Relaxation behaviors of Vickers indentations in soda-lime glass

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Introduction

Permanent deformation of glass can be divided into two modes.

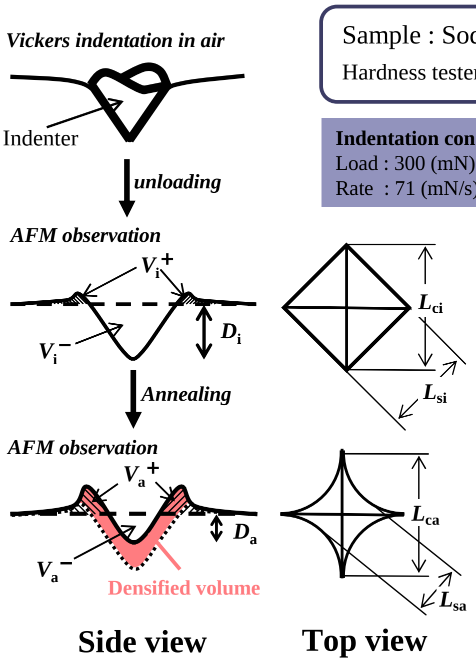
Permanent deformation of glass

Plastic flow (shear flow) | **Densification** (Recovered by thermal annealing)

There are no quantitative studies based on relationship between annealing conditions and recovery of indentation volume.

In this study, annealing recovery of indentation-induced densification was studied under several annealing conditions, using a Vickers hardness tester and an atomic force microscope (AFM).

Experimental procedure



Sample : Soda-lime silicate glass (Matsunami S-0050)
Hardness tester : Shimadzu DUH-201 AFM : Veeco NanoscopeE

Indentation conditions
Load : 300 (mN)
Rate : 71 (mN/s)

Annealing conditions

Annealing temperature	Annealing time
214℃ (Tg×0.6) K	15 min
295℃ (Tg×0.7) K	30 min
458℃ (Tg×0.9) K	2hr
539℃ (Tg)	

Shrinkage ratio of indentation volume

Densified volume

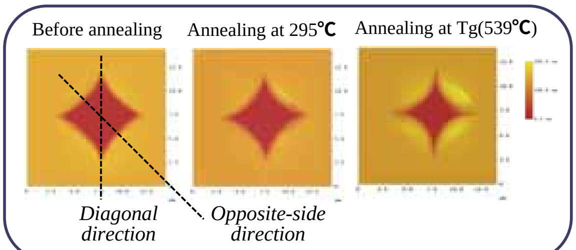
$$V_R = \frac{(V_i^- - V_a^-) + (V_a^+ - V_i^+)}{V_i^-}$$

Shrinkage ratio of length and depth

Shrinkage ratio of diagonal length L_c, opposite-side length L_s and depth D is

$$X_R = \frac{X_a - X_i}{X_i} \qquad X = L_c, L_s, \text{ or } D$$

Results and Discussion



These AFM images clearly show that the shrinkage of diagonal is very limited. And that large shrinkage is observed in opposite-side length.

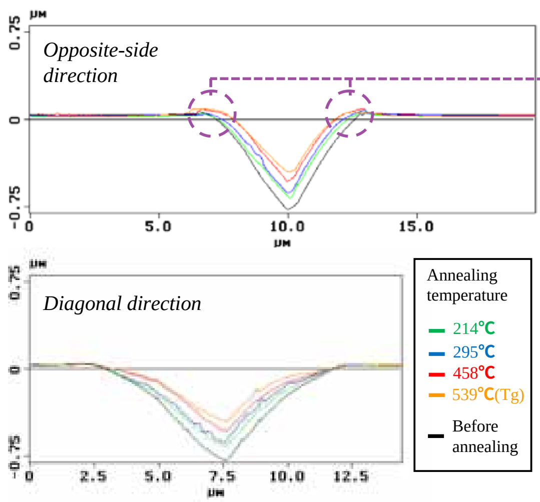


Fig.1 AFM cross-section profiles of a residual impression after annealing.

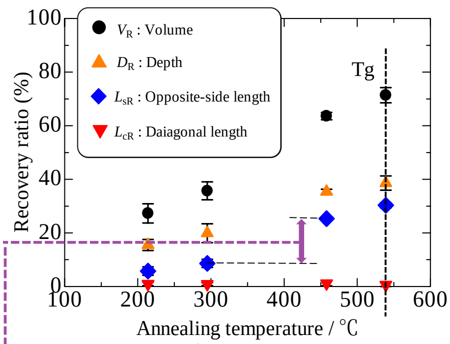


Fig.2 Variation of recovery ratio with annealing temperature

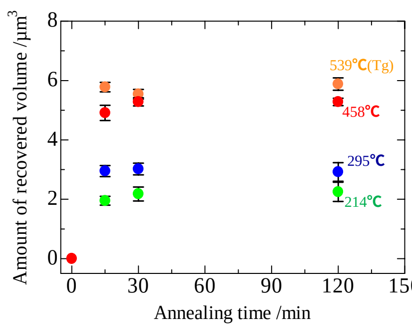


Fig.3 Variation of recovered volume with annealing time.

From Fig.2, shrinkage ratios of length and depth increase in the order of
Diagonal length < Opposite-side length < Depth

The recovery ratio of indentation volume by annealing increases up to **70%** with increasing the annealing temperature to Tg (539℃).

Fig.3 indicates that the relaxation behavior of densified volume mainly depends on annealing temperature. (The relaxation is completed in a short annealing time.)

Small density increment

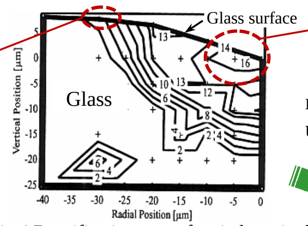


Fig.4 Densification map of an indentation[1]

Large density increment

In previous study[1], a quantitative densification map of an indentation-densified area was obtained using Raman microspectroscopy.

Large shrinkage of L_s is observed by annealing at high temperature.

The recovered region by annealing at a given temperature is much affected by a distribution of density increment.

Summary

○ Densification contribution of soda-lime glass accounts for 70% of the indentation volume.

○ Densification gives a major contribution to the indentation depth and a minor contribution to the diagonal.

○ The recovered region by annealing at a given temperature is determined from the microscopic glass density which is affected by the pressure distribution under a Vickers indenter.

References

[1] A.Perriot et al. , *J. Am. Ceram. Soc.*, 89 (2005)592.