

Rapid damage-free shaping of silicon carbide using Reactive Atom Plasma (RAP) processing

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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 relieve residual stress that can be detrimental to its performance. Typically, a sequence of subsequent polishing steps with finer and finer grit sizes is used to remove damage, but the process can be painfully slow especially for hard materials such as silicon carbide and often fails to remove all the damage. Reactive Atom Plasma (RAP™) processing, a 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 SiC substrates demonstrate RAP's ability to relieve the stress while at the same time improving surface form.

Keywords: reactive atom plasma, silicon carbide, RAP, sub-surface damage, optics

1. INTRODUCTION

The cost-effective manufacture of large optics is a fundamental requirement for the next generation of ground and space-based astronomical telescopes. While there have been an enormous range of improvements in the manufacturing processes for large optics over the last 40 years, the primary optic remains a principal cost, schedule and risk driver.

A fundamental contributor to the cost and schedule of large optics fabrication is the fact that these precision components must be fabricated from relatively hard, brittle materials such as glass or ceramics. The machining processes used to shape these materials involve numerous cycles of mechanical (grinding) steps and chemical-mechanical (polishing) steps.

1.1 Sub-surface damage: an inevitable consequence of brittle machining

Because of the brittle nature of glass and ceramics, sub-surface damage (SSD) is an inevitable consequence of modern optical fabrication processes [1]. The presence and amount of SSD can greatly influence the time required to achieve final optical specification not only because SSD can create surface defects during polishing but also because of the state of stress in the damage zone. SSD also can exert a strong control on the tensile strength of brittle materials because the microcracks and defects that constitute SSD can lower the strength of optical substrates by providing an abundance of nucleation sites that can grow catastrophically during transient stress [2]. Even with the tremendous care that is taken during large optics fabrication, testing, coating and installation, the chances that at least one damage site will "run away" into a through-going crack is significant. A number of large optics now installed in astronomical observatories have at least one macroscopic crack that required emergency mitigation during fabrication or testing. The catastrophic failure of a large optic during final finishing is a nightmare scenario for any program manager or research consortium. However the failure of a large optic to meet final figure or roughness specification can be nearly as devastating to the budget and schedule of a large astronomical program.

Sub-surface damage can be reduced through successive grinding (and polishing) with smaller and smaller grit sizes. Opticians often use rules of thumb such as removing 3x the depth of material corresponding to the size of the previous grit to eliminate the damage created by the prior grinding step. However considerable damage can lurk deeper than this

depending on the previous process steps. Polishing with fine ($< 1\sigma\text{m}$) abrasives is thought to prevent the introduction of new damage but nevertheless can leave a surface that is under substantial residual stresses [e.g. 3, 4]

Sub-surface damage can be detected and qualitatively assessed using a variety of techniques including wet chemical etching, microscopy, indentation and stress measurements [5]. The stress associated with SSD can be quantitatively determined by measuring the change in shape of a thin disk as the SSD layer is removed [6]. Etching with various acids has been used to relieve such stress and open up and arrest SSD – a process commonly referred to as “stabilizing” the optical substrate. This is commonly used in the manufacture of optics from glass or glass ceramics.

1.2 Silicon carbide and SSD

Silicon carbide (SiC) is an extremely attractive material for reflective optics for large ground and space-based telescope systems. SiC combines a high stiffness to weight ratio along with thermal properties far superior to beryllium which combine to make it an ideal mirror substrate material. Several SiC manufacturers have demonstrated the ability to rapidly manufacture lightweight substrates and supporting structures out of SiC, enabling the design of complex lightweight athermal optical systems [7].

The same properties that make SiC so attractive as a material for telescope optics also make it a manufacturing challenge. SiC’s high hardness requires diamond grinding tools and abrasives for shaping and polishing, often applied for prolonged periods of time. The high downward pressures required to effectively use these tools produces substantial sub-surface damage and stress. Because SiC is chemically inert to acids and solvents at room temperature there is no means to easily arrest the damage and stress caused by grinding using a chemical process. Unlike glass, which can be polished with a chemical-mechanical process, traditional polishing of SiC is almost entirely mechanical in nature.

1.3 Rapid damage removal using Reactive Atom Plasma (RAP™) Processing

Reactive Atom Plasma (RAP™) processing is a novel optical manufacturing technology with the potential to significantly reduce the time and cost of manufacturing astronomical optics. The process uses inductively coupled RF power to excite an argon plasma at atmospheric pressure. A non-reactive precursor chemical, in this case, CF_4 , is injected into the plasma and is broken down into individual carbon and highly reactive fluorine atoms (see Figure 1).

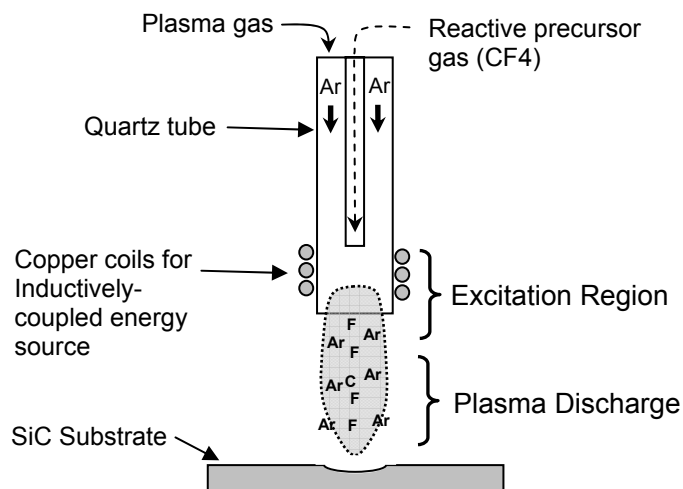


Figure 1. Schematic diagram of RAP tool. The reactive precursor gas is converted to highly reactive atomic radicals as it passes through the plasma excitation region.

The stream of reactive atoms is directed onto the surface of the substrate (in this case, SiC) and the silicon is chemically converted to SiF₄ and the carbon is converted to CO₂ and both are removed as a gas (see Figure 2). The surface left behind is undamaged and contaminant-free.

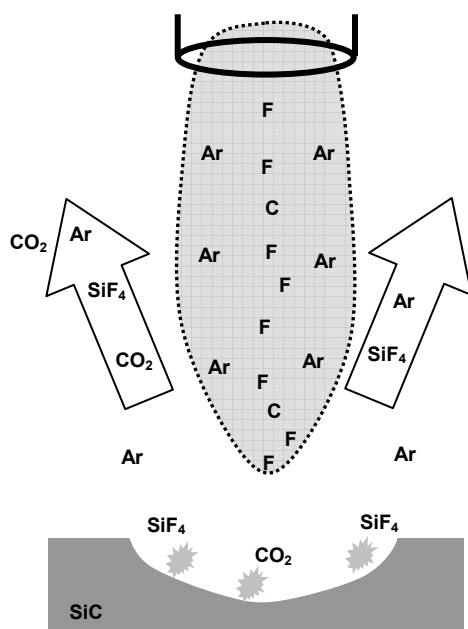


Figure 2. RAP removes material by chemically converting the solid material into a gas.

In this study we used RAP as a damage removal tool to assess the magnitude and distribution of sub-surface damage in glass and SiC optical substrates shaped by fixed abrasive lapping. RAP can be used both for bulk etching to reveal the presence of SSD, or as a precision damage removal tool to incrementally remove the damage layer across an optical substrate.

2. EXPERIMENTAL PROCEDURE

Thin disks (75 mm diameter by 2 mm thick) of fused silica glass (Corning 7980) and sintered SiC (Coorstek SC30) were obtained for this experiment. Both sides of each disk were ground on a 3 micron fixed abrasive lap to remove 200-250 microns of material and damage from previous process steps. The glass disks were then immersed in a buffered HF solution to remove 50-60 microns of material from all surfaces. The SiC disks were etched on both sides with RAP to remove at least 20 microns of material.

The frontside of each disk (designated as S1) was measured for flatness using either a phase shift interferometer (for glass) or using a Zygo New View 5000 white-light interferometer microscope (for SiC). Samples were then weighted to impose 0.6 to 2.6 psi of downward pressure and ground using either a 9 micron or 3 micron fixed diamond abrasive lap to remove 20 to 25 microns of material from the backside (designated as S2). The frontside shape (S1) of each disk was re-measured. The roughness produced on the S2 surface was also measured using a Zygo NewView 5000. The grinding step produced compressive stress in S2 which caused the S1 surface to bend to a concave shape. The shape change was measured and then RAP was used to remove an increment of material (typically 2 microns) off S2. The S1 surface was re-measured and the process was repeated until the S1 surface relaxed back to its original stress-free state and subsequent RAP removals produced no change in shape.

3. RESULTS

The change in shape of a disk can be directly and quantitatively related to a change in the state of surface stress through the Stoney equation [6] where:

$$S = \frac{4 C E d^2}{3(1-\nu)\#t_{SSD} D^2} \quad (i)$$

- S = Twyman stress (in MPa)
- $\nu\#$ = Poisson's Ratio
- E = Young's Modulus (Kgf/mm²)
- t_{SSD} = Damage layer thickness (nm)
- d = Substrate thickness (mm)
- D = Substrate Diameter (mm)
- C = Circular plate deflection (nm)#

The damage layer thickness is determined by the depth of material required to restore the disk to its original pre-ground shape. The circular plate deflection is simply the change in shape between process steps.

3.1 Results on glass

Figure 3 shows the decrease in stress with depth in glass disks subjected to grinding at 0.6, 1.3 and 2.6 psi using a 3

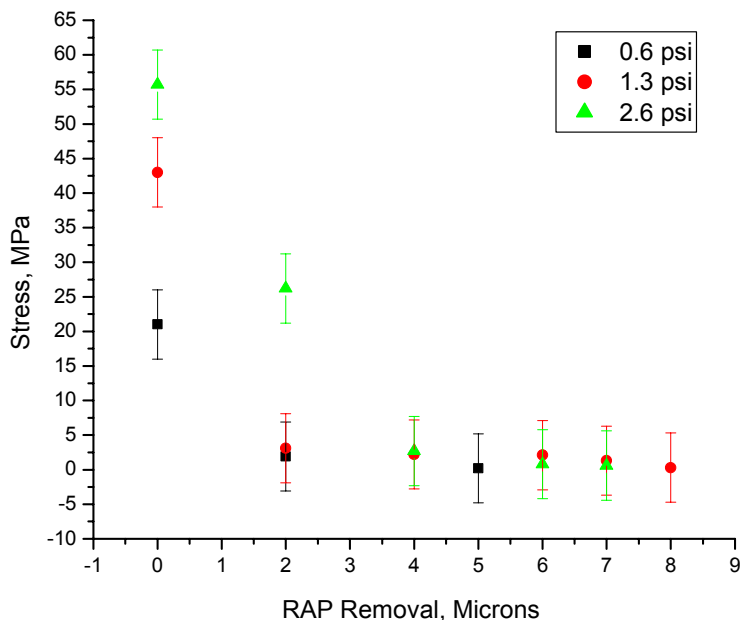


Figure 3. Residual stress in fused silica glass ground on 3 μm fixed abrasive lap under various pressures as a function of material removed by RAP processing.

micron fixed abrasive lap. The roughness produced by this lapping step was 90-100 nm Ra and did not change as a function of downward pressure. The surface stress prior to RAP treatment varies from 20 MPa for the disk ground at 0.6 psi to 50 MPa for the disk ground under 2.6 psi pressure. The thickness of the damage layer also varies from 2-3 microns for samples ground at 0.6 and 1.3 psi to 4-5 microns for the samples ground under 2.6 psi pressure.

Figure 4 shows the decrease in stress with depth in glass disks subjected to grinding at 0.6, 1.3 and 2.6 psi using a 9 micron fixed abrasive lap. The roughness produced by this lap was 140-150 nm Ra and did not vary with downward pressure. The surface stress prior to RAP treatment is 25-30 MPa for all samples: no variation in initial stress is observed as a function of grinding pressure. The thickness of the damage layer 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 monotonic – the first RAP removal removes more stress than subsequent RAP steps.

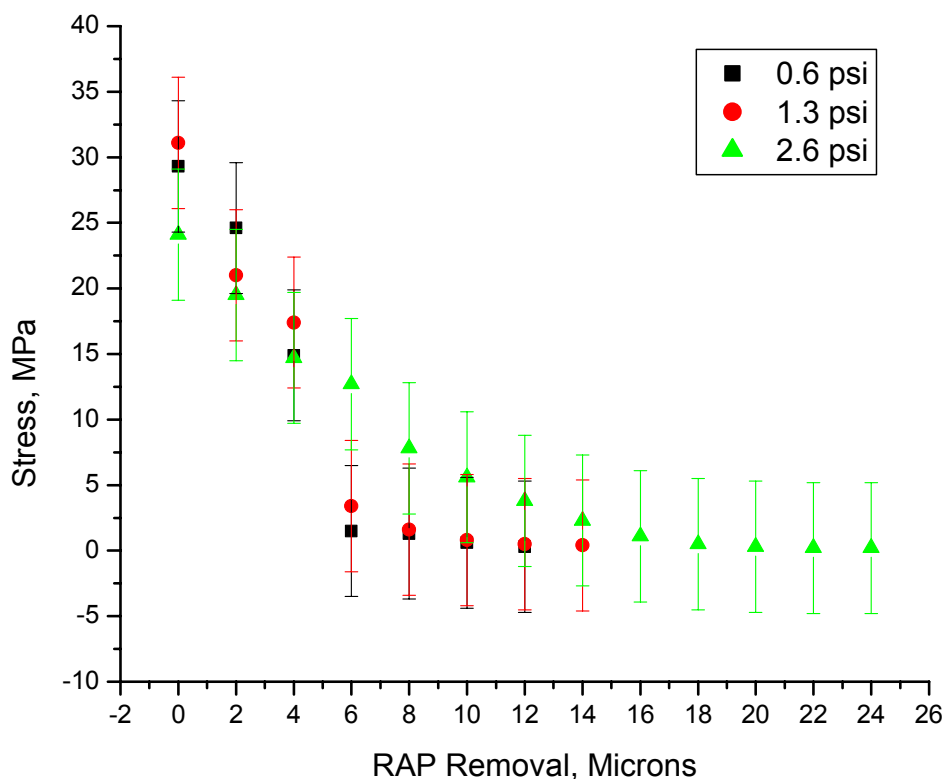


Figure 4. Residual stress in fused silica glass ground on 9 μ m fixed abrasive lap under various pressures as a function of material removed by RAP processing.

3.2 Results on SiC

Figure 5 shows the decrease in stress with depth in a SiC disk ground under 1.3 psi downward force using a 9 micron fixed diamond abrasive lap. The roughness produced by this lapping was 170-200 nm Ra. The initial 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 stress is roughly 14 microns.

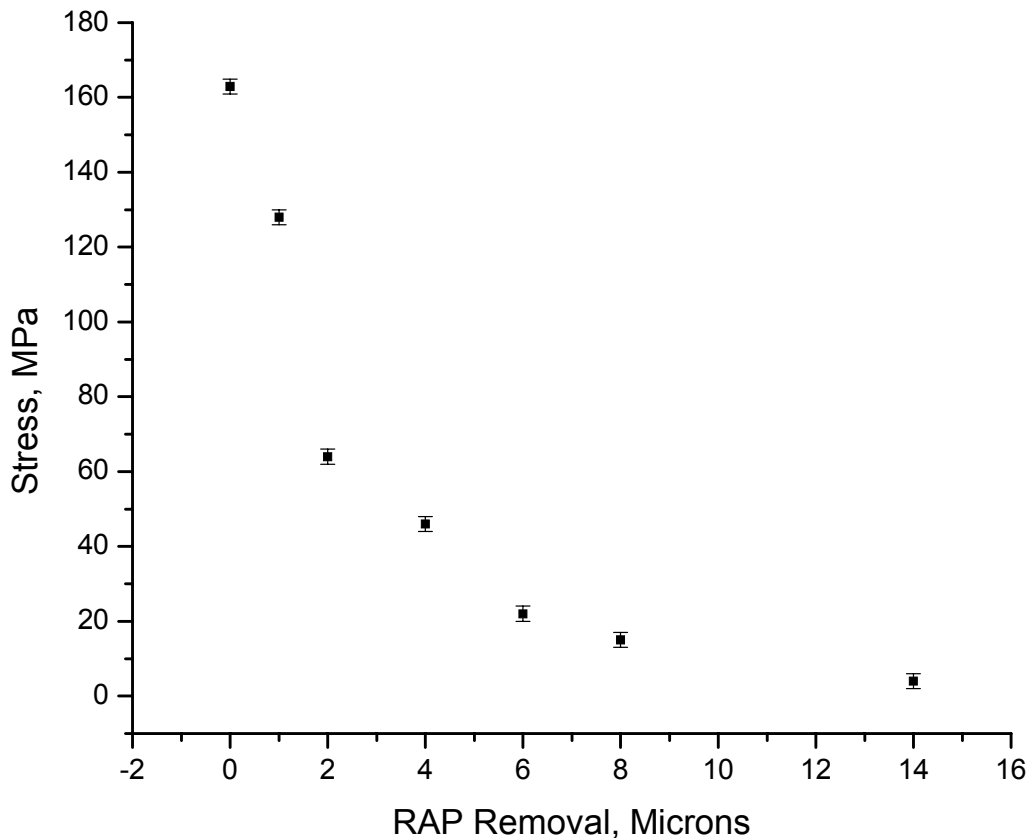


Figure 5. Residual stress in Coorstek SC30 SiC ground on 9 μm fixed abrasive lap under various pressures as a function of material removed by RAP processing.

4. DISCUSSION

Our results on glass are in accordance with a number of prior observations and measurements of damage in brittle materials. The depth of stress (and SSD) correlates with the pressure during lapping and the size of the abrasive used. However the magnitude of stress is inversely proportional to the abrasive size for the range investigated here. Lambropoulos [4] observed similar trends in data on fixed and loose abrasive ground glass. According to his model, different sized abrasives create a comparable amount of plastic damage during material removal and the amount of plastic damage is mostly controlled by the mechanical properties of the substrate material. But for surfaces ground with finer abrasives, the stress associated with this plastic damage is distributed across a thinner damage layer, creating a stronger gradient of stress and higher surface stresses.

Like previous studies, our results on both glass and SiC suggest that stress is not uniformly distributed within the sub-surface 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 [8].

Deriving an exact relationship between stress and depth from our data may be complicated by the fact that the RAP process is chemical in nature. Rather than precisely removing a specific depth of material, the plasma will

preferentially etch the most chemically active surface sites first which will coincide with the most damaged and stressed material. Thus the first few plasma exposures may release a disproportionate amount of stress.

We observe a roughly 5-fold higher level of stress in lapped SiC than glass. As noted by earlier studies, the magnitude of residual stress is largely controlled by the material properties of the substrate: higher strength materials require more force to remove material during grinding and can sustain larger residual stresses after lapping [4]. In comparison to Corning fused silica glass, the compressive strength of SC30 SiC is a factor of 3 higher (3.5 GPa vs. 1.14 GPa), the flexural strength is a factor of 9 higher (480 MPa vs. 52.4 MPa), and the elastic modulus is a factor of 5.6 higher (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. Thus, in order to manufacture precision low-damage SiC optics using traditional manufacturing processes, significant stock removal (comparable to that required for glass) using successively finer abrasives is required. 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 times for SiC optics at all and will increase the risk of catastrophic failure of the optical substrate during optical fabrication.

5. 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 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 and 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 at areas of concentrated damage as well as a 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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