, such as alcohols or glycerol. It can be safely mixed with certain saturated organic acids, but should not be mixed with acetic acid. Chromic acid solutions cannot be used in contact with plastic parts without their eventual destruction. Care should be taken to prevent its contact with the skin since repeated exposure to even dilute solutions of chromic acid or the chromates will cause persistent and painful ulcers that are slow to heal. Chromium trioxide is a human poison and is a carcinogen.

5.8.7 *Group VI—(Mixed Acids or Salts in Water or Organic Solutions):*

5.8.7.1 These mixtures are safe to mix and use providing the mixing is done properly. It must be remembered that in all cases, the acid is added to the solvent slowly and with constant stirring. If sulfuric acid is in the formula, it is added last and with particular care. If hydrofluoric acid or fluorides are part of a formula, polyethylene or other similar hydrofluoric acid-resistant vessels should be used. Particular care should be taken to avoid skin contact with acid fluorides since exposure to them, which may pass unnoticed at the time, may result in serious burns later. In those electrolytes containing anhydrous aluminum chloride, extreme care must be exercised. The reaction between this compound and water is almost explosive. Chromates and dichromates cannot be safely mixed with most organic liquids but can be mixed with saturated organic acids. Care should be taken to prevent contact with the skin.

5.8.8 *Group VII—(Alkaline Electrolytes):*

5.8.8.1 These mixtures can be grouped into two general categories, those containing cyanide, and those not containing cyanide.

(1) The use of cyanide by anyone not properly trained and familiar with it is extremely dangerous. Cyanides are among the quickest acting and most potent poisons likely to be encountered in the laboratory. Cyanide is so quick-acting and deadly that the administration of an antidote is usually ineffectual. Extreme care must be taken that no droplet of the solution or crystal of the salt is ever left around where it can be accidentally picked up and carried to the mouth.

(2) Solutions of the alkali hydroxides are very useful for the polishing of certain amphoteric metals. Their attack on the skin is drastic, so great care should be exercised in their use. The dissolution of alkali hydroxides, such as NaOH, in water produces substantial heat. Add the pellets to the water a little bit at a time with constant stirring until the required concentration is obtained. If the temperature becomes excessive, allow the solution to cool back to ambient before adding more hydroxide.

5.8.9 *Group VIII—(Mixtures of Methyl Alcohol and Nitric Acid):*

5.8.9.1 Nitric acid can be mixed with methanol with apparent safety (mixtures with up to about 33 % nitric acid can be safely stored). This is done by adding the acid to the alcohol with careful stirring. HNO_3 cannot be safely mixed and stored with any higher alcohol except in very dilute solutions (for example, do not store solutions of more than 3 % HNO_3 in ethanol). Under certain conditions, extremely unstable or explosive nitro compounds, azides or fulminates can be formed in alcoholic HNO_3 solutions. The spontaneous decomposition of the mixture can also be catalyzed by impurities or heat. It should always be discarded as soon as it has served its immediate purpose. Due to its dangerous nature, it should not be employed if its use can be avoided.

6. Apparatus

6.1 For the electropolishing of metal specimens in an appropriate electrolyte, a suitable electrolysis cell and a controllable power supply are needed. Simple laboratory apparatus, such as shown in Fig. 1, can be assembled to perform this function. Many such arrangements are described in the literature. There are also several commercially available models of electropolishing apparatus for either laboratory or field use.

6.2 Whenever an attempt is made to polish large surface areas, the problems of obtaining sufficient current density and cooling of the specimen and electrolyte become troublesome. An adequate volume of electrolyte should be used so that localized overheating does not occur. Supplementary cooling may be required. In general, electropolishing of areas larger than 1 cm^2 is not recommended for metallographic work because of the increased difficulties encountered, but it is possible to polish larger areas.

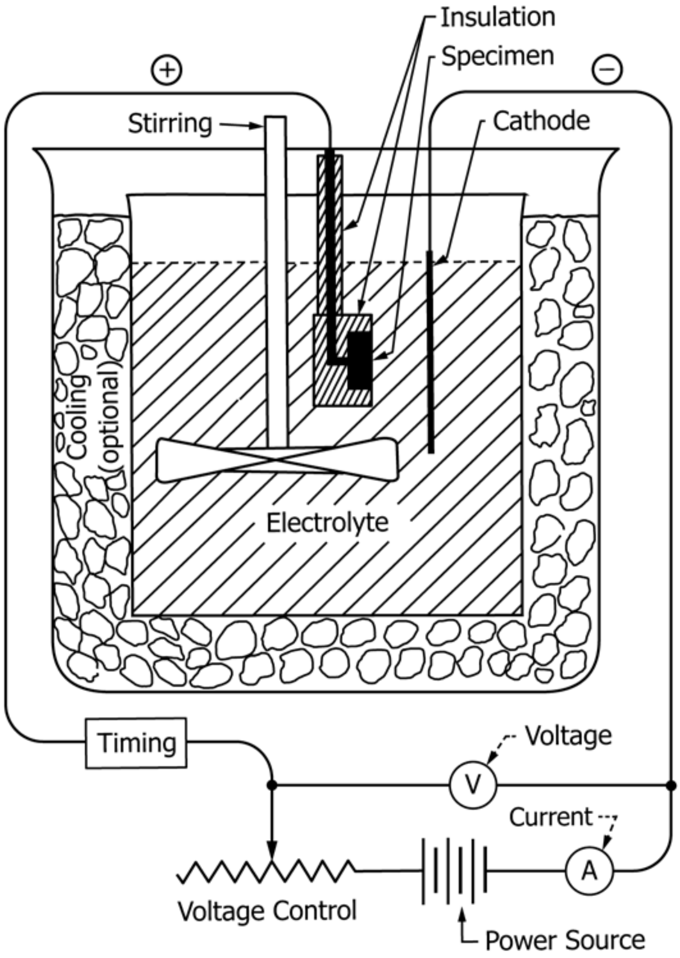

FIG. 1 Simple Arrangement for Electropolishing

6.3 Many variations are possible in the design of a cell for electrolysis. Because the current density is critical, predetermine the approximate area to be polished so that the conditions for optimum results can be achieved. This variable can be controlled by the use of simple fixed masks, such as are used in commercial units, or by the use of protecting masks of insulating paints or tapes.

6.3.1 The material used for the mask may be important as reactions with the electrolyte may occur which may simply degrade the mask material, or may lead to explosions (see discussions of electrolyte types I, II, V, and VIII in 5).

6.4 Suitable power sources may be batteries, rectifier power supplies, or direct-current generators. Because as much as 150 V dc may be required to polish some metals, precautions must be taken to avoid electrical shock. The ideal power source would be a true direct current supply with a closely controlled voltage. When alternating-current converted to direct-current power is used, the voltage and current characteristics needed for electropolishing can be controlled only by the use of resistance. One of the best ways of obtaining the power for electropolishing is to rectify the output of a variable-voltage transformer. When a single-phase, full wave, bridge-rectifier circuit is used, the output current is not satisfactory for all electropolishing unless the ripple voltage is reduced to some small value by filtering. This is because a particular ionization level must often be maintained within close limits in the electrolytic cell. Sufficient filtering for small amounts of power is obtained with small chokes and capacitors. When heavy currents are needed, a better source of power is a three-phase, full-wave, bridge recifier.

6.5 Certain electrolytes are strong etchants of the metal when the polishing current is off. Therefore, the provision for rapid insertion and removal of the specimen is necessary.

6.6 The electrolyte flow rate may be an important variable. Certain electrolytes polish best when a vigorous flow rate is employed, using special electropolishing cells, while other electrolytes may require gentle agitation or no agitation at all. When using pumped solutions with specially designed polishing cells, the manufacturer's practice for adjustment of the flow rate should be followed.

6.7 The cathode material should be relatively inert in the electrolyte and, for best results, should be more noble than the metal or alloy being electropolished. A stainless steel cathode is recommended for all of the electrolytes in Table 1 where no specific recommendation is given. Other useful cathode materials include copper, nickel, graphite and platinum. The surface area of the cathode should be much greater than that of the anode (surface to be polished).

6.8 Additional design features that are desirable in apparatus for the electropolishing of metallographic specimens are as follows:

6.8.1 Separation of the power and control portions from the polishing cell to protect them from corrosive fumes and solutions;

6.8.2 Meters for the continuous indication of voltage and current;

6.8.3 Polishing cycle controlled by an automatic timer;

6.8.4 Appropriate fume hood to vent gases formed while polishing;

6.8.5 Convenient changing of electrolytes and washing and drying of the apparatus; and

6.8.6 Corrosion resistant parts which are in contact with electrolyte.

7. Procedure

7.1 The metallographer may be required to electropolish some metal or alloy which has not previously been encountered. There are several general principles which can be applied advantageously in these cases. The problem should be viewed in comparison with known procedures and information gained through previous experience. It is generally helpful to compare the relationship of the major component with elements of the same general group in the periodic arrangement of the elements, and to study the phase diagram, if available, to predict the number of phases and their characteristics. Single-phase alloys are generally easier to electropolish than multiphase alloys. Minor alloying elements may affect the material's response to polishing in a given electrolyte.

7.2 The conditions required for optimum polishing of a metal in a given electrolyte can be ascertained by plotting current density versus voltage curves. The resulting curves will approximate one of the two forms shown in Fig. 2. Curve I is typical of electrolytes that either polish over a very wide range or will not polish at all. Curve II is characteristic of electrolytes that form an ionic film. The dotted portion of the curve is added in recognition of certain published data and the observation that the formation of a polishing film requires finite time. Polishing will occur between B and C and is usually best slightly before C.

7.3 After the polishing range is determined, other constants such as preparation, electrolyte flow and time can be determined experimentally. In the majority of cases, a mechanically prepared surface, as produced by grinding up to a 600-grit silicon carbide finish, is sufficient; however, with some alloys, a mechanically polished surface is desirable. Metal removal rates by electropolishing are low, typically around 1 µm per minute. To remove all prior damage from grinding, either some rough polishing should be performed after the P1200-grit grinding step, or the electropolishing time may be extended, which may not be desirable. The surface to be electropolished should be thoroughly cleaned to obtain uniform attack by the electrolyte.

7.4 Small specimens may be mounted in compression mounting media or in castable mounting media for ease in handling for mechanical preparation. Both types of mounting media are generally impervious to attack by the electrolyte (see 5.7). When mounted samples are to be electropolished, electrical contact can be made through a small hole drilled through the back of the mount into the metal.

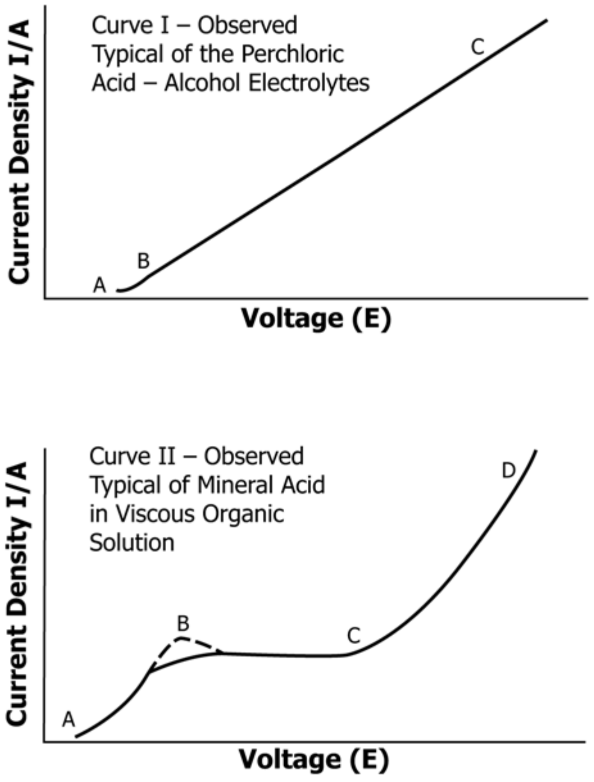

FIG. 2 Typical Curves for Determining Conditions for Electropolishing

7.5 To aid in selecting or developing an electrolyte for a new metal or alloy, the following characteristics should be considered:

7.5.1 The electrolyte should be somewhat viscous.

7.5.2 The electrolyte must be a good solvent of the anode under electrolysis conditions. Insoluble products that form adherent deposits on the face of the specimen may prevent polishing.

7.5.3 It is desirable that the electrolyte not attack the metal when the current is not flowing. However, this condition is not always achieved.

7.5.4 One or more ions of large radii [example: $(PO_4)^{-3}$, $(ClO_4)^{-1}$, or $(SO_4)^{-2}$ (and occasionally certain large organic molecules)] are usually required in electrolytes.

7.5.5 The electrolyte should be simple to mix, stable, and safe to handle.

7.5.6 It is desirable that the electrolyte function at room temperature and not be sensitive to temperature changes. If electropolishing is being done on a continuing basis, the electrolyte can be cooled by placing the cell in a water-cooled container having a water inlet and outlet.

7.6 The cathode material and size, and the anode-to-cathode spacing and orientation, are important variables. The cathode material should be more noble than the anode material (that is, the surface to be polished) and the cathode surface area should be substantially greater than the anode surface area to be polished. Generally, the cathode and anode surfaces should be aligned parallel to each other with a spacing of about 10–20 mm.

7.7 The depth of the specimen below the electrolyte surface during polishing may also influence polishing results. There should be adequate electrolyte above the area to be polished for best results.

7.8 Table 2 shows a listing of some of the most common troubles likely to be encountered in electropolishing and suggestions for solving the problems.

7.9 Table 1 contains a listing of the formulas and conditions for use of electrolytes suggested for the polishing of various metals. Table 3 is a cross listing of the various electrolytes for specific metals and generally for their alloys.

8. Keywords

8.1 electrolytic polishing; electropolishing ; polishing; specimen preparation (metallographic)

TABLE 2 Electropolishing Procedural Problems and Corrections

Trouble	Possible Cause	Suggested Correction
Center of specimen deeply etched	no polishing film at center of specimen	(*1*) increase voltage (*2*) decrease agitation (*3*) use more viscous electrolyte
Pitting or etching at edges of specimen	too viscous or thick film	(*1*) decrease voltage (*2*) increase agitation (*3*) use less viscous electrolyte
Sludge settling on surface	insoluble anode product	(*1*) try new electrolyte (*2*) increase temperature (*3*) increase voltage
Roughness or matte surface	insufficient or no polishing film	(*1*) increase voltage (*2*) use more viscous electrolyte
Waviness or streaks on polished surface	(*1*) insufficient time (*2*) incorrect agitation (*3*) inadequate preparation (*4*) too much time	(*1*) increase or decrease agitation (*2*) better preparation (*3*) increase voltage and decrease time
Stains on polished surface	attack after polishing current is off	(*1*) remove specimen while current is still on (*2*) try less corrosive electrolyte
Unpolished spots (bullseyes)	gas bubbles	(*1*) increase agitation (*2*) decrease voltage
Phases in relief	insufficient polishing film	(*1*) increase voltage (*2*) better preparation (*3*) decrease time
Pitting	(*1*) too long polishing (*2*) too high voltage	(*1*) better preparation (*2*) decrease voltage (*3*) decrease time (*4*) try different electrolyte

TABLE 3 Suggested Electrolytes for Metals and Generally Their Alloys

Metal or Alloy	Electrolyte No.
Aluminum	I-1, I-2, I-4, I-5, I-6, I-8, I-10, I-12, I-13, II-3, III-7, III-14, IV-6
Aluminum-silicon alloys	I-6, I-8
Antimony	II-4, VI-26, VI-27
Beryllium	I-4, I-13
Bismuth	VI-18, VI-25, VI-28
Cadmium	I-14, III-4
Cast iron	I-4, II-1
Chromium	II-1, II-6, VI-29, VIII-1
Cobalt	I-5, II-3, III-1, III-4, VIII-1
Copper	III-2, III-3, III-4, III-5, III-10, III-15, VIII-1
Copper-zinc alloys	III-3, III-4, III-5, III-10, V-2, VIII-1
Copper-tin alloys	III-10, VI-5, VI-6, VIII-1
Copper-nickel alloys	III-3, III-10, VIII-1
Germanium	I-9, VI-30
Gold	VII-1
Hafnium	II-7, VII-11
Iron, pure	I-5, II-1, IV-2, IV-3
Iron-silicon alloys	I-5, I-6, I-8, II-5
Iron-copper alloys	III-3, III-4
Iron-nickel alloys	I-5, II-1, II-2, II-4, IV-3, VIII-1
Lead	I-1, I-5, I-11, II-4, VII-5
Magnesium	I-1, III-7, III-12, VI-19
Manganese	III-9
Molybdenum	I-7, I-11, I-15, IV-4, IV-7, VI-20
Neptunium	III-16
Nickel-chromium	II-4, VIII-1
Nickel	I-4, I-13, I-14, II-4, IV-2, VIII-1
Nickel aluminide	I-13
Niobium	VI-31, VI-32
Silicon	VII-5
Silver	I-14, III-7, VII-1, VII-2, VII-3
Steel, austenitic, stainless, and super alloys	I-1, I-2, I-3, I-4, I-5, I-11, I-13, II-1, II-2, II-3, III-3, III-6, III-11, IV-1, IV-2, IV-3, IV-5, IV-6, IV-8, V-1, VI-1, VI-2, VI-3, VI-4, VI-7, VI-8, VI-9, VI-10, VI-11, VI-13, VI-15, VI-16, VI-17, VI-33, VIII-1
Steel, carbon and alloy	I-1, I-2, I-4, I-5, I-11, I-13, II-1, II-2, II-3, II-5, II-7, III-6, VI-3, VI-11
Tantalum	VI-12
Tin	I-4, I-13, VI-5, VI-6, VII-6
Titanium	I-4, I-9, I-13, II-1, II-2, II-3
Tungsten	VII-4, VII-5, VII-7
Uranium	I-4, I-7, II-1, II-2, II-3, III-8, III-13
Vanadium	I-9
Zinc	I-1, I-5, III-12, V-2, VI-14, VI-23, VII-6, VIII-1
Zirconium	I-4, I-7, I-9, II-2, VI-24

REFERENCES

(1) Jacquet, P. A., "Electrolytic and Chemical Polishing," *Met. Reviews*, Vol 1, Part 2, 1956, pp. 157–238.

(2) Wernick, S., *Electrolytic Polishing and Bright Plating of Metals*, 2nd ed., Alvin Redman Ltd., London, 1951.

(3) Tegart, W. J. McG., *The Electrolytic and Chemical Polishing of Metals in Research and Industry* , Pergamon Press Ltd., London, 1956.

(4) Shigolev, P. V., *Electrolytic and Chemical Polishing of Metals*, Corrected 2nd ed., Freund Publishing House, Tel-Aviv, Israel, 1974.

(5) Anderson, R. L., "Electrolytic Polishing of Metallographic Specimens," Westinghouse Research Laboratories, Pittsburgh, Research Report 60-94402-11-R2, April 20, 1955.

(6) Linke, U., "Procedures for Electrolytic Polishing of a Number of Special Materials," *Prakt. Met.*, Vol 17, February 1980, pp. 89–92.

(7) Schafmeister, P., and Volk, K. E., "The Electrolytic Polishing of Metals," *Arch. Eisenhutten*., Vol 15, 1941, pp. 243–246 (HB No. 1235).

(8) Jacquet, P. A., "Improved Electrolytes for the Anodic Polishing of Certain Metals," *Sheet Metal Ind.*, Vol 26, March 1949, pp. 577–584, 593.

(9) Anon, "Electrolytic Polishing and Etching," *Metal Progress*, Vol 66, July 15, 1954, pp. 169–173.

(10) Zmeskal, O., "Electrolytic Polishing of Stainless Steel and Other Metals," *Metal Progress*, Vol 47, April 1945, pp. 729–736.

(11) Merchant, H. J., "The Application of Electrolytic Polishing to Ferrous Metallography," *JISI*, Vol 155, 1947, pp. 179–194.

(12) Mott, B. W., and Haines, H. R., "The Application of Polarized Light to the Examination of Various Anisotropic Metals and Intermetallic Phases," *Journal Inst. Metals*, Vol 80, 1951–52, pp. 629–636.

(13) Tolley, G., "The Electrolytic Polishing of Metals," *Metallurgia*, Vol 37, December 1947, pp. 71–74.

(14) Hull, F. C., and Anderson, R. L., "Tentative Methods for Electrolytic Polishing of Metallographic Specimens," Westinghouse Research Laboratories, Pittsburgh, *Scientific Paper 6-94402-11-P2*, April 3, 1958.

(15) Elmore, W. C., "Electrolytic Polishing," *Journal of Applied Physics*, Vol 10, 1939, pp. 724–727; Vol 11, 1940, pp. 797–799.

(16) Tegart, W.J. McG., and Vines, R. G., "The Mechanism of Electrolytic Polishing of Metals," *Rev. Met.,* Vol 48, No 4, 1951, pp. 245-250.

(17) Jacquet, P. A., "The Principles and Scientific Applications of the Electrolytic Polishing of Metals," *Sheet Metal Ind*., Vol 24, October 1947, pp. 2015–2025, 2030.

(18) Michel, P., "A Report on Recent Research in Electrolytic Polishing," *Sheet Metal Ind*., Vol 26, October 1949, pp. 2175–2189.

(19) Hoar, T. P., and Mowat, J. S., "Mechanism of Electropolishing," *Nature*, Vol 165, No. 4185, Jan. 14, 1950, pp. 64–65.

(20) Walton, H. F., "The Anode Layer in the Electrolytic Polishing of Copper," *Journal of the Electrochemical Society*, Vol 97, July 1950, pp. 219–226.

(21) Hoar, T. P., and Farthing, T. W., "Solid Films on Electropolishing Anodes," *Nature*, Vol 169, No. 4295, February 23, 1952, pp. 324–325.

(22) Allen, J. A., "Oxide Films on Electrolytically Polished Copper Surfaces," *Trans. Faraday Society*, Vol 48, March 1952, pp. 273–279.

(23) Edwards, J., "The Mechanism of Electropolishing of Copper in Phosphoric Acid Solutions," *Journal of the Electrochemical Society*, Vol 100, July 1953, pp. 189C–194C; August 1953, pp. 223C–230C.

(24) Hickling, A., and Higgins, J. K., "The Rate-Determining Stage in the Anodic Dissolution of Metals (During Electrolytic Polishing of Copper)," *Trans. Inst. Metal Finishing*, Vol 29, 1952–53, pp. 274–301.

(25) Wagner, C., "Contribution to the Theory of Electropolishing," *Journal of the Electrochemical Society*, Vol 101, May 1954, pp. 225–228.

(26) Higgins, J. K., "The Anodic Dissolution and Electrolytic Polishing of Metals," *Journal of the Electrochemical Society*, Vol 106, December 1959, pp. 999–1005.

(27) Anon, "Electropolishing," *Metal Finishing*, Vol 66, May 1968, pp. 81–84; June 1968, pp. 123–126.

(28) Olefjord, I., "ESCA Studies on Films Formed on Stainless Steels During Oxidation and During Electropolishing," *Scand. J. Met*., Vol 3, 1974, pp. 129–136.

(29) Tousek, J., "The Polarization Curves of Metals in Polishing Solutions," *Prakt. Met* ., Vol 14, 1977, pp. 639–642.

(30) Mathieu, J. B., et al., "Electropolishing of Titanium in Perchloric Acid-Acetic Acid Solutions," *Journal of the Electrochemical Society*, Vol 125, July 1978, pp. 1039–1049.

(31) Honeycombe, R. W. K., and Hughan, R. R., "Electrolytic Polishing of Copper in Orthophosporic Acid," *J. Council Sci. Ind. Res*. (Aust.), Vol 20 , 1947, pp. 297–305.

(32) Jacquet, P. A., "Electrolytic Polishing of Metallic Surfaces," *Metal Finishing*, Vol 47, May 1949, pp. 48–54; June 1949, pp. 83–92; July 1949, pp. 58–64; September 1949 , pp. 60–67; October 1949, pp. 68–73; Vol 48, January 1950, pp. 56–62; February 1950, pp. 55–62.

(33) Hahn, T. S., and Marder, A. R.," Effect of Electropolishing Variables on the Current Density-Voltage Relationship," *Metallography*, Vol 21, November 1988, pp. 365–375.

(34) Nakada, Y., "Specimen Temperature During Electropolishing of Aluminum Crystals," *Trans. AIME*, Vol 233, January 1965, pp. 244–246.

(35) Brown, O. E., and Jimison, C. N., "An Improved Cell for Electrolytic Polishing," *Metal Progress*, Vol 40, September 1941, pp. 298–299.

(36) Gleekman, L. W., et al., "An Improved Cell for Electrolytic Polishing," *Metal Progress*, Vol 61, June 1952, pp. 92–93.

(37) Hancher, R. L., "An Improved Method for Routine Electrolytic Polishing of Microspecimens," *Metallurgia*, Vol 49, January 1954, pp. 47–51.

(38) Sykes, E. C., et al., "An Apparatus for Electropolishing Specimens for Metallographic Examination," *J. Inst. Metals*, Vol 83, 1954–55, pp. 166–168.

(39) Chawner, P. M. H., "Electrolytic Polishing Technique for Metallographic Specimens in Routine Quality Control," *Metal Treatment and Drop Forging*, Vol 22, October 1955, pp. 427–429, 432.

(40) Jacquet, P. A., "Rapid Method of Preparation of Surfaces for Metallographic Examination by Local Electrolytic (Tampon) Polishing," *Note Technique*, O.N.E.R.A. Publication No. 40, 1957, 26 pgs. (BISI No. 1084).

(41) Jacquet, P. A., "Polishing with the 'Ellopol' Apparatus," *Metal Progress*, Vol 75, January 1959, pp. 125–126.

(42) Asundi, M. K., and Tangri, K., "A Simple Electropolishing Cell," *Metallurgia* , Vol 61, No. 365, March 1960, pp. 139–140.

(43) Rowe, M., et al., "A Computer Controlled Electropolishing System," *Microstructural Science*, Vol 16, ASM and IMS, 1988, pp. 555–564.

(44) Paller, E., "Development of Metallographic Structure with the Aid of the Electrolytic Polisher Elypovist Made in Jena," *Jena Rev*., 1972, No. 5, pp. 243–247.

(45) Henning Strehblow, H., "An Apparatus for Electropolishing Metal Surfaces," *Prakt. Met.*, Vol 12, 1975, pp. 283–292.

(46) Venkataraman, G., "Improved Devices for In-Situ Metallographic Sample Preparation, Part 2, Electrolytic Polishing and Etching Devices," *Prakt. Met*., Vol 18, July 1981, pp. 342–353.

(47) Jacquet, P. A., "Nondestructive Techniques for Macro- and Micrographic Surface Examination of Metallic Specimens (Electrolytic Local Polishing and Replica Technique)," *Proceedings ASTM*, Vol 57, 1957, pp. 1290–1303.

(48) Rümmele, F., "A Combined Electrolytic Polisher and Microscope," *Engineers' Digest*, Vol 19, November 1958, pp. 479–481.

(49) Raemmele, F., and Veit, K., "A New Polishing Concept from East Germany," *Metal Progress*, Vol 81, January 1962, pp. 109–113.

(50) Pekarev, A. I., and Chistyakov, Yu. D., "Microscope Attachments for Observing Electropolishing and Etching," *Ind. Lab.*, Vol 31, October 1965, p. 1594.

(51) Arrowsmith, H. W., and Rencken, D. S., "Electrolytic Polisher-Etcher for Standard Metallographs," *Proc. First IMS Tech. Meeting*, 1969, pp. 17–22.

(52) Michel, P., "A Report on Recent Research in Electrolytic Polishing," *Sheet Metal Ind.*, Vol 27, February 1950, pp. 165–173.

(53) Samuels, L. E., "A Critical Comparison Between Mechanical and Electrolytic Methods of Metallographic Polishing," *Metallurgia*, Vol 66, No. 396, October 1962, pp. 187–199.

(54) Eckstein, H. J., "The Surface Quality of Electrolytically Polished Steels," *Radex-Rundschau*, 1967, No. 314, pp. 629–634.

(55) Hryniewicz, T., et al., "The Evaluation of Electrolytically Polished Surfaces," *Wear* , Vol 45, December 1977, pp. 335–343.

(56) Knuth-Winterfeldt, E., "Electropolishing with Special Reference to Two Commercial Types of Polishing Apparatus for Metallographic Purposes—Postscript," *Mikroscopie* , Vol 5, 1950, pp. 184–193.

(57) Jacquet, P. A., "The Electrolytic Polishing of Aluminum: The Application to the Micrographic Study of the Metal and its Alloys. I. The Technique of Electrolytic Polishing," *Metaux et Corrosion*, Vol 13, No. 153, 1938, pp. 86–91.

(58) Waisman, J. L., "Metallographic Electropolishing," *Metal Progress*, Vol 51, April 1947, pp. 606–610.

(59) Jacquet, P. A., "Electrolytic Polishing of Aluminum," *Comptes Rendus*, Vol 205, 1937, pp. 1232–1235.

(60) DeSy, A. L., and Haemers, H., "Electrolytic Rapid Method of Etch-Polishing Metallographic Specimens," *Stahl und Eisen*, Vol 61, 1941, pp. 185–187 (HB No. 1098).

(61) Hagemann, I., "Rapid Preparation of Aluminum and Aluminum-Alloy Specimens for Grain Size Determinations in Continuous Production Control," *Prakt. Met.*, Vol 9, May 1972, pp. 293–297.

(62) Larke, L. W., and Wicks, E. B., "A Method for the Electrolytic Polishing and Etching of Some Al-Ag Alloys, Commercial Purity Aluminum and Pure Magnesium," *Metallurgia*, Vol 41, January 1950, pp. 172–174.

(63) Knuth-Winterfeldt, K., "A New Electrolytic Polishing Method for Metallographic Investigations of Si-Rich Light Alloys," *Rev. Aluminum*, Vol 28, No. 175, March 1951, pp. 84–86.

(64) Yang, H. S., "An Electrolytic Etching/Anodizing Method for Revealing the Microstructures of Common Aluminum Alloys," *Praktische Metallographie*, Vol 27, 1990, pp. 539–545.

(65) Coons, W. C., "Preparing Bismuth and Antimony for Metallographic Examination," *Metal Progress*, Vol 84, December 1963, pp. 120–123.

(66) Jacquet, P. A., "Electrolytic Polishing of Zirconium, Titanium, and Beryllium," *Proceedings of the First World Met. Congress*, ASM, Cleveland, 1952, pp. 732–751.

(67) Wheeler, G. A., and Price, C. W., "A Metallographic Technique for Polishing and Etching Beryllium," *Trans. AIME*, Vol 239, October 1967, pp. 1657–1659.

(68) Hare, G. A., and Mallon, H. D., "Electrolytic Polishing of Bismuth," *Metallurgia* , Vol 63, April 1961, pp. 208–209.

(69) Jacquet, P. A., "Research on Electropolishing of Steels, Chromium, and Light Alloys as an Aid to their Micrographic Examination," *Rev. Met.*, Vol 46, April 1949, pp. 214–227.

(70) Jacquet, P. A., "A Practical Method for the Electrolytic Polishing of Steels and of Chromium for Micrographic Examination," *Comptes Rendus*, Vol 227, 1948, pp. 556–558.

(71) Coons, W. C., and Iosty, L. R., "Electrolytic Polishing System for Space Age Materials," *Metal Progress*, Vol 109, May 1976, pp. 36–40.

(72) Lorking, K. F., "The Electropolishing of Chromium and its Alloys," *Bulletin of the Institute of Metal Finishing*, Vol 5, 1955, pp. 119–126.

(73) Perry, E. R., "Electro-Polishing and Electro-Etching a Sintered Cobalt Alloy," *Metallurgia* , Vol 53, No. 319, May 1956, pp. 231–233.

(74) Powers, R. W., "The Electropolishing of Copper Specimens," *Electrochemical Technology*, Vol 2, September–October 1964, pp. 274–281.

(75) Pellisier, G. E., et al., "Electrolytic Preparation of Iron and Steel Micro-Specimens," *Metal Progress*, Vol 37, January 1940, pp. 55–57.

(76) Jacquet, P. A., "On the Anodic Behavior of Copper in Aqueous Solutions of Orthophosphoric Acid," *Trans. Electrochemical Society*, Vol 69, 1936, pp. 629–655.

(77) Foss, G. J., and Shiller, L., "The Electrolytic Polishing of Brass and Copper," *Metal Progress*, Vol 42, July 1942, pp. 77–79, 98.

(78) Perryman, E. C. W., "A New Solution for the Electrolytic Polishing of Copper and Copper-Base Alloys, Particularly Tin Bronze," *Metallurgia*, Vol 46, July 1952, pp. 55–57.

(79) deSy, A., and Haemers, H., "Rapid Electropolish and Etch," *Metal Progress* , Vol 53, March 1948, pp. 368–371.

(80) Knuth-Winterfeldt, E., "Electrolyte for Electrolytic Polishing and Etching," *Jern. Ann.*, Vol 134, October 1950, pp. 538–539.

(81) Bassi, G., "Electrolytes for the Polishing and Etching of Austenitic Chrome-Nickel and Manganese Steels," *Metalloberfläche*, Vol 52, 1961, No. 2, pp. 141–142 (HB No. 5164).

(82) Pow, J., "Electrolytic Polishing of Iron and Steel," *Metal Treatment and Drop Forging*, Vol 16, Spring 1949, pp. 31–37.

(83) Morris, C. E., "Electropolishing of Steel in Chrome-Acetic Acid Electrolyte," *Metal Progress* , Vol 56, November 1949, pp. 696–699, 710, 712, 714.

(84) Uhlig, H. H., "The Electrolytic Polishing of Stainless Steel," *Trans. Electrochemical Society*, Vol 80, 1940, pp. 265–277.

(85) Eilender, W., et al., "An Electrolyte for the Electrolytic Polishing of Steels," *Metalloberfläche*, Vol 3, No. 4, 1949, pp. 88–90 (HB No. 2327).

(86) Skoda, R. E., "Electrolytic Polishing of Nodular Cast Iron," *Metal Progress*, Vol 69, February 1965 , p. 66.

(87) Imboden, R. L., and Sibley, R. S.," Anodic Polishing of Plain Carbon Steels," *Trans. Electrochemical Society*, Vol 82, 1942, pp. 227–239.

(88) Parfessa, G. I., and Sidlyarenko, V. A., "Universal Electrolyte for the Polishing of Stainless and Heat-Resisting Steels, Low-Carbon Steels and Titanium Alloy VT-5," *Avtom. Svarka*, Vol 11, 1958, No. 7, pp. 83–84 (HB No. 4421).

(89) Verhaege, M., and DeWilde, R., "A New Method of Electrolytic Polishing and Etching of Lead and Lead Alloys," *Prakt. Met.*, Vol 10, 1973, pp. 220–226.

(90) Jones, E., and Thirsk, H. R., "Electrolytic Polishing of Lead in a Sodium Acetate-Acetic Acid Bath," *Nature*, Vol 171, May 9, 1953, p. 843.

(91) Moulen, A. W., "The Electrolytic Polishing of Lead-Tin Alloys for Microscopic Examination," *Journal of the Electrochemical Society*, Vol 99, June 1952, pp. 133C–136C.

(92) Gregory, P., et al., "A Method of Electropolishing Dilute Alloys of Lead," *Metallurgia* , Vol 60, No. 360, October 1959, pp. 171–172.

(93) Borradaile, J. B., et al., "An Electropolishing and Etching Solution for Magnesium Alloys," *Prakt. Met.*, Vol 12, December 1975, pp. 656–659.

(94) Cortes, F. R., "Electrolytic Polishing of Refractory Metals," *Metal Progress*, Vol 80, August 1961, pp. 97–100.

(95) Coons, W. C., "Simple Electrolytic Polishing Procedures for Molybdenum Metallographic Specimens," *Trans. ASM*, Vol 41, 1949, pp. 1415–1424.

(96) Zamin, M., et al., "On the Electropolishing of Molybdenum," *Journal of the Electrochemical Society*, Vol 124, October 1977, pp. 1558–1562.

(97) Wensch, G. W., “Electrolytic Polishing of Nickel,” *Metal Progress*, Vol 58, November 1950, pp. 735–736.

(98) Jacquet, P. A., “Electrolytic Polishing of Nickel,” *Metal Progress*, Vol 60, August 1951, p. 65.

(99) Krudtaa, O. J., and Stokland, K.,“ Electropolishing of Columbium and Tantalum,” *Metal Progress*, Vol 77, January 1960, pp. 101–103.

(100) Pelleg, J., “Electropolishing Niobium,” *Journal of Less-Common Metals*, Vol 12, May 1967, pp. 421–422.

(101) Hallas, C. E., “Electropolishing Silicon,” *Solid State Technology*, Vol 14, January 1971, pp. 30–32.

(102) Shuttleworth, R., et al., “A Note on the Electrolytic Polishing of Silver,” *Metal Treatment*, Vol 14, Fall 1947, pp. 161–163.

(103) Hesselberger, W. M.,“ Electropolishing Silver,” *Metal Industry*, Vol 75, No. 9, August 26, 1949, pp. 167 –168.

(104) Gilbertson, L., and Fortner, O. M., “Electrolytic Polishing of Silver,” *Tarns. Electrochemical Society*, Vol 81, 1942, pp. 199–211.

(105) Wensch, G. W., et al., “Polishing Tantalum,” *Metal Progress*, Vol 61, March 1952, p. 81.

(106) Booker, G. R., and Stickler, R., “Large-Area 'Jet’ Electrolytic Polishing of Ge and Si,” *Journal of the Electrochemical Society*, Vol 109, No. 12, December 1962, pp. 1167–1171.

(107) Whelan, E. P., “An Electropolishing Procedure for Tin and Dilute Tin-Lead Alloys,” *Metallography*, Vol 1, 1969, pp. 455–457.

(108) Puttick, K. E., “Electrolytic Polishing of Tin in Perchloric Acid,” *Metallurgia*, Vol 41, 1949, pp. 120–121.

(109) Jacquet, P. A., “Electrolytic Polishing and Oxidation of Titanium,” *Metal Treatment and Drop Forging*, Vol 18, April 1951, pp. 176, 182.

(110) Coons, W. C., “Preparing Titanium for Microscopic Examination,” *Metal Progress* , Vol 79, June 1961, p. 93.

(111) Sutcliffe, D. A., et al., “Electrolytic Polishing of Titanium,” *Metallurgia* , Vol 41, March 1950, pp. 283–284.

(112) Ogden, H. R., and Holden, F. C., “Metallography of Titanium Alloys,” *TML Report No. 103*, May 29, 1958, Battelle Memorial Inst. (reprinted April 1965).

(113) Pelleg, J. A., “Electropolishing of Titanium,” *Metallography*, Vol 7, August 1974, pp. 357–360.

(114) Coons, W. C., and Iosty, L. R., “Electrolytic Polishing of Titanium, Its Alloys and Various Other Metals and Alloys,” *Microstructural Science*, Vol 4, 1976, pp. 193–202.

(115) Hughes, J. M., and Coomes, E. A., “Electrolytic' Polishing’ of Tungsten,” *Phys. Rev.*, Vol 55, June 1, 1939, p. 1138.

(116) Coons, W. C., and Gleason, A. S., “Polishing Tungsten by Electricity,” *Metal Progress*, Vol 92, October 1967, p. 9.

(117) Mott, B. W., and Haines, H. R., “The Electrolytic Polishing and Etching of Uranium,” *Metallurgia*, Vol 43, No. 259, May 1951, pp. 255–257.

(118) Jacquet, P. A., and Caillat, R., “Electrolytic Polishing of Uranium for Physico-Chemical and Metallographic Study,” *Comptes Rendus*, Vol 228, April 4, 1949, pp. 1224–1226.

(119) Pelleg, J., “Electropolishing Pure Vanadium,” *Metallography*, Vol 3, 1970, pp. 457–460.

(120) Vernon, W. H. I., and Stroud, E. G.,“ Electrolytic 'Polishing’ of Zinc,” *Nature* , Vol 142, No. 3593, September 10, 1938, pp. 477–478; No. 3609, December 31, 1938, p. 1161 .

(121) Rodda, J. L., “Electrolytic Polishing of Zinc and Brass,” *Mining and Met.*, Vol 24, July 1943, p. 323.

(122) Powers, R. W., and Jerabek, E. C., “The Electropolishing of Zinc Specimens,” *Journal of the Electrochemical Society*, Vol 117, August 1970, pp. 1099–1100.

(123) Roth, H. P., “Metallography of Zirconium,” *Metal Progress*, Vol 58, November 1950, pp. 709–711.

(124) Jacquet, P. A., “The Electrolytic Polishing of Zirconium and its Application to a Study of the Effects of Abrasion Upon the Structure of the Metal,” *Metallurgia*, Vol 42, No. 252, October 1950, pp. 268–270.

(125) Roberson, A. H., “Metallurgy of Zirconium and Zirconium Alloys,” *Metal Progress*, Vol 56, November 1949, pp. 667–669.

(126) Hopkins, E. N., et al., “A 'Universal’ Electropolishing Method,” *Tech. Papers*, 19th AEC Metallographic Group Meeting, ORNL-TM-1161, 1965 , pp. 1–27.

(127) Gabe, D. R., “Toward a Universal Electropolishing Solution,” *Metallography* , Vol 5, 1972, pp. 415–421.

(128) Evans, U. R., and Whitwham, D., “Note on a Convenient Method of Electropolishing Aluminum Alloys,” *Journal of the Electrodepositors’ Technical Society*, Vol 22, 1947, pp. 24–28.

(129) Pellisier, G. E., et al., “Electrolytic Polishing of Metals,” *Metal Progress* , Vol 38, 1940, p. 554.

(130) Anderson, R. L., “Safety in the Metallographic Laboratory,” *Westinghouse Research Lab. Scientific Paper No. 65-1P30-METLL-P2*, March 29, 1965.

(131) Jacquet, P. A., “The Safe Use of Perchloric-Acetic Electropolishing Baths,” *Metal Finishing*, Vol 47, 1949, pp. 62–69.

(132) Comas, S. M., et al., “Hazards Associated with Perchloric Acid-Butylcellosolve Polishing Solutions,” *Metallography*, Vol 7, 1974, pp. 47–57.

(133) Dawkins, A. E., “Chromic-Acetic Anhydride 'Explosion’,” *JISI*, Vol 182, 1956, p. 388.

Electropolishing Reviews–References (1-14)

Electropolishing Theory–References (15-34)

Electropolishing Equipment–References (35-51)

Electropolishing Evaluations–References (52-56)

Electropolishing Solutions–References (57-130)

Safety–References (130-133)