

ELECTROCHEMICAL POLISHING OF DIAMOND-TURNED  
NICKEL MIRRORS

by

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A Thesis Submitted to the Faculty of the  
~~COMMITTEE ON OPTICAL SCIENCES~~ (GRADUATE)

In Partial Fulfillment of the Requirements  
For the Degree of

MASTER OF SCIENCES

In the Graduate College

THE UNIVERSITY OF ARIZONA

1 9 8 1



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## ABSTRACT

Selective electrochemical polishing of metal to produce high quality mirrors was investigated. This thesis describes an attempt to use electropolishing to remove the microripple intrinsic on diamond-turned metal mirrors. Several diamond-turned nickel mirrors were selectively electropolished under various conditions. The results showed no apparent change in the turning depth or profile. This technique does not appear to be a viable method for diamond-turned microripple reduction.

## CHAPTER 1

### INTRODUCTION

Electrochemical polishing of diamond-turned nickel mirrors is an attempt to significantly reduce the microripple of the diamond-turned surface and to control the figure of the surface. This process, which dates back to Faraday, has an inherent characteristic of reducing the surface roughness of metal. Also, due to the dissolution process, it does not introduce stress into the surface. Moreover, it reduces the stress caused by the turning process.

Incorporation of commercial techniques of electrochemical milling and electro-polishing will give the high degree of contour control of milling and the smoothing and brightening of polishing that will supply the fundamental requirements needed for precision mirror fabrication. The problems are in the mating of the two processes. Electrochemical milling does not normally lend itself to formation of the hydroxide layer and chemical film needed to smooth and brighten the metal surface.

It is the mating of these two processes that is dealt with primarily in this thesis to give some substantive answer to the principal question of whether electrochemical polishing can have substantial effect on the microripple with minimal material removal. Secondary questions on suitable surface quality and figure control need to be answered as well.

It is not the intent of this thesis to develop a complete process or technique. The intent is to establish the feasibility of this process for reducing the microripple of diamond-turned mirrors.

## CHAPTER 2

### ELECTROCHEMICAL MATERIAL REMOVAL PROCESS

#### Fundamental Concepts

Electrochemical metal removal has been considered the opposite of electroplating. There are, naturally, some obvious similarities. Both processes require an anode and cathode and an appropriate electrolyte. The finished product is the cathode in the plating operation and the anode in the dissolution operation. Three distinct phenomena occur in the circuit facilitating current flow for both reactions. The metal incorporates conduction, the motion of ions in the electrolyte solution and a complex chemical reaction bridge at the interface.

Similarities end at the interface because the conditions that exist here determine if the metal anode will be polished, etched, partially passivated, or even completely passivated. A continuous dissolution of metal such that irregularities are smoothed and brightened is desired. This describes the electrochemical polishing process. The exact mechanism of the electropolishing process has not been established for specific cases, but a general explanation has been developed. Two distinct processes are believed responsible for the smoothing and brightening effects. The formation of a thin film on the anode is usually required for brightening, and a formation on the anode of a thick layer usually viscous, of reaction products, for smoothing. This latter layer has a higher electrical resistance and tends to concentrate the current on the high areas where the layer is thinner.

The conditions under which proper polishing will occur are rather limited. Characteristic regions of surface quality can be found by plotting potential versus current density. The orthophosphate system as described by Tegert (1956) illustrated, by similarity, a characteristic curve.

The characteristic curve (Fig. 2.1) is for a potentiometric circuit on a copper anode in a 900 g/l orthophosphoric acid solution. At voltages lower than that at point A on the curve, etching occurs on the surface of the anode. Between point A to point B is a region where the surface is partially passivated and instability between voltage and current density is present. The polishing plateau is the region from B to C with C being the optimum. In the region of higher voltages and currents, gas evolution, which causes pitting and irregular finish, occurs. The regions shown by the orthophosphate system exist in many other systems although not usually as distinct or broad.

There are several fundamental conditions that have a direct effect on the best polishing region. The four primary variables are temperature, concentration, agitation, and viscosity of the electrolyte. Temperature does not change the length of the polishing plateau but does move it relative to the current. This is expected as the mobility of the molecules is inversely proportional to the resistance of the electrolyte. Increase in temperature can also severely affect the hydroxide layer by reducing the viscosity and subsequently increasing its dissolution.

Tegert (1956, p. 6) found that as the concentration increased the current density varied for the orthophosphate electrolyte. There

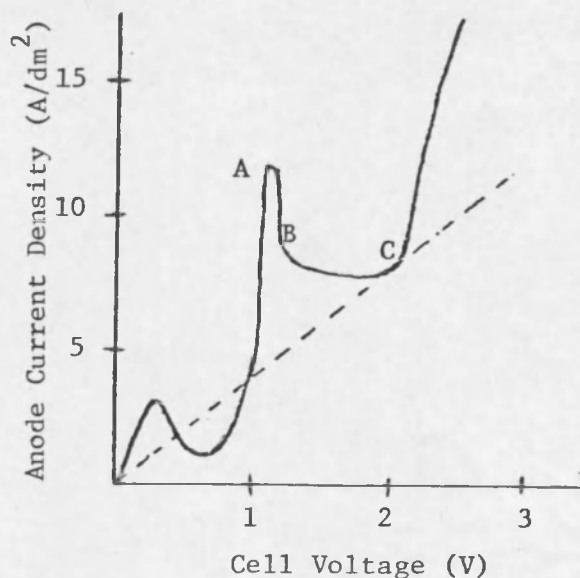


Fig. 2.1. Orthophosphate characteristic curve.

From Tegert (1956).

usually exists a concentration region for best surface results. This conforms to an optimum ion concentration in the electrolyte. During agitation or stirring of the electrolyte the current density usually significantly increases. The agitation increases the movement of the reaction products away from the anode by bringing in fresh electrolyte. In a related manner the viscosity of the electrolyte also affects the mobility of the reaction products and their dissolution from the hydroxide layer.

### Electrodics

By definition all electrochemical processes involve both electrical and chemical reactions. These processes can be usually considered

as "one" reaction at the electrode-solution interface for simplistic interpretation. The reactions are considered heterogeneous. The heterogeneous reactions of the electroactive species involve many distinct processes such as transport incorporating convection, electrical migration, and diffusion. The formation of crystals, partial or total dehydration of ions accompanied by acid-base reactions, further complicates the simplistic one-reaction concept.

Electrodics is the study of reactions occurring at the electrodes. These reactions are normally treated from the thermodynamic point of view. It must be understood that thermodynamics will not give the characteristics of a system to determine whether it will behave reversibly or not. It also does not fully explain observations obtained when appreciable current flows.

Experimentally, however, most reactions are found to follow the Tafel equation (Bauer, 1972, p. 15):

$$\eta = a \pm b \log i$$

where  $\eta$  is the overvoltage and  $i$  the current density. This equation does not hold up at low overvoltages and does not give a clear picture of what happens at the interface. At lower overvoltages the double-layer effect dominates. A dipole effect occurs between the charge on the electrode and the molecules in the electrolyte. This layer naturally affects the direction of the chemical reactions by presenting a particular molecular orientation to the electrode. The layer can be several molecules thick thus affecting the diffusion process, which in

most cases is the limiting dynamic factor in the entire process sequence.

Further complicating the electrochemical phenomena are the effects on surface tension called electrocapillarity and the double-layer influence on the faradaic process. Electrodeics involves the characterization of solid electrode surfaces relating to composition, crystallographic makeup, homogeneity, and time changes due to adsorption or corrosion and the basic geometry in the molecular dimension and macroscopically due to local polarization effects. All of which can be significant in their effects under various circumstances.

#### Material Requirements

The material requirements are simple. The anode, which is the mirror, should be fine-grained and usually of only one species. Preferential polishing, which tends to occur in materials of more than one species, causes a very rough surface enhancing intergranular attack. The mirror material should be low in stress and inclusions for the same reason.

The cathode, however, shapes the current distribution in electrochemical milling and is required to facilitate the exchange of the electrolyte. The material must be machinable and must not react with the electrolyte in such a way as to form precipitates to contaminate the mirror surface. Conduction between the cathode and the electrolyte must be high. A typical choice of material is stainless steel.

### Cleaning Requirements

For electropolishing, proper cleaning is usually ignored, but this can cause serious problems. There are some common methods used to remove foreign matter from the surfaces of both the anode and cathode. A strong cleaner can be used if the materials can tolerate the high alkalinity. Aluminum, for example, can be pitted by a strong cleaner. A soft or deionized water is best to prevent nonsoluble soap residue from forming. High temperatures are usually best, but not so high that cleaner deposits react with the surfaces. The cleaner solution should be agitated to facilitate dispersion of contaminants and to reduce redeposition. The faster one cleans the surface the better, but a slightly slower process lets the oils help lift off the solids from the surface. If the surface is clean, deionized water will run off or "break free" from the surface. It is common practice to assume that electropolishing or anodic electrocleaning is sufficient; however, only the oils are outgassed leaving the particulate matter.

### Potential Considerations

To be able to figure a mirror, a nonuniform distribution of current must be formed on the mirror. This is in direct contrast with most work done in the theory of commercial polishing. Because the amount of material removed is so critical, there must be a rapid development of the current distribution and conditions for polishing. There must also be a rapid shut-off of current flow to prevent low-current etching, which can also be a problem in outlying regions of the distribution.

The primary current distribution is the fundamental control of the deplating process (Graham, 1978). This distribution of current is based on the geometry of the situation and can be assumed to be linearly related to the electric field. Much work on the theory of potential has been done by Kasper (1940).

The theory of potential relates to the study of the distribution of the potential energy between geometrical configurations at specific potentials. The electrokinetic case is not as simple, however, as the electrostatic.

The current distribution can be thought of as combinations of basic geometrical situations to formulate an intuitive distribution of current. The simplest is two infinite planes where  $V_a - V_b = IR$  and for a linear electrolyte,

$$j = -\sigma \partial V / \partial X = I/A$$

where  $\sigma$  is the conductivity,  $j$  the current density and  $A$  the area of the cross section. Integrating one finds the resistance  $R = L/\sigma A$  where  $L$  is the separation between planes. This is trivial, however; tilt one of the planes and the problem is no longer even elementary, although solved for the current distribution by Busse (1932), Mantzell (1936), and Hall (1939).

The effect of  $F/\#$  on the figuring problems can be related to the tilt of a plane. The distribution will become nonsymmetrical in a nonelementary sense and the predictability extremely difficult. The practicality of the actual need for accurately calculating the primary

current density is offset by the polarization of the electrolyte. This polarization has the effect of reducing the current in regions of high current density, thus leveling the distribution. An insulating wall such as the surface layer of the electrolyte will also have a similar effect on the current distribution.

### Industrial Electrochemical Techniques for Material Removal

Electrochemical techniques for material removal used in industry are generally categorized into four groups, electrochemical machining (ECM), electrolytic grinding (ECG), electrochemical milling and electropolishing.

Electrochemical machining consists of metal removal by passing a direct current between work piece and precisely shaped cathode in close proximity. This method requires a swiftly flowing electrolyte under high pressure. This facilitates the removal of particles from the anode and improves the surface quality over what would normally occur. A simple salt solution is the electrolyte most widely used for this technique with other parameters such as electrode separation of 0.002 to 0.033 in. and electrolyte pressures of 10 to 350 psi being typical (Gurkis, 1965, p. 12). With voltages of only 3 to 25 volts, current densities range from 100 to 1500 A/in<sup>2</sup>, which will yield very high removal rates of about 0.15 in<sup>3</sup> in one minute for 1500 A/in<sup>2</sup> (Kleiner, 1963).

Electrolytic grinding is a mechanically assisted electrochemical machining technique that incorporates the use of a grinding wheel

## CHAPTER 3

### EQUIPMENT AND MATERIALS

#### Cathode Design and Manufacture

Selective removal with surface contour control dictates that a technique similar to electrochemical milling must be used. It must also produce surfaces of a quality equal to the best that electropolishing is capable. Because the shape of the mirror cannot be one of the parameters for control, the cathode must take total consideration.

The local current distribution is best established by using dielectric insulators such as air and easily machined acrylics. A specifically shaped puddle of electrolyte will force the current distribution within the confines of the puddle. This also tends to make the distribution of current uniform within this area. Time can therefore be used to create a nonuniform material removal distribution.

The puddle of electrolyte is small in volume and normal convection will not keep fresh electrolyte exchanging with the hydroxide layer on the mirror surface. A flow of electrolyte must occur through the cathode to exchange the old electrolyte and reaction products with fresh electrolyte. This must be done without destroying the hydroxide layer or creating localized disruption. The cathode-anode area ratio should also be greater than two to one based on commercial experience (Graham, 1978, p. 500). Filtration, preferably between the cathode

and anode, is needed to remove particles that may break off the cathode since plating will not occur. A conductive cathode material that machines well and will not react adversely with the electrolyte is necessary.

Plexiglas (acrylic) and stainless steel were chosen as materials for the cathode for their lack of chemical reaction with the electrolyte, and cotton was chosen for the filter and for the shaped wad for electrolyte puddle control to prevent destruction of the hydroxide layer during dynamic polishing. The electrolyte flows through the stainless steel cathode, which has one end filled with stainless steel lathe shavings similar to steel wool. These shavings give a large cathode-to-anode area ratio. A cotton wad cut to the desired shape (diamond) of the puddle is placed at the end of the stainless steel cathode and held in place by a Plexiglas shield. The used electrolyte is pulled back through the cotton wad around the outside of the Plexiglas-shielded cathode. The stainless steel cathode and the nickel anode were separated by approximately 0.25 inch. The cathode with the cotton wad (Figs. 3.1 and 3.2) resembles very much the brush electrode used in localized plating.

#### Electrolyte

Both electrochemical milling and electrochemical polishing electrolytes are usually acid types, typically a sulfuric acid base; however, not all electrolyte solutions need be acidic. A common industrial nickel polishing solution is a nickel sulfate-based solution. This electrolyte, which is much safer, was used.

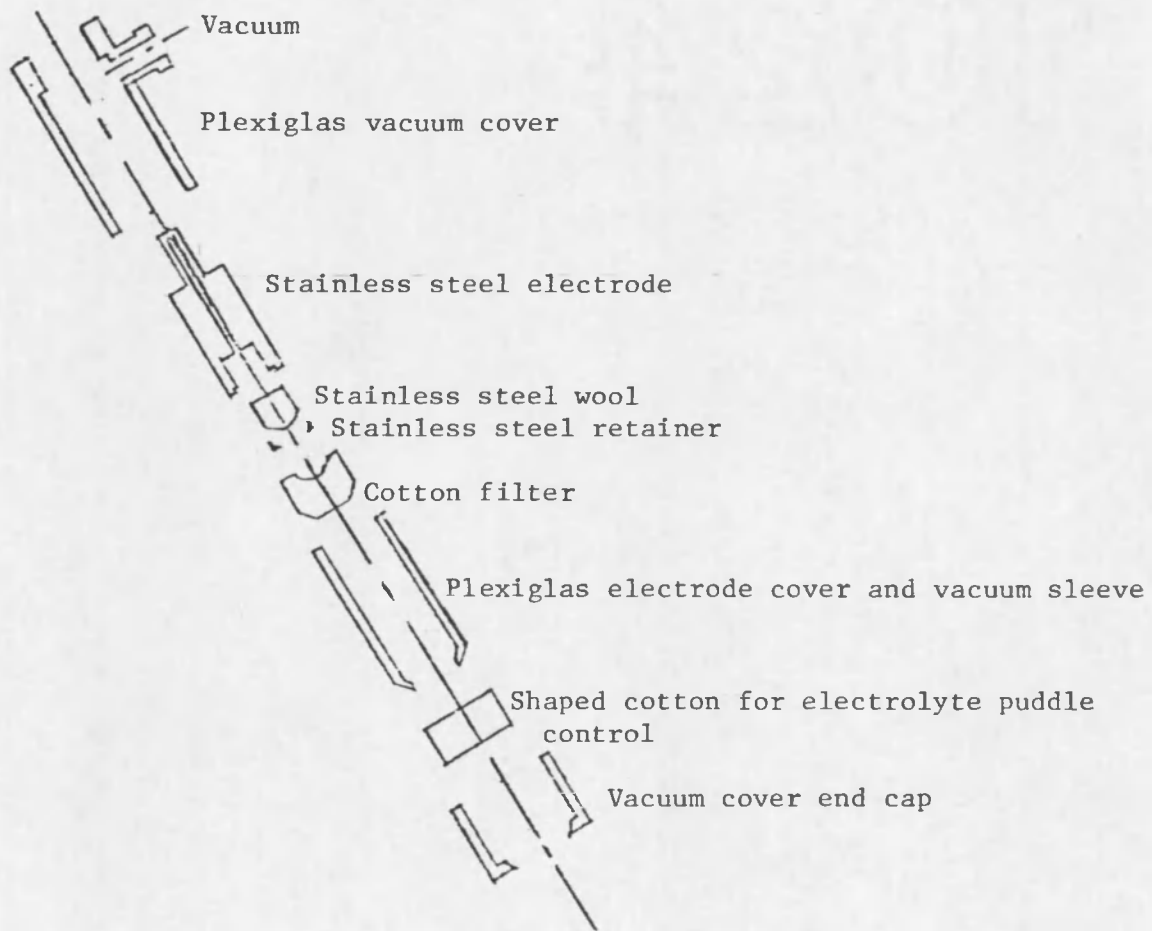


Fig. 3.1. Cathode components.

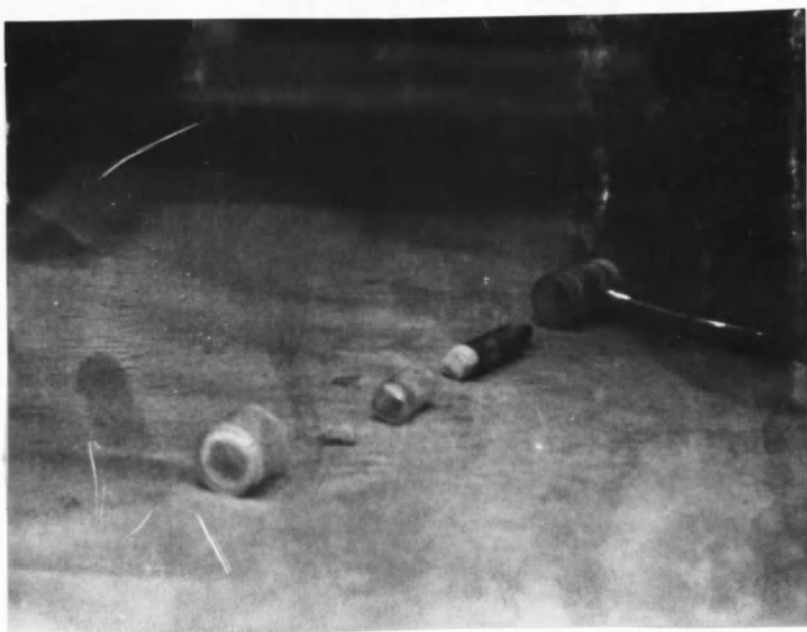


Fig. 3.2. The electrode used in the experiment dismantled.

The electrolyte, which is dissolved in deionized water, consists of a saturated solution of (Graham, 1978, p. 504):

Nickel sulfate	240 g/liter
Ammonium sulfate	45 g/liter
Potassium chloride	35 g/liter

The solution is mixed and then heated to about 90°C until dissolved, then cooled to room temperature. The excess reagents crystallize out at the bottom of the container in blue-green crystals. When more electrolyte is needed it is necessary to add deionized water to some of these crystals making sure that the solution is saturated.

#### Electrical System

The electrical requirement for effective polishing is sufficient current density. An area of approximately 0.25 square inch under the puddle and the need for current densities of up to 1000 A/ft<sup>2</sup> require currents of as much as 1.75 A.

A simple series circuit using a Lambda LE 104M power supply and a 0.825 Ω resistor for current measurements were used. A two-channel Tektronix oscilloscope was used for the voltmeter (Fig. 3.3). The expected voltage ranges were between 0-15 volts for the cell and 0-0.2 volts across the resistor for current readings.

#### Pump, Temperature, and Vacuum Systems

The electrolyte was temperature controlled and recirculated to keep the active puddle refreshed and the excess reaction products diluted (Fig. 3.4). A HAAKE FE constant temperature bath with circulation

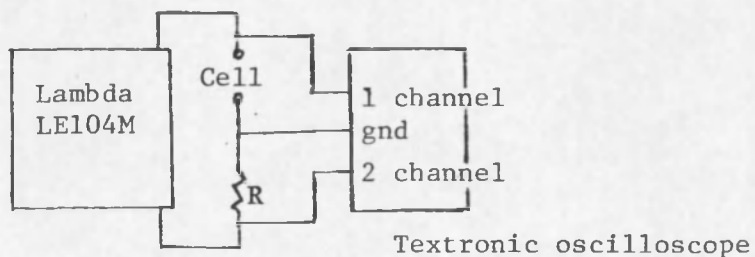


Fig. 3.3. Electrical control and monitoring layout for electrolytic cell.

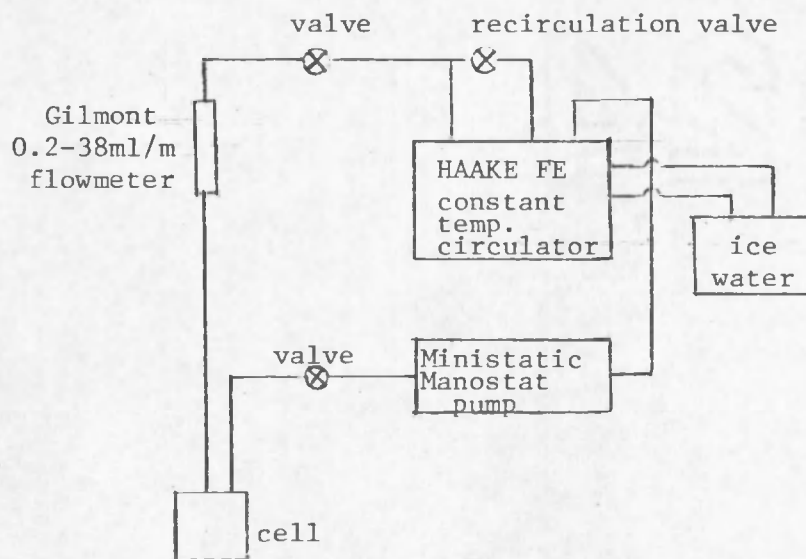


Fig. 3.4. Electrolyte flow system.

pump was used. It has a two-liter capacity and is made of materials that will not react with the electrolyte. It is rated by the manufacturer as being able to control temperature in the bath to  $\pm 0.01^\circ\text{C}$ . To assist the bath a bucket of ice water supplied cold water to the cooling coils of the device and the heater coils brought the electrolyte up to the desired temperature ( $15^\circ\text{--}20^\circ\text{C}$ ).

The electrolyte to the cell flowed through a Gilmont flowmeter with optimum flow at about 50% capacity or 20 ml/min. The electrolyte was pulled from the cell by a Ministatic manostat pump. The rate was adjusted, to keep a stable puddle between the electrodes. This required frequent manipulation.

#### Cathode X-Y Drive

The X-Y drive was put together to move the electrode and the puddle across the mirror (Fig. 3.5). With manual adjustment in the Z or vertical direction, it was powered to move in the horizontal plane. The velocity and direction were controlled by a "joy" stick with the velocity proportional to the angle of the stick (Fig. 3.6). It works smoothly in either the X or Y direction but is erratic in any but the orthogonal directions and hence could not be used to figure.

The drive has a constant speed override in the Y, or forward and back, directions so that dynamic removal rates can be measured. The sensitivity of the velocity to angle of the "joy" stick resulted in all dynamic measurements being done in the constant speed override mode. In this mode the velocity is 1.71 inches per minute forward and

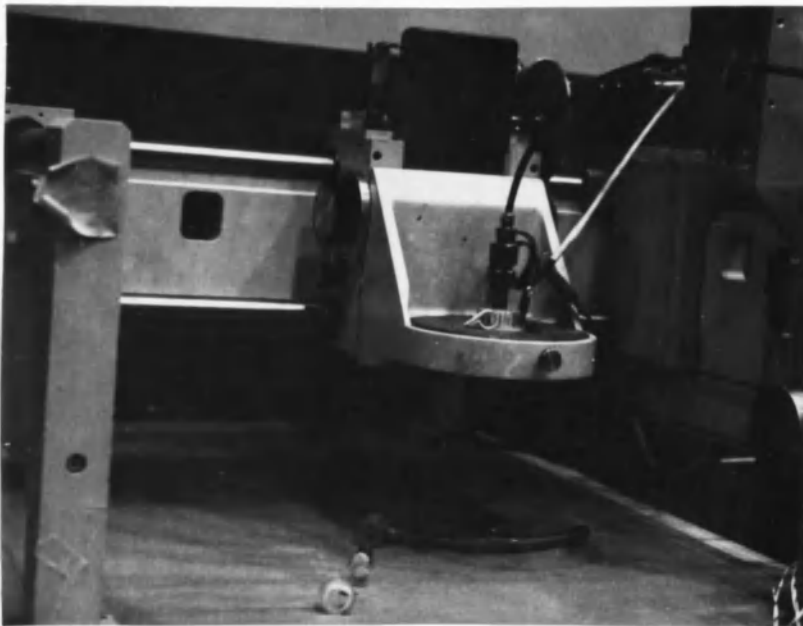


Fig. 3.5. X-Y drive carriage with electrode.



Fig. 3.6. X-Y drive "joy" stick control.

1.62 inches per minute backward. This allows a spatial distribution of polishing time by using the diamond-shaped puddle and varying from 0 to 17 seconds.

### Substrate Preparation

The experiment used two different substrate types, a mechanically polished electroless nickel plate on aluminum and a nickel-plated diamond-turned flat mirror. The classically polished substrates are for set up and the finding of the proper conditions for electropolishing.

The classically polished substrates were made of extruded aluminum cut into 2 inch diameter, 0.5 inch-thick slabs with a 6-32 tapped hole on the edge for electrical contact. These substrates were fine ground with 25  $\mu$  aluminum oxide prior to electroless nickel plating. The plating was done by Chem-Research Company of Phoenix, Arizona. The substrates were polished on 73 Gugalz pitch, first with Lindy C (1.0 $\mu$ ) then finished with Lindy A (0.3 $\mu$ ), a fine grit aluminum oxide. The substrates were rinsed with deionized water and reagent grade acetone while being lightly wiped with a saturated cotton ball. They were stored in plastic dust containers.

The diamond-turned substrates do not have a clear history. They were turned by Y-12 in Oak Ridge, and one was cycled through high temperatures and slowly brought back to room temperature.

Four substrates were available for this experiment of which only the backs of three could be used. The backs were also diamond-turned but not with the care of the good side. The turnings on the

backs were broader and deeper than on the fine-turned side. The fourth surface, which was fine tuned, was used for final evaluation.

These surfaces were cleaned with deionized water and cotton ball swabs and then finished with reagent-grade acetone.

## CHAPTER 4

### PROCEDURES

The measurement of the relationship of material removal to voltage and current requires that the temperature and concentration of the electrolyte and the electrode separation remain constant throughout the experiment. The electrolyte is a saturated solution; therefore, an excess of the electrolyte crystals was kept in the temperature bath and agitation kept the solution close to saturation. The temperature bath was set at about 15°C to compensate for the heating effect of the room that was almost always 24°C. The temperature of the electrolyte at the electrodes was about 19.5 C after about 10 minutes to bring both electrodes to this temperature.

The voltage was set by substituting an 0.825  $\Omega$  resistor for the electrolytic cell. A Tektronix oscilloscope was used to measure both the voltage and the current. A second 0.825  $\Omega$  resistor was placed in series with the cell and the voltage drop was assumed to be a measure of the current using Ohm's law.

The separation between the electrodes was about 0.25 inch, the substrate was replaced after removal, so that the distance between the two electrodes did not vary. A change here would make what could be a linear relationship a nonlinear one, or at least erratic.

The conventionally polished electroless nickel substrate was cleaned and air-blown dry and an interferogram was taken of the surface.

This substrate was then positioned under the cathode and good electrical contact made taking the resistor substitute out of the circuit. The electrolyte flow was then started and the vacuum adjusted to hold a stable puddle between the electrodes. After temperature equilibrium was established it was verified by placing a thermometer in the small puddle.

The time was set with a Gra-Lab timer with one rotation being 15 seconds divided in one-second intervals. Cycles of 5, 15, 30, and 60 seconds were arbitrarily chosen. After each cycle the substrate was removed and cleaned and an interferogram taken. Consecutive runs were taken until it started to become difficult to read the interferogram due to the high slope at the edge of the zone. The fringe change between runs corresponds to the material removed during the run. This procedure required precise positioning of the substrate under the cathode.

The interferograms of the substrates were only 16.5 mm in diameter. This left the zones of removal very small in size, about 7 mm across. A mask with a 7-mm-diameter hole was used over the interferogram and a photo was taken of this masked region using a 35 mm single lens reflex camera, a 50 mm lens and a bellows attachment on a tripod. These photos were enlarged to bring the 7 mm area to 63 mm in diameter, a much easier to read interferogram. All relevant data was recorded and the photos sequenced and interpreted. Three substrates were used.

The microripple of the diamond-turned substrates had to be evaluated under the microscope and would not follow the exact same procedures as the removal rates. By moving the electrode and a diamond-shaped puddle a general V-shaped trough could be made allowing, hopefully, a gradual improvement in the microripple. This procedure would also answer some of the questions about the motion of the cathode and its correlation to a stationary one.

Voltages of 4 and 6 were used and multiple passes of the cathode were made to increase the material removal. Visual observations with a 3X microscope were made after each set of multiple runs of about six each until hazing precluded further runs. A single-pass sequence was also done with the voltages varying in one-volt increments from 1 to 7 for static dynamic correlation. Interferograms were taken before and after each voltage change for total removal measurements on the Davidson Fizeau interferometer. Microphotographs with the 10X and 40X objectives were taken as representative of the surfaces and specific zones of removal both before and after electropolishing. The relative location of each record was mapped to a fiducial point. Microinterferograms were taken with some correlation to the microphotographs. There was little variation from one photo to another on the same mirror but obvious differences from mirror to mirror. The photos selected for presentation are representative of the particular mirror being evaluated.

## CHAPTER 5

### PRESENTATION AND ANALYSIS

#### Surface Characteristics

During the process of electropolishing the two different types of mirrors demonstrated distinct differences. The mechanically polished electroless nickel aluminum substrate mirrors showed characteristics of microfracture stress relief that seemed related to a Beilby-type layer surface effect. The diamond-turned nickel-plated mirrors, with the exception of one, did not show this characteristic, but showed a hazelike surface that appeared to be a deposit of granules, possibly of a nickel compound of some sort. Figures 5.1 through 5.4 are representative of the conventionally polished substrates with various effects caused by electropolishing. The figures A-1, A-3, A-8, A-11, A-16, A-20, and A-22 show the effects of electropolishing on the diamond-turned surfaces. Diamond-turned substrate 004 (Fig. A-1) shows some preferential etch that follows the stresslike patterns of conventionally polished substrates. This substrate went through a heating cycle of 400°F for one hour then a six-hour cool down at The University of Arizona. It is highly likely that stress was put into the mirror, thus making it different from the other diamond-turned mirrors.

A microfracture hazing developed wherever the hydroxide layer failed to materialize properly because of current density or temperature. Under the microscope the fracture pattern was characteristic of

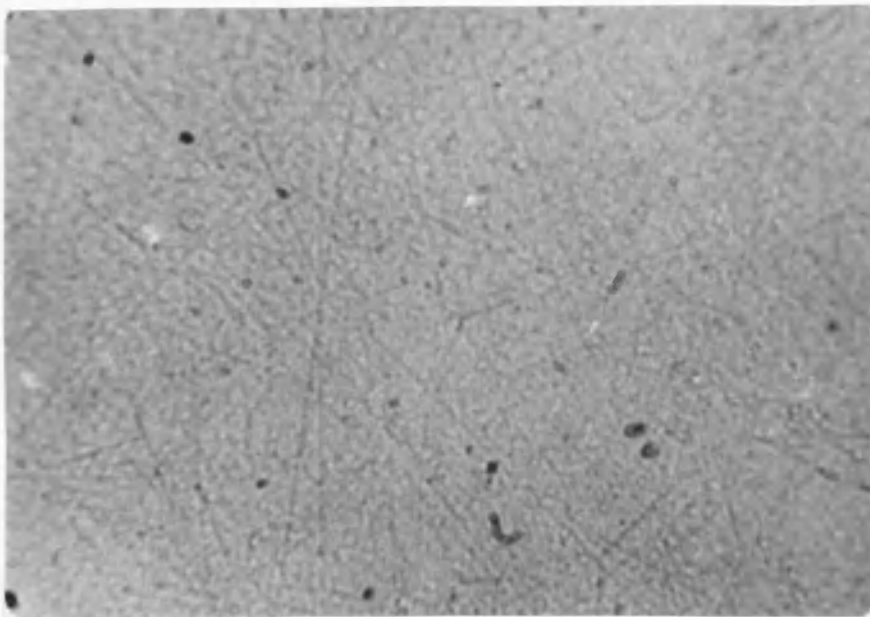


Fig. 5.1. Conventionally polished electroless nickel plate 10 X objective.

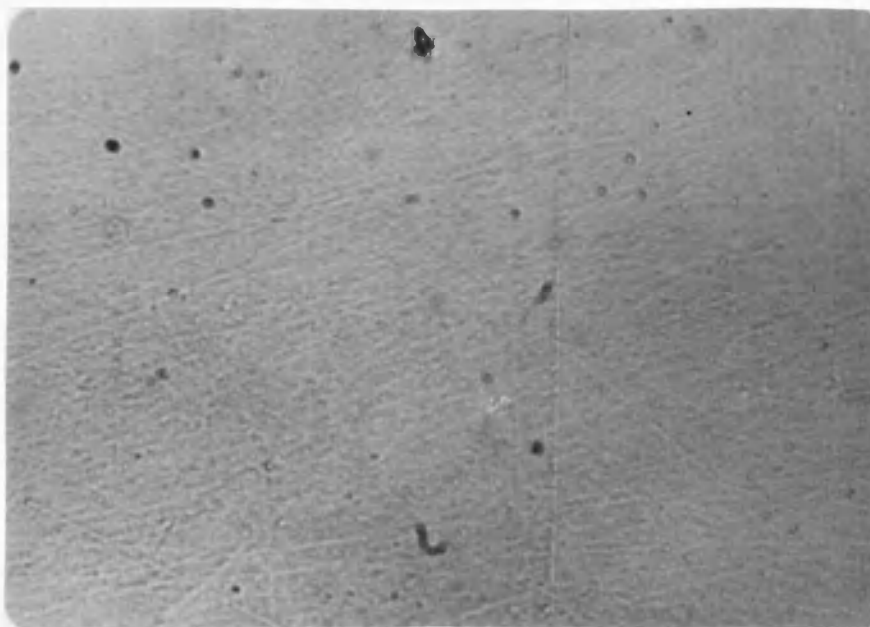


Fig. 5.2. Conventionally polished substrate, electropolished with light haze, 10 X objective.

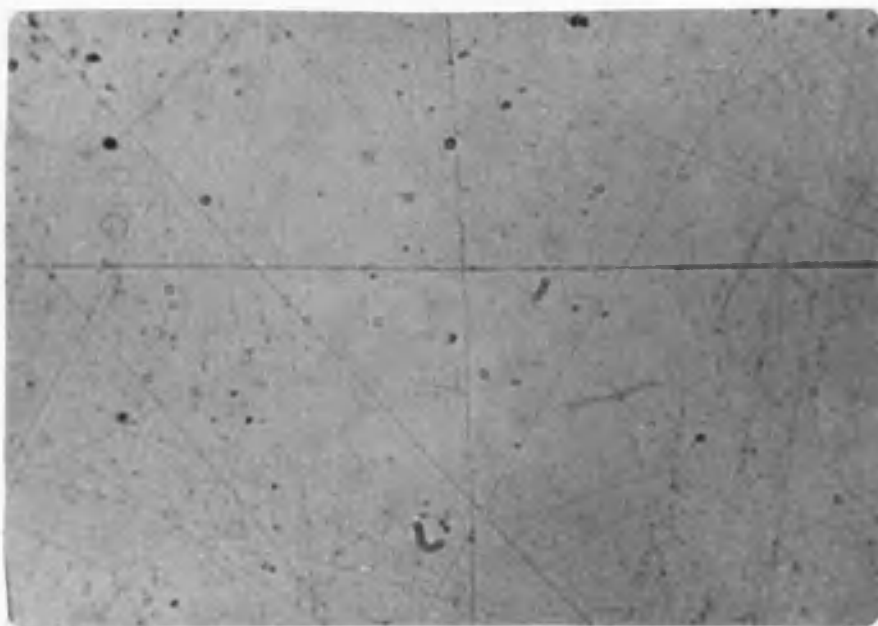


Fig. 5.3. Conventionally polished substrate, electropolished, 4.1  $\mu$  removal, very bright appearance, 10 X objective.

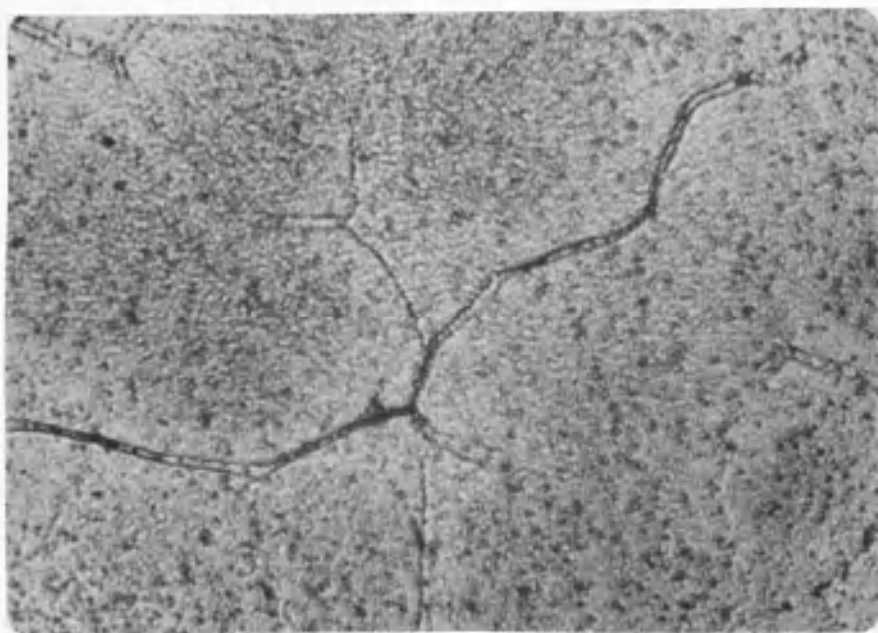


Fig. 5.4. Conventionally polished substrate, electrochemically polished, very hazy, heavy microfracture, 10 X objective.

that on the bottom of a dried-up mud puddle, which undergoes the same contractive stress normally found in electroless nickel plate. Also associated with the fractures is a Beilby-type layer, which was observed by first electropolishing a region then overlapping this "polished" area and an untouched region with a cycle at a higher temperature so that etching occurred in the previously untouched area. The previously polished area did not show any signs of microfractures. This implies a surface layer of contamination possibly caused by the conventional polishing process.

#### Material Removal

Material removal followed a general linear relationship (with voltage and time) because the electropolishing action is primarily dependent on current density and is supported by the linear relationship maintained between current and voltage (Fig. 5.5). The data presented in Table 5.1 represents stationary localized zones on the conventionally polished electroless nickel plate. Table 5.2 presents the maximum removal, representing a 17-second electrochemical polishing time, on a diamond-turned mirror by use of a moving cathode. With the limited number of substrates available, the data are quantitatively inconclusive but qualitatively suggestive.

As would be expected, the removal rate is linear relative to voltage, as shown in Fig. 5.6. There is a reasonable correlation for a linear removal as a function of time for a specific voltage, as illustrated in Fig. 5.7. With the exception of the value for the 6-volt

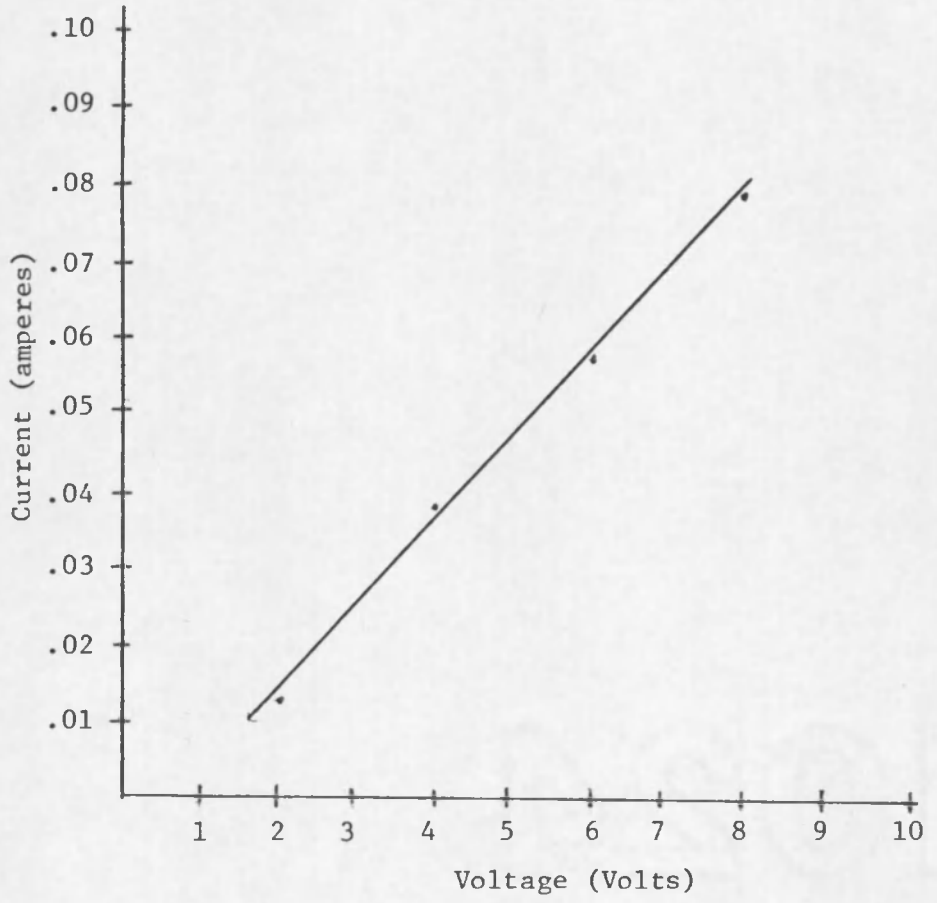


Fig. 5.5. Current versus voltage.

Table 5.1. Removal Rates.

<u>Voltage</u>	<u>Time</u> <sup>1</sup>	<u>Current</u> <sup>2</sup>	<u>Removal</u> <sup>3</sup>	<u>Rate</u> <sup>4</sup>	<u>Rate Avg.</u> <sup>4</sup>
2	4	.0125	.036	.009	
2	15	.0125	.095	.006	
2	30	.0125	.127	.004	
2	60	.0125	.091	.001	.005
4	5	.0375	.132	.026	
4	15	.0375	.41	.027	
4	30	.0375	1.11	.037	
4	60	.0375	1.89	.031	.030
5.8	5	.050	.23	.046	
6	15	.562	.89	.059	
6	30	.562	1.49	.050	
6	60	.500	2.21	.037	.048
8.2	5	.078	.41	.082	
8	15	.078	.975	.065	
8	30	.078	1.52	.051	
8	60	.075	3.44	.057	.064

1. Seconds

2. Amperes

3. Material removed in fringes, wavelength  $\approx 0.55 \mu$

4. Rate is in fringes per second.

Table 5.2. Dynamic Removal.

<u>Volts</u>	<u>Fringe pri.</u>	<u>Fringe aft.</u>	<u>Removal<sup>1</sup></u>
2	0	-.1	.1
3	+.077	-.15	.23
4	+.077	-.30	.38
5	+.077	-.50	.58
6	0	-.73	.73
7	+.077	-.73	.81

1. Removal in fringes, wvl .55  $\mu$

Moving electrode, velocity 1.7 inches/minute.  
 The maximum polishing duration at any one point  
 is 17 seconds, a diamond-turned surface was used.

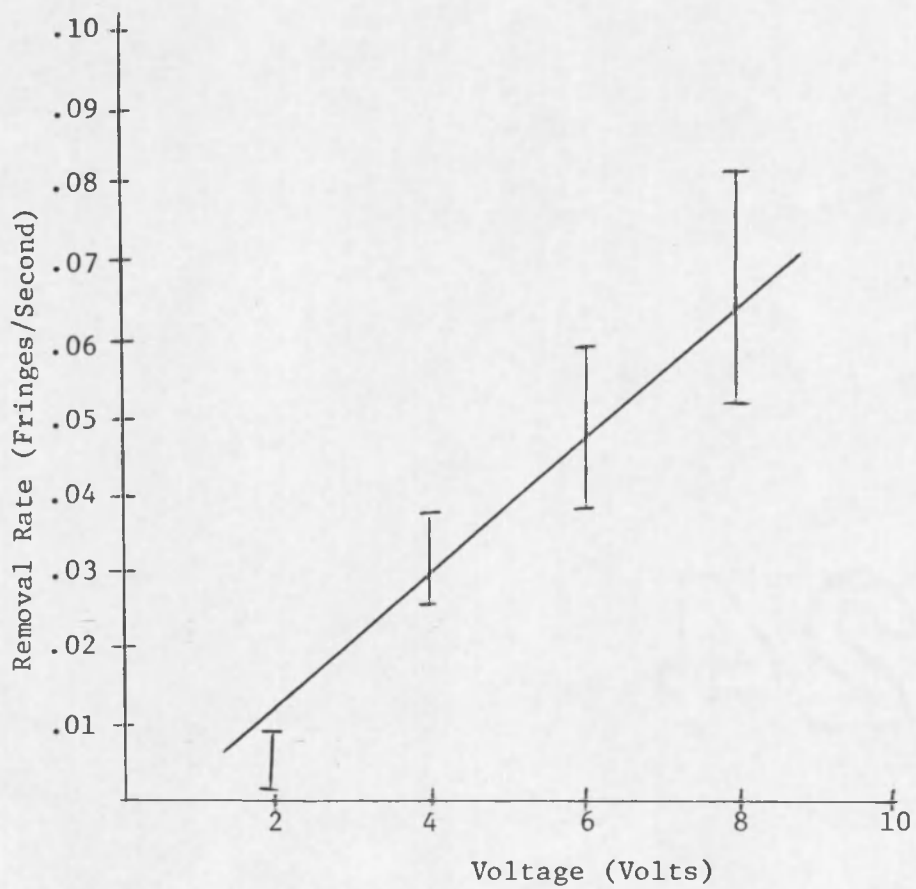


Fig. 5.6. Removal rate versus voltage.

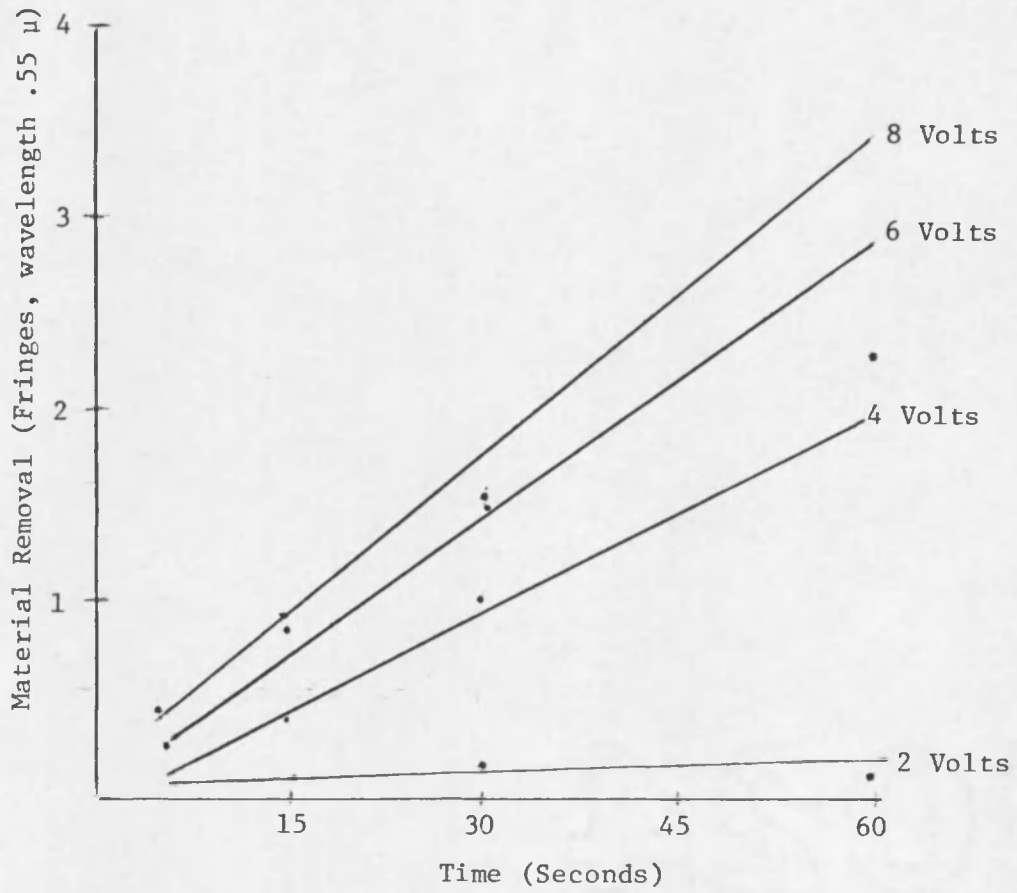


Fig. 5.7. Material removal versus time.

static run, which appears erroneous, the correlation between the 17-second dynamic and 15-second static runs is reasonably strong (Fig. 5.8).

The spatial distribution for the dynamic run is what would be intuitively expected, but it does not follow the linear geometry that the square mask implies because the slight distortion of the puddle gives a rounded appearance, as shown by the fringe patterns in Fig. A-7. The edges of the distribution were smoother if the surrounding area has been previously electropolished, allowing a better wetting of the surface by the electrolyte.

#### Diamond-turned Substrates

Evaluation of the microripple of the diamond-turned mirrors involved the use of a microscope and a Leitz interference objective. The microinterferograms were taken with a 10X objective and can be correlated with the 10X scale, Fig. A-26. The smallest increment on the scale represents 0.02 mm (20  $\mu$ ).

Substrate 004 (Figs. A-1, A-2, A-3) had very fine turnings but during electropolishing experienced localized attack and preferential etching that appear to follow the stress crack pattern of the conventionally polished substrates. This pattern is found only on this sample of diamond-turned mirrors. The microinterferograms, Figs. A-5 and A-6), although difficult to evaluate, do not show any improvement even though 10 times the estimated depth of the turnings, 0.07  $\mu$ , was removed.

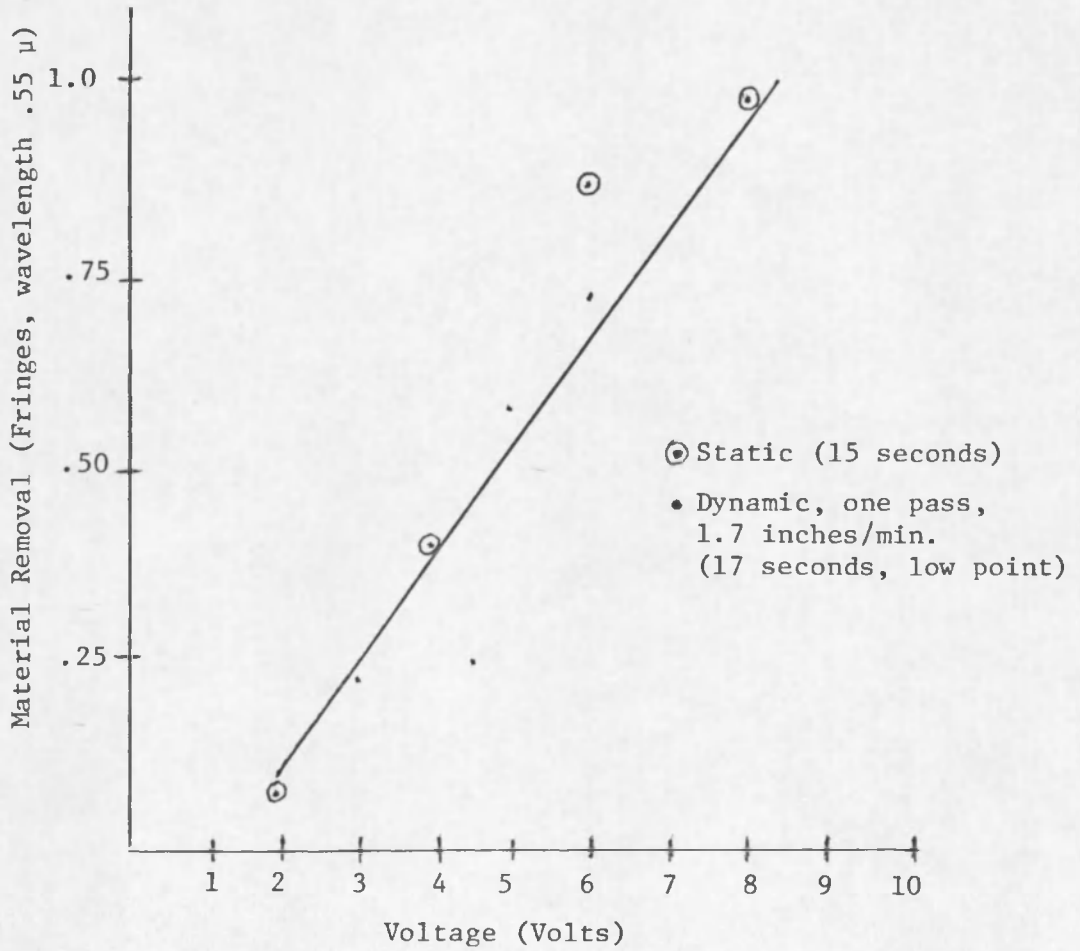


Fig. 5.8. Static and dynamic removal.

Substrate 018 (Figs. A-8 and A-12), had the coarsest turnings. During electropolishing the surface appeared to have undergone a grain attack or deposit that left the surface with a very hazy, granular appearance. The microinterferograms (Figs. A-13, A-15) shows a unique shape to the turnings with a peak to valley of  $0.28 \mu$ . There appears to be no change after electrochemical polishing in the shape or the magnitude of the turnings even though material up to 15 times the depth of the turnings was removed.

Substrate 022 (Figs. A-16 and A-17) indicates further hazing like an intergranular attack and deposit with localized spot attack. The microinterferograms (Figs. A-18 and A-19) shows a  $0.27 \mu$  peak to valley and again electrochemical polishing did not have any effect on the shape or depth of the turnings with up to 4.1 microns of material removed.

Substrate 025 (Figs. A-20-A-23), a fine-turned surface, suffered severely from a granular attack or deposit. The surface turned black and totally non-specular after only  $0.27 \mu$  of material had been removed. The heat treatment that this mirror had been given by Y-12 in Oak Ridge seems the likely cause of the effect. Examination with a 40X objective showed that the turnings were evidently completely obscured. This surface was lightly buffed with a soft foam and Lindy B, a  $0.06 \mu$  aluminum oxide, and after about 30 seconds of very light rubbing most of the granular cover was gone. The microinterferograms (Figs. A-24 and A-25), revealed no change in the approximate depth of  $0.07 \mu$ .

## CHAPTER 6

### CONCLUSIONS

Due primarily to the limited number of substrates, quantitative results are not possible but some qualitative results are.

The microinterferograms showed no observable effect in the depth of the turning grooves but more significantly there appeared to be no affect in the profile of the grooves, even the sharp peaks on the diamond-turned groove were not rounded or softened. The implication is that the electrochemical polishing has no effect on the microripple.

The answer to the question of figure control was qualitatively very positive. The tendency of the removal rates to be linear implies a strong probability of being able to figure a surface.

Evaluation of the effect of the electrochemical polishing on surface quality was inconclusive even though principally negative in this experiment. There were too many differences between the nature of the nickel plate from substrate to substrate to truly locate the best possible conditions for the best surface. Details of the surface character depend strongly on the structure of the hydroxide layer as evident in the localized surface distortions. The hazing characteristics of the substrates show that the results of the electropolishing even under apparently identical conditions vary significantly between the electroless nickel conventionally polished and the diamond-turned substrates. This problem

resulted in the surface quality being the limiting factor in the micro-ripple reduction analysis. The hazing limited the material removal to only at most 15 times the apparent depth of the turnings.

The technique simulated the normal commercial electropolishing since over 300 feed lines (grooves) were under the puddle during electropolishing. The local removal, possible because of the electrodes similarity to a brush electroplating electrode, allowed for easy optical measurement of material removed. The chemical limitations appeared to be associated with the plate and not the one electrolyte system employed. Environmental hazards of the alternatives restricted their use. If smoothing was to occur with reasonable material removal some effect should have been observed at least in the turning profile such as a rounding of the sharp peaks. This technique does not appear to be a viable method for diamond-turned microripple reduction.

It can be speculated that this technique could lend itself to electroplating and yield a high degree of figure control. The limited weight it places on the mirror should allow the figuring of very lightweight structured mirrors, and is an area for further research.

APPENDIX A

SUMMARY OF DATA FOR PHOTOGRAPHS

<u>SUBSTRATE</u>	<u>PHOTO</u>	<u>FIGURE</u>	<u>OBJECTIVE</u>	<u>REMOVAL</u>
004	4-8	A-1	10 X	0.7
004	4-7	A-2	10 X	0
004	4-11	A-3	40 X	0.7
004	4-12	A-4	40 X	0
004	3-14	A-5	Leitz	0.7
004	3-13	A-6	Leitz	0
004		A-7		
018	4-1	A-8	10 X	4.1
018	4-3	A-9	10 X	2.2
018	4-17	A-10	40 X	0
018	4-13	A-11	40 X	4.1
018	4-16	A-12	40 X	2.2
018	3-11	A-13	Leitz	4.1
018	3-12	A-14	Leitz	2.2
018	3-10	A-15	Leitz	0
022	4-6	A-16	10 X	4.1
022	4-5	A-17	10 X	0
022	3-7	A-18	Leitz	4.1
022	3-8	A-19	Leitz	0
025	3-20	A-20	10 X	0.3
025	3-22	A-21	40 X	0
025	3-17	A-22	10 X	0
025	4-18	A-23	40 X	0.3
025	3-4	A-24	Leitz	0.3
025	3-1	A-25	Leitz	0

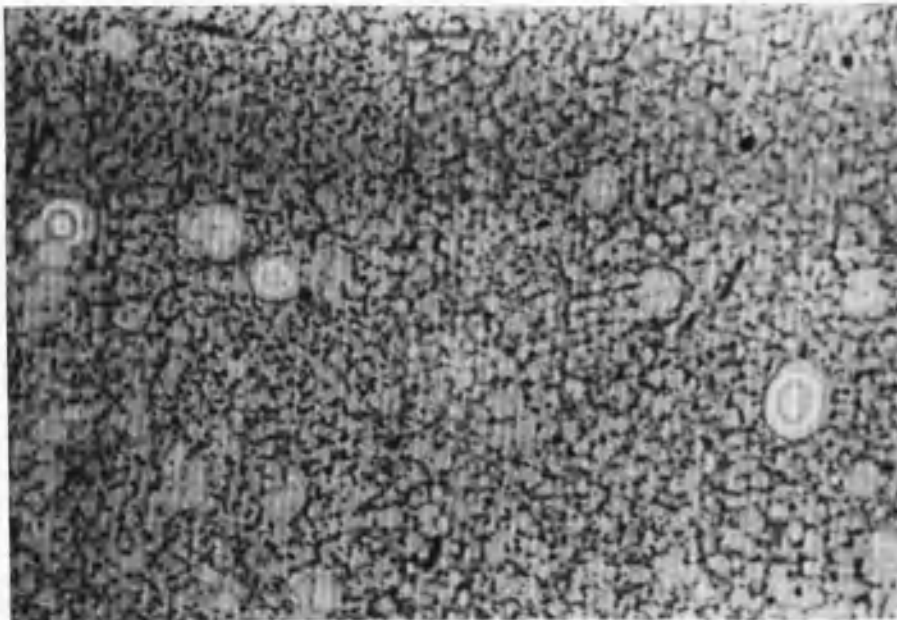


Fig. A.1. Diamond-turned substrate 004 with  $0.7\ \mu$  material removal; exhibits stress-related preferential etching, 10 X objective.

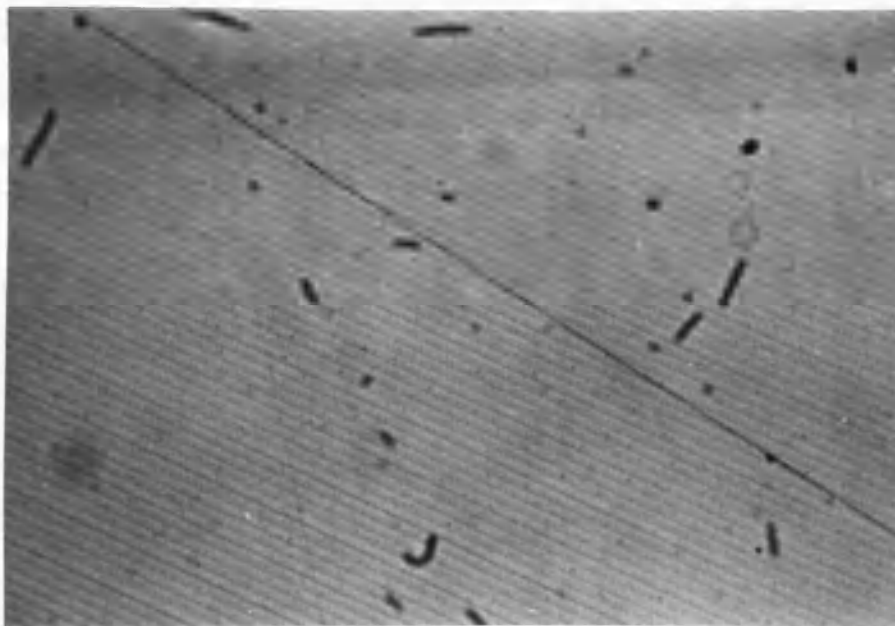


Fig. A.2. Diamond-turned substrate 004, untouched area; shows some dirt and light scratches; 10 X objective.



Fig. A.3. Diamond-turned substrate 004 with  $0.7 \mu$  material removal; exhibits stress-related preferential etching; 40 X objective.



Fig. A.4. Diamond-turned substrate 004, untouched area, 40 X objective.

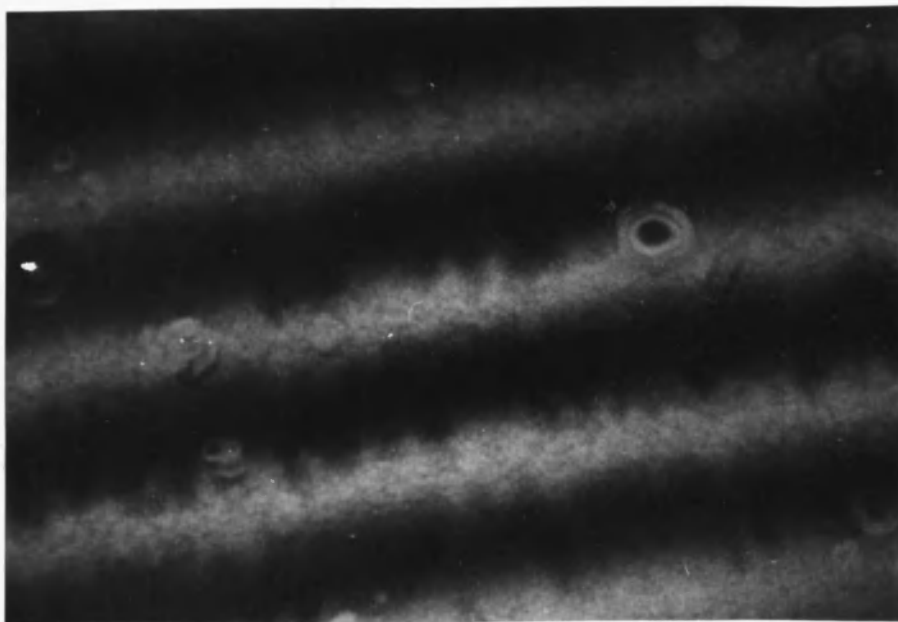


Fig. A.5. Diamond-turned substrate 004, microinterferogram with  $0.07 \mu$  material removal; shows localized etching, each band indicates a turning depth of  $0.07 \mu$ ; 10 X objective.

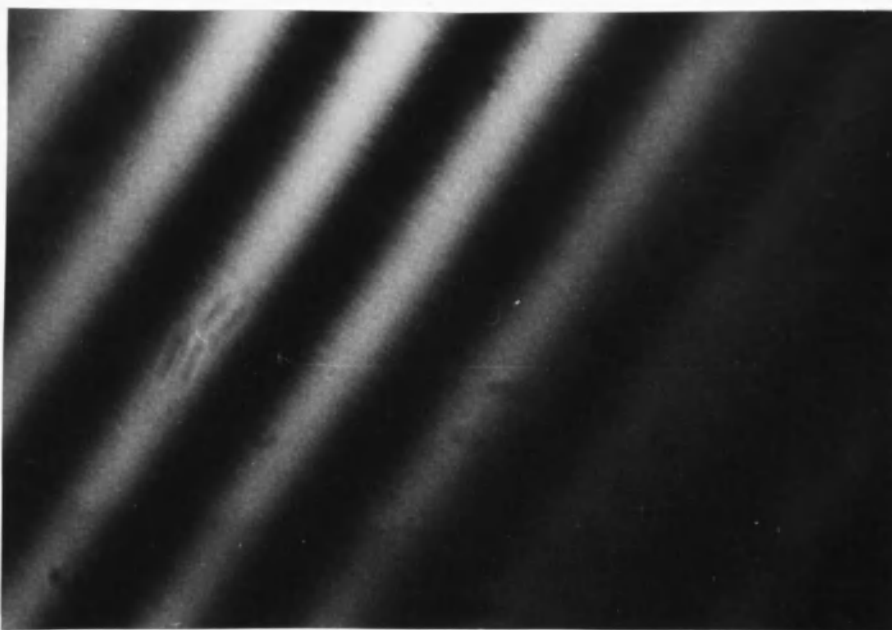


Fig. A.6. Diamond-turned substrate 004, microinterferogram, untouched area; each band indicates a turning depth of  $0.07 \mu$ ; 10 X objective.



Fig. A.7. Diamond-turned substrate 004, interferogram; two-fringe removal at 3 o'clock; the scallop in the fringes between 6 and 10 o'clock are local dynamic removal areas with voltage change from 2 to 7 volts at 6 o'clock.

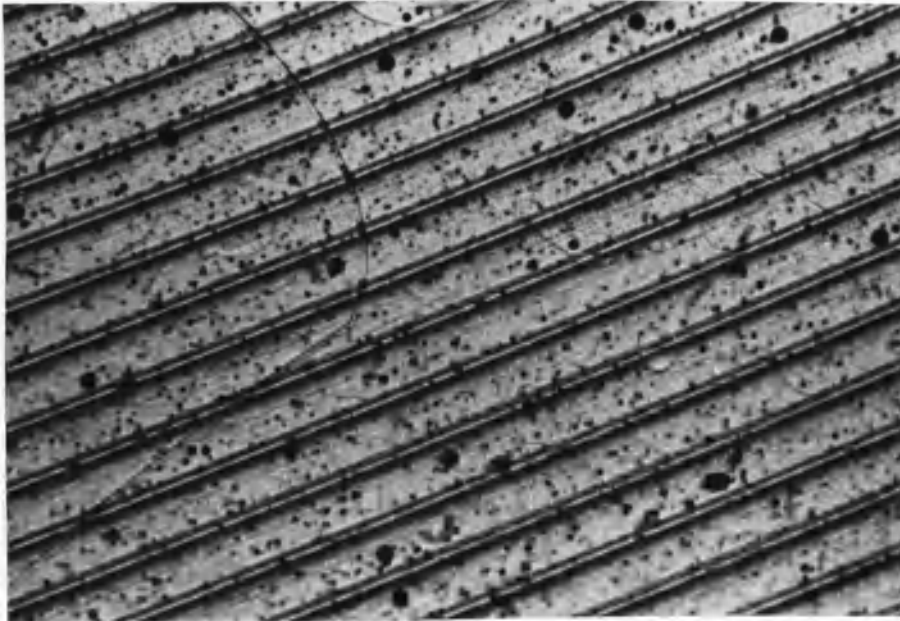


Fig. A.8. Diamond-turned substrate 018, 4.1  $\mu$  material removal, granular attack hazing; 10 X objective.

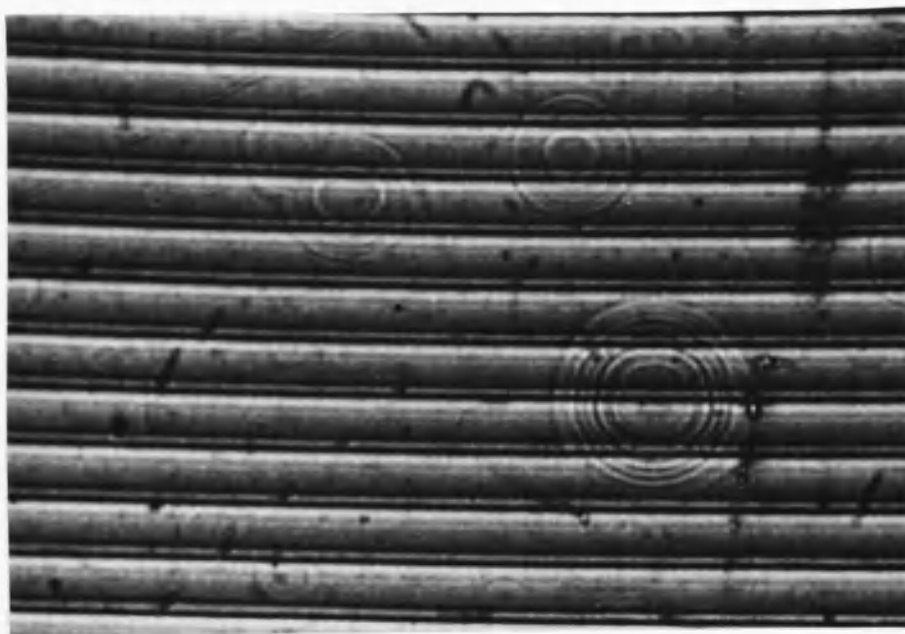


Fig. A.9. Diamond-turned substrate 018, 2.2  $\mu$  material removal, granular attack hazing, 10 X objective.

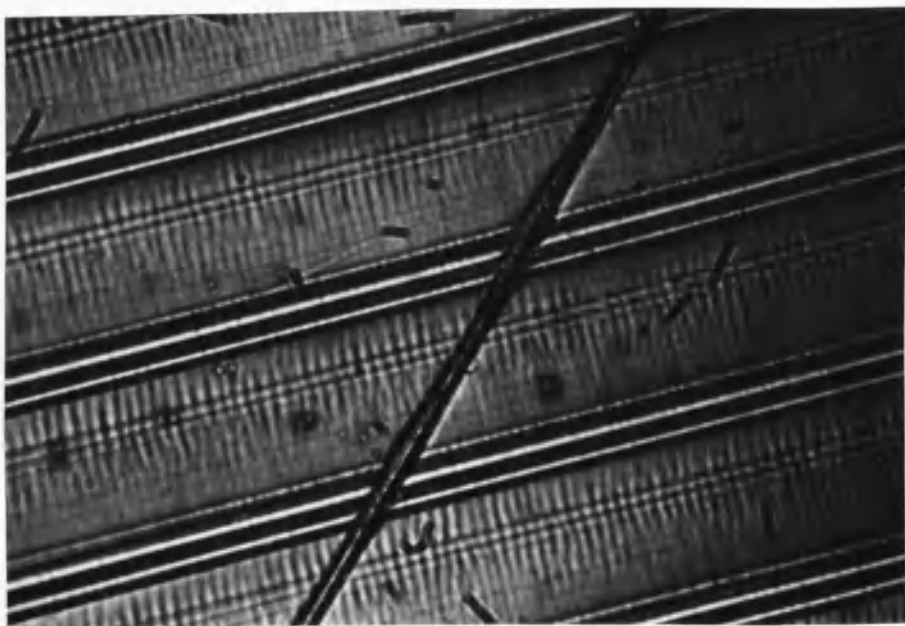


Fig. A.10. Diamond-turned substrate 018, untouched area, scratch evident, 40 X objective.

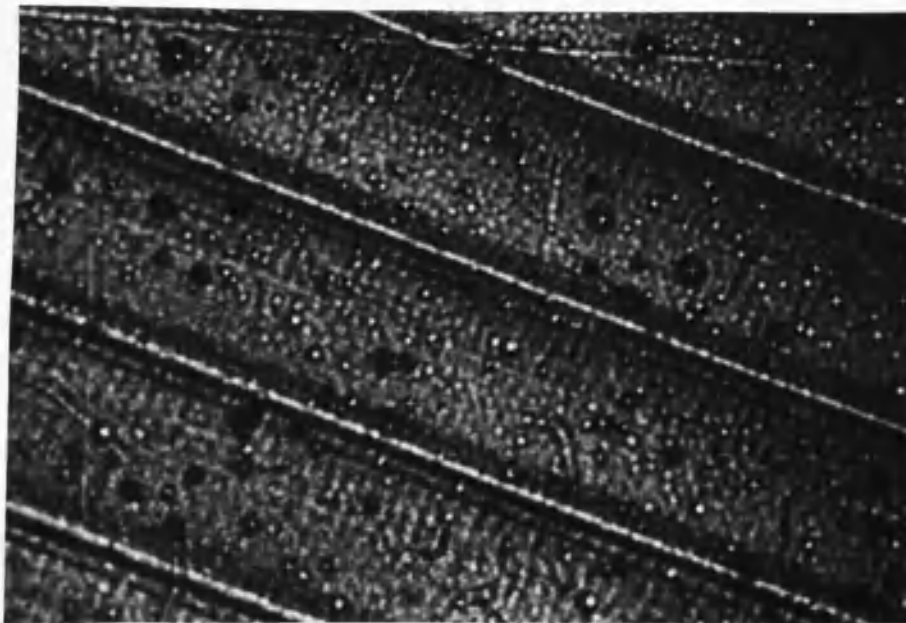


Fig. A.11. Diamond-turned substrate 018, 4.1  $\mu$  material removal, granular attack evident, 40 X objective.



Fig. A.12. Diamond-turned substrate 018, 2.2  $\mu$  material removal, some attack and burn spots, 40 X objective.

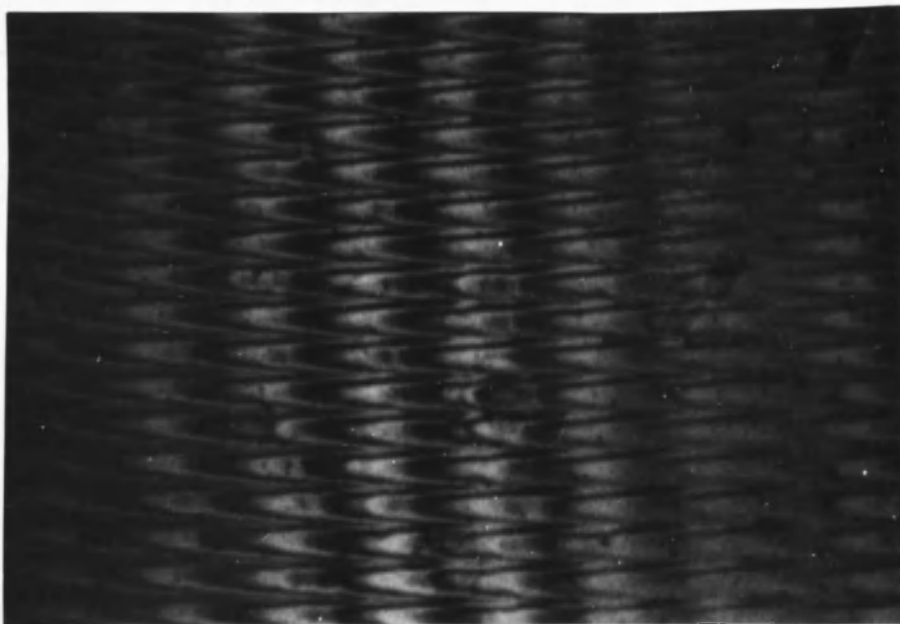


Fig. A.13. Diamond-turned substrate 018, microinterferogram  $4.1 \mu$  material removal, large unique shaped turnings evident, 10 X objective.



Fig. A.14. Diamond-turned substrate 018, microinterferogram  $2.2 \mu$  material removal, large unique turnings are evident, 10 X objective.

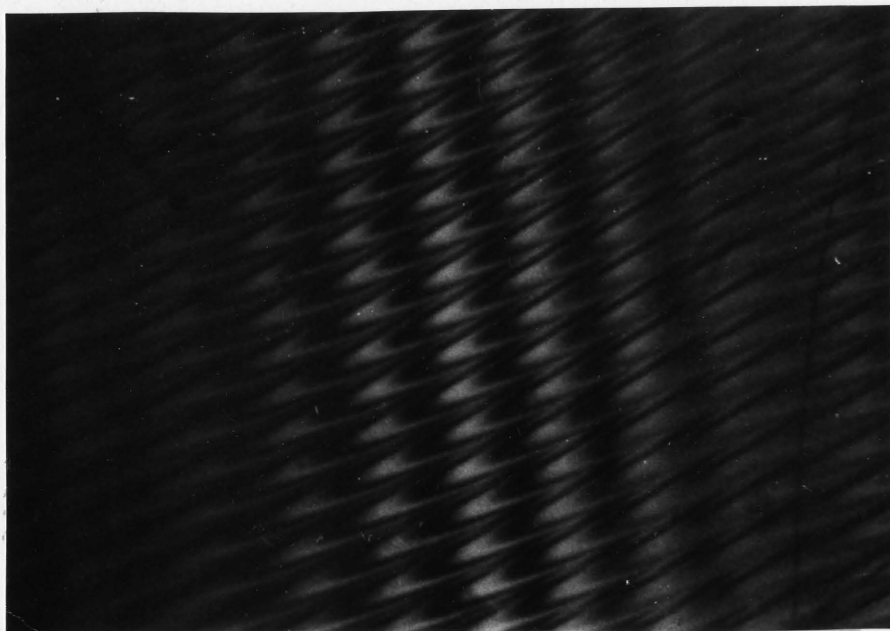


Fig. A.15. Diamond-turned substrate 018, microinterferogram untouched area, large unique shaped turnings observable, 10 X objective.

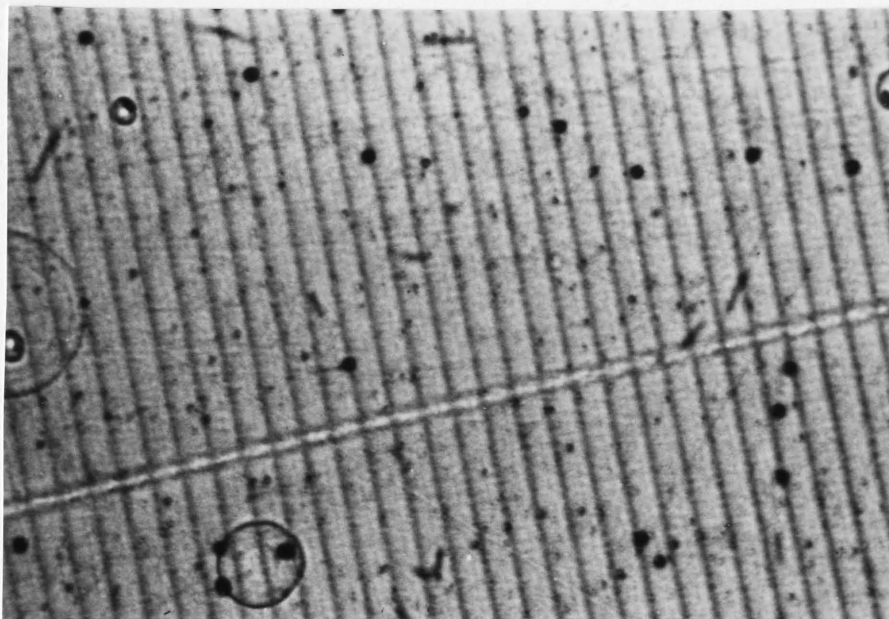


Fig. A.16. Diamond-turned substrate 022, 4.1  $\mu$  material removal, granular attack evident, light scratch, 10 X objective.

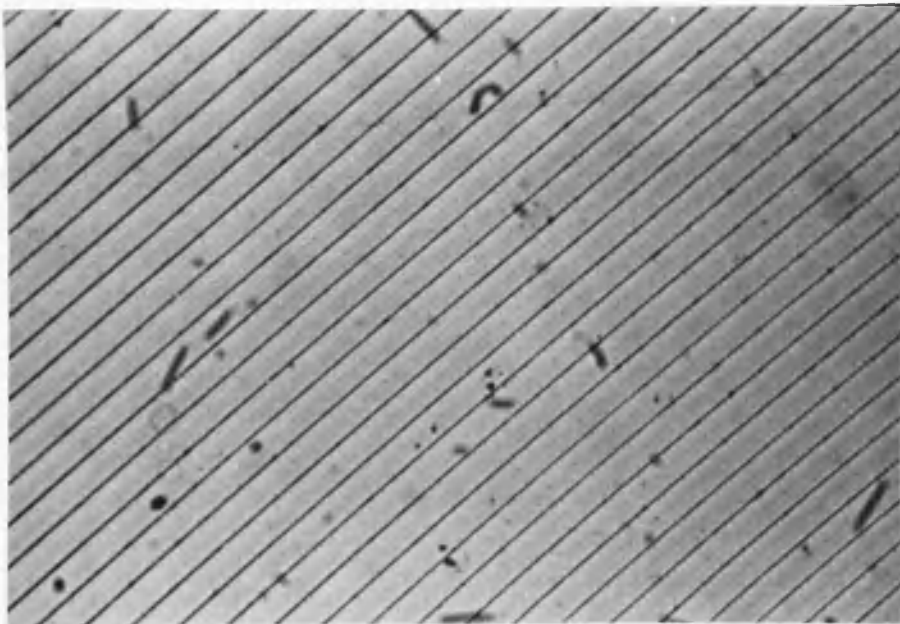


Fig. A.17. Diamond-turned substrate 022, untouched area, 10 X objective.

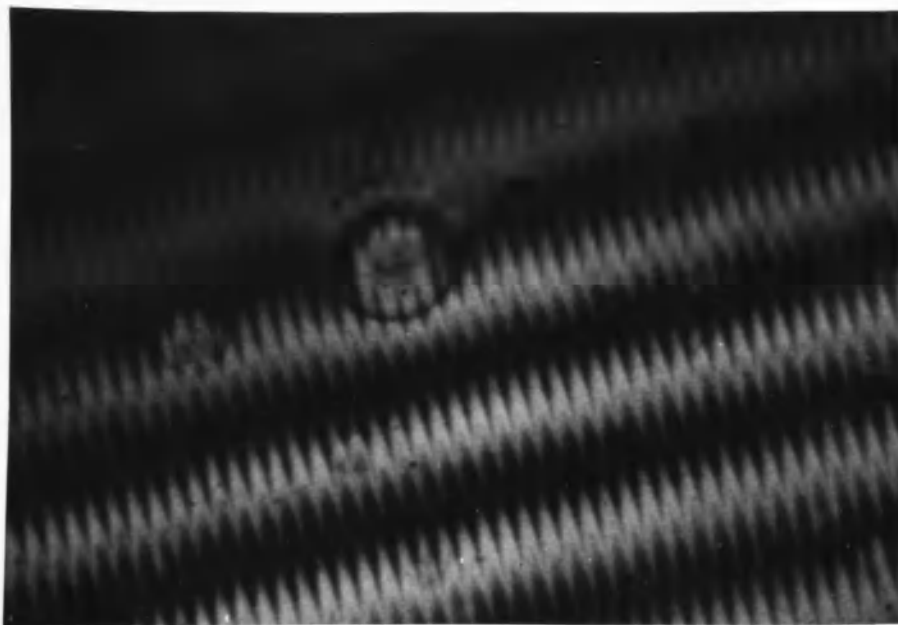


Fig. A.18. Diamond-turned substrate 022, 4.1  $\mu$  material removal, microinterferogram, localized attack evident, unique shaped turnings observable, 10 X objective.

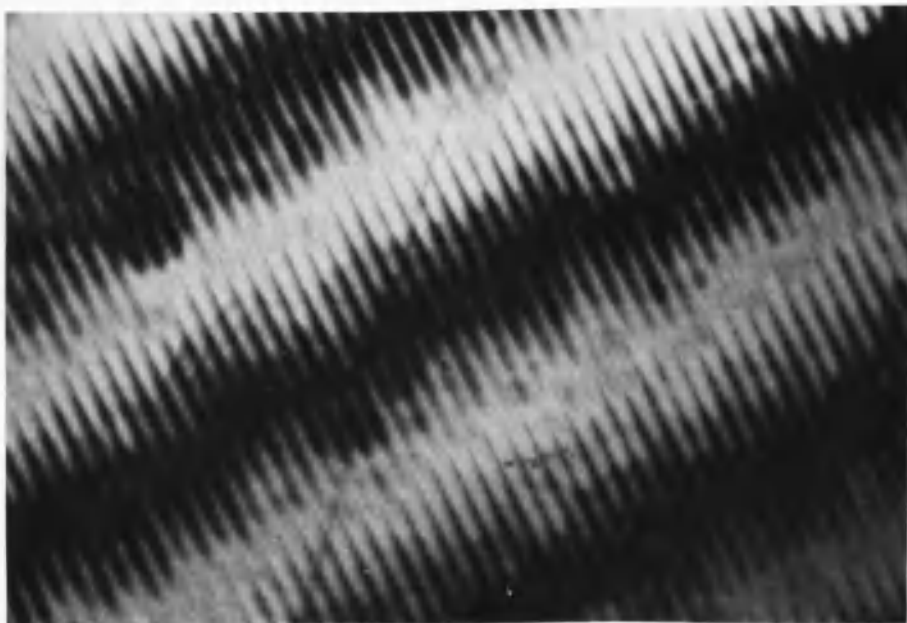


Fig. A.19. Diamond-turned substrate 022; untouched area microinterferogram, unique shaped turnings evident 10 X objective.

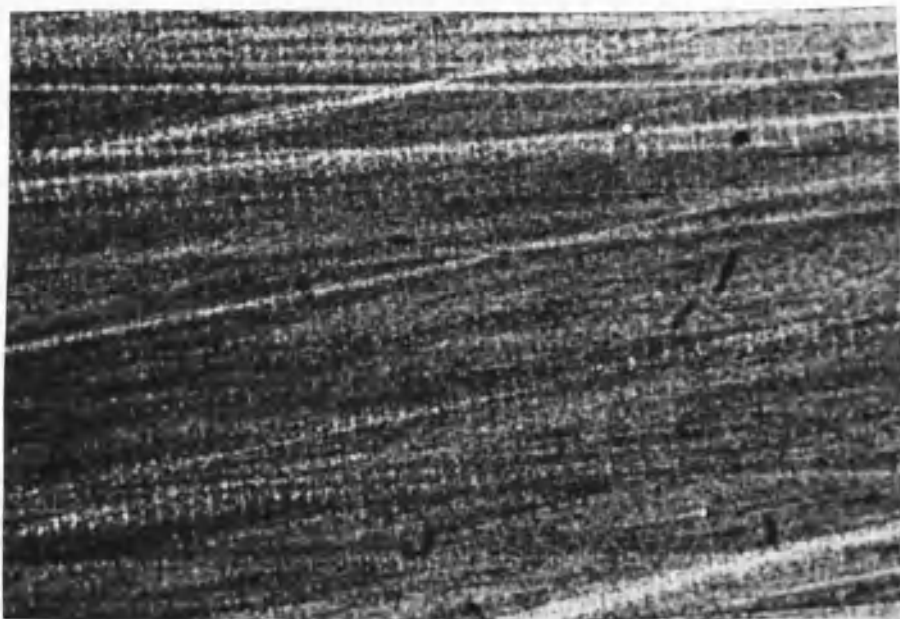


Fig. A.20. Diamond-turned substrate 025, 0.3  $\mu$  material removal, heavy granular deposit and attack, 10 X objective.

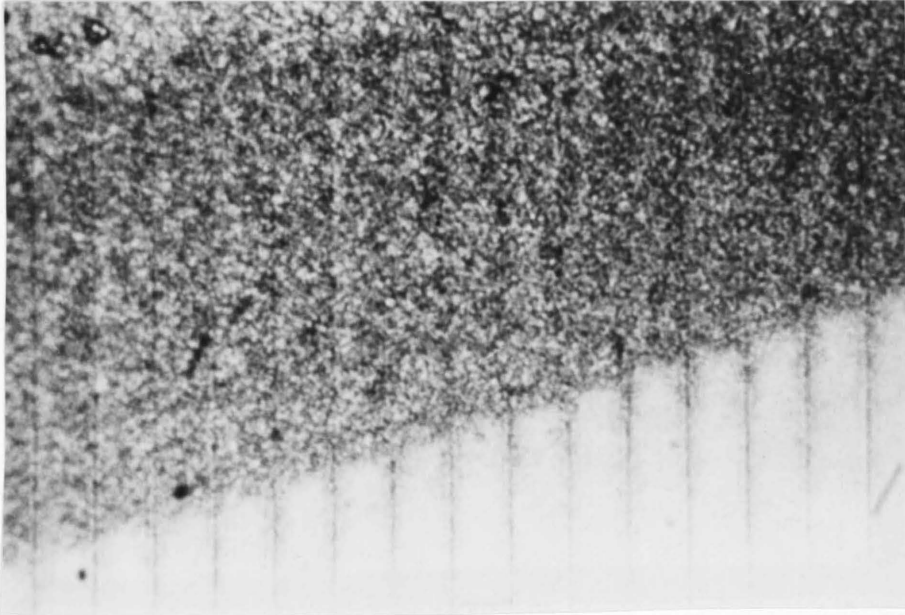


Fig. A.21. Diamond-turned substrate 025, untouched area and electro-polished attack area, 40 X objective.

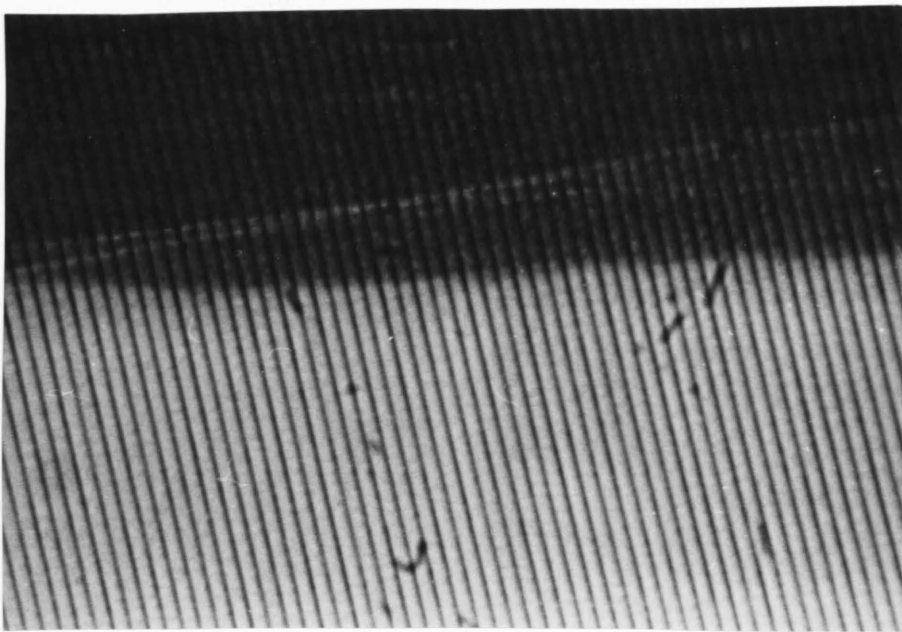


Fig. A.22. Diamond-turned substrate 025, untouched area and attack area, 10 X objective.



Fig. A.23. Diamond-turned substrate 025, 0.3  $\mu$  material removal, heavy granular deposit and attack, 40 X objective.

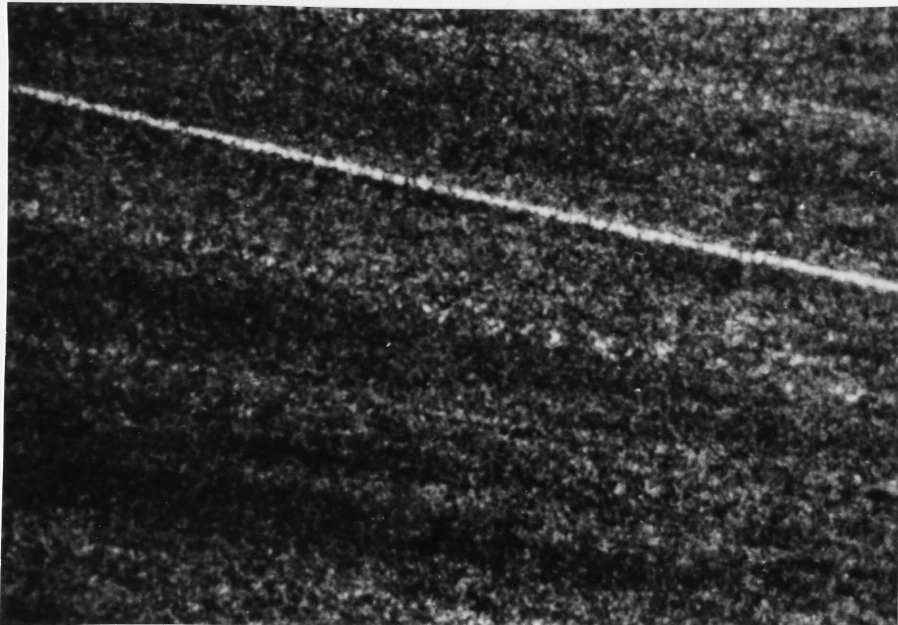


Fig. A.24. Diamond-turned substrate 025, 0.3  $\mu$  material removal, microinterferogram, very fine turnings observable in bands, 10 X objective.



Fig. A.25. Diamond-turned substrate 025, untouched area microinterferogram, very fine turnings observable in bands, 10 X objective.

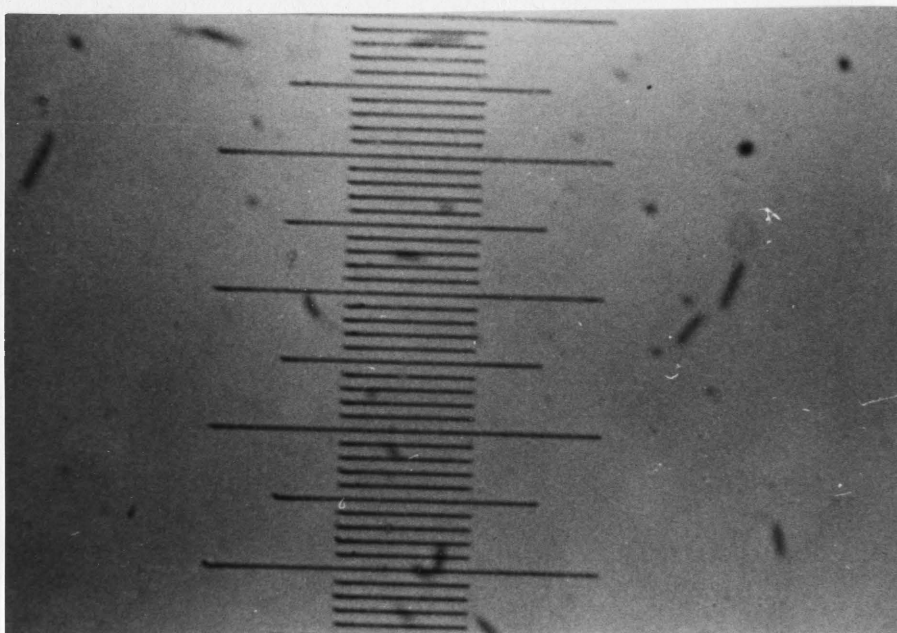


Fig. A.26. 10 X objective scale, 1 division = 20  $\mu$ .

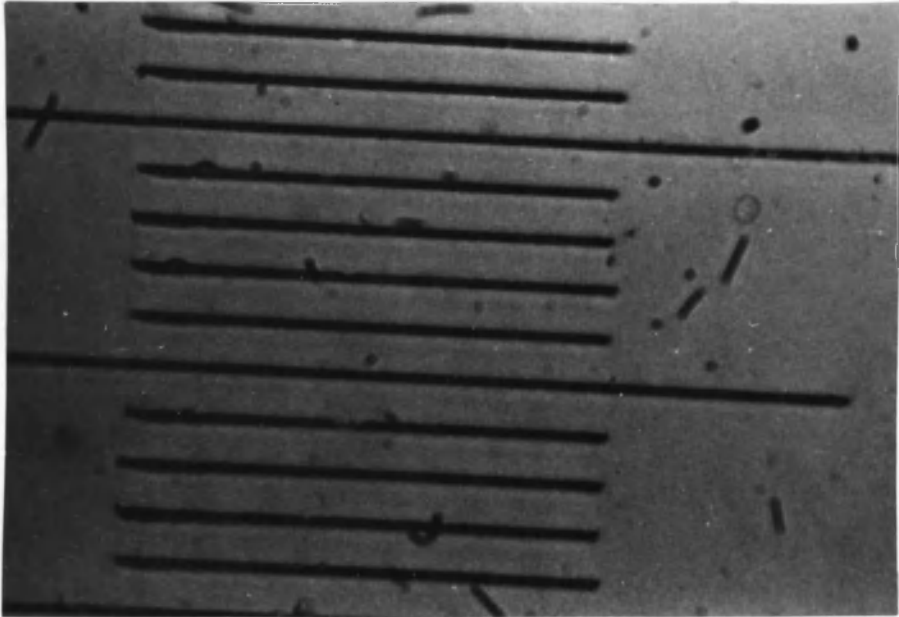


Fig. A.27. 40 X objective scale, 1 division = 20  $\mu$ .

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