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An account of the first experiments and results will be addressed. The efforts and results from an extensive fused silica grinding and polishing study will follow. The bulk of the research performed during the contract period was devoted to the fabrication of optical quality fused silica flats. A portion of the best effort fused silica samples were used in a thin-film coating study carried out by the Developmental Optics Facility of the Phillips Laboratory at Kirtland Air Force Base. Behaviors of the float polished fused silica substrates due to thin-film deposition can be found in that final report. The results of the optical fabrication of other materials, such as potassium deuterium phosphate (KD*P) and crystalline quartz, will be discussed. A list of publications and conference presentations associated with this effort will close the report.



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Float Polishing Final Report
February 1, 1990 - September 30, 1993
AFOSR - 90 - 0145

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# Final Report
## February 1, 1990 - September 30, 1993

### Introduction

This final report will cover all of the efforts and results during the awarded contract period. The format will generally be a chronological account of events, however, several projects were performed concurrently. These simultaneous projects will be discussed separately rather than chronologically to avoid confusion.

An account of the first experiments and results will be addressed. Following this is the efforts and results from an extensive fused silica grinding and polishing study. The bulk of the research performed during the contract period was devoted to the fabrication of optical quality fused silica flats. A portion of the best effort fused silica samples were used in a thin-film coating study carried out by the Developmental Optics Facility of the Phillips Laboratory at Kirtland Air Force Base. Behaviors of the float polished fused silica substrates due to thin-film deposition can be found in that final report. The results of the optical fabrication of other materials, such as potassium deuterium phosphate (KD*P) and crystalline quartz, will be discussed. A summary and conclusions section will draw together the concepts and results obtained during the effort. A list of publications and conference presentations associated with this effort will close the report.

## Prelude

In 1989 a float polishing machine was purchased be UNM from Toyoda Machine Works under AFOSR-89-0337 contract. The machine was purchased to perform a technology transfer of the float polishing technique invented in Japan by Dr. Y. Namba to finish electronic components without damage and contamination, and to investigate the fundamental parameters influencing float polishing in order to achieve low scatter, smooth, damage-free surfaces.

The machine was delivered in November 1989 and was installed the following month. The first three months after installation were devoted to learning how to cut the tin and copper laps, how to fabricate the proper lap grooves, and how to deburr the lap prior to polishing. Because the optical part being polished only floats approximately .5 μm above the lap, it is essential that all metal burrs larger than .25 μm be removed. The procedure to machine and deburr the lap properly was developed during this three month period. Polishing then proceeded.

## Polishing of Crystalline Quartz

Crystalline quartz was chosen for polishing based upon previous studies using a rather crude, single spindle polishing machine. The float polishing technique was utilized in the fabrication of disc-shaped quartz resonators, having a diameter of 6.35 mm and a polished thickness of 104 μm, to increase the fracture strength of the resonators. We were able to remove all the subsurface damage that manifests itself in lower stress failures. The stress fracture threshold was increased up to 4-10 times that of conventionally polished samples (approaching the bulk value). All of this work was reported at the Science of Optical Finishing Conference at Monterey, California on June 10-12, 1990.

In an attempt to fully quantify the surface roughness and the presence of sub-surface damage resulting from the polishing process, natural crystalline quartz substrates were polished and analyzed.

Natural quartz substrates, 25.4 mm in diameter, were polished to a thickness of 0.9 mm. The substrates were fixed with wax in a hexagonal pattern on a base that weighed 2000 gm. The substrates were first float polished on a diamond turned Cu lap with a 2% wt. $TiO_2$-deionized $H_2O$ slurry. The $TiO_2$ particle size is approximately 1000 Å in diameter. The removal rate in this procedure was close to 1 μm/hr, as measured by a digital micrometer. Approximately 10 μm were removed from one face (Side A) and 100 μm from the opposite face (Side B) of each samples. The substrates were then float polished with a slurry of the same composition as above on a Sn lap for 4 hr, and finally polished on a Sn lap with a 2% wt. 70 Å fumed silica-deionized $H_2O$ slurry. The slurry temperature was controlled at 20° C to prevent any lap warpage from occurring due to temperature changes during the polishing process. The rotational rate of the substrates and the lap were set at 75 rpm. The removal rate in this procedure was extremely slow and not measurable by standard techniques. The wax residue was removed by soaking in 111 tri-chloroethane, and an enamel paint mark was placed on the edge to distinguish the two sides. The substrates were inspected for overall surface quality using a Nomarski microscope. On Side A there were still many imperfections in the surface. Side B had fewer imperfections, but not all imperfections had been removed.

Photoacoustic spectroscopy using surface acoustic wave (SAW) generation was performed on both surfaces of the samples to determine the surface absorption. This absorption is determined by the properties of approximately the first 50 μm of material; the surface absorption is indicative

of the presence of substoichiometric material or incorporated impurities in these layer. Photoacoustic spectroscopy is a well know technique used for investigating physical properties of various media.[1] A system for SAW detection is described in the reference.[2] A pulsed optical beam in a one-dimensional grid pattern is imaged on the sample. The nonradiative fraction of the absorbed radiation results in a local rise in temperature and a corresponding decrease in the local density. This local thermal gradient propagates as a surface wave and is detected by a piezoelectric transducer coupled to the sample. The amplitude of this signal is proportional to the absorbed nonradiative fraction of power. The SAW measurements of the two sides of a float polished quartz sample are shown in Table 1.

Table 1

| Surface | Photoacoustic Signal |
|---|---|
| Side A | 1.3 ppm |
| Side B | 0.4 ppm |

The photoacoustic signal of Side B is approximately one-third that of Side A. In fact the 0.4 ppm is very near the noise floor of the instrument. Our experience with photoacoustic absorption of conventionally polished optical components is that absorption levels are usually 1 ppm or larger. It should be noted that the photoacoustic signal did not vary on Side B when the laser beam was incident on different regions of the surface. The absorption was the same when examining a region of a smooth surface area, and where surface imperfections existed as observed by Nomarski microscopy. Because of this, it is believed that the surface imperfections that were observed are due to

crystalline imperfections and not due to the sawing and lapping operations. In an effort to obtain better quality substrates, polishing of Corning 7940 fused silica was begun.

**Corning 7940 Fused Silica Polishing**

Polished samples of Corning 7940 fused silica, fabricated by General Optic, were selected for float polishing. Initially, the 1 inch diameter samples were closely examined under the Nomarski microscope. The surfaces appeared to be very good with no observable surface imperfections. These substrates were then float polished with a 2% wt. $TiO_2$-deionized $H_2O$ slurry. The particle size of the $TiO_2$ is approximately 1000 Å diameter. The rotational rate of the substrates and the lap were 80 rpm with a removal rate of approximately 1 μm/hr. After 125 μm were removed from the surface, defects were visible on the surface with a Nomarski microscope. The defects were randomly distributed on the surface. Also there seemed to be defects systematically distributed in an arc having a radius of curvature of approximately 2 inches (≈ 40 mils defect size). Two microns of material were removed by further polishing. Most of the defects that were previously visible had been removed, and new defects appeared. Subsequent polishing revealed previous defects being removed and new defects appearing. It appears that as material is removed, damage is removed in some areas but in other areas the damage increased, presumably the result of material fracture. This fracture may be due to stress in the material or to the shape of the microcracks in the material. When 230 μm of material had been removed form the surface, there were no signs of any surface defects.

The samples were then float polished for 12 hr with a 2% wt. 70 Å fumed silica-deionized $H_2O$ slurry to obtain a smoother surface. Surface

roughness measurements were taken of the three samples. These results are shown in Table 2.

Table 2

Wyko Optical Profilometer NCP-1000

RMS Surface Roughness Measurements (Å)

Limit of Resolution 3.5 Å

218 Hour 1000 Å Titanium Oxide Polish Followed by

12 Hour 70 Å Fumed Silica Polish

| Sample | Measurement 1 | Measurement 2 | Measurement 3 | Average |
|---|---|---|---|---|
| Control | 2.80 | 3.04 | 2.41 | 2.75 |
| C20 | 4.99 | 6.11 | 3.27 | 4.79 |
| C90 | 5.54 | 4.37 | 3.17 | 4.36 |
| C109 | 6.43 | 3.33 | 3.94 | 4.57 |

**Fused Silica Optical Flat Fabrication Study**

One of the primary goals of the contract was to investigate the fundamental parameters influencing float polishing in order to achieve the best surfaces possible. A study was conducted to identify and characterize these parameters for the fabrication of fused silica flats. A detailed investigation of the grinding phase of the fabrication process was carried out. Then parameters influencing the float polishing phase were investigated. Several diagnostic techniques were employed and developed to characterize the surface and subsurface features of the fused silica polished flats. The procedures and results of these efforts follow.

## Grinding

Rough grinding of fused silica samples is necessary for removal of previous damage, such as saw marks, and to insure relatively good planararity and flatness of samples. The latter requirements are crucial to the float polishing phase of the fabrication process. After several approaches the below grinding procedure was adopted.

Corning 7940 fused silica samples with a 5 cm diameter and a 1 cm thickness were used almost exclusively. Polished bevels are required to prevent chipping of the outer edges of the samples. A schematic of the grinding setup is shown in Figure 1. Oxygen free high carbon (OFHC) copper was used as the lap material because of its resistance to wear. The low amount of wear reduced the tendency of the lap surface to become concave during grinding. The copper lap was grooved concentrically 0.25 mm deep and 1 mm wide with 1 mm lands and faced on the float polishing machine. The tooling on the machine faces to ±1 μm over the surface of the lap. Three samples were mounted concentrically and equally spaced on a 15 cm diameter fused silica plate 1 cm thick with thermoplastic cement. The diameter of the circle circumscribing the samples was 12 cm. Strips of 1/8" thick 3/8" wide double sided tape held the glass plate to the work holder. Recently, the tape was eliminated and filament tape was adhered to the outside circumference of the glass plate and work holder bonding the two together. This reduced any possible flexing of the glass plate due to gaps between the double sided tape. The lap surface was cut such that the samples would overhang the lap a maximum of 4 mm. The overhang improves the figure of the samples. A 15 cm diameter fused silica plate 2.54 cm thick was attached to a separate spindle drive and this device was mounted to the float polishing apparatus. The

fused silica plate was centered on the lapping surface and ground at the same time as the samples thus providing greater uniform wear of the lap. This plate is referred to as the conditioning plate. The pressure of the conditioning plate is slightly greater than the pressure of the samples.

The distance from the lap center to the work spindle center was 17 cm. The angular velocity of the lap was 2.1 rad/s (20 rpm) and that of the work spindle and conditioning plate was 2.2 rad/s (21 rpm) to further promote even lap wear. A slurry of 2 μm synthetic polycrystalline diamond and deionized water (0.2 g diamond/2.5 cc deionized water) was applied to the lap in five drop quantities from a hypodermic needle every 15 minutes. The polycrystalline diamond was used over single crystal diamond by recommendation of the vendor. It was stated that single crystal diamond produces greater subsurface fractures than polycrystalline diamond. For a given grinding particle the pressure exerted on the sample is greater for a single point than for multiple points. Deionized water was applied every few minutes in amounts sufficient to keep the lap thoroughly wet. The first hour of grinding was at a pressure of 53 g/cm$^2$ to prevent any unstable motion of the work piece due to the differing heights of the samples. The pressure was increased to 84 g/cm$^2$ for the remainder of the grinding process. The average removal rate at the increased pressure was 25 μm/hr.

Two modes of material removal exist when grinding fused silica. The brittle or fracture mode results in appreciable removal rates. When plastic flow of the sample material occurs a ductile mode is present. The removal rate is negligible with this mode. A brittle mode of grinding was predominant at the two operating pressures. Figure 2 is a photograph of a brittle ground surface. Many conchoidal fractures can be observed in this figure. These two modes are not mutually exclusive, they can partially occur

simultaneously. Hourly observation with a Nomarski microscope indicated no saw marks or lateral cracks at a removal depth of ~100 μm as measured with a digital micrometer (1 μm precision). An additional 50 μm of material was removed to insure that no preexisting damage remained. Interferometric measurements of the samples after grinding indicated a flatness of ~1 λ (λ = 630 nm) with a slightly convex figure.

Without the conditioning plate a surface profile matching the concentric lands of the copper lap was observed on the outer 1/3 area of the samples after grinding. The profile was identified by placing a test lap on the surface and noting the steps in the fringes. The profile occurred due to the mounting geometry of the samples and the lap groove pattern. More sample area is in contact with the central region of the lap than the outer region, and therefore the central region of the lap annulus wears at a greater rate than the outer regions. A concave lapping surface is the result of this wear, and the pressure on the sample surfaces increase at the outer edges of the lap. Thus an imprint of the lap pattern will occur at the outer edges of the samples.

The lap geometry plays a critical role in the grinding process as stated above. Cutting a spiral groove in OFHC copper was attempted but not successful. The lap material is too hard to machine a spiral groove of reasonable depth on the float polishing apparatus. Such a groove would promote asymmetry during the grinding process and eliminate any lap patterns on the surface of the samples. A modified concentric pattern was adopted instead. This consisted of 2 mm and 1 mm lands and grooves in an antisymmetric pattern. See Figure A. The mean radius of the lapping surface is $R_m$, and the radial distance to the center of the designated groove and land is $R_g$ and $R_l$, respectively. The width of the designated groove and land are equal. The relation $|R_g - R_m| = |R_m - R_l|$ provides the antisymmmetric

geometry. This pattern along with a conditioning plate has proved very successful in eliminating lap patterns on the surfaces of the ground samples.

A phenomenon known as the zeta potential and its effect on grinding modes was investigated. A brief description of the $\zeta$ potential and how it can be used to control grinding modes follows.

When a solid-liquid interface is established an electrical potential is set up in the liquid near the interface. See Figure 3. This potential is mainly due to adsorption effects at the interface. The inhomogeneous distribution of positive charge is a result of diffusion properties. If a liquid undergoes laminar flow a shear plane is set up. In the region between the shear plane and the interface the fluid velocity is zero. Outside this region the fluid velocity increases with increasing distance from the interface. When an external electric field is applied tangentially to a fixed surface the mobile portion of the fluid will flow. A constant flow rate will be reached when the electric forces balance with the frictional forces. Considering the viscosity and velocity of the fluid, the potential at the shear plane, and the applied field, the $\zeta$ potential can be determined without further consideration of the structure of the charge distribution. This is a valid assumption because the fluid velocity is independent of distance from the shear plane for small geometries. Potentials of ~100 mV have been measured. Even with these small values fields on the order of $10^6$ V/m are present owing to the small dimensions involved. However, the $\zeta$ potential cannot reveal the structure of the diffuse layer with this assumption. The $\zeta$ potential is dependent on many parameters such as the potential determining layer and solubility of the solid, and the electrolytic nature, pH level, and dielectric constant of the liquid. Nevertheless, with the many variables associated with the $\zeta$ potential it is a

very useful quantity to predict the hardness of a material experiencing scratching by a hard, sharp object.

Hardness is a term describing how easily a solid material wears when subjected to scratching or grinding. Two types of scratching can occur. The first is known as brittle or fractile scratching. Bits of material exit the surface because of concoidal fracturing and this implies a "soft" material exists. On the other hand, ductile or plastic scratching is a smearing of the material owing to plastic flow. The amount of material removal during ductile grinding is small compared to brittle grinding indicating a "hard" surface. Past experiments have shown a correlation between hardness and $\zeta$ potential. When a non-zero $\zeta$ potential exists the hardness of the material is less than if a zero $\zeta$ potential were present. One idea suggests that the high fields present with a non-zero $\zeta$ potential induces stress at the surface of the solid thereby making it easier to produce brittle fracture. The importance of slurry types used during geological drilling operations can be appreciated. Studies have been conducted to control the type of grinding or scratching by controlling the $\zeta$ potential. Westwood has shown the relationship between slurry pH and hardness and concluded that the pH influences the $\zeta$ potential. He also controlled the $\zeta$ potential by applying an external electric field perpendicular to the interface. By varying the magnitude and direction of the field he noted the hardness response.

The isoelectric point (IEP) is defined as the pH of a solution at which the $\zeta$ potential is zero. Fused silica has an IEP of ~2. There are many ways to influence the $\zeta$ potential owing to the many parameters associated with it. Controlling the hardness of fused silica by varying the pH requires operating in a highly acidic region. Since tin and copper are soluble in acid, the use of

an external field to control the hardness seems to be a more promising approach.

To achieve high quality polished surfaces the grinding process previous to polishing is paramount. The manner in which the surface was ground greatly influences the required polishing time. The pressure, diamond size, lap material, and pH of the solution are some parameters that control the type of grinding performed. Brittle grinding produces fractures that can extend tens of microns below the surface and will leave a surface with a dull shine. These fractures are undesirable and must be removed. Float polishing has been shown to remove this type of damage but the removal rate is on the order of one micron per hour. Ductile grinding on the other hand does not produce fractures. Achieving ductile grinding requires operations at low pressures and small diamond size. This reduces the removal rate greatly when compared to brittle grinding. Therefore, a large amount of time is required to grind away previous damage from sawing. Grinding an optic in the brittle mode then switching to the ductile mode seems promising. Brittle grinding would remove saw marks in a quick manner and ductile grinding would remove these fractures in a reasonable amount of time. This scheme would reduce the overall time to fabricate a high quality optic.

The ductile removal of fused silica has not yet been achieved. It is believed that the fused silica undergoes a depolymerization and repolymerization process. However, ductile removal has been observed with composite glasses. An approach has been considered to obtain ductile removal of fused silica and will be discussed later.

An experiment was carried out to confirm the ability to control the grinding mode with an applied electric field. A schematic of the experiment

is shown in Figure 4. Three 1/4 inch thick, 1 inch diameter fused silica samples were ground with 3 μm polycrystalline diamond on a tin lap at a pressure of 56 g/cm$^2$. An average removal rate of 25 μm/hr was measured. Removing ~100 μm of material was required to eliminate saw marks and insure a stable mode of grinding. A predominantly brittle mode was present with a few ductile scratches as viewed with a Nomarski microscope. An electric field was then applied normal to the glass surface at different potentials and the removal rate and glass surface was observed. It was confirmed that the electric field did influence the mode of grinding. This was supported by a drop in removal rate and increased ductile scratching of the glass surface. A minimum removal rate was observed at an applied potential of 600 volts. This value is within an order of magnitude of the calculated value required to produce a zero ζ potential.

Cuthrell at Sandia National Labs has shown that the hardness of glass is influenced by the pH of a solution through acoustical monitoring of the drilling of the glass. He placed an acoustic sensor on the bottom of a Pyrex sample, drilled the glass on the top surface with a diamond bit in a solution and counted the acoustic events per unit time. Brittle fracture will produce more acoustic events than ductile. The wear rate of the glass was proportional to the acoustic event. He found that a minimum event count was observed when the pH level of the solution corresponded to a zero ζ potential. This means a reduction in wear rate was achieved indicating ductile grinding was present. In our case, by applying an external field during the grinding of glass and monitoring the acoustic events, the field can be tuned to control the mode of grinding.

## Polishing

A schematic of the float polishing arrangement is shown in Figure 5. A tin lap replaced the OFHC copper lap and was machined on the float polishing apparatus. The lap was faced with a diamond tool until no defects were visible with the eye and undetectable by touch. Concentric grooves 1 mm wide and 1 mm deep with 1 mm lands were cut into the lap. It was discovered after polishing that the lap pattern was evident on the surface of the optic. A spiral groove 1 mm wide and 250 μm deep with land widths ranging from 1 mm to 4 mm was cut to promote asymmetry. This spiral pattern eliminated the concentric pattern on the polished samples. A microgroove 50 μm deep was cut with a 100 μm radius diamond tool into the lands as a continuous spiral. The center-to-center distance between adjacent microgrooves was 250 μm resulting in microlands 76 μm wide. The microgrooves and microlands are necessary to facilitate hydroplaning. The lap surface was cut to give the samples a maximum overhang of 0.5 mm. The overhang is necessary to prevent any edge effects from occurring.

The distance from the lap center to the work spindle center was 17 cm. The angular velocity of the lap was 8.4 rad/s (80 rpm) and that of the work spindle was 8.6 rad/s (82 rpm). These speeds were chosen from previous literature on float polishing and seemed to provide stable operating conditions. The parameters controlling the removal rate have not been fully characterized. A hydrodynamic model is currently being developed to predict what influence the different parameters have on the removal rate, surface and subsurface features, and figure of the samples. It has been discovered that the loading pressure, the width of the lands, and the slurry type and temperature influence the removal rate and figure. There are other

parameters to consider also and with the modelling these issues will be addressed. The following trends have been noted. The removal rate increases with increased loading pressure, land width and temperature. The temperature of the slurry controls the figure of the samples by changing the figure of the lapping surface. A modelling of the lap figure as a function of temperature is now being conducted. Several different polishing slurries have been investigated.

A slurry of 2% by weight 300 Å fumed $TiO_2$ in 4 liters of deionized water and 5% vol. of glycerin (to facilitate a smooth start up) was initially used in the polishing phase providing a removal rate of ~1 μm/hr. The pH of this slurry was 4.0. The high removal rate allows the majority of grinding damage to be rapidly polished out. The surfaces are polished for ~100 hours to insure removal of subsurface damage caused by grinding. Inspection of the surface with a Nomarski microscope revealed very few pits (~1 μm diameter) and scratches (~1 μm wide) with a slight texture over the entire surface. These surface features are typical with the $TiO_2$ slurry. To prevent agglomeration of the $TiO_2$ particles the slurry pH must be in the neighborhood of 6 because the IEP of $TiO_2$ is 6.6. However, adding glycerin may change the suspension characteristics, however, a change in pH is not measurable with a .1 pH resolution meter. The added glycerin may be causing agglomeration for other reasons based on the observable surface features.

A 2% by weight 70 Å fumed silica slurry was made by mixing 80 gm of Aerosil 300 fumed silica with 4 liters of deionized water giving a pH of 4.0. This slurry was used to remove the surface features present after polishing with the $TiO_2$ slurry. A removal rate of ~.4 μm/hr was measured at 56 g/cm$^2$ load. After several hours of polishing the number of pits and scratches increased. The defect sizes were the same as the $TiO_2$ slurry defects. This

behavior suggested that agglomeration of the silica particles may have occurred. It was discovered that the manufacturing of fumed silica forms irreversible agglomerates up to 60 times the individual particle size. Colloidal silica does not agglomerate during synthesis . We abandoned the use of fumed silica in favor of colloidal silica.

A 4% by weight mixture of 500 -700 Å colloidal silica (Rodel 2360) and deionized water giving a pH of 8.5 was used as a polishing slurry. A removal rate of ~.6 μm/hr was measured at 155 g/cm$^2$ load. The pressure increase was necessary to achieve reasonable removal rates. A removal of ~ 10 μm was performed with this phase. The surface defects were polished out with no new damage sites visible with a Nomarski microscope at 400X. The ambient temperature was 26° C and the slurry temperature was 23° C due to evaporation. The drop in temperature produces a concave lapping surface.

Temperature control of the slurry was required to control the figure of the samples. Fluid temperature controllers for $TiO_2$ and colloidal silica slurries were added to the float polishing apparatus. Starting with samples having a figure of ~ .25 λ (obtainable with grinding) requires temperature ranges within a few degrees of ambient to hold flatness better than .25 λ. For ground samples with initial figures greater than .25 λ, the operating temperature range must increase to improve the optical figure. Ground samples with optical figures several waves from flat require more polishing time and are more difficult to reduce figure error. Typically, the ground samples have a convex figure and require a convex lap surface during polishing to produce a flat surface. Increasing the temperature of the slurry above ambient causes the lap to form a convex surface. Likewise, decreasing the slurry temperature below ambient produces a concave lap surface.

## Diagnostics

A Total Internal Reflection Microscopy (TIRM) apparatus was used as a qualitative indication of the surface and subsurface nature of the samples. See Figure 6. A 1 Watt CW argon ion laser operating at a 513 nm wavelength is directed into the sample via a prism and index matching fluid. The angle of incidence at the sample surface is slightly greater than the critical angle. A microscope with a 5X objective providing a large viewing field is focused on the sample surface over the point where the reflection is occurring. The eyepiece and camera lens magnifications are 10X and 5X, respectively. Any imperfections due to surface pits, subsurface fractures, inclusions, etc. in the illuminated area will be indicated by light entering the objective viewing field. For qualitative surface evaluation a Nomarski differential phase microscope was used.

Figures 7A and 7B show the comparative TIRM results of two different commercial optical flats and a float polished optical flat. The float polished sample has significantly less features than commercial flat type A and slightly more than commercial flat type B. The type B flat is the best fused silica flat commercially available. When these areas were viewed with a Nomarski microscope at the same magnification no surface features were visible. This indicates the majority of features were subsurface.

The High Resolution Scatter Measuring Instrument (HRSMI) at the Phillips Laboratory provided quantitative information of the surface and subsurface features of the samples. See Figure 8. A continuous wave helium-neon laser operating at a 630 nm wavelength with a .1 mm diameter spot size is incident

on the sample surface at Brewster's angle. A photodetector is rotated a constant distance from the sample about the incident point in the plane of incidence. The normalized intensity value, the solid angle of the detector, and the angle of measurement with respect to specular is used to calculate the bidirectional reflectance distribution function (BRDF) verses detector angle.

Figure 9 shows the average scatter levels of the three sample types measured with the HRSMI. One sample of each type was measured five times at one point, and the five measurements averaged. The peak at 110° is a result of the detector passing through the specular beam. A beam of p-polarized light was incident on the samples and p-polarized light was selected by the detector. This arrangement was used to highlight the subsurface features by reducing the surface reflectance. The specular beam is a result of the surface microroughness and the subsurface scattering sites. Separating these contributions is difficult. Also, there is no guarantee the detector will pass through the center of the specular beam for each sample. Therefore, the specular scatter levels do not give an indication of the surface and subsurface scatter levels. The float polished scatter level is an order of magnitude lower than commercial type A in the near specular angular range and within an order of magnitude across the entire angular range compared to commercial type B.

A combined effort with the Phillips Laboratory required the fabrication of fused silica substrates by float polishing for a thin-film coating study. The fabrication of these substrates will be discussed. A joint effort with IBM was conducted to investigate the feasibility of polishing Cu and polyimide flats for planarization purposes. A brief description of the joint effort will follow. Ductile grinding of fused silica and Zerodur was investigated. The advantage of ductile grinding is that no fracturing takes place during the removal of material. This removal mode does not produce subsurface damage. Therefore, after ductile removal of material the polishing phase of the fabrication process can be shorten since surface damage is the only damage that exists. A discussion of these results follows. An investigation into the cause of subsurface damage produced by float polishing will be addressed. It was discovered that an HF acid etch of float polished fused silica revealed subsurface damage.

**Substrate Fabrication:**

Twelve 1.5 inch diameter and six 2 inch diameter General Optic fused silica substrates were ground and polished for the coating study in conjunction with the Phillips Lab. The 2 inch diameter samples were made first. Three samples were optically contacted to a 6 inch diameter 1 inch thick fused silica plate. Optical contacting of the samples to the plate was used so in-process Total Internal Reflection Microscopy (TIRM) could be performed. Several conclusions were drawn from this mounting scheme. First, the plate thickness raised the drive point of the workpiece higher than normal thereby introducing instability in the floating characteristics. This was confirmed by the observation of the samples during polishing, and the eventual crash of one set of samples into the

lap. Second, it was difficult to optically contact the samples because General Optic polished surfaces exhibit marginal contacting results. And third, the mounting geometry of three samples did not facilitate constant floating conditions. With three samples mounted 120° from each other there is a fair amount of space between samples. It is perceived that a sample undergoes maximum lift when moving perpendicular to a lap groove. With this in mind it was realized that one sample is moving virtually parallel to the lap grooves while the other two have some component of its surfaces moving perpendicular to the groove. This would set up an unstable floating condition. In fact, the sample moving parallel to the groove could come in contact with the lap. Contact was evident by scratches seen on the lapping surface, and microgroove patterns on the outer edges of the samples after a polishing run. A new mounting scheme was adopted to facilitate more stable floating conditions. Four 2 inch samples were mounted with optical epoxy to a 6 inch diameter plate with a thickness of 3/8 inches. The samples were evenly spaced about a common circumference. The samples were ground with 2 μm synthetic diamond on a copper lap, float polished with $TiO_2$ for ~20 hours to remove grinding damage then float polished with colliodal 500 Å $SiO_2$ until optical profilometry and TIRM measurements indicated a well polished surface. The floating characteristics improved for this mounting scheme. Two sets of 1.5 inch diameter samples were then made. Six samples were epoxied to a 6 inch diameter 3/8 inch thick plate about a common circumference. The spacing between samples was kept to a minimum. The grinding and polishing parameters for the 2 inch samples were used on the 1.5 inch samples so that consistent surfaces would exist from set to set. Out of the six 2 inch samples fabricated, two did not undergo the same conditions as the four mounted together. These two were the best of the three polished with the

former mounting scheme. The best samples were determined by optical profilometry and TIRM. These characteristics were close to the four polished samples on one plate.

**IBM Joint Effort:**

IBM Research at Yorktown Heights, New York expressed interest in a joint effort concerning float polishing of copper and polyimides. During a two week period Dr. Jeffery Carr of IBM, and UNM members conducted studies on the feasibility of float polishing these materials. The preliminary results indicated that float polishing reduced the surface roughness in regions where scratching did not occur. The scratching was believed to be caused by agglomerated polishing particles. The copper polished best on a nylon lap and the polyimide polished best on a tin lap. Dr. Carr was pleased with the initial results and ordered a float polishing machine from Toyoda Machine Works of Japan.

**Hydrodynamic Modelling:**

A first order hydrodynamic model was delivered by Dr. Randy Truman of the Mechanical Engineering Department at UNM. The model stated that maximum lift would occur with microgroove depths of ~1 μm. All polishing had been performed with 50 μm deep microgrooves according to Dr. Namba's recommendation. When 10 μm grooves were cut the amount of scratching increased on the sample and lapping surfaces. It was not possible to cut a shallower groove due to the accuracy of the tool mount. With the original groove depths (50 μm) the model stated that floating should not occur. It was realized that concave cusps ~.1 μm deep result from the final facing cut with a 3 mm radius diamond tool. These cusps could be the key geometry of the lap to

provide floating. In any case the microgroove depth was increase to 50 μm and the scratching on the sample and lapping surfaces decreased compared to the 10 μm grooves. It is believed that the deeper grooves allow slurry and removed material to be carried away from the work surface without contacting the sample and lapping surfaces. The lapping surface is currently being cut such that at least two full cusps exist between microgrooves.

Ductile Grinding:

Five 2 inch diameter General Optic fused silica samples were mounted with optical epoxy to a 6 inch diameter 3/8 inch thick fused silica plate in a circular arrangement. A 1 inch diameter sample was mounted in the center of the arrangement to reduce the amount of space between samples. Five 1.75 inch diameter Zerodur samples were mounted in the same arrangement as the five 2 inch diameter fused silica samples using optical epoxy. During the entire grinding process of the Zerodur samples a predominantly ductile mode of removal was evident up to the greatest possible pressure of 350 g/cm$^2$. Similar pressures for fused silica exhibited a brittle grinding mode. This suggested that the chemistry of the two glass may influence the mode of grinding. Presently, removal rates for fused silica in the ductile mode is virtually zero. It is accepted that a smearing of the fused silica is occurring, namely a removal and redeposition of material. On the other hand, composite glasses have appreciable removal rates in the ductile mode. The solution to achieving workable removal rates during ductile grinding of fused silica is to introduce a carrier or binding agent that would prevent redeposition. Several carriers were tried; ceria, zirconia, titania, and a mixture of ceria and zirconia. These compounds were chosen because of their ability to polish glass. No significant removal rates were

achieved with these materials when mixed with 2 μm polycrystalline diamond. It was decided to charge the lap with Zerodur to see if its chemistry could act as a carrier agent. This was achieve by simultaneously grinding fused silica and Zerodur on the same lap. Preliminary results indicated that a removal rate of ~2 μm/hr was obtained for fused silica in the ductile mode.

It was also noted that the diamond slurry concentration plays a significant role in the fracture mode of fused silica. Figure 1A is a plot of the rms roughness vs. slurry concentration. Brittle fractures sites will produce higher roughness values than ductile scratches. Note that the error bars overlap in the minimum region. However, it is believed that a minimum roughness will exist for a specific slurry concentration because of the following reasons. At a concentration less than the the minimum roughness the load experienced by each diamond particle is large which exerts a great enough localized pressure to exceed the fracture point of the glass. For concentrations greater than the minimum roughness diamond particles have saturated the surface area of the lap and extra particles lie on top of the first layer of particles. These extra particles do not penetrate the copper lap and are therefore free to tumbles o shearing for ductile removal can occur under these conditions. The same situation of increased localized pressure also exists. As the concentration continues to increase the amount of brittle fractures increase until, in this case, the removal is almost entirely in the brittle mode. The concentration that produces the minimum roughness is when the lapping surface is in a saturated state. Any other state promotes increases brittle fracture. It was at the concentration for minimal roughness that the carrier agent study was performed on fused silica.

Subsurface Damage:

A float polished sample was etched in 10% HF for 5 minutes to reveal the amount of subsurface damage. A disturbingly large amount of damage was evident. This damage was not revealed by TIRM. The etching of a General Optic sample showed very little damage. Two types of subsurface damage may exist; cracks and inclusions, and residual stress due to the healing of cracks. Since TIRM did not indicate the amount of subsurface damage revealed by etching, the sensitivity of the diagnostic is in question. To determine the sensitivity a study is being conducted to compare the results of TIRM and the scatterometer of the Phillips Lab at common points on a float polished and General Optic polished fused silica sample. These samples have been laser marked with crosshairs one inch apart to serve as reference points. By using a vernier stage to position the samples with respect to the crosshairs similar areas can be measured on the optic with different diagnostic techniques. Five points on each samples was characterized with TIRM and scatterometry. The samples are being etched at 2 minute intervals (~ 130 nm removal) and characterized with TIRM at the five points. If damage sites are exposed in these measured areas by etching that was not seen initially with TIRM, the scatterometry measurements should indicate whether the damage was subsurface cracks (scattering sites) or residual stress. It was decided to investigate the cause of this damage to float polished optics. The first aim was to determine what step of the polishing process produced the damage. The five 2 inch diameter General Optic fused silica samples that were optically contacted to a 6 inch fused silica plate were used for this study. The samples were polished with $TiO_2$ slurry to remove grinding damage observable with a Nomarski microscope. The samples were acid etched to reveal any damage, then polished again to see if the exposed damage could be removed.

This was done because etching relieves residual stress, and opens up and terminates subsurface cracks. After several polish-etch procedures the following observations were made. Several distinct damage sites opened up by etching were monitored for size and depth changes. The depth of a damage site was determined by focusing first on the surface then on the bottom of the crack with a 100 X objective (depth of focus = .5 μm) and noting the change in height with the vernier on the microscope stage (2 μm divisions). The initial deepest depth of one monitored fracture site was 24 μm. Subsequent polishing reduced this depth to 5 μm. However, the damage was never removed. This suggests that $TiO_2$ slurry can remove damage only to a certain depth and thereafter propagates the damage. As the polishing time increased a crazed film developed on the lapping surface filling in the microgrooves, and the removal rate dropped to zero. There is a strong correlation between these two behaviors. It seems that the microgrooves play an important role in the removal process. The lapping surface had to be faced and grooved periodically to obtain appreciable removal rates. This behavior had not been observed previously. Because of the deposition problem with $TiO_2$ no further polishing was conducted with this slurry.

A 3% wt. $SiO_2$ slurry consisting of 800 Å colliodal particles was investigated. This was a commercial product manufactured by Fujimi Corp. The pH of this slurry was 10.4 as opposed to a $TiO_2$ slurry pH of 4. After polishing for 4 hours the lap showed no residue but the damage to the surface of the samples increased dramatically. The next polishing run was conducted at a lower pressure (43 vs. 56 $g/cm^2$) and the damage sites reduced significantly. The monitored damage site depth did not change. After 20 hours of total polishing time the samples decontacted from the plate during a polishing run and severe

damage occurred to the lap and samples. Five 2 inch diameter General Optic fused silica samples were mounted to a 6 inch diameter 3/8 inch thick fused silica plate with optical epoxy in a circular arrangement. A 1 inch diameter General Optic fused silica sample was mounted in the center of the arrangement to further reduce the spacing between samples. This was the set of samples used previously for the ductile grinding experiment.

A paper authored by Dr. Aleta Tesar of LLNL reported results from a polishing study were pH was varied. The outcome of the experiment showed that conventional polishing of fused silica with a ceria slurry at pH 4 gave the smoothest surfaces. It so happens that the isoelectric point of fused silica (the pH at which zero charges exists on the surface) occurs at pH 4. This suggests that polishing at the isoelectric point of a material can improve the surface roughness because redeposition of material cannot occur due to electrostatic attraction. This approach was adopted for float polishing. Colloidal slurries of ceria were procured from Solution Technology, Inc. with submicron size particles. The mean size of the particles was .3 μm. This size is currently being verified by Leeds and Northrop, Inc. using a doppler effect particle size distribution device. The first ceria slurry used had a pH of ~ 9. After a standard polishing run of 7 hours, the samples showed no polishing damage as observed with a Nomarski microscope. The removal rate at 18 g/cm$^2$ was in the neighborhood of 1 μm/hr with 4.5 Å rms surfaces measured with an optical profilometer. A surface roughness of 10 Å rms was measured with an atomic force microscope (AFM). The difference in the two measurements is due to their bandwidths. The AFM images revealed a surface with some random scratches and a slight texture. To date these are the best surfaces achieved with the corresponding removal rate. Comparable removal rates were obtained with titania at ten times the pressure

but the surfaces were five times as rough. Collodial ceria at pH 4 was obtained from Solution Technology but these slurries had agglomeration problems. Because of these problems the polishing results were inconsistent.

A set of six fused silica samples were mounted for grinding and polishing. Colloidal silica, ceria, and zirconia were investigated to characterize the surface quality of the fused silica samples. During this study is was noticed that the viscosity of the polishing slurry play an important role in the surface quality. A viscosity measuring instrument was obtained to quantify the viscosity of the polishing slurries. A lap pattern was always evident with the zirconia slurry. However, the surface roughness measured at the smooth areas of the zirconia polished surfaces was less than the ceria polished surfaces. The ceria polished surfaces exhibited much less lap pattern features than the zirconia. A slurry of Nalco 2360 colloidal silica produced the worst surfaces of the three polishing compounds. It is believed that the low viscosity of the colloidal silica was the cause of this damage.

During this polishing compound study the pH of the slurries was varied (by the manufacturer). It was discovered that a basic zirconia slurry did not remove material whereas an acidic zirconia slurry did. Changing the percent weight of the slurry effectively changed the viscosity of the slurry. Because a lap pattern was always evident with the zirconia slurry, a percent weight study with ceria was conducted. This study showed a best surface of 5 Å rms roughness being obtained with a 2% wt. ceria slurry with a pH of 9. The study was put on hold in order to investigate the polishing of KD*P.

The polishing of KD*P for Lawrence Livermore National Laboratory (LLNL) was initiated due to the possible funding available. The KD*P crystals are used for frequency doubling in the Nova Laser at LLNL. The problem with these crystals is that the surfaces are diamond turned. This leaves

tooling marks behind after facing that behave like a grating when a laser beam is incident on the surface of the crystal. Hence, the focusing of the laser beam after passing through the crystal does not concentrate all of the beam energy at a desired point. Since high energy densities are required for the Nova Project, a smooth KD*P crystal surface with good figure is required.

The polishing of these crystals has been attempted by LLNL. They have achieved surfaces with 5 Å rms roughness but streaks are always left behind. The streaks are most likely a result of the contact nature of the polishing process. This prompted us to investigate the float polishing of KD*P. The difficulty with the polishing of this crystal is the softness of the material (2.5 Moh hardness; fused silica has a Moh hardness of 7), and the fact that the material is water soluble. Several polishing fluids were tried.

The one recommended by LLNL was silicone oil. The studies done with the different slurries on fused silica indicated that a kinematic viscosity of 10 centistokes produced the best surfaces. Therefore, five gallons of 10 centistoke silicone oil was purchased. However, this fluid did not produce any removal when mixed with Linde A and Linda B polishing powder (.3 μm and .05 μm alumina). Plus silicone oil is impossible to remove by chemical means. So cleaning of the crystal surfaces to remove the fluid was not possible. Previous literature recommended the use of ethylene glycol (EG) with Linde A polishing powder. This was investigated.

### Polishing of KD*P

The EG by itself was found to dissolve the crystal. It was discovered that EG is hydroscopic. The water picked up from the atmosphere would dissolve the crystal. When the crystal dissolves one of the products is KOH. The KOH reacts with the EG to form a sub EG and water. This results in a

runaway situation. The result was confirmed by measuring the material removal of the crystal in EG over time. The removal rate increased with time. Therefore it was necessary to find a fluid that could be chemically removed from the crystal surface and have low hydroscopic properties. One such fluid is polypropylene glycol (PPG).

The PPG was found to be a suitable fluid for polishing when Linde B polishing powder was added. This combination produced solely a mechanical removal of material. It has been accepted that to achieve optically smooth surfaces there must be a balance between the mechanical and chemical removal mechanisms. To promote a chemo-mechanical removal of KD*P, a study was done where EG was added in controlled amounts. Without the EG the removal rate of the PPG slurry was .5 μm/hr. Set amounts of EG were added to this slurry to obtain a chemo-mechanical removal rate of 1 μm/hr. This removal rate has been found to produce high quality optical surfaces. Initially, three 2 inch square crystal samples were symmetrically mounted on a six inch diameter fused silica plate. However, this mounting geometry does not facilitate stable floating conditions. Consequently, the samples were momentarily touching the lap thereby causing damage. Four crystal samples will be mounted to obtain a more continuous work surface thus promoting a more stable floating condition. Another material being tested for float polishing is titanium doped sapphire.

### Polishing of Titanium Doped Sapphire

The Crystal Division of Union Carbide was contacted to see if they would be interested in evaluating float polished titanium doped sapphire. One use of this type of crystal is a laser gain medium. Union Carbide expressed an interest in the float polishing method to produce superior crystal

surfaces. Twenty one inch diameter samples of titanium doped sapphire have been procured from Union Carbide. The samples will be beveled, ground, and float polished to obtain the best possible surfaces.

## Conclusions

The efforts put forth the past three years have yielded valuable information. The float polishing process has been shown to produce optical quality surfaces. Much of the emphasis of this effort was to quantify and characterize the parameters involved with the float polishing process. Studies in the areas of hydrodynamics, kinetics, and slurry chemistries has led to much insight to the polishing process. A more scientific approach has dispelled some to the black art associated with the fabrication of optical surfaces. In addition, a technique for identifying subsurface damage by a nondestructive method was developed during this effort. This method allows the viewer to image subsurface features through a microscope. The technique, known as total internal reflection microscopy (TIRM), has been used in the past to image surface features of optically transparent materials. Other nondestructive methods such as scatterometry can detect subsurface damage but the results are more difficult to interpret and does not provide an image of the damage site. Consequently, the TIRM technique for identifying subsurface damage of polished substrates proved to be a fast and reliable method that played an important role in this effort.

The potential of the float polishing process has not yet been realized. This is the reason for the continued effort with materials other than fused silica. The experience and knowledge gained from this effort has given us the ability to approach polishing problems in a scientific manner rather than a trial and error method. It is our goal to continue the quest for optically

superior surfaces of different materials using the float polishing technique through a scientific approach.

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1. A. C. Tam, "Applications of Photoacoustic Sensing Techniques," Rev. Mod. Phys., 58 (2), 381 (1986).

2. M. Y. A. Raja, D. W. Reicher, S. R. J. Bruech, J. R. McNeil, D. I. Oates, "High Sensitivity Surface-Photoacoustic Spectroscopy," Optics Letters, 15 (1), 66 (1990).

## Publications

1. "Float-Polishing Process and Analysis of Float Polished Quartz," Optical Society of America, Optical Technology Issue, 32 (1993).

2. "Subsurface Damage Identification in Optically Transparent Material using a Total Internal Reflection Technique," Optical Society of America, Optical Technology Issue, to be published.

## *Conference Presentations*

1. "Float Polished Quartz Substrates," Science of Optical Finishing, June 1990.

2. "Fabrication of Optical Surfaces with Low Subsurface Damage Using a Float Polishing Technique," Laser Damage Symposium, October 1991.

3. "A Non-destructive Method of Assessing Subsurface Damage in Optically Transparent Materials," Society of Precision Engineers, April 1993.

# DIAMOND LAPPING SCHEMATIC

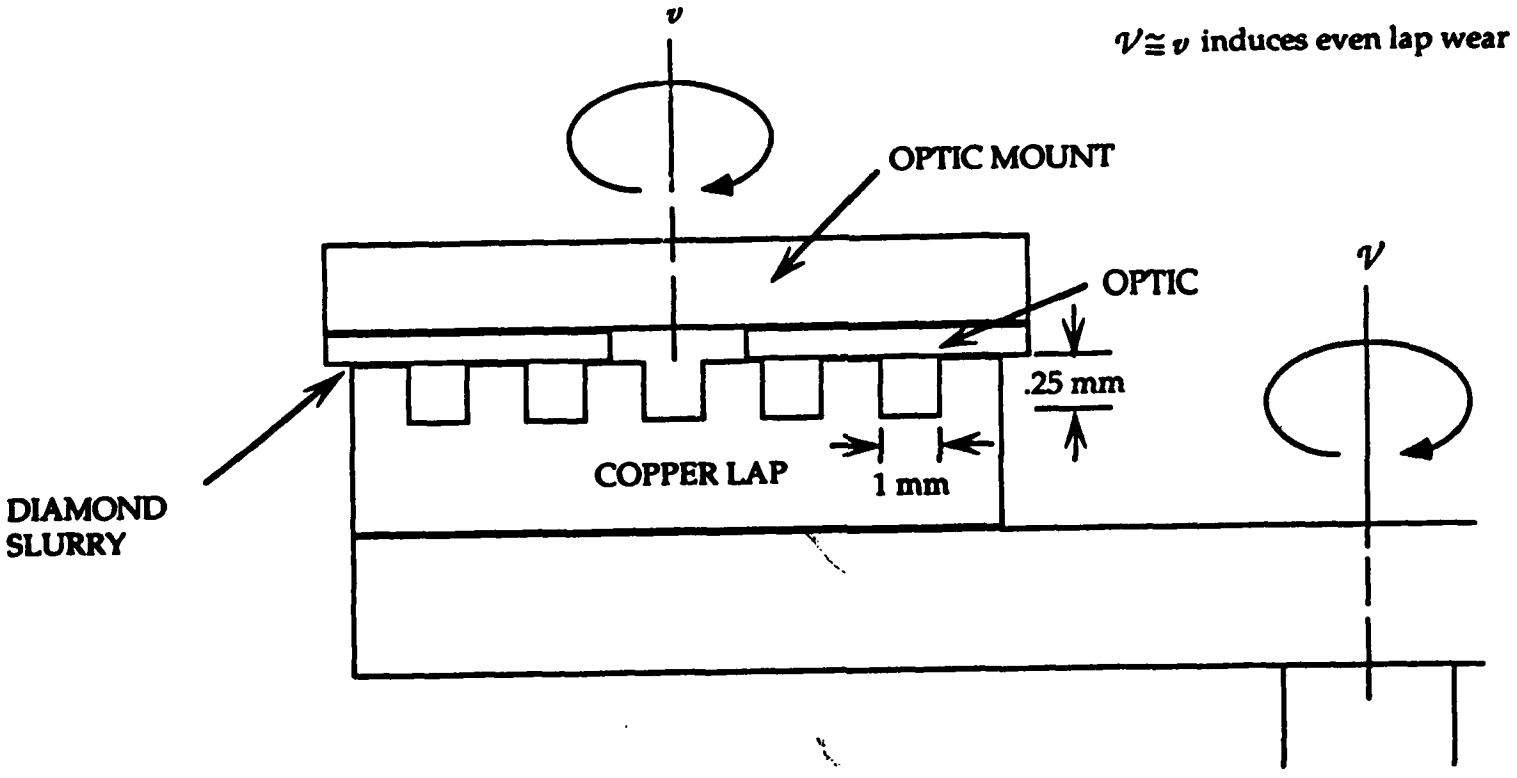
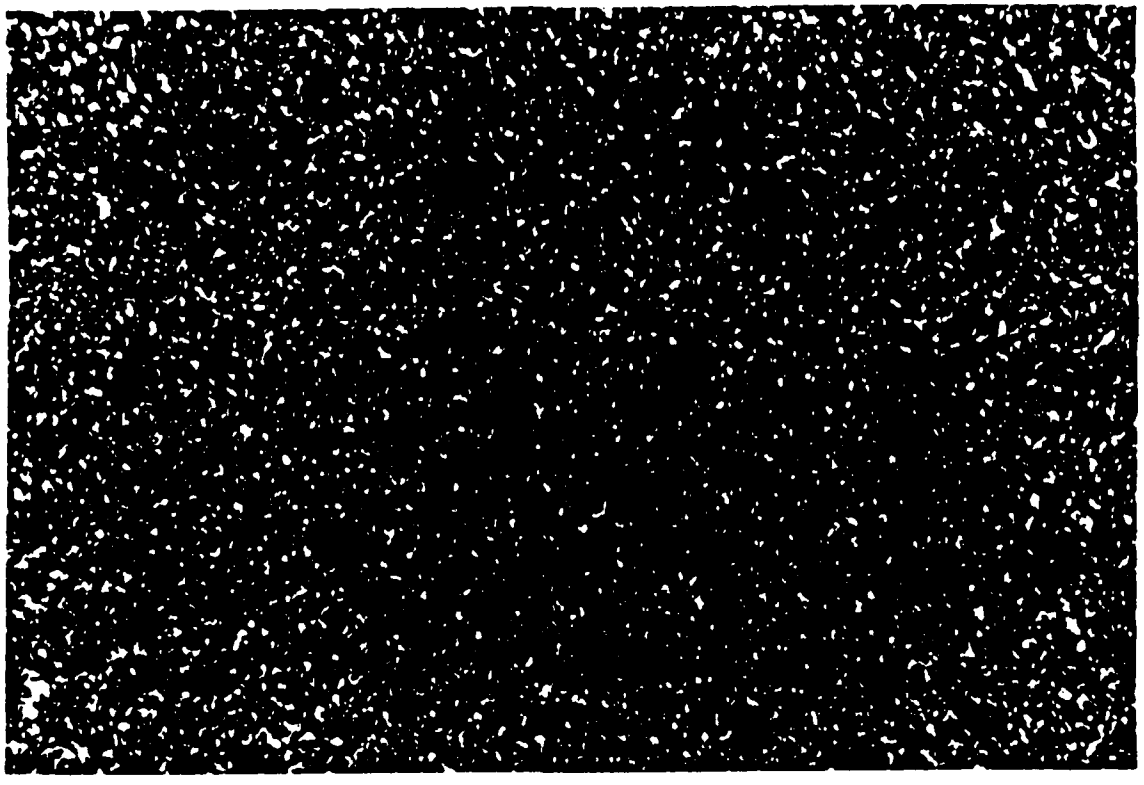



**Figure 1**

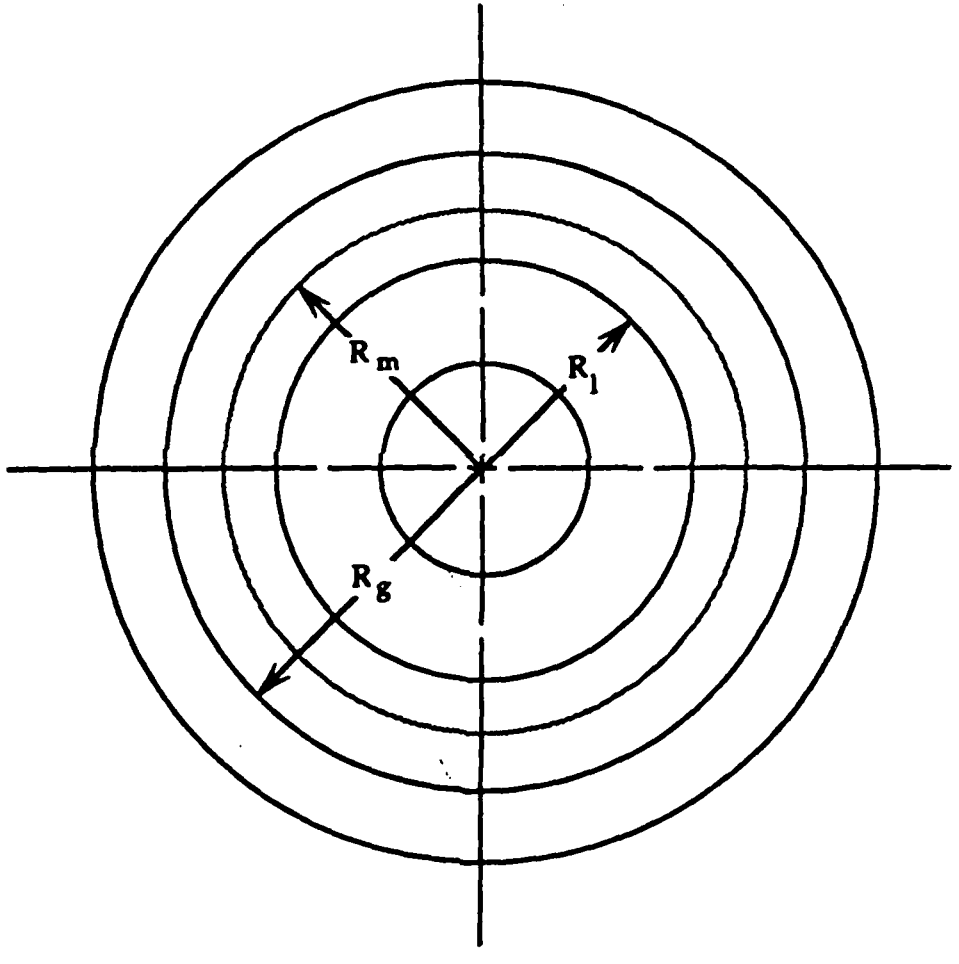
NOMARSKI MICROGRAPH
2 μm POLYCRYSTALLINE DIAMOND GRIND
400 X



**Brittle Fractures**

**Figure 2**

**GROOVE-LAND PATTERN**
**FOR**
**DIAMOND LAPPING**




$$|R_g - R_m| = |R_m - R_l|$$

**FIGURE A**

## THE ZETA POTENTIAL

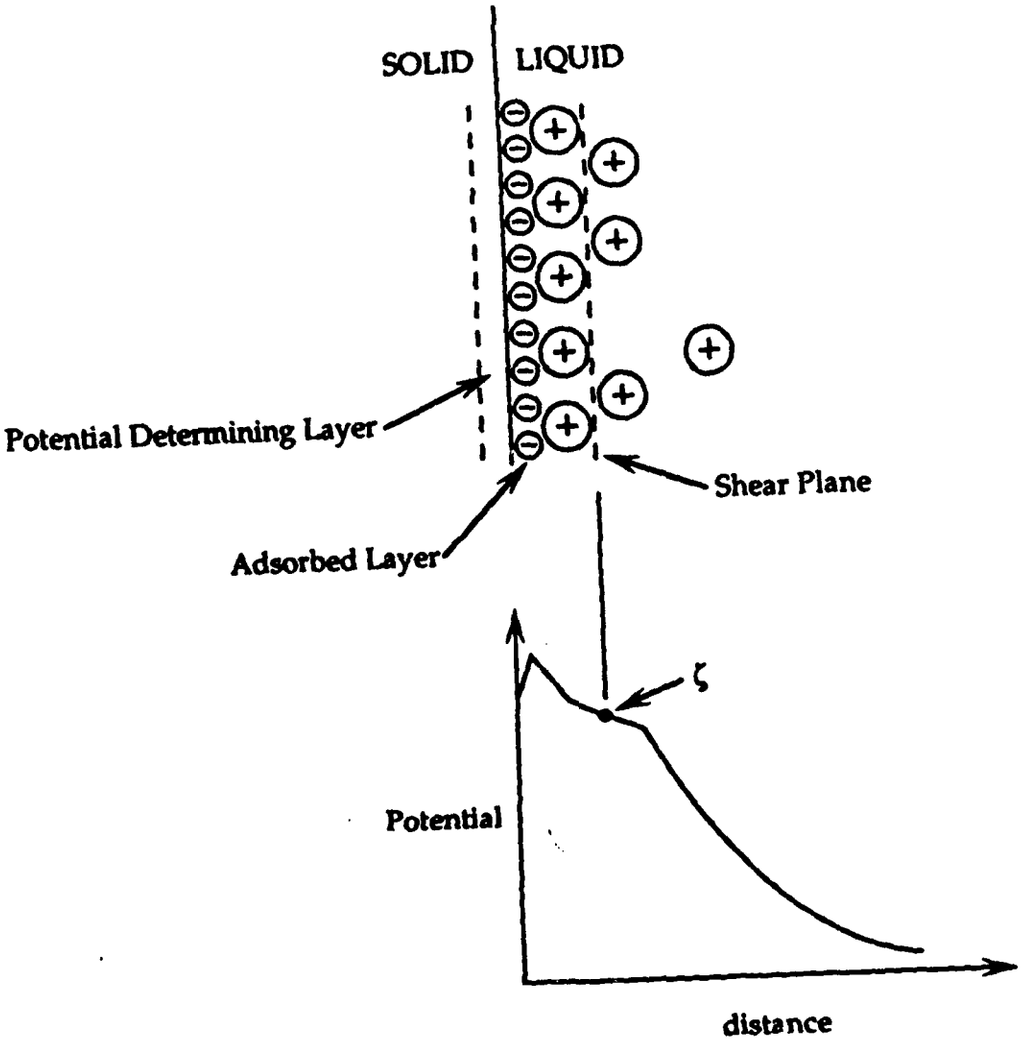



Figure 3

# GRINDING MODE EXPERIMENT

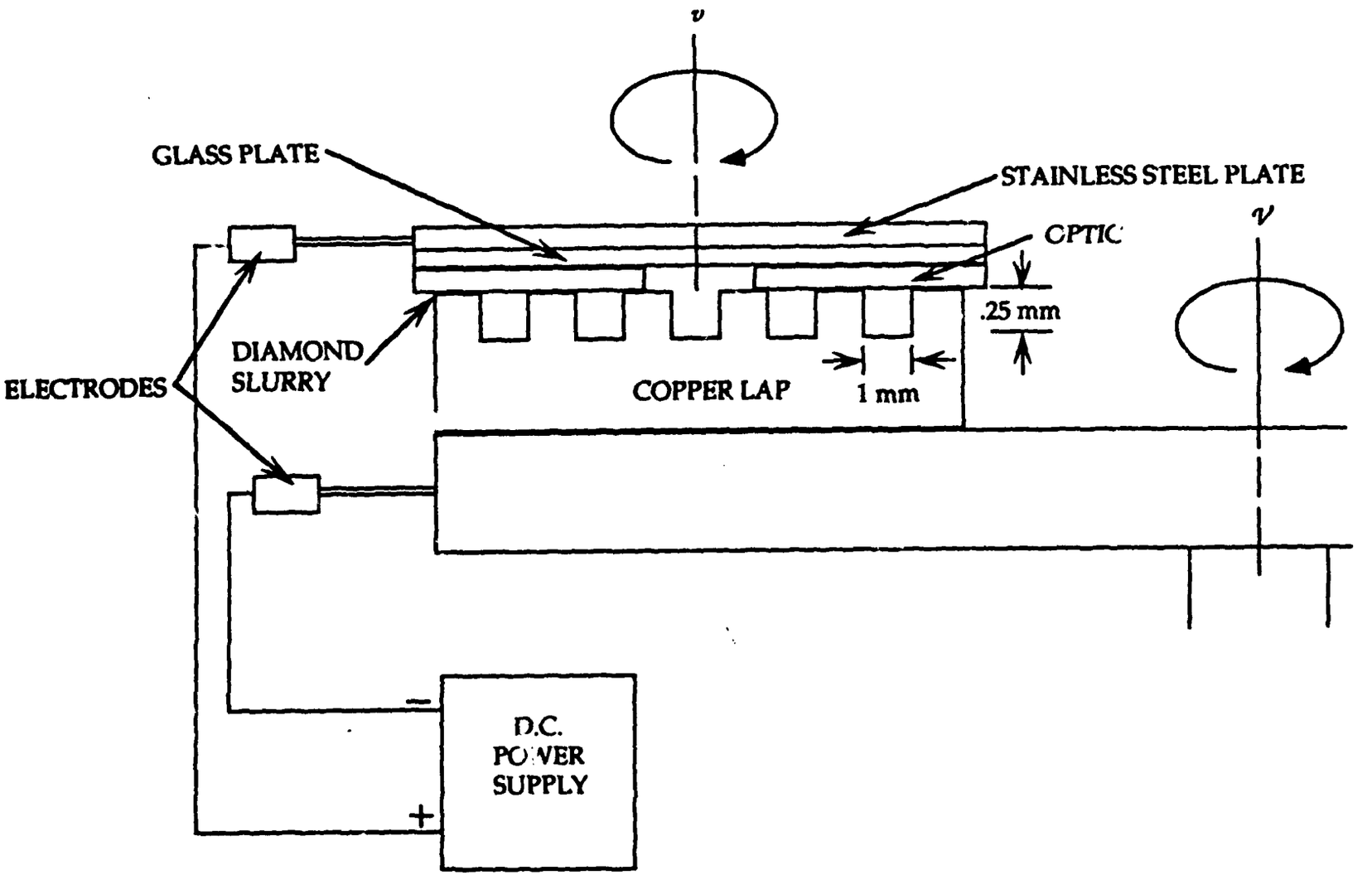



Figure 4

# FLOAT POLISHING SCHEMATIC

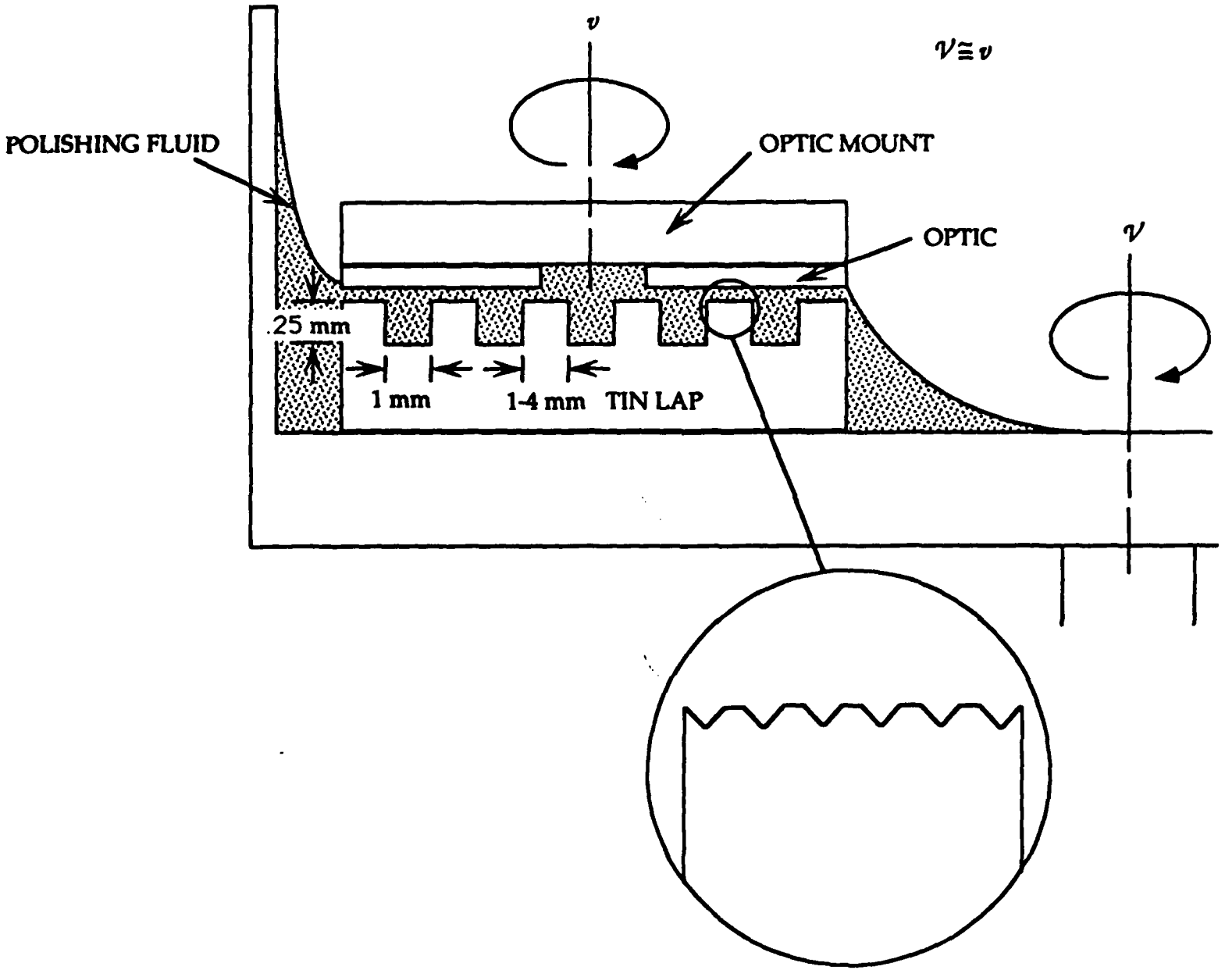
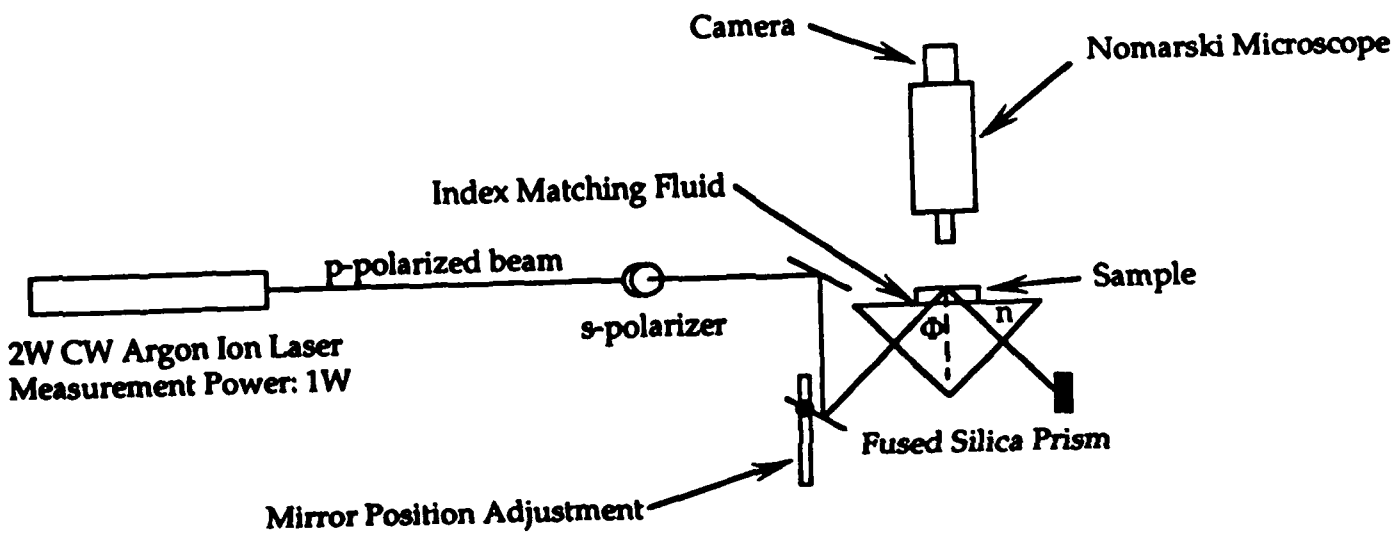



Figure 5

# TOTAL INTERNAL REFLCTION MICROSCOPY




The phase change Δ upon reflection at an angle of incidence Φ, and sample index of refraction n.

$$\tan\left(\frac{\Delta}{2}\right) = n\frac{\sqrt{n^2\sin^2\Phi - 1}}{\cos\Phi} \quad \text{(p polarization)}$$

$$\tan\left(\frac{\Delta}{2}\right) = \frac{\sqrt{n^2\sin^2\Phi - 1}}{n\cos\Phi} \quad \text{(s polarization)}$$

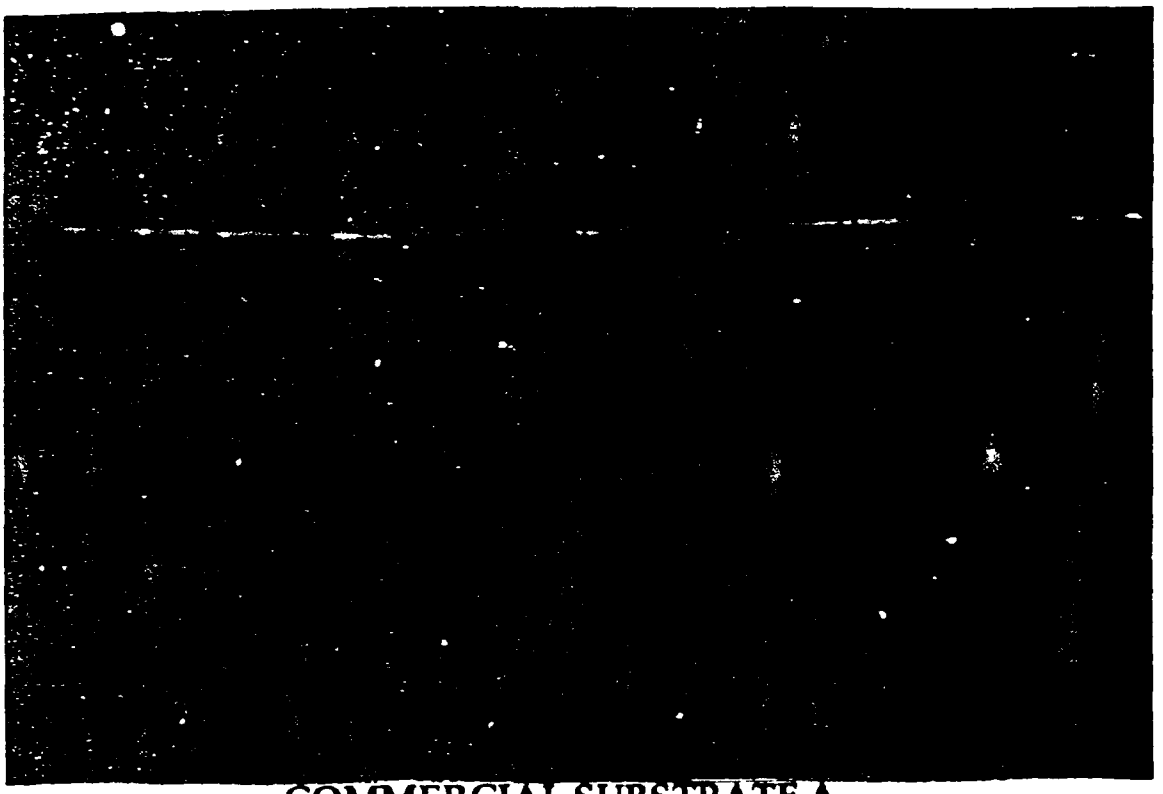
The nodal and antinodal locations l below the surface. $\lambda_o$ is the free space wavelength. Setting δ equal to 0, π, 2π, 3π,... determines the nodal and antinodal locations.

$$l(\delta) = (\delta - \Delta)\left(\frac{\lambda_o}{4\pi n\cos\Phi}\right)$$

Developed by Paul Temple, NWC 1979

**Figure 6**

TOTAL INTERNAL REFLECTION MICROSCOPY
25 X



COMMERCIAL SUBSTRATE A

COMMERCIAL SUBSTRATE B

FIGURE 7A

TOTAL INTERNAL REFLECTION MICROSCOPY
25 X

FLOAT POLISHED SUBSTRATE

FIGURE 7B

# High Resolution Scatter Measuring Instrument

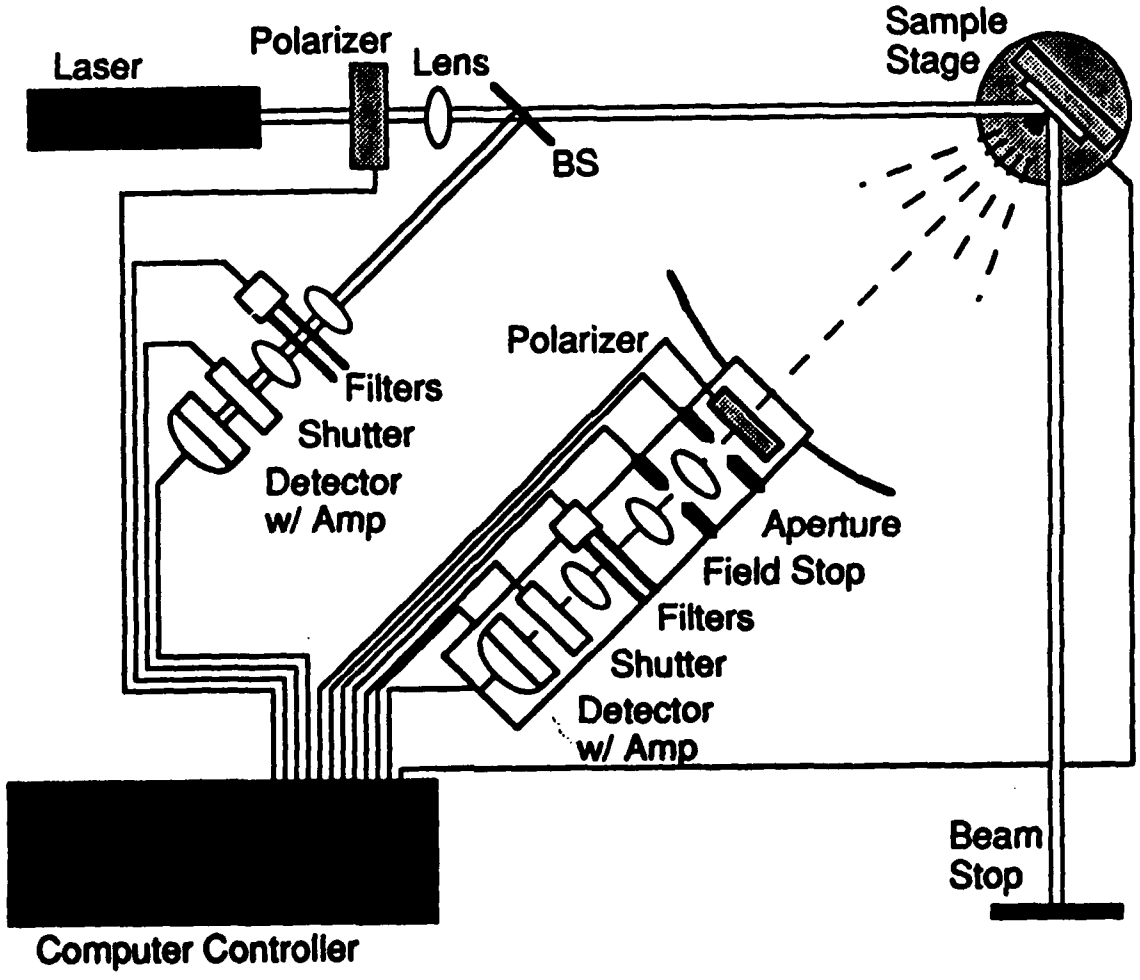



Figure 8

# BRDF (BREWSTER'S ANGLE) OF UNCOATED SUBSTRATES

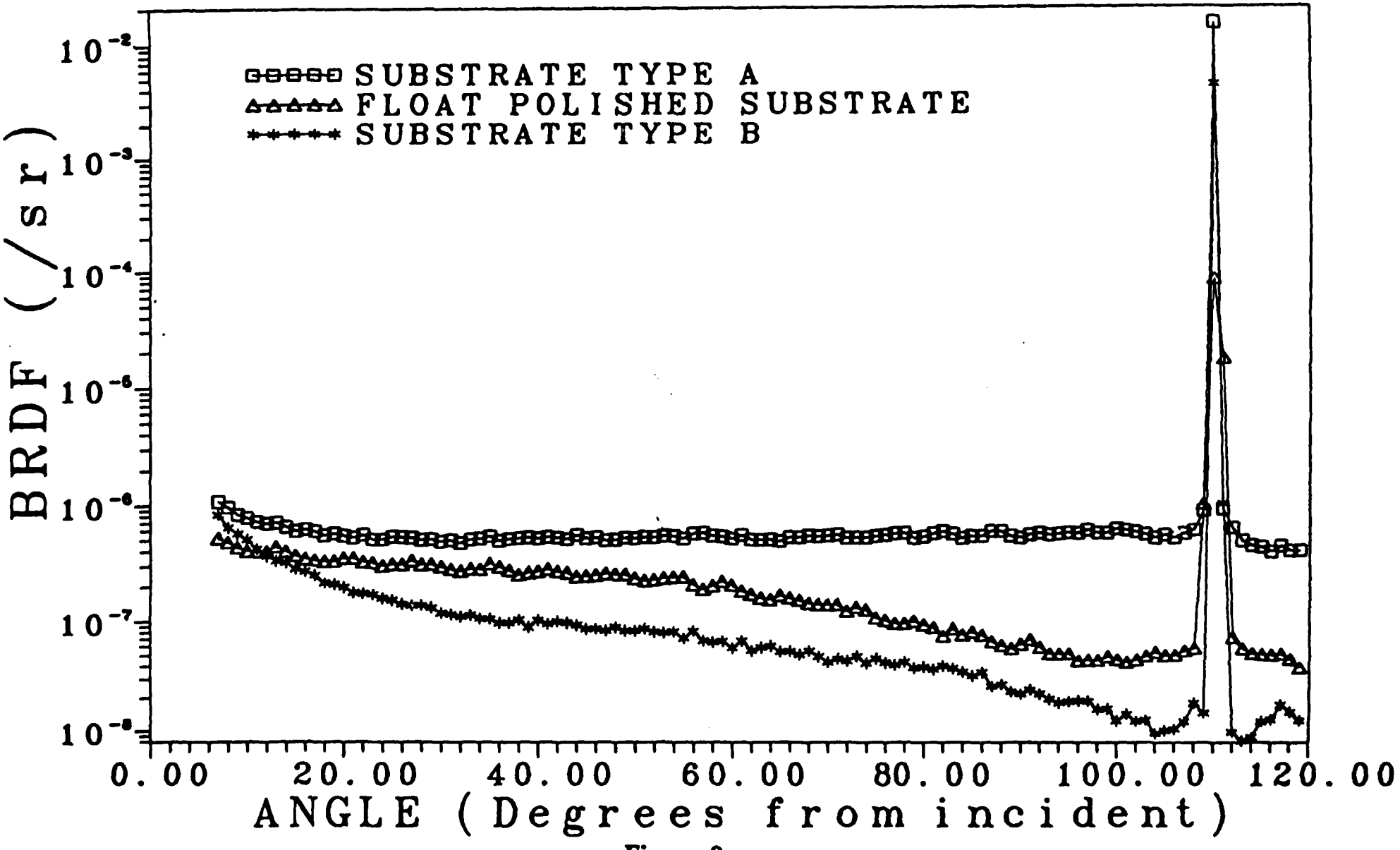



Figure 9

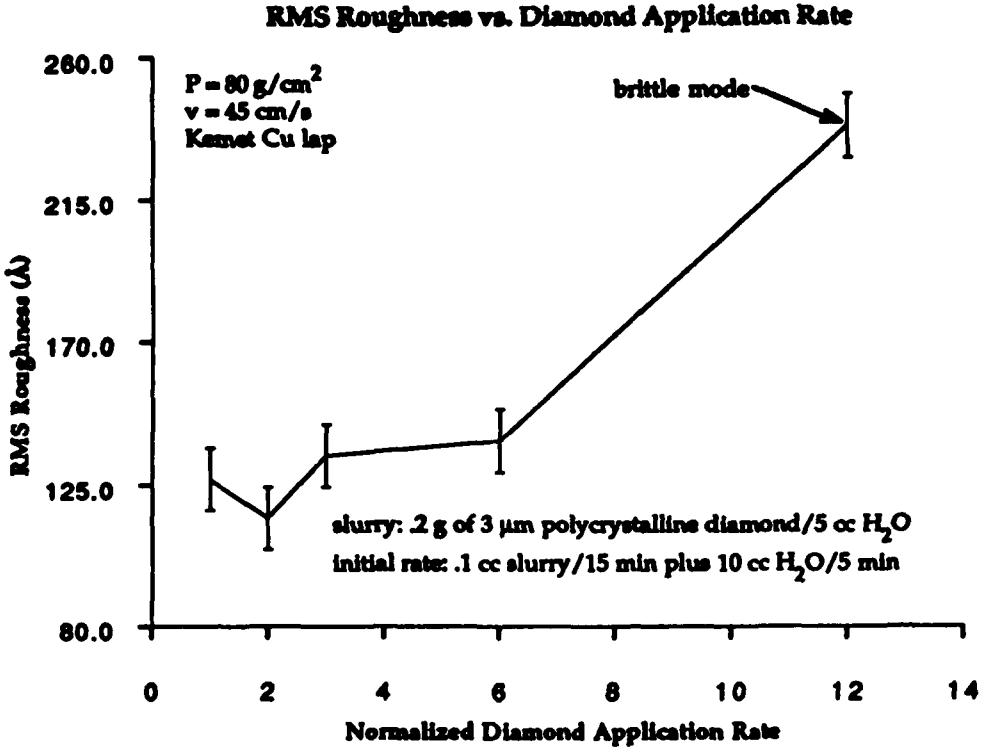

RMS Roughness vs. Diamond Application Rate
P = 80 g/cm2
v = 45 cm/s
Kemet Cu lap
brittle mode
slurry: .2 g of 3 μm polycrystalline diamond/5 cc H2O
initial rate: .1 cc slurry/15 min plus 10 cc H2O/5 min
RMS Roughness (Å)
260.0
215.0
170.0
125.0
80.0
0
2
4
6
8
10
12
14
Normalized Diamond Application Rate

Figure 1A

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1. A. C. Tam, "Applications of Photoacoustic Sensing Techniques," Rev. Mod. Phys., 58 (2), 381 (1986).

2. M. Y. A. Raja, D. W. Reicher, S. R. J. Bruech, J. R. McNeil, D. I. Oates, "High Sensitivity Surface-Photoacoustic Spectroscopy," Optics Letters, 15 (1), 66 (1990).

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