Fundamentals of Indentation Cracking in Glass: A Measure of Strength?

Satoshi YOSHIDA
Associate professor
Center for Glass Science and Technology,
The University of Shiga Prefecture, Hikone,
Shiga, Japan
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Outline

1. Background
   » Strong glasses around us
   » What factors determine glass strength? ⋅⋅⋅ Cracks

2. Indentation cracking
   » What factors affect indentation cracking?
     ⋅⋅⋅ Densification

3. Micro-photoelastic imaging technique
   » Elastic and residual stresses around a ball indentation
   » Compositional variation of the residual stress

4. Summary
Background

Strong glasses around us

Apple Store, New York City

Glass House, Milan, Italy

Glass Violin, Hario Glass, Japan
Background

Strong glasses around us

Corning Gorilla (ion-exchanged)

Schott AG Xensation (ion-exchanged)

Asahi, AGC Dragon trail (ion-exchanged)

NEG Thin-Film Glass $t = 0.05$ mm
Background

Fracture of glass is one of the crucial issues.

iPad

Aquarium glass tank (Tempered) in Toyohashi, Japan
A sea otter broke it using a shell.
We need a simple evaluation method of glass strength.

We must know

What determines the glass strength?
A larger crack results in a lower fracture stress.

\[ \sigma_f = Y \frac{K_{ic}}{\sqrt{c}} \]

- \( K_{ic} \): Fracture toughness
- \( Y \): depends on the crack and loading geometries.
- \( c \): Crack size

Background

$K_{IC}$ of glass shows a less compositional variation.

<table>
<thead>
<tr>
<th>Glass</th>
<th>Fracture toughness SEPB (MPam$^{1/2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LCD backlight tube</td>
<td>0.73</td>
</tr>
<tr>
<td>LCD substrate</td>
<td>0.79</td>
</tr>
<tr>
<td>Microscope slide</td>
<td>0.76</td>
</tr>
<tr>
<td>CRT tube</td>
<td>0.71</td>
</tr>
<tr>
<td>PDP substrate</td>
<td>0.73</td>
</tr>
<tr>
<td>X-ray shield (lead glass)</td>
<td>0.66</td>
</tr>
<tr>
<td>Mother glass of glass-ceramic(Li-Al-Si)</td>
<td>0.84</td>
</tr>
</tbody>
</table>

$\sigma_f = Y \frac{K_{IC}}{\sqrt{C}}$

$K_{IC}$: Fracture toughness

$Y$: depends on the crack and loading geometries.


Crack size ($\sqrt{C}$) is a critical factor of glass strength!
Indentation cracking

» One measure to evaluate Crack Resistance

» One of the simplest fracture tests
Indentation Cracking

Indentation is used to model Contact Damage, or Crack Nucleation.

Vickers indenter

Soda-lime glass

Increasing load

Radial Crack

0.1 N

5 N
Comp. dependence of indentation cracking


Figure 4. Crack resistances of various glasses.

- Mother glass of Vycor
- Mother glass of Li-Al-Si Glass ceramics
- Lead glass
  \( K_{ic} = 0.66 \text{ MPam}^{1/2} 
- E-glass
- Window glass
  \( K_{ic} = 0.84 \text{ MPam}^{1/2} 

Wide variety of cracking
Comp. dependence of indentation cracking

What factors determine the crack initiation load?
Relation between crack initiation load and Ring-on-Ring fracture stress

We are on the right track.

But, the compositional variation of ROR fracture stress is not so large.
No relation between crack initiation and other mechanical properties

- A: SiO$_2$-B$_2$O$_3$-K$_2$O
- B: SiO$_2$-B$_2$O$_3$-Na$_2$O
- C: SiO$_2$-Al$_2$O$_3$-B$_2$O$_3$
- D: SiO$_2$-CaO-Na$_2$O
- E: SiO$_2$-SrO-Na$_2$O
- F: SiO$_2$-SrO-K$_2$O
- G: SiO$_2$-B$_2$O$_3$-PbO
- H: SiO$_2$-Al$_2$O$_3$-Li$_2$O
- I: Li-Al-Si Glass-ceramics

\[ (80-x)\text{SiO}_2-x\text{B}_2\text{O}_3-20\text{Na}_2\text{O} \]
Even though the indentation load is identical, the driving force for crack initiation would be different among glass compositions.
Crack initiation load decreases with increasing the estimated residual stress.

Residual stress = Bulk modulus \times Volume strain

Y. Kato, JNCS (2010)
How can we estimate the residual stress?
**Indentation Fracture (Median/Radial Crack)**

Lawn, Evans, Marshall (1980)

Median/Radial cracks are generated by the residual force.

- **P**: Indentation load
- **P_r**: Residual force for crack initiation
- **a**: Contact size
- **b**: Radius of plastic zone
- **c**: Median crack length
- **d**: Depth of impression

P: Residual force for crack initiation
Indentation Fracture (Median/Radial Crack)

Residual stress = Bulk modulus $\times$ Volume strain

$$\sigma_R = k \frac{\Delta V}{V}$$

$\Delta V \propto a^3$, $V \propto b^3$

$\kappa$: Bulk modulus

Lawn, Evans, Marshall (1980)
Indentation on glass @RT results in both

1. Shear flow (Volume conservative)

and

2. Densification (Shrinkage)

Densification does not contribute to expansion of plastic zone.
**Indentation-induced flow and densification**

**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!
What is Densification?

Glass increases in its density (or index) under a high compressive stress.

Under hydrostatic stresses

Fig. 3. Relative increase (percent) of density of vitreous silica as a function of applied pressure.


Raman spectra of hydrostatically densified silica glass

Decrease in the bond angle (Si-O-Si bending)

Increase in the Si-O bond length because of Si-Si repulsion (Si-O stretching)


Indentation also induces densification

Raman spectra of silica glass
How do we estimate the densification contribution to total indentation deformation?
Determination of 'Densification'

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


Densification contribution (%) = \frac{\text{Densified volume}}{\text{Initial volume}}

Annealing

\[ T_g \times 0.9 \ (K) \]

Temp. is high enough for almost full recovery, and low enough for viscous flow.

Shrinkage

AFM image

Densified volume

Initial volume
Raman spectra of silica glass before and after annealing

The densified structure is relaxed by annealing at $T_g \times 0.9$. 

Annealing

$T_g \times 0.9, 2 \text{ h}$

Wavenumber (cm$^{-1}$)
Every glass is densified under Vickers indenter.

YBC6: Oxynitride glass

BMG: Bulk metallic glass

Densification contribution decreases with increasing Poisson’s ratio.

Higher % Densification, Better Crack Resistance!!

because densification reduces the residual stress.

Indentation-induced densification is affected by

2. Indenter geometry (not shown today), \( \text{J. Mater. Res., 25 (2010) 2203.} \)
4. Fictive temperature (not shown today). \( \text{I.C.G., Salvador (2010).} \)
5. Water in glass (not shown today).
The stress is a tensor quantity, not a simple scalar. We should know stress components.
A wide variety of crack morphology comes from different stress states.

Indentation imprints (1 kgf) on different glasses

60SiO$_2$-20Al$_2$O$_3$-20CaO (mol%)

80SiO$_2$-10Al$_2$O$_3$-10CaO

100SiO$_2$

Median / Radial

Edge

Ring / Cone

One solution to obtain stress components is Birefringence technique.

With Dr. C.R. Kurkjian (Univ. Southern Maine)  
Dr. A. Errapart (Tallinn Univ. Tech.)
Birefringence, or Photoelasticity

2-Dimensional

\[ \delta = (n_1 - n_2)t = C(\sigma_1 - \sigma_2)t \]

Stress Optical Coefficient: \( C \)

\( \delta \): Retardation \( \leftrightarrow \) \( \sigma_1 - \sigma_2 \)

Principal stresses: \( \sigma_1, \sigma_2 \)
(Membrane stresses)

Principal refractive indices: \( n_1, n_2 \)

The stress state is biaxial.
Determination of stress distribution

3-Dimensional

\[ \delta = C \int (\sigma_1 - \sigma_2) \, dt \]

Ball indenter

Cross-section:

Top view:

Schematic of transmitted light through a square fiber

Onion peeling method

Stresses are calculated in layer-by-layer manner.

Optical path in the 1\textsuperscript{st} ring

Optical path in the 2\textsuperscript{nd} ring


In-situ imaging system with an indenter

# Mechanical responses of glasses (Ball indentations)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ball indenter</th>
<th>Indentation load /N</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
<td>1.0</td>
</tr>
<tr>
<td>Soda-lime</td>
<td>0.05R</td>
<td>Elastic deformation</td>
</tr>
<tr>
<td>0.05R</td>
<td>5.0N</td>
<td>20 µm</td>
</tr>
<tr>
<td>0.1R</td>
<td>Elastic deformation</td>
<td>Cracking</td>
</tr>
<tr>
<td>3.3N</td>
<td>20 µm</td>
<td></td>
</tr>
<tr>
<td>Silica</td>
<td>0.05R</td>
<td>Elastic deformation</td>
</tr>
<tr>
<td>3.0N</td>
<td>20 µm</td>
<td></td>
</tr>
<tr>
<td>0.1R</td>
<td>Elastic deformation</td>
<td></td>
</tr>
</tbody>
</table>
BR images during indentation

Soda-lime glass
\[ R = 0.1 \text{ mm indenter} \]
Indentation load = 3.0 N

During loading
Only Elastic.

Retardance
0 \sim 250 \text{ nm}
Black to White

Slow axis orientation
0 \sim 180 \text{ }^\circ
Black to White
Elastic stresses (SLS)

Soda-lime, \( R = 0.1 \) mm, Load = 3.0 N

Stresses from BR images
\( z = 0.004 \) mm

- **Tensile**
- **Compressive**

\( \sigma_z \): Axial stress
\( \tau_{zr} \): Shear stress
\( \sigma_r \): Radial stress
\( \sigma_\theta \): Circumferential, or hoop, stress

Indenter

Retardance

\( R \) is 0.004 mm
Comparison with analytical solution

Soda-lime, $R = 0.1$ mm, Load = 3.0 N

BF exp. ($z = 0.004$ mm)
- Max. $\tau_{zr} = 1.5$ GPa
- Min. $\sigma_z = -4.9$ GPa

Hertzian solutions ($z = 0.008$ mm)
- Max. $\tau_{zr} = 1.3$ GPa
- Min. $\sigma_z = -5.0$ GPa

Obtained stresses are in agreement with Hertzian solutions.
Evaluation to Residual indents

- Silica (Anomalous)
- $25\text{Na}_2\text{O}-75\text{SiO}_2$ (mol\%) (Normal)
Residual stresses

Retardation maps with coordinates for stress calculation

Ball (R=0.05mm)
Max. load = 3.0 N

Quite different!
**Residual stresses**

**Stress mapping (Radial stress)**

Ball (R=0.05mm)  
Max. load = 3.0 N

- **Silica 25Na<sub>2</sub>O-75SiO<sub>2</sub>**
- **Tensile Compressive Tensile Compressive**

**Residual stresses**

**Stress mapping**

- **Radial, \( \sigma_r \)**
- **Tensile**
- **Compressive**
- **Silica**

- **Radial, \( \sigma_r \)**
- **Compressive**
- **Stress (MPa)**
- **Silica**
- **25Na<sub>2</sub>O-75SiO<sub>2</sub>**

**Plastic zone**
Residual stresses and crack morphology

Stress (MPa)

Radial, $\sigma_r$

Distance from the loading axis, $r$ (mm)

Depth, $z$ (mm)

Silica

25Na$_2$O-75SiO$_2$

Tensile

Compressive

Ring/Cone crack

Median/Radial crack

Restoring force?

Densification (Shrinkage)

Flow (Expansion)

7.5N 20μm

4.0N 20μm

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The University of Shiga Prefecture
BR (Birefringence) stresses after unload tell us where a crack will initiate.
Summary

1. Residual stress after indentation is critical for understanding the compositional variation of glass strength.

2. Densification of glass affects the residual stress.

3. Microscopic BR (birefringence) technique is useful in order to evaluate stress components around the indent.

4. Our BR work has just started, but important. We have various unsolved questions.