THE DRIVING FORCE FOR INDENTATION CRACKING IN GLASSES

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ABSTRACT

The indentation cracking behavior of glasses depends much on the glass composition and on the loading conditions (temperature, speed). This is partly because, in contrast to other inorganic solids, glasses may be permanently compacted beneath the indenter, and the silica content of soda-lime-silica glasses governs the amplitude of the phenomenon. Besides, many inorganic glasses exhibit indentation-creep at room temperature. In addition, the elastic recovery observed upon unloading during indentation experiment varies much from one glass to the other. All these phenomena (flow densification, indentation creep and elastic recovery) reduce the amount of energy available for crack initiation and propagation. As a result, for a given indentation experiment (given load, loading time, indenter shape and environment), the driving force for indentation cracking depends much on the glass composition. So will also the indentation fracture toughness parameter (K_c), which may thus greatly differ from the intrinsic fracture toughness (K_{1c}).

1. INTRODUCTION

The experimental determination of the fracture toughness may give rise to different values depending on the way the measurement is achieved. The Single-Edge Notched Beam (SENB) test is a self-consistent method suitable to obtain a critical stress intensity factor, K_{1C} , close to the intrinsic value, provided the notch tip is sufficiently sharp. The Vickers Indentation Fracture (IF) technique allows for an estimation of the "so-called" indentation fracture toughness, K_{C} .

Glasses	Window-type					Aluminosilicate		
	WG1	WG2	WG3	WG4	WG5	CaSiAlO	YSiAlO	YSiAlON
SiO ₂	70.8	79	68.0	71	80	41.9	65.6	52.7
Si ₃ N ₄	-	-	-	-	-			7.7
Li ₂ O			4.2			-		
Na ₂ O	12.8	13	5.4	17.5	12.1		-	
MgO	5.9	4	4.8	-	-			-
CaO	10.2	1	9.2	11.5	7.9	41.9		
B_2O_3	-	-	4.3			-		
Al ₂ O ₃	0.4	2	1.2	-	-	16.2	12.5	14.4
Y ₂ O ₃	-	-	-			-	21.9	25.2
Other	-	1	2.1				-	-

Table 1: Glass composition expressed in molar percentages.

This paper discusses the differences observed between the IF and SENB fracture toughness values. Different parameters have a direct and significant influence on the Kc values, among which: (i) the indentation load, (ii) the indentation loading time and (iii) the time which elapses between the indentation and the crack-length measurements. It is thus necessary to study the influence of such parameters in order to compare the indentation fracture toughness values of different glasses. Besides, the environment may also affect the crack-growth kinetics and the fatigue resistance was therefore also characterized. The effect of both the indentation environment and the fatigue (post-indentation) duration is discussed. The compositions of the investigated glasses are given in Table 1.

2. RESULTS AND DISCUSSION

Glasses densify more or less under an indenter depending on their compositions, so that different crack systems may form. Moreover, a given glass may exhibit a load-dependent behavior, with ring cracks preferentially forming at high loads (Fig. 1), revealing an increasing trend to densification flow process with the load. Fracture toughness values were measured by the SENB (except for YSiAlO and YSiAlON glasses) and by the indentation fracture (IF) techniques. For silica-rich glasses, the indentation fracture toughness value was higher than the K_{1C} value obtained from the SENB technique. The opposite trend was observed for harder and stiffer (high Young's modulus) glasses such as CaSiAlO glasses. For standard window glasses, similar fracture toughness values were obtained by means of IF and SENB experiments. These results show that the indentation fracture toughness was overestimated in the case of the silica-rich glasses. In fact, it is suggested that a flow densification process occurs and that as a Hertzian-type contact prevails, radial crack extension is limited. It was previously shown that radial crack opening is governed by the elastic stress relaxation caused by the indenter removal.



Fig. 1 - Scanning electron micrographs of indentations performed under 4.91 N (left) and 49.05 N (right) indentation loads for the WG2 glass (79 mol. % SiO₂).

The flow densification process leads to structural changes at the atomic or molecular scale and was already deduced from refractive index change beneath the indenter. In "anomalous-like" glasses such as silica-rich glasses, the occurrence of flowdensification results in much lower residual stress in the area surrounding the indentation print, which causes the reductions of the radial crack length and consequently to the overestimation of the fracture toughness. Normal glasses, for which $K_C < K_{1C}$, thus differ from anomalous-like glasses (here silica-rich glasses) for which, $K_C > K_{1C}$. The anomalous character is enhanced both by a low compactness of the vitreous network (large free volume fraction) and a low modifier oxide content.



Fig. 2 - Change in the glass brittlness as a function of the density

No evidence for flow densification was observed in the CaSiAlO glass, nor in the YSiAlO(N) glasses by the present authors, consistently with the fact that these glasses exhibit relatively high network compactnesses, typically over 0.4, where the compactness parameter reflects the free volume content and thus the susceptibility of a glass to densification. Rare-earth aluminosilicate glasses are much more resistant to sub-critical crack growth than soda-lime-silica glasses. Nitrogen enhances this trend: the YSiAlON glass is more resistant than its oxide counterpart. The silica-rich glasses are less sensitive to indentation fatigue than standard window glasses, which means that glass fatigue is a function of both the modifier oxide content and the modifier type present in the glass susceptibility to moisture attack, while alkaline-earth cations moderate this trend. Glass composition has a marked effect on the rate of crack growth.

4. CONCLUSIONS

The indentation fracture toughness parameters were studied for different silicate glasses. The radial crack length increased as a function of the indentation load, but at loads larger than 49 N circular cracks were observed, giving evidence for a Hertzian-type contact. At constant indentation loads, the post-indentation radial crack length increased as a function of the loading time, and this fatigue phenomenon depends much on the glass composition. Fracture toughness values are different depending on considered experimental technique, i.e. IF or SENB. For the silica-rich glasses, the indentation fracture toughness was overestimated while for stiffer and harder glasses, the opposite trend was observed. The flow densification process occurring in low-compactness (i.e. silica-rich) glasses is thought to be responsible for this trend, some of the indentation-loading energy being dissipated in the flow densification process and therefore unavailable for cracking. The static fatigue behavior was also investigated from the indentation crack length versus time data and the following conclusions were drawn: (i) indentation cracks propagate in the same velocity range as long cracks from constant load experiments on notched specimens, (ii) the glass fatigue resistance depends strongly on the composition, and (iii) both the nitrogen content in aluminosilicate glasses and the silica content in the silicate glasses increase the resistance to sub-critical crack growth.

5. REFERENCES

- A.N. Georoffand C.L. Babcock, "Relation of microindentation hardness to glass composition", J. Am. Ceram. Soc., 56 2 97-99 (1973).
- [2] G.R. Anstis, P. Chantikul, B.R. Lawn and D.B. Marshall, "A critical evaluation of indentation techniques for measuring fracture toughness: I, direct crack measurements", J. Am. Ceram. Soc., 1981, 64, 9, 533-538.
- [3] K.W. Peter, "Densification and Flow Phenomena of Glass in Indentation Experiments", J. Non-Cryst. Sol., 5 103-115 (1970).
- [4] A. Arora, D.B. Marshall, B.R. Lawn and M.V. Swain, "Indentation deformation/fracture of normal and anomalous glasses", J. Non-Cryst. Sol., 31 415-428 (1979).
- [5] S. Dériano, T. Rouxel, M. Lefloch and B. Beneu, "Structural and mechanical properties of alkali-alkaline earth-silicate glasses", J. Phys. Chem. Glasses, 45 37-44 (2004).
- [6] S. Dériano, A. Jarry, T. Rouxel, J.-C. Sangleboeuf and S. Hampshire, "The indentation fracture toughness (K_c) and its parameters: case of silica-rich glasses", J. Non-Cryst. Sol., 344 44-50 (2004).