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Mechanical Properties of Annealed Soda Lime Silica Glass with Various Potassium Salt by Ion Exchange

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ABSTRACT

This work studied the effect of ion exchange parameters on the glass properties. The regular soda lime silica glass was annealed at 600 ºC for 16 hours, followed by the chemical treatment process. The chemical treatment was carried out using different types of potassium salts at two different temperatures. Different ion exchange media in salt paste were prepared using KNO_3, KCl, and K_2SO_4 and heat treated at 425 ºC and 465 ºC for 4 hours. The mechanical properties of the glass before and after ion exchange treatment were analyzed using the 3-point bending and Vickers hardness method. The results show the highest flexural strength was obtained after the annealed glass was treated with KCl for 4 hours at 425 ºC. Meanwhile, treatment with KNO_3 at 425 ºC resulted in the highest Vickers hardness strength and glass fracture toughness compared to samples treated with other potassium salts. From both characterization techniques, the K_2SO_4 salt medium showed the lowest flexural strength, hardness, and fracture toughness values. Additionally, ion exchange treatment at 465 ºC reduced the glass's mechanical properties as higher treatment temperatures lead to surface structure stress relaxation.

Keywords: Ion exchange; soda lime silica; potassium salt; mechanical properties; compressive stress

1. Introduction

Glass is the most useful ancient material found everywhere in our daily lives. Glass's aesthetic appearance, rigid structure, and chemical inertness act as good surface protection, which makes it suitable as a device screen material [1]. However, the glass's amorphous structure and highly brittle properties caused it to be managed gently. As such, it can easily break with the presence of small voids on its surface [2]. Some treatment can be done to improve the glass surface structure for it to withstand any external force or internal voids.

This study focused on chemical strengthening involving the ion exchange method. Chemical strengthening is preferable to thermal strengthening due to the higher mechanical strength result, and it is suitable for applications involving thin and irregular glass structures [1]. Chemical strengthening also reduces the glass's tendency to optical distortion and structure deformation. The ion exchange process involves replacing the smaller alkali ions within the glass structure with larger alkali ions from the molten salt bath [3]. The procedure is usually performed at 50 to 200 ºC below the glass transition temperature (T_g). Generally, soda lime silica glass has T_g around 550 ºC, and the ion exchange procedure usually was done below 500 ºC. Potassium nitrate (KNO_3) is preferable as a medium for salt baths for ion exchange since KNO_3 is highly soluble in water and has a low melting temperature. Potassium chloride (KCl) and potassium sulfate (K_2SO_4) are less soluble in water; thus, the treatment temperature needs to be maintained high for a successful diffusion process [4]. Besides chemical treatment, the annealing process can help in relaxing the residual stress inside the glass structure. By heating the glass structure nearly to its glass T_g and slowly cooling it to a controlled temperature, the trapped locked-in residual stresses can be released [5]. The annealing treatment before chemical strengthening can help in increasing the rate of ion diffusion during the ion exchange process [6].

Usually, the ion exchange treatment is done in a molten salt bath. However, salt bath preparation requires advanced appliances and is also time-consuming when dissolving the salt [7]. Moreover, the vapor from the salt bath is said to be corrosive and requires a highly practical washing-off procedure to clean up the salt residue [4]. In the past, some studies have already tried using the paste method during the ion exchange process to solve these issues [8]. Even though the solid phase diffusion is slower compared to the liquid phase, the procedure still improves the glass properties. Furthermore, the preparation of salt paste requires a simple procedure and only a small amount of salt will be used. Thus, it is more cost-effective compared to the salt bath procedure [9]. The objective of this study was to evaluate the effectiveness of using different types of potassium salt using the salt paste technique to improve the mechanical properties of soda lime silica. The ion exchange process took place in two different treatment temperatures, 425 ºC and 465 ºC, which is under the soda lime silica glass transition temperature. Finally, the glass's mechanical properties were studied by using a 3-point bending and hardness test.

2. Materials and Methods

2.1 Sample Preparation

The glass used was flat soda lime silica that was manually cut with a dimension of 75 mm × 25 mm and a thickness of 1.8 mm. The glass samples were cleaned by using distilled water and left to dry at room temperature. The glass samples were annealed at 600 ºC for 16 hours. After total cooling, the annealed glass samples were chemically treated.

Table 1. Samples codes of ion-exchanged glass samples.

Sample Code	Ion Exchange Temperature (ºC)	Potassium salt
AN425		KNO_3
AC425	425	KCl
AS425		K_2SO_4
AN465		KNO_3
AC465	465	KCl
AS465		K_2SO_4

Based on Table 1, the samples were chemically treated with three different types of potassium salt at two different temperatures. The ion exchange treatment was carried out in a semi-automatic furnace at 425 ºC and 465 ºC for 4 hours with a heating and cooling rate of 5 ºC/min. The salt paste was prepared by mixing 6 g of potassium salt with 2 ml of distilled water at room temperature. The salt paste was applied evenly on one side of the glass samples. The samples were then kept preheating and post-cooling over the salt paste for 20 minutes before and after heating. After total cooling achieved the room temperature, all samples were rinsed carefully by using distilled water. Before undergoing sample characterization, all the samples were reannealed at 550 ºC for one hour.

2.2 Sample Preparation

2.2.1 3-Point Bending Test

The mechanical strength of the glass samples was determined by using a 3-point bending test. The test was measured by using a Universal Testing Machine (UTM). The sample flexural strength was determined by applying a constant load of 1 N with a test speed of 2 mm/min at room temperature. The glass's flexural strength can be calculated by using the standard formula Equation (1).

$$\sigma = \frac{3FL}{2bd^2} \tag{1}$$

Where;

F= the load at a given point on the load-deflection curve (N)
L= length of support span (mm)
b= width of the test beam (mm)
d= thickness of the test beam (mm)

2.2.2 Vickers Hardness Test

The hardness properties of glass samples were identified by using Vickers hardness indentation at Pusat Penyelidikan Mineral, Jabatan Mineral & Geosains, Ipoh, Perak. The clear glass surfaces were indented with a load of 1 kgf for 10 seconds of dwell time. The instrument used was the Hardness Tester model MMT-3. Five indentations were made on each glass sample at room temperature. Crack formation on the glass surface was measured and recorded using an optical microscope. The hardness values were calculated by using Equation (2).

$$H_v = 1.8544\frac{p}{d^2} \tag{2}$$

Where;

P= load (kg)

d= average of diagonal length (mm)

2.2.3 Fracture Toughness

The fracture toughness (K_{IC}) was measured directly from Vickers indentations cracks made with 5 different indentations. The length of the median cracks was measured to determine the glass fracture toughness. The glass sample fracture toughness was calculated using Equation (3) according to the median crack model [10]. The constant 0.016 was proposed by Anstis et al. [11] and supported by Žmak et al. [12].

$$K_{IC} = 0.016\left(\frac{E}{H_v}\right)^{1/2}\left(\frac{P}{C^{3/2}}\right) \tag{3}$$

Where;

P= indentation load (N)
C= half crack length (m)
E= Young's modulus
H_v= Vickers hardness

3. Results and Discussions

3.1 Flexural Strength

Fig. 1 shows the flexural strength of pure glass sample and annealed glass samples after chemically treated at 425 ºC and 465 ºC. The flexural strength of the pure glass sample is 68.40 MPa. After ion exchange treatment at 425 ºC, the glass flexural strength increases to the highest value for sample AC425 (111.80 MPa), followed by AN425 (108.07 MPa) and AS425 (99.53 MPa). The increase in flexural strength was due to potassium ions diffusion into the glass structure, which led to compressive strength [13]. Meanwhile, samples treated at 465 ºC also show an increase in flexural strength for samples AC465 (90.17 MPa), AN465 (80.93 MPa) and AS465 (71.00 MPa). However, the strength value is slightly lower compared to the sample treated at 425 ºC. The lower flexural value at high treatment temperature was due to stress relaxation that occurred near the glass T_g [14]. Stress relaxation reduces the stress of the sample at the constant strain. Fig. 1 shows that treatment using KCl produced the highest strength as potassium from KCl salt sufficiently diffused into the glass structure and improved the glass's flexural properties. Meanwhile, K_2SO_4 salt had the lowest strength recorded. The lowest strength obtained by samples AS425 and AS465 was due to the high thermal stability and lower water solubility of K_2SO_4 [4]. A previous study by Beall et al. [15] also achieved an increase in mechanical strength after conducting an ion exchange treatment using salt mixtures of KCl and K_2SO_4. This proved that KCl and K_2SO_4 still can be used as ion exchange agents besides the common KNO_3 salt.

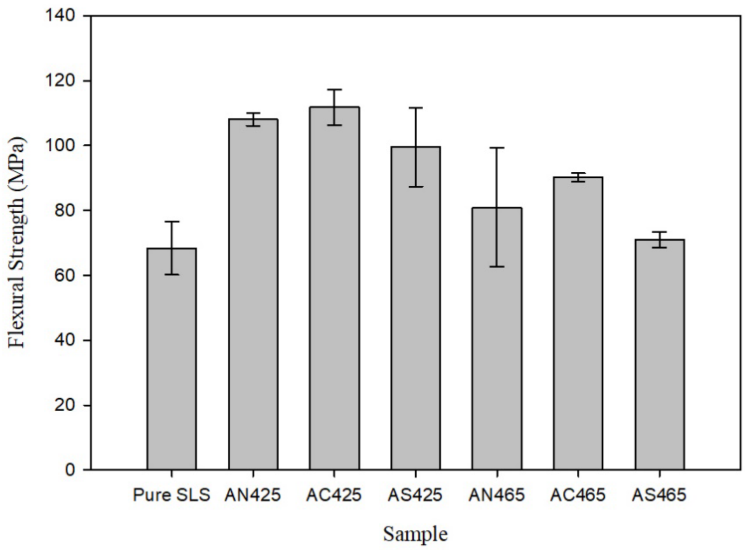

Fig. 1. Flexural strength of pure glass sample and ion-exchanged glass samples treated at 425 ºC and 465 ºC by using different types of potassium salt

3.2 Hardness

The hardness properties of pure glass samples and chemically treated samples at temperatures 425 ºC and 465 ºC are presented in Fig. 2**.** The hardness value of pure glass samples is 5.11 GPa. The glass hardness increases after being treated with ion exchange for samples AN425, AC425, and AS425 with values of 6.3, 6.2 and 6.15 GPa, respectively, with treatment at 425 ºC. The significant increase in hardness properties was caused by the formation of surface structure compression due to the stuffing effect of large potassium ions into the smaller site of sodium ions [16]. This compression increases the sample resistance from indentation at a load of 1 Kgf. Meanwhile, samples treated at 465 ºC, shows a slightly lower hardness value compared to samples treated at 425 ºC. Sample AN465 has a hardness value of 6.13 GPa, while samples AC465 and AS465 have 5.95 GPa and 5.87 GPa, respectively. The decrease in hardness value when treated at 465 ºC was due to the stress relaxation that occurs at high temperatures or usually near the glass transition temperature [17]. However, the glass's hardness strength treated at 465 ºC still improved compared with the pure glass sample, which means that the exchanging of ions still happened. Moreover, the result shows the highest value when treated with KNO_3 salt. KNO_3 has the lowest melting point and also higher water solubility, which makes it a preferable and popular salt for ion exchange agents [18]. The diffusion of potassium from KNO_3 salt into the glass structure produces a higher surface hardness on the glass surface to prevent further damage.

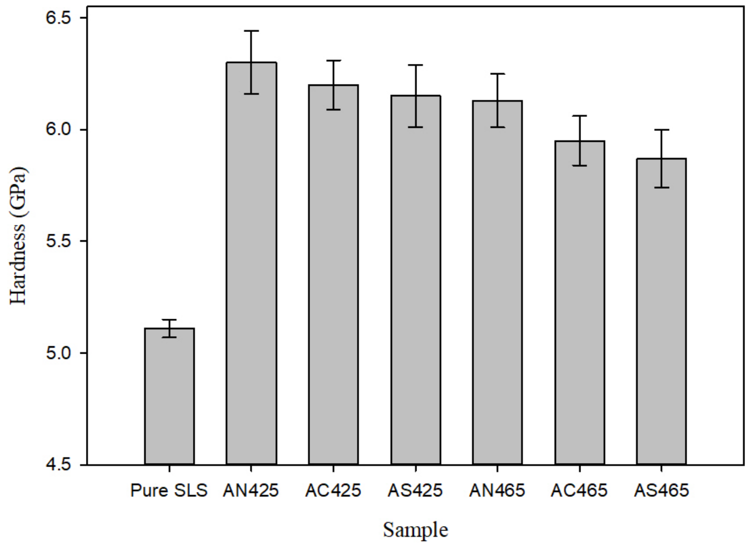

Fig. 2. Hardness of pure glass sample and ion-exchanged glass samples treated at 425 ºC and 465 ºC by using different types of potassium salt

3.1 Fracture Toughness

Fig. 3 shows the fracture toughness of pure glass and chemically treated glass samples at 425 ºC and 465 ºC using different types of potassium salt. The fracture toughness of the pure glass sample is 0.89 MPa $m^{1/2}$. After glass samples are treated with ion exchange at 425 ºC, the sample's toughness increases to 0.95 MPa $m^{1/2}$ for sample AN425, 0.94 MPa $m^{1/2}$ for sample AC425, and 0.93 MPa $m^{1/2}$ for sample AS425. For samples treated at 465 ºC, sample AS465 has the lowest value of toughness, which is about 0.91 MPa $m^{1/2}$. Meanwhile, samples AN465 and AC465 have a similar value to the sample treated at 425 ºC, which is 0.95 MPa $m^{1/2}$ and 0.94 MPa $m^{1/2}$, respectively. The improvement of glass toughness after ion exchange treatment is influenced by the stuffing effect of the large K^+ ions into the Na^+ ions sites [16]. The significant increase in sample fracture toughness proved that compressive stresses are introduced into the glass surface. The increase in fracture toughness was also said to depend on the more connected and compact glass structure [19]. The lower hardness value of the sample treated with K_2SO_4 was supposed to be due to the thermal stability of salt and surface stress relaxation that occurs at high temperatures [20].

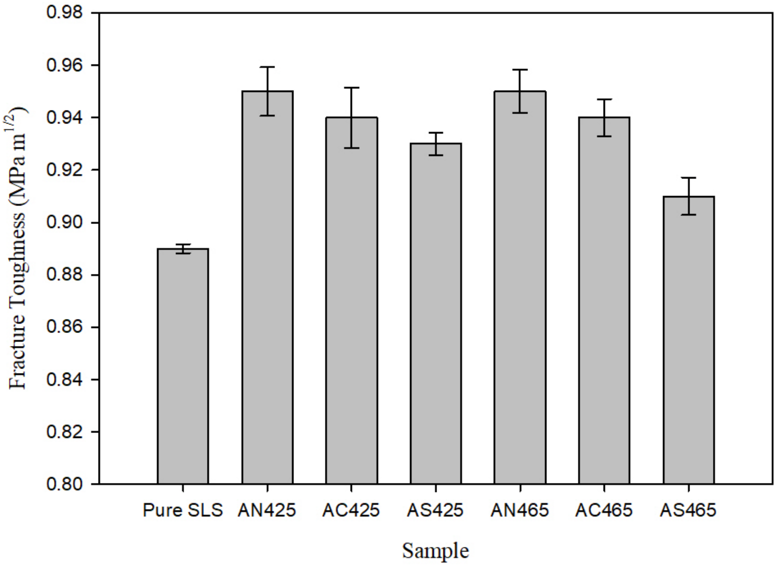

Fig. 3. Fracture toughness of pure glass sample and ion exchanged glass samples treated at 425 ºC and 465 ºC by using different types of potassium salt

4. Conclusion

The mechanical properties of conventional soda lime silica glass can be improved by using ion exchange treatment. In this study, the use of different types of potassium salts was proved to increase the mechanical properties of the glass samples. The diffusion of large potassium ions from the salt paste can lead to the introduction of compressive stress on the glass surface. The presence of compressive stress increases the glass's mechanical properties. The highest flexural strength was obtained by the sample treated with KCl, while the highest hardness strength and glass toughness were obtained by the sample treated with KNO_3. For both mechanical properties, the sample treated with K_2SO_4 resulted in the lowest mechanical value. Moreover, in this study, the optimum temperature for ion exchange was 425 ºC. The lower treatment temperature can reduce the tendency for stress relaxation to happen. This is because the higher treatment temperature can cause stress relaxation to occur in the glass structure, thus reducing the sample's mechanical strength.

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