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SEMIAUTOMATIC PREPARATION OF SPECIMENS FOR TRANSMISSION ELECTRON MICROSCOPY

C. K. H. DuBose
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OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

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METALS AND CERAMICS DIVISION

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C.K.H. DuBose J. O. Stiegler

FEBRUARY 1967

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SEMIAUTOMATIC PREPARATION OF SPECIMENS FOR TRANSMISSION ELECTRON MICROSCOPY

C.K.H. DuBose J. O. Stiegler

ABSTRACT

A rapid expansion in the use of transmission electron microscopy at the Oak Ridge National Laboratory has led to the development of new equipment designed to produce specimens of a variety of materials, often after neutron irradiation, in a quick and reliable manner. Specimens are cut, in a hot cell if necessary, to a thickness of about 20 mils and a maximum diameter of 1/8 in. These are first "dimpled" on both sides by an electrolytic jet technique to reduce the thickness of the center of the specimen to about 2 mils. The shape of the dimple bottom is controlled by varying the current density; and under appropriate conditions large, flat-bottomed dimples can be formed. Final polishing is accomplished in a standard electrolytic polishing cell in which breakthrough in the dimpled region is detected by an extremely sensitive photocell device. Incident light is piped to the specimen and the light transmitted through the hole is delivered to the photocell by a fiber-optics system. This system provides a small, intense light beam and also eliminates interference from thermal fluctuations of the polishing solution and radiation effects on the photocell. Activation of the photocell stops the polishing action in the final step.

With this technique, extremely small holes can be detected, which usually are surrounded by reasonably large areas transparent to 100 kv electrons. The technique is extremely rapid compared to standard techniques using large foils, and it does not require close attention of the operator. Its semiautomatic nature permits handling of radioactive materials with little radiation exposure. It is now possible to go from bulk sample to a finished specimen in about 30 min. Full details on design of the equipment and operations of the technique are included.

INTRODUCTION

An increase in the application of transmission electron microscopy to a large number of metallurgical problems at the Oak Ridge National Laboratory has led to the development of new equipment and techniques for preparing thin foils of a wide variety of materials, often after neutron irradiation, in a quick and reliable manner. Standard techniques, such as Bollman[1] or window[2,3] are slow, demand close attention of an operator, and require foils initially 1/2 in. square by 10 mils thick as starting material, which precludes studies of many interesting engineering materials.

In designing the equipment we have kept in mind a number of requirements of the Metals and Ceramics Division. Since interest is increasing in studies of irradiated structural and fuel materials, any equipment must be capable of working with specimens having radiation levels up to several roentgens per hour. The expense of reactor irradiations and spatial limitations of research reactors make it desirable that the technique be capable of using bulk specimens on which other tests are run. The technique must be applicable to a wide variety of materials and must be simple and reliable enough that it can be used by technicians having little training in electron microscopy.

This report describes in detail the technique that has evolved from our experience. The technique consists of three separate operations. First, a wafer about 1/8 in. in diameter, or square, by about 20 mils thick is mechanically cut from a bulk specimen. Next, a jet of electrolyte is used to produce depressions on opposite flats leaving an area about 2 mils thick surrounded by a thick ring of unpolished material. Finally, this piece is inserted in a standard electropolishing cell where it is illuminated from one side by an intense light beam transmitted by a fiber-optics system; when a hole is formed by further polishing, the transmitted light activates a detector which stops the polishing action and sounds an alarm.

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[1]W. Bollman, Phys. Rev. 130, 1588 (1956).

[2]R. B. Nicholson, G. Thomas, and J. Nutting, Brit. J. Appl. Phys. 9, 25 (1958).

[3]H. M. Tomlinson, Phil. Mag. 3, 867 (1958).

JET POLISHING

A number of jet polishing techniques[4–12] have been reported for machining or dimpling specimens for electron microscopy. They all employ a fine jet of electrolyte to remove material from a limited area. Gravity, air pressure, or mechanical pumps have been used to force the electrolyte through the orifice. No details have been reported on the nature of the polished surface; dimples have generally been described as dish shaped.

Difficulties with control of electrolyte flow rate with mechanical pumps and with corrosion of hypodermic needles led us to all glass systems using air pressure, as shown in Fig. 1. The unit is built around an erlenmeyer flask that serves as a storage reservoir for electrolyte. A thermometer port is built in for measuring temperature, which is controlled by a heater tape or large dish of a cooling liquid in which the entire assembly may be placed. Air pressure at about 2 psig forces the electrolyte up through a tube containing a platinum wire cathode and out through an orifice approximately 20 mils in diameter. A micromanipulator positions the specimen with respect to the jet; specimen-to-nozzle distances are on the order of 1/2 in. The specimen is clamped in platinum-tipped tweezers or in a special Teflon holder which allows more reproducible positioning. The specimen is set in a beaker which is attached to the reservoir flask by a check valve, so that when the pressure is removed the electrolyte will flow directly back to the reservoir.

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[4]P. R. Strutt, Rev. Sci. Inst. 32, 411 (1961).

[5]R. P. Riesz and C. G. Bjorling, Rev. Sci. Instr. 32, 889 (1961).

[6]G. Blankenburgs and M. J. Wheeler, J. Inst. Metals 92, 337 (1963-64).

[7]Eugene S. Meieran and David Thomas, Trans. AIME 227, 284 (1963).

[8]G. R. Booker and R. Stickler, Brit. J. Appl. Phys. 13, 446 (1962).

[9]R. Stickler and R. J. Engle, J. Sci. Instr. 40, 518 (1963).

[10]L. I. VanTorne and G. Thomas, Rev. Sci. Instr. 36, 1042 (1965).

[11]R. C. Glenn and R. D. Schoone, Rev. Sci. Instr. 35, 1223 (1964).

[12]R. D. Schoone and E. A. Fischione, Rev. Sci. Instr. 37, 1351 (1966).

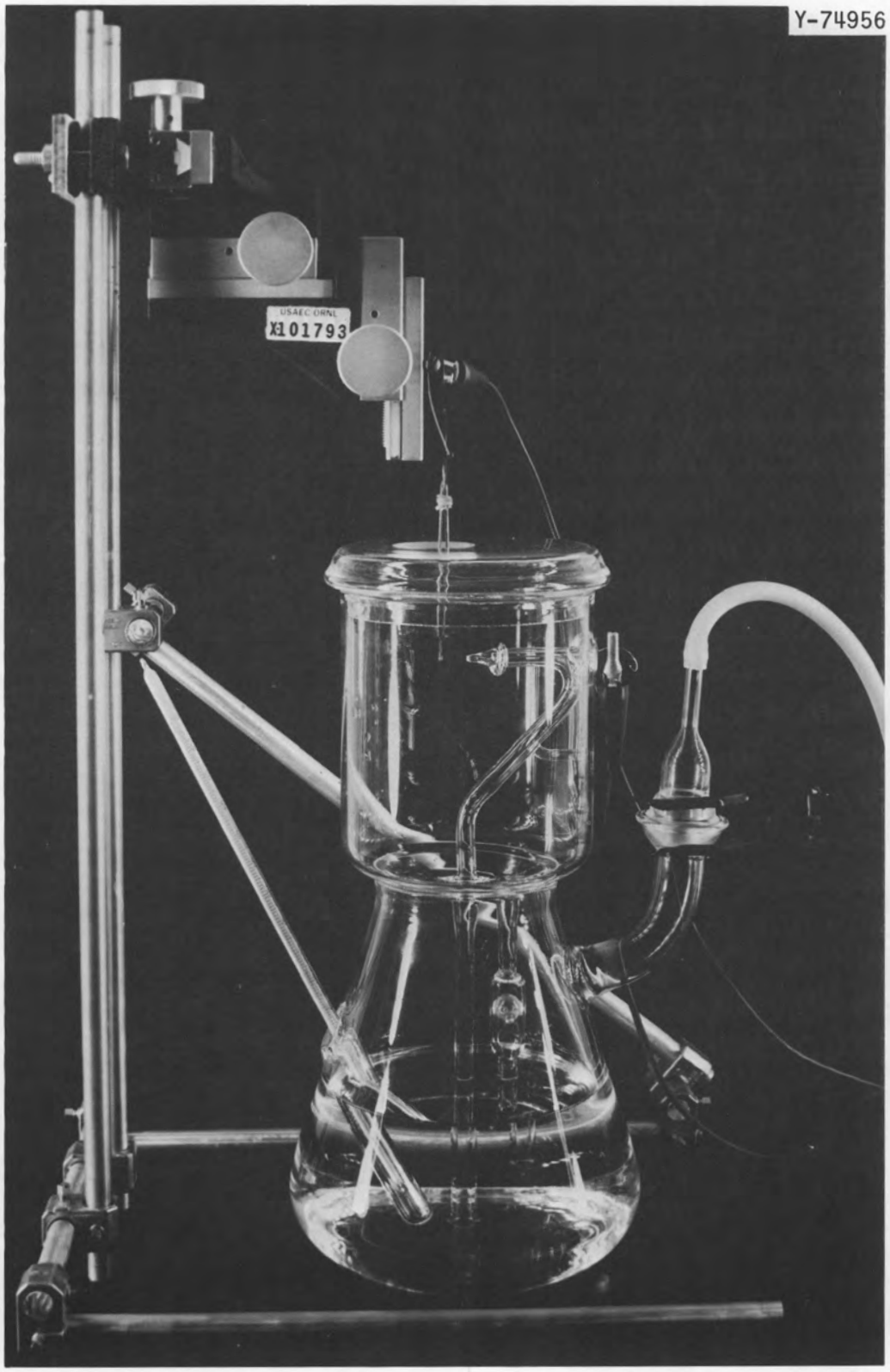

Fig. 1. Jet Polishing Rig. The unit is constructed from standard laboratory glassware. See text for details on operation.

Conditions for jet polishing are generally different from those for standard electropolishing. In the following sections we will discuss the effects of the polishing variables.

Voltage

Current-voltage characteristics of standard low resistance electropolishing cells in which electrodes are on the same order of size as the specimen are of the form shown in Fig. 2. As the voltage applied across the cell is increased, the current also increases to a point where it remains relatively independent of voltage (the so-called polishing plateau-region CD). Above the plateau, current increases rapidly with an increase in voltage and gas evolution usually occurs. Etching of the specimen usually occurs at voltages below the plateau (AB) and uneven polishing or pitting above it (DE). Reasonably good polishes can be obtained across the plateau although at the low voltage end specimen edges are polished preferentially, while at the high voltage end the specimen centers receive the best polish. The nature and extent of the plateau are dependent on the electrolyte and on the electrode configuration, disappearing as the resistance of the electrolyte increases and as the cathode size decreases.[13]

The situation is quite different for the jet polishing rig described here. A polishing plateau is never observed, but rather the behavior depicted in Fig. 3 in which current always increases with an increase in voltage. Surfaces in the path of the electrolyte are polished over the entire range of voltage; the shape of the polished region is, however, very sensitive to the voltage, as is shown in the inserts in Fig. 3. At low voltages the centers of the polished regions are preferentially polished, creating the well-known dish-shaped depressions. At intermediate voltages polishing is uniform over the cross section of the jet leading to flat-bottomed dimples. At high voltages the current density is highest at the perimeter of the jet giving rise to depressions that are crowned at the center and low at the edges. Dimples produced by the jet polishing unit described here are about 1/16 in. in diameter.

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[13]Pierre A. Jackquet, Met. Rev. 1, 157 (1956).

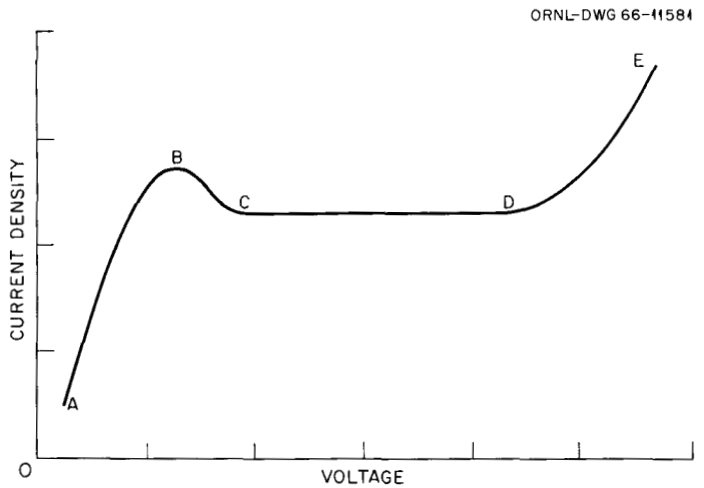

Fig. 2. Schematic Illustration of the Dependence of Current on Voltage Applied Between the Specimen and the Cathode(s) in a Low-Resistance Electropolishing Cell. Etching occurs in the region AB, polishing in CD, and pitting and rapid gas evolution in DE.

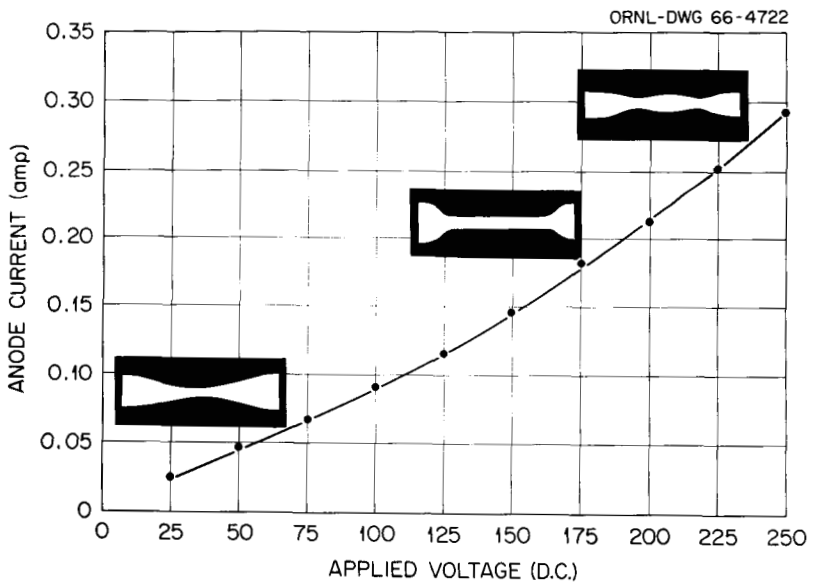

Fig. 3. Anode Current vs Applied Voltage for the Jet Polishing Unit Shown in Fig. 1. Polishing occurs at all voltages. The shapes of the polished areas are shown in the inserts. Data are for type 304 stainless steel. See Appendix A for polishing solution.

With the jet technique it is possible to work at what would normally be considered high voltages for electropolishing. The jet evidently prevents buildup of the viscous anolyte that controls polishing under conditions of lower voltage. Material is removed at a rate of a few mils per minute.

Specimen-to-Nozzle Distance

The dimple shape is largely a function of current density, for similar behavior can be obtained at constant voltage by varying the specimen-to-nozzle distance. The relationship between these variables is illustrated in Fig. 4. Figure 5 is a plot of anode current against applied voltage for several different distances. The actual value of current for a particular voltage also depends on the location of the cathode with respect to the tip of the nozzle.

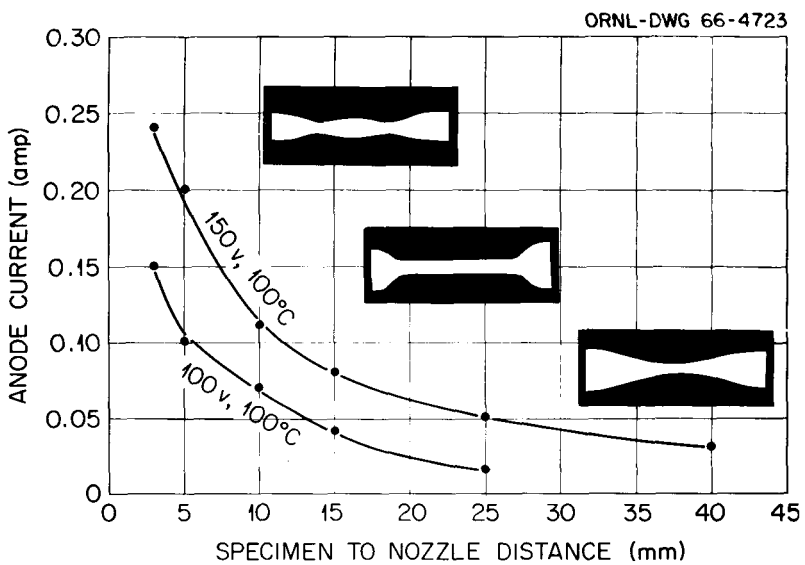

Fig. 4. The Effect of Specimen-to-Nozzle Distance on Current and Shape of the Polished Region. Data are for stainless steel (see Fig. 3).

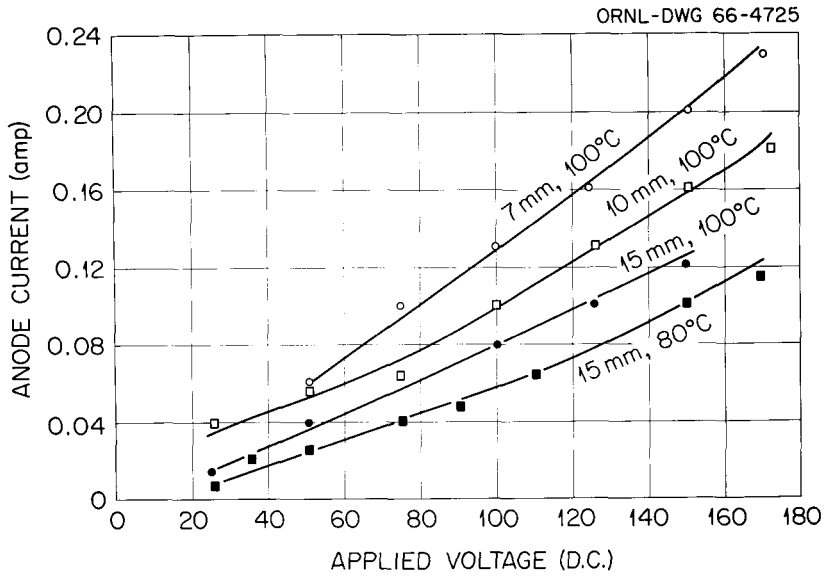

Fig. 5. Effect of Specimen-to-Nozzle Distance and Solution Temperature on Current-Voltage Characteristics for the Jet Polishing Unit. Data are for nickel.

Pressure or Flow Rate

The voltage-current characteristics are relatively independent of the flow rate of electrolyte although the quality of the polish is generally poor at extremely high or extremely low rates. At normal rates of flow (50 ml/min at 2 psig) a round, uniformly polished dimple can be obtained, Fig. 6. At low flow rates the polished region becomes tear shaped, and at very high rates inclusions cause the star-shaped effect shown in Fig. 7. At high rates splashing also becomes a problem.

Electrolytes

In many cases electrolytes used for standard electropolishing can be used for jet polishing although temperature ranges for obtaining good polishes often are significantly different. In many cases different solutions produce better results or are more desirable from a safety standpoint. Since the high voltages used in jet polishing can cause arcing, solutions containing alcohol and percaloric acid are potentially dangerous. Appendix A lists electrolytes and polishing conditions that we have found to be successful.

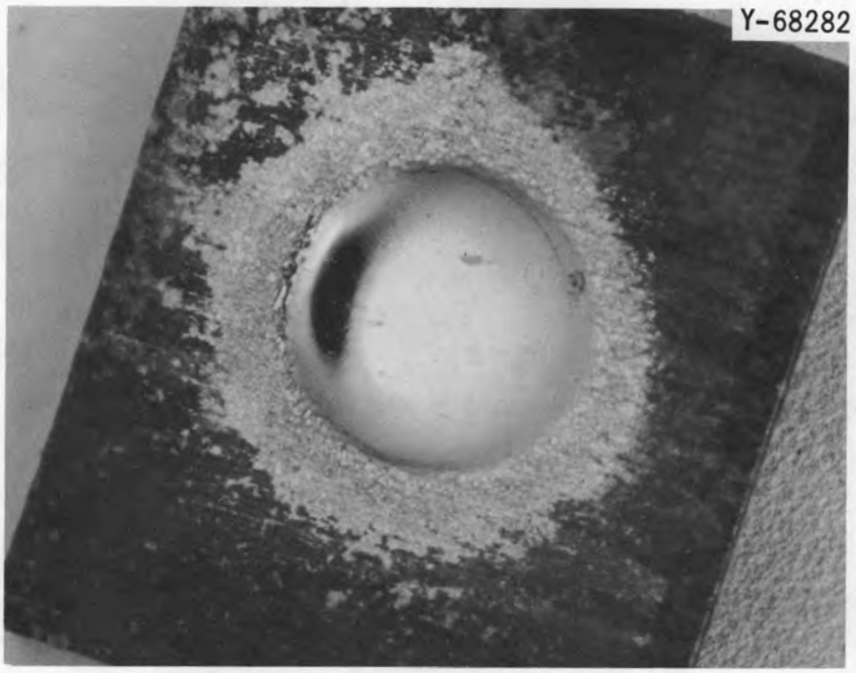

Fig. 6. Macrophotograph of Jet Polished Stainless Steel. Note the confinement of the polish to the area in the path of the jet and the flat-bottomed nature of the dimple.

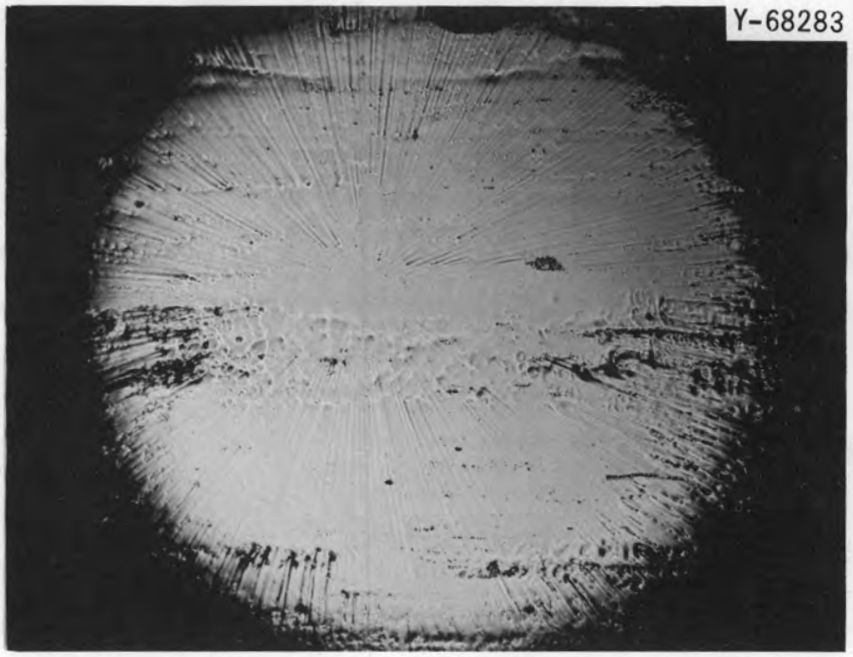

Fig. 7. Dimpled Area of a Stainless Steel Specimen Containing Large Second Phase Particles that was Subjected to a High Electrolyte Flow Rate.

Procedure

The technique because of its simplicity is extremely reproducible and easy to apply. Once polishing conditions have been determined, a specimen should be polished until the jet polishes a hole through it. The thickness divided by the time to cause perforation gives a good estimate of removal rate. The specimen should be clamped with tweezers or mounted in a holder and carefully positioned with respect to the jet. Both sides should be dimpled until a thickness of about 2 mils is left. This eliminates the possibility of mechanical damage from the jet. By controlling the relative times of polishing from each side it is possible to select sections from about any depth in the specimen, the only limitation being that sufficient material be removed to eliminate regions damaged by the cutting operation. We have tried some double jet rigs but found it difficult to reproduce jet geometry and electrode placement in the two jets, leading to different polishing conditions on the two sides of the specimen. This could be overcome by using different power supplies for the two sides, but the difficulties associated with defining the polishing conditions make use of the single jet easier and more desirable.

The technique has the advantage that it requires a minimum amount of handling and attention by the operator. The specimen does not need to be masked; it can be clamped in tweezers or held in a simple holder and aligned with respect to the jet in a matter of a few seconds. Figure 6 shows that the polishing is confined to the area of the path of the jet. Once polishing conditions have been defined, specimens can be handled routinely in a matter of a few minutes. In addition, we have used it on specimens reading up to 10 r/hr with only slight exposure (few milliroentgens) to the operator.

FINAL ELECTROPOLISHING WITH A LIGHT DETECTOR

As was mentioned earlier, samples are obtained by electropolishing the dimpled wafers in a standard electropolishing cell until a hole forms. We have adapted the cell slightly to incorporate a light source and detector to halt automatically the polishing action at the time of

perforation. Although this seems to be a fairly straightforward operation, it did require several unique modifications.

Polishing Cell and Cathodes

The cathodes and light source compete with each other for space in the cell in the sense that they should both face the surface being polished. Schoone and Fischione[14] solved this problem by positioning submerged jets containing the cathodes normal to the specimen surface and by illuminating the specimen with a beam of light incident at an angle of about 45° from the normal. We felt that the illumination system demanded the highest priority and designed our polishing cell around it. The cell is constructed from a 90-mm crystallizing dish with two 10-mm-diam tubes having optically flat ends extending in about 30 mm from opposite sides, as shown in Fig. 8. The sample is then located midway between the tubes that serve to introduce and remove light from the cell.

The cathodes are flat plates having circular holes equal to the outer diameter of the light tubes and thus fit around the tubes in the manner shown in Fig. 9. We have found that this cathode design does not have a significant effect on the nature or the extent of the polish as long as the specimen is small compared with the cathodes, which is always the case in this arrangement. The cathodes are mounted in a bar that rests on the polishing cell which allows them to be adjusted with respect to each other and the specimen. The negative lead is attached to a screw on the bar.

The entire cell normally rests on a hot plate having a built-in magnetic stirring device. For solutions requiring a low operating temperature, the electropolishing cell sits in a larger dish that can contain a liquid held at the desired temperature. The specimen is gripped with platinum-tipped tweezers that can be positioned with respect to the light beam with a micromanipulator. No masking is required if the specimen has been dimpled as described previously. Figure 10 shows the assembly with a specimen in position for polishing.

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[14]R. D. Schoone and E. A. Fischione, Rev. Sci. Instr. 37, 1351 (1966).

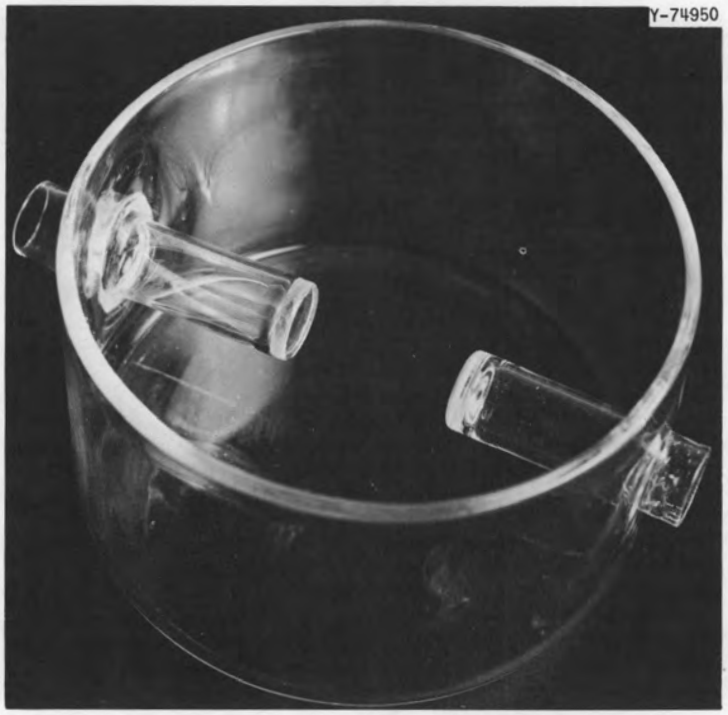

Fig. 8. The Final Polishing Cell. Tubes having optically flat ends have been inserted into the cell to admit and remove the light.

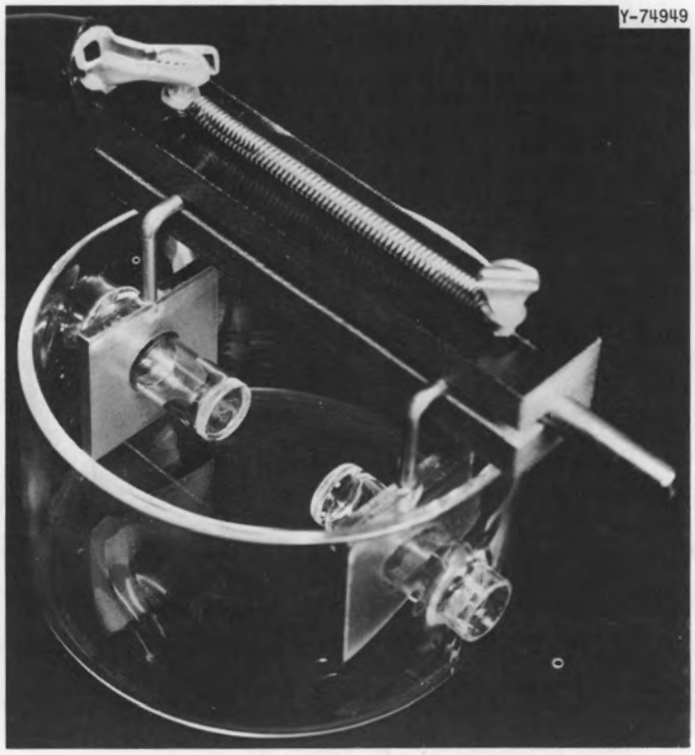

Fig. 9. Final Polishing Cell with Cathode Assembly in Place.

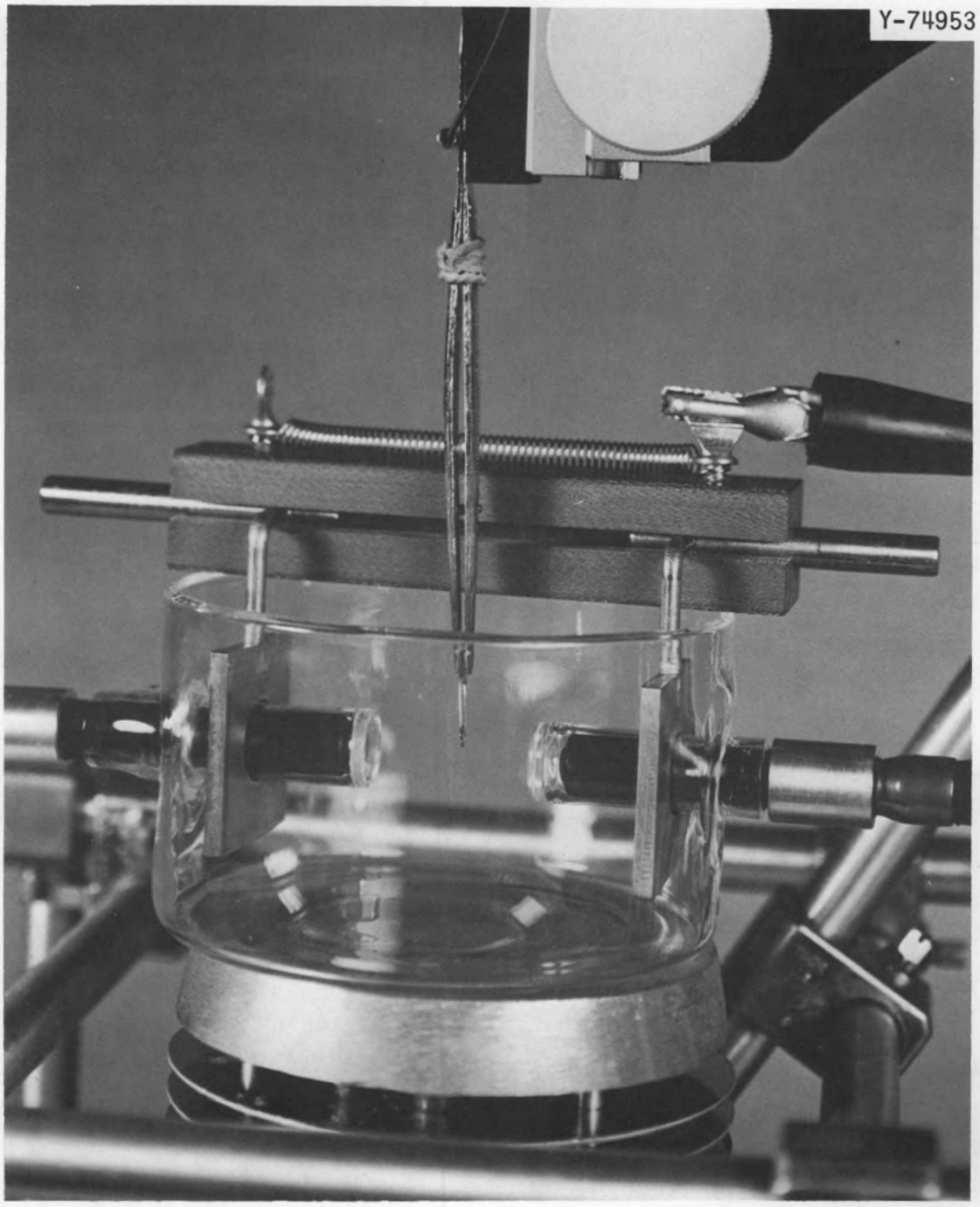

Fig. 10. Polishing Cell with Fiber Optics and Specimen in Place. Note that the specimen is clamped by platinum-tipped tweezers. No masking is required.

Light Source

Initially, we located a small bulb in one of the light tubes but found that with bulbs that would fit in the tube we were unable to obtain sufficiently high light intensities. This led to the concept of a high intensity source from which light is piped to the polishing cell by means of a fiber-optics system.

The light source, shown in Fig. 11, is built around a type DAH high intensity projection lamp (500 w). To dissipate heat the lamp is mounted in a chimney, and cooling air is forced around it with a rotary blower. In addition, a vacuum tube heat shield, having a hole cut out for the light beam, is used on the lamp to enhance heat removal. An interlock prevents operation of the light without first turning on the blower. In order to prolong lamp life, the lamp filament is brought up to rated current gradually with an unijunction transistor-triac circuit. A running time meter is included in the circuit to allow replacement of the lamps before their rated lifetime of 200 hr is reached.

The light beam is coupled to the fiber-optic bundle through a clad-glass rod, which absorbs enough of the infrared component of the light beam to keep the temperature at the face of the epoxy-bonded fiber-optic bundle down to an acceptable level. The bundle transmits the light to the polishing cell where a lens system at the end of the light tube focuses it to a spot about 1/16 in. in diameter at a point midway between the light tubes, Fig. 12. A fine focus lens with a built-in aperture, focusing at 1/2 in., is fitted to the terminal end of the fiber-optic bundle from the light source, as can be seen in Fig. 10. Another lens, less the aperture, is attached to the bundle leading to the photocell. This second lens is focused on the rear of the specimen, thus reducing the amount of ambient light reaching the photocell. These lenses are attached to the fiber-optic bundles by means of an aluminum tube, approximately 3/4 in. long and threaded inside. The metal end of the bundle is wrapped with rubber tape, and thus can be self-threaded.

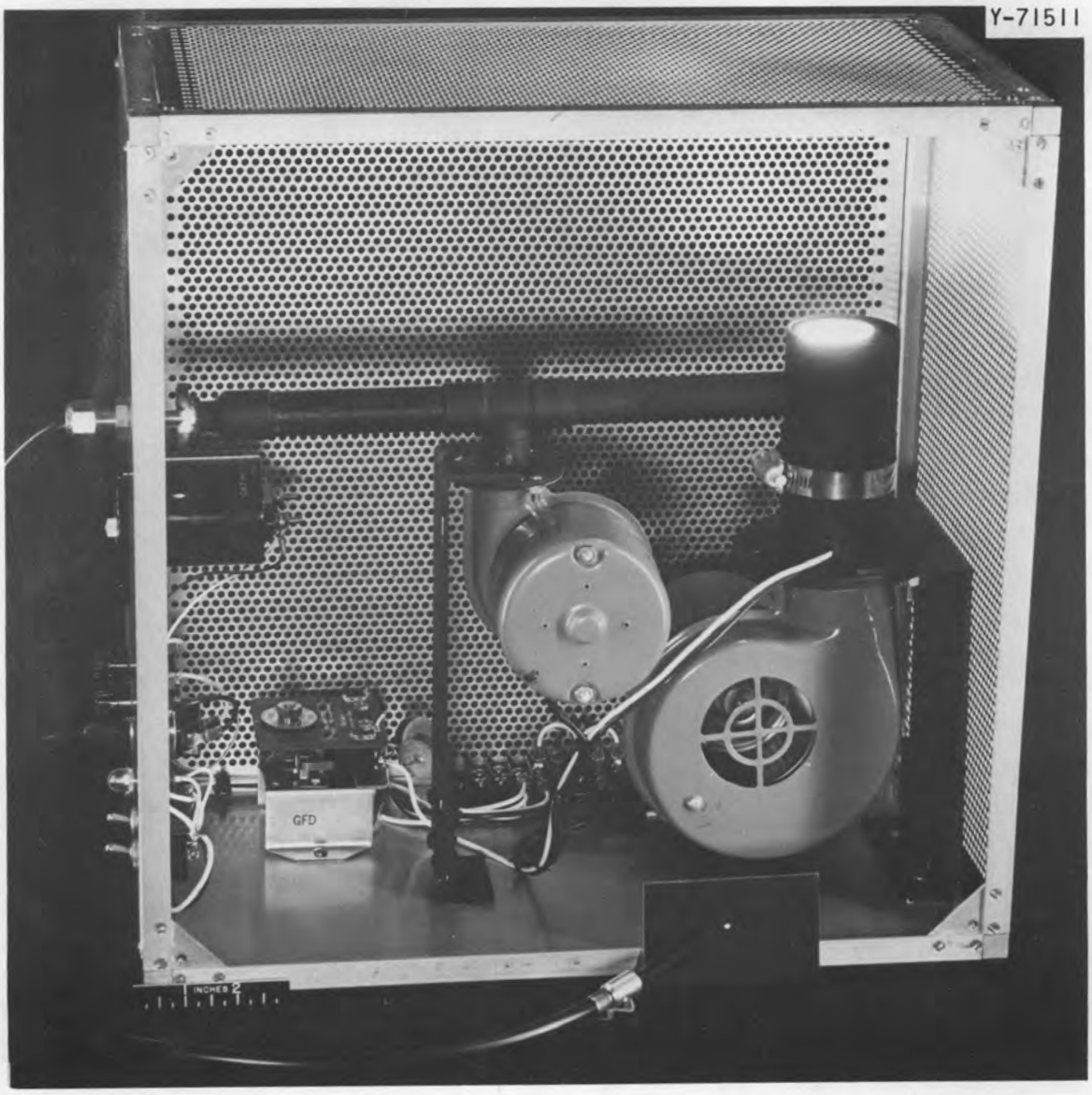

Fig. 11. Light Source. See text for description.

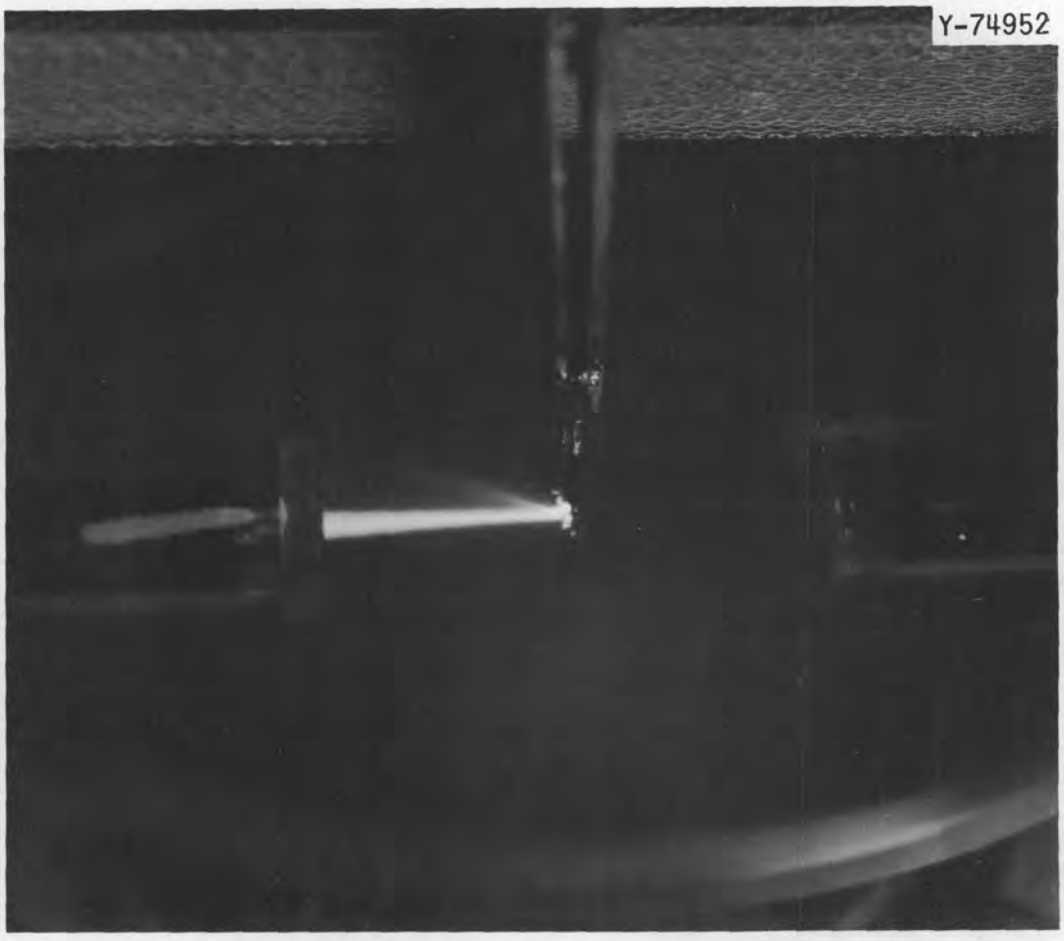

Fig. 12. Polishing Cell in Operation. The light beam can be seen striking the specimen. Notice the focusing action of the lens system.

Light Detector and Controller

In our initial experiments, cadmium-sulfide cells were located in one of the light tubes to receive and detect light transmitted by the sample. Two difficulties prevented successful operation: (1) temperature fluctuations in the polishing solutions led to fluctuations in the output of the photocell that interfered with normal operation and (2) the cadmium-sulfide cell was sensitive to a fairly narrow range of wavelengths that were heavily absorbed in many of our standard polishing solutions. We overcame the thermal fluctuations by extracting the light from the cell by another fiber-optics system and obtained broader response by using a photosensitive field-effect transistor to detect the transmitted light. The wide spectral response of this detector (Siliconix P-102, which has usable sensitivity of 0.4 to 1.1 μ) ensures that absorption

bands on particular polishing solutions have minimum effect on the operation of the controller. The filed-effect transistor is used in a source follower circuit[15] that is noncritical in adjustment, yet has a photosensitivity which approaches that obtainable with photomultiplier tubes. Several sensitivity ranges are provided to accommodate various polishing solutions with different optical densities. Output from the detector circuit drives an optically coupled meter relay with latching contacts. When the meter reading goes above a preset limit at the time a hole forms in the sample, the meter relay contacts open to de-energize a power relay and stop the polishing action. An alarm can be sounded at the same time. A schematic diagram of the electronic circuits is shown in Fig. 13. The relay switch, shown in the schematic, is used to halt the direct-current flow to the specimen when polishing is completed. In this way the direct-current supply is left on which prevents any emf feedback to the specimen as would occur if the entire power supply were cut off. The entire assembly is shown in Fig. 14.

The potential at which final polishing is accomplished is very critical, and we have incorporated a chart recorder to determine more reliably the polishing curve. By recording the specimen current while increasing the applied voltage at a constant rate the proper polishing plateau can be established within a few seconds. A typical chart recording showing the plateau is shown in Fig. 15.

Procedure

With the light source on, the specimen is placed at the midpoint between the lenses The specimen is then more accurately positioned with the micromanipultor until a minimum reading is recorded on the light meter coupled to the photocell. A range or sensitivity selector switch is provided to compensate for differences in light transmittance of various electrolytes. The electronic zero adjust should be used to set the pointer near midscale. The cutoff pointer should then set slightly above this reading to prevent minor fluctuations from cutting

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[15]M. Shipley, Solid State Design 5, 28 (April 1964).

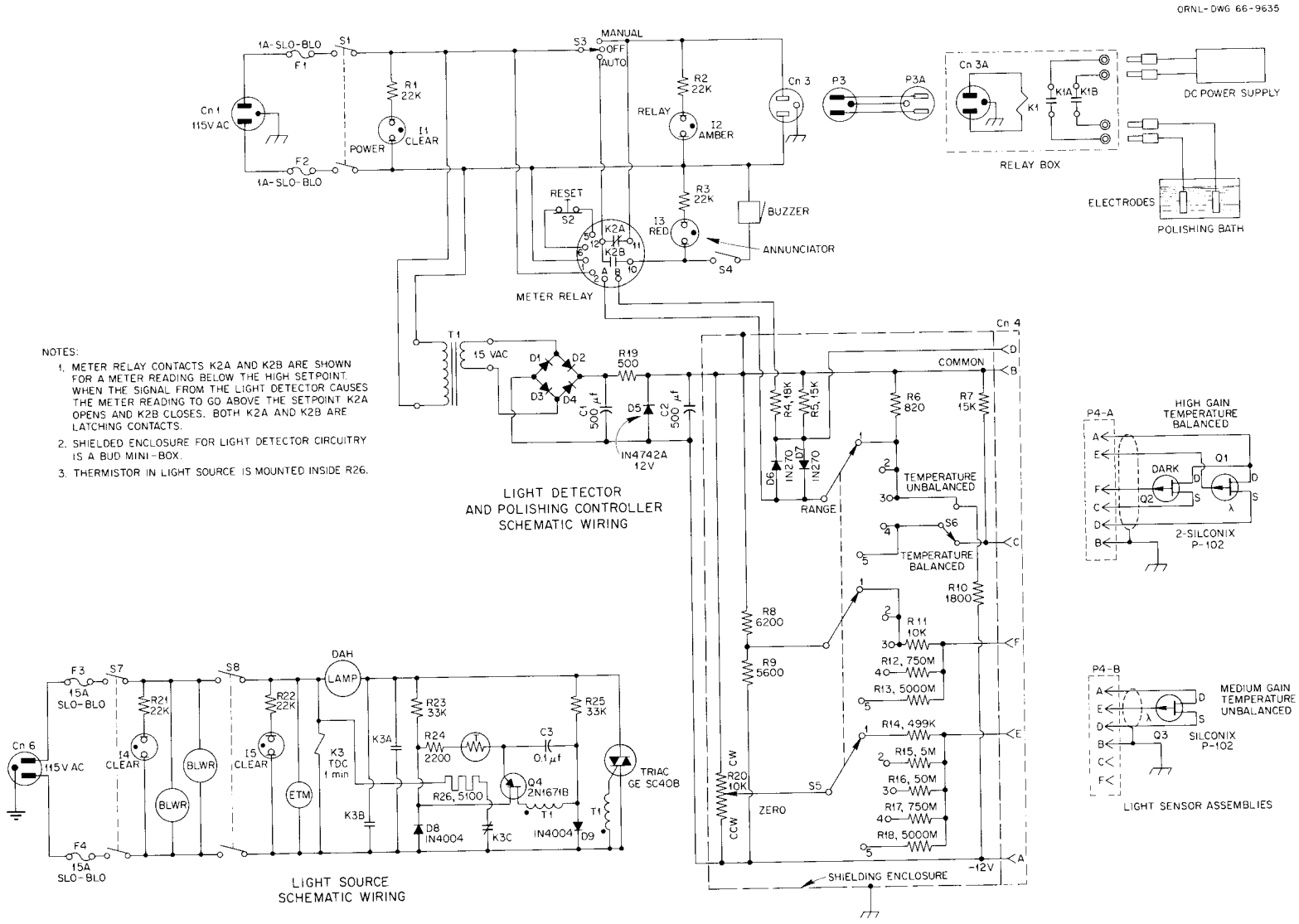

Fig. 13. Schematic Diagram of the Electronic and Illumination Systems. Appendix B contains a parts list.

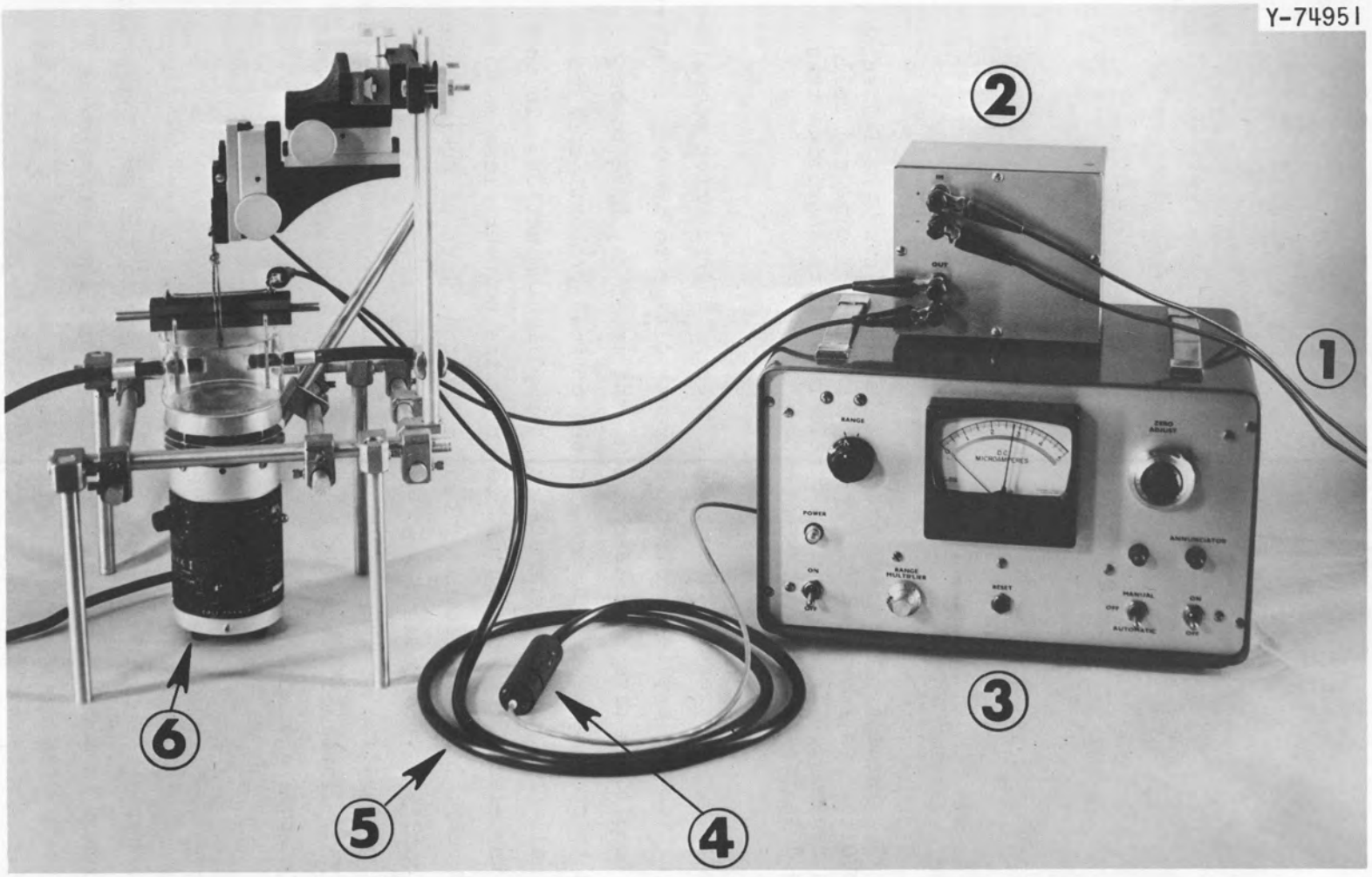

Fig. 14. The Polishing Cell and Controller. Leads to direct-current power supply, relay box, controller, field-effect transistor, fiber-optic bundle, and hot plate with magnetic stirrer.

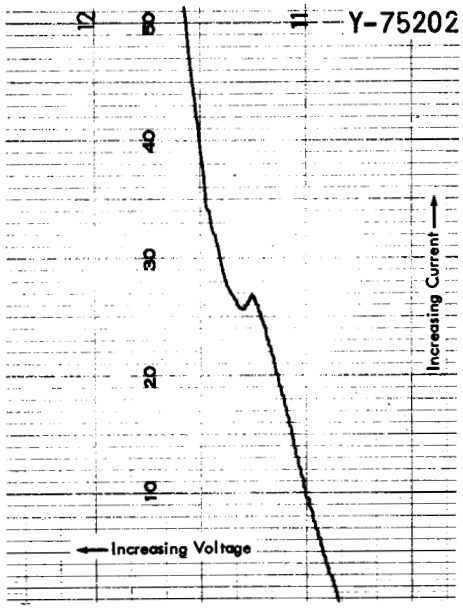

Fig. 15. Portion of Strip Chart Used in Determining the Polishing Plateau. Voltage is increased slowly as a function of time and cell current is recorded. This method is considerably more sensitive than making the determination by eye.

off the voltage. The reset button should then be pushed which energizes the relay switch and applies a direct-current voltage to the cell. After polishing conditions have been determined, the meter may have to be readjusted to compensate for changes in the optical properties of the polishing cell from bubbles generated by the polishing action. In this case, use the electronic zero adjust to bring the pointer close to the cutoff pointer. Light refracted by bubbles that collect on the specimen may occasionally give a false alarm.

After perforation of the sample, we use a standard washing technique of warm (55°C) distilled water, Versene in water, distilled water again, and finally ethyl alcohol.

SUMMARY

The techniques described here have evolved during the past two years from experience in our laboratory. They have shown themselves to be applicable to a wide variety of materials and to be extremely rapid as well as reliable. Their semiautomatic nature has permitted routine

handling of specimens having radiation levels up to several roentgens per hour by technicians having no prior experience in electron microscopy. Since starting material requirements are not very stringent, approximately 20 mils thick by about 1/8 in. in diameter, specimens may be prepared from bulk pieces on which other observations or tests have been made. Although the electronic equipment is quite sophisticated, both the jet polishing and photo detector units can be built for a combined cost of well under $1000. Detailed drawings can be obtained from the authors.

ACKNOWLEDGMENTS

T. M. Gayle and J. T. Hutton of the ORNL Instruments and Controls Division designed and developed the light source and detector systems. R. W. Poole made several suggestions that led to the final form of the jet polisher. J. T. Houston also made valuable suggestions based on his use of the equipment and developed a polyethylene jet polisher for use with hydrofluoric-acid containing solutions.

APPENDIX A

ELECTROLYTES AND POLISHING CONDITIONS

APPENDIX A

Electrolytes and Polishing Conditions

Material	Solution, Parts		Current (ma/mm^2)		Direct Current (v)		Temperature (°C)		Removal (mils/min)
	Jet[a]	Cell	Jet[b]	Cell[c]	Jet	Cell	Jet	Cell	Jet
Stainless Steel	60 H_3PO_4 40 H_2SO_4		48	10	160	2.5	100	60	3
Nickel	86 H_3PO_4– 10 gr CrO_3– 5 H_2SO_4		31	5	100		85	80	1
Uranium Nitride	4 H_2O– 1 H_3PO_4	4 C_2H_5OH– 1 H_3PO_4	18	15	125	50	RT	RT	4
Hastelloy N	60 H_3PO_4– 40 H_2SO_4	37 H_3PO_4– 56 glycerol– 7 H_2O	79	10	250	11	100	80	5
Tungsten and W-Re Alloys	250 H_2O– 32 gr Na_3PO_4		16	2	400	12	RT	RT	1
UO_2	53 H_2SO_4– 2.5 gr CrO_3–10 H_2O		2	0.2	150	9	RT	40	7
Aluminum	4 $HClO_4$– 6 H_2O– 35 C_2H_5OH– 5 butyl cellosolve	5 $HClO_4$– 95 C_2H_5OH	50	4	325	18	RT	10	4

[a]Flow rate: 50 ml/min at 2 psi.

[b]Area of dimple: 3.14 mm^2.

[c]Area of 1/8-in-diam disk: 20 mm^2.

APPENDIX B

PARTS LIST

APPENDIX B

PARTS LIST (See Fig. 13)

R1, R2, R3	Resistor, 22 kilohm, 0.5 w, 5%, carbon composition
R4	Resistor, 18 kilohm, 0.5 w, 5%, carbon composition
R5, R7	Resistor, 15 kilohm, 0.5 w, 1%, deposited carbon
R6	Resistor, 820 ohm, 0.5 w, 1%, deposited carbon
R8	Resistor, 6.2 kilohm, 0.5 w, 1%, deposited carbon
R9	Resistor, 5.6 kilohm, 0.5 w, 1%, deposited carbon
R10	Resistor, 1.8 kilohm, 0.5 w, 1%, deposited carbon
R11	Resistor, 10 kilohm, 0.5 w, 5%, carbon composition
R12, R17	Resistor, 750 megohm, 5.0%, Victoreen "Hi Meg"
R13, R18	Resistor, 5000 megohm, 5.0%, Victoreen "Hi Meg"
R14	Resistor, 499 kilohm, 0.5 w, 1%, deposited carbon
R15	Resistor, 5 megohm, 0.5 w, 1%, deposited carbon
R16	Resistor, 50 megohm, 1.0%, Victoreen "Hi Meg"
R19	Resistor, 500 ohm, 0.5 w, 5%, carbon composition
R20	Potentiometer, 10 kilohm, 10-turn, Bourns 3500S-1
R21, R22	Resistor, 22 kilohm, 0.5 w, 5%, carbon composition
R23, R25	Resistor, 33 kilohm, 0.5 w, 5%, carbon composition
R24	Resistor, 2.2 kilohm, 0.5 w, 5%, carbon composition
R26	Resistor, 5.1 kilohm, 3 w, 5%, Ohmite 2801 Brown Devil
C1	Capacitor, 500 μf, 50 WVDC, electrolytic
C2	Capacitor, 500 μf, 25 WVDC, electrolytic
C3	Capacitor, 0.1 μf, 200 WVDC, Mylar
Q1, Q2, Q3	Transistor, photosensitive, field effect, Siliconix P-102
Q4	Transistor, unijunction, 2N1671B
D1, D2, D3, D4	Rectifier, 100 PIV, 1 amp, IN4002
D5	Diode, zener, IN4742A
D6, D7	Diode, IN270
D8, D9	Rectifier, 400 PIV, 1 amp, IN4004
T1	Transformer, 115 v: 15 v, 100 ma

T2	Transformer, Sprague 11Z12
K1	Relay, 115 VAC, DPDT, General Electric CR2790E 100A2
K3	Relay, time delay, 1 min, Heineman BN4-522-XBX
I1, I2, I3, I4, I5	Lamp, neon, Dialco NE2J
	Meter relay, 10 μa dc, single high setpoint, optically coupled, Assembly Products, Inc. 503K Compack I
	Flexible fiber optic light guide, 72 in. long, approximately 6000 fibers, nominal 1/4-in. diam, with metal end fittings, LGM-5-72 (two each), and one image conduit clad rod, 1/4-in. diam × 12 in long, CR-2-12. American Optical Company
	Lens holders with lenses, one Fine Focus 17-183 and one Standard 17-210. Farmer Electric Products Company, Inc.
	Octal tube shield, T12-2027H, International Electron Research Corporation
	Triac, 200 PIV, 10 amp, General Electric SC40B
	Thermistor, 300 kilohm, VECO 53A9
	Lamp, high-intensity projection, Sylvania DAH

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