Engineering Conferences International ECI Digital Archives

Functional Glasses: Properties And Applications for Energy and Information

Proceedings

Winter 1-8-2013

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

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

Follow this and additional works at: http://dc.engconfintl.org/functional_glasses Part of the <u>Materials Science and Engineering Commons</u>

Recommended Citation

Satoshi Yoshida, "Fundamentals of Indentation Cracking in Glass: A Measure of Strength?" in "Functional Glasses: Properties And Applications for Energy and Information", H. Jain, Lehigh Univ.; C. Pantano, The Pennsylvania State Univ.; S. Ito, Tokyo Institute of Technology; K. Bange, Schott Glass (ret.); D. Morse, Corning Eds, ECI Symposium Series, (2013). http://dc.engconfintl.org/functional_glasses/8

This Conference Proceeding is brought to you for free and open access by the Proceedings at ECI Digital Archives. It has been accepted for inclusion in Functional Glasses: Properties And Applications for Energy and Information by an authorized administrator of ECI Digital Archives. For more information, please contact franco@bepress.com.

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





Acknowledgment

» Nippon Electric Glass Co., Ltd., Japan Continuous support for our works on mechanical properties of glass

» Dr. C.R. (Chuck) Kurkjian (Univ. Southern Maine)

» Dr. A. Errapart (Tallin Univ. of Tech., Estonia)

» Colleagues in Shiga, Japan
 Prof. J. Matsuoka, Prof. T. Sugawara, Prof. Y. Miura, Prof. N. Soga
 S. Iwata, H. Sawasato, and BS and MS students





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

Technology

4. Summary

Background

Strong glasses around us



Apple Store, New York City Glass House, Milan, Italy



Glass Violin, Hario Glass, Japan









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?





Background

<u>A larger crack results in a lower fracture stress.</u>



R.E. Mould, "The Strength of Inorganic Glasses," pp. 119 to 149 in Fundamental Phenomena in the Materials Sciences, V. 4 (1967)

Background

<u> K_{Ic} of glass shows a less compositional variation.</u>

Glass	Fracture toughness SEPB (MPam ^{1/2})	$oldsymbol{V}$
LCD backlight tube	0.73	$\sigma_{f} = Y \frac{\kappa_{\text{Ic}}}{\sqrt{c}}$ $\kappa_{\text{Ic}}:$ Fracture toughness
LCD substrate	0.79	
Microscope slide	0.76	
CRT tube	0.71	
PDP substrate	0.73	
X-ray shield (lead glass)	0.66	Y: depends on the crack and loading geometries.
Mother glass of glass-ceramic(Li-Al-Si)	0.84	

Y. Kato et al., J. Non-Cryst. Solids 356(2010)1768.

<u>Crack size (\sqrt{c}) is a critical factor of glass strength !</u>





Indentation cracking

» One measure to evaluate Crack Resistance

» One of the simplest fracture tests







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







Comp. dependence of indentation cracking



Figure 4. Crack resistances of various glasses.





Comp. dependence of indentation cracking



J. Sehgal & S. Ito, J. Am. Ceram. Soc. 81(1998)2485.





What factors determine the crack initiation load?



SHIGA PREFECTURE

<u>Relation between crack initiation load and</u> <u>Ring-on-Ring fracture stress</u>



But, the compositional variation of ROR fracture stress is not so large.

No relation between crack initiation and other mechanical properties



Even though the indentation load is identical, the driving force for crack initiation would be different <u>among glass compositions. (E/H are almost identical.)</u> (The driving force affects Brittleness (H/K), Indentation toughness, or Crack initiation load.)





<u>Crack initiation load decreases with</u> increasing the estimated residual stress.

S. Yoshida, *XIXth I.C.G. (2007)* 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.





Indentation Fracture (Median/Radial Crack)



Center for Glass Science and Technology The University of Diga Predi-



Indentation on glass @RT results in both

1. Shear flow (Volume conservative)

and

Expansion of plastic zone

2. Densification (Shrinkage)

Densification does not contribute to expansion of plastic zone.





Indentation-induced flow and densification

Plastic flow and/or Densification K.W. Peter, J. Non-Cryst. Solids 5(1970) 103.



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

Sharp indenter Piling-up ! (Shear flow)



Ball indentation on soda-lime glass (Radius = $20 \ \mu$ m, Load = $100 \ g$ f)

Blunt indenter Densification !





What is Densification?

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



P.W. Bridgman and I. Simon, *J. Appl. Phys.*, **24**(1953)405.

Fig. 1. Index of refraction of the quenched phase as a function of the pressure of the run. Each point represents a spread of ± 0.005 index of refraction units.

H.M. Cohen and R. Roy, J. Am. Ceram. Soc., 44(1961)523.



Raman spectra of hydrostatically densified silica glass





SHIGA PREFEC

Indentation also induces densification



Center for

Glass

Science and Technology THE UNIVERSITY OF

SHIGA PREFEC

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 Mackenzie(1963), Neely & Mackenzie(1968), Yoshida (2001, 2005, 2007, 2010)



Raman spectra of silica glass before and after annealing

The densified structure is relaxed by annealing at Tg x 0.9.



Comp. dependence of densification contribution



<u>Every glass is densified under</u> <u>Vickers indenter.</u>

YBC6: Oxynitride glass BMG: Bulk metallic glass

Densification contribution decreases with increasing Poisson's ratio.

Yoshida, J.-C. Sangleboeuf, T. Rouxel (2005), J. Mater. Res. 20, p. 3404.





Higher %Densification, Better Crack Resistance!!

, because densification reduces the residual stress.



Y. Kato et al., J. Non-Cryst. Solids 356(2010)1768.





Indentation-induced densification is affected by

- 1. Glass composition, J. Mater. Res., 20 (2005) 3404.
- 2. Indenter geometry (not shown today),

J. Mater. Res., 25 (2010) 2203.

3. Indentation load (not shown today),

Int. J. Mater. Res., 98 (2007) 360.

- 4. Fictive temperature (not shown today).
 - *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.





<u>A wide variety of crack morphology comes from</u> <u>different stress states.</u>



Indentation imprints (1 kgf) on different glasses

T.M. Gross et al., J. Non-Cryst. Solids 355(2009)563.





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

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

The stress state is <u>biaxial</u>.

Stress Optical Coefficient: C

 $\delta : \text{Retardation} \implies \sigma_1 - \sigma_2$ $\overset{\checkmark}{}$ Principal stresses: σ_1 , σ_2

(Membrane stresses)

Principal refractive indices: n_1 , n_2





Determination of stress distribution

3-Dimensional

H. Aben, C. Guillemet, *Photoelasticity of Glass*, Springer (1993) J. Anton, A. Errapart, H. Aben, L. Ainola, *Exp. Mech.* **48**(2008)613.



In-situ imaging system with an indenter







SHIGA PREFECTURE

Mechanical responses of glasses (Ball indentations)



BR images during indentation

Soda-lime glassDuring loadingR = 0.1 mm indenterOnly Elastic.Indentation load = 3.0 N



Retardance 0 ~ 250 nm Black to White



Slow axis orientation 0 ~ 180 ° Black to White





Elastic stresses (SLS)

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





 $\tau_{\rm zr}$: Shear stress $\sigma_{\rm r}$: Radial stress σ_{θ} : Circumferential, or hoop, stress Retardance Indenter 0.004 mm 20um

JUICA FREFECTOR

Technology

Comparison with analytical solution τ_{x}

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



Obtained stresses are in agreement with Hertzian solutions.



 σ_{z}

 σ_r

Evaluation to Residual indents

» Silica (Anomalous)» 25Na₂O-75SiO₂ (mol%) (Normal)





Residual stresses

Retardation maps with Coodinates for stress calculation

Quite different !













Residual stresses and crack morphology



Science and Technoloav

BR(Birefringence) stresses after unload tell us where a crack will initiate.







- 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.







The University of Shiga Prefecture

Keynote Topic Discussion Points

Material questions:

-Why glass for this application? Good transparency and high scratch resistance

-What other materials could serve this purpose?

Polymers

-What trends will influence product development in the future? How will the products change? Watch the Corning's Video ! Mobile items and Tablets

-How will product trends impact the future use of glass and property/processability requirements?

Watch the Corning's Video more !

Key challenge/Key breakthrough questions:

-What are the key scientific challenges related to the topic of your presentation, and more generally the field of your expertise?

Toward Ultra-tough glasses !

-How critical are they to the advancement of the relevant technology?

They promote the market for glass products.

-Can you identify whether the challenge is with regard to limitations of specific property, performance or fabrication process? Establish target values, where possible.

Theoretical strength X 1/10 would be a goal.





Key challenge/Key breakthrough questions: (continued)

-What would be a key breakthrough and when might it occur?

Glasses would replace Polymers.

-How are these breakthroughs likely to occur? University discovery? Industry? University-Industry collaboration?

From Industry, because processing technology (especially surface. Polishing or coating) is a key issue. But, I hope that University will help the success.

Success questions:

-What will be a topic stating an exceptional success to be published in a well-known high-ranked research journal in 2025 concerning your R&D field?

(please give a title of a publication in 2025, you would like to read) 'New evaluation techniques of fracture strength and fracture toughness of $10-\mu m$ glass film'





Related Broad Discussion Points

Future R&D directions questions:

-What are the most critical scientific questions (be specific and list not more than 2 or 3 per topic);

What is the structural flaw?

How, Where, and When does a crack nucleate?

-With the given/limited R&D resources, on which R&D topics should we concentrate/reinforce? Which activity should be reduced?

Every topic is important, should not be reduced.

Continuous efforts (even under the limited condition) are important.

Do not make the missing years for each research.

-How much glass research is needed or optimum?

I have no idea. Depending on the scale of glass industry. (In Japan, \$17 billion/year) -What kind of education is needed to enable glass research? International workshop in order to exchange skills and ideas -What are strategies for better communication and collaboration in the glass community?

International meeting for a limited research area (like ICG's TC or FFAG)





Related Broad Discussion Points (continued)

Boundary conditions questions:

-Which changes in boundary conditions are foreseeable in your R&D field (legal, economical, ...)?

Collaboration between Surface and Mechanical Or between Structure(modeling) and Mechanical

-Do you see a possibility to influence/change/improve the boundary conditions (funding, networks,...)?

The project by Prof. Lothar Wondraczek @ Jena is one example. "Topological Engineering of Ultrastrong Glasses"

Glass community questions:

-which actions do you suggest to improve the situation in your field? Collaborations with other industries (energy? medical?).

-More lobbying activity in the political arena?

I have no idea. Sometime, it is important to get a big project.

-topical position papers?

Works of NGF(New Glass Forum in Japan) or GIC(Glass Indusctrial Conference in Japan) in my country ?





Unsolved Questions

- What is the origin of the residual RADIAL stress of silica glass ? The cavity model is not enough to explain it.
- 2. What is the crack nucleation mechanism ? From flaw-free surface or From indentation-induced shear defects ?
- 3. How can we calculate the stresses inside the plastic zone. (e.g. "black hole" in a retardation image)
- 4. How can we calculate the stress components around a non-axisymmetrical indent (e.g. Vickers) ?







The University of Shiga Prefecture (USP), Japan

1995 - Research Associate of USP

2004-2005 Guest Professor of Univ. Rennes, France (with Profs. T. Rouxel and J.-C. Sangleboeuf)

2007 - Associate Professor of USP

Member of TC06(mechanical properties) in I.C.G. Commitee member of FFAG (Flow and Fracture of Advanced Glasses)



Annealing weakens E-glass fiber, but

HF etching recovers its strength



(2), before etching; △, after etching.
(2), before etching; △, after etching.

S. Sakka, J. Ceram Soc. Jpn. 65(1957)190.





Indentation Fracture toughness, K_{c}



SHIGA PREFECTURE

Indentation fracture toughness

 $K_c \propto \frac{P_r}{c^{3/2}} \propto \sigma_R \frac{b^2}{c^{3/2}} \propto \frac{E}{H} \left(\frac{a}{b}\right) \frac{P}{c^{3/2}}$ $K_c = 0.016 \left(\frac{E}{H}\right)^{1/2} \frac{P}{c^{3/2}}$ $\frac{a}{b} \propto \left(\frac{E}{H}\right)^2$ Empirical relationship

Anstis *et al.*(1981)

Technoloav

Indentation toughness is not Real toughness



Center for

Glass

Science and Technology THE UNIVERSITY OF

SHIGA PREFECTURE

Anstis et al., J. Am. Ceram. Soc. 64(1981)531.

Relation between Crack initiation load and

<u>Ring-on-Ring fracture stress normalized by</u>

<u>Residual stress</u>







Determination of stress distribution

H. Aben, C. Guillemet, *Photoelasticity of Glass*, Springer (1993) J. Anton, A. Errapart, H. Aben, L. Ainola, *Exp. Mech.* **48**(2008)613.



Estimation of residual stresses (Yoffe's model)

E.H. Yoffe, *Philos. Mag. A* **46**(1982)617. R.F. Cook & G.M. Pharr, *J. Am. Ceram. Soc.* **73**(1990)787.

BR residual stresses

Yoffe's model (Silica glass) Stresses at z = 0



Yoffe's model reproduces (only) the Hoop residual stress, σ_{θ} . A modified model will be needed for σ_{r} .

In-situ observation of Vickers indentation

Soda-lime glass



In-situ observation of Vickers indentation





The University of Shiga Prefecture

Schematic model of stress state of SLS under Vickers

T. Wilantewicz, Ph.D thesis, Alfred Univ (2005)



(b) Unloading



indenter loading (a) and after complete unloading (b). Negative signs designate compression, and positive signs tension. F_r is the wedge-opening force due to the residual stress field, and wedges open the lateral and median-radial (M/R) cracks, particularly on unloading. F_e is the elastic force which also wedges open median-radial cracks on loading. After Lawn et al.²⁰ Lawn, Evans, Marshall(1980)

Ring/Cone crack after/during unloading













Sample 1: Soda-lime glass (Matsunami 0050)

Stress optical coefficient2.7 TPa-1Poisson's ratio0.20Young's modulus73 GPa

Sample 2: Silica glass

Stress optical coefficient3.5 TPa-1Poisson's ratio0.16Young's modulus72 GPa

Sample dimensions:

0.5 x 0.5 x 35 mm³ Square fibers

SHIGA PREFECT

Indenter: Diamond ball indenter (R=0.05, 0.1 mm) Why ball indentation ?

... The obtained stress field has an axial symmetry.



Soda-lime glass, 2.09 N, Indenter R=0.05 mm





AFM cross-section Max. depth \sim 0.2 µm Diameter \sim 22 µm





Comparison with analytical solution $\tau_{\mathbf{x}}$

<u>Soda-lime</u>, R = 0.1 mm, Load = 3.0 N <u>Deeper under the indenter</u>







 σ_{z}

 $\sigma_{\!_{ heta}}$

 σ_r
Elastic stresses (Silica)

Silica, R = 0.05 mm, Load = 1.5 N







 σ_{z}

 σ_r

 au_{zr}



Elastic stresses (Silica)





 σ_{z}

 au_{zr}



Soda-lime glass, 2.09N, Indenter R=0.05mm



Raman spectra before and after indentation













Silica glass, 7.0 N, Indenter R=0.05mm



Determination of stress distribution







Determination of stress distribution

Determination of radial stress distribution

Equilibrium eq.

Written in a discrete form

$$\frac{\partial \sigma_{r}}{\partial r} + \frac{\sigma_{r} - \sigma_{\theta}}{r} + \frac{\partial \tau_{rz}}{\partial z} = 0,$$

$$\sigma_{r,i} = \sigma_{r,i-1} - \left(\frac{\sigma_{r,i-1} - \sigma_{\theta,i-1}}{r_{i-1}} + \frac{\Delta \tau_{rz,i-1}}{\Delta z}\right) \Delta r.$$





Determination of stress distribution

Determination of circumferential stress distribution $\frac{\partial \varepsilon_{\theta}}{\partial r} - \frac{\varepsilon_r - \varepsilon_{\theta}}{r} = 0$ Compatibility equation: $\varepsilon_r = \frac{1}{E} [\sigma_r - \mu (\sigma_\theta + \sigma_z)],$ Hooke's law: $\varepsilon_{\theta} = \frac{1}{F} [\sigma_{\theta} - \mu (\sigma_r + \sigma_z)]$ $\frac{\partial \sigma_{\theta}}{\partial r} - \mu \frac{\partial}{\partial r} (\sigma_r + \sigma_z) - \frac{1}{r} (1 + \mu) (\sigma_r - \sigma_{\theta}) = 0$ $\frac{\Delta \sigma_{\theta}}{\Delta r} - \mu \frac{\Delta (\sigma_r + \sigma_z)}{\Delta r} - \frac{1}{r} (1 + \mu) (\sigma_r - \sigma_z)$ $\sigma_{\theta,i} = \sigma_{\theta,i-1} + \mu (\sigma_{r,i} - \sigma_{r,i-1} + \sigma_{z,i} - \sigma_{z,i-1}) + \frac{\Delta r}{m} (1 + \mu) (\sigma_{r,i-1} - \sigma_{\theta,i-1})$ Important constraint: in the centre of solid specimen $\sigma_r = \sigma_{\theta}$ σ_{θ} and σ_{r} $\tau_{\rm rz}$ and $\sigma_{\rm z}$ SHIGA PREF Science and

Comparison of BF with Hertzian stresses

Hertzian stresses - Photoelastic data Photoelastic stresses



<u>Relation between crack resistance and</u> <u>residual stress</u>



Y. Kato et al., J. Non-Cryst. Solids 356(2010)1768.



