UCRL--95882

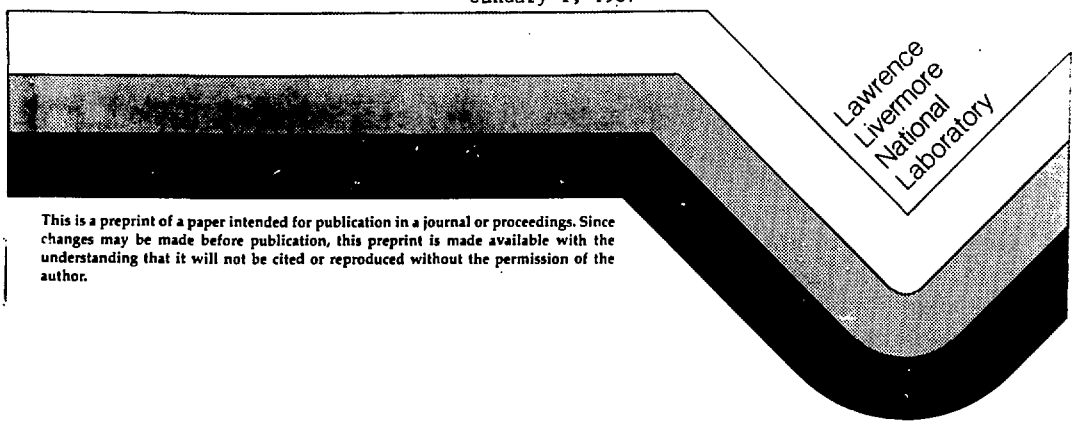
DE87 005055

Development of Polishing Methods for Chemical Vapor Deposited Silicon Carbide Mirrors for Synchrotron Radiation

Baruch A. Fuchs
Norman J. Brown

Optical Society of America
Seattle, Washington
October 20-24, 1986

January 1, 1987



DISCLAIMER

Development of Polishing Methods for Chemical Vapor Deposited Silicon Carbide Mirrors for Synchrotron Radiation

B. A. Fuchs
N. J. Brown

October 17, 1986

Abstract

Material properties of Chemical Vapor Deposited Silicon Carbide (CVD SiC) make it ideal for use in mirrors for synchrotron radiation experiments. We developed methods to grind and polish flat samples of CVD SiC down to measured surface roughness values as low as 1.1 Angstroms rms. We describe the processing details, including observations we made during trial runs with alternative processing recipes. We conclude that pitch polishing using progressively finer diamond abrasive, augmented with specific water based lubricants and additives, produces superior results. Using methods based on these results, a cylindrical and a toroidal mirror, each about 100 x 300mm, were respectively finished by Continental Optical and Frank Cooke, Incorporated. WYCO Interferometry shows these mirrors have surface roughness less than 5.7 Angstroms rms. These mirrors have been installed on the LLNL/UC X-ray Calibration and Standards Facility at the Stanford Synchrotron Radiation Laboratory.

Introduction

For very high energy density x-ray beam reflectors, such as used in synchrotron radiation experiments, silicon carbide appears to be an extremely attractive candidate material. It has a very high melting point, is chemically inert, has a high thermal conductivity, and a low coefficient of thermal expansion. It's ratio of thermal conductivity to thermal expansion coefficient, the principle figure of merit for such applications, is almost

*This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

three times that of copper and 30 times that of fused silica (see Figure 1). In the form produced by chemical vapor deposition, it has extremely low porosity and appears quite homogeneous. Its density and hardness are such that it can be polished to very low roughness values.

We have recently received three specimens of CVD silicon carbide from Dr. Richard Gentilman of the Raytheon Company. The specimens were 5 cm diameter flats about 4 mm thick. The mandrel side, which Dr. Gentilman suggested was the more homogeneous, was marred by hemispherical bubbles, the largest of which was about 150 micrometers diameter. Using this material we prepared a number of specimens and conducted a series of grinding and polishing runs aimed at producing a flat surface with minimal roughness.

Blanchard Grinding

We Blanchard ground the mandrel surface after epoxy mounting the specimens on other silicon carbide blanks that were approximately 7 mm thick. The Blanchard coolant was water with "Rust-Lick" added.

The ground surfaces were quite interesting. Portions appeared near specular to the naked eye. Under the Nomarski microscope these seemed mottled with a raised film that had the appearance of melted wax deposits. Some of these patches were quite extensive. The very small fleck seen in Figure 2 was selected to illustrate these, since the Nomaraski shadowing clearly show it is raised above the surrounding region. We suspect from other experience that this film is a repolymerized hydrolized silica, but we made no other measurements to verify this. It did lead us to speculate that hydrolysis could be involved and that aqueous chemistry effects might contribute to surface processing. (Surface chemistry can be quite different from bulk chemistry).

Free Abrasive Grinding

Next, the samples were free abrasive ground. For this step we selected a copper Kemet lap, and 9 micron diamond abrasive with 20% ethylene glycol and water as a lubricant. We wanted a soft lap material to minimize depth of cracking in this very hard material. We removed 40 micrometers. The surface under the Nomarski microscope seemed very fine ground, a collection of random pits, a typical free abrasive ground surface (see Figure 3). The Blanchard operation had given a half micrometer background with isolated pits about 1.5 to 2 micrometers in depth. The 9 micrometer free abrasive grind gave a very uniform 1.0-1.3 micrometer peak-to-valley rough surface, leading us to suspect 5 to 7 micrometers of subsurface damage.

Consequently, we removed an additional 13 micrometers on a Kemet lap with 3.0 micrometer diamond. This surface was quite different from that produced by the 9 micrometer diamond (see Figure 4). This was essentially a surface of very clean crisp scratches with very few pits, possibly the residue from the earlier free abrasive grind. We are far from certain as to the cause of the change in behavior. One possibility is that we exceeded the critical pressure described by Tsesnek[1], thus preventing the rolling of particulants. Another possibility is that we entered another region of surface mechanics that we have encountered elsewhere. We are very near a critical threshold region on both counts.

Initial Pitch Polishing Runs

At this point we began pitch polishing. The indications of aqueous chemistry effects led us to try pitch with an aqueous coolant (20% ethylene glycol in water). For the primary abrasive we tried diamond, which had been shown to score by plowing in the last grind. In some of the runs we also attempted to activate additional surface chemistry, by adding small amounts of silica or alumina to the diamond. We selected half micrometer synthetic

diamond, which we knew would deeply penetrate the pitch, exposing 10 to 20% of its height above the surface for most of the polishing. This would leave a part to lap gap of 0.1 to 0.05 micrometers. Thus, we would be restricted to colloidal silica or 0.05 micrometer gamma alumina, neither of which would penetrate the pitch.

In our first runs we used only diamond. Initially the surface was removed rapidly. We quickly obtained a surface in the 4-6 Angstroms rms vicinity but it had numerous small pits and traces of scratches. Subsequent runs were quite different. Not only did we not improve the surface, but weighings capable of detecting 200-400 Angstroms of surface removal showed no perceptible material removed.

We suspected that dulling of the diamond and loss of "tooth" was degrading material removal. We have encountered this before when diamond was used with carbide formers. While the surface is rough, even dull diamond can find sufficient purchase to permit shear. Once this roughness is removed however, we believe carbide transformations reduce the sharp edges sufficiently to preclude a grip necessary for shearing. We tried soap additions to provide the wetting necessary to cool the diamond to prevent this supposed transformation. However, this produced no effect.

Next, we decided to try inducing chemical effects, adding first colloidal silica and, on a subsequent run, the alumina referred to earlier. The silica produced a surface that appeared rough due to a smeared deposit, and weighings showed nearly a thousand Angstroms of deposit (see Figure 5). We suspected the deposit was silica and wondered if the possibility existed for a surface sealing phenomenon. Just as plastic is used to seal a pitted but otherwise smooth granite, permitting a smooth essentially granite surface to be obtained by mild abrasion with steel wool, so we felt we might be able to remove the silica with ceria. But the ceria produced no perceptible wear on this surface. Fine finger-like structures were clearly visible after the run just as before, indicating virtually zero wear. We later confirmed this fact by

careful weighings. Subsequently, half micrometer diamond alone easily removed this surface but would proceed no farther than before once we had reobtained the SiC surface. We then tried the alumina with a result similar to that obtained with the silica. A rough smeared deposit was laid down, confirmed both by weighings and by Nomarski.

Next, we decided to try lubricating the diamond with silicon oil. This had worked well with metals. We tried 2 centistoke silicon oil and encountered great difficulty with pitch lap deterioration. We had used this effect to advantage on metals where the silicon oil attack on the pitch simply re-exposed the sinking diamond. Here, however, the removal was so slow that we obtained a sticky gummy lap before we had removed any significant material.

Final Pitch Polishing

At this point, we decided to return to an aqueous coolant that would not attack the pitch and to look for a soluble organic that would lubricate the diamond and also not attack the pitch. We experimented with several standard coolants, trying these first on pitch. Rodel Corporation Geonite Diamond Extender emerged as an interesting candidate.

With the half micrometer diamond and silicon oil, the surface had varied from 2.38 to 3-4 Angstroms rms in spots. While the removal rate was not good, it was perceptible. Both pitting and scratch residuals were reduced. When the new coolant was next mixed at 5% in water directly with quarter micron diamond, the results were immediately improved. We got perceptible removal with no perceptible pitch attack. The roughness quickly reduced in two runs on the same lap to below 2 Angstroms rms with very few traces of pitting or sleeking. Several deep scratches were perceptibly decreased virtually to the vanishing point. Two and one-half hours of polishing with quarter micrometer diamond heavily hand charged onto the lap and lubricated with 5% Geonite in

water reduced the background roughness to about 2.5 Angstroms rms. An additional hour reduced it to about 1.6 Angstroms rms.

We then used the same recipe with tenth micrometer diamond. Within one hour we had consistent readings in the 1.1-1.2 Angstroms rms range over the central 70% area (see Figures 6, 7 and 8). We must emphasize that these readings characterize background conditions on the surface. We do not feel that they accurately document the substantial number of sleeks that are clearly visible under strong illumination. The process at this point appears to be one of pure abrasion with no chemical redeposition to seal over micro sleeks. We believe the effects of sleeks might be minimized by optical coatings that are applied later. But this conclusion remains to be verified.

We are disturbed that our quantitative instruments seem ineffective in characterizing the large number of sleeks that are present. These sleeks may be so small that they escape detection within the 2 micrometer diameter focal spot on the Optical Heterodyne Profiler. Unfortunately, they are visually large enough to cause concern for their potential effect on x-rays impinging on a mirror surface. Work is continuing on CVD SiC sample preparation in the hope of developing a very hard, damage resistant specimen for use in round robin tests of different surface roughness instruments. Data gathered from such a test program could make a major contribution toward the improvement of our surface metrology methods.

Applications

The process we developed using small CVD SiC samples has recently served as a basis for finishing work on actual mirror components. Continental Optics has produced a 110 x 300mm cylindrical mirror with a 925mm radius, while Cooke Optical has produced a 100 x 300 mm toroidal mirror with a 114 mm traverse radius and a 419 mm long axis radius (see Figures 9 and 10). These mirrors were manufactured for use in the Lawrence Livermore National

Laboratory/University of California (LLNL/UC) X-ray Calibration and Standards Facility at the Stanford Synchrotron Radiation Laboratory. After final polishing and before coating, the surface finish of each of these mirrors was inspected at Brookhaven National Laboratory by Dr. Peter Takacs. Using a WYKO Interferometer, Dr. Takacs reported a roughness of 2.8-3.5 Angstroms rms for the cylindrical mirror and 3.2-5.7 Angstroms rms for the toroidal (Figure 11).

These two mirrors are sizeable optics rather than small laboratory flats. They were finished on a best effort basis without the advantage of previous polishing experience with SiC in complex configurations such as these. Moreover, neither facility producing these mirrors had immediate access to metrology capable of guiding the process and determining the optimum point at which to cease operations. Under these circumstances, the results are quite remarkable and extremely promising for this type of applicaton.

Acknowledgements

The authors wish to extend sincere appreciation to the following individuals and organizations for their participation, contributions and support of this work: Mr. Frank Cooke, Frank Cooke, Inc.; Manfred Grindle, Continental Optics Incorporated; Dr. Richard Gentilman, Raytheon Company; Mr. Satish Kulkarni and Mr. Victor Karpenko, Lawrence Livermore National Laboratory X-ray Calibration Facility Project; and Dr. Peter Takacs, Brookhaven National Laboratory.

References

1. Tsesnek, L. S., Physical Laws of Abrasive Disintegration, Generation of Optical Surfaces (Kumamin, Editor), Focal Press, 1962.

Thermal-distortion figure of merit

Common mirror materials	Relative figure of merit K/α
CVD SiC	27
Copper	10
Aluminum	4
Fused silica	1

The material of choice for the fabrication of synchrotron mirrors subject to thermal loading is silicon carbide

FIGURE 1 Comparison of the ratio of thermal conductivity to thermal expansion for selected materials

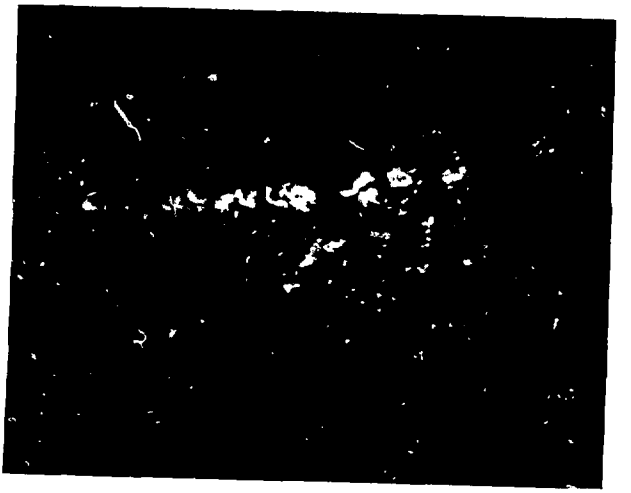


FIGURE 2 Nomarski photomicrograph of a CVD SiC Blanchard ground surface showing raised deposits that are suspected to be repolymerized hydrolized silica.

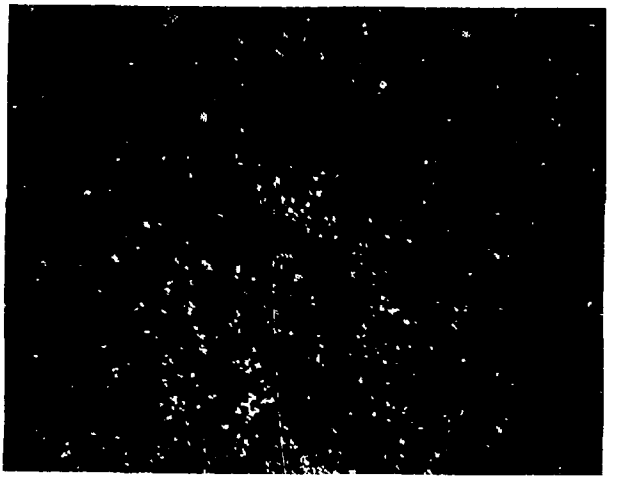


FIGURE 3 Nomarski photomicrograph of a SiC surface that was free abrasive ground using a copper Kemet lap, 9 micrometer diamond and 20% ethylene glycol and water as a lubricant.

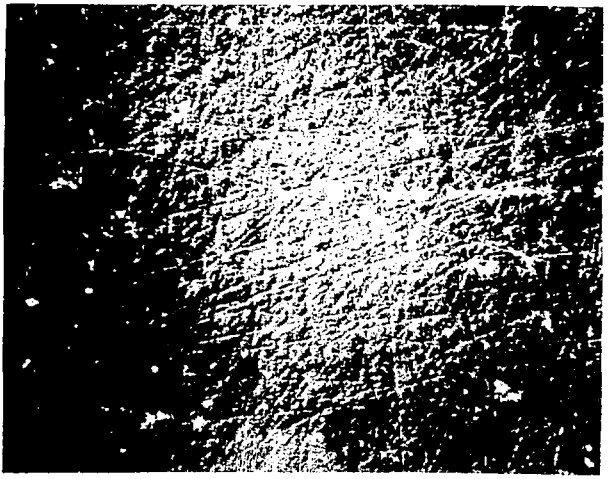


FIGURE 4 Nomarski photomicrograph of SiC further lapped using 3 micrometer diamond. Differences from Figure 3 are curious.

FIGURE 5 Nomarski photomicrograph of SiC that was pitch polished using diamond and added silica. The smeared layer is believed to be silica. Further polishing with 0.5 micrometer diamond alone removed this layer.

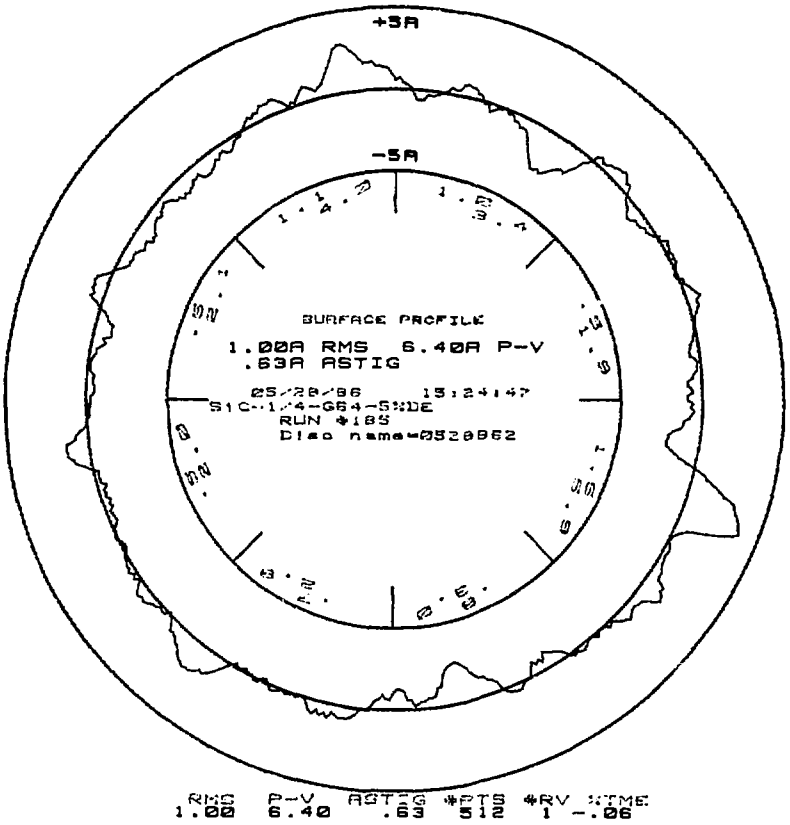


FIGURE 6 Optical Hetrodyne Profilometer trace of a SiC surface after pitch polishing with 0.1 micrometer diamond: Test sample Number 1 of three.

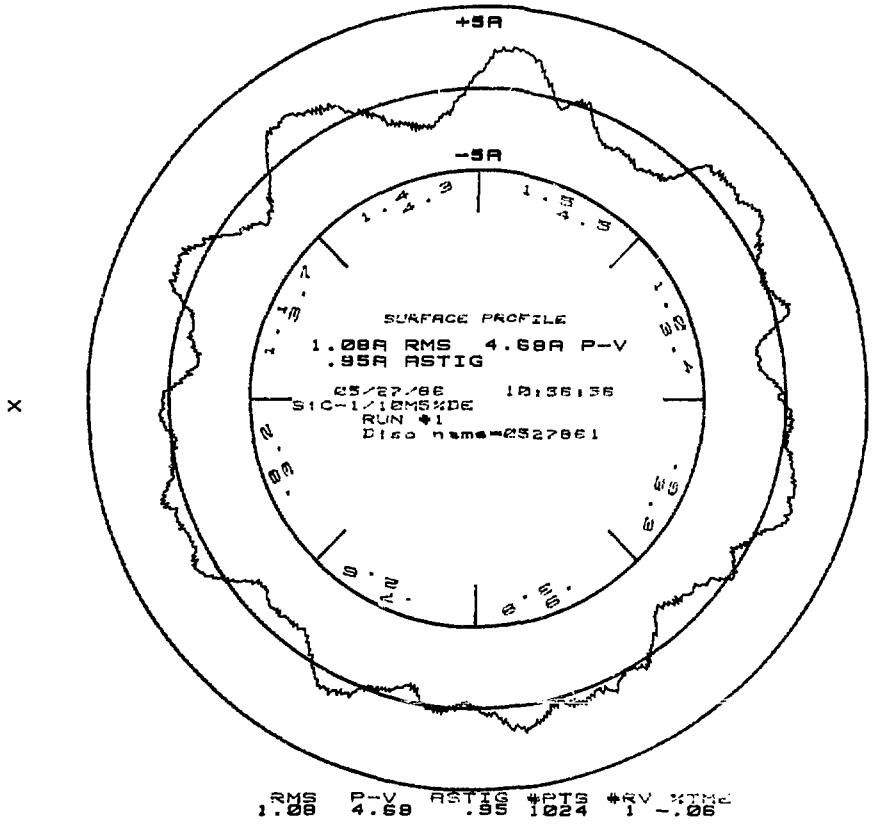


FIGURE 7 Optical Hetrodyne Profilometer trace of a SiC surface after pitch polishing with 0.1 micrometer diamond: Test sample Number 2 of three.

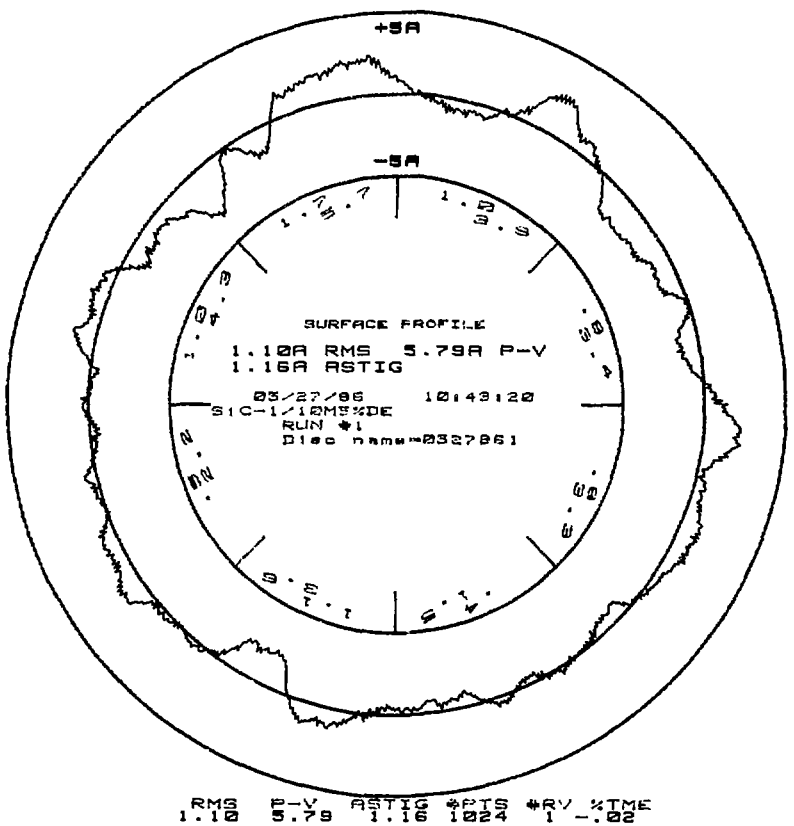
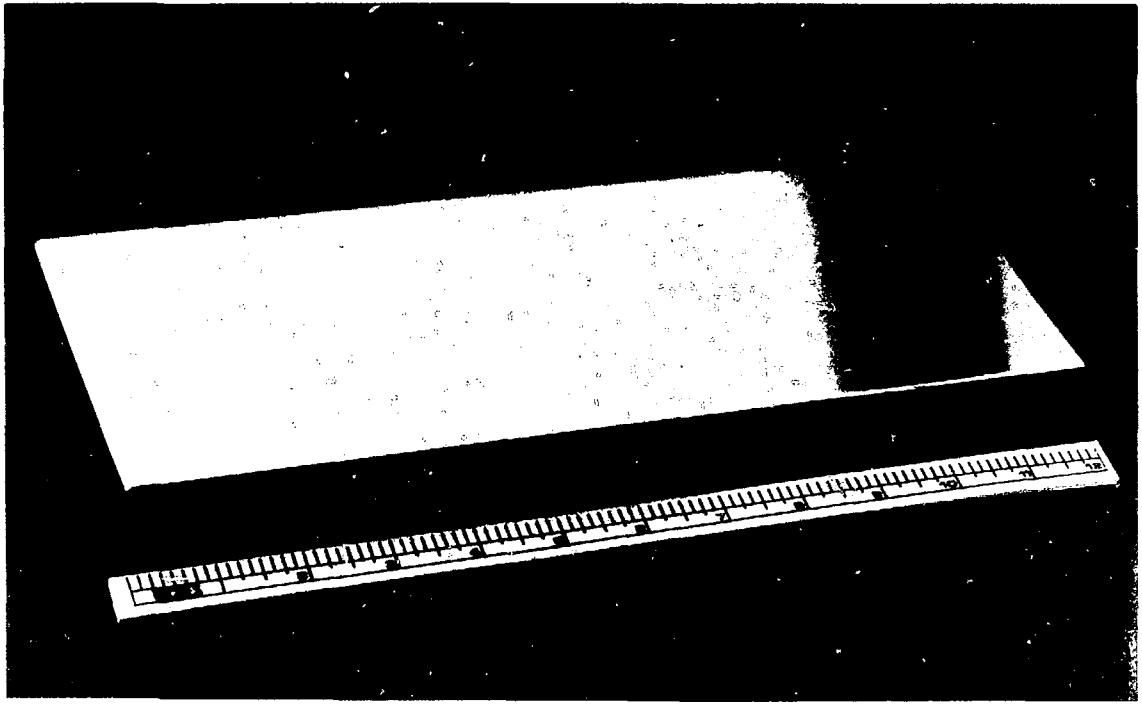


FIGURE 8 Optical Hetrodyne Profilometer trace of a SiC surface after pitch polishing with 0.1 micrometer diamond: Test sample Number 3 of three.

MO cylindrical mirror



Manufactured by continental optics

FIGURE 9 SiC cylindrical mirror, 110 x 300 mm, finished by Continental Optics to a measured surface roughness of 2.8 to 3.5 Angstroms rms.

Alpha-sintered SiC mirror holder assembly

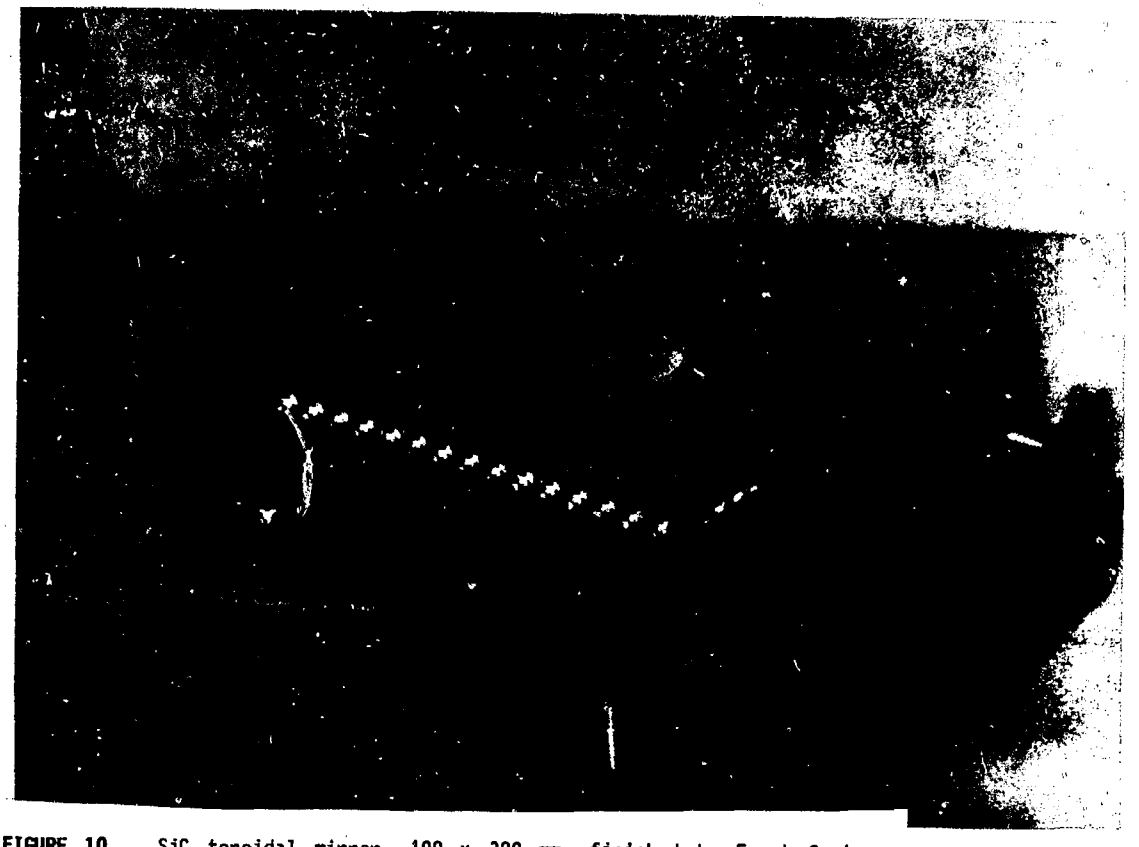


FIGURE 10 SiC toroidal mirror, 100 x 300 mm, finished by Frank Cooke, Incorporated, to a measured surface roughness of 3.2 to 5.7 Angstroms rms.

Comparisons of surface roughnesses

	Plano	Sphere	Cylinder	Toroid	Paraboloid	Ellipsoid
Fused Silica	2.6/4.7	4 4	41.0/83.3	4.3/6.9 2.1/4.4		4.5
Zerodur			16.0			
REFEL SiC		62.1 59.4		59.5		
Float Glass	(2.8/17.2) 3.5/3.7					
Single-crystal Si			25.3/28.6			
EMP on Cu	18.0					
EMP/Al (SPDT)					87.5/399.0	

1 After Pt coating

Peter Takacs NSLS

FIGURE 11 Comparisons of surface roughness on selected parts used in Synchrotron Radiation Experiments. Data provided by Dr. Peter Takacs, Brookhaven National Laboratory.