

Increased UV transmission by improving the manufacturing processes for FS

Jessica DeGroot^{*a}, Tobias Nitzsche^a, Daniel E. Savage^a, Jonathan T. Watson^a, Donald K. Henry^a, Andrew A. Haefner^a and Robert A. Wiederhold^a

^aOptimax Systems Inc, 6367 Dean Parkway, Ontario, NY, USA 14519

ABSTRACT

Optical designers have been designing ultraviolet (UV) systems at wavelengths in the UV region for many years. With increasing demand for deep UV applications, special considerations that are not applicable to traditional visible optics must be taken to produce the optics. Specifically as the wavelength of incident light decreases, the importance of very smooth surfaces increases. The intent of this project is to increase the performance of UV optics in a four-phase project. The first phase consists of characterizing sub-surface damage using destructive methods to enable process control, the second phase (presented here) focuses on polishing methods, the third phase will include cleaning and possible etching protocols and the fourth phase will be improving thin film coating performance.

Keywords: Ultraviolet, fused silica, polishing, coating

1.0 INTRODUCTION

As trends in UV optical system design shift to shorter UV wavelengths, optical manufacturing has to be more conscious of the effect that subsurface damage, surface features, residual contamination from polishing and cleaning and coating have on the residual performance of the optics in their systems. For many years, researchers have tackled partial aspects of these problems. For example, Bloembergen¹ stated that cracks and pores on an optical surface will lead to laser damage (LD) when incident with a laser beam. Neauport et al.² spoke to two of the main damage initiators of LD, sub-surface damage (SSD) and nano-absorbing centers, focusing mainly on the latter. They used fused silica optics in high power laser applications at 351nm. Higher cerium concentration on the surfaces strongly correlated with increased damage density. Aluminum, copper and iron did not have similar correlations. Neauport et al. also tried to correlate the presence of cerium with damage morphology but the results were inconclusive. Yoshiyama et al.³ studied the effects of polishing, etching, cleaving and water leaching on the UV damage of fused silica. The surfaces were all exposed to a Nd:YAG laser at 355nm. Micropits were found on the polished surface. Their analysis found high concentrations of Al, B, Ce and Zr. The concentrations of the Al, B and Zr all decreased rapidly to less than 10% of the maximum value at a depth of 50nm, but the Ce required ~100nm before decreasing to less than 10% of its maximum value. A second sample etched with a buffered HF solution had a lower pit density than the polished surface. The pit density decreased exponentially with the etched layer thickness indicating that the cerium is a precursor to laser damage. Micropits found on the cleaved surface indicated that cerium contamination is not the only cause of damage. It is hypothesized that damage initiated because of residual stresses and permanent mechanical damage from the cleaving process. Hydrolyzed cleaved surfaces were found to decrease the laser damage threshold. Camp et al.⁴ determined that the zirconia conventionally polished surfaces have a higher laser damage threshold at 355nm compared to ceria polished surfaces. They also observed that damage typically centered around scratches or digs on the surface of the parts. Neauport et al.⁵ tried to improve laser damage threshold (LDT) of fused silica at 351nm. They performed experiments whose results prove the importance of a proper step grind to eliminate all SSD. They also determined that MRF followed by a chemical etch increases the LDT.

* jnelson@optimaxsi.com; phone: (585) 265-1020 x276; www.optimaxsi.com

All of these advances prompted us to expand the work by the above researchers and Cumbo et al.⁶ who studied the chemo-mechanical effects during optical polishing. The intent of this project is to increase the performance of UV optics in four phases. The first phase consists of characterizing sub-surface damage using destructive methods to enable process control, the second phase (presented here) focuses on polishing methods, the third phase will include cleaning and possible etching protocols and the fourth phase will be improving thin film coating performance.

2.0 EXPERIMENT

Phase one of this project entailed implementing the COM ball test⁷ to destructively measure sub-surface damage (SSD) as a function of material, abrasive type and size, pressure and speed. Figure 1 is a picture taken of the COM ball testing apparatus constructed at Optimax.

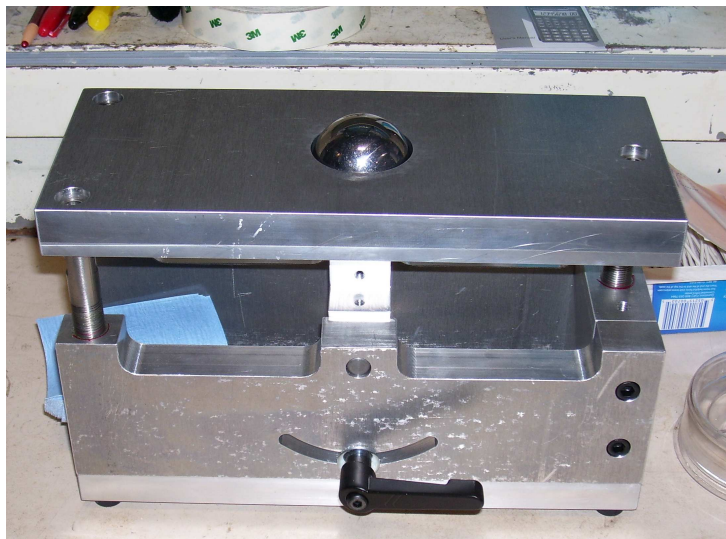


Figure 1: Photograph of the COM ball SSD testing apparatus

This test is a destructive test where a ground surface is first lightly etched to expose all of the sub-surface cracks, and then a small circular hole is polished into the surface using diamond paste and the chrome steel ball shown in Figure 1. The resulting surface has a circular polished area, the sub surface damage is calculated with a delta sag equation shown as Equation 1. D_1 is the diameter of the circle of the edge of the ground surface, D_2 is the diameter of the circle of the last visible portion of damage, R is the radius of the chrome-steel ball. Figure 2 contains a schematic drawing of a dimple and a photograph of a portion of the dimple. The diameters were measured on a white light microscope.

$$\text{SSD depth} = \Delta\text{sag} = [R - \sqrt{R^2 - (D_1/2)^2}] - [R - \sqrt{R^2 - (D_2/2)^2}] \quad (1)$$

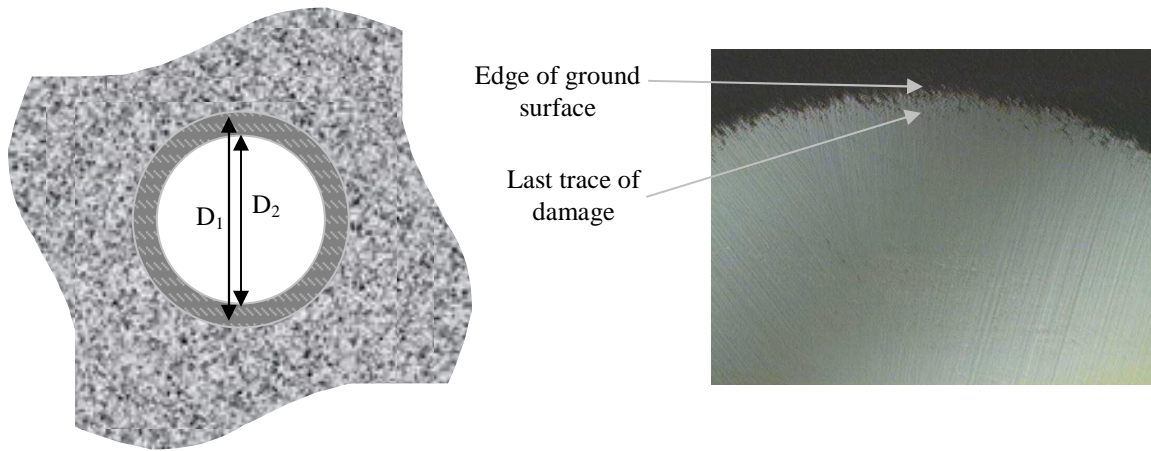


Figure 2: Schematic drawing of a COM ball dimple and photograph indicating edge of ground surface

In addition to destructively measuring the depth of sub-surface damage, the ground (unetched) surfaces were measured on a white light interference microscope. Similar to Lambropoulos et al.⁸ our experimentation discovered that there was a correlation between SSD and the peak-to-valley (PV) surface roughness measured on a white light microscope. Our experimentation was limited to loose abrasive lapping, and we found that removing at least 1.2 times the PV areal surface roughness was sufficient to remove all damage on optical glass. Lambropoulos' factor of 2 times the PV areal surface roughness also included deterministic microground surfaces, which according to his data had more damage for the same PV surface roughness value. Using this information, we implemented a loose abrasive grinding process to ensure that for each subsequent layer all of the SSD is entirely removed leaving a pristine surface ready for polishing. This grinding process was used for all of the glass samples used for this work.

For phase two, we focused on the UV material fused silica, reducing surface roughness and optimizing removal rate and transmission. The variables we chose to adjust in our experimental design were abrasive, pH and polishing lap material. There were four different types of abrasives used, all were approximately 1 μ m mean particle size and the Mohs hardness values were 6 (Abrasive 1), 6.5 (Abrasive 2), 9 (Abrasive 3) and 10 (Abrasive 4). The pH values ranged from 6 to 10, depending on the abrasive type, and we chose to use both a traditional natural pitch and a synthetic pitch with similar viscosity. Figure 3 contains a picture of the side by side experimental set up. Two parts were run at the same time, each with the same abrasive but different pitch types. Polishing conditions such as pressure, speed, concentration, abrasive size and part diameter were all kept constant. Material removal rates were measured by weight change utilizing an analytical balance. Surface roughness measurements were made on a white light interferometer, and transmission measurements were made using a spectrophotometer.

The surface cleaning and thin film coating phases have not yet been completed.



Figure 3: Photograph of the polishing test set-up

3.0 REMOVAL RATE RESULTS

The removal rate data for three of the abrasive types are shown in the plot of polishing slurry pH versus average material removal rate in Figure 4. At the time of this paper, Abrasive 1 was the only polishing abrasive to vary the polishing slurry pH. Upon closer inspection of Abrasive 1, the removal rate results for pH 6 and 8 were very similar, independent of pitch type. The removal rate results at pH 10 however differed significantly indicating a very high dependence on pitch type for removing FS material with Abrasive 1 in basic environments. Abrasives 2, 3 and 4 all had lower removal rate values compared to Abrasive 1, other than the natural pitch at pH 10 condition. In fact, Abrasive 4's extremely low removal rates caused the experiment to be stopped before all of the grinding damage was removed. Removal rates that low would not prove to be economically feasible in production.

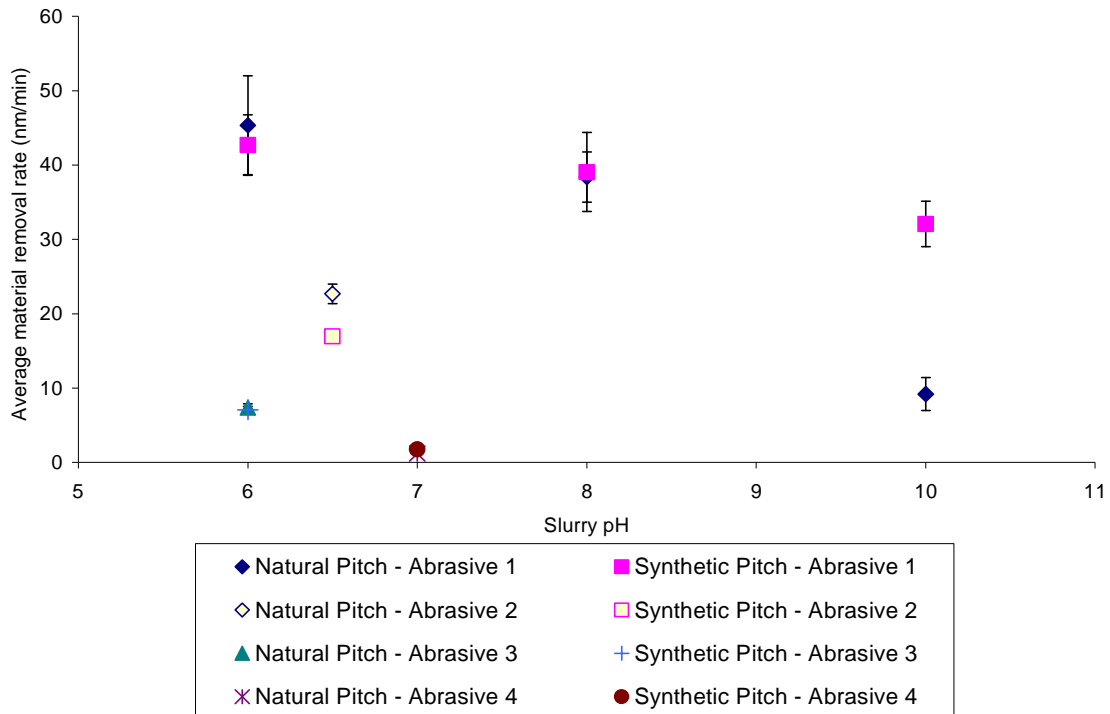


Figure 4: Polishing slurry pH versus average material removal rate for fused silica for four different abrasive types

In order to have a better view of this experimental data as a function of abrasive type, the same average material removal rate data has been plotted versus abrasive hardness in Figure 5. Although the Abrasive 1 data has a large range due to changes in slurry pH, the general trend is quite pronounced that the average material removal rate decreases as the polishing abrasive hardness increases.

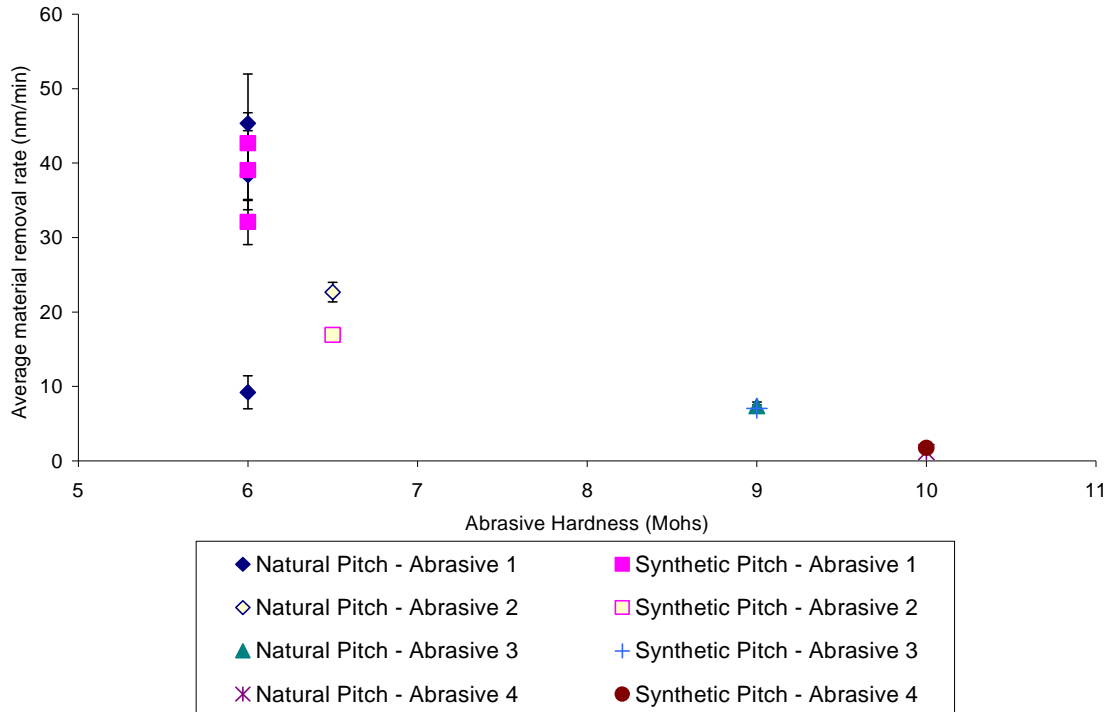


Figure 5: Polishing abrasive hardness versus average material removal rate for fused silica

4.0 SURFACE ROUGHNESS RESULTS

The average areal surface roughness for the Abrasive 1, 2 and Abrasive 3 (natural pitch) are shown in Figure 6 as a function of slurry pH. The remaining three surfaces, Abrasive 3 (synthetic pitch) and Abrasive 4 (natural and synthetic pitch) results are not shown in this plot because the surface roughness values were so high [98 \pm 35Å, 270 \pm 65Å and 255 \pm 74Å respectively] that they biased the graph.

One very interesting trend found with the surface roughness results were the large difference between the natural and synthetic pitch for Abrasive 1. At pH 6 the surface roughness values were comparable with error, but as the pH became more basic the surface polished with Abrasive 1 and natural pitch had much smoother surfaces compared to surfaces polished with synthetic pitch. Recalling the removal rate data shown in Figure 4, the removal rate was also significantly lower at pH 10 with the natural pitch versus synthetic pitch. At first glance, the Abrasive 2 surface roughness data appears to fall in line with the Abrasive 1 data, but the pitch types are flipped, meaning the synthetic pitch and Abrasive 2 produced a smoother surface compared to natural pitch and Abrasive 2. The Abrasive 3 and natural pitch data point has also been included in Figure 5 indicating comparable surface roughness values to Abrasive 1 and Abrasive 2.

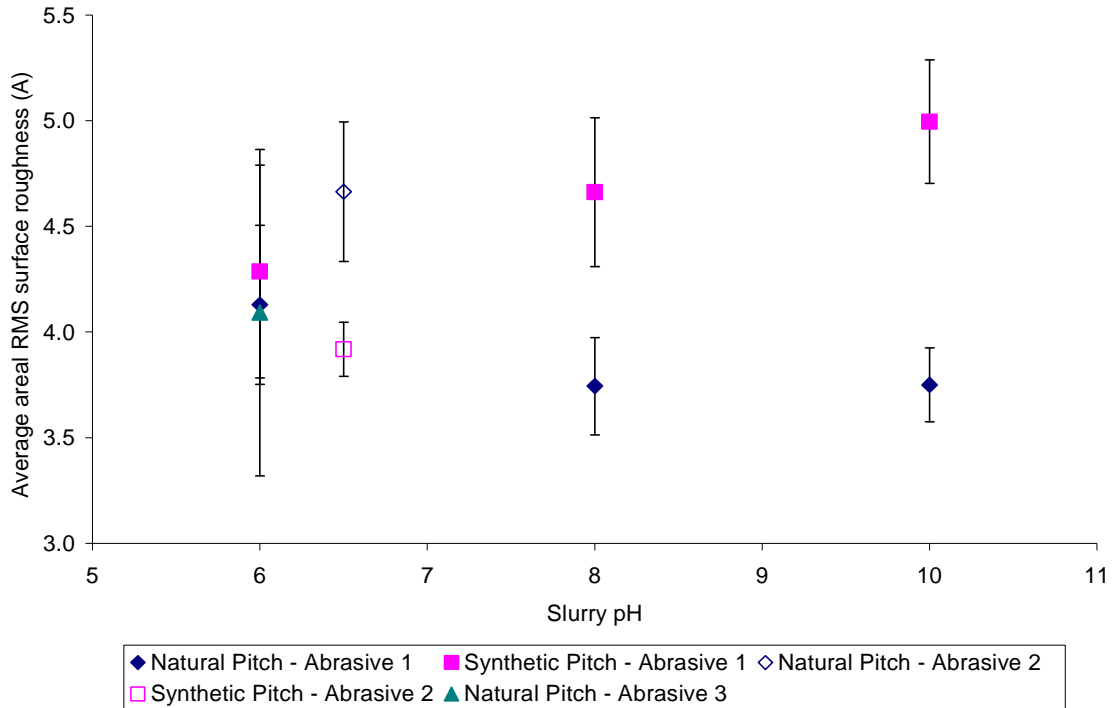


Figure 6: Slurry pH versus average areal rms surface roughness

5.0 TRANSMISSION RESULTS

The main goal of this work is to determine the optimum polishing method to ensure the highest transmission possible for fused silica at UV wavelengths. The wavelength that was chosen for comparison was 266nm. This wavelength was chosen for three main reasons: it is a commonly used wavelength in the UV industry, Abrasive 1 is a known absorber at this wavelength, and the spectrophotometer will still accurately give transmission values. Similar to Abrasive 1, Abrasive 2 also absorbs in the UV, but its absorption levels are higher at wavelengths shorter than 266nm.

Figure 7 contains a plot of the slurry pH versus the average transmission at 266nm for fused silica polished (bare window substrate) with Abrasive 1 and 2. At the time of this presentation, transmission measurements had not been made on the surfaces polished with Abrasive 3. They will be published at a later date. Due to the fact that only side 1 of the Abrasive 4 surface was partially polished, transmission measurements were not possible.

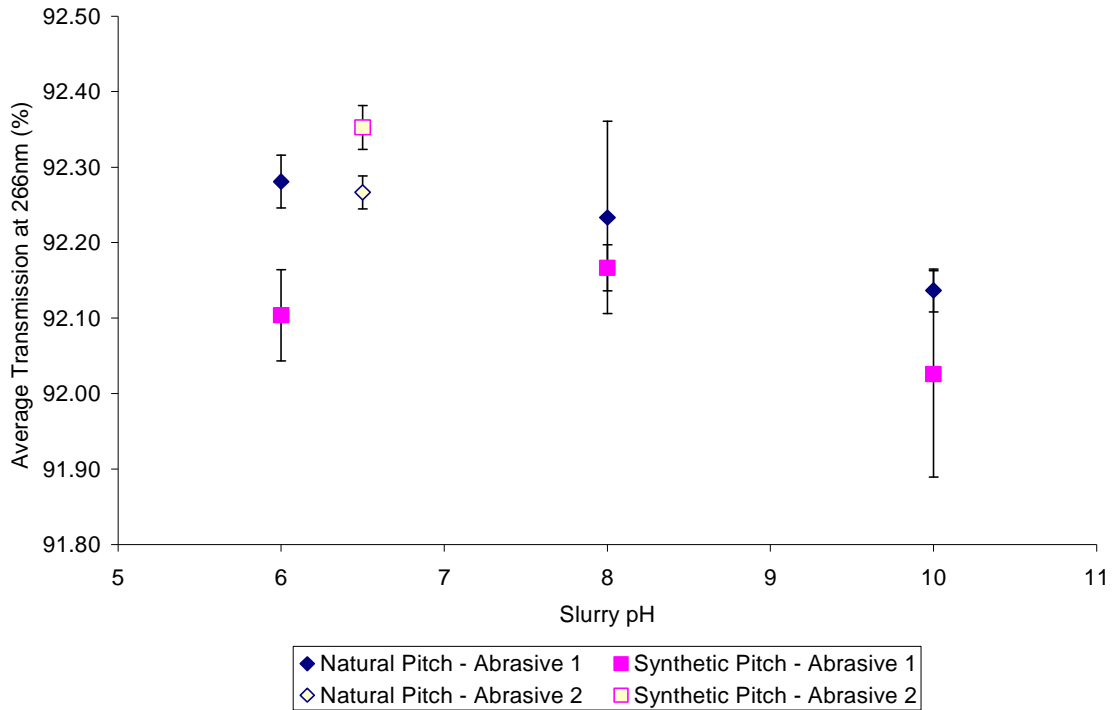


Figure 7: Average transmission of FS (bare window substrate) at 266nm as a function of slurry pH

Light incident on a surface will either transmit, reflect, scatter, or absorb. A bare substrate (measured here), will lose approximately 4% at each surface from reflection. The majority of this loss will be recovered with the application of anti-reflection coatings. The work presented here is focused on ensuring the highest possible transmission of the bare substrate to allow for the highest possible transmission of a final coated surface. The data presented in Figure 7, supports our hypothesis that the surfaces polished with Abrasive 2 have the higher transmission levels compared to surfaces polished with Abrasive 1 due to the high absorption of Abrasive 1 at 266nm. All of the surfaces were thoroughly cleaned with a standard lens cleaning procedure, and inspected to make sure that there was no residual abrasive/slurry contamination on the surface. The hypothesized absorption due to the abrasive type is not due to large abrasive particles but rather small nano-size particles not visible by the naked eye, or 20x magnification. These nano-size absorbers are hypothesized to be tightly adhered to the surface in such a way that traditional cleaning methods will not be able to remove them without mechanical abrasion. The cleaning portion of this work, referred to as Phase 3, will be the next phase of this study.

In addition to absorption, it is also important to consider the effect of surface scatter (reflection) on the transmission quality of an optic. The average transmission at 266nm is plotted versus the average areal RMS surface roughness in Figure 8. The first observation from Figure 8 is that the surfaces polished with Abrasive 2 have higher transmission compared to those polished with Abrasive 1 for similar surface roughness values, as was shown in Figure 7. The second observation is that there is a trend, where the smoother the surface, the higher the transmission. This shows a direct relationship between the amount of light lost to scatter to the surface roughness value, a relationship that is very intuitive and especially important to applications in the UV.

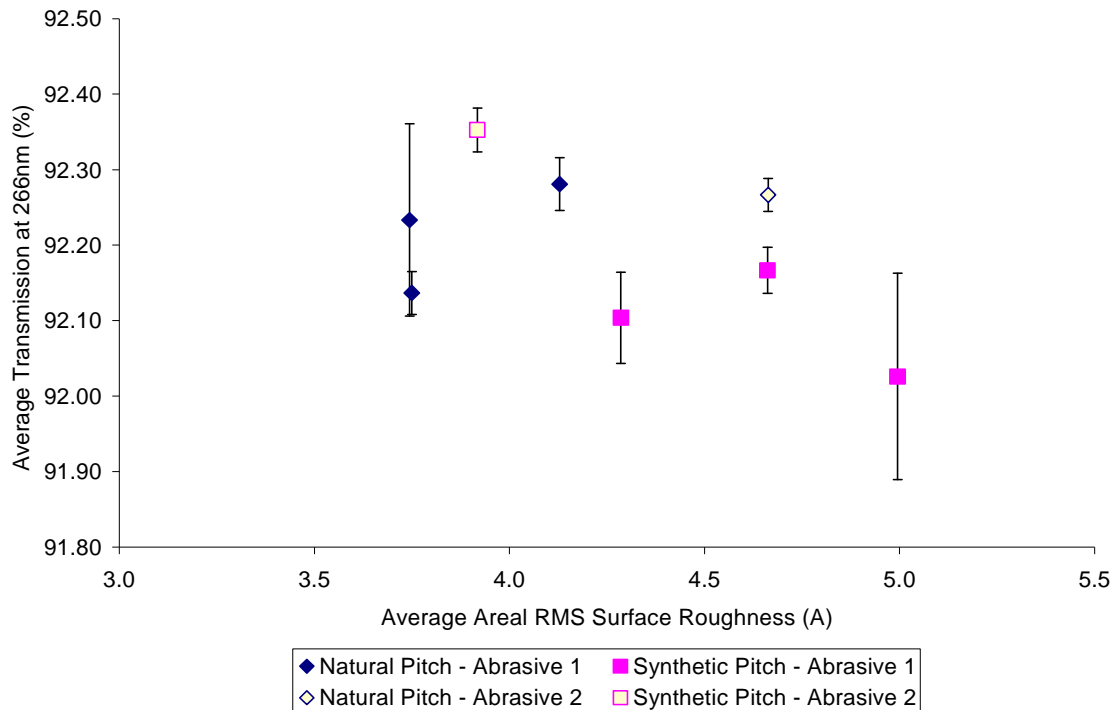


Figure 8: Average transmission of FS (bare window substrate) at 266nm as a function of surface roughness

6.0 CONCLUSION/FUTURE WORK

The intent of this project is to increase the performance of UV optics in a four-phase project. The first phase consists of characterizing sub-surface damage using destructive methods to enable process control, the second phase (presented here) focuses on polishing methods, the third phase will include cleaning and possible etching protocols and the fourth phase will be improving thin film coating performance.

The amount of sub-surface damage was measured destructively and correlations were made to non-destructive tests to allow for in-process measurements of sub-surface damage. A polishing study is underway that to date, has shown a dependence on abrasive type and pitch type to material removal rate, surface roughness and transmission.

Future work will include completion of the polishing study and then work will start on a cleaning protocol followed by a study to improve the thin film coating performance for UV optics.

7.0 ACKNOWLEDGEMENTS

The sub-surface damage study was performed by Joshua Briggs during his summer co-op as part of the Kettering University Engineering program. In addition, Stephen Goodridge performed the initial polishing experiments for this project during his summer internship. Stephen is now attending The Institute of Optics Masters program at the University of Rochester.

8.0 REFERENCES

1. N. Bloembergen, "Role of Cracks, Pores, and Absorbing Inclusions on Laser Induced Damage Threshold at Surfaces of Transparent Dielectrics," *Applied Optics* **12**, 4, 661-664 (1973).
2. J. Neauport, L. Lampaignere, H. Berceogol, F. Pilon, and J.-C. Birolleau, "Polishing-induced contamination of fused silica optics and laser induced damage density at 351 nm," *Optics Express* **13**, 25, 10163-10171 (2006).
3. J. Yoshiyama, F. Y. Genin, A. Salleo, I. Thomas, M. R. Kozlowski, L. M. Sheehan, I. D. Hutcheon, and D. W. Camp, "A Study of the Effects of Polishing, Etching, Cleaving, and Water Leaching on the UV Laser Damage of Fused Silica," in *29th Annual Symposium on Optical Materials for High Power Lasers*, (Boulder, CO, 1997).
4. D. W. Camp, M. R. Kozlowski, L. M. Sheehan, M. Nichols, M. Dovik, R. Raether, and I. Thomas, "Subsurface damage and polishing compound affect the 355-nm laser damage threshold of fused silica surfaces," in *29th Annual Boulder Damage Symposium*, (Boulder, CO, 1997).
5. J. Neauport, D. Valla, J. Duchesne, P. Bouchut, L. Lampaignere, J. Bigarre, and N. Daurious, "Building high damage threshold surfaces at 351nm," in *Optical Fabrication, Testing and Metrology*, R. Geyl, D. Rimmer, and L. Wang, eds. (SPIE, 2004), pp. 131-139.
6. M. J. Cumbo, D. Fairhurst, S. D. Jacobs, and B. E. Puchebner, "Slurry Particle Size Evolution during the Polishing of Optical-Glass," *Applied Optics* **34**, 19, 3743-3755 (1995).
7. Y. Zhou, P. D. Funkenbusch, D. J. Quesnel, and A. Lindquist, "Effect of etching and imaging mode on the measurement of subsurface damage in microground optical glasses," *Journal of American Ceramic Society* **77**, 12, 3277-3280 (1994).
8. J. C. Lambropoulos, Y. Li, P. Funkenbusch, and J. L. Ruckman, "Non-contact estimate of grinding-induced subsurface damage," in *Optical Manufacturing and Testing III*, (SPIE, Denver, CO, 1999), pp. 41-50.