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McCarter Superfinish Grinding for Silicon An Update

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McCARTER SUPERFINISH GRINDING FOR SILICON-AN UPDATE

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ABSTRACT

A grinding technique, referred to as the McCarter Superfinish, for grinding large size optical components is discussed and certain surface characterization information about flatness and the relative magnitude of the subsurface damage in silicon substrates is reported. The flatness measurements were obtained with a Zygo surface analyzer, and the substrate damage measurements were made by x-ray diffraction and acid etching.

Results indicate excellent control of flatness and fine surface finish. X-ray measurements show that the diamond wheels with small particle sizes used in the final phases of the grinding operation renders surfaces with relatively small subsurface damage.

Keywords: Silicon, Silicon Carbide, Grinding, Superfinish, Residual Strain, Etching, and X-ray diffraction

1. INTRODUCTION

Initial efforts at McCarter Technology, Inc., focused on improving the surface finish that could be obtained on silicon with conventional machine shop grinding equipment. It was found that a surface finish approaching that of a lapped surface could be obtained using a series of grinding operations leading to what we term a McCarter Superfinish (MS). The adaptability of MS grinding to meter length components and complex shapes has been demonstrated, (Figures 1, 2 and 3) as discussed earlier[1]. These components were produced to conventional grinding tolerances. Recently, emphasis was placed on producing a number of small flat plates for a non-optical application. Lessons learned during the machining of these plates allowed the machining of larger plates to be much flatter than the 8 wave peak-to-valley (P-V) requirement. Characterization of the resultant surfaces is presented here. Another important factor in characterization of grinding surfaces is subsurface damage (SSD). Small single crystal silicon samples were machined to compare the relative magnitude of SSD left by grinding with several different grinding wheels and that of a McCarter Superfinish ground surface. Preliminary results are reported here.

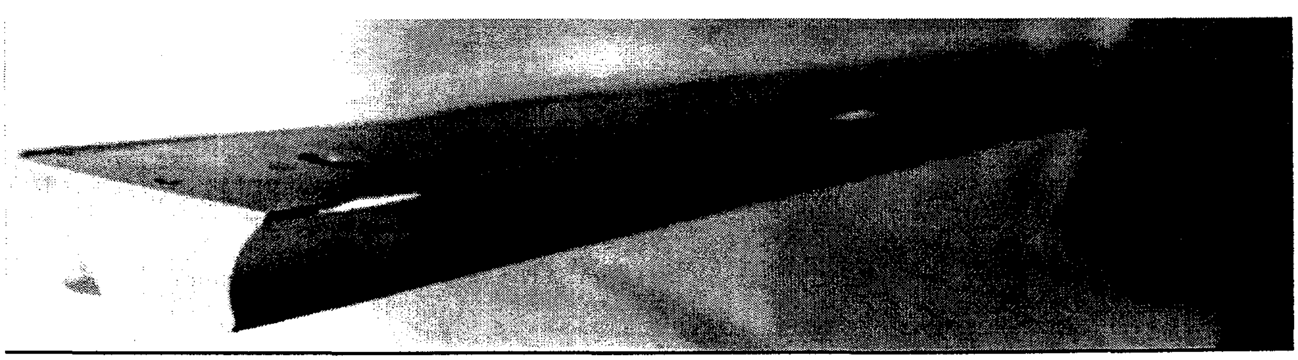

Figure 1: Superfinish silicon blank, 60 mm x 1200 mm in size with 75-mm sagittal cylindrical radius.

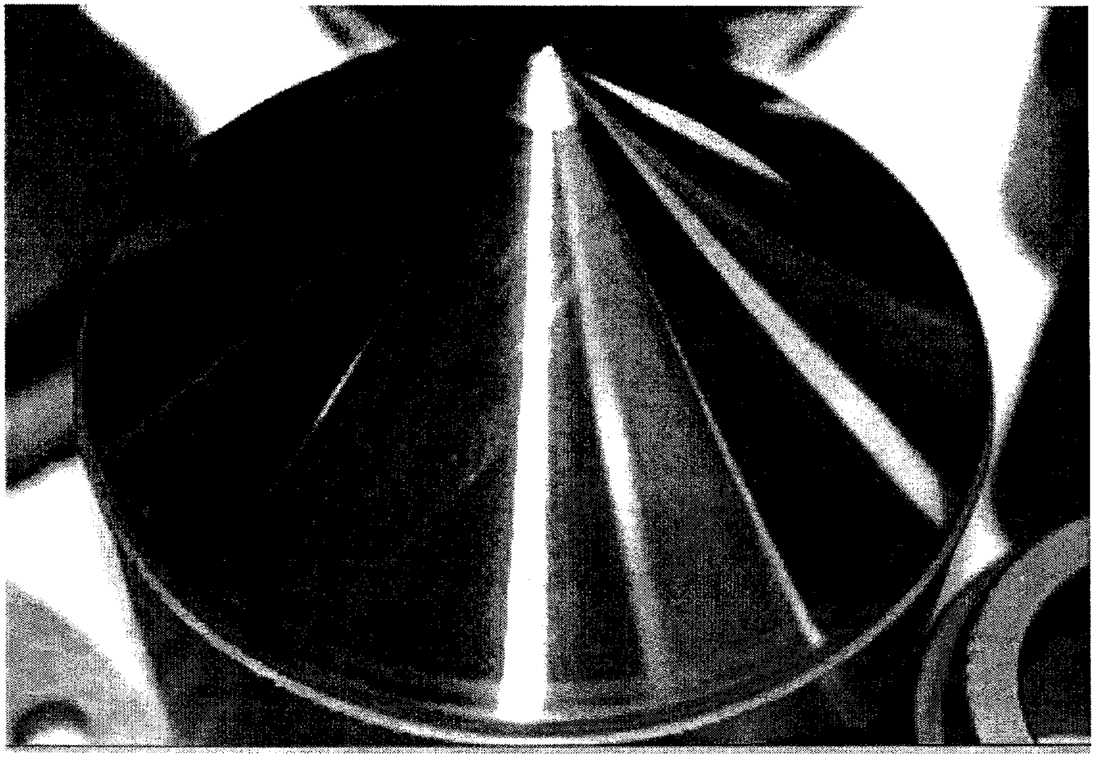

Figure 2: Superfinish silicon cone with a 125-mm base and a 45-degree half angle.

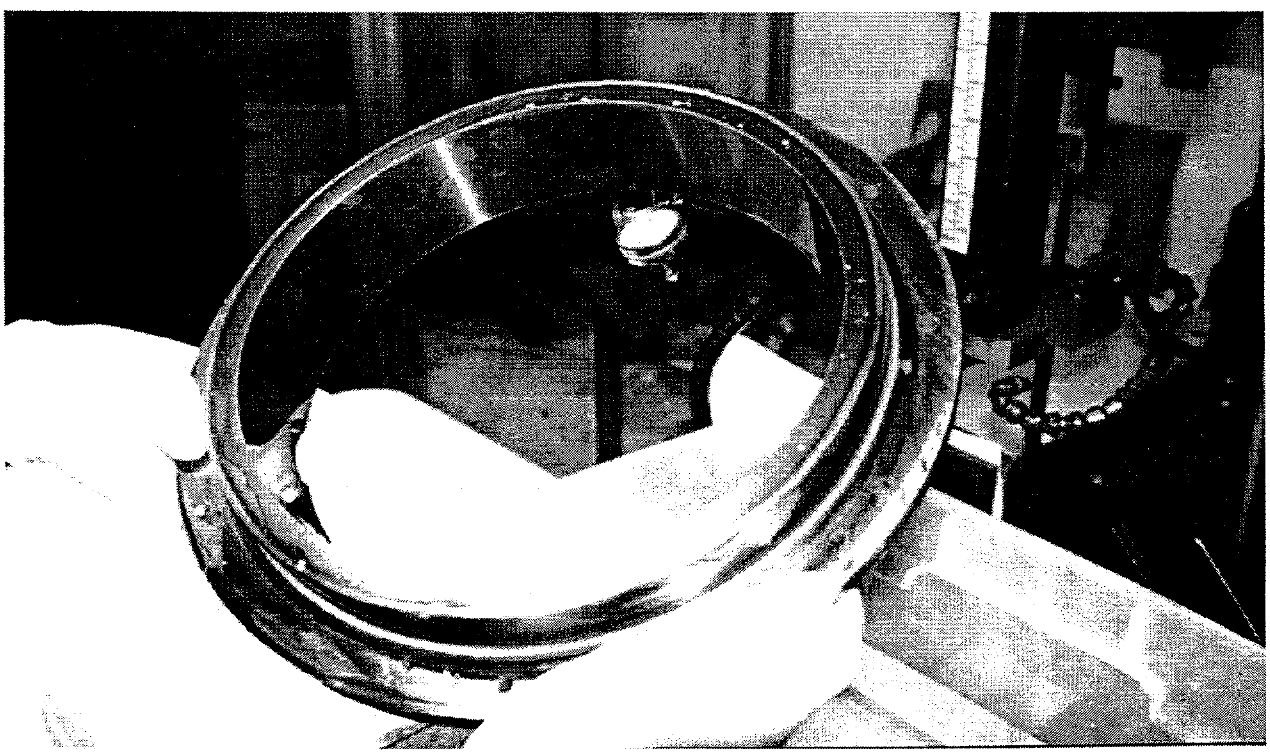

Figure 3: Superfinish silicon carbide flange, 300-mm diameter and 50 mm thick.

2. SURFACE FLATNESS

Two sizes of flat plates were ground and supplied to a customer who measured flatness with a Zygo model GPL interferometer at 6328 angstroms. Insight gained from the surface characterizations was used to improve the quality of subsequent works. A summary of the initial measurements is provided in Table 1. Samples are identified as to size and serial number. All were 25 mm (1") thick.

The RMS flatness values for the 38 mm x 38 mm (1.5" x 1.5") samples was quite good, 1 to 3 waves, as machined. Figure 4, for sample number A-2 depicts the reason for the significant difference between the RMS and p-v results; there is significant edge roll-off. The major portion of the surface is very flat and smooth. Roll-off contributes about 5 to 6 waves for A-1 and A-2 and about 10 waves for A-3. The specification for these plates was 8 waves, so the first two were acceptable and the third was accepted, although somewhat out of specification.

Table 1: Surface flatness results for the square plates using Zygo interferometer.

Sample	Size (mm)	Flatness (Waves)		Comments
		P-V	RMS	
A-1	38 x 38	7.1	1.1	For these three small samples, the entire surface area could be examined with the 100-mm-diameter Wyko profiler.
A-2	38 x 38	6.6	1.3	
A-3	38 x 38	13.5	2.9	
B-1	150 x 150	4.4-5.8	0.7-1.2	Measurements over overlapping 100 mm apertures.
B-2	150 x 150	3.0-4.3	0.1-0.6	Measurements over 100 mm and 38 mm apertures.
B-3	150 x 150	*	*	Surfaces flat beyond Wyko sensitivity; Estimated P-V (waves): 4 (entire surface) & 1 (without roll-off).
B-6	150 x 150	*	*	
B-4	150 x 150	2.5-4.1	0.3-0.5	Measurements over 30 mm apertures.
B-5	150 x 150	1.2-4.7	0.2-0.7	Measurements over 100 mm and 38 mm apertures.

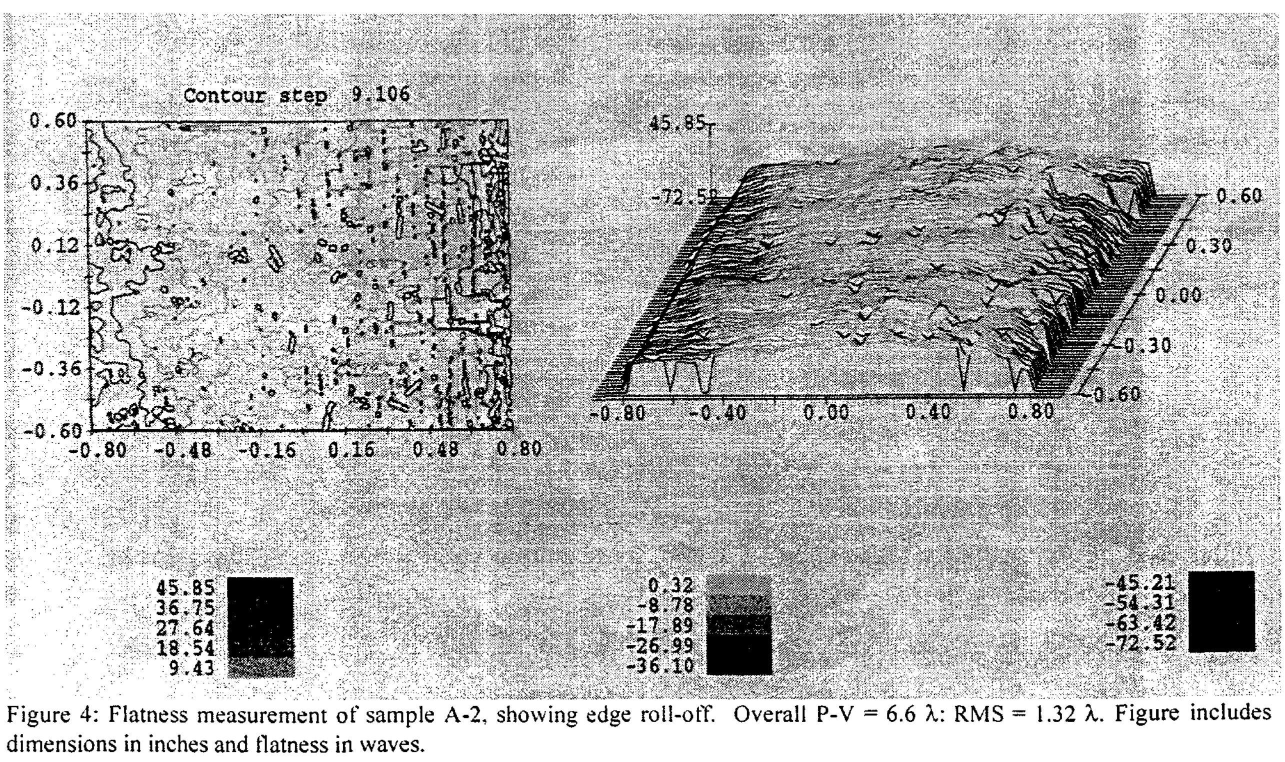

Figure 4: Flatness measurement of sample A-2, showing edge roll-off. Overall P-V = 6.6 λ: RMS = 1.32 λ. Figure includes dimensions in inches and flatness in waves.

Because the maximum aperture size of the Wyko instrument used is 100 mm, each set of measurements for the larger 150 mm x 150 mm (6" x 6") plates listed in Table 1 pertains to only a portion of each of the plate but locations overlap. Most locations were selected to avoid roll-off regions. Sample B-2 measurements were made with and without the roll-off regions. With roll-off, the value of 6.9 waves is within the range observed for all other the plates and is within the customer's 8 wave P-V specification.

Some plates showed concavity and although they were within the specification, they were reground. Plate B-3 produced no significant print out when examined, the reason being the excellent flatness of the surface and removal of surface damage introduced by prior roughing cuts. There are regions that are very flat, less than 1 wave, as apparent in Figure 5 with a better than 0.25 wave P-V. Analysis of all measurements suggests that a limiting value of smoothness of about 2 μm (100 μ in) P-V as seen by typically orthogonal line scans shown in Figure 6. The excellent flatness of plates B-3 and B-6 was confirmed by measurements with an electronic dial indicator, although there was still as much as 4 waves of roll-off.

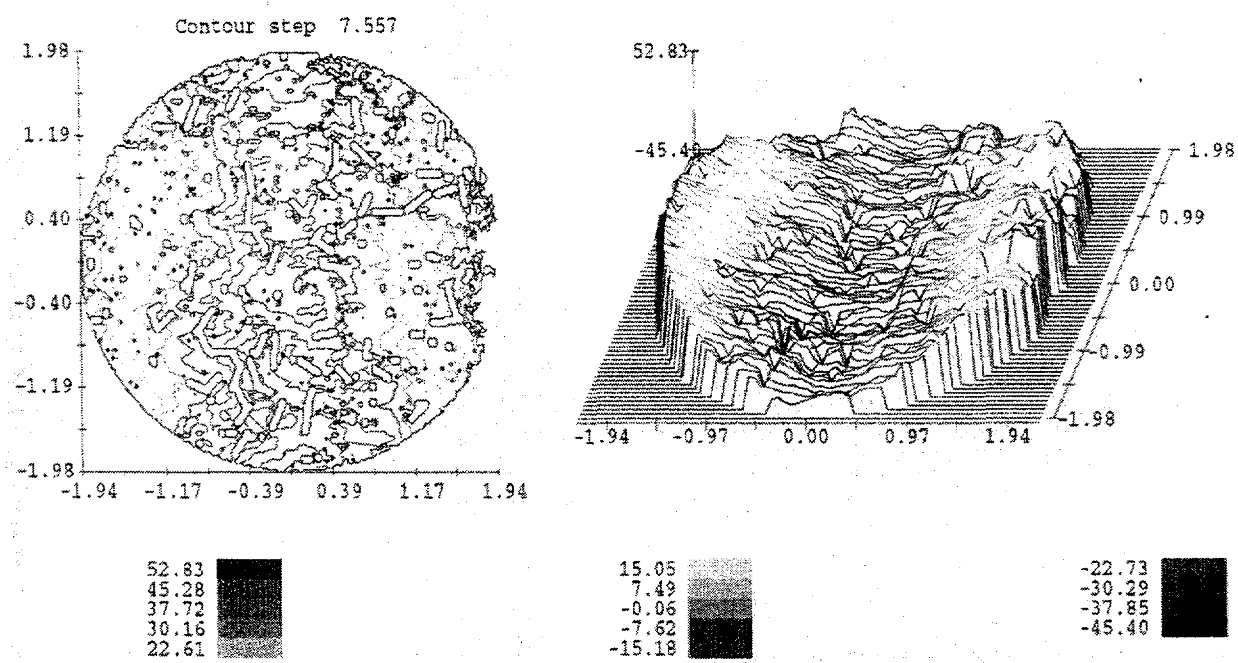

Figure 5: Initial surface profile of the sample B-2 over a 100 mm (4") diameter region, indicating an RMS of 0.75 waves and P-V of 3-4 waves.

Concave plates were rotated 90° and ground, after the initial Wyko examination, to remove the concavity. Subsequently they were too flat to be measured, better than 0.7 wave P-V. The surface inspection data was a key element in achieving flatter surfaces. It provided insight into the surface features that needed improvement. Such an interactive approach warrants consideration when demanding tolerances are required, even at grinding level..

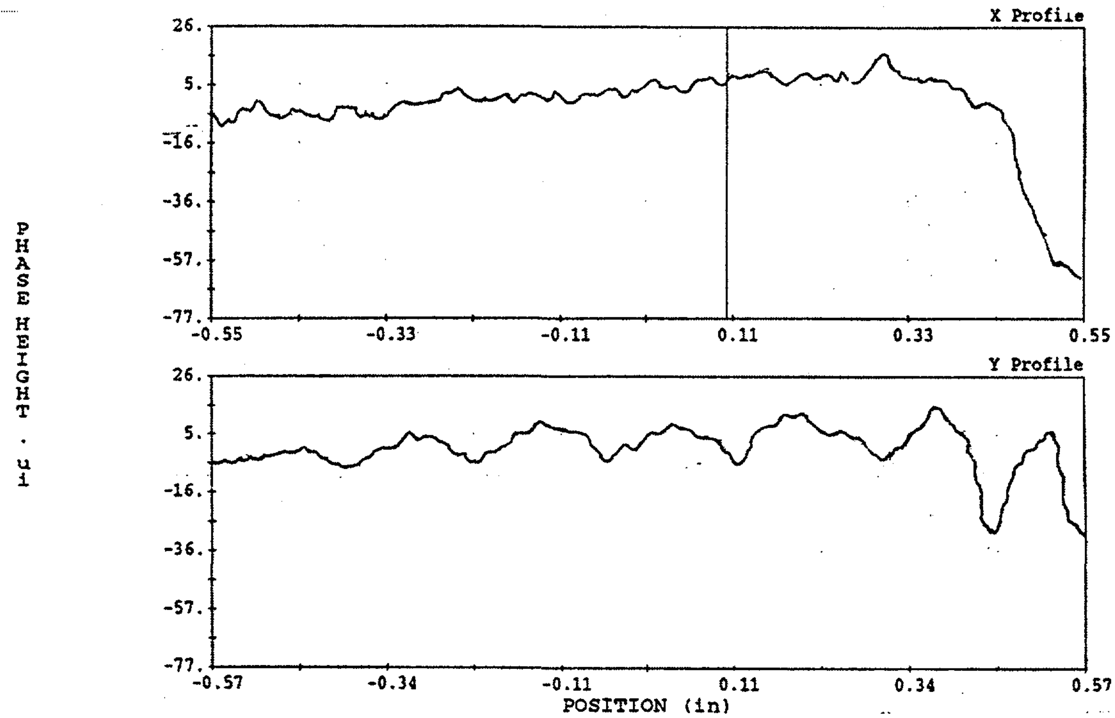

Figure 6: Line profiles of a typical surface suggesting an attainable surface smoothness of under 2.5 μm (100 μin) P-V on the surface.

3. QUANTITATIVE ANALYSIS OF SUBSURFACE DAMAGE

In order to determine the subsurface damage, four single crystal silicon samples were prepared, ground, and evaluated using the Advanced Photon Source (APS) x-ray topography system.

The purpose of the analysis was to quantitatively estimate the subsurface damage (SSD) introduced by the grinding of silicon using wheels with bonded abrasives of various particle size. A more careful analysis, which is planned, would determine the thickness of the SSD layer and also shed light on the nature of this damage: mosaicity, residual stress, and dislocation. Mosaicity in a single crystal would broaden the rocking. An angular shift in the rocking curve peak of a crystal suggests a change in the d-spacing, i.e., residual stress in the crystal. Loss of photons, determined by integrating the area under the rocking curve and comparing it with a perfect crystal, can be due to destruction of crystalline structure in the damaged layer.

Four silicon substrates, each 70 mm in diameter and 22 mm thick were prepared. They are cut from a disk perpendicular to the growth direction of a (111) single crystal boule. They were all ground flat on both faces and then completely etched in a hydrofluoric/nitric acid solution to remove all damage introduced during machining. The measured full-width-half-maximum (FWHM) of the rocking curves of the samples at copper Kα energy (8048 eV) was about the ideal value of about 3.6 arcseconds.

The etched samples were then ground on one side with resin bonded grinding wheels having different imbedded particle sizes. The grinding history of the samples is shown in Table 2. Figure 7 shows the surface texture of these samples at 20 x magnefication. The backsides of the samples were all (inadvertently) ground with the 400 grit wheel to flatten the etching introduced waviness. Given the thickness of the disks, grinding of the backside of the samples was not expected to affect subsequent measurements of front sides rocking curves.

TABLE 2: Gridning history of the single crystal silicon samples and the measured rocking curve FWHMs at8 keV photon energy.

Sample	Diamond abrasive particle (μm)/(mesh size) used & the thickness of layers removed in the operation				Rocking curve FWHM (arcsec)
	46/400	30/600	25/800	*	
	Thickness of the material removed (μm)				
A	~ 1000	-	-	-	91
B	~ 1000	680	-	-	40
C	~ 1000	680	160	-	36
D	~ 1000	680	160	75	25
Perfect Crystal	-	-	-	-	4

*Proprietary information. McCarter Superfinish Grinding.

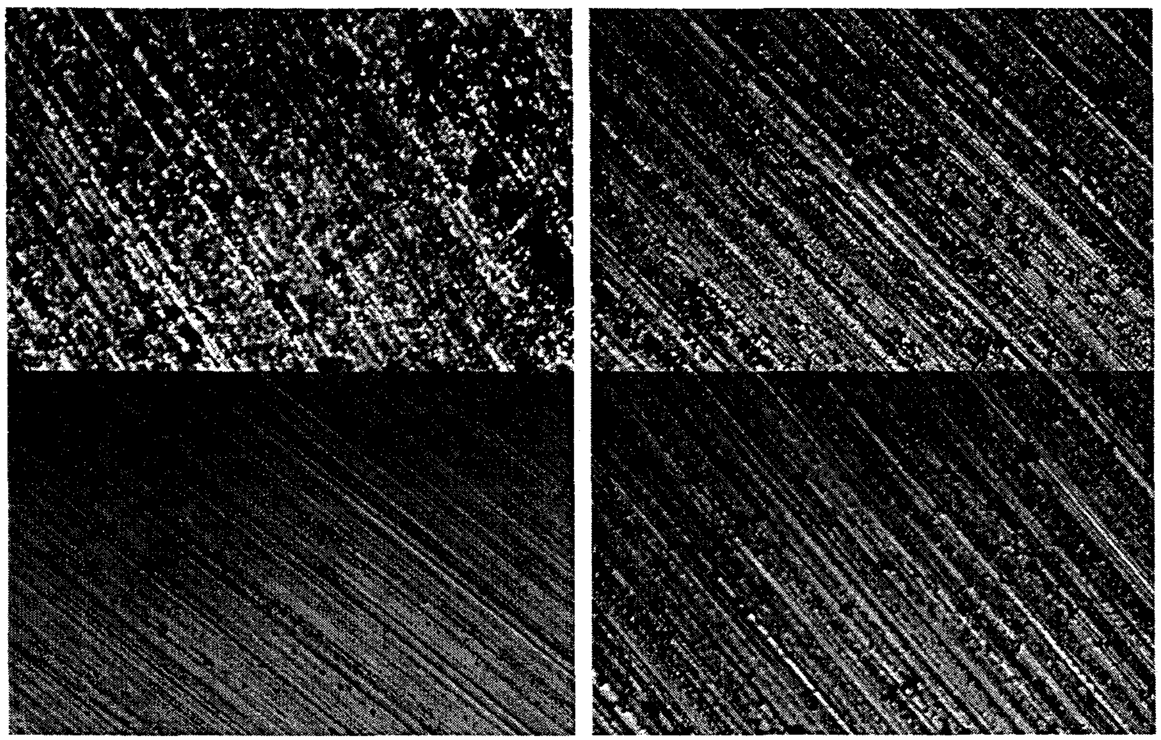

Figure 6: Ground single silicon crystal samples A, B, C, and D (clockwise from Top Left) at 20x magnification. Each picture shows a 0.3 mm x 0.4 mm area.

After the grinding operations noted in Table 2, the samples were evaluated one by one at the APS x-ray topography lab. Figure 8 shows the raw rocking curves of the samples measured at Cu Kα energy. The areas under the curves, normalized to 100 for sample A, are also noted. In these measurements, the entire surface of each crystal was sampled. Other measurements examining smaller areas of the sample produced the same results, indicative of the uniformity of damage across the sample surface. The angular shifts in the rocking curves seen in Figure 8 are due to sample mounting and have no significance.

In Figure 9, these same rocking curves are shown with their peak intensity normalized to unity. Their FWHMs, indicative of SSD, correlate well with the abrasive sizes (see Table 2) used in their grinding. The FWHM values ranging from 91 arc seconds for sample A to 25 arc seconds for the (Superfinish) sample D are substantially higher than 4 arc seconds value for an undamaged crystal. A more detailed analysis of these rocking curves, particularly their wings, can provide valuable information about subsurface damage. However, the data in terms of the FWHM presented here are sufficient to provide qualitative information about the damage introduced by various grinding operations. While the SSD in the Superfinish sample is substantially smaller than in other samples, there remains a thin damaged layer.

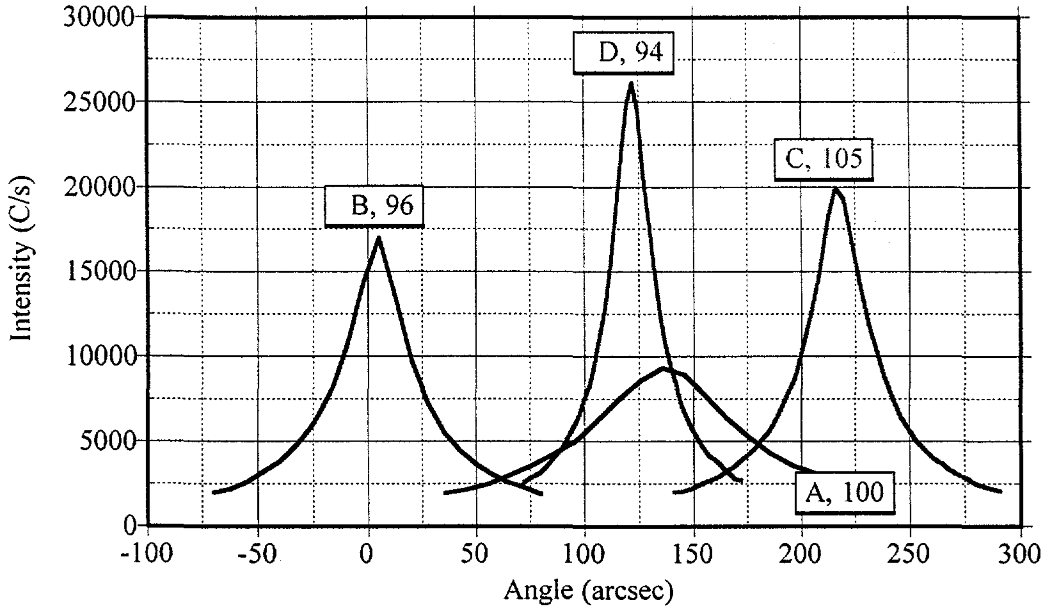

Figure 8: Rocking curves for the four Si (400) samples. Areas under the curves are normalized to 100 for sample A. Higher intensity (smaller curve width) indicates lower subsurface damage. Shift in position of the curves along the x-axis is of no significance and is due to mounting of samples.

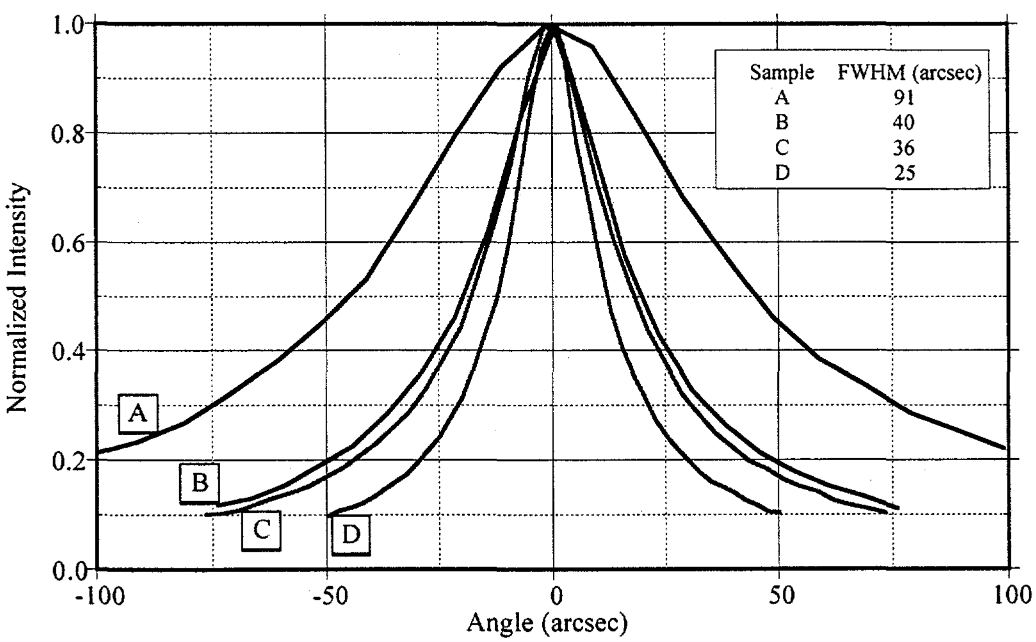

Figure 9: Normalized rocking curves of the four samples. The insert shows their FWHM values.

In an attempt to provide additional qualitative information about the depth of SSD, x-rays from a molybdenum (Mo) target (17 keV) were used to obtain the rocking curve for sample A. The extinction and attenuation depths of photons from a Cu target for Si (400) are 15 μm and 60 μm, respectively. For a Mo target, these are 40 and 655 μm, respectively. The FWHM of the rocking curve for a perfect Si (400) crystal at Mo energy is 1.4 arc seconds compared with 3.6 at Cu energy. The normalized rocking curves for sample A at 8 and 17 keV photon energies are shown in Figure 10. Because a higher fraction of the more penetrating 17 keV photons diffract from the depth of the silicon sample, the much smaller FWHM of the rocking curve at 17 keV is a further indication that the SSD is confined to a thin layer at the surface. The rocking curve measurements here and the values of the extinction depths indicated above seemed to suggest that subsurface damage depth might be about the abrasive particle size as in glass substrates[2]. In fact, an emperircal equestion SSD (μm) $=1.07\ L^{3/4}$ is suggested, where L is the abrastive particle size[2].

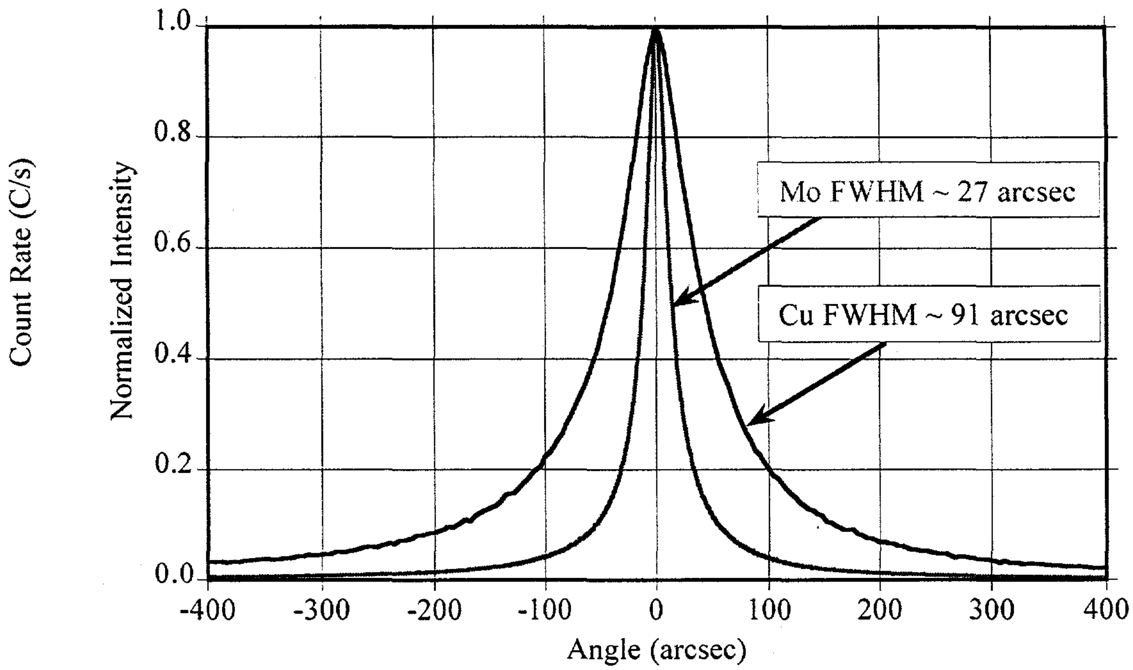

Figure 10: Normalized Rocking Curves of Sample A (Top Side) Measured in APS Topography Lab at Cu (8 keV) and Mo (17 keV) Energies.

In order to provide a precise estimation of sub-surface damage in the samples, the front sides of all samples were etched in a hydrofluoric/nitric acid solution. As depicted in Figure 11, the front surface of each sample was partially painted to avoid being etched. All four samples were then mounted on a Teflon holder and dipped into the etchant stepwise such that four different layer thickness are etched away from each sample. In each sample, the bottom segment was etched 15 minutes, the next segment for 9 minutes, the following one for 6 minutes, and the top segment, dipped last into the etchant for 3 minutes. The reason for painting parts of the surface to avoid etching was to provide a reference for measuring the thickness of the etched layers, and to allow a second etching operation should that become necessary. Measurements indicated that layers ranging from 8 to 45 μm in thickness had been removed from each sample, somewhat more than what was intended to etch. The samples were analyzed on the x-ray topography system. Results indicated that essentially all of the damage had been removed; only a very small residual strain was observed in the least etched part of sample A, indicating that the most coarse grinding operation (with the 45 μm mesh) seems to have introduced a 10-15 μm deep SSD. This confirmed expectations based upon estimates of between 1 and 15 micrometers presented in last year's paper[1], based on the work or Kerstan[3] and Ball[4]. Kerstan correlated surface roughness with SSD as determined by scanning infrared depolarization, photothermal, and high-resolution transmission electron microscopy techniques. Ball correlated SSD with surface roughness by observing the depth of microcracking at high magnification. The infrared data of Kerstan was favored for the correlation of the Superfinish grind while the Ball data was used directly for all surfaces. This approach bracketed the estimated range of SSD for each surface.

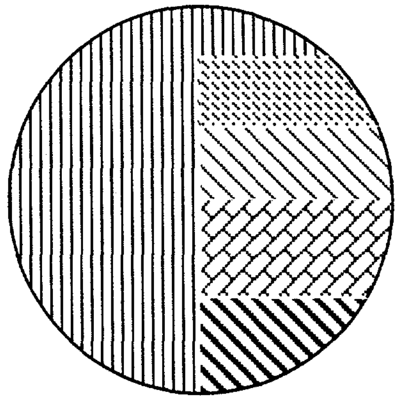

Figure 11: Front sruface of each silicon test sample is partially painted (vertical hatching) and the exposed area etched to varrying degrees (four shaded areas on the right side) to remove four different thicknesses.

4. CONCLUSIONS

We expect to etch the other half of the samples with a more diluted acid etch and determine the SSD depth accurately. The results will be reported in a later publication. Detailed inspection of ground surfaces is a valuable tool for improving the machining process. It was used to improve the McCarter Superfinish technique to the point where the surfaces produced were within a few waves of the desired flat configuration. Although the characterized parts were not for an optical application, their quality suggests that grinding technology may be approaching the point where lapping time can be reduced or eliminated for some types of optics. Two significant lessons were learned from the interferometer examinations of the plates that were characterized. First, the use of wasters to surround the plates should be considered as a means of minimizing roll-off at edges. Second, the amount of material removal required to eliminate surface damage from diamond wheels employed for roughing cuts can be determined. Improvement in the visually observed smoothness of the surface is not an adequate indication that prior surface damage has been removed but interferometer techniques appear to provide such information. Optical evaluation of the surfaces does not quantitize the subsurface damage, however. Other techniques such as the measurement of residual strain can provide guidance in this regard. Results of the x-ray analysis of the ground surfaces demonstrate the value of this technique for the definitive information about SSD.

4. ACKNOWLEDGEMENT

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5. REFERENCES

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