THE UNIVERSITY OF SHIGA PREFECTURE

Inelastic Deformation of Glass beneath Sharp Diamond Indenters

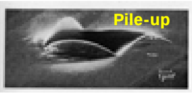
○S. Yoshida, H. Sawasato, T. Sugawara, Y. Miura, J. Matsuoka

Center for Glass Science and Technology, The University of Shiga Prefecture, JAPAN

Introduction

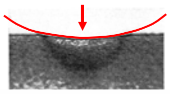
What is the deformation mechanism of brittle material, such as glass, especially at room temperature ?
It has been a controversial problem from 1970's.

K.W. Peter, *J. Non-Cryst. Solids* 5(1970) 103.



Pyramidal indentation on soda-lime glass (Opposite face angle = 70 °) Cf. Vickers 136 °

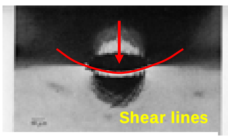
Sharper indenter



Change in refractive index

Ball indentation on soda-lime glass (Radius = 20 μm, Load = **100 gf**)

Larger load



Ball indentation on soda-lime glass (radius = 80 μm, load = **1100 gf**)

Shear flow — Densification — Shear flow

Although the deformation mechanism is surely related with cracking, or brittle behaviors of glass even in sub-micro scales, only a few reports focus on deformation of glass beneath an indenter.

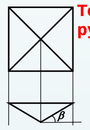
The purpose of this study is :
1. To propose a separation method of the permanent densification part from the total indentation volume.
2. To clarify the effects of glass composition on densification.
3. To evaluate relationship between densification and indenter geometries or indentation conditions.

Experimental Procedures

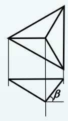
Samples: Soda-lime silicate glass, and other glasses (Microscope slide, Matsunami S-0050)

Indentation test:
Indentation load = 50 ~ 300 mN
Holding time = 15 ~ 999 sec.

Diamond indenter:



β = 22° (Vickers)

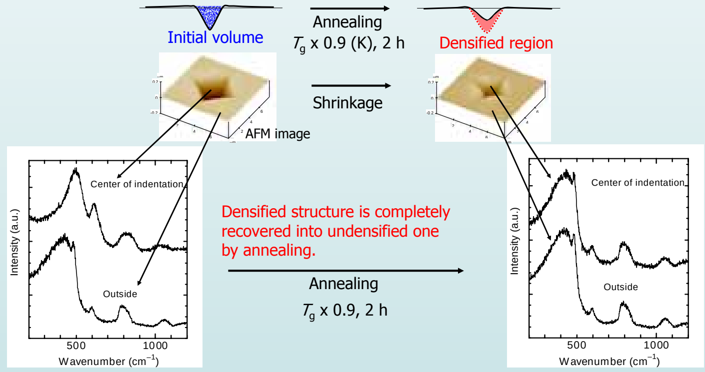


β = 10°
β = 25° (Berkovich)
β = 55° (Cube-corner)

Separation method of densified part from total indentaton volume :

Densified region can be relaxed by annealing at around T_g

Mackenzie(1963), Neely & Mackenzie(1968), Yoshida (2001, 2005, 2007, 2009)



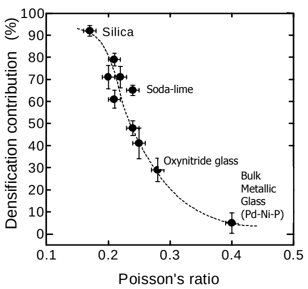
Raman spectra of silica glass before annealing

Raman spectra of silica glass after annealing

Densification contribution (%) = Densified volume / Initial volume

Results and Discussion

Effect of glass composition (Vickers indenter):



Densification contribution vs. Poisson's ratio

1. Various kinds of glasses except for BMG experience densification beneath a Vickers indenter.
2. Densification contribution for silica glass is > 90 % !
3. Densification contribution decreases with increasing Poisson's ratio, or free volume in glass.

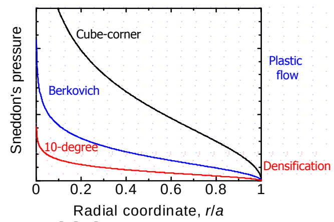
S. Yoshida *et al., J. Mater. Res.,* 20(2005) 3404.
S. Yoshida *et al., Phys. Chem. Glasses,* 50(2009)63.

Sneddon's pressure distribution for an equivalent rigid cone

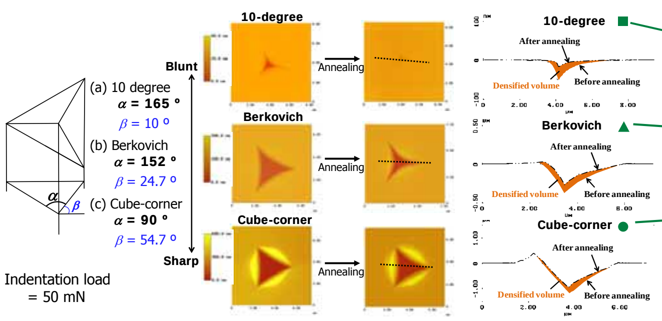
I.N. Sneddon, *Int. J. Eng. Sci.,* 3(1965) 47.

$$p(r) = \frac{E}{2(1-\nu^2)} \frac{\cosh^{-1}(a/r)}{\tan\Psi} \quad 0 \le r \le a$$

E : Young's modulus, ν : Poisson's ratio, a : contact radius, r : radial coordinate in the surface, Ψ : half included angle

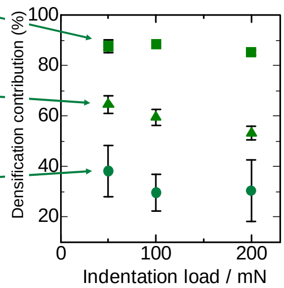


Effect of indenter geometry (Trihedral pyramids):



AFM top-views — AFM cross-sections

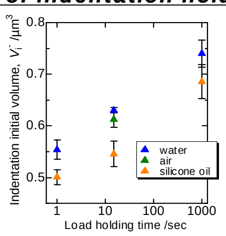
Effect of indentation load (Trihedral pyramids):



Densification contribution vs. Indentation load

1. A Sharper indenter results in plastic flow, while a blunter indenter causes densification.
2. Higher Sneddon's pressure is the origin of smaller densification contribution beneath a Cube-corner indenter.
3. Similar to phase transformation of crystal, glass changes its structure depending on pressure.

Effect of indentation holding time (Berkovich indenter):



Indentation volume vs. Load holding time

1. Indentation volume increases with increasing holding time in any environment. (Indentation Creep!)
2. Densification contribution is almost constant (63 % for 1 sec in water, 67 % for 1000 sec in water).

Conclusion

- Inelastic deformation of glass was investigated using some types of diamond indenters.
- Densified volume can be estimated from AFM measurements before and after annealing.
- Densification is a major mechanism of indentation deformation in oxide glass, and depends on glass composition, indenter geometry, and indentation conditions.