



Surface modification of ion-exchanged float aluminosilicate glass during deposition of amorphous alumina coatings by e-beam evaporation

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ABSTRACT

The main objective of this study was to investigate the physico-chemical and mechanical properties of ion-exchanged float aluminosilicate glasses deposited the Al_2O_3 films. The concentration gradients of potassium (K), sodium (Na), aluminum (Al) and oxygen (O) elements near Al_2O_3 film/ion-exchanged glass interface were analyzed by electron probe X-ray micro-analyser (EPMA). Compared with the ion-exchanged glasses, the peak of potassium concentration profile in the ion-exchanged glasses deposited Al_2O_3 films decreases and moves from glass surface to the inner part. Potassium ion diffusion is accompanied by aluminum (Al) and oxygen (O) diffusion from Al_2O_3 film into the ion-exchanged glass during deposition. In-depth analyses are performed using micro-Raman spectroscopy on sample cross sections in the aim to investigate the microstructure variation of the ion-exchanged glasses after deposited Al_2O_3 film on it. The results show that the change of chemical composition throughout ion-exchanged glass surface region can cause a gradual change in microstructure and physical properties.

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1. Introduction

Display devices have become a very important human-machine interface in information technology [1] and the display industry is second only to the semiconductor industry [2]. For a display device, a cover glass (protective glass) has been used in many cases in order to protect the display and to improve the appearance [3–8]. Ion-exchanged glasses pose a special challenge, since the integrated compressive stress in the surface region of the glass must be balanced by an equivalent magnitude of integrated tensile stress in the interior of the glass.

Ion exchange is a well-known technique to modify the mechanical and optical [7–12] properties of glasses at temperatures below the strain point of the glass. Typically, the exchange of small ions such as sodium in an alkali-containing glass, with larger ions such as potassium from a molten potassium nitrate bath below glass transition temperature, is responsible for the creation of compressive stress in the surface region of the material. Because glass articles usually break due to excessively applied tension acting on surface flaws, the introduction of a high surface compression into glass surface region strengthens the glass [4, 13]. This process of chemical strengthening or ion-exchange strengthening was originally described by Kistler [14] and Aclouque and Tochon [15] in 1962. Recently, demand for multifunction leads to most of the ion-exchanged glasses enduring subsequent treatment processes. Further, to a display device, surface treatment, for example, formation of

a film having a function such as antireflection, impact failure prevention, electromagnetic wave shielding, near infrared ray shielding or color tone correction may be applied in some cases. Some process might give rise to the structure modification and stress relaxation which will reduce the strength of the ion-exchanged glasses. The high temperature and particle impact process is an enormous challenge for the preservation of stress of the ion-exchanged glasses. Therefore, to investigate the surface structure modification of the ion-exchanged glasses during deposition of coatings is an important issue.

In the past, apparently little attention has been paid to the role of the physical vapor deposition treatment on the physical and mechanical properties of the ion-exchanged glasses. Some recent results [16–18] indicated the ion-exchanged glasses exhibited anomalous temperature dependence for stress relaxation process. With a liquid water or water vapor atmosphere, Tomozawa et al. [19] found glass surfaces exhibited faster relaxation than the bulk at a constant temperature. Nevertheless, there are no much studies on surface modification of the ion-exchanged glasses under coatings and particle impact, particularly below the glass transition temperature.

In this paper Al_2O_3 thin films deposited on the ion-exchanged glasses were prepared using e-beam evaporation assisted with ion source technique. The treated samples were characterized in terms of both concentration gradients and mechanical strength to define the influence of physical vapor deposition on the final performances. Moreover, Raman spectroscopy measurements were carried out from the glass slides beneath the surface in order to investigate the microstructure of the silica network of the different samples.

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2. Experimental

Float aluminosilicate glasses (glass code: EFL0.7TN, Asahi Glass Co., Ltd., Japan), from a single original commercial sheet 0.7 mm thick which was cut in 120×60 mm slides by straight cutter (CIE-7260, Chin I Machinery Co., Ltd. Taiwan), were used in this work. The composition of the glass (in wt.%) is: 71.2% SiO_2 , 13% Na_2O , 0.8% K_2O , 9% CaO , 4.5% MgO , 1% Al_2O_3 , and 0.5% trace elements. Ion exchange was performed by immersing the glass into molten pure KNO_3 (purity >99.9%, Changsha Xinben Chemical Co. Ltd. China) in a small automatic furnace at 450°C for 8 h. Then samples were washed with ethanol and acetone in an ultrasonic cleaning system (KDAB8000, Sanho-Potent (Hong Kong) Ltd.) respectively, and dried in the end.

The Al_2O_3 films were prepared on ion-exchanged glass substrates by electron beam evaporation in ZZSX-800F vacuum coating system, which equipped with an EH1000 ion source when the acceleration energy of Ar was 100 eV and the ion current density was over $120 \mu\text{A}/\text{cm}^2$, respectively. An Al_2O_3 target (99.99%, Beijing Laibaoli Coating Technology Co. Ltd. China) was used and Ar ion bombardment using ion beam assisted deposition system was carried out to deposit the Al_2O_3 thin films. The background pressure was about 3.0×10^{-3} Pa, whereas the working pressure was 1.0×10^{-2} Pa. The thickness of Al_2O_3 film was measured by a model NKD7000 spectrophotometer. Before deposition, the substrates were baked at 160°C for 30 min in ZZSX-800F. Following a 5 min pre-sputtering, the Al_2O_3 films were deposited on ion-exchanged glass substrates with a thickness of 350 nm.

Moreover, in order to discriminate the physical vapor deposition contribution to the ion-exchanged glass modification, electron probe X-ray micro-analyser (EPMA, JXA-8230) with tungsten filament was used to measure the elemental in depth profiles of chemical species near Al_2O_3 film/ion-exchanged glass interface. The accelerating voltage was 15 kV and the probe current was about 5 nA for back scattered electron imaging and secondary electron imaging, and about 10 nA for quantitative composition analysis. Concentration distributions were measured by performing elemental line scans over smooth fresh cross-section of glass plates in the $140 \mu\text{m}$ range normal to the plate surfaces with a $0.5 \mu\text{m}$ interval. Concentration distributions were measured in the Al_2O_3 -coated ion-exchanged glasses and in the ion-exchanged glasses.

Field emission scanning electron microscopy (FE-SEM) was performed by a JEOL JSM-7600F for the analyses of cross-section surface features and morphological investigations. The surface compression stress of the ion-exchanged glasses before and after deposition was measured by surface stress meter FSM-6000LE, which is a standard optical method based on the photoelastic properties of the glass. Studying specific molecular vibrations associated with the structural modifications in the superficial region was induced due to the diffusion during the deposition, micro-Raman spectra were taken at room temperature by using a Labram HR800 micro-Raman spectroscopy in the 450–1250 nm range with a step-size of 2 nm. For excitation, the 514.5 nm line from a mixed Ar-Kr ion gas laser was used. The laser beam was focused onto the cross section of a cut sample near the surface region and the scattered radiation from this region was recorded with a Peltier-cooled CCD camera. A $100\times$ microscope objective (N.A. = 0.9) was used to excite and collect the Raman spectra. The laser power on the sample cross section was kept below 5 mW in our measurements.

3. Results and discussions

In Fig. 1 the potassium (K), oxygen (O), sodium (Na) and aluminum (Al) concentrations versus depth are given. Quantitative line scans for the elements potassium (K), oxygen (O), sodium (Na) and aluminum (Al) across the structures of the ion exchanged glasses are presented. The potassium concentration in the ion-exchanged glasses decreases gradually with depth, which a peak exists on the surface of the ion-exchanged glasses. However, the oxygen, sodium

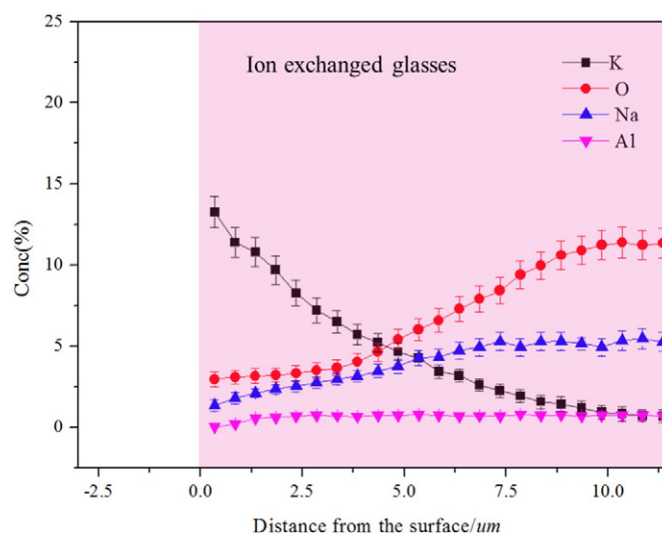


Fig. 1. Potassium, oxygen, sodium and aluminum concentration profiles of the ion exchanged glasses.

and aluminum concentrations in the ion-exchanged glasses increase firstly and then tend to be stable with depth.

The process deposited the Al_2O_3 films, which appears to be accompanied by potassium ion diffusion from the surface region of the ion-exchanged glasses, has a noticeable effect on the potassium concentration distribution in the ion-exchanged glasses. Baking heats the substrate and enhances the mobility of potassium ion on the ion-exchanged glass surface before deposition, resulting in an increase of the potassium ion diffusion on the glass surface. Also, the vapor depositing particles and argon (Ar) ions bombardment on the substrate is one of the causes to change the potassium concentration distribution near surface region of the ion-exchanged glasses.

Electron probe X-ray micro-analyser (EPMA) in-depth profiles of potassium (K), sodium (Na), aluminum (Al) and oxygen (O) elements near the Al_2O_3 film/glass interface are shown in Fig. 2. In the Al_2O_3 -coated ion exchanged glasses, measurements displayed remarkable gradients in aluminum, potassium, and oxygen concentrations in the absence of concentration gradients of sodium element. The concentration distribution of potassium element varies violently after the Al_2O_3 -coated treatment. Compared to the ion-exchanged glasses, the

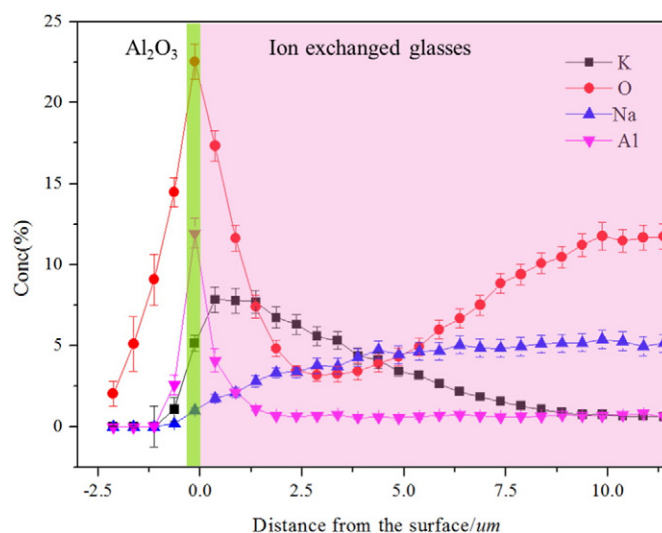


Fig. 2. Potassium, oxygen, sodium and aluminum concentration profiles of the ion exchanged glasses coated with Al_2O_3 .

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