

Mechanical behaviour of glass during cyclic instrumented indentation

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Indentation techniques are largely used nowadays for characterizing the intrinsic mechanical properties of brittle materials. A cyclic indentation tests have been carried out on soda lime glass and borosilicate glass using an instrumented indentation apparatus. Repeated post-threshold Vickers indentations were made using various peak loads. The hardness, elastic modulus and fracture toughness of the two glasses were evaluated using the consecutive load–displacement curves. Their properties of were compared and discussed.

Keywords: *glass; hardness; fatigue crack propagation; stress analysis*

1. Introduction

Indentation techniques are appropriate for characterizing the fracture behaviour of brittle materials. They are simple, economical and efficient methods that can be used to determine the material fracture parameters (toughness, and sub-critical crack-growth characteristics) and to analyse brittle contact damage problems (erosion, wear) [1]. Their applications have been extended over the last two decades with the development of instrumented equipments that allow the continuous measurement of the indenter depth under loading [2–4]. Instrumented indentation is a sensitive technique that can probe the mechanical properties of materials (Young modulus and hardness) at submicron scales from the load–displacement data (Fig. 1). Loads at the milli-Newton scale and displacements lower than 1 nm can be measured. It was used for studying metal dislocations [5, 6], toughened ceramics fracture behaviour [7], material coatings [8, 9], residual stresses [10] and the tribological behaviour of materials [11–13].

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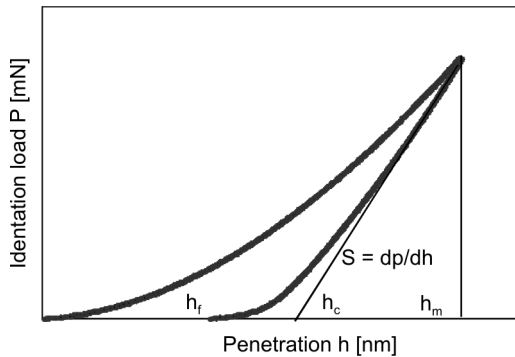


Fig. 1. Typical load–displacement indentation curve

When a Vickers indenter is loaded against a brittle material surface, it induces a plastic zone under the residual imprint. Above a critical loading level, in the post-threshold domain, two crack systems form during a loading–unloading cycle. A radial–median cracks system evolves normal to the surface along the imprint diagonals followed by a lateral crack system parallel to the surface [1] (Fig. 2).

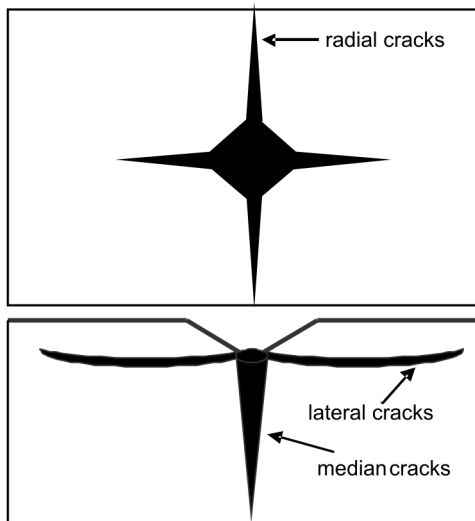


Fig. 2. Vickers indentation crack systems in glass

The material fracture toughness can be evaluated by measuring the radial cracks and the lengths of imprint diagonals with the knowledge of the applied load and the material elastic modulus using formula of Anstis et al. [14]:

$$K_{Ic} = 0.016 \sqrt{\frac{E}{H_v}} \frac{P}{C^{3/2}} \quad (1)$$

where P is the peak applied indentation load, E is the elastic modulus, H_v is the material hardness and C is half the length of the two radial cracks (measured from the imprint centre).

Both the hardness and the elastic modulus can be derived from the load–displacement curve. Figure 1 shows a typical load–displacement indentation curve during a loading–unloading cycle. In this figure, h_m represents the displacement at the peak load $P_{\max}$, h_c is the depth along which the indenter is in contact with the sample during loading, h_f is the final displacement after complete unloading, S is the initial unloading contact stiffness. From the load–displacement curve, the hardness obtained at the peak load is defined as:

$$H = \frac{P_{\max}}{A} \quad (2)$$

where A is the projected contact area. Determining the projected contact area from a load–displacement curve requires the contact depth h_c to be known. The elastic modulus of the indented sample can be inferred from the initial unloading contact stiffness [15]:

$$S = \frac{dP}{dh} \quad (3)$$

Brittleness is crucial to ceramic and glass materials in contact damage issues. The ranking of susceptibilities of brittle materials to contact damage is based on indentation brittleness indexes. With the use of the Vickers indentation, Lawn and Marshall [16] proposed the brittleness index B , defined as the ratio of the hardness H to the fracture toughness K_{Ic} :

$$B = \frac{H}{K_{Ic}} \quad (4)$$

Cyclic fatigue characterization is usually used for describing the degradation of the mechanical properties of metals with time under repeated subcritical loads. Cyclic fatigue on brittle materials has not been extensively studied as for metals because it was thought insignificant in terms of their lack of plasticity. Some ceramics exhibit the mechanical fatigue effect related to the microstructure toughening mechanisms present in these materials [17–19]. Without any toughening mechanisms, glasses are considered resistant to mechanical cyclic fatigue. The observed glass mechanical strength deterioration under cyclic loading was explained by stress corrosion (environmentally assisted crack growth process).

However, recent works on borosilicate glass showed an evidence of a mechanical effect in addition to the stress corrosion effect at very low sub-critical crack growth velocities, using a standard fatigue test method [20]. In the context of brittle contact damage problems, repeated indentation was used to study the cyclic fatigue of ceramics [21–23]. The method is based on the relation between the applied indentation load and the number of subsequently repeated indentations needed to produce radial cracks (for sub-threshold loads) [23], or chipping (for post-threshold loads) [21].

2. Experimental

CSM indentation equipment (Micro-Combi-Tester, CSM Instruments, Switzerland) was used for our tests. It is essentially composed of three parts, as shown in Fig. 3. The first part corresponds to the indentation and scratch instrument. It has a sample holder table (4), an indenter holder (3) connected to the amplification and signal transformation system (1) and an acoustic probe (2). The second part consists of an optical system that enables imprint observation and magnification with the use of different objectives (6) and a display screen. The third part is the computer (7) used for test programming and data acquisition.

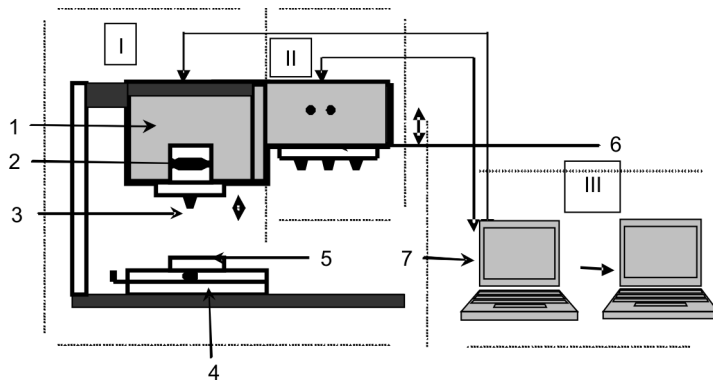


Fig. 3. Scheme of the CSM indentation tester. See the text for details

Table. 1. Chemical compositions [wt. %] of the tested glasses

Soda lime glass		Borosilicate glass	
Oxide	Content	Oxide	Content
SiO ₂	72.851	SiO ₂	69.734
Al ₂ O ₃	1.354	Al ₂ O ₃	2.549
Na ₂ O	12.729	Na ₂ O	6.761
K ₂ O	0.478	K ₂ O	3.071
CaO	8.249	CaO	1.508
MgO	4.097	B ₂ O ₃	12.086
Fe ₂ O ₃	0.098	ZrO ₂	0.399
SO ₃	0.151	SO ₃	0.033
BaO	0.007	BaO	2.318
TiO ₂	0.469	ZnO	1.115

Soda lime and borosilicate flat glass samples were used in this study. Their chemical compositions are given in Table 1. The sample thicknesses were 3 and 5 mm for the soda lime and borosilicate glasses, respectively. Samples of the dimensions 40×20 mm² were fabricated. They were annealed at 530 °C for 40 min in order to eliminate residual

stresses. Each sample was glued to a metallic pellet fixed on the sample holder. Its surface was previously cleaned before testing.

Indentations have to be spaced apart by 2 mm intervals in order to avoid their mutual interactions. The peak loads applied for the different indentations were: 1 N, 2 N, 3 N, 4 N and 5 N. For each load, the number of loading–unloading cycles was varied from 1 to 5. The loading and unloading speed was maintained at a constant rate of 1 N/min. A dwell time of 5 s at the peak load was used at every cycle. The minimal contact load during unloading was 0.2 N. Every test was repeated three times in order to get reliable results. Imprint micrographs were systematically taken for both glasses.

3. Results and discussion

The load–displacement curves obtained during repeated indentations under the 5 N peak load for the two glasses are presented in Fig. 4. It can be noticed that there is a hysteresis effect and a looping shift toward higher displacements as the number of repeated indentations increases, which are more pronounced for soda lime glass than for borosilicate glass.

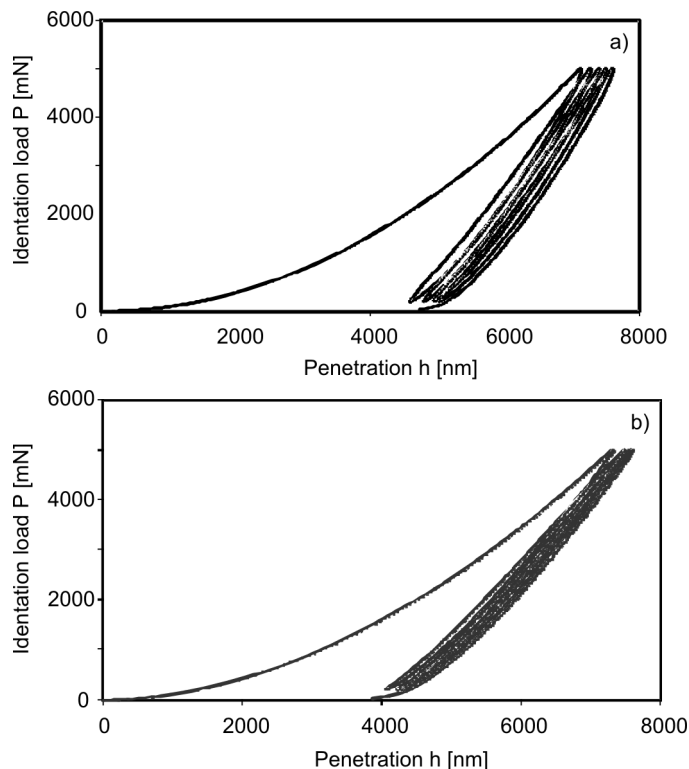


Fig. 4. Cyclic load–displacement curves for:
a) soda lime glass, b) borosilicate glass

The hysteresis effect reveals that the unloading is not only due to elastic recovery but includes also a certain amount of plastic reversibility. The dissipated energy related to non-elastic effects during the hysteresis process can be measured from the obtained closed loop surfaces. This energy, more important for soda lime glass, decreases as the indentation cycle number increases. The permanent penetration depth after the fifth cycle is 5 μm and 3.9 μm for the soda lime and the borosilicate glass, respectively. This leads to a smaller elastic recovery E_r for soda lime glass (E_r is the ratio of the recovered to the total surface of the loading displacement curve) [24].

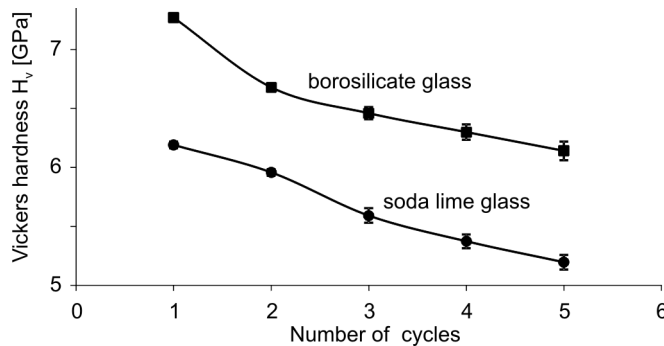


Fig. 5. Dependence of hardness on the number of indentation cycles for 5 N peak load

The hardness values (Fig. 5) reveal a similar decreasing trend upon the increasing number of indentation cycles for both glasses. It is probably related to the hysteresis effect. Such a trend was not observed with subthreshold loads in other papers when the three first cycles were avoided [3]. The measured values of hardness from the first load–displacement curve (6.2 GPa and 7.27 GPa for the soda lime glass and the borosilicate glass, respectively) are higher than the previously reported values (5 GPa and 6 GPa). The lower values obtained at the fifth indentation are closer to these values. Based on the mean value obtained from three measurements in every test, it is noticed that the hardness values obtained with instrumented indentation exhibit an insignificant variation between 0.4% and 0.94% for the first indentation and the fifth one, respectively.

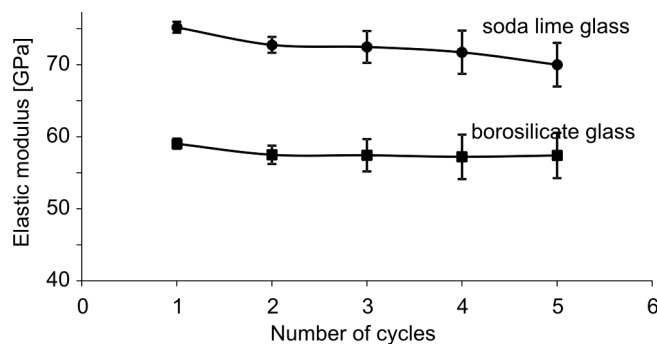


Fig. 6. Dependence of the elastic modulus on the number of indentation cycles for 5 N peak load

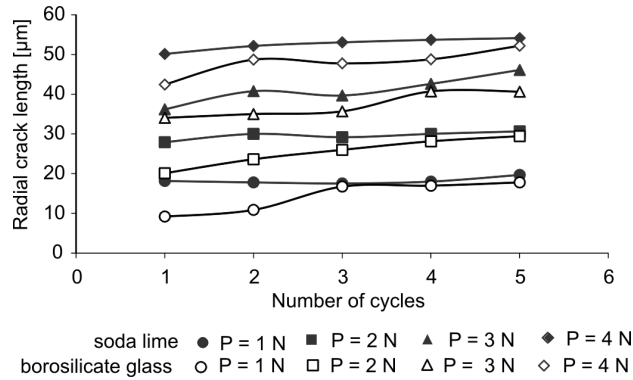


Fig. 7. Dependence of radial crack length on the number of indentation cycles using different peak loads

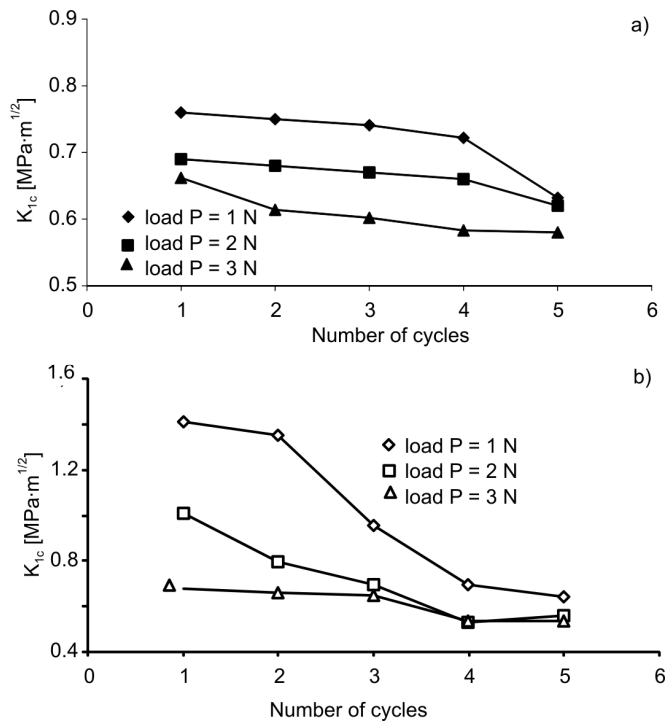


Fig. 8. Dependence of fracture toughness on the number of indentation cycles for: a) soda lime glass, b) borosilicate glass

The obtained elastic moduli for the borosilicate glass seem to stabilize after the first indentation cycle, as seen in Fig. 6. Contrarily, the values obtained for the soda lime glass appear less consistent with the more pronounced hysteresis effect. It can be noted that Young's moduli obtained using instrumented indentation for both glasses, exhibit a significant scatter. Typical variations are in the range from 1% for the first

indentation to 6% for the fifth indentation. Nevertheless, it remains acceptable if one considers the fact that Young's moduli, obtained by instrumented indentation, rely on localized measurements, highly sensitive to local defects like micro-cracks, and cumulative residual stresses induced during the indentation cycles.

Figure 7 shows the radial crack length evolution with the cycle numbers for various peak loads corresponding to the two glasses. The same increasing trend of the radial crack lengths clearly appears for all peak indentation loads. This is probably due to the increase in the residual stresses as the number of cycles increases.

For both glasses, the obtained values of fracture toughness from the Anstis et al. formula (Fig. 8) are much lower than previously accepted values. The determination of fracture toughness is not only influenced by the variation of the hardness and Young modulus, but also by the cumulative residual stresses induced during the indentation cycles. With regard to the obtained variations of hardness and the Young modulus, the observed decrease in the fracture toughness with the cycle number can be explained by the effect of cumulative residual stresses on the propagation of the radial cracks. This effect is more apparent on the borosilicate glass.

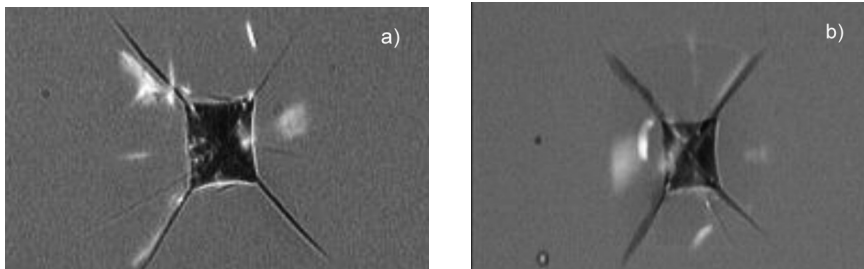


Fig. 9. Vickers indentation imprints for 4 N peak load after 5 cycles ($\times 400$): a) soda lime glass, b) borosilicate glass

For a peak load of 5 N, soda lime glass was found to have a brittleness index that increases from $9.98 \mu\text{m}^{-1/2}$ at the first cycle up to $10.60 \mu\text{m}^{-1/2}$ at the fifth cycle. For the same testing conditions, the borosilicate glass showed a variation from $10.19 \mu\text{m}^{-1/2}$ up to $11.24 \mu\text{m}^{-1/2}$. This increase of the brittleness index is clearly related to the changes in fracture toughness, which is more important than that of the hardness. Observations of the two glasses indentation imprints generated during the cyclic loading showed more pronounced scaling in the borosilicate case. The contact damage characteristics (imprints, radial cracks, etc.) for the soda lime glass did not vary that much during cycling. The photographs in Fig. 9 show longer radial cracks for soda lime glass under the same peak load (4 N).

4. Conclusions

For the mechanical properties obtained during repeated indentations, we observe a similar qualitative behaviour for both soda lime and borosilicate glasses. The hardness

evaluation is clearly influenced by the hysteresis effect affecting the load–displacement curves. The values obtained during the first four cycles are higher than those usually accepted. The values of the Young modulus seem to stabilize after the first cycle and are closer to expected values. It is therefore important to reduce the hysteresis effect, as was suggested in the literature, by using a longer dwell time at the peak load and avoid the first cycles, where this effect is preponderant. The obtained values for the fracture toughness are much lower than the accepted values for both glasses. They decrease, particularly for the borosilicate glass, as the cycle number increases. This is probably due to the cumulative residual stress effect on the radial crack propagation. From the brittleness index values, it appears that the use of instrumented Vickers indentation is appropriate for detecting the variation in the brittleness index.

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