Effects of the indenter shape on the indentation-induced densification of soda-lime glass

Satoshi YOSHIDA ¹, ²
Hiroshi SAWASATO ¹, Toru SUGAWARA ¹, ², Yoshinari MIURA ², Jun MATSUOKA ¹, ²

¹ Department of Materials Science,
² Center for Glass Science and Technology,
The University of Shiga Prefecture, Hikone, Shiga, Japan
1. Background
   Indentation-induced densification of glass
   … Compositional variation
   … Relation between cracking and indentation-induced densification

2. Experimental procedure

3. Results and Discussion

4. Summary
   A blunter indenter results in larger contribution of densification.
1. Background

*Indentation-induced densification of glass*

2. Experimental procedure

3. Results and Discussion

4. Summary
1. **Background**

**Indentation impression on glass**

**Plastic flow** and/or **Densification**?


---

Pyramidal indentation on soda-lime glass

(Opposite face angle = 70°)

Cf. Vickers 136°

Ball indentation on soda-lime glass

(Radius = 20 µm, Load = 100 gf)

---

**Sharp indenter**

Piling-up! (Shear flow)

**Blunt indenter**

Densification!
1. Background

**Indentation impression on glass**

**Plastic flow and/or Densification?**


---

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

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

---

**Blunt indenter**

**Smaller load**

**Densification!**

**Larger load**

**Shear lines!**
1. Background

**Indentation impression on glass**

Deformation properties by indentation

<table>
<thead>
<tr>
<th></th>
<th>Plate glass</th>
<th>SiO$_2$ 74% Na$_2$O 14% CaO 10%</th>
<th>SiO$_2$ 85% Na$_2$O 15%</th>
<th>SiO$_2$ 100%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Densification</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>Piling-up</td>
<td>+</td>
<td>+</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Slip(shear) lines below</td>
<td>+</td>
<td>+</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>the indentation</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### 1. Background

**Indentation impression on glass**

#### Deformation properties by indentation

<table>
<thead>
<tr>
<th></th>
<th>Plate glass</th>
<th>SiO$_2$ 74% Na$_2$O 14% CaO 10%</th>
<th>SiO$_2$ 85% Na$_2$O 15%</th>
<th>SiO$_2$ 100%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Densification</td>
<td></td>
<td>All Glasses can be compacted!</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Piling-up</td>
<td>+</td>
<td>+</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Slip(shear) lines below the indentation</td>
<td>+</td>
<td>+</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

*Flow properties are correlated to a minimum percentage of network modifiers.*

1. **Background**  
**Indentation-induced structural change**

A decrease in Si-O-Si bond angle under the indentation

Raman spectra of *silica glass*
1. **Background**

*Indentation-induced structural change*

A decrease in the Si-O-Si bond angle under the indentation. This is also the case for soda-lime float glass.

Raman spectra of *soda-lime float glass*
1. **Background**

**Indentation-induced structural change**

A structural change can be also detected by infrared spectroscopy.


The increase in density corresponds to the increase (or decrease) in fictive temperature.
1. Background

Indentation impression on glass

Is it possible to estimate a densification contribution to total indentation deformation?
1. **Background**

*Indentation impression on glass*

Densified region can be relaxed by annealing at around $T_g$


![Initial volume](image1)

![Annealing](image2)

$T_g \times 0.9$ (K), 2 h

![Densified region](image3)

**Shrinkage**

Densification contribution (%) = 

\[
\frac{\text{Densified volume}}{\text{Initial volume}}
\]
1. **Background**

**Indentation impression on glass**

Densified region is completely recovered by annealing.

Annealing $T_g \times 0.9, 2$ h
1. **Background**

**Indentation-induced densification**

Densification is a general property for oxide glasses.

1. Various kinds of glasses experience densification beneath an Vickers indenter.

2. Volume recovery (densified volume) decreases with increasing Poisson’s ratio.

3. Sodium borate series show different dependence on Poisson’s ratio.

---

1. Background

**Indentation-induced densification**

Densification affects radial cracking of some commercial glasses.

![Diagram showing the relationship between densification contribution and crack initiation load](image)

- **Kato (2007)**
- **Radial crack**
- **Aluminosilicate glass**
- **Recovery of indentation depth (%)**
- **Crack initiation load (gf)**
- **Less brittle, or Less fragile**

- **A**
- **B**
- **C**
- **D**
- **E**
- **F**
- **G**
- **H**

100µm scale bar
1. **Background**

*Indentation impression on glass*

1. Indentation-induced densification is a general property of glass.

2. Indentation-induced densification is related to the cracking behavior of glass.

3. Indentation-induced densification correlates with Poisson’s ratio of glass.

No report on the indenter shape dependence of indentation-induced densification of glass.
Objective of this work

The effects of the indenter geometry on indentation-induced densification of soda-lime glass are investigated.

This may give some information on mechanical durability of glass against cracking, rubbing, tipping, and so on.
1. Background

2. Experimental procedure

3. Results and Discussion

4. Summary
2. Experimental procedure

Sample:

Soda-lime glass (Matsunami S-0050, Japan)

Vickers indentation test:

Load = 50 ~ 300 mN, Duration = 15 s, in air

Diamond indenter:

- Tetrahedral pyramid: $\beta = 22^\circ$ (Vickers)
- Trihedral pyramid: $\beta = 25^\circ$ (Berkovich), $\beta = 55^\circ$ (Cube-corner)
- Cone: $\beta = 20^\circ$
2. Experimental procedure

Observation of indentation imprints:

Atomic Force Microscope (AFM, Veeco Nanoscope E)
Contact mode
Scan area = \( \sim 15 \ \mu m^2 \)

Annealing conditions:
Temp. = \( T_g \times 0.9 \) (K), Time = 2h., in air
2. Experimental procedure

AFM volume measurements before and after annealing

Diamond indenter

AFM observation

Initial volume

Pile-up volume

Depth

unloading

Annealing ($T_g \times 0.9$, 2h)

AFM observation

Densified volume

Corner-to-Corner distance

Densification contribution =

Densified volume

Initial volume
1. Background

2. Experimental procedure

3. Results and Discussion

4. Summary
3. Results and Discussion

Three types of trihedral pyramids

Trihedral pyramid

\[ \beta = 10^\circ \]
\[ \beta = 25^\circ \) (Berkovich) \]
\[ \beta = 55^\circ \) (Cube-corner) \]
**Trihedral pyramids**

Indentation load = 50 mN

(a) 10 degree
   \[ \alpha = 165^\circ \]
   \[ \beta = 10^\circ \]
(b) Berkovich
   \[ \alpha = 152^\circ \]
   \[ \beta = 24.7^\circ \]
(c) Cube-corner
   \[ \alpha = 90^\circ \]
   \[ \beta = 54.7^\circ \]
Annealing recovery is remarkable at the faces of 10-degree and Berkovich indentation.

Distinct pile-up is observed around Cube-corner indentation.

Indentation load = 50 mN

Trihedral pyramids

10-degree

Berkovich

Cube-corner

Annealing

Annealing

Annealing

Annealing

Annealing

Pile-up
Trihedral pyramids

Trihedral pyramid

(a) 10 degree
\[ \alpha = 165^\circ \]
\[ \beta = 10^\circ \]

(b) Berkovich
\[ \alpha = 152^\circ \]
\[ \beta = 24.7^\circ \]

(c) Cube-corner
\[ \alpha = 90^\circ \]
\[ \beta = 54.7^\circ \]

Indentation load
= 50 mN

10-degree

After annealing
Densified volume
Before annealing

Berkovich

After annealing
Densified volume
Before annealing

Cube-corner

After annealing
Densified volume
Before annealing
Trihedral pyramids

Load dependence of annealing recovery of volume

Densification contribution

Recovery ratio /

Indentation load / mN

10-degree > Berkovich > Cube-corner

No load dependence for 10-degree
**Trihedral pyramid indenters**

Change in indentation volume by annealing

<table>
<thead>
<tr>
<th></th>
<th>10-degree</th>
<th>Berkovich</th>
<th>Cube-corner</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial volume ($\mu m^3$)</td>
<td>0.20 ± 0.01</td>
<td>4.53 ± 0.10</td>
<td>16.05 ± 1.3</td>
</tr>
<tr>
<td>Densified volume ($\mu m^3$)</td>
<td>0.17 ± 0.01</td>
<td>2.41 ± 0.13</td>
<td>4.86 ± 1.4</td>
</tr>
<tr>
<td>Densification contribution (%)</td>
<td>85 ± 1.0</td>
<td>53 ± 2.7</td>
<td>30 ± 12</td>
</tr>
</tbody>
</table>

With an increase in face-angle (or sharpness), both initial and densified volumes increase.

Densification contribution

10-degree > Berkovich > Cube-corner
**Trihedral pyramid indenters**

Sneddon’s pressure distribution for an equivalent rigid cone:

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

Sneddon (1965)

\( E \): Young’s modulus, \( \nu \): Poisson’s ratio, \( a \): contact radius, \\( r \): radial coordinate in the surface, \( \Psi \): half included angle

High pressure results in large indentation.
**Trihedral pyramid indenters**

Sneddon’s pressure distribution for an equivalent rigid cone

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

Sneddon (1965)

\(E\): Young’s modulus, \(\nu\): Poisson’s ratio, \(a\): contact radius, \(r\): radial coordinate in the surface, \(\Psi\): half included angle

---

High pressure results in large indentation, and in large contribution of plastic flow.

2\textsuperscript{nd} yield point

1\textsuperscript{st} yield point
3. Results and Discussion

Three types of equivalent indenters with different number of edges

These indenters have the same projected area to depth ratio.

- Tetrahedral pyramid: \( \beta = 22^\circ \) (Vickers)
- Trihedral pyramid: \( \beta = 25^\circ \) (Berkovich)
- Cone: \( \beta = 20^\circ \)
Equivalent indenters

Berkovich

Vickers

Cone

Densified volume

Before annealing

After annealing
Equivalent indenters

Annealing

Densification contribution

Berkovich < Vickers < Cone

Densified volume

Before annealing

After annealing
## Equivalent indenters

Change in indentation volume by annealing

<table>
<thead>
<tr>
<th></th>
<th>Berkovich</th>
<th>Vickers</th>
<th>Cone</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Initial volume (µm³)</strong></td>
<td>9.81 ± 0.31</td>
<td>8.30 ± 0.11</td>
<td>3.77 ± 0.15</td>
</tr>
<tr>
<td><strong>Densified volume (µm³)</strong></td>
<td>5.29 ± 0.54</td>
<td>5.28 ± 0.15</td>
<td>3.05 ± 0.18</td>
</tr>
<tr>
<td><strong>Densification contribution (%)</strong></td>
<td>54 ± 2.8</td>
<td>64 ± 1.4</td>
<td>81 ± 1.6</td>
</tr>
</tbody>
</table>

Indentation load = 300 mN

The edges of indenter promotes not only plastic flow but also densification.

Stress singularity at edges results in, if anything, plastic flow.
Equivalent indenters

<table>
<thead>
<tr>
<th></th>
<th>Berkovich</th>
<th>Vickers</th>
<th>Cone</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial volume (µm³)</td>
<td>9.81±0.31</td>
<td>8.30±0.11</td>
<td>3.77±0.15</td>
</tr>
<tr>
<td>Densified volume (µm³)</td>
<td>5.29±0.54</td>
<td>5.28±0.15</td>
<td>3.05±0.18</td>
</tr>
<tr>
<td>Densification contribution (%)</td>
<td>54±2.8</td>
<td>64±1.4</td>
<td>81±1.6</td>
</tr>
</tbody>
</table>

The large face angle, β, and/or the small edge included angle, γ, are the origin of smaller contribution of densification for Berkovich indenter.
Summary

The effects of the indenter geometry on indentation-induced densification of soda-lime glass are investigated.

1. A trihedral pyramid indenter with a smaller face-angle shows a larger contribution of densification.

2. An indenter with edges promotes irreversible plastic flow as well as densification.

3. We do not care about shear yielding and tip roundness. These effects remain open for further investigation.