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

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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

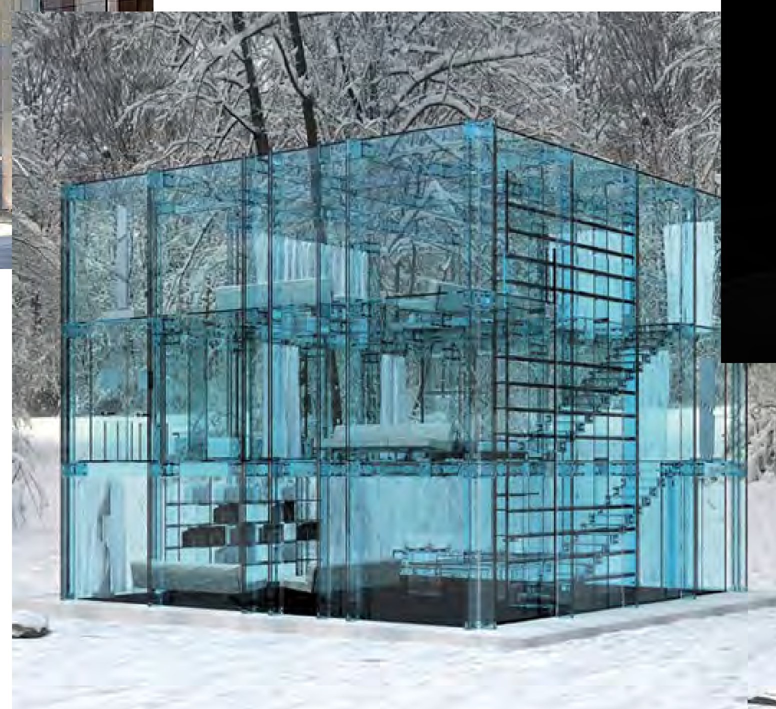
4. Summary

Background

Strong glasses around us

Apple Store,
New York City

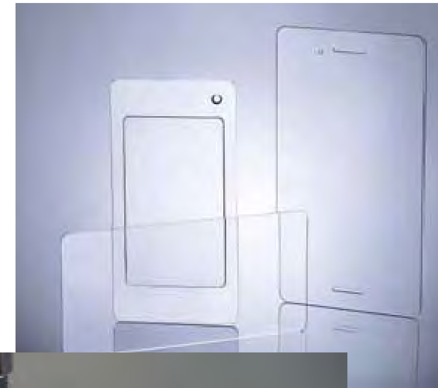
Glass House,
Milan, Italy



Glass Violin,
Hario Glass, Japan

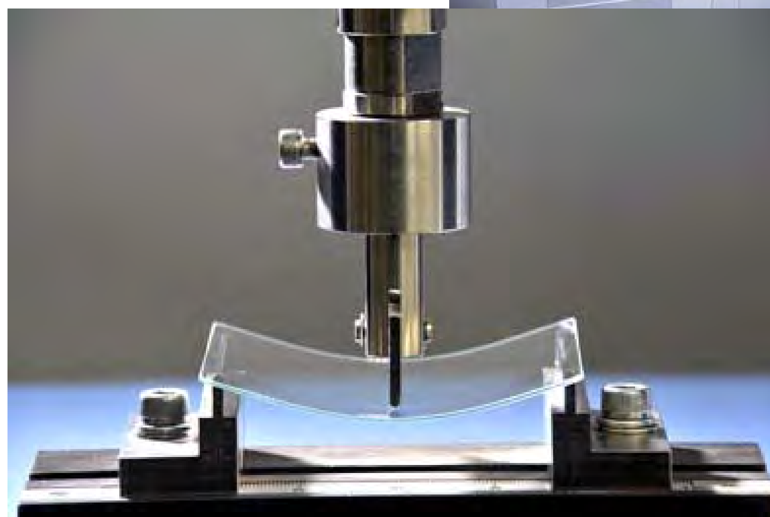
Background

Strong glasses around us



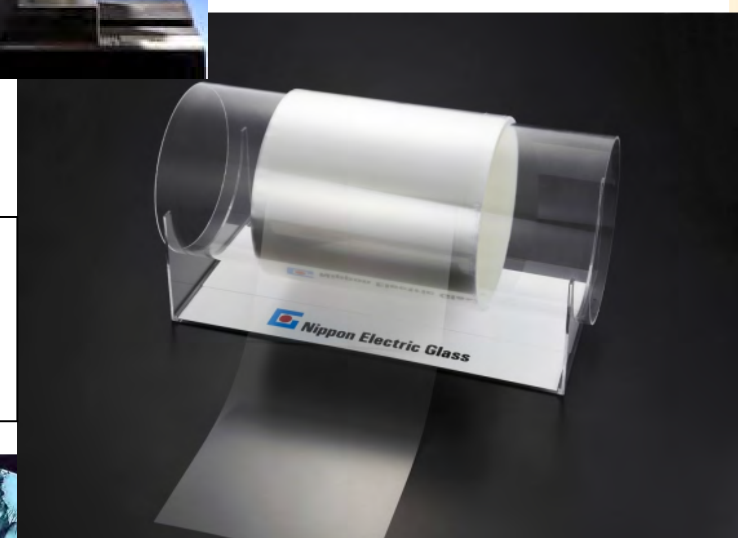
Schott AG
Xensation
(ion-exchanged)

Corning
Gorilla
(ion-exchanged)



Asahi, AGC
Dragon trail
(ion-exchanged)

NEG
Thin-Film Glass
t = 0.05 mm



Background

<u>Fracture of glass is one of the crucial issues.</u>

iPad

Aquarium glass tank (Tempered)
in Toyohashi, Japan
A sea otter broke it using a shell.

Background

We need a simple evaluation method of glass strength.

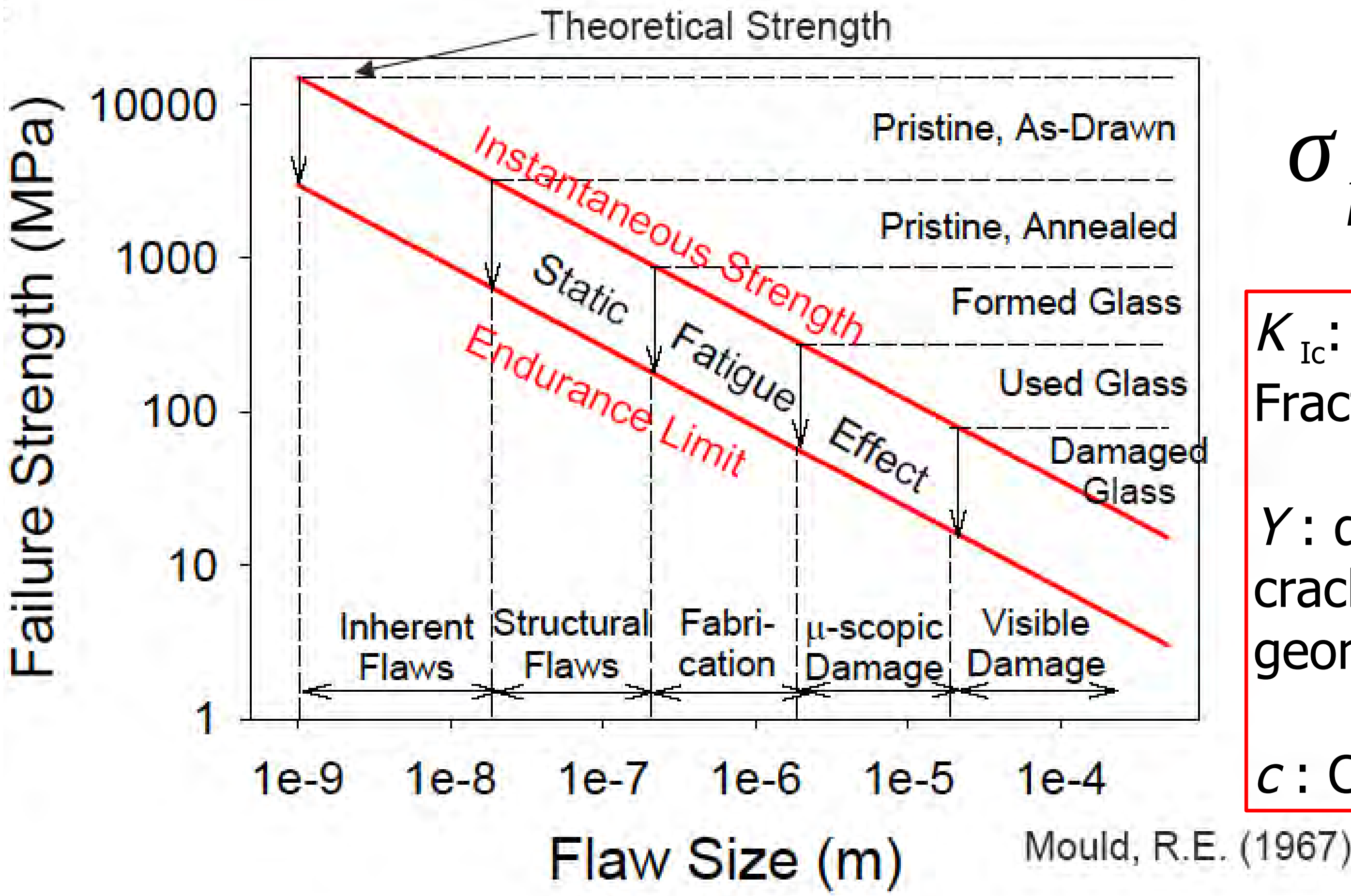
↓

We must know

What determines the glass strength?

Background

A larger crack results in a lower fracture stress.



$$\sigma_f = Y \frac{K_{\mathrm{Ic}}}{\sqrt{c}}$$

K_{Ic}:
Fracture toughness

Y : depends on the crack and loading geometries.

c : Crack size

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

Background

K_{Ic} of glass shows a less compositional variation.

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

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

$$\sigma_f = Y \frac{K_{Ic}}{\sqrt{c}}$$

K_{Ic}:
Fracture toughness

Y : depends on the crack and loading geometries.

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

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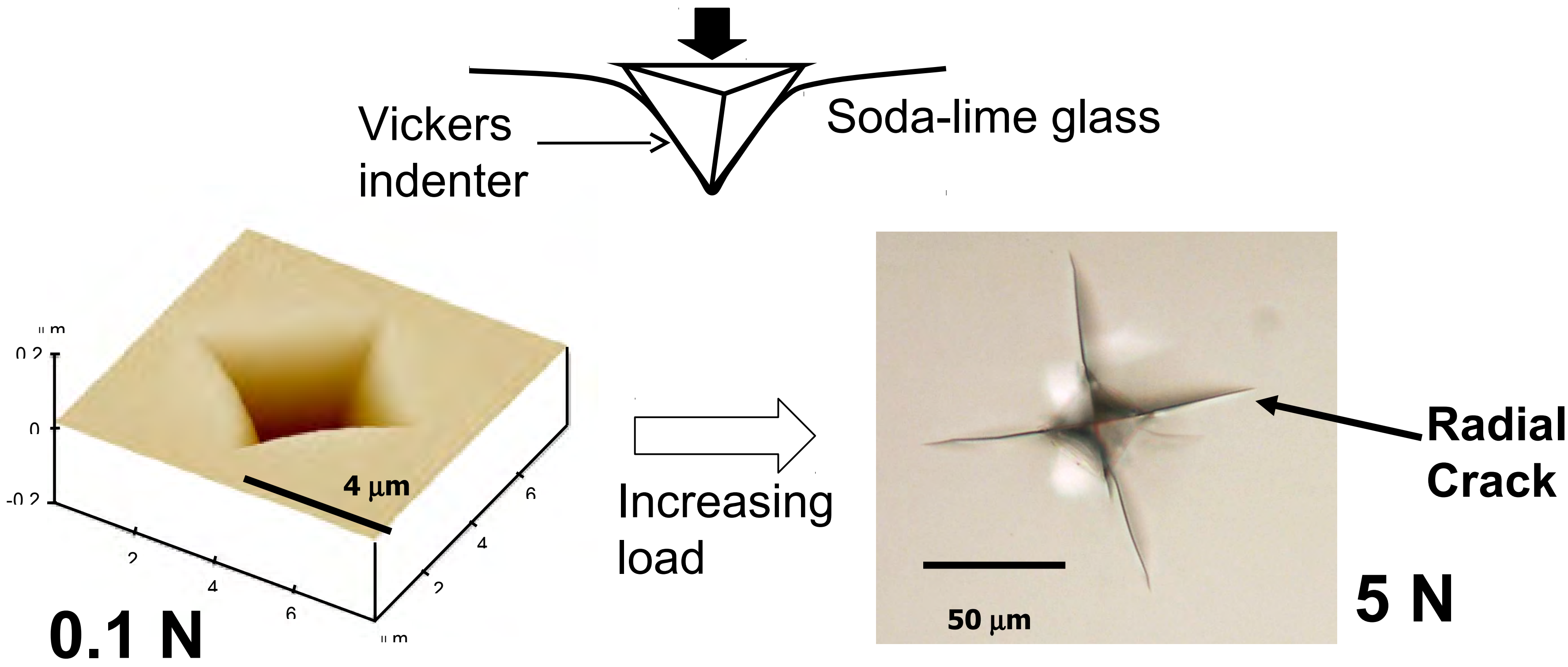
Indentation cracking

» One measure to evaluate Crack Resistance

» One of the simplest fracture tests

Indentation Cracking

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



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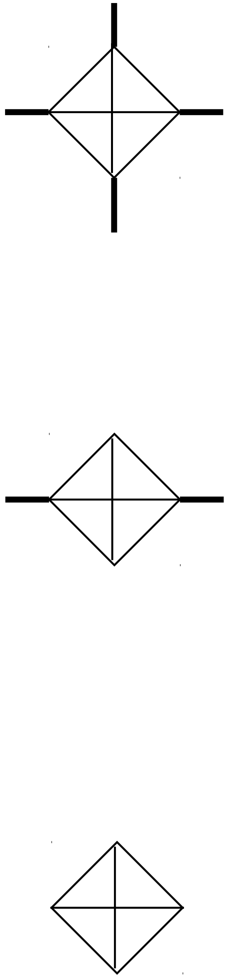
Comp. dependence of indentation cracking

M. Wada *et al.*, *Proc. Xth I.C.G.* **10**(1974)39.



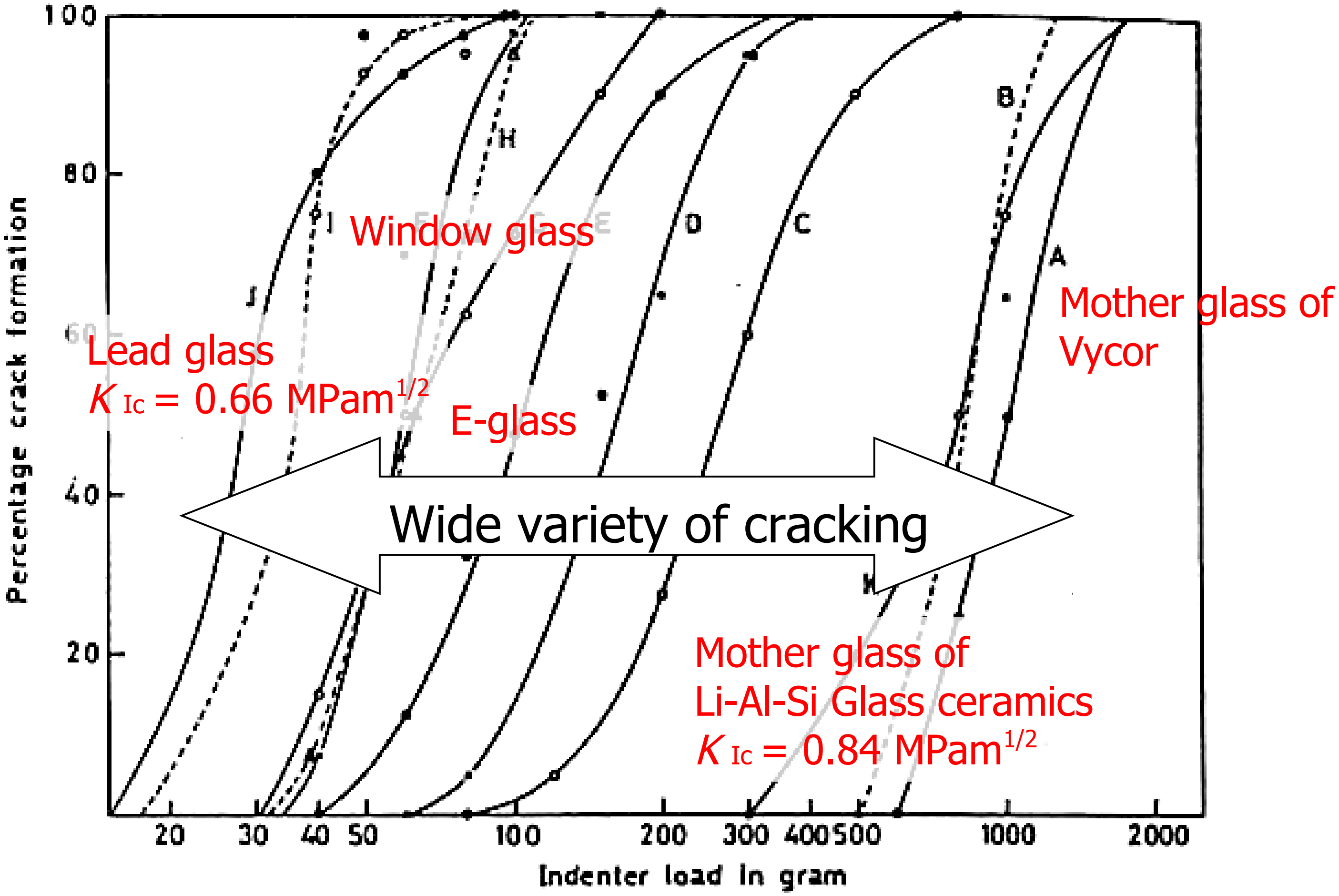
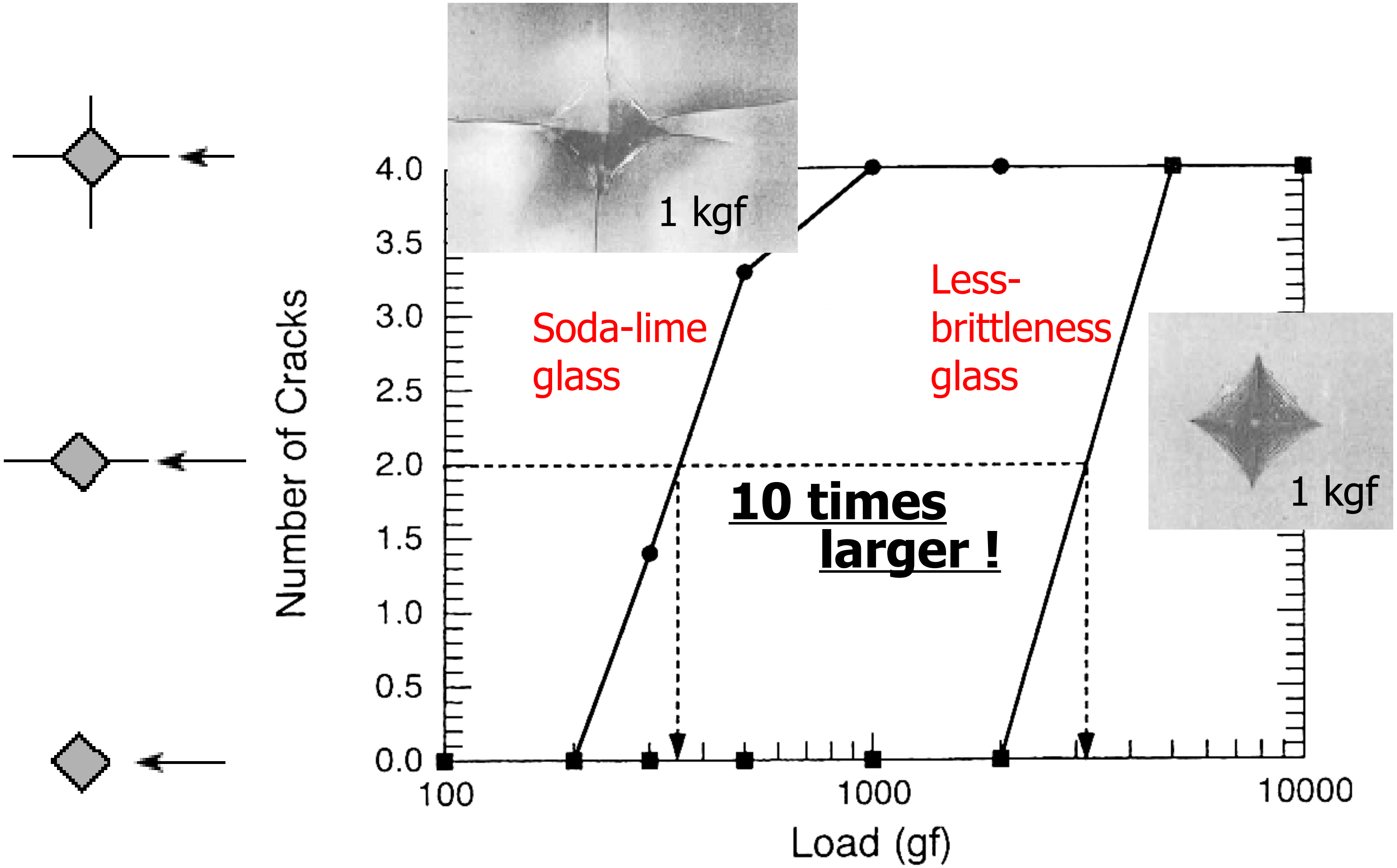


Figure 4. Crack resistances of various glasses.

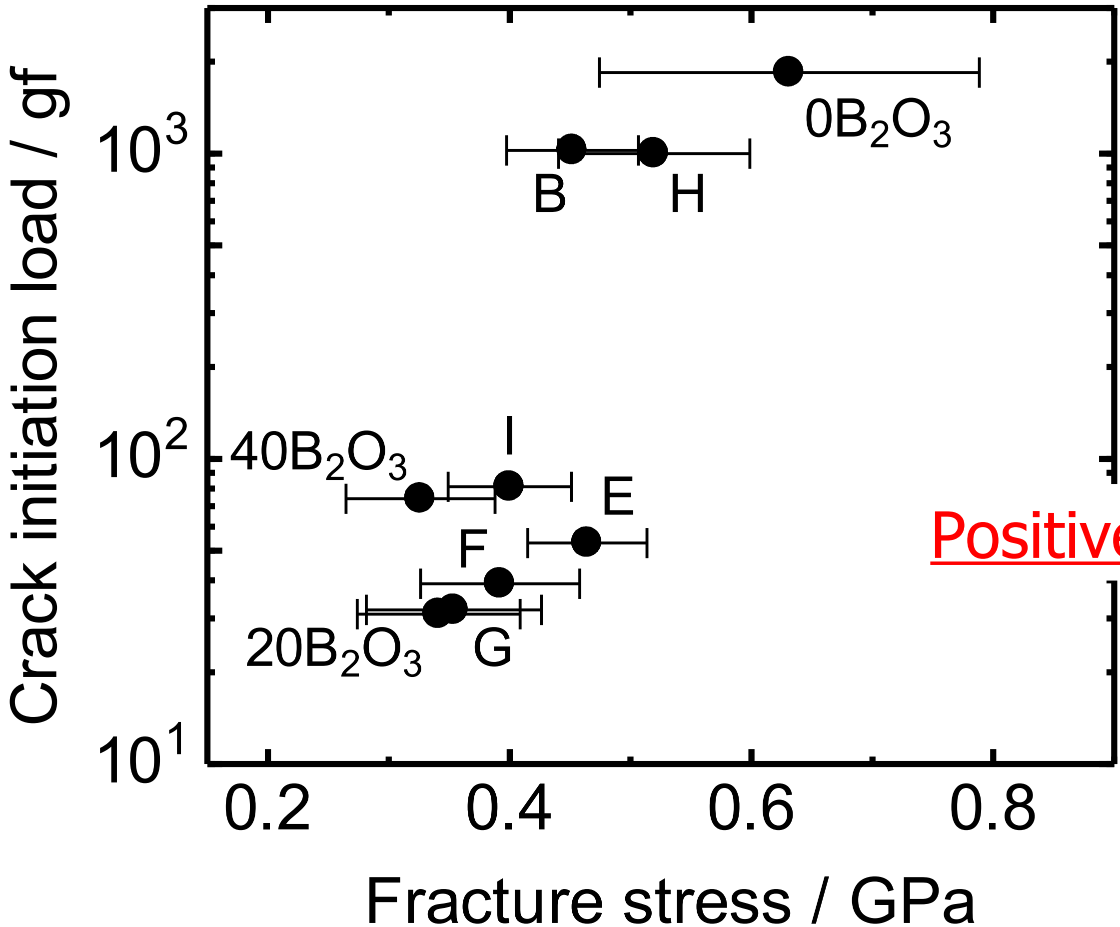
Comp. dependence of indentation cracking



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

What factors determine the crack initiation load?

Relation between crack initiation load and Ring-on-Ring fracture stress



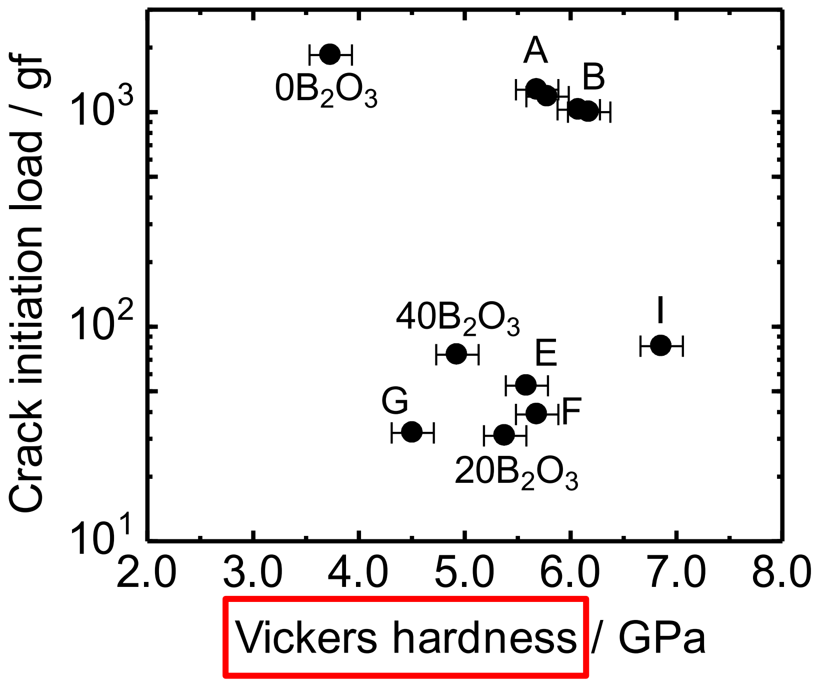
Positive relation ?

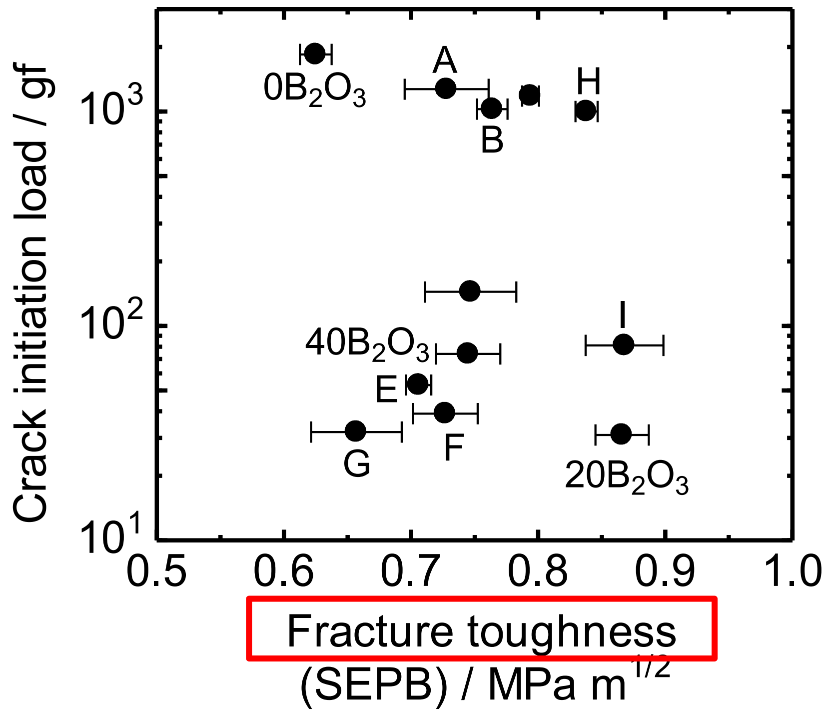
We are on the right track.

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

No relation between crack initiation and other mechanical properties

S. Yoshida, *XIXth I.C.G. (2007)*
Y. Kato, *JNCS (2010)*





A: SiO_2-B_2O_3-K_2O	B: SiO_2-B_2O_3-Na_2O	C: SiO_2-Al_2O_3-B_2O_3
D: SiO_2-CaO-Na_2O	E: SiO_2-SrO-Na_2O	F: SiO_2-SrO-K_2O
G: SiO_2-B_2O_3-PbO	H: SiO_2-Al_2O_3-Li_2O	I: Li-Al-Si Glass-ceramics

$0B_2O_3$, $20B_2O_3$, $40B_2O_3$: (80-x)SiO_2-x B_2O_3-20Na_2O

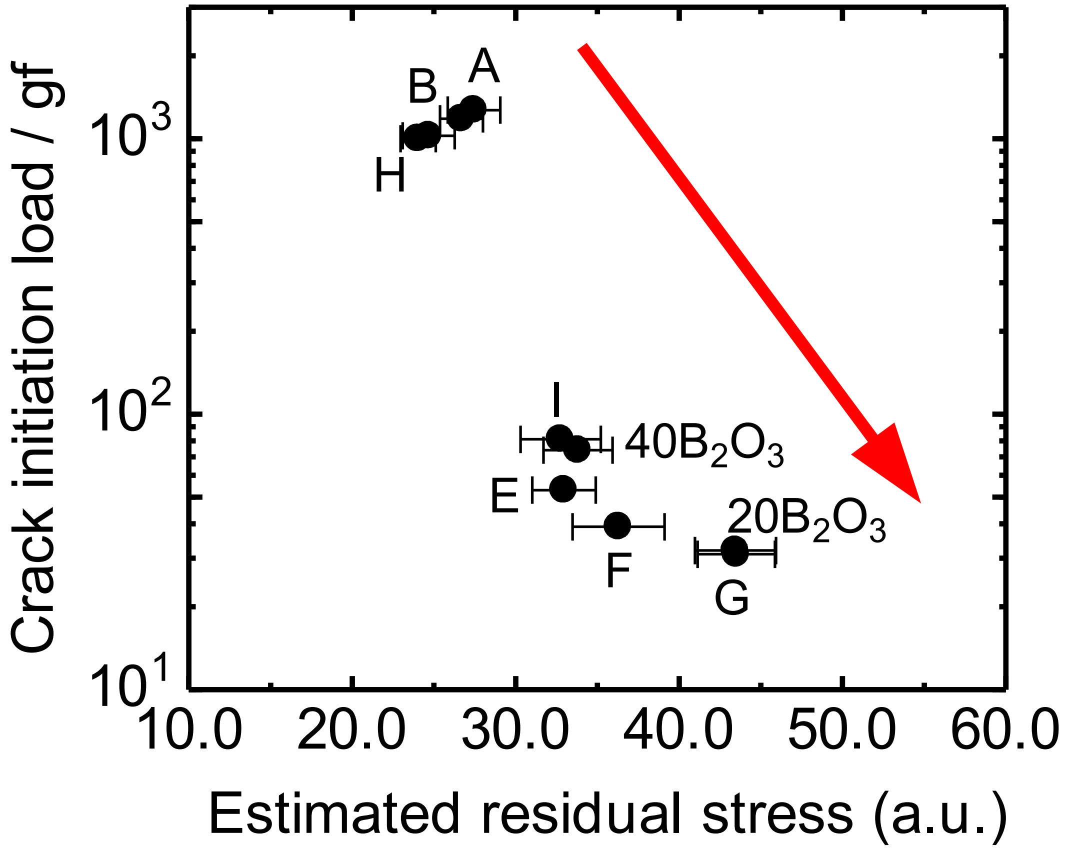
Even though the indentation load is identical,

the driving force for crack initiation would be different

among glass compositions.

Crack initiation load decreases with increasing the estimated residual stress.

S. Yoshida, *XIXth I.C.G. (2007)*
Y. Kato, *JNCS (2010)*

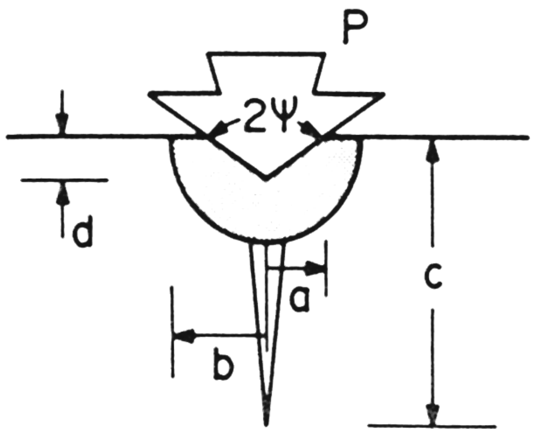


Residual stress = Bulk modulus x **Volume strain**

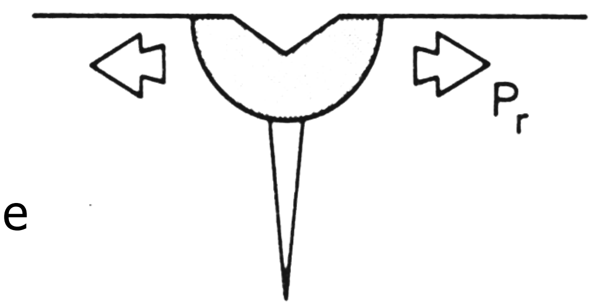
How can we estimate the residual stress?

Indentation Fracture (Median/Radial Crack)

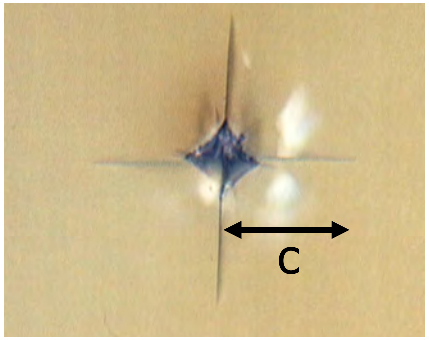
Lawn, Evans, Marshall(1980)



P_r: Residual force
for crack initiation



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



Median/Radial cracks are generated by the residual force.

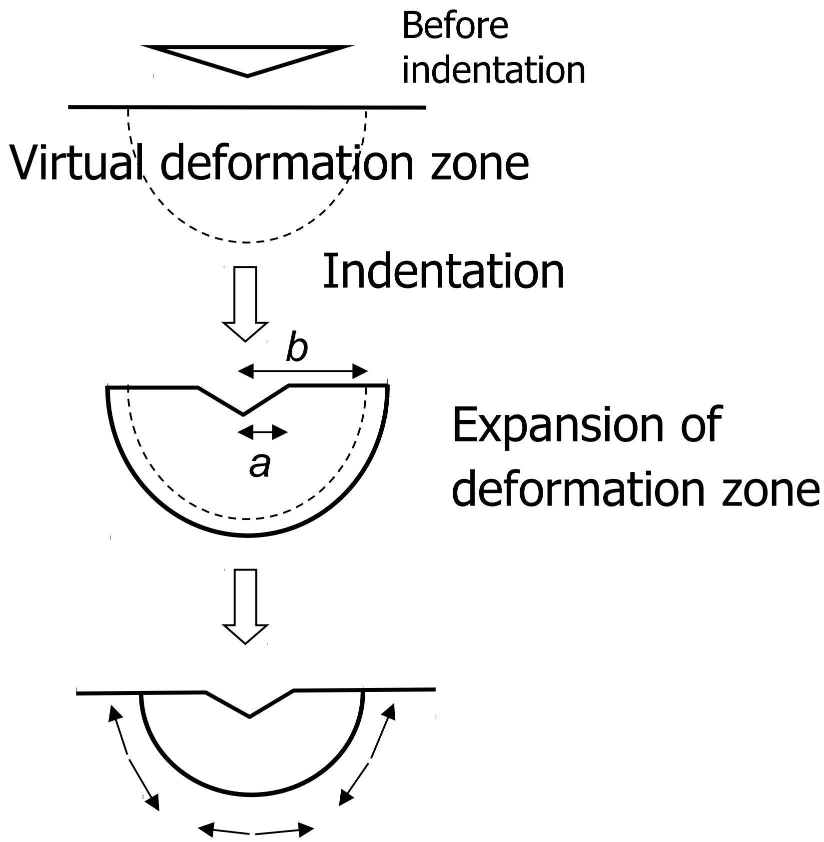
Indentation Fracture (Median/Radial Crack)

Residual stress = Bulk modulus x Volume strain

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

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

κ : Bulk modulus



Tensile stress
at the elastic/plastic interface

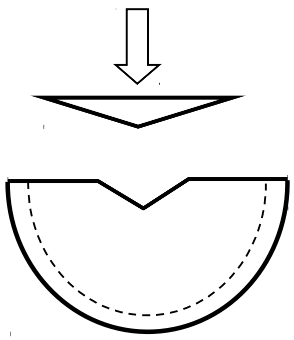
Lawn, Evans, Marshall(1980)

Indentation on glass @RT results in both

<u>1. Shear flow (Volume conservative)</u>

and

<u>2. Densification (Shrinkage)</u>



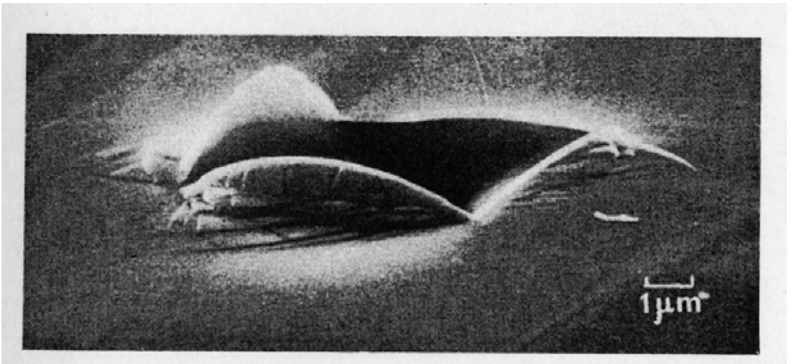
Expansion of plastic zone

Densification does not contribute to expansion of plastic zone.

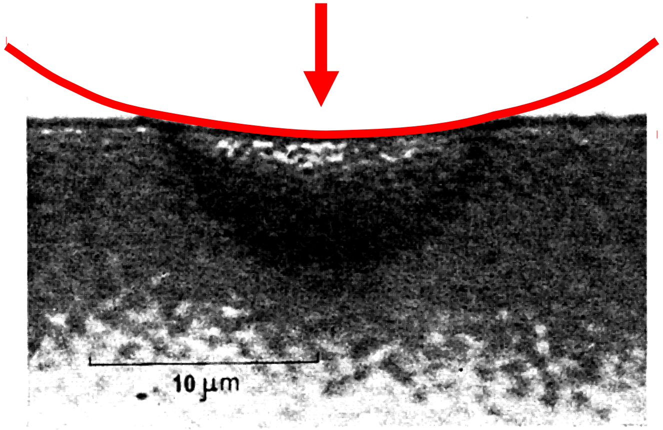
Indentation-induced flow and densification

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 °



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

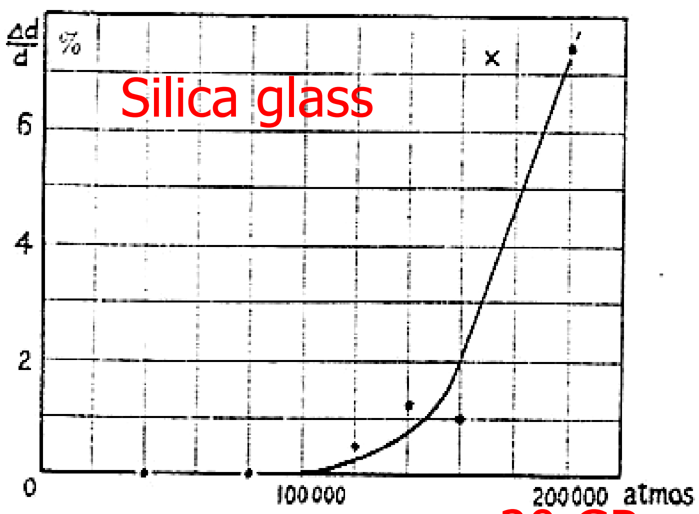


FIG. 3. Relative increase (percent) of density of vitreous silica as a function of applied pressure.

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

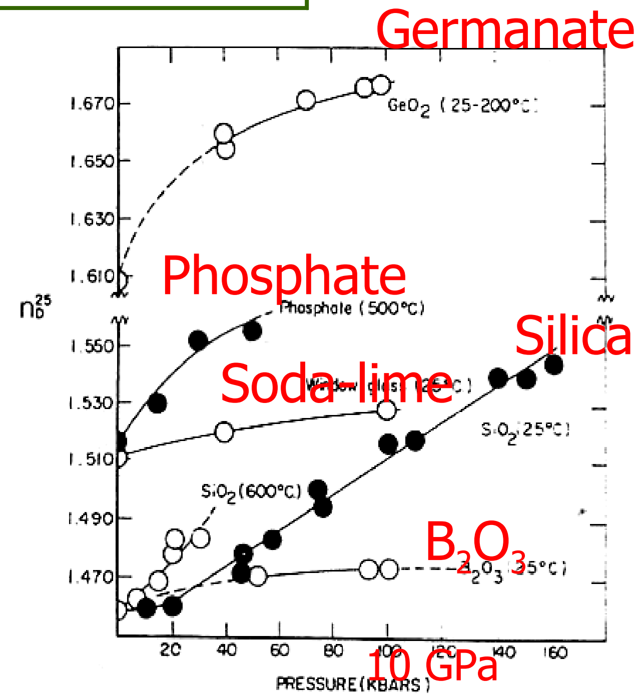


Fig. 1. Index of refraction of the quenched phase as a function of the pressure of the run. Each point represents a spread of ±0.005 index of refraction units.

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

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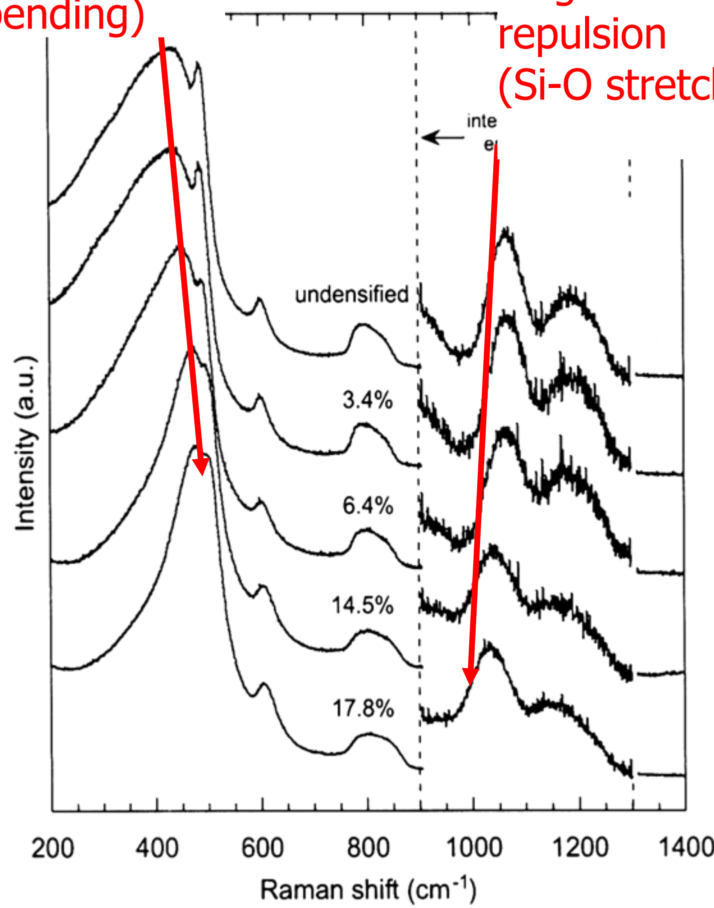
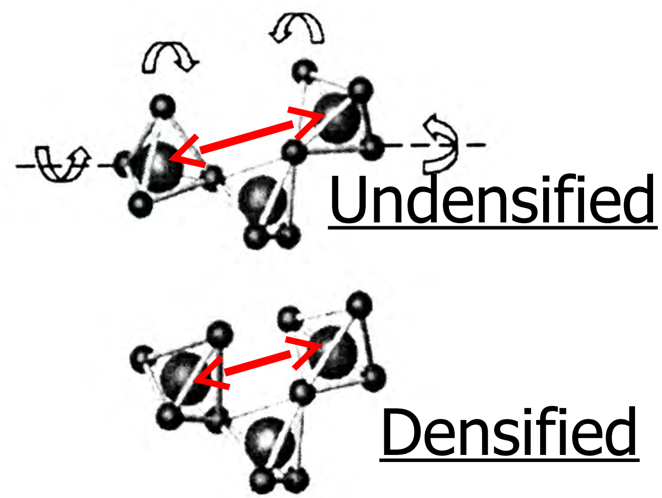
Raman spectra of hydrostatically densified silica glass

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

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

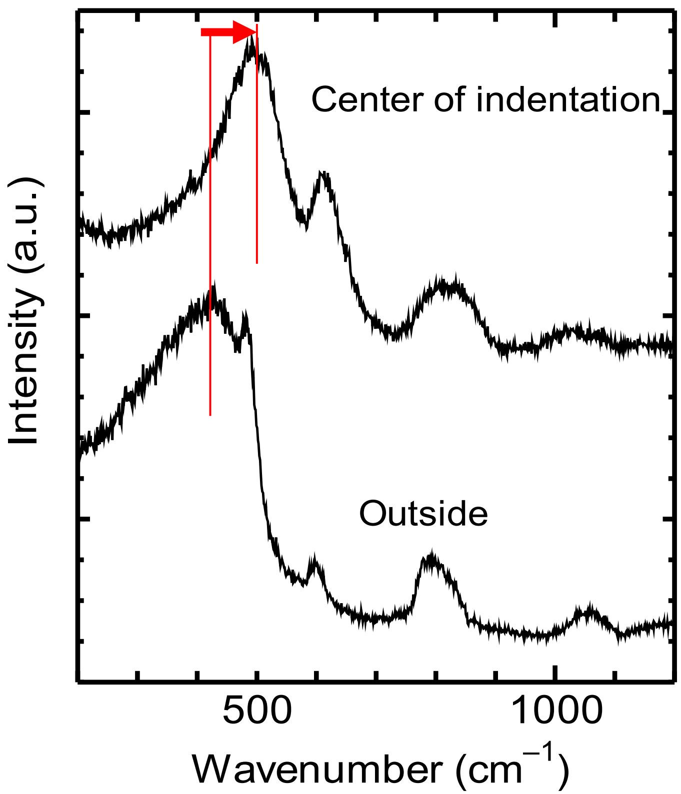


Poe *et al. J. Non-Cry.* (2004)

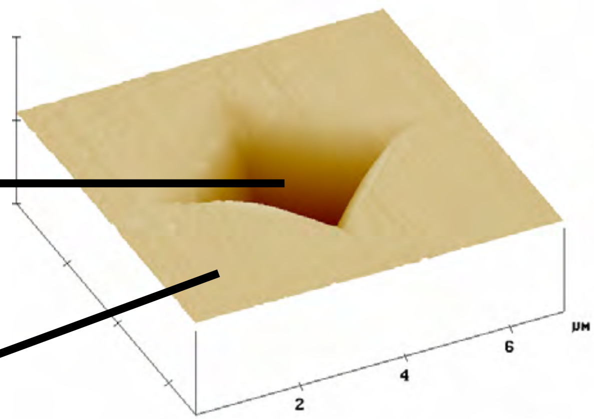


Sampath *et al.*, *Phys. Rev. Lett.* (2003)

Indentation also induces densification



Raman spectra of silica glass



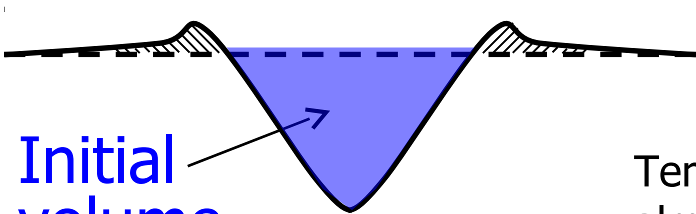
AFM image of Vickers indentation

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)

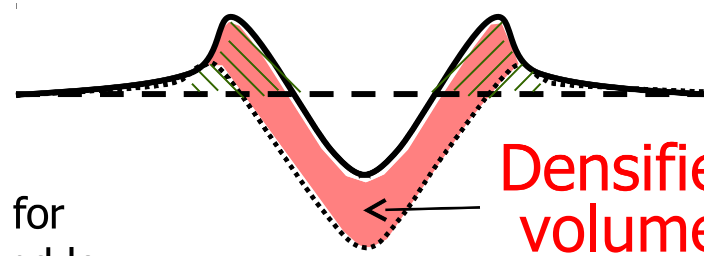


Initial volume

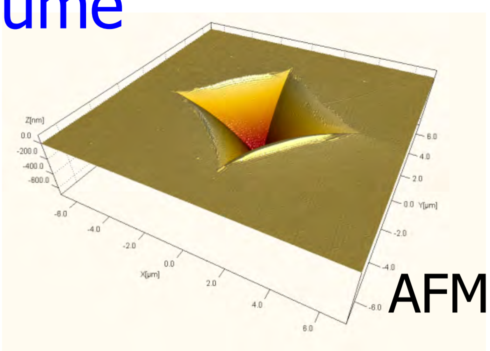
Annealing

$T_g \times 0.9$ (K)

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

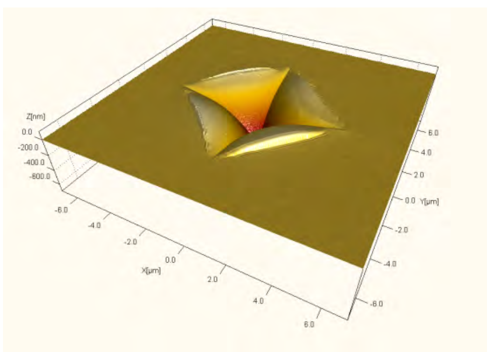


Densified volume



Shrinkage

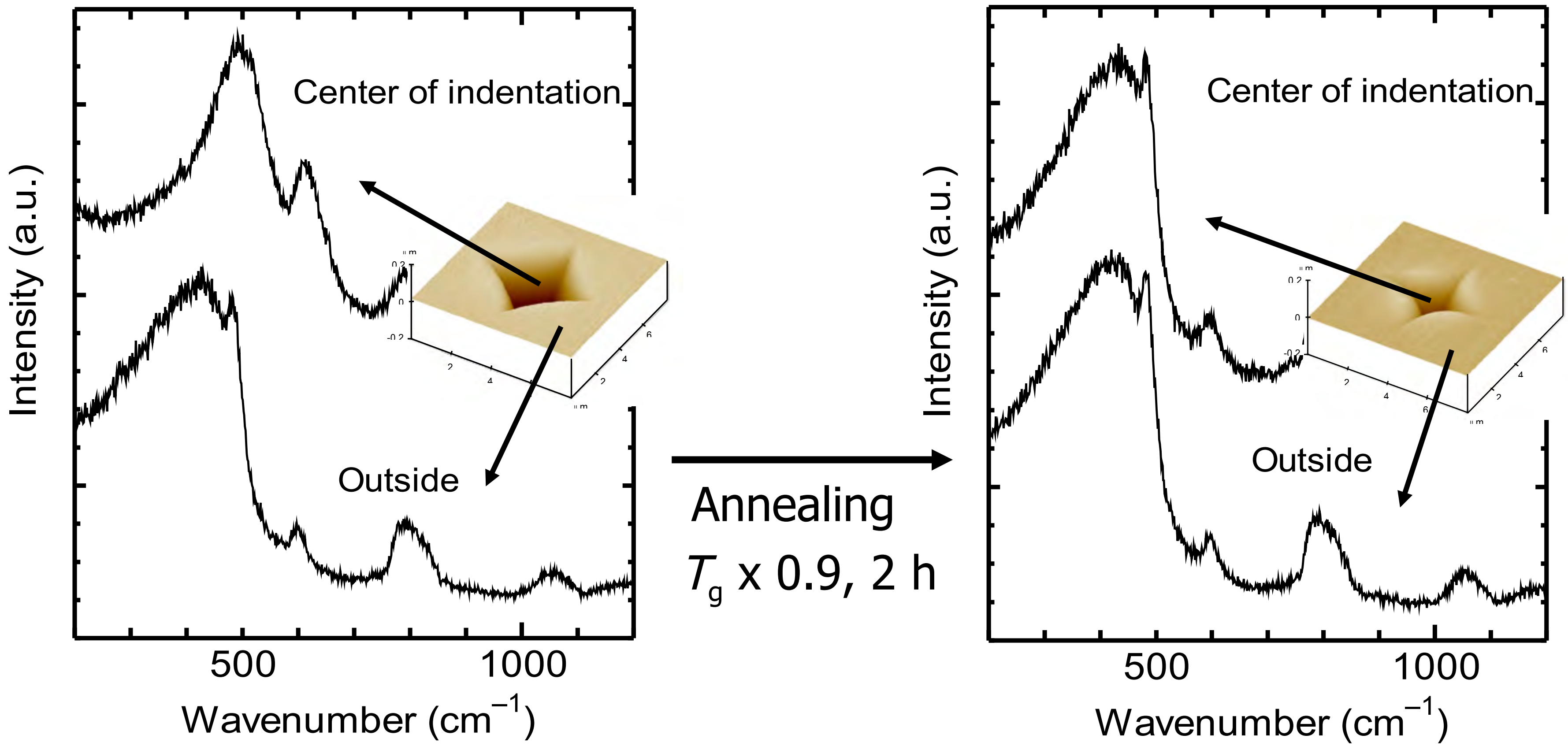
AFM image



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

Raman spectra of silica glass before and after annealing

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



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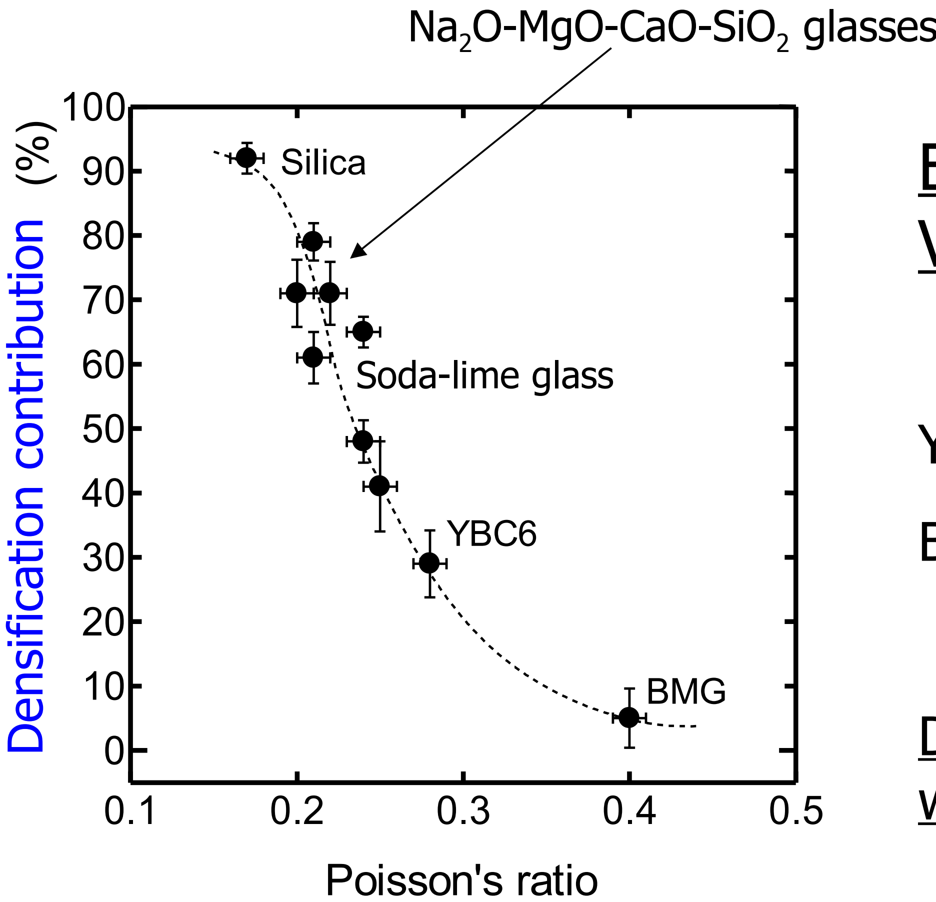
Comp. dependence of densification contribution



Every glass is densified under Vickers indenter.

YBC6: Oxynitride glass

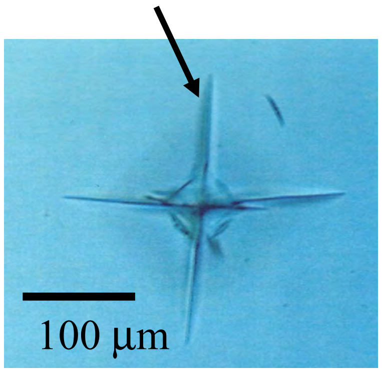
BMG: Bulk metallic glass

Densification contribution decreases with increasing Poisson's ratio.

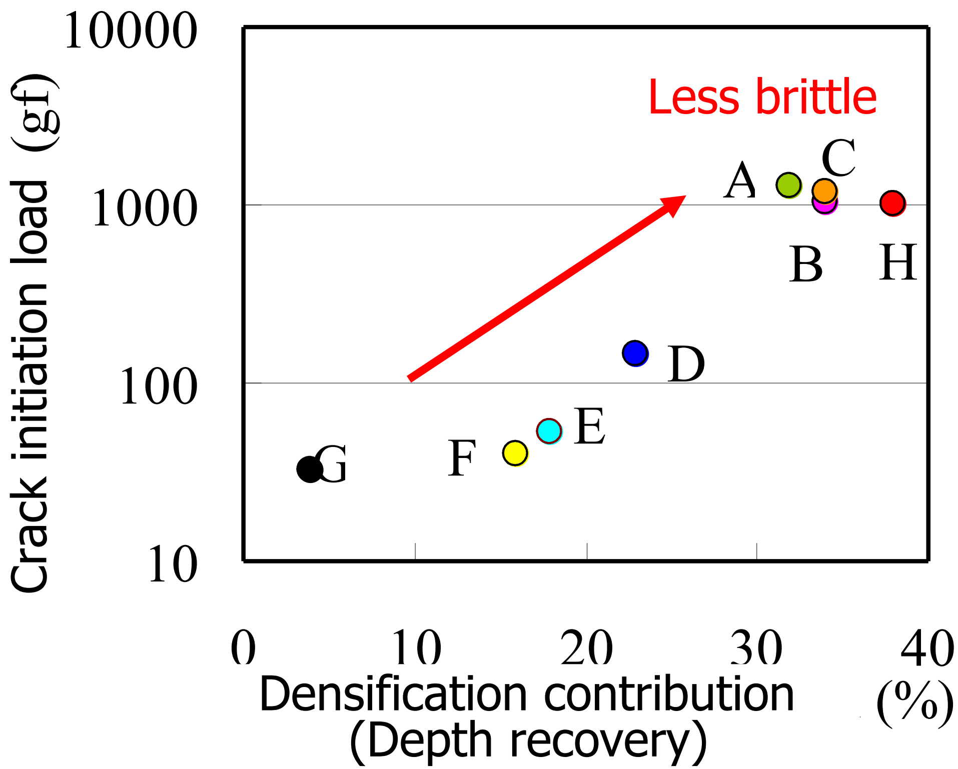
Yoshida, J.-C. Sangleboeuf, T. Rouxel (2005), *J. Mater. Res.* ***20****, p. 3404.*

Higher %Densification, Better Crack Resistance!!

, because densification reduces the residual stress.



Aluminosilicate glass



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

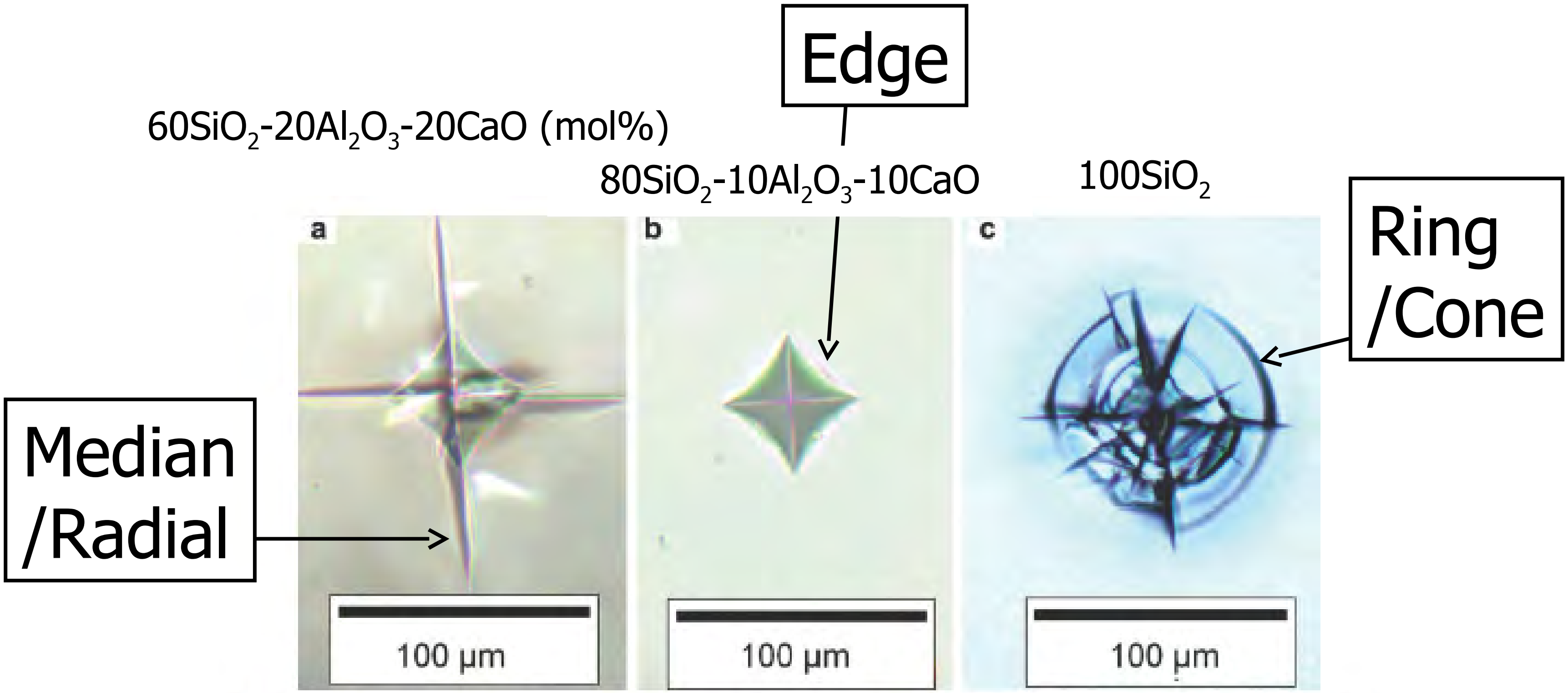
Indentation-induced densification is affected by

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.

A wide variety of crack morphology comes from different stress states.



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.)

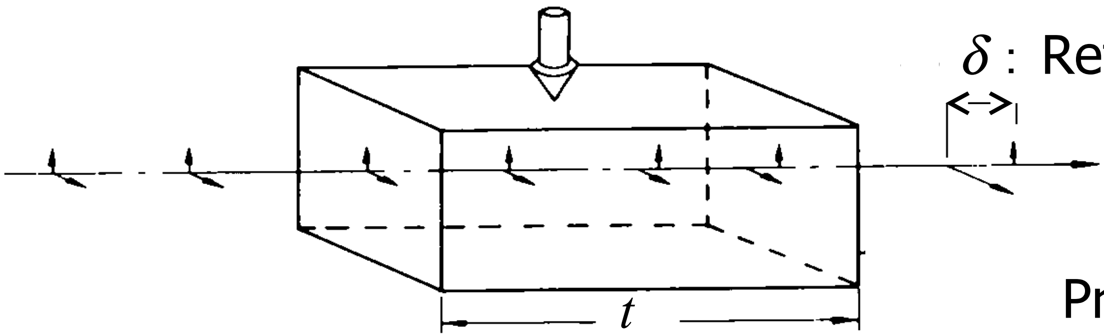
Birefringence, or Photoelasticity

2-Dimensional

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

Stress Optical Coefficient : C

δ : Retardation ⇨ $\sigma_1 - \sigma_2$



The stress state is biaxial.

Principal stresses : σ_1 , σ_2
(Membrane stresses)

Principal refractive indices : n_1, n_2

Determination of stress distribution

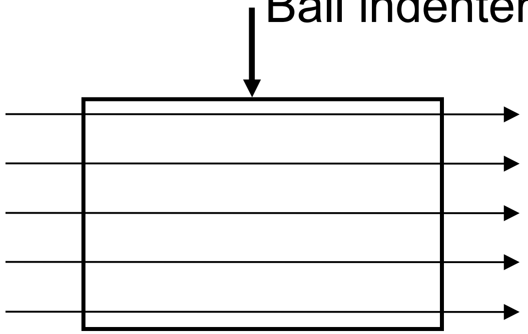
H. Aben, C. Guillemet, *Photoelasticity of Glass* , Springer (1993)
J. Anton, A. Errapart, H. Aben, L. Ainola, *Exp. Mech.* **48**(2008)613.

3-Dimensional

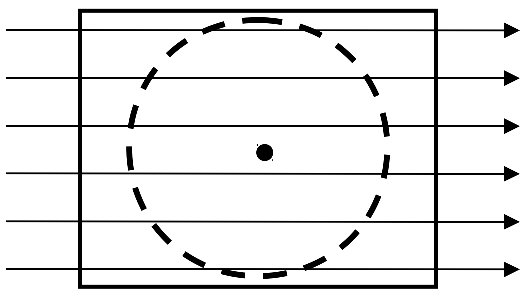
$$\delta = C\int(\sigma_1 - \sigma_2)dt$$

Ball indenter

Cross-section:



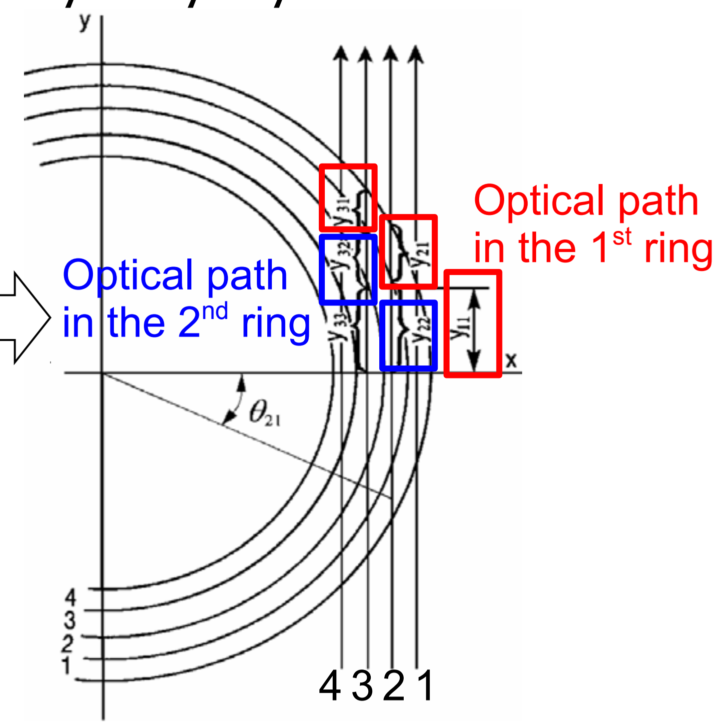
Top view:



Schematic of transmitted light through a square fiber

Onion peeling method

Stresses are calculated in layer-by-layer manner.



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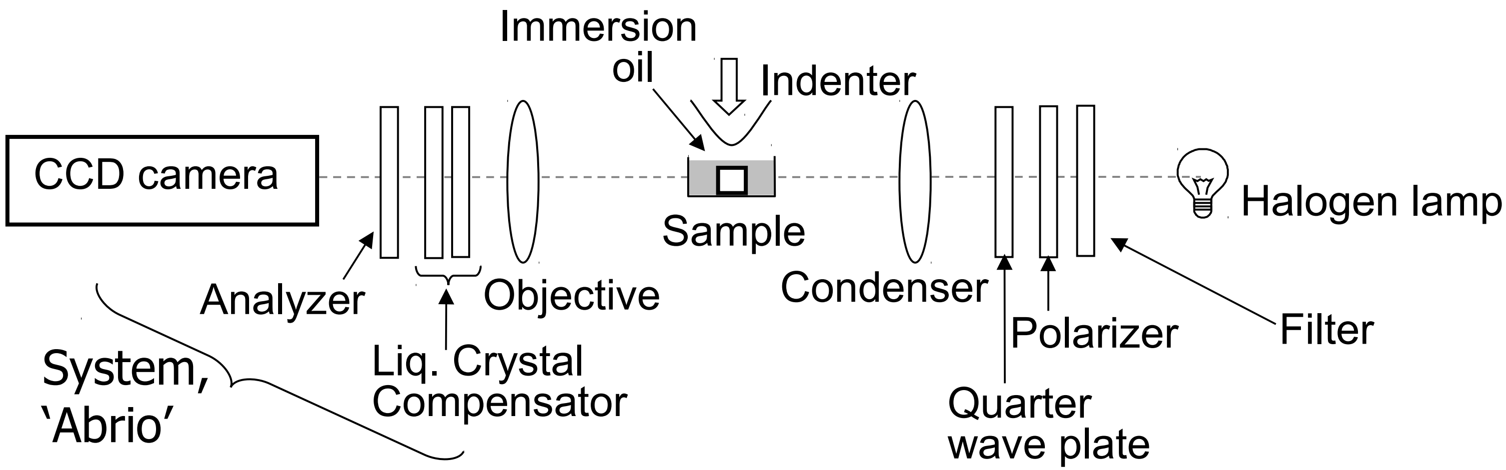
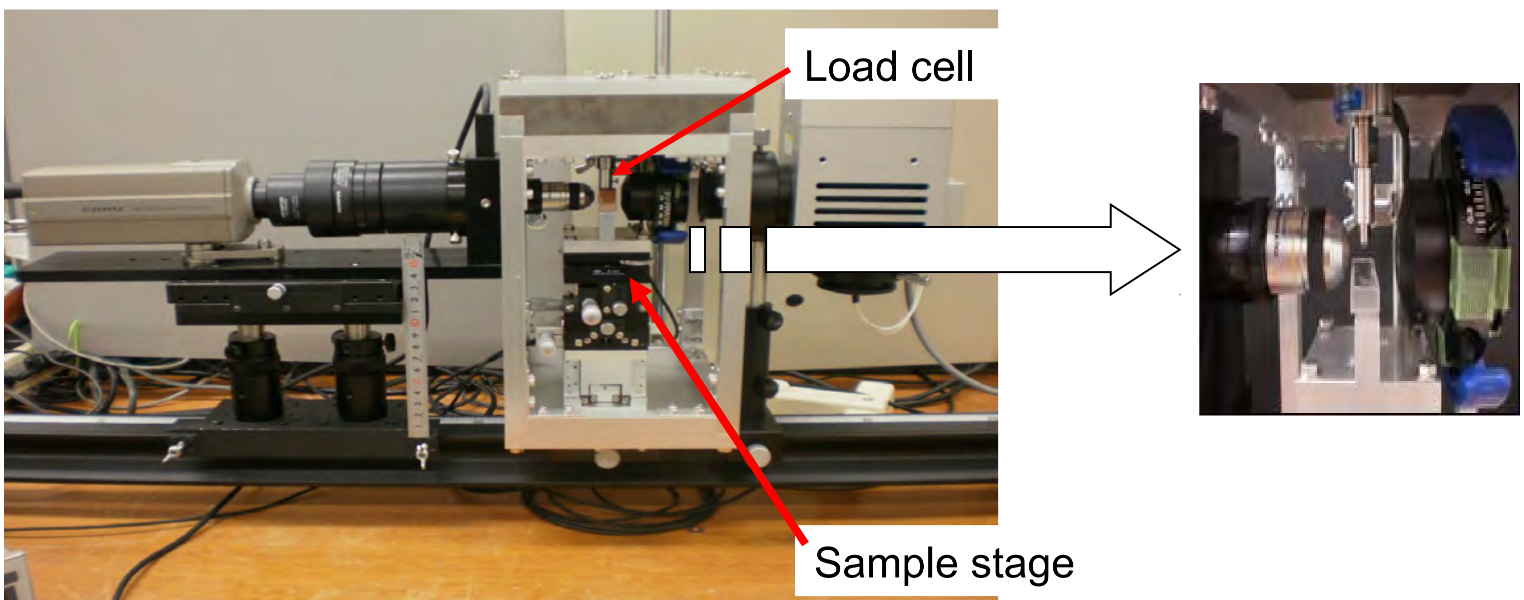
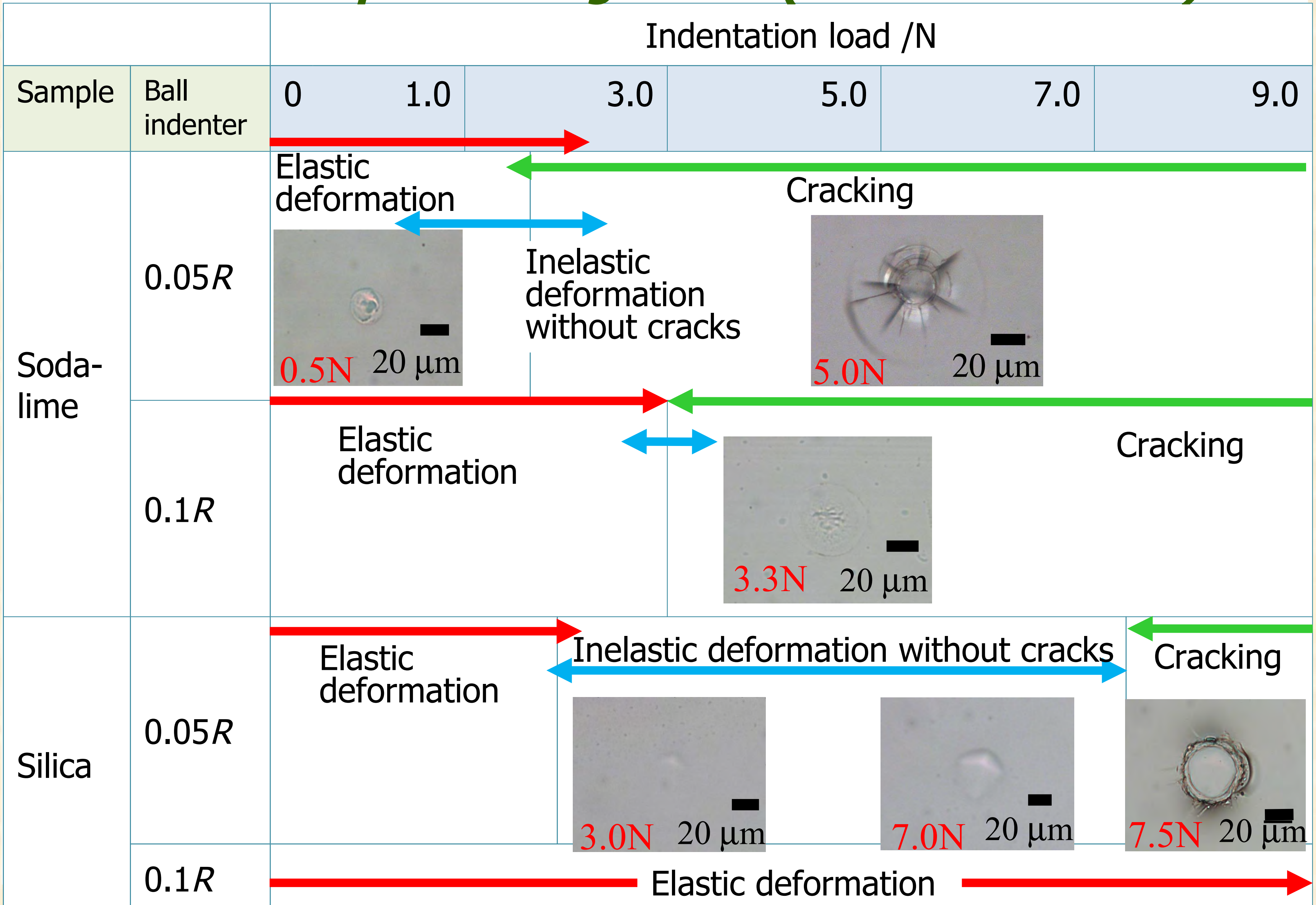
In-situ imaging system with an indenter

S. Yoshida *et al., J. Non-Cryst. Solids* **358** (2012)3465.





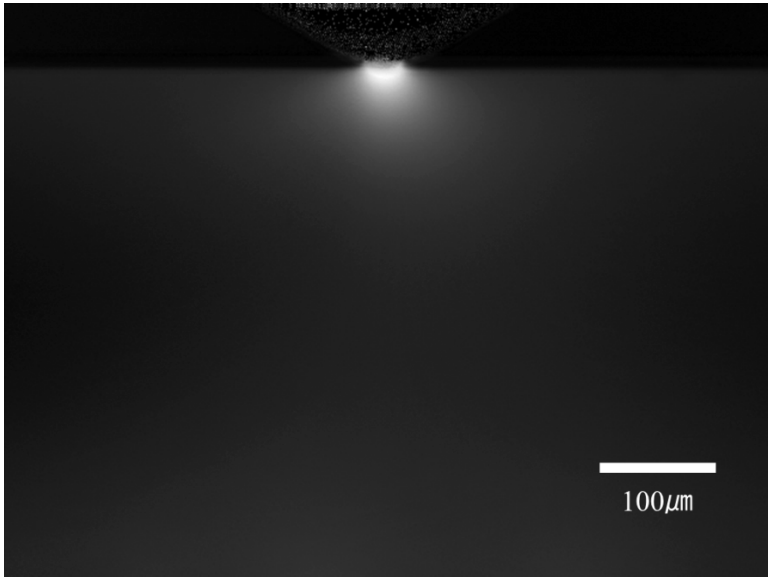
Mechanical responses of glasses (Ball indentations)



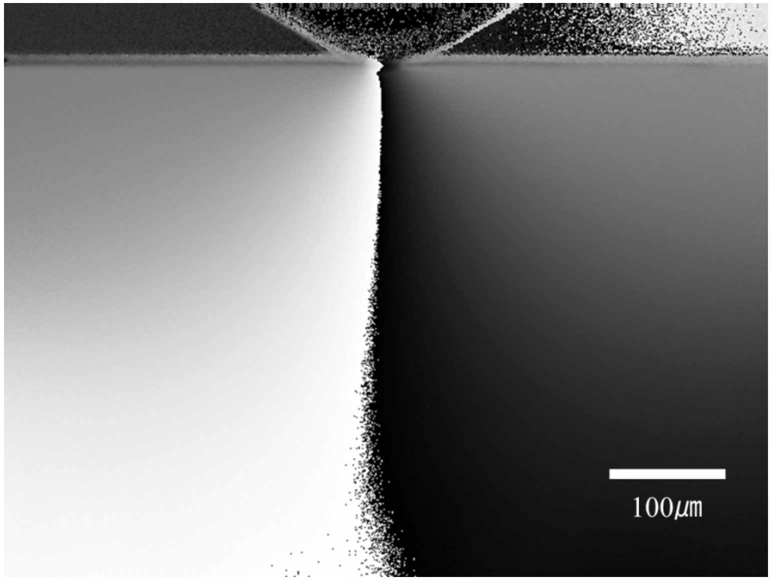
BR images during indentation

Soda-lime glass
R = 0.1 mm indenter
Indentation load = 3.0 N

During loading
<u>Only Elastic.</u>



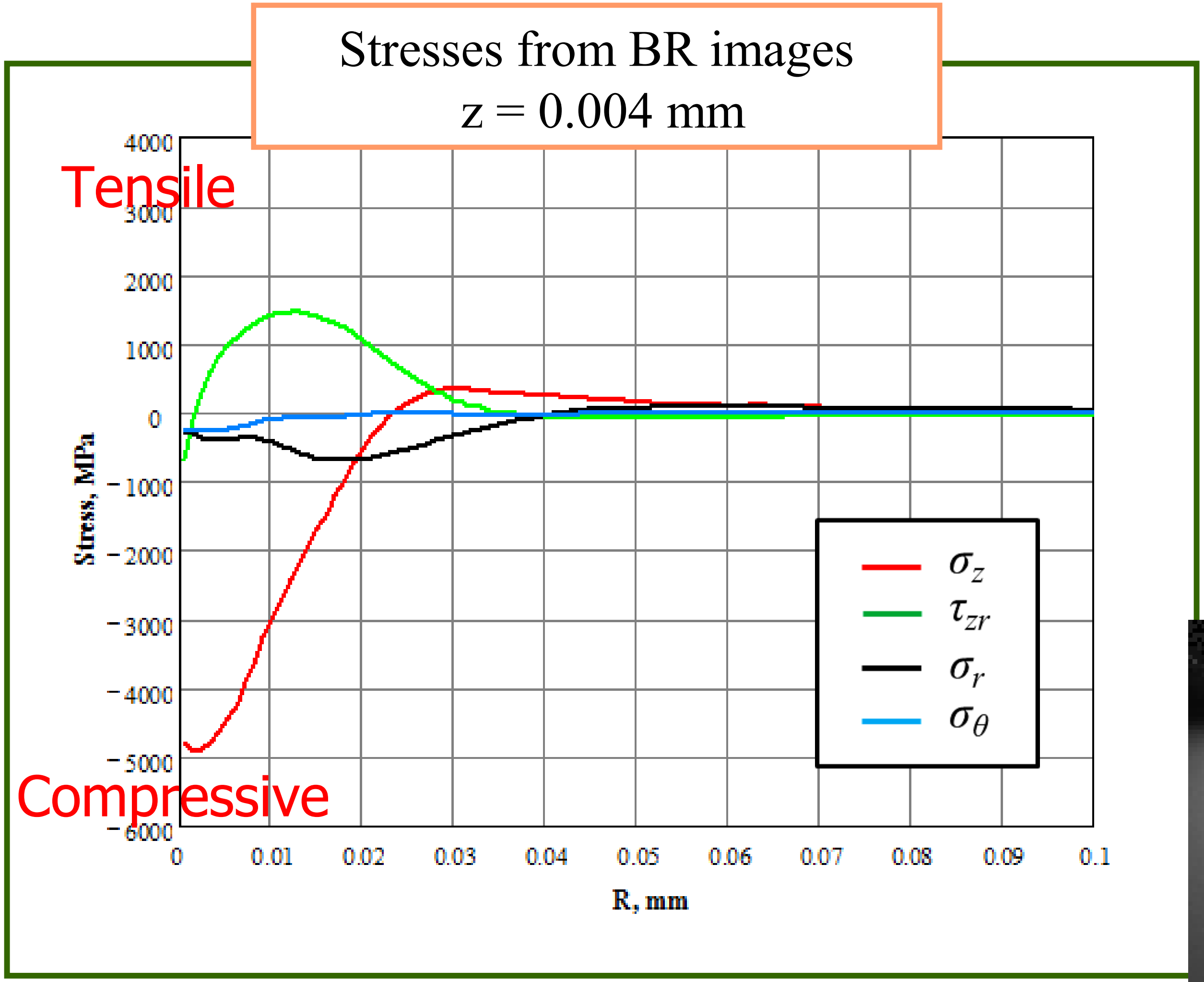
Retardance
0 ~ 250 nm
Black to White

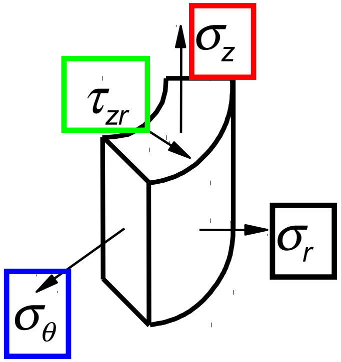


Slow axis orientation
0 ~ 180 º
Black to White

Elastic stresses (SLS)

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



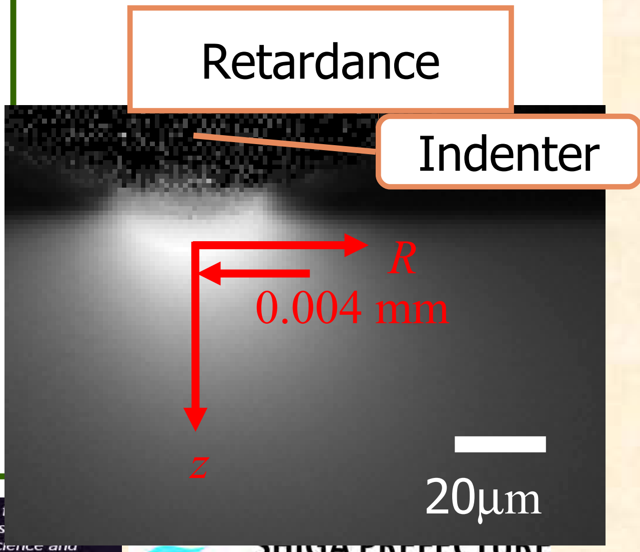


σ_z : Axial stress

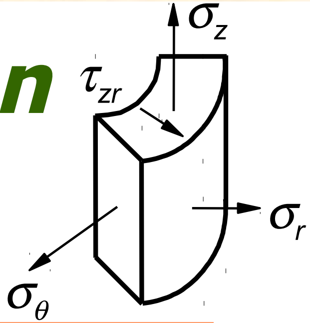
τ_{zr} : Shear stress

σ_r : Radial stress

σ_θ : Circumferential, or hoop, stress

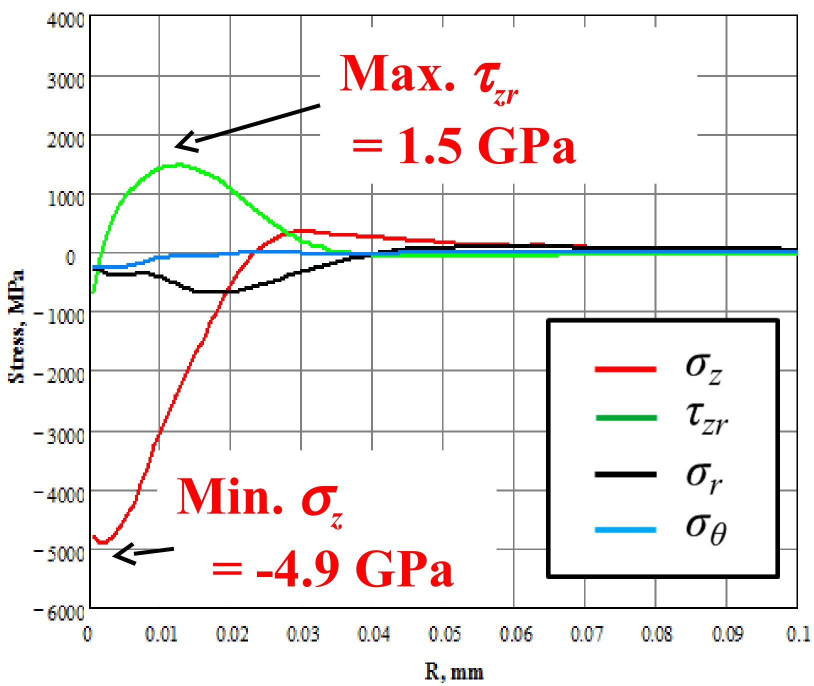


Comparison with analytical solution

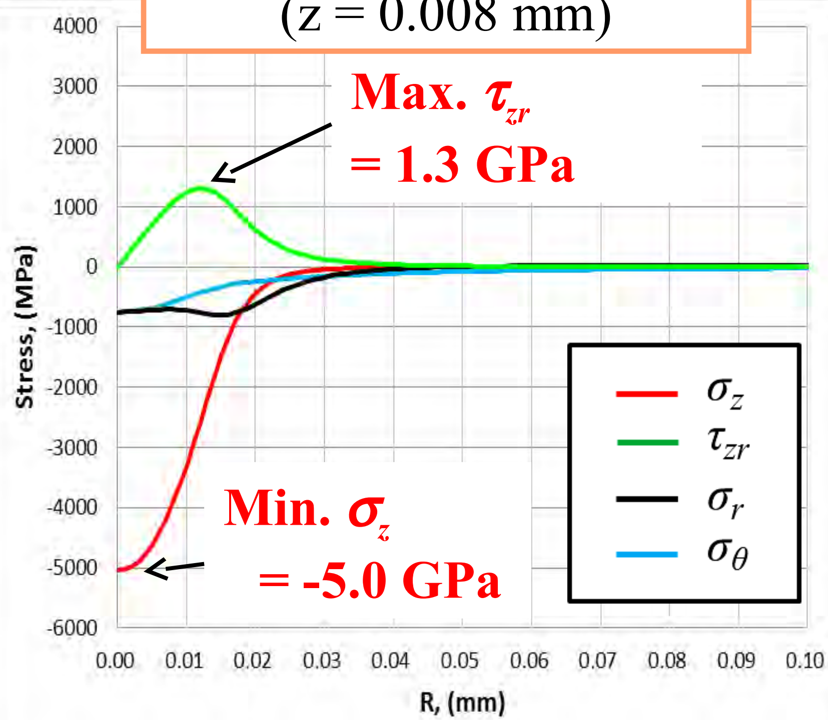


Soda-lime, R = 0.1 mm, Load = 3.0 N

BF exp. (z = 0.004 mm)



Hertzian solutions (z = 0.008 mm)



Obtained stresses are in agreement with Hertzian solutions.

Evaluation to Residual indents

» Silica (Anomalous)

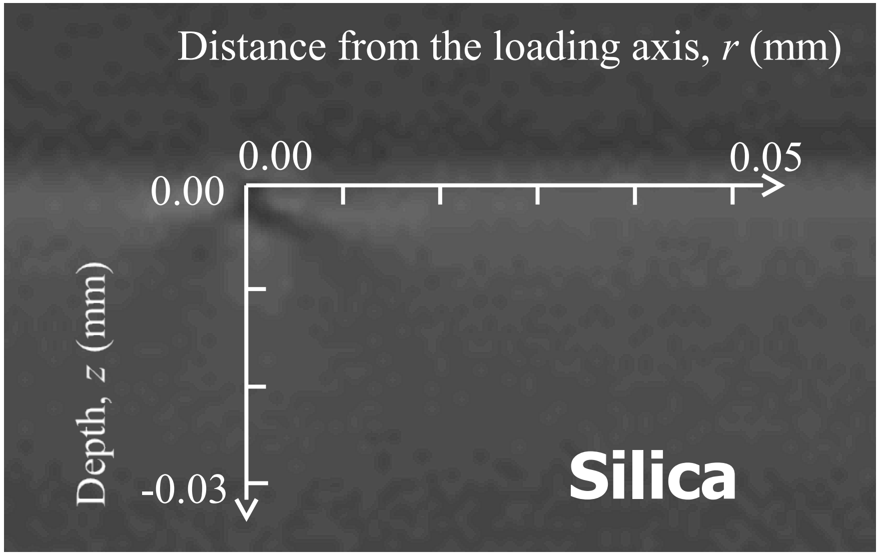
» $25Na_2O$-$75SiO_2$ (mol%) (Normal)

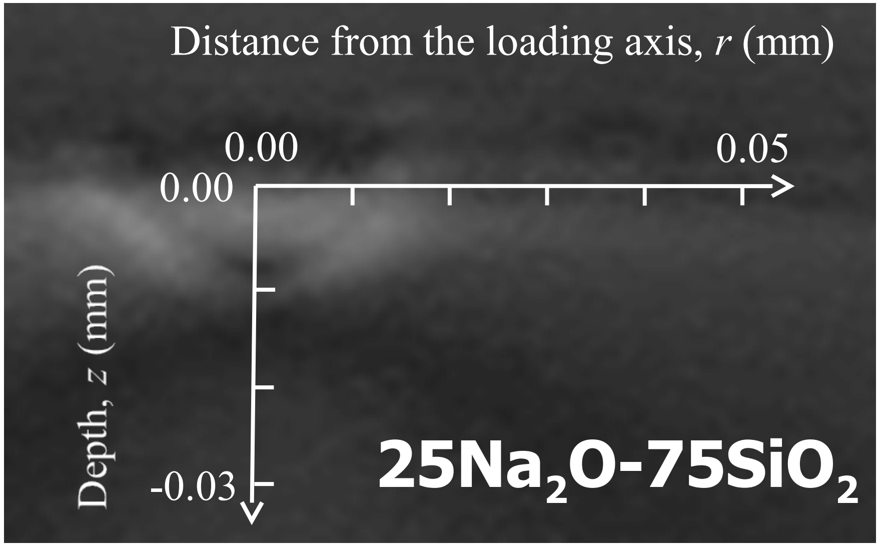
Residual stresses

Retardation maps
with
Coodinates for stress calculation

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

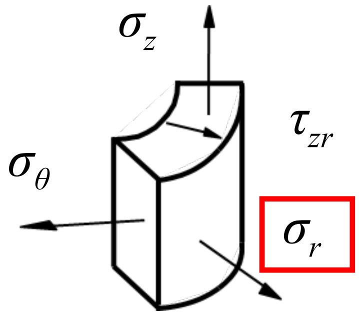
Quite different !



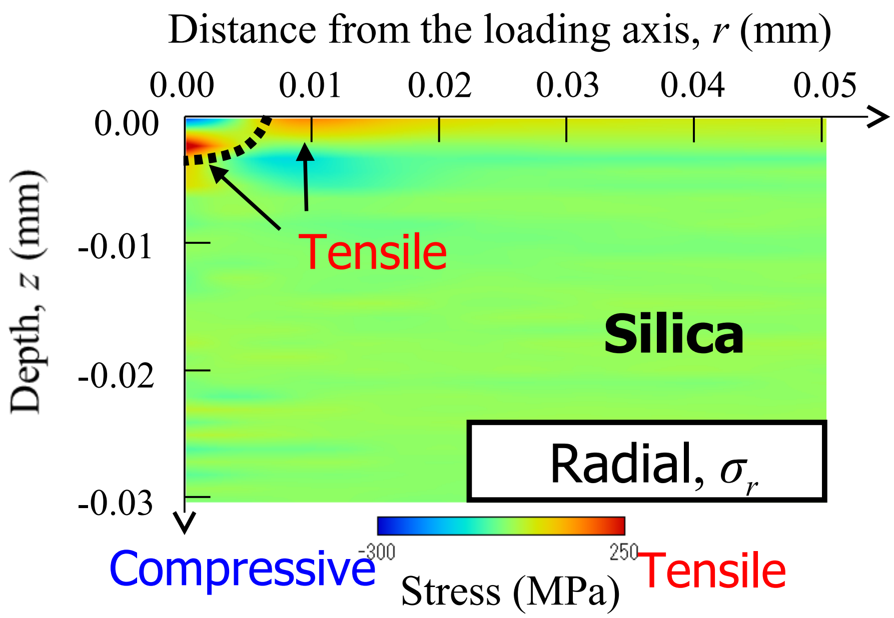


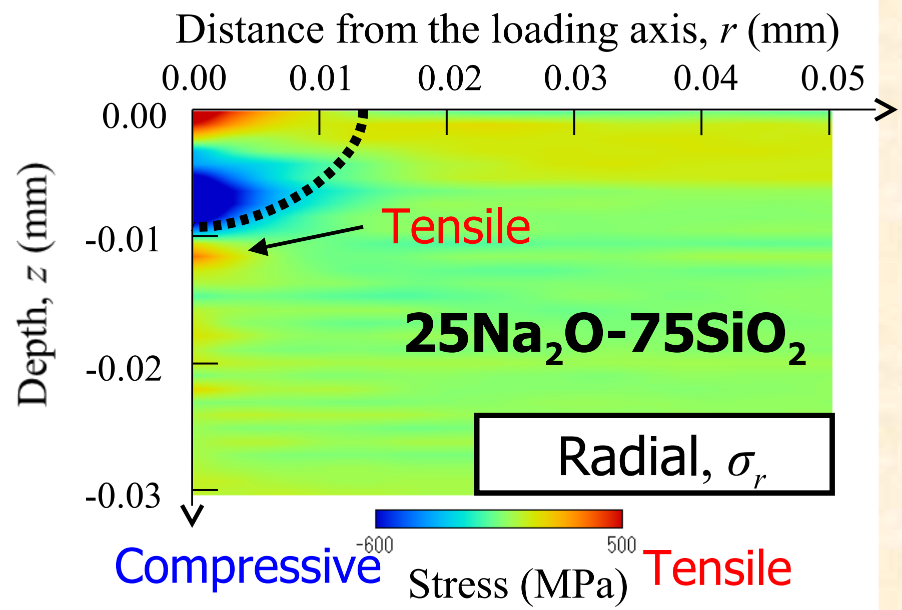
Residual stresses
Stress mapping (Radial stress)

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



: Plastic zone





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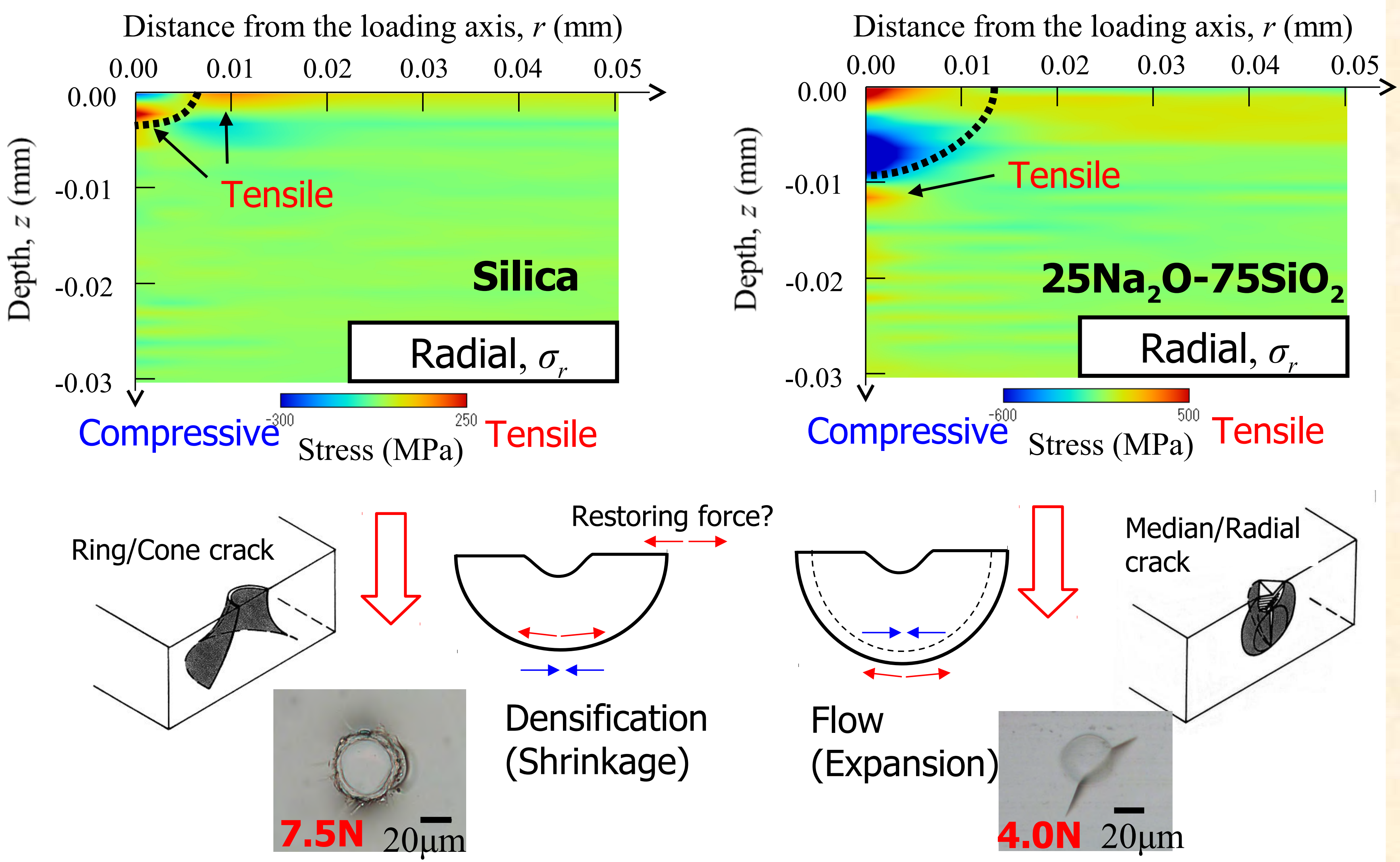
Residual stresses and crack morphology



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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.

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