THE ELECTROLYTIC POLISHING OF METALS AND ITS APPLICATIONS TO METALLOGRAPHIC SPECIMENS

by

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THE ELECTROLYTIC POLISHING OF METALS AND ITS APPLICATIONS TO METALLOGRAPHIC SPECIMENS

I. INTRODUCTION

The preparation of metallographic specimens by mechanical methods has involved the use of a highly developed technique and considerable skill; the polishing must be of a high order to make possible efficient examination. In the past few years, however, electrolytic methods have been developed that make the polishing of metallographic specimens a simple and routine operation.

Electrolytic polishing has been defined as "the preparation of smooth, scratch-free surfaces by anodic corrosion in a fluid electrolyte" (1-819). By proper control of the composition and temperature of the electrolyte, the voltage and current applied across the cell, the area of the article to be polished, and the time of polishing, material can be removed from a metal surface selectively; the high portions are removed first, leaving an approximately plane surface which, in some cases, is slightly undulated.

Anodic methods of electrolytically cleaning metals, commonly known as electropickling, have been in use for

over 30 years (6-57). In this process, only foreign materials are removed from the surface. It was not until the findings of Jacquet were published (2-1076) that electrolytic polishing was introduced for metallurgical purposes.

The field of stainless steel gave electrolytic polishing industrial importance. A lustrous finish on stainless steel requires difficult and costly grinding and buffing operations; the difficulty increases with the complexity of the object. Electrolytic polishing will produce a mirrorlike finish on all portions of an object regardless of shape, with an ultimate saving in time and labor, without the use of any such mechanical methods. Electrolytic polishing has found industrial application in the brightening of aluminum reflectors, steel and brass wire, stainless steel refrigerator trays, and many other commercial metal products.

Classed as either being of the high or low voltage types. The former solutions usually use perchloric acid as the base constituent; these solutions give a polished surface in a matter of seconds. The solutions requiring a low voltage for operation use phosphoric acid, sulphuric acid, or combinations of the two in most instances as the base constituents. The low voltage solutions require a much longer time for polishing than do those of the high voltage

type. Many solutions in use contain special additives to give faster results; as a rule, these have been patented.

The purposes of this investigation were, namely: to design and construct an electrolytic polishing cell, to choose suitable electrolytes, and to determine the necessary conditions and best procedure with which to polish metal specimens for metallographic examination.

The metals chosen for polishing were those alloys of aluminum, copper, and steel having greatest industrial importance. Lead was added to the list of the metals to be polished electrolytically because of the difficulty encountered polishing it mechanically; the surface of the lead being soft will be caused to flow, thus giving a false picture of the grain structure.

II. THEORY

As yet the theory of electrolytic polishing is not completely understood. Essentially, the process consists in making the article to be polished the anode in an acid electrolyte bath capable of forming a soluble salt with the constituent metal. The cathode is usually of stainless steel, copper, or some other conductive, inactive material about 10 to 20 times the size of the anode.

The surface of any specimen polished on metallographic paper consists of a series of peaks and ridges or minute scratches. The soluble salt that is formed by the electrolyte in contact with the metal is a viscous, poorly conducting layer that is thinner at the peaks than in the troughs of the scratches; this soluble salt has a higher electrical resistance than the remainder of the electrolyte. The film covering the peaks will have a lower resistance than that in the troughs; thus, the current density at the peaks will be many times that of the troughs. In this way the peaks are removed first, leaving a plane surface.

The current density required to perform the polishing operation has to be accurately controlled and held constant during the entire procedure; it will vary with the electrolyte and metal being polished. Sufficient current density must be had "to form and to maintain a

solid oxidation product on the anode and permit it to have a viscous flow from the surface of the anode" (8, 730).

The electrolytes that are used consist of two general types. The first is composed of those solutions of low electrical resistance, requiring low voltage of from 4 to 25 volts. The second type is made up of those solutions requiring a high voltage of from 10 to 220 volts. With the first type of solution polishing is done by controlling the voltage; whereas, with the second type of solution polishing comes from current control.

Solutions of the first type are those based upon phosphoric acid, sulphuric acid, and acids such as arsenic, chromic, boric, citric, acetic, and lactic with or without the addition of certain organic reagents.

The greater number of solutions of the high resistance type contain perchloric acid as the base constituent with reagents such as acetic acid, acetic anhydride, and ethyl alcohol with or without ether added. Solutions containing nitric acid and methyl alcohol are also of the high resistance type.

The sketch in Fig. 1 shows the general form of the current density-voltage curve for the solutions of the low electrical resistance type. Up to point B metal is removed from the anode but a dull etched finish results. At point B the current density has become sufficient to

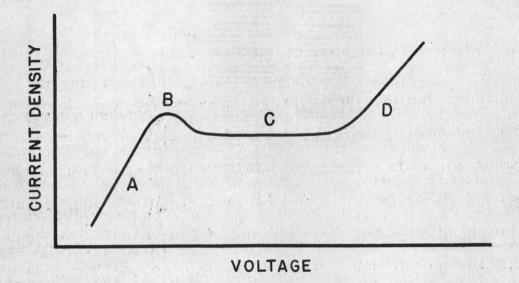


Fig. 1 General form of Current Density-Voltage Curve for Electrolytes that Polish with an almost Constant Current Density.

form a solid oxidation product on the anode and polishing begins. An increase in the voltage at point B will cause the current to drop slightly due to the added resistance of the film. The surface of the anode will continue to be polished with an increase in voltage and very little, if any, increase in current density. When the increase in voltage reaches point D, the current density will increase sharply with little change in voltage, and gas bubbles will be given off from the anode. At point D, pitting of the surface of the anode will result from the gas bubbles breaking up the polishing film sufficiently to cause non-uniform attack.

Electrolytic solutions of the second or high resistance type follow the general form of the curve shown in

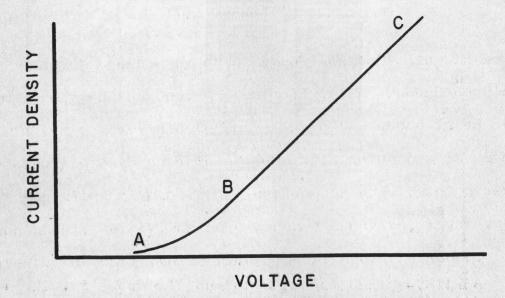


Fig. 2 General form of Current Density-Voltage Curve for Electrolytes that Polish Over a Wide Range of Voltages and Currents.

Fig. 2. At point A the voltage is great enough to overcome the back emf of the film that begins to form at very
low voltages. At this point a current will begin to flow
and will continue to increase in nearly direct proportion
to an increase in voltage.

For a short distance up the curve from point A, etching will occur because the polishing film is not thick enough to permit an even selective removal of metal from the high points on the surface. Polishing begins to take place at point B and will continue up to point G. At point C, gas bubbles will begin to accumulate on the surface of the anode and pitting will occur. Solutions in this category are also known as the "straight-line" type.

A simple sketch of an electrical circuit is shown in

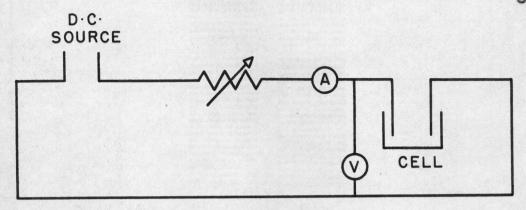


Fig. 3 A Simple Polishing Circuit.

Fig. 3. The direct current may be obtained from a generator, rectifier, or battery.

The total applied voltage across the cell and the external circuit is

$$E_t = E_f + I(R_f + R_e + R_r)$$

where E_f is the back emf of the polishing film; I is the total current flowing; R_f is the film resistance, and R_r is the external resistance of the circuit.

Re will vary with the electrolyte composition, temperature, and the cell size. Rr will be determined by the cathode area and the external resistance of the circuit. The ease with which the most satisfactory polishing current and voltage can be determined will depend upon the number of the above items held constant.

III. APPARATUS

Electrolytic Cell -- The cell shown in Fig. 4 is of the "spot-polish" type, that is, the area of the electrolytically polished surface is held constant by allowing the electrolyte to come in contact with the specimen through a hole of a known diameter. With the area constant, the current density for any one metal will be constant to obtain the best polishing results. The cell was formerly a glass battery jar, the inside dimensions of which are 3½ inches wide, 6 inches deep, and 4½ inches long. A ½ inch hole is drilled in one end, over which a gasket, having a ½ inch hole, and the specimen to be polished are placed. The tilting base that supports the cell is constructed of wood and held together with screws.

The gasket shown to the left of the cell in Fig. 4 consists of two pieces of rubber, stapled together with a sheet of 20 gauge, type 302 stainless steel between them. The hole in the gasket which the specimen covers is \(\frac{1}{4} \) inch in diameter and is used for all specimens.

The specimen is clamped to the gasket with a screw clamp which also makes electrical contact; the anode wire is attached to the clamp with a battery clip.

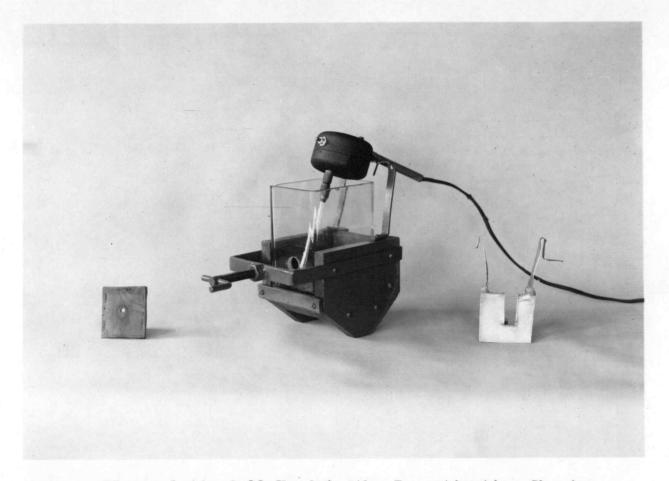


Fig. 4 Electrolytic Cell Used in the Investigation Showing Rubber Gasket and Stainless Steel Cathode.

The electrolyte level in the cell is raised and lowered above and below the gasket hole by tilting the cell forward or backward. When the cell is tilted to the back position, the operator is free to insert the specimen in place and see that it is firmly seated without any contact with the electrolyte, whatsoever. Tilting the cell to the forward position raises the electrolyte level over the specimen, thus closing the electrical circuit. A stirring motor is fixed permanently in place above the cell and operates a glass stirring rod approximately 1 inch from the specimen.

The cathode is shown to the right of the cell in Fig. 4; it is made of 20 gauge, type 302 stainless steel with dimensions that are 3 inches wide and 4 inches long. A slit inch wide and linches long is cut down the center so as not to interfere with the operation of the stirring rod. The cathode can be moved the entire length of the cell so as to obtain the best polishing results; it clamps to the top edge of the cell and is suspended in the electrolyte. Electrical contact is made on one edge with a battery clip.

The cell does not have any provision installed for regulating the temperature of the electrolyte. The volume of the electrolyte used in the operation of the

cell is 550 co; this quantity is sufficiently large to keep the temperature from varying more than 3 degrees during the preparation of a number of specimens. The use of electrolytes that must be used hot to give best polish results are not recommended with this apparatus; the glass is not pyrex and the temperature could not be maintained.

Power Source -- The power required for the operation of the cell was supplied by a 44 volt, 250 ampere direct current generator shown in Fig. 5. A 110 volt ac source provides power for the operation of the stirrer.

Instruments -- A wiring diagram of the electrical circuit is shown in Fig. 6. A 0-1.5 and a 0-5 ampere d.c. ammeter are included in the circuit with a double throw switch. In this arrangement values of low and high currents can be accurately read. A 0-50 volt d.c. voltmeter is connected across the cell. Protection of the ammeters and rheostat is provided by the 4 ampere fuse included permanently in the circuit.

The external resistance of the circuit is made up of an 80-ohm button type rheostat with a 10-ohm potentiometer type rheostat placed in series for fine adjustment. A filter was included in the circuit to smooth out some of the ripple effect of the current supplied by the generator.

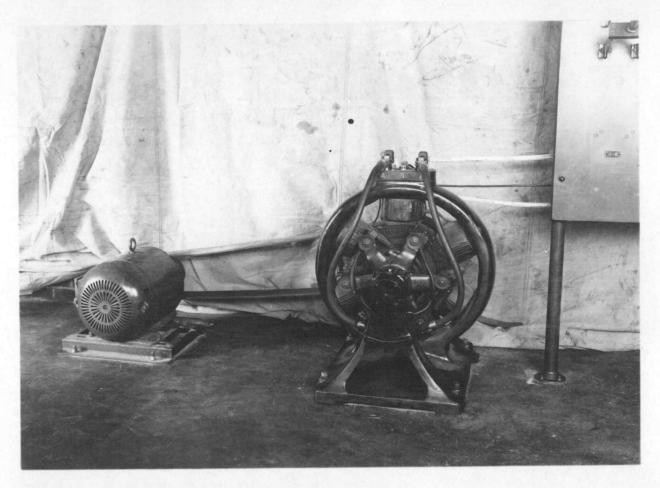


Fig. 5 Generator Used for Power Source.

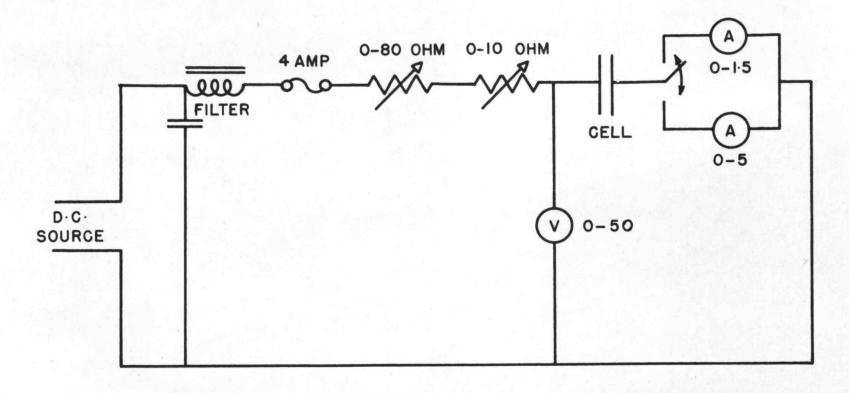


FIG. 6. WIRING DIAGRAM OF THE ELECTROLYTIC POLISHING CIRCUIT.

Electrolytes -- For the polishing of steel and aluminum alloys a solution consisting of perchloric acid, ethyl alcohol, water, and ether is used in the following proportions: (4, 209).

Perchloric Acid (70-72%) ----- 54 cc
Water ------ 146 cc
Ethyl alcohol with 3% ether ----- 800 cc

The solution used for the polishing of copper and its alloys is a mixture of nitric acid and methyl alcohol absolute in the following proportions: (5, 756).

Nitric Acid (con) ----- 600 cc

Methyl Alcohol (Abs) ----- 300 cc

Lead was found to be satisfactorily polished with an electrolyte of perchloric acid and glacial acetic acid, the proportions of which are given below: (8, 734).

> Perchloric acid (70-72%) ----- 300 cc Glacial acetic acid ----- 700 cc

IV. POLISHING PROCEDURE

Specimen Preparation -- In order to obtain satisfactory finishes for metallographic observation by electrolytic methods, it is necessary to start with a specimen that has a plane surface. The initial preparation of specimens for this investigation included rough grinding on grinding belts followed by further finishing on No. 1, 0, 00, and 000 metallographic emery paper. After the last finishing operation on metallographic emery paper, the specimen is washed in alcohol to remove any traces of grease and dirt. The specimen is then ready to be given its final polish electrolytically. Operation of Polishing Cell -- The prepared specimen is clamped in place over the gasket and the hole in the end of the cell. The cathode is adjusted to the proper position in the cell as given in the Tables of Operating Conditions further on in this thesis. The cell is tilted to its forward position allowing the electrolyte to cover the specimen. The stirrer is next switched on and the electrolyte given a swirling motion. The current is then permitted to flow and is adjusted and held at the proper value with the external resistance. The length of the polishing operation is

determined with the second hand of a watch or clock and

must be observed quite accurately in some cases.

After the polishing operation has proceeded for a definite period of time the cell is tilted to its back position and the stirrer switched off. The specimen is removed from the clamp, washed in water or alcohol, and dried. After etching with the required etchant if necessary, the specimen is ready for microscopic examination.

V. METALS POLISHED

As stated in the introduction the metals chosen to be polished in this investigation were those having greatest industrial importance. The specimens considered as representative are the following:

- 1. 0.10% carbon steel
- 2. 0.43% carbon steel
- 3. 0.97% carbon steel
- 4. White cast iron
- 5. Medium carbon cast steel
- 6. Stainless Steel (Type 302)
- 7. 2S aluminum
 - 8. 24S-T aluminum
- 9. 75S-0 aluminum
- 10. 195 casting aluminum
- 11. Cartridge brass (70-30)
- 12. Naval brass
- 13. Everdur bronze
- 14. Monel metal
- 15. Copper
- 16. Lead

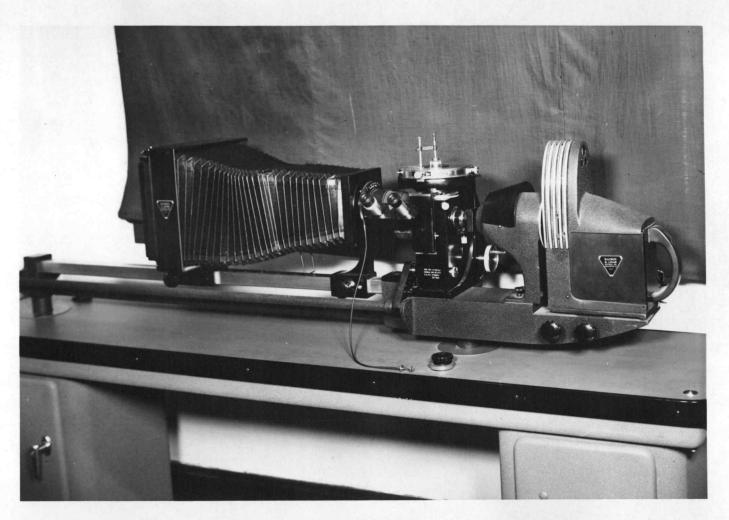


Fig. 7 Bausch and Lomb Micrometallograph.

VI. DISCUSSION OF RESULTS

The primary purposes of polishing specimens for microscopic examination are to study the grain structure, nonmetallic inclusions, surface, and constituents of the metal in question. In this investigation the photomicrographs of the metals polished were made at 250 diameters magnification to enable comparison of the effects of electrolytic polishing on each of the metals and to observe how well the primary purposes listed above are accomplished. The photomicrographs shown in the following illustrations were made on the Bausch and Lomb micrometallograph in Fig. 7.

The 0.10%, 0.43%, and the 0.97% carbon steel samples had been annealed for three hours prior to polishing. The photomicrograph of the 0.10% carbon steel in Fig. 8 shows some surface undulation. It was etched with 2% nital solution in order to get an even etch over the entire surface. The electrolyte has a tendency to etch the specimen as well as polish it, but in some cases this etching is not sufficient to show clearly the entire grain structure.

The very low carbon steels will have a dirty appearance when polished electrolytically because of the excessive attack on the nonmetallic inclusions; this

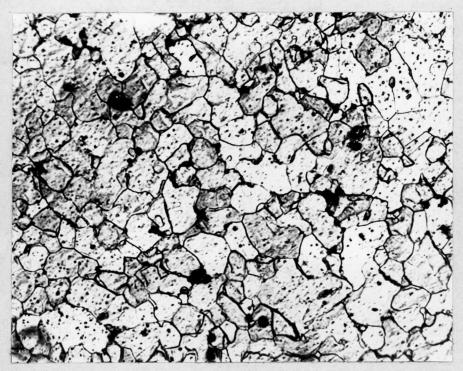
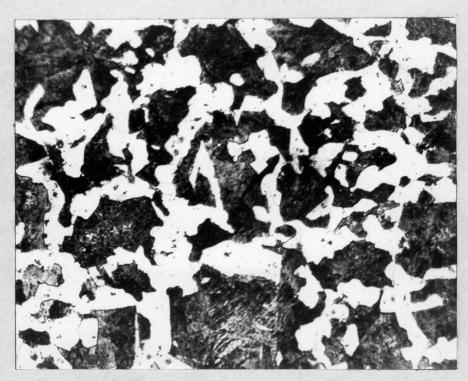


Fig. 8 Annealed 0.10% Carbon Steel, Electrolytic Polish 20 Seconds, 2% Nital Etch, 250X.



Pig. 9 Annealed 0.43% Carbon Steel, Electrolytic Polish 20 Seconds, 2% Nital Etch, 250x.



Fig. 10 Annealed 0.97% Carbon Steel, Electrolytic Polish 20 Seconds, 2% Nital Etch, 250X.

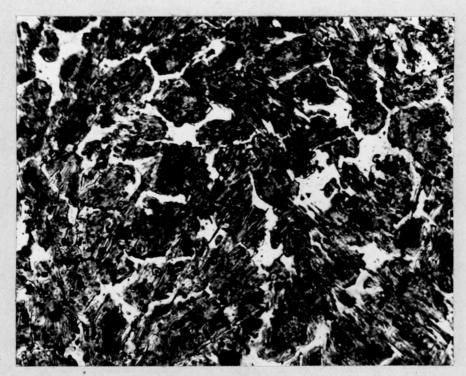


Fig. 11 White Cast Iron, Electrolytic Polish 10 Seconds, 2% Nital Etch, 250X.



Fig. 12 Medium Carbon Cast Steel, Electrolytic Polish 30 Seconds, 2% Nital Etch, 250X.

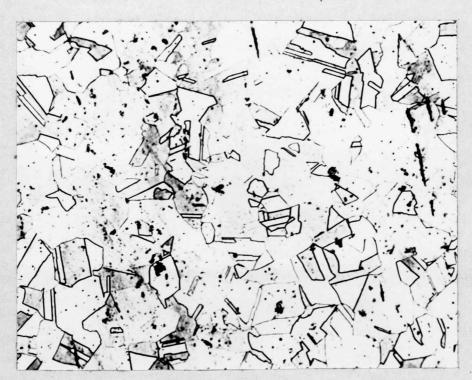


Fig. 13 Type 302 Stainless Steel, Electrolytic Polish 60 Seconds, Aqua Regia Etch, 250X.

causes pitting of the metal. The carbides will be left in high relief because they are not attacked by the electrolytic action. The photomicrograph of the 0.10% carbon steel gives the impression that the steel is dirty which actually is not the case.

The higher carbon steels polish very well electrolytically with results that are comparable with those of mechanical polishing. Several checks for smoothness of the pearlitic grains were made on 0.43% carbon steel with the Tukon Tester. Small indentations were made on the individual grains with a diamond point; if the indentation appeared even without being enlarged at either end, the grain would be considered smooth. The results of this check showed that most of the pearlitic grains were polished smooth. The hardness of the grains could be determined with the use of the Tukon Tester with as much accuracy on electrolytically polished specimens as with those polished entirely by mechanical methods; average values should be used, however.

In Fig. 12 the Widmanstätten structure of cast steel is shown. The specimen was taken from a broken tooth of a gear that was part of the swing draw mechanism of the Morrison Street Bridge in Portland, Oregon. This is representative of cast steel before annealing with approximately 0.30% carbon.

Stainless steel is very difficult to polish by mechanical methods without taking an excessive amount of time. The metal cold-works easily and the grain structure will be distorted for quite some depth below the surface. Aside from being difficult to polish easily, it is also difficult to etch because of its stainless properties; in many cases repolishing has to be done in order to obtain a satisfactory etch. Electrolytic polishing will give a polished surface in 60 seconds after preliminary preparation without flowed metal to obscure the true grain structure. The greatest difficulty experienced with the stainless steel came while trying to obtain a satisfactory etch with aqua regia.

Table I shows that the steels polished with nearly the same current density and the same voltage. The time varied somewhat, with stainless steel taking the longest as would be expected. The results obtained on steels by electrolytic polishing are very easily duplicated. Extensive experience is not necessary to polish any metal electrolytically after the initial conditions are established.

Aluminum proved to be one of the hardest metals to polish satisfactorily by electrolytic methods. Certain of the intermetallic compounds in aluminum such as silicon,

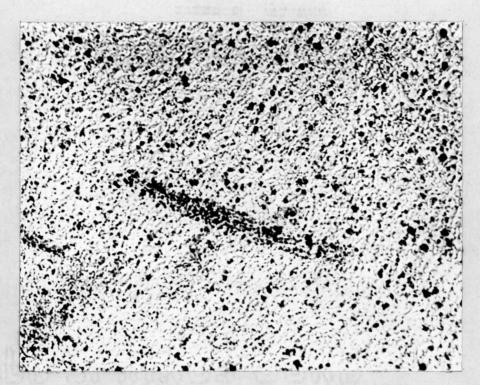


Fig. 14 2S Aluminum Bar Stock, Electrolytic Polish 60 Seconds, HF-HC1-H₂O Etch, 250X.

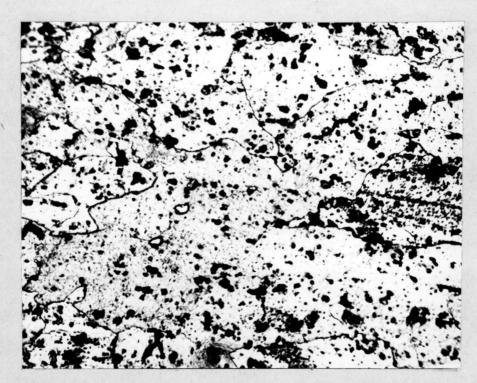


Fig. 15. 24S-T Aluminum Sheet, Electrolytic Polish 15 Seconds, HF-HCl-H₂O Etch, 25OX.

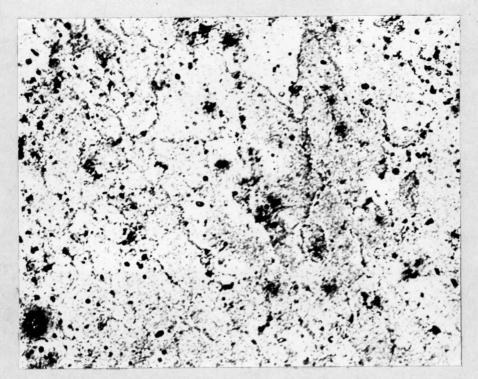


Fig. 16 75S-O Aluminum Bar Stock, Electrolytic Polish 30 Seconds, HF-HC1-H₂O Etch, 250X.

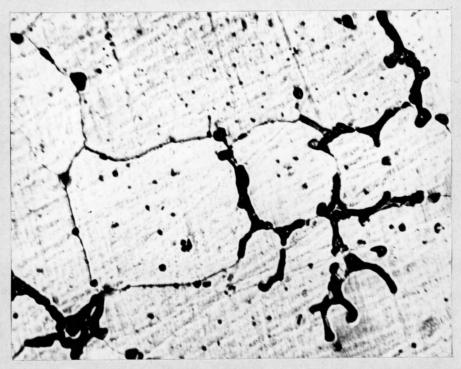


Fig. 17 195 Casting Aluminum, Electrolytic Polish 15 Seconds, No Etch Required, 250X.

FeAl₃, and AlMn may be left in relief while there will be "excessive attack on CuAl₂, MgSi, and Al-Cu-Mg" (1, 25). Electrolytic polishing is therefore impractical for aluminum alloys that are to be examined for cleanliness.

The attack of the CuAl₂ in the 2S (99% Al) aluminum specimen, Fig. 14, gave it a somewhat rough appearance, but the structure of the metal is brought out very clearly. In the 24S-T specimen sheet aluminum was used rather than the bar stock that was used for the other aluminum specimens. The photomicrograph of 24S-T in Fig. 15 shows very little pulling of the CuAl₂ and other constituents of the alloy. The results obtained by polishing 24S-T bar stock electrolytically were much poorer. The added rolling of the sheet stock seems to have the effect of making the nonmetalic inclusions harder and less subject to excessive attack from the electrolyte.

Difficulty was experienced obtaining a satisfactory polish on the 75S-0 specimen of aluminum shown in Fig. 16. Some of the alloying constituents show excessive attack while others are left in relief.

The 195 casting alloy of aluminum shown in Fig. 17 was the easiest to polish of the aluminum alloys used in this investigation. The surface of this metal will

polish with a finish that is somewhat undulated, but the copper alloying constituent will be brought out very clearly.

Steel and aluminum alloys were polished with the same electrolyte without any noticeable detrimental effect to either metal. The steel seems to polish better after the electrolyte has been used for a while for polishing aluminum specimens.

From Table II it is readily noticeable how the temper and alloying constituents affect the current density and time required to obtain a satisfactory polish on aluminum. The aluminum alloys will in most cases be etched satisfactorily by the electrolytic polishing process, however, an etchant solution of HF-HCl-H₂O was used to increase the etch when required.

The brasses polished very easily with slight etching; further etching was accomplished with a solution of hydrogen peroxide and ammonium hydroxide. Photomicrographs of cartridge brass and naval brass are shown in Figs. 18 and 19 respectively. The grain structure and the twinned grains are very pronounced and clear.

The Everdur bronze, a copper-silicon alloy also known commercially as Herculoy, Duronze, Olympic, etc., required a very high current density in order to be polished satisfactorily. The generator used in this

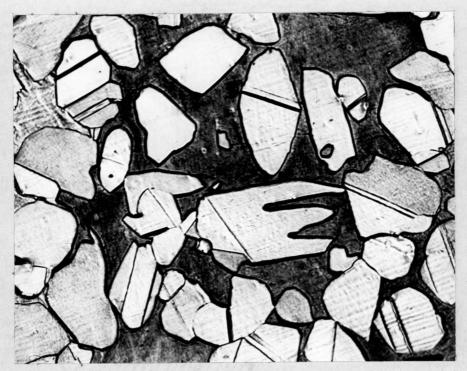


Fig. 18 Cartridge Brass, Electrolytic Polish 15 Seconds, NH₄OH-H₂O₂ Etch, 250X.

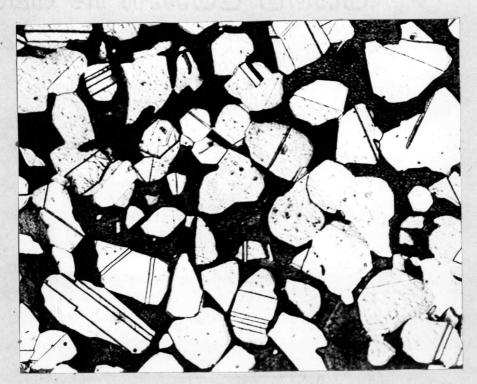


Fig. 19 Naval Brass, Electrolytic Polish 5 Seconds, NH₄OH-H₂O₂ Etch, 250X.

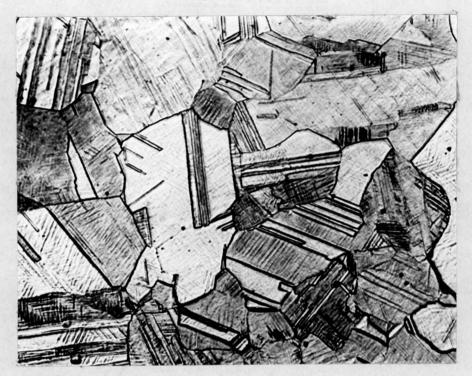


Fig. 20 Everdur Bronze, Electrolytic Polish 5 Seconds, NH₄OH-H₂O₂ Etch, 250X.

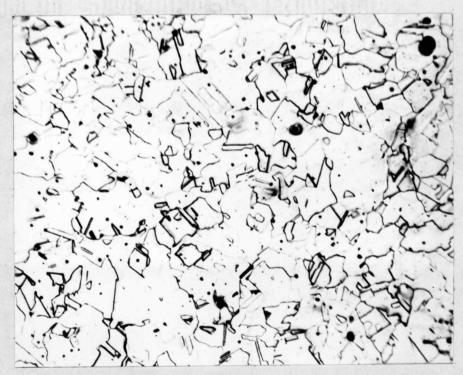


Fig. 21 Monel Metal, Electrolytic Polish 10 Seconds, HNO3-H.C2H3O2 Etch, 250X.

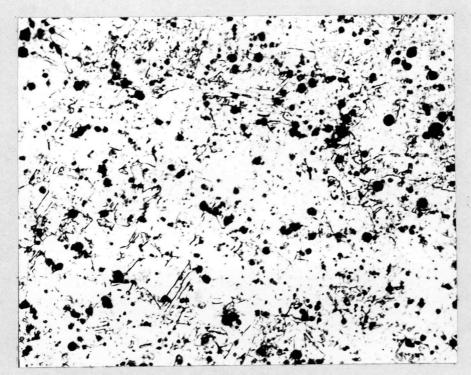


Fig. 22 Copper Bar Stock, Electrolytic Polish 5 Seconds, NH₄OH-H₂O₂ Etch, 250X.

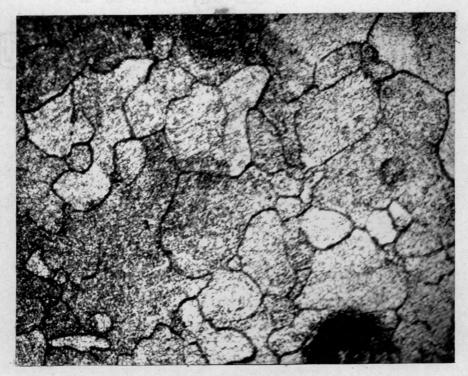


Fig. 23 Lead Sheet, Electrolytic Polish 60 Seconds, HNO₃ Etch, 250X.

investigation was unable to furnish enough current density to polish the entire surface of the ½ inch diameter spot on the specimen; a frosty portion was left at the center. The edge around the small spot was sufficiently large and polished for the taking of photomicrographs and study of the grain structure.

Monel metal, a copper-nickel alloy, gives a bright, unetched surface with numerous small inclusions when polished electrolytically. The specimen of Monel shown in the photomicrograph in Fig. 21 was given a flat etch after polishing with an etching solution consisting of glacial acetic acid and nitric acid.

copper proved difficult to polish electrolytically and obtain a clean, flat surface. The copper oxide that is prevalent in copper and noticeable in the photomicrograph of copper in Fig. 22 is attacked excessively by the polishing action; the resulting surface of the metal after polishing is quite dirty in appearance. Copper does not require any further etching after it is polished electrolytically.

Polishing lead by mechanical methods will make the surface metal flow because the lead is so soft; this flowed metal is removed by dipping in an acid; this operation usually proves difficult for the beginner to perform satisfactorily. Fig. 23 shows the microstructure of lead sheet

after being electropolished for 1 minute in a perchloricglacial acetic acid solution. Lead will polish electrolytically with some pitting leaving parts of the surface with a dirty appearance.

After the electropolishing operation, the lead specimen is dipped in concentrated nitric acid and immediately washed under a stream of running water; this will clean and etch the polished surface. The electrolyte has a tendency to discolor the surface of the metal; this discoloration can be removed satisfactorily with nitric acid.

The preliminary polishing of the surface of the specimen with metallographic emery paper to ready it for the electrolytic polishing was tried with the 00 and 000 papers on each metal. The specimens given a preliminary finish with the 000 paper polished faster and with less surface undulation than those given a preliminary finish with the 00 paper. With aluminum there was less attack on the nonmetallic inclusions because the polishing time was shortened. The low carbon steels appeared less dirty for the same reason.

The electrolytic polishing solutions finally decided upon were selected for the following reasons: Ease of preparation, safe to use, heating not required for polishing, would polish several metals, were of the

straight-line type, and gave fast results. Other electrolytes used in this investigation but discarded in favor of those listed previously in the paper were:

Steel:	1.	Phosphoric acid Sulphuric acid Chromic acid Water	65 15 5 15	(wt)
	2.	Phosphoric acid Sulphuric acid Water	48 40 12	(wt)
Brass:	1.	Phosphoric acid Water	70 30	(wt)
Aluminum:	1.	Nitric Acid (con) Methyl alcohol (abs)	2	(vol)

The phosphoric acid solutions require temperatures ranging from 90F to 300F in order to polish effectively. The polishing cell was not designed to hold electrolytes that required heating. The phosphoric acid solutions also require a relatively long time to perform the polishing operation satisfactorily; they use a low current density thus removing metal slowly. Controlling the voltage within proper limits for polishing with the phosphoric acid solutions proved much more difficult than controlling the current required for polishing with the straight-line type solutions. The cell used in this investigation gave much better results with the straight-

line type solutions than with those of the phosphoric acid type.

"Perchloric acid is a violent explosive" (3, 430).

Several of the perchloric acid electrolytes used in electrolytic polishing have been known to explode when coming in contact with some organic substance.

The perchloric acid solution and the nitric acid solution used in the polishing of steel, aluminum, and copper alloys are easily mixed and are not explosive; "they do not attack bakelite specimen mounts" (7, 609). With specimens mounted in lucite, electrolytic polishing in the nitric acid-methyl alcohol solution was found to turn the lucite a milky white color on the surface. Lucite is not recommended for mounting specimens that are to be polished in this solution. In this investigation mounted specimens were not polished in the perchloric acid solutions.

The electrolyte containing nitric acid and methyl alcohol was used to polish aluminum as well as brass; the results on aluminum were comparable with those obtained with the perchloric acid electrolyte. The same solution, however, could not be used for both copper and aluminum alloys without staining the surfaces of both metals when polished. The nitric acid-methyl alcohol

solution was discarded in favor of the perchloric acid solution for the polishing of aluminum alloys.

The electrolytic solutions will become tinted after usage; this is no cause for alarm. The solution will become more stable and give better polishing results after it has developed a tinted color. When the required current density cannot be obtained without an excessive increase in voltage, the solution is getting old and should be renewed. Small additions to the electrolytic polishing solutions to make up for that lost through evaporation and spilling should give them a long life. Only one batch of each solution was used in this investigation with good results still being obtained when the experimentation ended.

VII. CONCLUSIONS

Electrolytic polishing proves to be a fast method of obtaining a surface finish suitable for micrographic examination; in some cases the finish will be comparable with good mechanical polishing. The saving of time and energy with less skill are the principal advantages.

As yet no universal electrolyte has been developed for the polishing of all metals; at best, only several different metals can be polished with any one electrolyte. The operating conditions of the polishing cell will also vary with the alloy of a metal and in some cases, the temper.

The pure metals or those in solid solution will be best polished by electrolytic action; heterogeneous alloys are best polished mechanically. In some cases the nonmetallic inclusions will be excessively attacked during the polishing action while others will be left in relief; this action will leave some of the aluminum alloys and the lower carbon steels with a dirty appearance.

For photomicrographic examination specimens must be given a preliminary smoothing of the surface with metallographic emery paper; the finer the preliminary finish the faster the electrolytic polishing action can be performed, and the better will be the resulting finish. Undulation of the polished surface which is characteristic in electrolytic polishing is lessened with fine preliminary preparation of the specimen.

Electrolytic polishing is best adapted to industry or a metallurgical laboratory where large amounts of one alloy or a number of one type of specimens have to be polished. Mechanical polishing apparatus will still be needed in every laboratory where micrographic work is being done; electrolytic polishing can only supplement.

TABLES

Table I. Electrolytic Polishing Conditions For Steel And Its Alloys

Electrolyte:	Perchloric acid (70-72%) Ethyl alcohol with 3% ether	800 ec	Cathode: Stainless steel Temperature: Room
	Water	146 cc	Spot diameter: 1/4 inch

Specimen	Voltage	Amperes	Time Seconds	Cathode-anode Distance Inches
0.10% Carbon	40	0.65	20	1 3/4
0.43% Carbon	41	0.55	20	1 1/2
0.97% Carbon	40	0.55	20	1 1/2
White Cast Iron	40	0.70	10	1
Low Carbon Cast	40	0.60	30	1
Type 302 Stainless	38	0.50	60	1 3/4

Table II. Electrolytic Polishing Conditions For Aluminum Alloys

Electrolyte:	Perchloric acid (70-72%)		ec	Cathode: Stainless steel	L	
		Ethyl alcohol with 3% ether	800	cc	Temperature: Room	
		Water	146	cc	Spot diameter: 1/4 inch	

pecimen	Voltage	Amperes	Time Seconds	Cathode-anode Distance Inches
28	30	0.25	60	2
24S-T	20	0.06	15	2
758-0	34	0.35	30	2
195	40	0.08	15	2 1/4

Table III. Electrolytic Polishing Conditions For Copper And Its Alloys

Electrolyte:	Nitric acid (Con) Methyl alcohol (Abs)	600 cc 300 cc	Cathode: Stainless Steel Temperature: Room Spot diameter: 1/4 inch
			ppo aramoter a/ a anon

Specimen	Voltage	Amperes	Time Seconds	Cathode-anode Distance Inches	
Cartridge Brass	15	0.65	15	2 1/4	
Naval Brass	15	1.0	5	2 1/4	
Everdur Bronze	41	3.0	5	3 1/2	
Monel Metal	28	2.5	10	1	
Copper	30	1	5	2 1/2	

Table IV. Electrolytic Polishing Conditions For Lead

Electrolyte:	Perchloric Acid	(70-72%)	300 700		Cathode: Stainless Temperature: Room Spot diameter: 1/4	
Specimen	Voltage Amperes		Time Seconds		Cathode-anode Distance Inches	
Lead	40	0.65		50	1	

BIBLIOGRAPHY

VIII. BIBLIOGRAPHY

- 1. American Society for Testing Materials. Electrolytic Polishing of Metallographic Specimens. A.S.T.M. Standards 1-B: 819-25, 1946.
- 2. Jacquet, P. Electrolytic Methods of Obtaining Bright Copper Surfaces. Nature 135: 1076, 1935.
- 3. Merchant, M. E. Warning! Perchloric Reagents
 May Explode. Metals And Alloys 13: 430,
 April 1941.
- 4. Parcel, R. W. Simplified Electropolishing of Steel Specimens. Metal Progress 42: 209-12, August 1942.
- 5. Thum, E. E. Speedy Polishing Without Abrasives.
 Metal Progress 36: 755-6, December 1939.
- 6. Tour, S. Pickling and Polishing of Metals. The Iron Age 145: 56-60, May 23, 1940.
- 7. Waisman, J. L. Metallographic Polishing. Metal Progress 51: 606-10, April 1947.
- 8. Zmeskal, 0. Electrolytic Polishing of Stainless Steel and Other Metals. Metal Progress 47: 729-36, April 1945.