Indentation cracking of glass II: Relationship between crack resistance and equibiaxial flexural strength

S. Yoshida[1], T. Shimizu[1], Y. Kato[2], H. Yamazaki[2], J. Matsuoka[1]
[1] *The University of Shiga Prefecture, 2500, Hassaka, Hikone, Shiga 522-8533, Japan*
[2] *Nippon Electric Glass Co., Ltd., 2-7-1, Seiran, Otsu, Shiga 520-8639, Japan*

The indentation-induced crack initiation test is a simple and useful method in order to evaluate brittleness of glass. However, no clear physical meaning is given to the critical load of the crack initiation. In our previous studies, it was shown that the crack initiation load, which is defined as crack resistance (CR), was affected much by densification, in other words by residual stress field beneath an indenter. The purpose of this study is to investigate relationship between indentation-induced crack initiation and equibiaxial flexural strength. CR of glass is determined from Vickers indentation tests, and the equibiaxial flexural strength is obtained from Ring-on-Ring flexural test. From the results of crack initiation loads and flexural strength, it is found that CR of glass is affected not only by the residual stress field but also by the flexural strength of the glass. The indentation cracking occurs when the residual stress is larger than the critical fracture stress.

1. Introduction

It is considered that indentation cracking of glass is one model case of chipping, erosion, or abrasion of glass. These mechanical failures of glass are severe problems in cutting and handling of thin sheet glass, such as an LCD (Liquid Crystal Display) or PDP (Plasma Display Panel) glass. It is, therefore, important to put in light relationship between the indentation-induced cracking and macroscopic fracture phenomena in glass. In Part 1, we discussed relationship between load dependence of indentation deformation and indentation cracking, and pointed that load dependence of densification under a Vickers indenter was related with indentation cracking. In Part 2, it is shown that Crack Resistance (CR), which is the crack initiation load in Vickers indentation test, can be determined both from the residual stress around an indentation impression after unloading and from equibiaxial flexural strength. The driving force of indentation cracking is the residual stress around the indentation impression, and the critical fracture stress without any deformation is estimated from the flexural strength of a sheet glass. The indentation cracking of glass is a fracture-related phenomenon, and occurs when the residual stress after unloading is larger than the critical fracture stress without any deformation.

2. Experimental procedures

Samples employed in this study are listed in Table 1. They are 9 types of commercial glasses and three ternary borosilicate glasses. Vickers hardness, fracture toughness (SEPB method), and elastic moduli of all the samples were determined.

CR is determined from Vickers indentation tests.[i] Vickers indentation tests were performed in air (25ºC, r.h. 30 %). The indentation load increased step-by-step from 10 gf to 2000 gf, and 20 indentations were performed at each load. In the indentation tests, some cracks appeared at the corners of the indentation mark. The number of the corners with cracks depended on the indentation load. The crack forming probability was determined as the ratio of the number of the corners with cracks to the total number of the corners. The crack forming probability was plotted against the applied load to determine CR, which is defined as the indentation load at which the crack forming probability would be 50 %.

Table 1 Glass systems*.

Name	Glass system	Name	Glass system
A	SiO_2-B_2O_3-K_2O	G	SiO_2-PbO-B_2O_3
B	SiO_2-B_2O_3-Na_2O	H	SiO_2-Al_2O_3-Li_2O
C	SiO_2-Al_2O_3-B_2O_3	I	Transparent Glass Ceramics
D	SiO_2-Na_2O-CaO	$0B_2O_3$	$80SiO_2 \cdot 20Na_2O$
E	SiO_2-Na_2O-SrO	$20B_2O_3$	$60SiO_2 \cdot 20B_2O_3 \cdot 20Na_2O$
F	SiO_2-SrO-K_2O	$40B_2O_3$	$40SiO_2 \cdot 40B_2O_3 \cdot 20Na_2O$

* The glass system shows three of the most components oxides in glass.

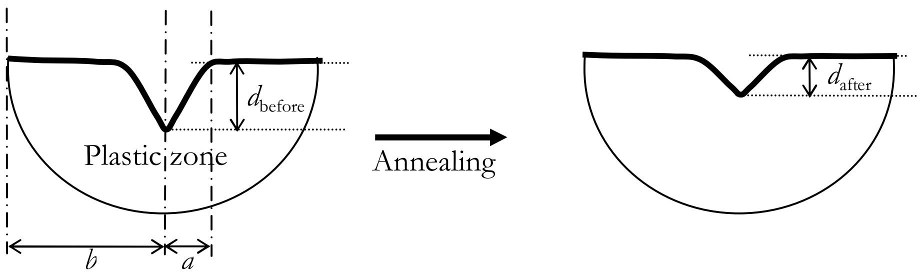

Fig. 1 Schematic model of indentation impression before and after annealing.

The residual stress around an indentation impression after unloading can be estimated by using the model of plastic zone embedded in a perfect elastic matrix.[ii] As shown in Fig. 1, only a half-sphere region, called as plastic zone, of characteristic radius of b can be deformed, and the deformed region is embedded into an elastic medium. The lost volume by indentation, ΔV, which is proportional to a^3, is an origin of a volume strain, $\Delta V/V$, where V is the volume of plastic zone, and is proportional to b^3. The residual stress at the elastic/plastic interface, σ, is given by Eq. (1).

$$\sigma = \kappa(\Delta V/V)\,, \qquad (1)$$

where κ is the bulk modulus of material. This model is applied to evaluate fracture toughness of brittle materials. The indentation deformation is divided into mainly two deformation mechanisms, shear flow and densification. The densification is a non-volume conservative process. This means that the densified volume does not contribute to the volume strain and does not contribute to the residual stress. In our previous studies, we reported that the volume deformed by shear flow was determined from measuring the indentation volume after annealing, because only the densified volume can be recovered by annealing.[iii, iv] In this study, we assume that only the indentation depth is recovered by annealing, and that the volume of plastic zone is proportional to the indentation volume. By using Eq. (2), the residual stress around the indentation impression can be estimated from the indentation depth after annealing, d_{after}, and the indentation depth before annealing, d_{before}.

$$\sigma \propto \kappa(a^2 d_{\mathrm{after}} / a^3) \propto \kappa(d_{\mathrm{after}} / d_{\mathrm{before}}), \qquad (2)$$

where a is the indentation diagonal. In Eq. (2), it is assumed that there's no compositional dependence of elastic recovery during unloading half cycle. Vickers indentation tends to produce various cracks, which obstructs the measurement of the indentation depth. Therefore, a Knoop indenter was used because of its less tendency of crack formation. The bulk modulus was determined from sound velocities, and the depth of indentation was measured using a laser scanning microscope (Keyence, VK-9500) before and after annealing. The annealing was performed at Tg X 0.9 (K) for 2 hours. Other experimental details were reported in elsewhere.[v]

Eqibiaxial flexural strength of the sample was measured using the Ring-on-Ring biaxial flexural test geometry. Both faces of the samples with thickness of 1 mm were polished quite carefully. The radius of the load ring and the radius of the support ring are 5 mm and 10 mm, respectively. Teflon sheets (0.1 mm thick) were placed between the rings and sample in order to reduce friction. The displacement rate of the cross head (Instron 1362) was 2.5 mm/min. Fracture stress was calculated according to the reported analysis.[vi]

3. Results

Figure 2 shows the relationship between CR and Vickers hardness. There is no relation with each other. This is also the case for fracture toughness (Fig. 3). CR is independent of the resistance to deformation and of the resistance to crack propagation. In Fig. 4, it is found a good negative relationship between the residual stress estimated from Eq. (2) and CR. Larger residual stress after unloading results in smaller CR. Figure 5 shows relation between CR and biaxial flexural strength. CR of glass increases with increasing fracture stress of the glass.

4. Discussion

As shown in Figs. 1 - 4, indentation cracking of glass can be explained not from conventional mechanical parameters, but from the residual stress and the fracture stress. The fracture origin of biaxial fracture test would be a surface flaw. The origin of indentation cracking is still unclear at the present stage, but the surface and/or subsurface conditions would affect cracking behaviors. CR depends on a finishing condition. CR of top surface of float method is larger than that of mechanical polishing surface of the identical glass[vii]. Taking into consideration of stress concentrating at the corner of indentation impression, the surface condition affects cracking behavior, or CR. Therefore, the biaxial fracture stress is a measure of crack initiation stress without any deformation. It is considered that CR is the indentation load, at which the residual stress takes precedence over the fracture stress of the glass.

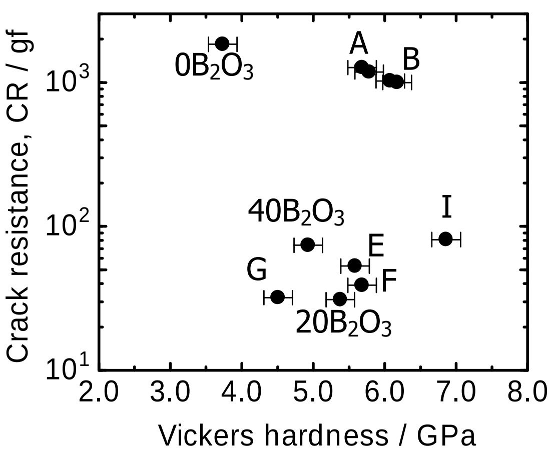

Fig. 2 Vickers hardness vs CR.

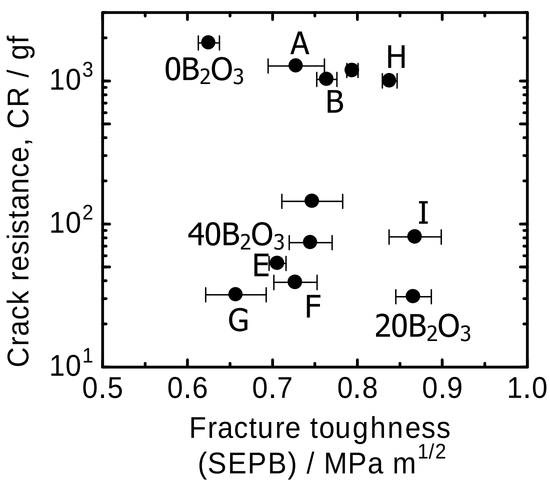

Fig. 3 Fracture toughness vs CR.

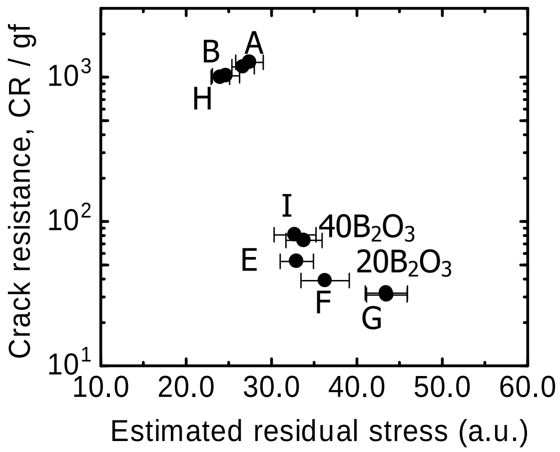

Fig. 4 Estimated residual stress vs CR.

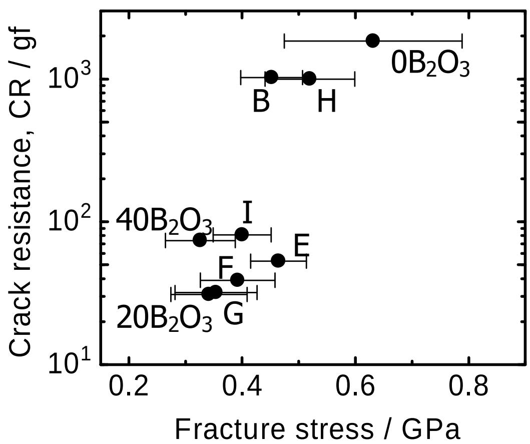

Fig. 5 Fracture stress vs CR.

The residual stress varies with the indentation load, because the ratio of densified volume to the total indentation volume changes with the load. With increasing the indentation load, the ratio of densified volume to the total indentation volume decreases. This means that ΔV in Eq. (1) increases with the indentation load, and that the residual stress increases with the load. So, we define the criterion of indentation cracking as shown in Eq. (3).

$$\sigma_{\mathrm{R0}}\left(\frac{P}{P_0}\right)^x = \sigma_{\mathrm{f}} \qquad (3)$$

where σ_{R0} is the residual stress at the indentation load of P_0, P is the crack initiation load, x is constant, and σ_f is fracture stress, which is the biaxial flexural fracture stress. Substituting CR for P in Eq. (3), Eq. (4) is obtained and plotted in Fig. 6.

$$CR \propto \left(\frac{\sigma_f}{\sigma_{R0}} \right)^{1/x} \qquad (4)$$

CR is determined from the residual stress around the indentation impression after unloading and from the fracture stress of the glass. In Eqs. (3) and (4), however, it is assumed that x value does not depend on glass composition. The compositional variations of this x value and the size of plastic zone in Fig. 1 must be taken into consideration in order to apply this relationship to a wide variety of glasses.

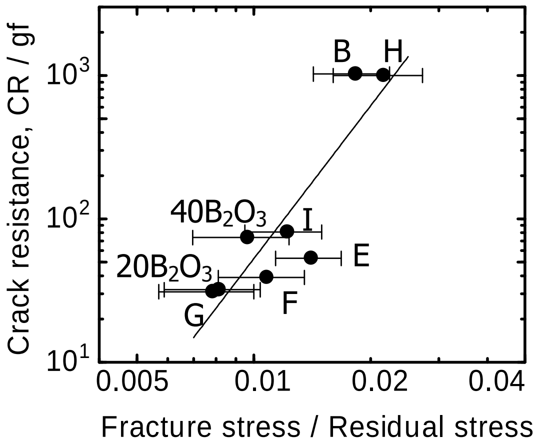

Fig. 6 Variation of CR with the ratio of fracture stress to residual stress.

5. Conclusion

In order to clarify the relationship between indentation cracking and macroscopic fracture, the equibiaxial flexural test was performed for various types of glass. The crack initiation load (CR) on indentation tests is affected both by the residual stress around the indentation impression and by the equibiaxial flexural stress. Assuming that the residual stress depends on the indentation load, CR can be derived from the residual stress and the fracture stress.

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[v] Y. Kato, H. Yamazaki, S. Yamamoto, S. Yoshida, J. Matsuoka in *Proc. 11th International Congress on Fracture,* 2005 (CD-ROM)

[vi] C1499-05, in *Annual Book of ASTM Standards*, edited by ASTM International (West Conshohocken, PA, 2005).

[vii] Unpublished data.