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Enhancement in nanohardness of soda–lime–silica glass

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ABSTRACT

For a thin commercial soda–lime–silica glass cover slip a significant increase in nanohardness (e.g. up to 74%) occurred with variations in the loading rate (10–20,000 $\mu N \cdot s^{-1}$) along with presence of serrations in load–depth plots and deformation band formation inside the nanoindentation cavity. These results are explained in terms of shear stress acting at positions of structural weakness close to the tip of the nanoindenter and the time scale of interaction between the nanoindenter and the local microstructure of the glass.

1. Introduction

Effect of loading rate on micro- and nanohardness of glass and ceramics is an area of very active current research due to its obvious technological importance [1–15]. However, there are many contradictory reports. For instance, the microhardness of glass has been reported to increase [1,2], decrease [3,4] or remain constant with variation in loading rate [5]. Nanohardness of soda–lime glass has been reported to slightly reduce [9], remain load independent [10,12,13], or exhibit indentation size effect [11,14] and that of soda–lime–silica glass to slightly increase [15] with variation in loading rate (1–1000 $mN.s^{-1}$).

In an attempt to resolve such controversies, the objective of the present study was to examine the effect of loading rate variation (10–20,000 $\mu N \cdot s^{-1}$) on the nanohardness of a thin (330 μm) commercial soda–lime–silica glass.

2. Materials and methods

The material used in the present study was a commercial soda–lime–silica (SLS) glass with the principal components of ~70 wt.% of SiO_2, 14 wt.% of Na_2O and 8 wt.% of CaO. It was in the form of a thin (~330 μm) microscopic cover slip polished to optical quality finish. The reason for choosing a very thin glass was to minimize the chance of interaction between extraneous bulk defects (which could be present more in a thicker rather than in a thinner glass) with the penetrating nanoindenter tip. The nanoindentation experiments were conducted with a TriboIndenter (Ubi 700, Hysitron Inc., Minneapolis, USA) having load and depth resolutions of 1 nN and 0.04 nm along z-axis, respectively while the thermal drift was $<0.05\ nm \cdot s^{-1}$. The machine provided a surface topography of constant contact force in scanning probe microscopy (SPM) mode and a load versus depth of penetration plot in nanoindentation mode using the same tetrahedral diamond Berkovich tip with a ~150 nm tip radius and a semi-apex angle of ~65.35°. The sample was mounted on a motorized table that allowed for a movement in the plane normal to the axial motion of the tip. The transducer that measured both load and depth consisted of a three-plate capacitor with the same Berkovich tip as mentioned above attached to the central plate. The instrument was calibrated by performing indents of increasing depth in a standard fused quartz sample that had a known nanohardness of 9.25 ± 0.93 GPa and indentation modulus of 69.6 ± 3.48 GPa. The diamond tip had a Poisson's ratio of 0.07 and Young's modulus of 1140 GPa [16]. The nanohardness and Young's modulus of the present soda–lime–silica glass was calculated using the method of Oliver and Pharr [17].

In the present experiments, the peak load was kept constant at 10,000 μN while both the loading and the unloading times were varied from 0.5 to 1000 s to change the loading rates in the range of 10 to 20,000 $\mu N \cdot s^{-1}$. At least five nanoindents were made at each of the five randomly chosen different locations of the sample at a given loading rate for a given peak load. Thus, each reported value of nanohardness was an average of at least 25 or more individual data points.

3. Results

The typical SPM based surface profile of the present glass sample prior to the nanoindentation experiment is shown in Fig. 1a. This data

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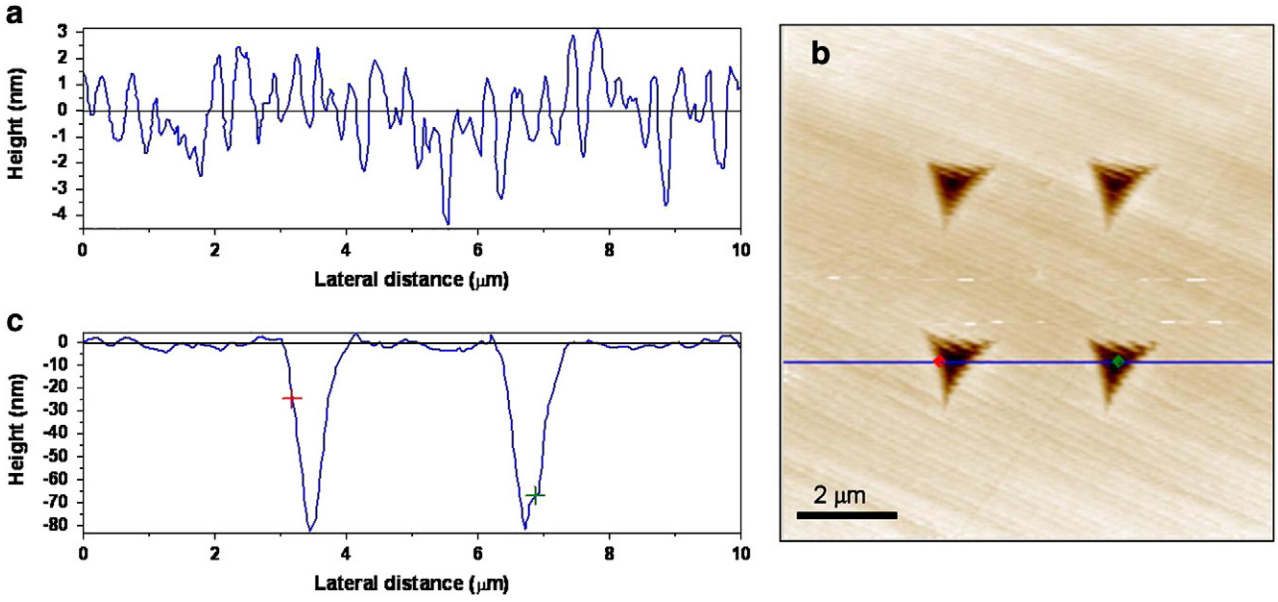

Fig. 1. (a) Surface profile prior to nanoindentation, (b) typical SPM image of nanoindentation array and (c) corresponding depth profile of nanoindentation for the soda–lime–silica glass.

confirmed the very low surface roughness of the soda lime silica glass sample used in the present work. It had a RMS roughness ~2.1 and a CLA of ~1.8 nm, typical mean asperity height was ~2.9 nm, with a maximum of ~9.3 and a minimum of ~−3.1 nm whereas the typical peak-to-valley distance of an asperity on the surface was ~12.3 nm. The SPM image of the nanoindents and corresponding depth profile of the marked indents are shown in Fig. 1b and c, respectively. Depth profile clearly showed evidence of the sink-in effect which is expected for brittle materials like glass and ceramics [17]. Further, the ratio between final depth of penetration and the maximum depth of penetration (h_f/h_{max}) was ~0.25 ± 0.08 which was the average value of all loading rates and well within the ratio of 0.70 suggested in [18] for the Oliver and Pharr [17] method to be applicable. The average h_f/h_{max} values obtained at the different corresponding loading rates are shown in Table 1. The data showed that the average h_f/h_{max} value decreased with increase in loading rates (Table 1). Moreover, the maximum and minimum h_f/h_{max} values were ~0.6 and ~0.12, respectively.

Fig. 2a–c showed the data on variation of nanohardness (H), projected area of contact (A_c) and Young's modulus (E) as a function of loading rate (LR); all plotted in a logarithmic scale. The fitted lines in Fig. 2a–c were obtained by the conventional linear least square fitting technique. The correlation coefficients (R) were e.g. 0.87, 0.88 and 0.82 respectively, for the data shown in Fig. 2a–c. For Fig. 2a, the fitted line represents the empirical equation $H = 0.76 + (0.06 \times LR)$. For Fig. 2b, the fitted line represents the empirical equation $A_c = 1.29 - (0.07 \times LR)$. For Fig. 2c, the fitted line represents the empirical equation $E = 1.85 + (0.01 \times LR)$. With increase in loading rate, nanohardness and Young's modulus of soda–lime–silica glass increased respectively by ~74% (Fig. 2a) and ~15% (Fig. 2c) while the average projected area of contact decreased in a corresponding manner (Fig. 2b). Averaged over all loading rates used in the present work, the Young's modulus value was ~76.76 ± 4.79 GPa. This data was comparable to the range e.g. 70 to 85 GPa of reported data for Young's modulus of soda–lime–silica glass measured by different techniques [12,17,20]. For instance, the nanoindentation technique gave Young's modulus values of 67–85.5 GPa [12], 67–79 GPa [19] and 70 GPa [17] for soda–lime–silica glass. On the other hand the biaxial flexure test gave a value of 72.4 GPa [20] while the ultrasonic pulse-echo method gave a value of 85 GPa for soda–lime–silica glass [20].

Typical load–depth (P–h) plots for nanoindentations on the soda–lime–silica glass sample at loading rates of 10, 100, 2000 and 20,000 $\mu N \cdot s^{-1}$ are shown in Fig. 3a–d. It is observed from the data presented in Fig. 3 that pronounced serrations appeared in the P–h plots more so often at lower loading rates e.g. 10 $\mu N \cdot s^{-1}$ (Fig. 3a) than at higher loading rates e.g. 20,000 $\mu N \cdot s^{-1}$ (Fig. 3d). Similarly, the corresponding SPM images, Fig. 4a–d, confirm that a large number of shear bands were present in the nanoindents more often in those made at lower loading rates e.g. 10 $\mu N \cdot s^{-1}$ (e.g. Fig. 4a) rather than that made at higher loading rates e.g. 20,000 $\mu N \cdot s^{-1}$ (e.g. Fig. 4d).

Table 1
Loading rate and the corresponding average h_f/h_{max} values.

Loading rate ($\mu N \cdot s^{-1}$)	Average h_f/h_{max}
10	0.34
20	0.39
100	0.23
1000	0.25
2000	0.21
10,000	0.17
20,000	0.16

4. Discussions

The major interesting results are that for the SLS glass used in the present work, (a) the nanohardness increased by 74% with loading rate, (b) the Young's modulus increased by about 15% and (c) comparatively more occurrence of pronounced serrations were seen in the P–h plots at lower loading rates e.g. 10 $\mu N \cdot s^{-1}$ (Fig. 3a) rather than at higher loading rates e.g. 20,000 $\mu N \cdot s^{-1}$ (Fig. 3d).

The dependence of hardness as a function of loading rate has been reported by a few researchers [1,2,15]. The works reported in [1,2] deal with Vicker's indentation. To the best of the author's knowledge; our effort reported for the first time [15] about the dependence of both hardness and the occurrence of serration in the load–depth plot on loading rate of soda–lime–silica glass measured by nanoindentation where the load chosen spanned in the range of 100 to 1000 mN. In the present work, however, a much lower load range e.g. 10,000 μN was chosen in conjunction with in situ scanning probe microscopy to gather deeper scientific understanding about the deformation behavior of a typical brittle amorphous solid like glass subjected to point contact force at a range of higher loading rates. Only for the sake