



Characteristics of hydrolyzed layer and contamination on fused silica induced during polishing

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Abstract

Optical polishing is a chemo-mechanical process which can induce a near-surface hydrolyzed layer with contamination. To date, it is not clear whether the contaminants in the hydrolyzed layer are present as a uniform dopant or as discrete particles. The purpose of the present work is to try to clarify this contamination form. A field emission scanning electron microscope (FE-SEM) was used to determine the micro-topography and composition mapping of the polished fused silica. The relative density of the hydrolyzed layer and the bulk has been assessed using a combination of focused ion beam (FIB) milling and FE-SEM. The results suggest that the dopant contamination is located in the upper layer (tens of nm thick), which has a lower density compared to the bulk. Also, discrete contaminant ‘islands’ are present in the hydrolyzed layer.

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1. Introduction

Material removal during polishing takes place because of synergistic chemical and mechanical interactions [1–5]. Characterization of chemistry process has been largely dependent on the measurement of the properties, specifically the thickness, density and hardness, of the hydrolyzed layer on optic surfaces. Investigations in semiconductor CMP processes for planarization of inter-metal dielectric SiO₂ have presented many instructive findings [6]. Nevot and Croce demonstrated the existence of a 2 nm surface region possessing lower density than the bulk, below which the density increased to a value greater than the bulk, gradually returning to the bulk density at a depth of 15–20 nm below the surface [7]. Wallace et al. claimed the presence of a 20 nm compacted region possessing increased density [8]. Trogolo and Rajan revealed chemical/structural modification of SiO₂ to be 100–200 nm from the polished surface and more heavily altered or deformed regions extending to a few tens of nm in depth [9]. In contrary, Tadjiev et al. investigated hydrated extent of silicate glasses by simply exposing them to distilled water at 22 ± 1 °C for various periods of time [10,11]. It is shown that hydration has little

if any effect on high durability glasses even at long immersion times. Whereas for low durable glasses hydration reduces the near-surface density, modulus and hardness when compared to the bulk glasses. On the other hand, residual contaminants within hydrolyzed layer have been determined using various means. Adsorption of cerium hydroxide by silica, while tearing away individual silica molecules, also brings residual cerium in the hydrolyzed layer. Kozłowski firstly revealed an exponential decay of contaminant concentration (i.e., Ce) to a depth of 100–200 nm on fused silica surfaces [12]. More recently, Neauport et al. used the inductively coupled plasma atomic emission spectrometer (ICP-AES) to determine the composition and concentrations of HNO₃/HF solution which had etched fused silica, to reveal the contaminant concentrations [13,14]. Secondary ion mass spectroscopy (SIMS) was also employed to determine the concentration of polished-induced contaminants as a function of depth within the hydrated layer. By using SIMS, Liu et al. observed a cerium and iron contaminated layer of approximately 50 nm depth, where the concentration of iron is much higher than that of cerium, on the fused silica surfaces polished with magneto-rheological finishing (MRF) process [15].

Although much work has been done to characterize the hydrolysis process during optical polishing of fused silica, it is still not clear whether the contaminants in the hydrolyzed layer

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are present in the form of uniform dopant or as discrete particles. In the present work, FE-SEM has been used to determine the micro-topography and composition mapping of the polished fused silica surface. Combined with the milling function of the FIB, composition of the hydrolyzed layer has also been determined. The results will be systematically analyzed.

2. Experimental

The Helios NanoLab™ 650 system including FE-SEM, FIB and their combined use was employed. Its through-the-lens detector possesses highest collection efficiency of secondary electrons (SE) and on-axis backscattered electrons (BSE). During SEM analysis, electrons are emitted onto the specimen surface yielding various output signals. SE are the scattered atomic electrons of the specimen from the near-surface region within several nanometers [16]. Collection of SE presents the topography, in micro- and nano-scales, of the specimen. BSEs result from the bounce of primary electrons by atomic nucleus of the specimen within the depth of several hundreds of nanometers. An important utility of BSE signal is composition mapping. The BSE intensity increases with atomic number, yielding brighter images [16].

Fused silica and K9 glass samples ($\Phi 25 \times 5$ mm) were polished on a continuous polisher using a polyurethane pad (GR-35 by Universal Photonics Inc.). Fused silica contains higher than 99.9 wt% SiO_2 in terms of silicate tetrahedrons.

Whereas the extensively used alkaline K9 glass generally comprises 69.13% SiO_2 , 10.75% B_2O_3 , 0.36% As_2O_3 , 3.07% BaO , 10.40% Na_2O , and 6.29% K_2O . Some samples then underwent wet etching of hydrofluoric acid removing a depth of 1 μm . Both polished and etched samples were detected using FE-SEM in the modes of SE and BSE, respectively. A Pt layer was initially plated on the other fused silica samples so as to prevent the hydrolyzed layer from damage by scattered ions. FIB was, then, used to mill a dent on the plated surface with a depth of several microns. Afterwards, FE-SEM focuses on the cross-section of the dent to determine composition mapping of the layer in the mode of BSE.

3. Results and analysis

Fig. 1(a)–(c) shows the composition mapping of the milled near-surface layers of polished fused silica, etched fused silica and polished K9 glass, respectively. The upper layer (right side in the images) is the sputtered platinum. In Fig. 1(a), the hydrated layer is obvious, with a thickness of less than 100 nm, below the platinum layer in lighter contrast. Furthermore, the brightness of this layer in SEM image decreases as the depth increases until the pristine zone. This indicates that the hydrated layer has a reduced density when compared to the bulk silica, and further, the reduced density increase as the layer approaches to the pristine zone. It is suggested that extent of hydrolyzation decreases as the depth is increased. The results are consistent with the investigation conducted by

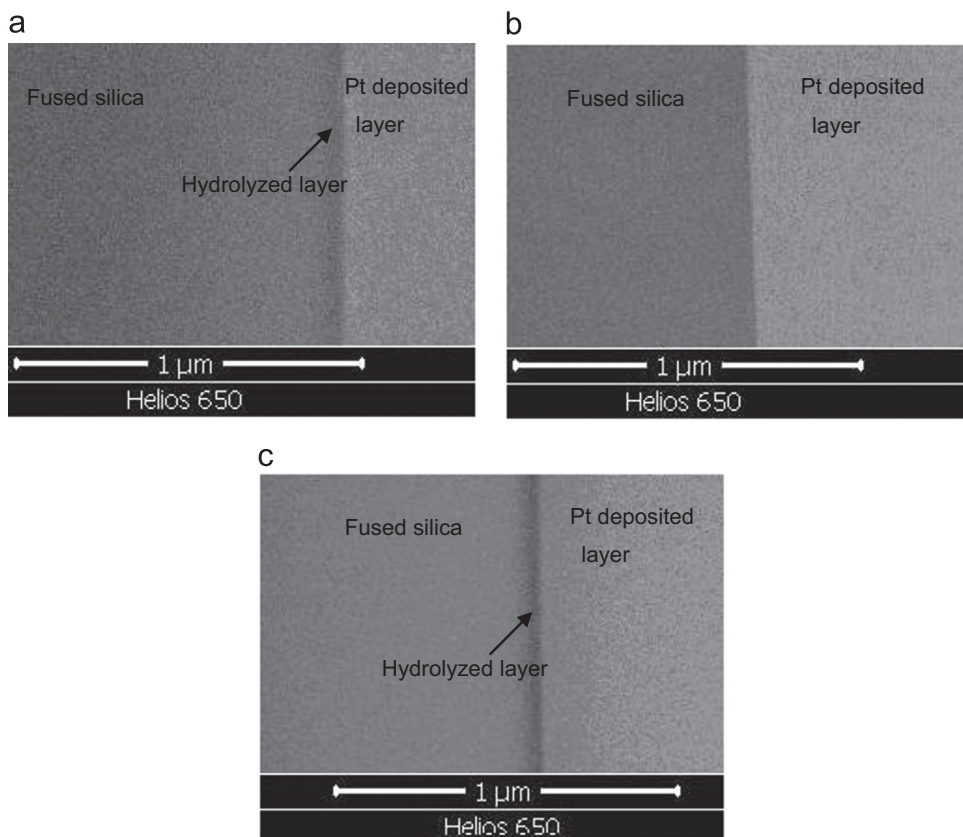


Fig. 1. Cross-section near the top surface of polished fused silica. (a) Polished surface of fused silica, (b) Etched surface of fused silica and (c) Polished surface of K9 glass.

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