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Influence of different process conditions on mechanical, optical and surface properties of silver ion exchanged soda-lime silicate glass

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ABSTRACT

Recently, surface modified functional glasses have been the focus of attention for researchers. Among the other surface modification methods, ion-exchange process has become prominent in terms of cost effectiveness of the process, ease of production, not causing damage to the glass matrix. The aim of this work is to investigate how certain ion exchange and further heat treatment processes affect the mechanical, optical and surface properties of soda-lime silicate glass. The glass samples have been subjected to ion exchange at 320 °C in a molten mixture of $AgNO_3$ and $NaNO_3$ with molar ratios of 2:98, 5:95, respectively and heat treatment under reducing atmosphere (Ar/H_2 with a 5% H_2 volume fraction mixture). The study is conducted in two parts. The first part involves a batch of characterization on the untreated and the treated glass samples. The characterizations include optical transmittance, absorbance and reflectance measurements by UV–vis-NIR spectrophotometer, FTIR analyses to examine surface properties. Scratch resistance with an increasing load and flexural strength measurements were conducted on the glass samples to determine the mechanical features of the glasses. The second part of the study includes the determination of possible corrosion mechanisms and optical properties changes of the glass surfaces after environmental test. Before and after the environmental test, optical transmission and colour analysis of the specimens were realized by UV–vis-NIR spectrophotometer, FTIR analyses were conducted on the surface of the silver ion-exchanged glass samples in order to observe corrosion reaction by using the reflection technique in the 400–4000 cm^{-1} wavenumber range. As a result of the study, glass samples with controllable optical, surface and mechanical properties by process parameters, containing silver nanoparticles and resistant to atmospheric conditions are obtained.

1. Introduction

In recent years, the use of surface modified glass by the ion exchange process has increased considerably. Ion exchange in glass has been used for many years to modifying the glass surface structure [1,2]. In 1913, Schulze [1,2] examined that the diffusion of silver in soda lime glass which is the first monovalent cation that was exchanged. It is known that ion exchange process at a desired temperature below the glass transition temperature followed by annealing in several temperature range and in various atmosphere is well established method for producing metal nanoclusters in alkali containing silicate glasses [3–7]. Correspondingly, silver ion is the most commonly used metal ion in optoelectronics, waveguides, photonics, biotechnology, solar technologies and glass colouring applications by the virtue of its high mobility, antibacterial efficacy and the optical absorption (surface plasmon resonance (SPR)) properties [3,8,9].

In the most literature studies, silver ion exchanged glasses were investigated according to the features such as diffusion kinetics, photoluminescence, refractive index and SPR absorption properties, colouring and antimicrobial effect [1,3,9,10]. But for the usage of the silver ion exchanged silicate glasses in many of these application areas, it is mandatory to understand the effect of applied processes on surface structures of glass and the behavior of these type of modified glasses against to atmospheric conditions and mechanical effects. Therefore in this study, it is aimed to investigate optical, mechanical and local glass network structural properties of glass samples subjected to different process parameters (silver concentration, ion exchange time and heat treatment). Furthermore, the effect of atmospheric conditions (such as

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humidity) on glass surface structure and some optical properties were evaluated. According to our literature survey, there is limited number of publications which approach to this topic in this respect, so it was thought that the results of this study will contribute to the silver ion exchange literature.

2. Experimental procedures

Commercial float soda-lime silicate glass with a composition specified as (wt%) 72.09 SiO_2, 13.46 Na_2O, 0.40 K_2O, 8.28 CaO, 4.24 MgO, 1.20 Al_2O_3, 0.035 TiO_2, 0.20 SO_3, 0.09 Fe_2O_3 was used in this study. The glass transition temperature of the soda-lime silicate glass was determined as 575 °C. The measurement was performed in Seiko Exstar 6300 model DTA/TG instrument by using the differential thermal analysis (DTA) method with a constant sample weight of 50 mg, in a static air atmosphere with Al_2O_3 pans. After the glass samples were ultrasonically cleaned and preheated, ion exchange was performed by immersing glass samples into molten salt bath of $AgNO_3$ and $NaNO_3$ with molar ratios of 2:98 and 5:95, respectively for 1 h. and 3 h. The ion exchanged samples were cleaned ultrasonically in distillated water after cooling to room temperature and subsequently heat treated under reducing atmosphere (Ar/H_2 with a 5% H_2 volume fraction mixture) at 250 °C for 5 mins. The prepared samples are listed in Table 1. According to our previously reported work [8], the minimum process temperatures, which silver nanoparticles formed in glass structure are 320 °C and 250 °C for ion exchange and heat treatment, respectively. Therefore, these temperatures have been chosen for this study by taking into consideration the duration and cost of the process. So, ion exchange processes and additional heat treatments were realized in temperatures as lowest as possible.

UV–vis-NIR spectroscopy measurements were realized to investigate the effect of silver-sodium ion exchange on the optical transmittance, absorbance and reflectance of the glasses between 250 and 2500 nm wavelengths (graph results were given in different ranges to provide better sight). Light transmittance, solar direct transmittance, total solar energy transmittance, ultraviolet transmittance and related glazing factors are the essential parameters for determining the light and energy performance of glazing in building. Several international standards have been published to evaluate the performance of windows and glasses such as thermal transmittance and solar parameters. In order to determine the optical properties of the glass samples, light transmittance, solar direct transmittance, infrared and ultraviolet transmittance and light and solar reflectance values were calculated based on the European Standard EN 410 after the spectral measurements [11–14]. The spectral measurements were carried out using Perkin Elmer, Lambda 950 UV–vis-NIR spectrophotometer, with a tungsten and deuterium lamp and at room temperature. The average radii of silver nanoclusters were calculated from the absorption peaks. Transmission electron microscopy observation of nanoparticles/nanoclusters was performed on JEOL-2100 HRTEM operating at 200 kV (LaB6 filament). Image was taken by Gatan Model 794 Slow Scan CCD Camera and also by Gatan Model 833 Orius SC200D CCD Camera.

Table 1
Samples prepared in this work.

Sample	% molar ratio of $AgNO_3$:$NaNO_3$	Ion exchange treatment T (°C), t		Heat treatment conditions T(°C), t	
a	Untreated soda lime silicate glass				
b	2:98	320	1 h.		
c	2:98	320	1 h.	250	5 min.
d	2:98	320	3 h.	250	5 min.
e	5:95	320	1 h.		
f	5:95	320	1 h.	250	5 min.
g	5:95	320	3 h.	250	5 min.

Infrared spectra of the samples were collected in reflection mode of the FTIR (Bruker Hyperion 3000) spectrometer, in the 400–4000 cm^{-1} wavenumber range.

Scratch tests have been performed by a Nanovea-M1 nanomechanical test instrument with diamond sphero-conical stylus (tip radius 5 μm), a rate of 312 mN/min progressive load between 0,1–200 mN, scratch length of 0,4 mm and 5 scratches per sample.

The flexural strength measurements of the ion exchanged and heat treated samples was carried out via four point bending test with a distance for loading pins and supporting pins of 10 and 30 mm, respectively. The measurements were examined according to ASTM Test Standard (C158-02-ASTM Standard Test Methods for Strength of Glass by Flexure (Determination of Modulus of Rupture)) by using an Instron 3366 Testing Instrument. Glass samples with 50 mm × 10 mm × 4 mm dimensions were subjected to the test by 0.5 mm/min displacement rate at room temperature.

Environmental testing was performed in a humidity test chamber (ESPEC-SH641) at 40 °C, under 95% relative humidity for 21 days to investigate of the optical and structural behavior of the glass surfaces against a certain humidity and temperature. The FTIR reflectance and optical transmission spectra were collected before and after environmental aging test. Colour analyses were detected using UV–vis-NIR spectrophotometer and parameters have been calculated in accordance with International Commission on Illumination (CIE) L*a*b* guidelines.

Errors were derived by using the well-known sample standard deviation formula:

$$S = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}} \quad (1)$$

where x is the measurement data, x̄ is the mean average (measurement 1, measurement 2, …) and n is the sample size (number of measurements).

3. Results

Prior to the ion-exchange process, the glass samples were colourless and had no measurable absorption in the visible region of the electromagnetic spectrum. Fig. 1 shows the UV–vis optical absorption spectra for the samples as-exchanged and heat treated at 250 °C for 5 min. under reducing atmosphere. After the ion exchange, the glass samples were slightly yellowed and no significant SPR of the silver nanoclusters was observed. The SPR absorption peaks have characteristic positions and shapes that depend on the optical constants, size, shape and volume fraction of the particles and on the optical constants of the dielectric matrix. The peak position is mainly related to the dielectric properties

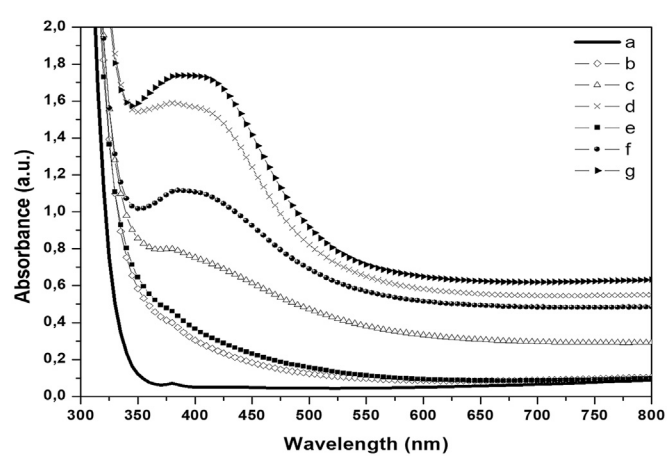

Fig. 1. Optical absorption spectra of the untreated and treated glass samples (a–g).

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