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Effect of rogue particles on the sub-surface damage of fused silica during grinding/polishing

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Abstract

The distribution and characteristics of surface cracks (i.e., sub-surface damage or scratching) on fused silica formed during grinding/polishing resulting from the addition of rogue particles in the base slurry has been investigated. Fused silica samples (10 cm diameter × 1 cm thick) were: (1) ground by loose abrasive grinding (alumina particles 9–30 μm) on a glass lap with the addition of larger alumina particles at various concentrations with mean sizes ranging from 15 to 30 μm, or (2) polished (using 0.5 μm cerium oxide slurry) on various laps (polyurethane pads or pitch) with the addition of larger rogue particles (diamond (4–45 μm), pitch, dust, or dried Ceria slurry agglomerates) at various concentrations. For the resulting ground samples, the crack distributions of the as-prepared surfaces were determined using a polished taper technique. The crack depth was observed to: (1) increase at small concentrations ($>10^{-4}$ fraction) of rogue particles; and (2) increase with rogue particle concentration to crack depths consistent with that observed when grinding with particles the size of the rogue particles alone. For the polished samples, which were subsequently etched in $HF:NH_4F$ to expose the surface damage, the resulting scratch properties (type, number density, width, and length) were characterized. The number density of scratches increased exponentially with the size of the rogue diamond at a fixed rogue diamond concentration suggesting that larger particles are more likely to lead to scratching. The length of the scratch was found to increase with rogue particle size, increase with lap viscosity, and decrease with applied load. At high diamond concentrations, the type of scratch transitioned from brittle to ductile and the length of the scratches dramatically increased and extended to the edge of the optic. The observed trends can be explained semi-quantitatively in terms of the time needed for a rogue particle to penetrate into a viscoelastic lap. The results of this study provide useful insights and 'rules-of-thumb' relating scratch characteristics observed on surfaces during optical glass fabrication to the characteristics of the rogue particles causing them and their possible source.
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1. Introduction

The creation of sub-surface mechanical damage (SSD) (i.e., surface micro-cracks) can be thought of as the repeated indentation of mechanically loaded hard indenters (abrasives) sliding on the surface of a brittle substrate (workpiece) during various cutting and grinding processes. These surface cracks are commonly identified as scratches and digs. During grinding operations, the removal of material is governed by the intersection of multiple surface cracks.

For static indents, the loads needed to initiate fracture (lateral, radial, Hertzian) are finite and can be analytically expressed in terms of the size of particle for blunt indentors and material properties for sharp indentors [4]. The critical load for a sharp indentor to create fracture in fused silica is ~0.02 N [4]. For sliding indents (i.e., leading to scratching), the types of features (trailing indent cracks, median cracks, lateral cracks, and plastic deformation/compaction) [4,9,10]

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are a function of the local shape of the particle at contact (sharp vs blunt) and the applied load. At low loads ($P < 0.05$ N) a plastic trench is formed without fractures. At intermediate loads ($0.1\ \mathrm{N} < P < 5$ N) well defined radial (or trailing indent) fractures along with lateral cracks are observed. At higher loads ($P > 5$ N), the plastically deformed track fractures into a rubble-like appearance, and lateral and trailing indent cracks are less pronounced.

In contrast, the general material removal mechanism responsible for polishing is less straightforward. Various mechanisms have been proposed including surface melting [1], plastic removal or abrasion [2,3], brittle fracture [4,5], adhesion [6], and chemical [2]. However, the most widely accepted mechanism involves the removal of material by a chemical reaction between the polishing particle and the substrate resulting in molecular level removal [2,5,7,8].

One aspect of the transition from grinding to polishing can be thought of in terms of the decrease in particle size of the slurry and the subsequent decrease in the load/particle. During grinding larger particles are used (greater than ~10 μm), resulting in fewer loaded particles per unit area between the workpiece and the lap and thus, resulting in loads/particle that exceed the initiation load for fracture. During polishing smaller particles are used (less than 3 μm), resulting in more loaded particles per unit area and loads/particle below the fracture initiation load. For a typical ceria based polishing slurry (0.5 μm), assuming a 0.3 fill fraction at the interface and all the particles are load bearing, one estimates a load/particle of 10^{-9}–10^{-6} N. Clearly, the load on an average polishing particle is many orders of magnitude lower than needed to initiate fracture. Thus any scratch formed on a polished surface implies a particle (i.e. a larger (rogue) particle) that is holding a much higher load than the average particle.

The presence of rogue (i.e. large) particles in the slurry during grinding or polishing is known to strongly influence surface properties of the workpiece either in terms of deeper damage or isolated scratching [2,4,11]. Several studies for polishing integrated circuits have investigated the effects of rogue particles for wafers ground or polished using chemical mechanical polishing. Basim et al. [12] spiked colloidal silica slurries with rogue silica particles and found that scratch densities increased with rogue particle size and concentration. By characterizing the slurry size distribution using dynamic light scattering, Basim also illustrated that rogue particles present even at a very low concentration (<1 out 100 000) can degrade surface quality. Ahn et al. [11] and Kallingal et al. [13] compared different colloidal silica and colloidal alumina based slurries of different pH, filtering, or ultrasonic preparation to reduce micro-scratches; the results were explained in terms of how the process parameters affect the large particles in the distribution.

In our previous studies, the characteristics and statistical distribution of SSD as a function of different grinding processes using a polished taper technique have been measured and analyzed [14–16]. These results suggested that only a small fraction (1 out of tens of thousands of particles) of the abrasive particles were participating in the material removal by comparing the measured crack depth with that expected from static indentation models. Hence only a small fraction of the particles were being mechanically loaded. In the present study, the effects of rogue particle additions during both grinding and polishing and their effect on the SSD depth and the characteristics of scratching have been investigated. Understanding the impact of the presence of rogue particles during grinding and polishing can ultimately be used to develop finishing processes resulting in minimal or no SSD. This is important in areas which require high quality polished brittle surfaces including integrated circuits, high strength windows, and optics for use in high power laser applications [17].

2. Experimental

2.1. Ground samples

Round fused silica samples (10 cm diameter × 1.0 cm thick) were ground or polished using slurries that were intentionally contaminated with various concentrations and sizes of rogue particles. Ground samples were prepared on a rotary 8″ borosilicate glass lap (0.3 psi, 1 h, 15 rpm lap) using an alumina abrasive particle (Microgrit 9T or 15T) slurry. Rogue particles (Microgrit 15T or 30T) were added to the base slurry at various concentrations. The SSD distributions of the as-prepared surfaces were then determined by: (1) creating a shallow (10–100 μm) wedge/taper on the surface by magneto-rheological finishing; 2) exposing the SSD by HF/NH_4F acid etching; and (3) performing image analysis of the observed cracks from optical micrographs (Nikon Optiphot, reflectance) taken along the surface taper (see Fig. 1). Details of this characterization technique to determine SSD depth and length distributions are provided elsewhere [14–16]. Photographs of the grinding setup is shown in Fig. 2(a) and the particle size distributions of the loose abrasives used in the rogue particle experiments are shown in Fig. 3(a). The matrix of experiments for the ground samples are shown in Table 1.

2.2. Polished samples

The pitch lap was prepared by heating 500 g of Gugolz 73 ground in ~mm sized pieces in an aluminum foil covered stainless steel container (12 cm diameter × 16 cm) in a convection oven at 76.7 °C for 60 min. The pitch was stirred after 30 min and 50 min. The molten pitch was then poured immediately after removal from the oven onto a preheated (76.7 °C) aluminum plate block (20 cm diam) with tape (3M black duct) wrapped around the edge on a flat, level surface until the lap formed the desired thickness. After 10 min, that tape was removed and the groove pattern (3.8 cm triangles with 3 mm wide, 3 mm deep, 60° V-grooves) was embossed using a rubber mask. The embossing was accomplished by placing the mask on the

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