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Influence of chemical treatment on microstructure and mechanical properties of chemically-toughened glass by ion exchange process

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ABSTRACT

In this study, an attempt was made to investigate the influence of immersion temperature and time of ion-exchange on the microstructure and mechanical properties of chemically toughened soda-lime glass (CTG). Glass samples of 120 × 50 × 40 mm were immersed in potassium nitrate (KNO_3) salt placed inside a salt bath and subjected to heating at temperatures of 450 °C and 500 °C with the immersion time varied for 4 h and 5 h for each temperature inside a muffle furnace. Scanning Electron Microscopy (SEM) was used to evaluate the microstructure of the chemically toughened glasses while tests such as hardness, flexural strength and impact strength were used to assess the mechanical properties of the chemically toughened glasses. The results obtained show that hardness value decreased as temperature and time increases with maximum hardness of 26.35 BHN for CTG_1 (450 °C for 4 h). The impact strength increased as temperature and time increases with maximum impact strength of 36.72 J for CTG_4 (500°C for 5 h). However, the maximum flexural strength of 95.87 MPa was obtained for CTG_2 (450 °C for 5 h).

1. Introduction

Glass is a general term which is commonly used to refer to any solid which lacks long range structural order [1]. Theoretically, glass is a very strong material with estimated strength of approximately 7000 MPa but fails in service below its estimated theoretical strength value as a result of inherent surface flaws and defects present within the glass matrix [2,3]. Over the past few decades, glass has been used in a large proportion for many applications in automobiles, aircraft, structural challenging field etc. however, recent demands for glass to possess a more reliable structural behavior to fit in for many advanced applications has resulted to strengthening of this material either by toughening or laminating. Toughening is performed either thermally or chemically which provides the glass with a pre-stress (surface compression) which inhibits flaws from growing until the surface pre-compression is exceeded [4,5].

Ion exchange strengthening which is sometimes referred to as stuffing is a process in which ions of a larger size than that which already present in the glass matrix are stuffed into the matrix, resulting to stress on the surrounding atomic bonds [1]. The induced compressive stress on the surface is widely accepted as the mechanism by which the ion exchange process enhances the overall strength value of glass as the compressive stress inhibits crack propagation from the sharp points of the flaws through the bulk glass. Ion exchange is a two-way process in which at the interface between the glass and molten salt, the two types of alkali ions (Na^+ and K^+) exchange places with each other by liquid-surface adsorption approach [6] which is temperature and time dependent (diffusion-controlled process). Asides temperature and time, glass composition also plays a major role in the ion exchange process.

In the past few years, ion-exchange has gained great interest as strengthening method of glass due to its possibility to improve glass strength and to work effectively on articles of varying shapes and limited thickness, thus eliminating some of the problems associated with thermal toughening such as optical distortions of the surface and premature failure [7]. Many recent technologies and applications took the fundamental advantages from the ion-exchange applications in glass components in area such as transparent lightweight armors, automobiles, aircraft and photovoltaic modules among others [7–8].

Recently, several works have been carried out with the sole aim of improving the mechanical properties of glass through ion-exchange process or combining two methods of glass strengthening in other to develop a glass for better advanced applications.

Bartholomew and Garfinkel [9] considered two methods of enhancing the strength of glass by either producing devoid of any surface defects or rendering the surface flaws inactive. They however, employed fire polishing and etching applications but reported they only resulted in temporal enhancement of the glass strength. Bogart and Dilliard [10] also investigated the approach of combined strengthening

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method using ion exchange and fire polishing in improving the strength property of glass. It was reported that there was improvement in glass strength as the surface flaws were rendered dormant.

Akamatsu et al. [11] studied the new method to strengthen glass edges using CO_2 laser irradiation. It was reported that the flexural strength and the thermal fracture strength of glasses with CO_2 laser processed edges was found to be much higher compared to conventional glasses with dry or wet polished edges. Tyagi and Varshneya [12] investigated and reported that the introduction of any ion with size varying from the original ions in the glass matrix resulted in significant changes in the glass matrix structure giving rise to increment in glass strength, thermal shock resistance and closure of surface flaws and micro-cracks.

However, since several works investigated and reported on glass strengthening by ion exchange have been beneficial, there is therefore need to determine at what immersion temperature and time better mechanical properties are going to be attained. Therefore, this present work aims at investigating the influence of ion exchange with respect to immersion temperature and time on the microstructure and mechanical properties of chemically toughened soda-lime glass.

2. Materials and method

2.1. Sample preparation

The material utilized for this present work is a sheet soda-lime silicate glass with nominal thickness of 4 mm which was commercially obtained in its as-received state. The mean composition of the soda-lime silicate glass taken into accordance with [13] is SiO_2–72.5%, Na_2O–15%, Al_2O_3–1%, CaO–9% and MgO–2.5%. The as-received commercial soda-lime glass was initially rinsed with distilled water in order to remove any surface dirt and later dried. A total number of 26 sample glasses of 120 × 50 × 4 mm were cut from the sheet glass and were prepared for the ion-exchange process at 450 °C and 500 °C for immersion period of 4 h and 5 h respectively for both temperatures. The sample designations are shown in Table 1.

2.2. Ion-exchange process

The prepared sample glasses having a measured glass transition temperature (T_g) of about 530 °C were fully immersed inside a salt bath containing potassium nitrate salt (KNO_3). The salt bath was then placed inside an electric power muffle furnace equipped with an accurate temperature monitoring and regulatory device. The furnace was then powered on for heating schedule at different immersion temperatures and soaking times under a static condition as shown in Table 1. After completion, the chemically toughened glasses were removed from the molten salt bath using a pair of tong.

2.3. Characterization

The microstructure evaluation was carried out using scanning electron microscope (SEM, ASPEX 3020) with generator voltage set at 16.0 kv. The glass samples to be viewed were initially sputtered with gold of about 5 nm size and then clamped on the sample holder attached to the machine and the surface to be viewed was then exposed at varying display magnification.

The mechanical behavior such as hardness, flexural strength and impact strength was examined by Brinell hardness indenter (BHN), Universal testing machine and Izod impact tester respectively. The sample preparation and the test procedure for the hardness evaluation Brinell performed by micro indentation was carried out in accordance with ASTM E-92 standard in which the samples were exposed to a direct load of 250 kg for about 15 s after which the indented diameter was measured by a special low-powered microscope utilizing a scale. The hardness values were then obtained using a conversion table according to standard. Flexural strength test was performed and evaluated in accordance with ASTM 8 M – 91 standard at 25 °C (room temperature) by a 3-point bending strength test with the distance set according to standard as stated by ASTM 8 M – 91 standard using Instron universal testing machine (Instron 3369, 50 kN load capacity) operated at a strain/load rate of 5 mm/min. The sample glasses were placed respectively on the two supporting rods of the machine, in a way that the two projecting ribs of the samples had equal distance from the two supporting rods. Load was then applied on the sample to obtain a rate of stress increase of 0.2 N/mm per sec. The load was applied until the sample fracture and force at the point of fracture noted and the flexural strength automatically obtained from the machine. Impact strength test was conducted and evaluated using Izod impact tester at room temperature under a standard specified condition of strain rate and applied load. The samples were fixed respectively and an arm held at a specified height was released, this arm hits the sample and breaks it. The impact strength was then determined from the energy absorbed by the sample using the difference from the initial and final height of the hammer.

Table 1
Sample designation for ion-exchange process at different temperature and time.

Sample designation	Temperature (°C)	Time (hours)
Control	–	–
CTG_1	450	4
CTG_2	450	5
CTG_3	500	4
CTG_4	500	5

3. Results and discussion

3.1. Microstructure analysis

The result of the representative microstructure obtained is shown in Figs. 1 and 2 respectively. Fig. 1 indicates the microstructure of the non-toughened glass sample (control) while Fig. 2 represents chemically toughened glass (CTG). From Fig. 1, it can be observed that the non-chemically toughened glass displayed a good uniform morphology with spatial distribution of seed-like grain indicated by somewhat white pattern and few defects portion indicated by a dark-like bubble; which might however affect its mechanical behavior. From Fig. 2 which represents the chemically toughened glass sample (CTG_2) having features generally common to the remaining chemically toughened samples, it can be observed that the stuffing of potassium ions on the glass resulted into surface compressive stresses which are uniformly distributed throughout the glass matrix indicated by several grain boundaries formation. However, a lateral crack was observed at the upper portion of

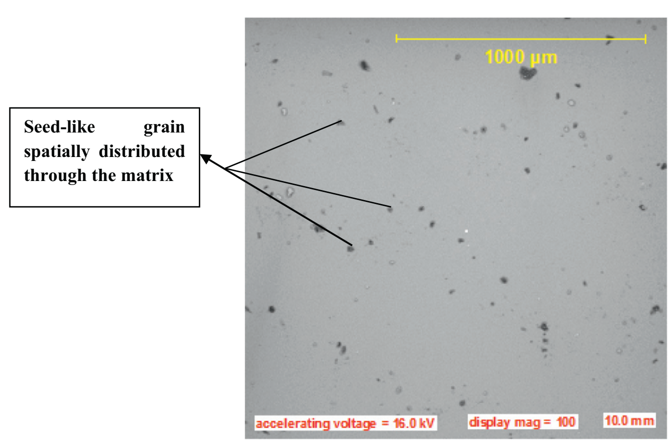

Fig. 1. SEM image of the non-chemically toughened glass sample (control).