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Hardness and toughness of sodium borosilicate glasses via Vickers's indentations

Marina Barlet [a,b], Jean-Marc Delaye [c], Thibault Charpentier [d], Mickael Gennisson [c], Daniel Bonamy [a,b], Tanguy Rouxel [e], Cindy L. Rountree [a,b,*]

[a] CEA, IRAMIS, SPEC, SPHYNX, CEA Saclay, CNRS UMR 3680, F-91191 Gif-sur-Yvette, France
[b] CEA, IRAMIS, SPCSI, Complex Systems and Fracture, CEA Saclay, F-91191 Gif-sur-Yvette, France
[c] CEA, DEN, DTCD, SECM, LMPA, CEA Marcoule, BP17171, 30207 Bagnols-sur-Cèze, France
[d] CEA, IRAMIS, NIMBE, LSDRM, CEA Saclay, CNRS UMR 3685, F-91191 Gif-sur-Yvette, France
[e] Département Mécanique et Verres, Institut de Physique de Rennes, UMR 6251, Université de Rennes 1, Campus de Beaulieu, 35042 Rennes cedex, France

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ABSTRACT

This study investigates the mechanical response of sodium borosilicate (SBN) glasses as a function of their chemical composition. Vickers's indentation tests provide an estimate of the material hardness (H_V) and indentation fracture toughness (K_C^{VIF}) plus the amount of densification/shear flow processes. Sodium content significantly impacts the glass behavior under a sharp indenter. Low sodium glasses maintain high connected networks and low Poisson's ratios (ν). This entails significant densification processes during deformation. Conversely, glasses with high sodium content, i.e. large ν, partake in a more depolymerized network favoring deformation by shear flow. As a consequence, indentation patterns differ depending on the processes occurring. Densification processes appear to hinder the formation of half-penny median–radial cracks. Increasing ν favors shear flow and residual stresses enhance the development of half-penny median–radial cracks. Hence, K_C^{VIF} decreases linearly with ν.

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1. Introduction

Portable electronic devices frequently require thin lightweight glass used to protect the internal electronics. As such these glasses need to be resilient to external pressures. A common test to study these protective glasses is micro-indentation from which two important and standard measurements are extracted: (1) hardness (material's resistance to permanent deformation, H_V) and (2) indentation fracture toughness (material's resistance to fracture, K_C). These tests classify the glass's mechanical response into two groups: *anomalous* and *normal* behavior. *Anomalous* glasses predominantly densify under high external pressures. These glasses have a low atomic packing density; thus, the relative movement of the Si–O–Si linkage under pressure leads to the volume shrinkage [1–4]. On the other hand, *normal* behavior implies volume conserving shear flow. This is evidenced by a plastic flow generating pile-up of matter in the vicinity of the indentation without volume change [5–8]. Typically broken bonds and cations favor this phenomenon [9]. The degree at which a glass behaves *normally* and *anomalously* significantly depends on the glass' chemical composition [9–11].

Residual indention patterns vary significantly with the chemical composition [12]. Typically, *anomalous* glasses exhibit cone crack, whereas *normal* glasses predominantly exhibit radial–median cracks [9]. Previous, in-situ indentation studies of *normal* and *anomalous* glasses emphasize variations in deformation processes and effects in the residual stress levels [13]. Variations in contribution of densification versus shear flow alter the indentation shape, the crack appearance and the toughness measurements [5,11,13]. Furthermore, Hagan [7,8,14] highlighted that flow lines which appear in the indentation imprints can pile-up to produce seed cracks for median and radial cracks. In order to discriminate between shear flow and densification in glasses, researchers developed a simple test to estimate the amount of permanent densification under an indenter in glasses and the amount of plastic flow [15–18].

This paper investigates the mechanical response due to indentation in eight SBN glasses of modulated chemical composition. The glass's mechanical response depends on the glass structure. Imaging of the indents provides a means to obtain the hardness (H_V), the crack appearance probability (P_C) and the indentation fracture toughness (K_C^{VIF}, *VIF* implies Vickers's indentation fracture). To understand how matter flows beneath the indenter, AFM imaging before and after annealing discriminates between densification and shear flow mechanisms. For the reader's convenience, Appendix A provides a list of symbols, there meaning, and when appropriate the equation used to calculate them.

The following sections detail experimental techniques: (1) glass fabrication, (2) techniques used in understanding the glass properties (density, elastic moduli, MAS NMR); and (3) measuring and extracting information on the glass's response to Vickers's indenter. The Results

* Corresponding author at: CEA, IRAMIS, SPEC, SPHYNX, CEA Saclay, CNRS UMR 3680, F-91191 Gif-sur-Yvette, France.
E-mail address: cindy.rountree@cea.fr (C.L. Rountree).

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Table 1
Target and measured ICP-AES molar compositions (where [·] ≡ mol%) of elaborated glass samples with their R_{SBN} and K_{SBN} values and physical properties: density (ρ); glass transition temperature (T_g); Young's modulus (E); Poisson's ratio (ν); $\langle CN \rangle$ is the mean coordination number of the boron atoms; the concentration of $^{[4]}B$ per volume unit deduced from NMR measurements and ICP-AES results and number of NBO per volume unit (N_{NBO}) deduced from Eq. (4). Target and ICP-AES measured molar composition and densities were previously published by Barlet et al. [19]. It was not possible to fabricate just one batch of glass to produce all samples; thus multiple batches were fabricated. All batches are within the 10% error of the ICP-AES measurement. Each SBN glass composition has an associated symbol in the subsequent figures. Glass's names won't be recalled on the figures below for clarity.

Name	Target values			Measured via ICP-AES			R_{SBN} measured	K_{SBN} measured	ρ $\left(\frac{g}{cm^3}\right)$	T_g (°C)	E (GPa)	ν	$\langle CN \rangle$	$^{[4]}B \cdot 10^{21}$ (cm^{-3})	$N_{NBO} \cdot 10^{21}$ (cm^{-3})	Symbols
	$[SiO_2]$	$[B_2O_3]$	$[Na_2O]$	$[SiO_2]$	$[B_2O_3]$	$[Na_2O]$										
SBN 12	59.6	28.2	12.2	59.6	23.9	16.5	0.69	2.5	2.463	543	80.1	$0.209_{\pm 0.004}$	3.60	6.78	1.022	△
SBN 25	50.7	23.9	25.4	52.6	20.6	26.8	1.30	2.5	2.545	535	80.3	$0.238_{\pm 0.001}$	3.70	7.22	5.91	▷
SBN 30	47.3	22.3	30.4	51	20.1	28.6	1.44	2.5	2.541	494	74.7	$0.255_{\pm 0.002}$	3.68	6.77	7.36	◁
SBN 35	44	20.6	35.4	46.9	18.6	34.5	1.85	2.5	2.537	467	76.7	$0.264_{\pm 0.0014}$	3.62	5.65	11.21	▽
SBN 14	67.8	18	14.2	70	15.8	14.2	0.89	4.4	2.474	588	81.8	$0.212_{\pm 0.004}$	3.72	5.49	1.34	✳
SBN 63	63.2	16.8	20.0	66.7	14.1	19.2	1.35	4.7	2.524	573	81.9	$0.226_{\pm 0.001}$	//	//	//	◆
SBN 59	59.2	15.8	25	61.1	13.3	25.5	1.91	4.5	2.534	539	77.2	$0.230_{\pm 0.01}$	3.79	5.22	7.39	●
SBN 55	55.3	14.7	30	58.0	12.9	29.1	2.25	4.5	2.538	505	72.8	$0.251_{\pm 0.006}$	3.76	4.86	7.49	★

section presents structural properties, H_V, P_C, K_C^{VIF}, and variations in contribution of densification and shear flow processes in the permanent deformation of sodium borosilicate (SBN) glasses. The Discussion section expounds the glass structure with their mechanical response to a Vickers's indenter. This part also compares and contrasts results presented in Sellappan et al. paper [18]. Furthermore, this section estimates the residual stresses induced during loading and after total unloading.

2. Experimental procedure

This section contains three subsections. It first describes the elaboration process of the glasses studied herein. Then, it details tests used to analyze the glasses' structural/material properties. Finally, it presents tests to understand the material response to microindentation.

2.1. Glass elaboration

Studies herein employ eight sodium borosilicate (SBN) glasses elaborated in-house [19]. During the elaboration process, manual homogenization of the silica (SiO_2), orthoboric acid (H_3BO_3), and sodium carbonate (Na_2CO_3) powders occurs. Platinum/gold (Pt/Au) crucibles retain the homogenized powder during the glass melting process. The formation of the glass melt undergoes three principle steps. Initially, the dehydration of the orthoboric acid takes place at 200 °C for 2 h. Next, the decarbonation of sodium carbonate Na_2CO_3 occurs at 800 °C for 3 h to avoid bubble formation. The final stage produces the glass melt. Depending on the glass composition, this stage occurs between 1100 °C and 1300 °C for 3 h. To avoid residual stress during the cooling process, the glass melt is transferred into a preheated carbon crucible whose temperature is approximately T_g (glass transition temperature). Subsequently, the glass melt enters in a second furnace and cools at a slower rate (10 °C/h) to release the residual stresses.

ICP-AES measurements (conducted by a third party, Prime Verre) verify the chemical compositions of the SBN glasses. Table 1 summarizes the target and measured values. ICP-AES measurements give approximately 10% error for each oxide. Several batches were fabricated to produce all samples. All of them are within the 10% error of the ICP-AES measurement. The glasses are classified depending on their $R_{SBN} = \frac{[Na_2O]}{[B_2O_3]}$ and $K_{SBN} = \frac{[SiO_2]}{[B_2O_3]}$ ratios.

2.2. Structural investigation

A glass's mechanical response is linked intrinsically to its structure. Thus, it is important to understand and quantify several glass parameters including density, elastic moduli, and the environment around the boron atoms.

2.2.1. Density, ρ

The densities of the glasses are estimated by Archimedes' principle. The geometry is a cylinder of thickness 10 mm and diameter 30 mm. Tests are conducted at ambient conditions using a hydrostatic balance. Initially, the glass samples are weighted in air (m_d) and water (m_w). Then by multiplying by the density of water (ρ_w) one can arrive at the density of the sample (ρ):

$$\rho = \frac{m_d}{m_d - m_w} \times \rho_w \tag{1}$$

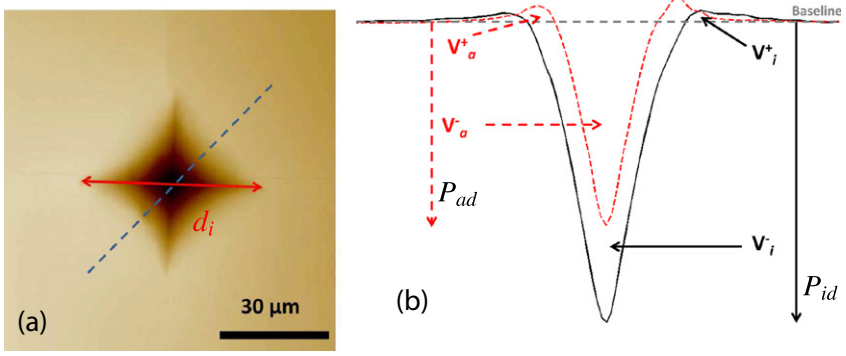

Fig. 1. (a) A typical indentation imprint used to determine the indentation diagonal length, (d_i and marked by a continuous line) and to estimate the pile-up profile (dotted line). (b) Sketch showing the evolution of indentation prints before (black solid line) and after annealing (red dotted line). V^+ and V^- represent the volume above and below the baseline (gray dotted line), respectively.

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