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Thermal and mechanical characterization of borosilicate glass

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Abstract

The aim of this work is to characterize thermally (dilatometric analysis) and mechanically a Pyrex type borosilicate glass. The mechanical tests (Vickers indentations, mechanical strength and fracture toughness) were made on the glass in an annealed state and after a chemical strengthening treatment by ionic exchange. The indentations imprints morphologies and details were observed by optical and scanning electron microscopy.

The dilatometric analysis shows that the thermal expansion variation with temperature is essentially non linear, increasing rapidly up to 200°C and slowing down beyond. The optimal glass chemical strengthening was obtained for a bath duration of 15 hours. This corresponds to a relatively moderate increase of the mechanical strength (~70%). The fracture toughness measured by indentation was appreciably improved by the chemical treatment. It seems also to increase with the applied indentation load.

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Key words : Borosilicate glass, Structure, Indentation Vickers, Thermal expansion, Strengthening

1. Introduction

Borosilicate glasses are well known for their excellent chemical durability and for their resistance to heat. The Pyrex glass brand with a weak linear expansion coefficient of about $3.3 \times 10^{-6} \text{ }^\circ\text{K}^{-1}$ is very resistant to thermal shock besides its chemical durability [1, 2, 3, 4]. It is designated by the abbreviation (BS 3.3) according to the international standardization organization (ISO). It is commonly used for ovenware and laboratory glassware. Other borosilicate glass types, with a linear expansion coefficient that varies between $3 \times 10^{-6} \text{ }^\circ\text{K}^{-1}$ and $6 \times 10^{-6} \text{ }^\circ\text{K}^{-1}$ and with different compositions are also used for glass-to-metal sealing applications and for specific electric, electronic and optical purposes.

The objective of this work is to characterize thermally and mechanically the behavior of a Pyrex glass.

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2. Experimental procedure

2.1. Material Characteristics

The glass used is a (BS 3.3) Pyrex glass which was polished by fire and has a thickness of 5mm. Its chemical composition and its principal physical properties are respectively given in table 1 and 2. The material was submitted to an annealing treatment at 565 °C during 30 minutes in order to eliminate any residual stresses.

Table 1: Material chemical composition (mass %) [5].

oxydes	SiO ₂	CaO	Na ₂ O	MgO	Al ₂ O ₃	B ₂ O ₃	Fe ₂ O ₃	autres
BS 3.3	80.6	0.10	4.2	0.05	2.25	12.6	0.04	//////

Table 2 : Material principal properties [5].

Physical properties	Units	
Poisson Coefficient	//	0.22
Density	ρ [g/cm ³]	2.23
Linear expansion coefficient	[°K ⁻¹]	3.3 x 10 ⁻⁶
Elastic modulus	[GPa]	64
Transition temperature	[°C]	565

2.2. Equipments Used and Tests Operations

For the dilatometric analysis, we used a NETZSCHDILL 402 C type apparatus. Equipped with a computer program that restitutes the dilatometric curve, it enables to deduce the linear expansion coefficient variation with temperature. Samples of dimensions (50 mm × 7 mm × 5 mm) were used for that purpose.

Chemical strengthening treatments and annealing operations were undertaken in a FILLMANFREDI electric furnace that can reach 1100°C. The ion exchange treatments were made using KNO₃ salt baths and different durations. The ionic exchange by diffusion of the smaller size ions (Na⁺) in the glass by the larger size ions (K⁺) from the bath lead to compressive stresses in the glass superficial modified layer. Consequently, it increases the glass mechanical strength.

Indentation tests were made on glass samples of dimensions (50mm × 10mm × 5 mm) using a Vickers hardness equipment with loads varying between 5 and 50 N and a dwelling time of 15 s. The obtained imprints with their crack systems were observed on a metallographic microscope (Neophot) and by scanning electron microscopy (SEM). The typical radial cracks obtained by Vickers indentation were also measured for determining the fracture toughness K_{IC} according the following relation [6]:

$$K_{IC} = 0.016 \left(\frac{E}{H} \right)^{0.5} \cdot \frac{P}{C^{3/2}} \quad (1)$$

C : half crack length.

E : Elastic modulus.

H : Vickers hardness.

P : Applied indentation load.

The glass mechanical strength σ_C was evaluated using a 4-points bending apparatus on a universal traction machine of the type Heckert Fu 1000e type, according to the following relation:

$$\sigma_c = \frac{3P_c \cdot (L - l')}{2 \cdot b \cdot w^2} \quad (2)$$

P_c : critical load at fracture.

L, l' : distances between respectively the outer and inner supports.

b : sample width.

w : sample thickness.

Prismatic samples of dimensions (50 mm x 12.5 mm x 5 mm) were previously chamfered on their longitudinal sides in order to reduce the strength values dispersion.

3. Results and discussion

3.1. Dilatometric Analysis

The glass thermal dilatometric curve obtained (figure 1) is characterized by a sharp increase up to a temperature of 200°C followed by a much more slower increase rate until 450°C. At this level a new rapid increase is observed up to nearly the transition temperature. In comparison with the linear thermal dilatometric curves usually obtained on soda lime glasses, the variation on the tested borosilicate glass is essentially non linear on the same working temperature range [7].

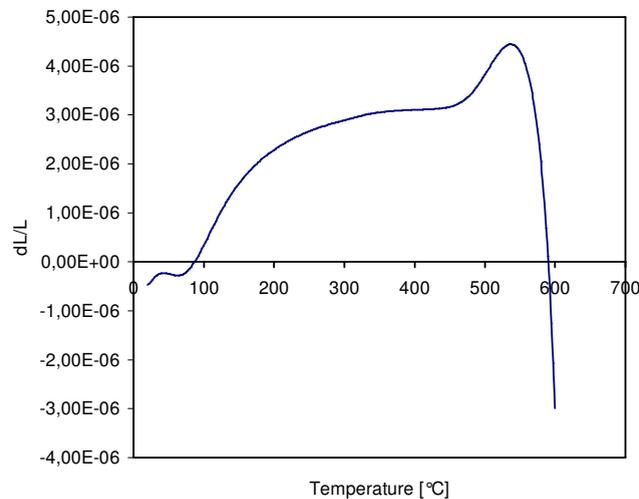


Fig. 1. Dilatometric variation curve of the borosilicate glass.

3.2. Indentation Imprints Observation

The induced Vickers indentation imprints morphology obtained on annealed glass were observed by optical (figure 2a) and by scanning electron microscopy (figure 2b). Besides the presence of the radial cracks system with some secondary cracks, we can also notice on these micrographs regular fault lines along the imprint sides. These fault lines become more separated with depth (figure 2b). This is typical of the anomalous behavior of the borosilicate glass whose plastic deformation at the imprints is characterized by material densification [8,9]. It is related to its particular structure. With a small amount of Na_2O modifier relatively to soda lime glass, it has

more empty space that promotes densification. It is also caused by the O-Si-O and Si-O-Si changing bonding angles [10].

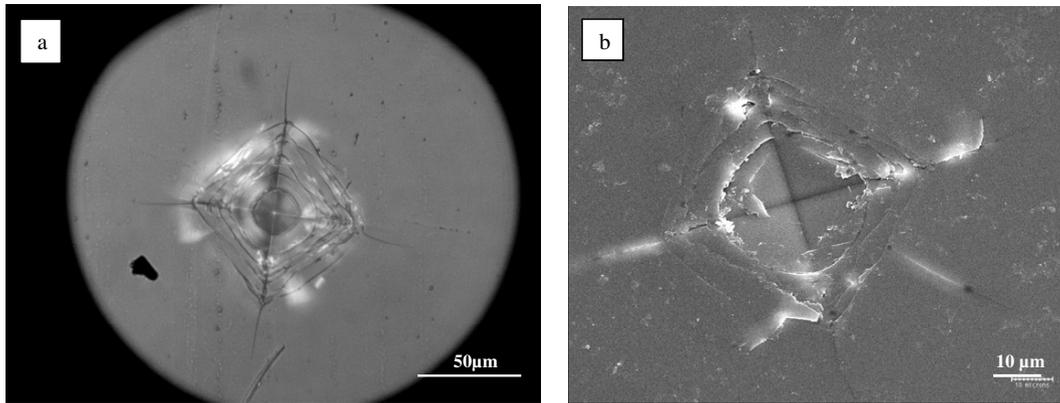


Fig. 2. (a) Vickers imprint obtained on borosilicate glass with a 10 N load. (x 200). (b) SEM micrograph of a similar Vickers imprint obtained on borosilicate glass with a 10 N.

3.3. Chemical Treatment Duration Effect On Mechanical Strength

As we can observe on figure 3, the strengthening duration in the chemical KNO_3 bath has an influence on the obtained mechanical strength. The ion exchanges for different durations (5h, 8h, 15h and 24h) at 480°C show that the maximum strength is reached for treatment duration of 15 hours. With a lesser time, the ion exchange process is incomplete. With a greater duration, the strength is reduced instead after a saturation of the ion exchange process. This is probably due to the fact that the maintained heat treatment after saturation leads to a relaxation of compressive residual stresses [11]. Besides, we also noticed that the maximal strength reached remains relatively low in comparison with soda lime glass treated in the same conditions (75 MPa against 350MPa). This can be explained by the small amount of Na_2O modifier present in the borosilicate chemical composition.

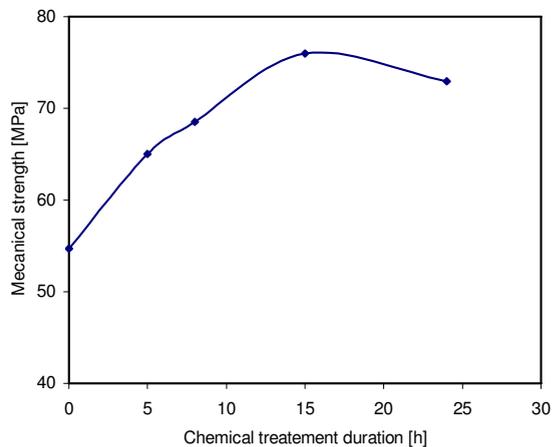


Fig. 3. Effect of ionic exchange treatment duration at 480°C on glass mechanical strength.

3.4. Effect of Chemical Treatment on Fracture Toughness

Figure 4 shows that the chemical treatment has a beneficial effect on the material fracture toughness. It increases by more than 50%.

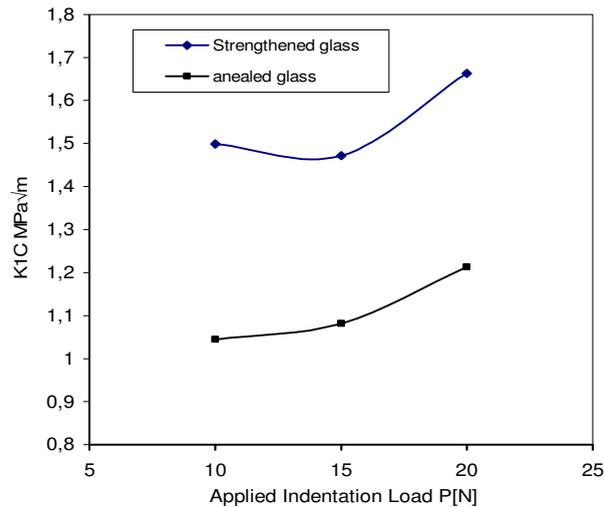
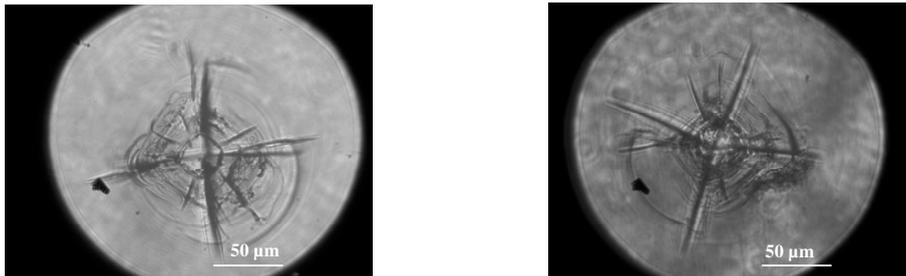


Fig. 4. Effect of ionic exchange treatment on glass fracture toughness.

We also notice that the fracture toughness increases with the indentation load. This apparent increase of the fracture toughness obtained by Vickers indentation technique is related to the Borosilicate glass behavior under sharp indentation. It is generally characterized by the presence of short radial and predominant conical cracks [8]. These later cracks are not visible on surface.

Because of this particular behavior, the indentation technique doesn't give reliable values on intrinsic fracture toughness [10]. However, for comparison between the annealed and the strengthened state glasses, Vickers indentation is a simple and accessible method.

The borosilicate glass indentation imprints present also many secondary cracks besides the typical radial cracks as we can notice on figure 5 for both annealed and treated cases. These reduce further the extension of the radial crack lengths needed for the fracture toughness by energy dissipation.



a) Treated glass

(b) Annealed glass

Fig. 5. Indentation imprints obtained with a 10N load showing secondary cracks.

4. Conclusion

The thermal and mechanical behavior of the borosilicate glass (BS 3.3) studied is quite different from the usually observed behavior of normal silicate glasses (Soda lime glass).

The dilatometric curve shows a non linear variation with temperature with a rapid changing rate up to 200°C.

An annealing treatment of the borosilicate glass samples was made prior to indentation in order to eliminate any residual stresses. The Indentation imprints reveal the presence of secondary cracks and faults lines along the sides. These are typical of the anomalous behavior (densification effect) of the borosilicate glass due to its particular structure.

In comparison to what is usually observed on soda lime glass, a relatively weak improvement of the mechanical properties of the borosilicate glass was observed after a chemical treatment. The maximal mechanical strength reached on the chemically treated glass is obtained for an ion exchange duration of 15 hours. The strength increase remains low (70% increase) because of the poor amount of sodium modifier in the glass chemical composition. Despite the fact that the indentation technique used is not very reliable on our material because of its particular behavior, it shows an evidence of the beneficial effect of the chemical treatment on the fracture toughness.

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