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Evaluation of Indentation Induced Residual Stress in the Surface of Float Glass

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圧子によるフロートガラス表面残留応力の評価

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Residual stress field around a Vickers diamond indentation was studied using float glass as a model specimen. The basis of the experimental technique is to use cracks from a small indentation as a microprobe to measure the residual stress at a specific point around a large indentation and to study the change of the residual stress by heat treatment. Residual tensile and compressive stresses around a large indentation produced by applying the load of 19.6 N were distributed up to the distance of about 300 μm from the indentation center. The tensile and compressive stresses were 10 MPa and -40 MPa, respectively, at a distance of 200 μm from the indentation center. The residual stress decreased by heat treatment and became zero by keeping the indented specimen at 550°C, a temperature which is close to the annealing point of the glass, for 2h. The depth of the indentation also decreased by heat treatment. This technique can be widely applied to study residual-stress-related phenomena in ceramics as well as in glass.

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Key-words : Float glass, Vickers indentation, Residual stress, Fracture mechanics, Heat treatment

1. Introduction

It is well known that the residual stress in glass and ceramics affects the mechanical, electrical and magnetic properties of materials. Extensive studies have been carried out of the measurement of residual stresses based on various techniques.^{1)–4)} Uchida recently reported an analytical technique to determine residual stress in thin film by asymmetric X-ray diffraction.⁴⁾ Zeng and Rowcliffe developed an indentation technique²⁾ to measure the residual stress field around Vickers indentations. The technique uses a small indentation as a microprobe to measure the stress field at a specific position around a large indentation. The crack length of the small indentation changes under the influence of the residual stress field created by the large indentation. The residual stress is calculated based on a simple fracture mechanics model.

In the present study, the indentation technique was applied to evaluate the change of the residual stress field; creation of the stress field by an indentation and reduction by subsequent heat treatment. The rise and fall of the residual stress was discussed taking the viscous flow and elastic strain of glass into consideration.

2. Experimental procedure

Experiments were made using the top surface of float (soda-lime) glass, the thickness of which was 5 mm. The Vickers diamond indentation experiment was carried out at room temperature (24–25°C, with 42–50% humidity) with Akashi AVK hardness tester. The primary indentation was made with a load of 19.6 N. The second indentation was made with a load of 4.9 N. In each indentation, the load was applied for 15 s. The second indentation was oriented so that the cracks form in the radial and tangential directions relative to the primary indentation. The introduction of the second indentation will affect the stress field created by the primary indentation. Therefore the load to make the second indentation should be small so that the effect on the stress field created by the primary indentation can be minimized.

The length of the cracks from the second indentation in the radial and tangential directions with regard to the primary indentation was measured on a TV monitor which was connected to the hardness tester. The center-to-center distance between the two indentations was also measured on the screen. The sequence of indentation experiments was repeated at positions of different center-to-center distance. The length of the cracks from the second indentation was measured and the tensile and compressive residual stresses were calculated.

The heat treatment was carried out in an electric furnace after the primary indentation. The glass specimen was heated at either 450°C for 2h or 550°C for 2h, respectively, in air. The specimen was cooled to room temperature and the second indentation was made. Crack length was measured and the residual stress was calculated in the same way as was adopted in the as-indented specimen. The indentation profiles were characterized with a DEKTAK3 Version 2.01 a/J instrument for as-indented and heat-treated specimens.

3. Results and discussion

3.1 Crack profiles under residual stress

Figures 1(a) and (b) show examples of crack patterns of second indentations made under the influence of the primary large indentation. Figure 1(a) shows that in the area near the primary indentation, there is a tangential tensile stress which propagates the crack in the radial direction. A compressive stress is developed in the radial direction and the length of cracks in the tangential direction decreases. Figure 1(b) shows that at a far distance from the primary indentation, the influence of the residual stress diminishes to zero and the second indentation behaves as a normal indentation.

Figure 2 gives an example of crack patterns of second indentation which is located close to the primary indentation which had been heat-treated at 550°C for 2h before the second indentation was made. No or little influence of the

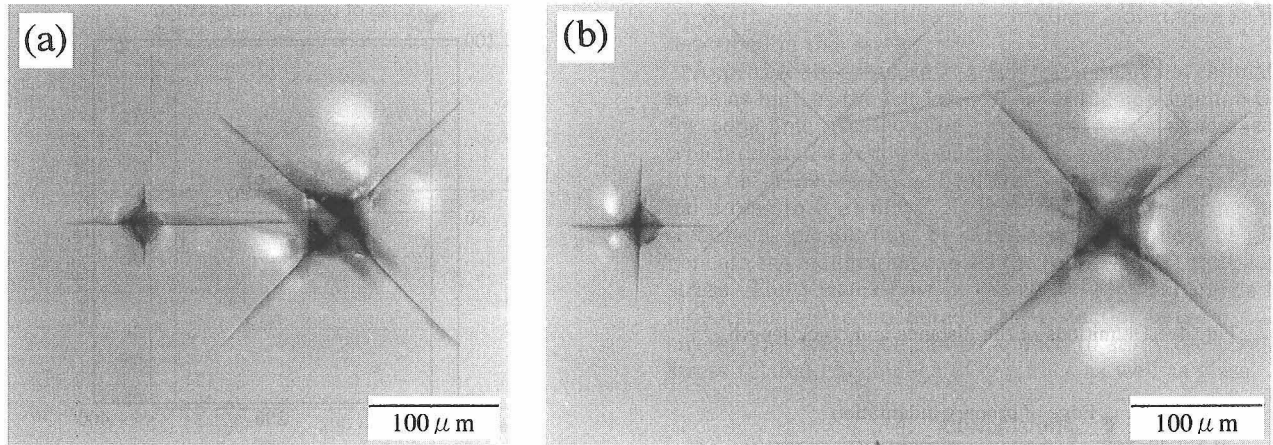


Fig. 1. (a) Optical micrograph of indentations; as-indented, near distance.
(b) Optical micrograph of indentations; as-indented, far distance.

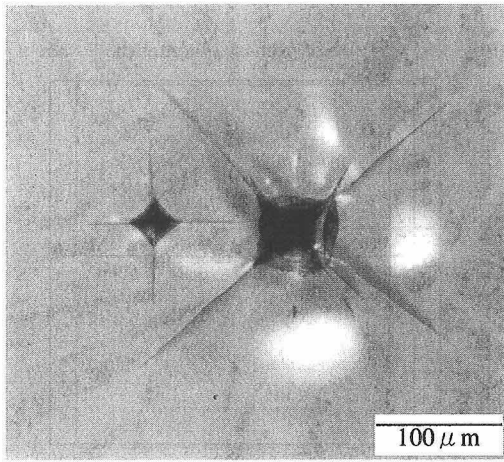


Fig. 2. Optical micrograph of indentations; after heat treatment at 550°C.

residual compressive and tensile stresses from the primary indentation could be observed and the second indentation behaves as a normal indentation.

3.2 As-indented residual stress

By measuring the secondary crack length, it is possible to calculate the residual stress field around the primary indentation. In order to calculate the residual stress around an indentation, fracture mechanics analysis has been proposed by Zeng and Rowcliffe.²⁾ Their analysis is based on the original derivation of indentation fracture toughness by Anstis et al.⁵⁾

The residual stresses are calculated by using the following equations.

$$\sigma_r = K_c \frac{1 - (C_0/C_1)^{3/2}}{\Phi C_1^{1/2}} \quad (1)$$

for tensile residual stress

$$\sigma_c = -K_c \frac{1 - (C_0/C_2)^{3/2}}{\Phi C_2^{1/2}} \quad (2)$$

for compressive residual stress

where K_c is the stress intensity factor at the indentation crack tip which is not affected with residual stress, C_0 is the crack length at a load P without residual stress and Φ is a geometrical constant and $\Phi = \pi^{1/2}$ for surface cracks. A

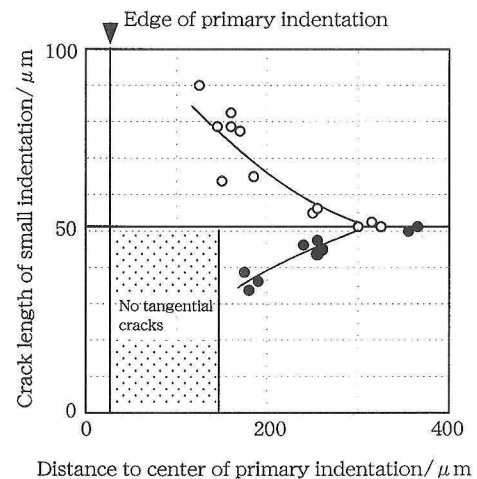


Fig. 3. Crack length of 4.9 N indentations under the residual stress of 19.6 N indentation; as-indented. ○ Cracks in radial direction, ● cracks in tangential direction.

value of K_c of $0.75 \text{ MPa} \cdot \text{m}^{1/2}$ was used in the subsequent calculation.⁶⁾ It is assumed that both tensile and compressive stresses are constant over the secondary crack. This assumption is good only if the secondary crack is small. However, for simplification, this assumption will be made in the present paper. The crack from the second indentation extended from C_0 to C_1 under tensile residual stress σ_r from the primary indentation. While, the crack length decreased from C_0 to C_2 under compressive residual stress σ_c .

In Fig. 3, crack length from small indentation (with a load of 4.9 N) is plotted against the distance, which is defined as the distance between the crack tip of small indentation and the center of the primary indentation (see Fig. 4). The plot shows that, near the primary indentation, the secondary cracks in the radial direction relative to the primary indentation are extended and longer than a normal indentation crack ($C_0 = 52.4 \mu\text{m}$ with standard deviation of $1.4 \mu\text{m}$). On the other hand, no or shorter cracks appear in the tangential direction relative to the primary indentation. Especially, no tangential cracks are observed in the area where the distance is less than about $150 \mu\text{m}$.

Figure 5 shows that residual tensile and compressive stresses from the primary indentation distributed to the distance of about $300 \mu\text{m}$. The stress decreases as the dis-

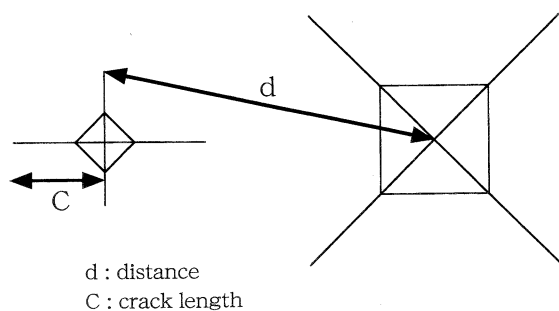


Fig. 4. Definitions of the distance and crack length.

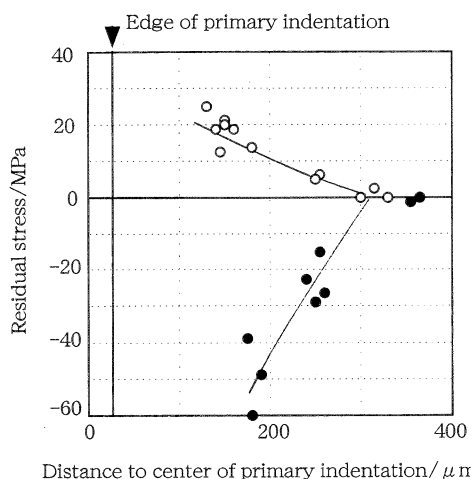


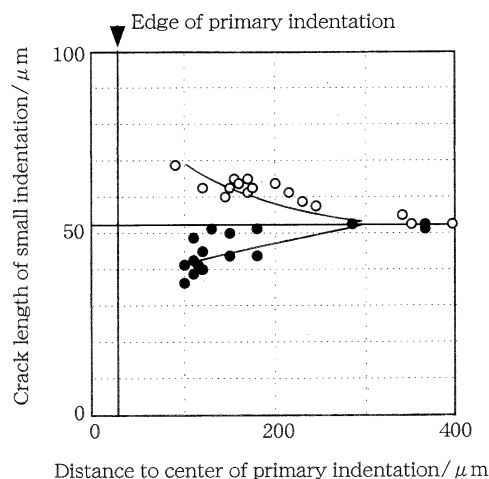
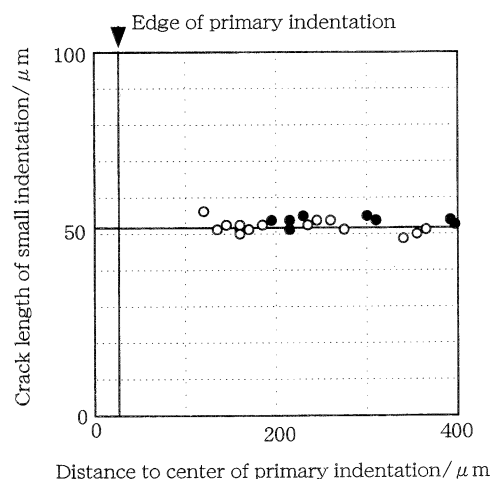
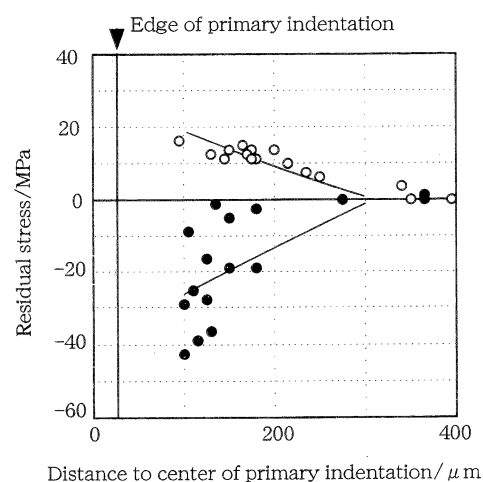
Fig. 5. Residual stress around a 19.6 N indentation; as-indented. ○ Residual stress in tensile direction, ● residual stress in compressive direction.

tance from the center of the first indentation increases. In the area where the distance is below $130\ \mu\text{m}$, the cracks from the second indentation interfere with the cracks from the primary indentation and the accuracy of the calculation of residual stress is apparently affected. It was a general trend that the compressive stress was larger than the tensile stress; at the distance of $200\ \mu\text{m}$, the compressive stress was about $-45\ \text{MPa}$ while the tensile stress was about $10\ \text{MPa}$.

3.3 Residual stress after heat treatment

In Figs. 6 and 7, crack length from the second indentation after heat treatment is plotted against the distance. The plot shows that the difference between stressed and unstressed crack lengths is reduced both at 450°C and 550°C . Figures 8 and 9 give the calculated residual stress based on the plots in Figs. 6 and 7. Residual tensile and compressive stresses from the primary indentation are reduced after heat treatment and decreased to zero after heat treatment at 550°C ; annealing point where the viscosity of glass is about 10^{13} poise, for 2h. The profiles of the primary indentation; as-indented and after heat treatment at 450°C or 550°C , are shown in Fig. 10 along with the summary of residual stress. Each profile is representing the average of two profiles. The depth of Vickers indentation after $19.6\ \text{N}$ loading was $6.3\ \mu\text{m}$ and was reduced to $4.2\ \mu\text{m}$ and $3.7\ \mu\text{m}$, respectively, after 450°C and 550°C heat treatment. Ridges were observed at the edges of indents. They were about $0.7\ \mu\text{m}$ high for both as-indented and heat-treated specimens.

The basic mechanism for the creation of indenter induced residual stress can be discussed based on Hill's elastic/plastic internal cavity analysis.⁷⁾ The analysis was

Fig. 6. Crack length of $4.9\ \text{N}$ indentations under the residual stress of $19.6\ \text{N}$ indentation; 450°C , 2h-heated. ○ Cracks in radial direction, ● cracks in tangential direction.Fig. 7. Crack length of $4.9\ \text{N}$ indentations under the residual stress of $19.6\ \text{N}$ indentation; 550°C , 2h-heated. ○ Cracks in radial direction, ● cracks in tangential direction.Fig. 8. Residual stress around a $19.6\ \text{N}$ indentation; 450°C , 2h-heated. ○ Residual stress in tensile direction, ● residual stress in compressive direction.

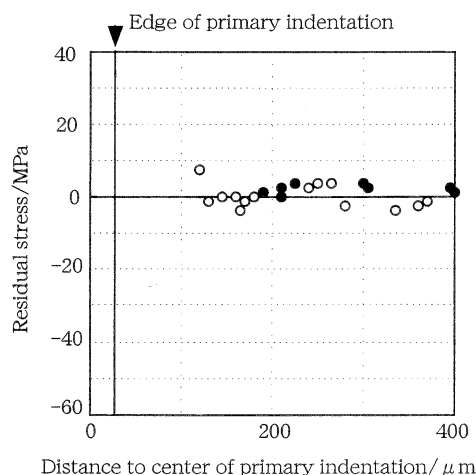


Fig. 9. Residual stress around a 19.6 N indentation; 550°C, 2h-heated. ○ Residual stress in tensile direction, ● residual stress in compressive direction.

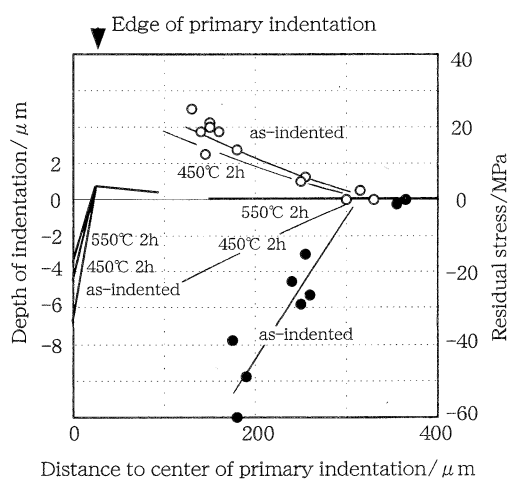


Fig. 10. Summary of residual stress and indentation profile. ○ Residual stress in tensile direction, ● residual stress in compressive direction.

further extended by Lawn et al.⁸⁾ whose analysis is based on a model in which the elastic/plastic field beneath the indenter is resolved into elastic and residual component. The radially expanded plastic zone, after unloading, exerts a hydrostatic pressure across the outerboundary of the plastic zone hence causing a tangential elastic tensile stress and a radial elastic compressive stress outside the boundary. The residual stress is detected and calculated by mea-

suring the crack length of the secondary indentation as was described in this study.

Approximate pressure beneath the indenter is estimated to be as high as the hardness value of the specimen, 5 GPa for soda-lime glass.⁶⁾ The pressure causes plastic zone which created a residual elastic stress field which extended to as far as 300 μm . The heat treatment reduced the residual stress to 0 at 550°C. It is assumed that, during heat treatment, plastic flow or precisely viscous flow of glass beneath the indentation caused the reduction of the residual stress. The viscous flow in the area which surrounds the indentation also contributed to the reduction of the residual stress. This technique can be applied to study the residual stress related phenomena of ceramics as well as glass.

4. Conclusions

Residual stress field around a Vickers diamond indentation was studied using float glass as a specimen. The basis of the experimental technique is to use a small indentation to measure the residual stress at a specific point around a large indentation which was formed by applying the load of 19.6 N. The results are summarized as follows.

(1) Residual tensile and compressive stresses distributed around the large indentation to the distance of 300 μm from the center of the indentation. The tensile and compressive stresses were 10 MPa and -40 MPa, respectively, at the distance of 200 μm .

(2) The residual stress decreased by heat treatment and became zero by keeping the indented specimen at annealing point (550°C for 2h). The depth of indent also decreased by heat treatment.

(3) This technique can be applied to study the residual stress related phenomena of ceramics as well as glass.

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