Indentation cracking of glass
I: Load-dependence of deformation during Vickers indentation test

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The indentation test is widely used for evaluating susceptibility to crack initiation. Crack initiation by indentation is affected much by deformation during the indentation, which includes three modes; elastic deformation, plastic flow, and densification. In our previous study, there was close correlation between crack initiation load and densification. However, difference in crack initiation load among glasses was much larger, compared with that in densification. In this study, each deformation during indentation and their load-dependence were evaluated by using a dynamic hardness tester and atomic force microscope (AFM). In Glass showing high crack initiation load, large elastic deformation and large densification at high load were found. It is supposed that small Young's modulus under high compression and large densification at high load lead to the large difference of crack initiation load among glasses.

Introduction

Susceptibility to crack initiation is one of the most important properties determining strength of glass products. The indentation test is widely used for evaluating susceptibility to crack initiation due to its convenience. Wada[1] proposed "Crack resistance (CR)" to evaluate the susceptibility to crack initiation. Crack resistance is defined as the load at which crack initiates at 50% of corner of indentation. When glass is being indented, deformation occurs around the indenter. Crack initiation by indentation is affected much by deformation during the indentation, which includes three modes; elastic deformation, plastic flow, and densification. In our previous study[2], relationship between CR and densification was investigated. Degree of densification was evaluated by "Recovery of indentation depth (RID)", which is the ratio of change in indentation depth after heat-treatment to the depth before the heat-treatment. (Details are described in the next section). It was found that there was close correlation between CR and RID (Figure 1). Densification does not generate residual stress around the indenter, so glass with large RID value shows high crack resistance.

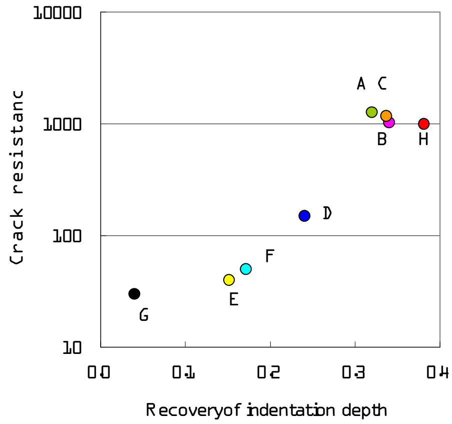


Figure 1. Relationship between crack resistance and recovery of indentation depth among various glass compositions (Glass A to H)[1].

As you can see in Figure 1, however, difference in crack resistance among glasses was much larger, compared with that in RID. The value of RID ranged from 0.05 to 0.4 (as much as 8 times), while crack resistance ranged form 10 to 1000 gf (about two orders!). Therefore, some other characteristics should affect CR.

Since the mode and the amount of deformation change with load, the amount and distribution of stress change during indentation. However, the change of deformation during indentation with load was not taken into account in the previous study. In this study, the change in each deformation mode was evaluated by a dynamic hardness tester and AFM, and relationship between CR and each deformation was discussed.

Experimental

Three types of glass composition were selected from the glasses in Figure 1 to evaluate deformation during the indentation: that is, Glass C (aluminoborosilicate glass, CR = 1200gf), Glass D (soda-lime glass, CR=120gf), and Glass G (Lead glass, CR=30gf) . The glass system and properties of the glasses are shown in Table 1. These glasses show much different crack resistance from each other. The glass sheets with a size of about 30mm x 30mm x 1mm were prepared and used for the indentation test.

The indentation test was conducted with a dynamic hardness tester attaching a Vickers indenter. One cycle of the indentation test consists of loading, holding for 15 sec, and unloading. The load and displacement of the indenter were recorded during the cycle of indentation, so that the load–displacement curve can be obtained. The maximum load varied from 5 gf to 200 gf, and the indentation speeds were 1.35 gf/sec and 2gf/sec for 5-10 gf and 50-200gf, respectively, which were the higher limit of the hardness tester for each load.

Degree of densification was evaluated by RID. After indented by the dynamic hardness tester, the depth of the indentation, d_{before}, was measured by AFM. The indented specimens were heat-treated at temperature of 0.9 x Tg (Tg: glass transition temperature in ℃) for 2 hours, and the indentation depth, d_{after}, was measured again. The ratio of the depth difference, d_{before} - d_{after}, to the depth before the heat-treatment, d_{before}, was defined as RID.

Table 1. The glass system and properties of the glasses used in this study.

	System	d (g/cm^3)	CR (gf)	Hv (GPa)	E (GPa)	Tg (℃)
Glass C	SiO_2-Al_2O_3-B_2O_3	2.48	1200	590	70	710
Glass D	SiO_2-Na_2O-CaO	2.49	120	570	68	540
Glass G	SiO_2-PbO-B_2O_3	4.44	30	460	64	470

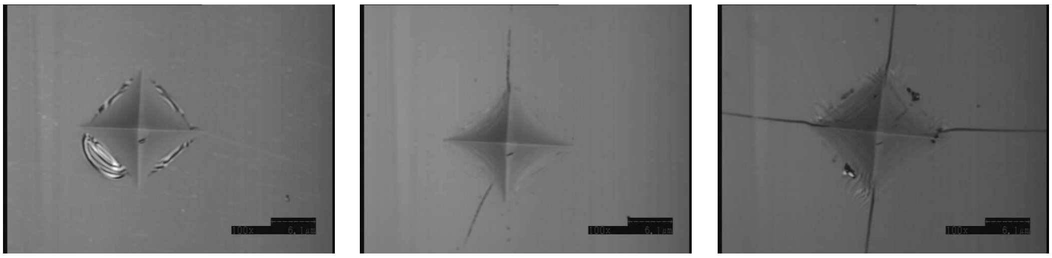
* d: density, CR: crack resistance, Hv: Vickers hardness, E: Young's modulus, Tg: glass transition temperature.

Results

The microscopic images of the indentation are shown in Figure 2. They are the indentations at a load of 100gf. There was difference in shape and position of cracks among the glasses, as well as difference in shape of indentation. In Glass C, which shows higher CR, no radial crack was initiated, but some cracks parallel to the indentation edge were found. In Glass D, radial cracks were initiated near the corners of the indentation, and the indentation edge lines were concave. In Glass G, radial cracks were initiated at all of the indentation corners, and lateral cracks were initiated at the bottom. The edge lines were less concave than that in Glass D.

The load-displacement curves of these glasses are shown in Figure 3, where the maximum load was 200 gf. The displacement after unloading corresponds to Vickers hardness, Hv. Glass with higher value of Hv showed larger displacement after unloading. There was also difference in the maximum displacement among glasses, and the difference is due to the difference in the amount of elastic deformation. However, Glass C, which has the highest value of Young's modulus, E, showed the largest maximum displacement, and Glass G, which has the lowest value of E, showed the smallest. Because the E value varies with load, the E value under high compression is different from the one under zero stress. From the slope of the load-displacement curve (Figure 3) near the maximum load during unloading and the maximum depth, the E value of Glass C at 200gf is estimated to be smaller than that of Glass G. It is thought that the E value of Glass C under high compression is the smallest.

Relationship between RID and the maximum indentation load is shown in Figure 4. Glass C and Glass D show almost the same value of RID at load less than 20gf. The RID values decreased with increasing the maximum indentation load, and Glass D showed larger decrease than Glass C. Glass C showed larger value of RID at 200 gf than Glass D. The RID value of Glass G was smaller than the other glasses at all the maximum indentation load. It was found that Glass C has the largest RID value at high load among the glasses.



(a)Glass C (b) Glass D (c) Glass G

Figure 2. Microscopic image of the indentation at a load of 100gf: (a) Glass C, (b) Glass D, (c) Glass G. (The width of the images is 45 μm.)

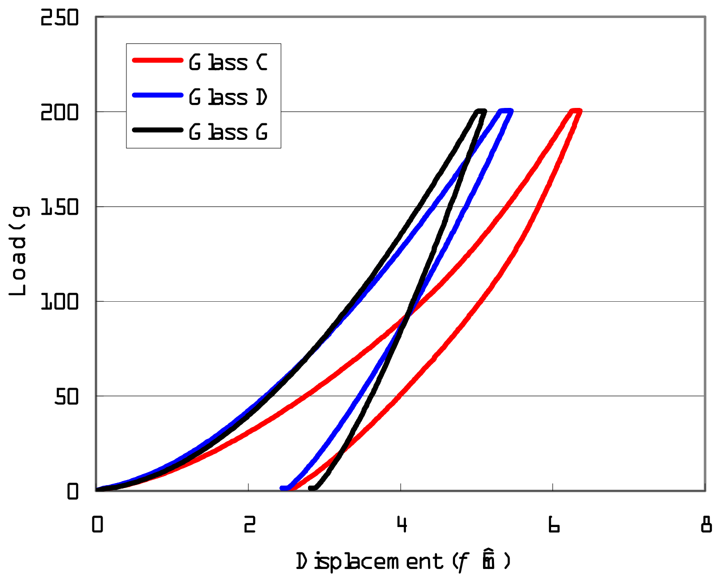


Figure 3. Load-displacement curve of Glass C, D and G. The maximum load is 200gf.

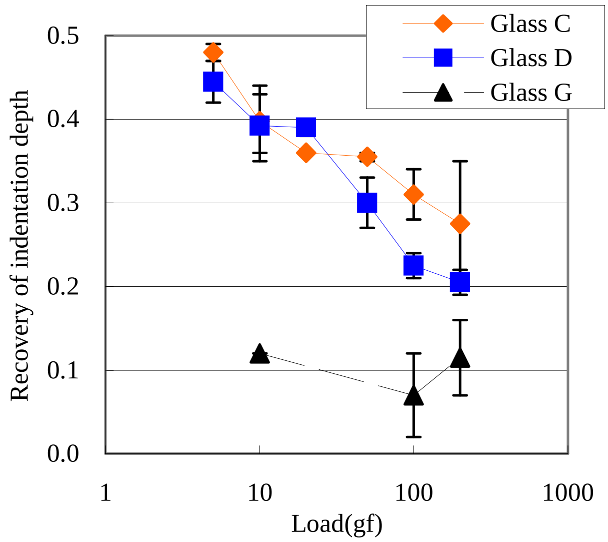


Figure 4. Relationship between load and recovery of indentation depth.

Discussion

It was found that Glass C showing higher value of CR has two characteristics about deformation during indentation. One is the small E value under high compression and the other is the large RID value at high load. The two characteristics are thought to raise the value of CR.

The radial cracks are initiated by the residual stress, and then appear at the corners of the indentation like Figure 2(c). The residual stress after indentation was described as follows[3]:

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

where σ, κ, ΔV, and V are residual stress, bulk modulus, plastic flow volume, and volume of plastic zone, respectively. The RID value affects the ratio of the plastic flow volume to the plastic zone. Glass with larger RID value shows smaller ratio or smaller residual stress. The RID value decreases with increasing load, so even if Glass C and Glass D show the same RID value at small load, there is large difference in the RID value at high load due to difference in load-dependence of RID value. That is supposed to be one cause of large difference in CR among glasses.

The E value under high compression affects the stress distribution around the indentation. Since Glass C shows the smaller E value, the region under high stress is hard to occur. So the stress distribution broadens around the indenter. The broad distribution of stress results in the small residual stress. Therefore, the small E value under high compression leads to high value of CR. However, if much large elastic deformation occurs during loading, large surface tension occurs during loading just outside of the contact area, resulting in initiation of cracks parallel to the edge line, like Figure 2(a).

Both of the small E value under high compression and the large RID value at high load are thought to be due to large free volume in glass structure. The E value under high compression is attributed to the packing of glass structure. So glass with large free volume is thought to show the small E value under high compression. Moreover, it is understood easily that glass with large free volume shows large RID value.

Conclusion

Deformation during indentation test and their load-dependence were evaluated by using a dynamic hardness tester and atomic force microscope (AFM). It was found that glass showing high crack resistance has the small E value under high compression and large RID value at high load. These characteristics lead to broad distribution of stress during loading and small residual stress. The characteristics of glass under high stress are very important to evaluate the susceptibility to crack initiation in glass.

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[1] Wada in *International Congress of Glass,* 1974, Vol.11, p.39.

[2] Y. Kato, H.Yamazaki, S. Yamamoto, S.Yoshida, and J.Matsuoka in *Proc. 11th International Conference on Glass,* 2005 (CD-ROM).

[3] B.R.Lawn, A.G. Evans and D.B. Marshall, Journal of the American Ceramic Society **63**, p. 574 (1980).