

DAMAGE-FREE PROCESSING OF SILICON CARBIDE OPTICS

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ABSTRACT

Mechanical grinding and shaping of optical materials imparts damage that manifests itself as defects and cracks that can propagate well below the surface of the optic. Mitigation of damage is necessary to preserve the integrity of the optic and to relieve residual stress that can be detrimental to its performance. This damage can also cause instability in optical performance with changes in the ambient conditions. In certain high laser fluence applications or under transient stresses, it can also lead to catastrophic failure. This residual stress is usually relieved by grinding with progressively smaller grit sizes but often times this process will still leave behind a small amount of stress from the final polishing step.

Reactive Atom Plasma (RAP), a novel non-contact, atmospheric pressure plasma-based process, has been shown to reveal and mitigate sub-surface damage in optical materials. Twyman stress tests on thin glass and Silicon Carbide (SiC) substrates demonstrate RAP's ability to relieve this stress. We will show how RAP processing can enhance and accelerate fabrication of precision SiC optics.

INTRODUCTION

Optics fabrication relies on various machining, grinding and polishing steps that remove material through either brittle or ductile mode shearing [1]. This not only leaves behind a fractured surface but most of those fracture points have cracks that extend well below the surface. This is usually referred to as sub-surface damage (SSD). The presence and amount of SSD influences the amount of time required to polish the optic to final specification not only because SSD can create surface defects but also because of the high state of stresses in the damaged zone. SSD can also be a limiting factor in the ultimate tensile strength

of brittle materials by providing nucleation sites for catastrophic failure [2].

Sub-surface damage is typically reduced through a series of grinding steps using successively finer grit sizes that remove the damage from prior grinding step but leave behind a thinner damage zone which has to be removed in subsequent grinding steps. Opticians often use rules of thumb such as removing 3x the depth of material corresponding to the previous grit size used. However, depending on the process parameters, there is damage that is lurking deeper than this. Final polishing with sub-micron abrasive particles is thought to prevent the introduction of new damage but will leave behind a surface that has substantial residual stresses from plastic deformation [3, 4].

Sub-surface damage can be detected and qualitatively assessed using a variety of techniques including ball-dimple tests, optical and acoustical microscopy and a variety of other non-destructive tests. The stress associated with SSD can be quantitatively determined by measuring the change in shape of thin disks before and after a grind step. This type of analysis is referred to as Twyman analysis and was first observed by an Englishman by the same name in the early 20th century [5].

SiC and SSD

SiC is a material finding increased adoption in the optical community due to a number of reasons. It has excellent thermal and mechanical properties with a specific stiffness of >120 N-m/KG (second only to Beryllium and without the accompanying toxicity issues). Its thermal stability is better than Zerodur in environments where there is rapid and large fluctuation in temperatures. However, its widespread adoption has been limited by manufacturability issues. SiC's hardness is such that the only known

technique to mechanically polish the surface is to use diamond based abrasives and employ high contact forces. This process ends up putting a significant amount of sub-surface damage into the substrate and, in the case of lightweight optics, the high contact force causes print-through. In the case of high-performance glass optics, a wet acid-etch step is sometimes inserted between hard machining steps in order to arrest further propagation of sub-surface damage. In the case of SiC, the only known (hazardous and slow) chemistry for an etching step is using molten potassium hydroxide at an elevated temperature.

Rapid damage removal using Reactive Atom Plasma (RAP™) processing

Reactive Atom Plasma processing is a novel manufacturing technology with the potential to significantly reduce the time and cost of manufacturing optics [6]. The process uses an atmospheric pressure inductively coupled Ar plasma to breakdown CF₄ gas into carbon and fluorine atoms and is discussed in detail in a companion paper [10].

EXPERIMENTAL PROCEDURE

For these tests thin disks (75mm diameter by 2mm nominal thickness) of fused silica (Corning 7980) and sintered SiC (CoorsTek SC30) were used. Both sides of each disk were ground on a 3 micron fixed abrasive lap to remove about 250 microns of material so that any damage from prior process steps is removed. The glass disks were then acid etched in a buffered HF solution to remove 50-60 microns of stock from both sides. In the case of SiC, the RAP process was used to strip off about 20 microns of material on both sides. At this point the disks had no sub-surface damage and no residual stress.

The front side of each disk (designated S1) was measured for shape either on a phase shifting interferometer (in case of fused silica) or an optical profiler microscope (in case of SiC). Samples were then ground on the back side (designated S2) using down pressures of 0.6 to 2.6 psi on fixed abrasive laps with either 9 micron or 3 micron grit. A total of 25 microns was removed in this grinding step. The grinding step produced a uniform compressive stress in S2 which caused it to bend in a convex shape, resulting in a concave shape change on S1.

The shape change on S1 was measured. This change in shape is radially symmetric and is commonly referred to as sag. Opticians refer to it as power change. Subsequently a RAP removal was performed on the back side to remove a small amount of damage layer. This caused a slight stress relaxation and a resultant change in sag. This change in sag of S1 was again measured. This procedure was repeated until further RAP removals didn't change the shape any further. The cumulative amount of RAP removals (based on weight loss measurements) gives the depth of SSD.

The Stoney equation can be used to correlate the change in sag of a disk to a change in the state of surface stress [6] where:

$$\frac{4 E d^2}{3 (1 - \nu) t_{ssd} D^2}$$

= Twyman stress (MPa)

= Poisson's ratio

E = Young's Modulus (MPa)

t_{ssd} = Damage layer thickness (nm)

= Circular plate deflection (nm)

d = Substrate thickness (mm)

D = Substrate diameter (mm)

This is the biaxial form of the equation and is valid for aspect ratios greater than 1:50 between depth of SSD and thickness of the substrate. The equation above further approximates the radius of curvature and is valid for small plate deflection. t_{ssd} is determined by the depth of removal required to restore the disk to its original pre-ground shape.

RESULTS

Using the Stoney equation and plugging in the total depth of SSD (t_{SSD}) the stress change after each RAP hit was determined based on the amount of sag change. The following results are shown as plots of change in stress as a function of RAP assisted material removal.

Results on glass

Figure 2 shows the decrease in stress as a function of RAP removal in glass disks subjected to grinding at 0.6, 1.3 and 2.6 psi

on a 9 micron fixed abrasive lap. The resulting roughness after lapping on the 9 micron lap was 140-150 nm Ra and did not vary with downward pressure. The surface stress from grinding with the 9 micron lap ranges from 25-30 MPa for all samples: no variation in initial stress is observed as a function of grinding pressure. The depth of SSD is around 10 microns in the surfaces ground at 0.6 and 1.3 psi but the surface ground under 2.6 psi of pressure shows stress and damage extending at least 16 microns into the material. In general, the relaxation of stress is not linear – the initial few RAP removals remove more stress than later RAP steps.

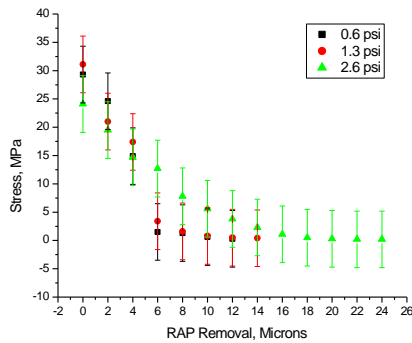


Figure 2. Residual stress in fused silica glass ground on 9 micron fixed abrasive lap under various pressures as a function of material removed by RAP processing.

Results on SiC

Figure 3 shows the decrease in stress as a function of RAP removal in a SiC disk ground under 1.3 psi using a 9 micron fixed diamond abrasive lap.

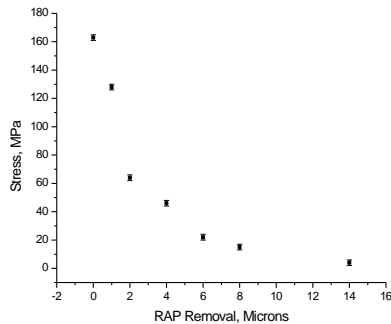


Figure 3. Residual stress in Coorstek SC30 SiC ground on 9 micron fixed abrasive lap under various pressures as a function of material removed by RAP processing.

Our results on glass are in accordance with a number of prior observations and measurements of damage in brittle materials [4]. The depth of SSD (and associated stress) correlates with the pressure during lapping.

DISCUSSION

The roughness produced by this lapping step was 170-200 nm Ra. The post-grinding state of stress is 160 MPa and falls off with depth in a relationship similar to that observed in the glass disks. The depth of SSD is roughly 14 microns.

Like previous studies, our results on both glass and SiC suggest that stress is not uniformly distributed within the damage zone but rather is concentrated at the surface and falls off with depth in a rough power law relationship. Similar behavior is observed in the distribution of micro-cracks in Corning 7980 fused silica ground under a variety of conditions [9].

Since the RAP process is chemical in nature it works by attacking the most (chemically) active sites first, so rather than remove a specific depth of material it would remove the most damaged and stressed material first. This makes it difficult to derive an exact relationship between stress and depth of SSD from our data.

We observe that under comparable lapping conditions SiC exhibits about 5.5x the stress associated with SSD compared to glass. As shown in earlier studies the magnitude of stress is dependent on physical material properties of the substrate and SiC has a modulus of elasticity that is about 5.5x higher than glass (410 GPa vs. 72.1 GPa).

Despite the differences in physical properties between glass and SiC, the thickness of the damage layer in this variety of SiC appears to be comparable to that in glass lapped under similar conditions. This suggests that using an all conventional process, opticians will have to remove at least as much material in SiC as in glass to achieve a surface with low damage. Using higher downward force during grinding may increase the material removal rate but it will also increase the depth of damage and necessitate larger volumes of stock removal

during subsequent process steps. Thus, higher pressure grinding may not reduce overall manufacturing cycle times for SiC optics at all but will increase the risk of catastrophic failure of the optical substrate during optical fabrication.

CONCLUSIONS

Residual stress in ground surfaces of SiC are roughly a factor of 5 times higher than those measured in glass ground under identical conditions. The depth of subsurface damage in ground SiC can extend at least as deep as that observed in ground glass. Manufacturing precision, low-damage SiC optics using purely conventional processes will thus require extremely long cycle times.

Irrespective of the magnitude or origin of the stresses observed in these materials, we have demonstrated that RAP can be used to quickly and deterministically mitigate the stress by removing the damage that causes it. In the case of glass, RAP provides an alternative to buffered etching – with the added advantage of allowing for localized removal in areas of concentrated damage as well as shape control and figure improvement.

In the case of SiC, RAP is the only method we are aware of that can rapidly and cost-effectively remove the damage caused by abrasive grinding while simultaneously improving the shape of the optical surface, thus greatly reducing the time required to finish high-performance SiC optics

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