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Elastic-plastic deformation in ion-exchanged aluminosilicate glass by loading rate dependent nanoindentation

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ABSTRACT

The elastic-plastic deformation in raw and ion-exchanged aluminosilicate glass is investigated by loading rate dependent nanoindentation. The nanohardness and Young's modulus of the raw and ion-exchanged glasses at different loading rate (100–20,000 μN.s^{-1}) are explored up to a maximum load of 9000 μN. The nanoindentations are scanned with AFM to observe the morphology of the indents. The results show that Young's modulus of the aluminosilicate glass increases after ion exchange and that the nanohardness of the raw and ion-exchanged aluminosilicate glass increases linearly with the loading rate, when plotted on a double logarithmic axes. However, the nanohardness of the raw glass is more sensitive to the variation of loading rate. The compressive stress on the ion-exchanged glass can inhibit plastic deformation. These results are explained in terms of shear stress underneath the indenter and the number of the flow lines in the nanoindentations. These findings are useful for better understanding the dynamic contact-induced damage growth mechanisms of ion-exchanged glass.

1. Introduction

Ion exchange, also known as chemical strengthening, is a process whereby the original glasses are immersed into a molten alkali salt at a temperature below the glass transition and this results in glass strengthening [1–4]. Ion-exchange has been used for one century to modify the surface properties of glass [5]. As the main material for fabricating touch panels of popular display devices, as well as forward facing aircraft and other windscreens, the scratch-proof property is an important requirement [3,6]. The hardness, which is apparently related to abrasion resistance, has often been used as an approximate measure of the scratch-proof resistance property [7]. Indentation is a convenient method to evaluate mechanical properties such as hardness and Young's modulus [8]. The indentation process involves the elastic-plastic deformation of materials [9,10]. According to Johnson [11,12], the indentation process can be divided into three distinct regimes, including elastic, elastic-plastic and fully plastic. The elastic-plastic transition regime is hard to understood due to its complexity of the combination of elastic and plastic deformation process. The indentation method can be applied to research the elastic-plastic transition phenomenon. For example, Wright et al. [13] indicated that the onset of plasticity during nanoindentation of a Zr-based bulk metallic glasses (BMGs) occurred at a discrete displacement burst (named as “pop-in”). The elastic-plastic transition on single crystals of platinum was studied statistically with nanoindentation as a function of temperature and indentation rate by Mason et al. [14]. The “pop-in” phenomenon which is related to the elastic-plastic transition regime in indentation experiments is also observed in ceramic and glass materials. The onset of plasticity was seen as a sudden displacement discontinuity in the load-displacement curves in low-load indentation experiments in ceramic single crystals by Page et al. [15]. Mao et al. [16] indicated that the first “pop-in” indentation loads for the C plane of sapphire single crystal gradually decreases as loading rates increase. Dey et al. [17] observed “pop-in” phenomenon in load-displacement plots in soda-lime-silica glass, and related those displacement bursts with the formation of deformation bands inside the nanoindentation cavity.

During the indentation tests, the rate of loading is an important parameter especially for brittle materials as it strongly affects the

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Table 1

Compressive stress (CS) and depth of stress layer (DOL) results for aluminosilicate glasses with different ion-exchange time.

Ion-exchange time (h)	1	12	48
CS (MPa)	747 ± 20	710 ± 20	640 ± 20
DOL (μm)	15 ± 2	43 ± 2	77 ± 2

damage evolution [18]. Clear understanding of the effect of loading rate on the elastic-plastic deformation of the glass will provide valuable insight into the damage growth mechanisms especially during dynamic contact events of brittle solids [19,20]. Rate dependence in the response of the near-surface region probed by indentation on glasses and ceramics has been intensively studied [17,19,21–31] in different ranges of applied load with some interesting results being obtained. For nanoindentation, the nanohardness of glasses and ceramics is reported to remain unchanged [32], slightly reduced [33] or increased with loading rate [19,22]. Schuh et al. [34] indicated that the indentation load-displacement curve in their study on a Pd-40Ni-20P BMG is strongly dependent on the indentation strain rate. For the ion-exchanged glass, the surface compressive stress can inhibit crack propagation, and lead to increased strength [35,36] and hardness [18,37]. However, the influence of loading rate on the nanohardness of ion-exchanged glasses still remains unknown.

The main object of this study is to investigate the effect of the loading rate variation (100–20,000μN.s^{-1}) on the nanohardness of the raw and ion-exchanged glasses with different surface compressive stress. In this study, it is shown that the nanohardness of the raw aluminosilicate glass increases linearly with loading rate in double logarithmic axes. The effect of loading rate on the nanohardness of the ion-exchanged glass is similar to that on the raw glass. However, the nanohardness of the raw glass is more sensitive to the variation of the loading rate. These results are explained in terms of shear stress underneath the indenter and the flow line phenomena in the nanoindentations.

2. Experiments

The glass used was a float glass of thickness, 4 mm, with the same composition as in our previous published research [36–38]. Ion exchange involved suspending the aluminosilicate glasses in a molten potassium nitrate bath held at 420 °C for different times (1, 12 and 48 h). Then the glass samples were removed, cooled, and rinsed with deionized water after ion exchange.

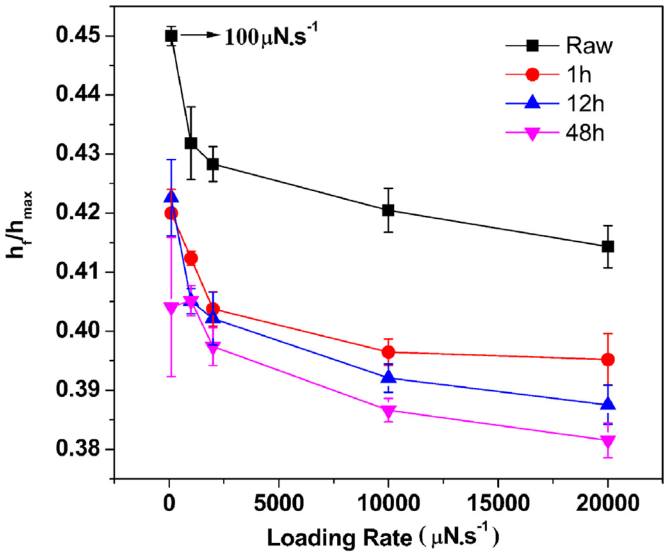

Fig. 2. Recovery ratio (h_f / h_{max}) of raw and ion-exchanged glass, with 1, 12 and 48 h immersion time, as a function of the loading rate.

The compressive Stress (CS) and depth of stress layer (DOL) of glass samples were measured by the surface stress meter (FSM-6000LE, ORIHARA, Japan) which is based on the theory of photoelasticity [39]. The glass sample was placed in the measuring area and care is taken to make sure the glass surface fits with the triple prism to ensure the birefringence fringes were detectable and clear. Parameters such as thickness, refractive index, photoelastic constant of the glass are input into the analysis [40]. Then, the CS and DOL values are obtained. The systematic error for the CS and DOL was ± 20 MPa and ± 2 μm, respectively. Each glass sample was measured at five random positions and the average CS and DOL values were obtained.

A nanoindenter (TI 950 Triboindenter, Hysitron) with a full-scale capacity of 10 mN was used for all indentation experiments. A Berkovich diamond indenter was used for measuring the nanohardness and Young's modulus. Calibration was performed using a standard fused silica sample prior to the nanoindentation of the glass samples. The method used in the experiments and calculations was based on ISO 14577 [41]. The nanohardness and Young's modulus data were calculated using the well-known method developed by Oliver and Pharr (referring to it as OP method) [42]. In the present experiments, the load was increased up to 9000 μN. This is with loading rate changed in the

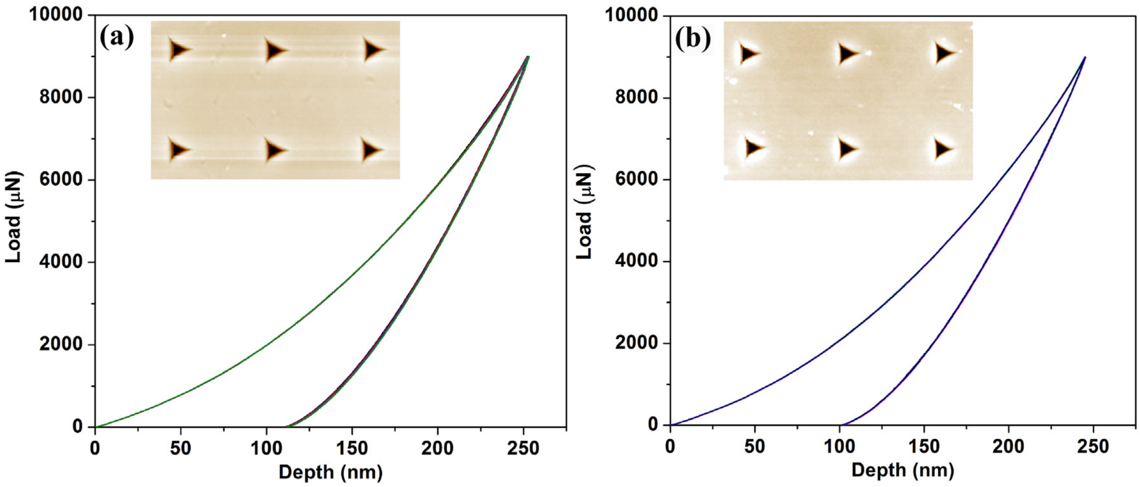

Fig. 1. Typical load-depth curves for: (a) loading and unloading of the raw glass;
(b) ion-exchanged specimen, with 1 h immersion time. For both cases, the top left inserted picture shows the corresponding SPM images of the six nanoindentations.