

Indentation Deformation and Fracture of Glasses

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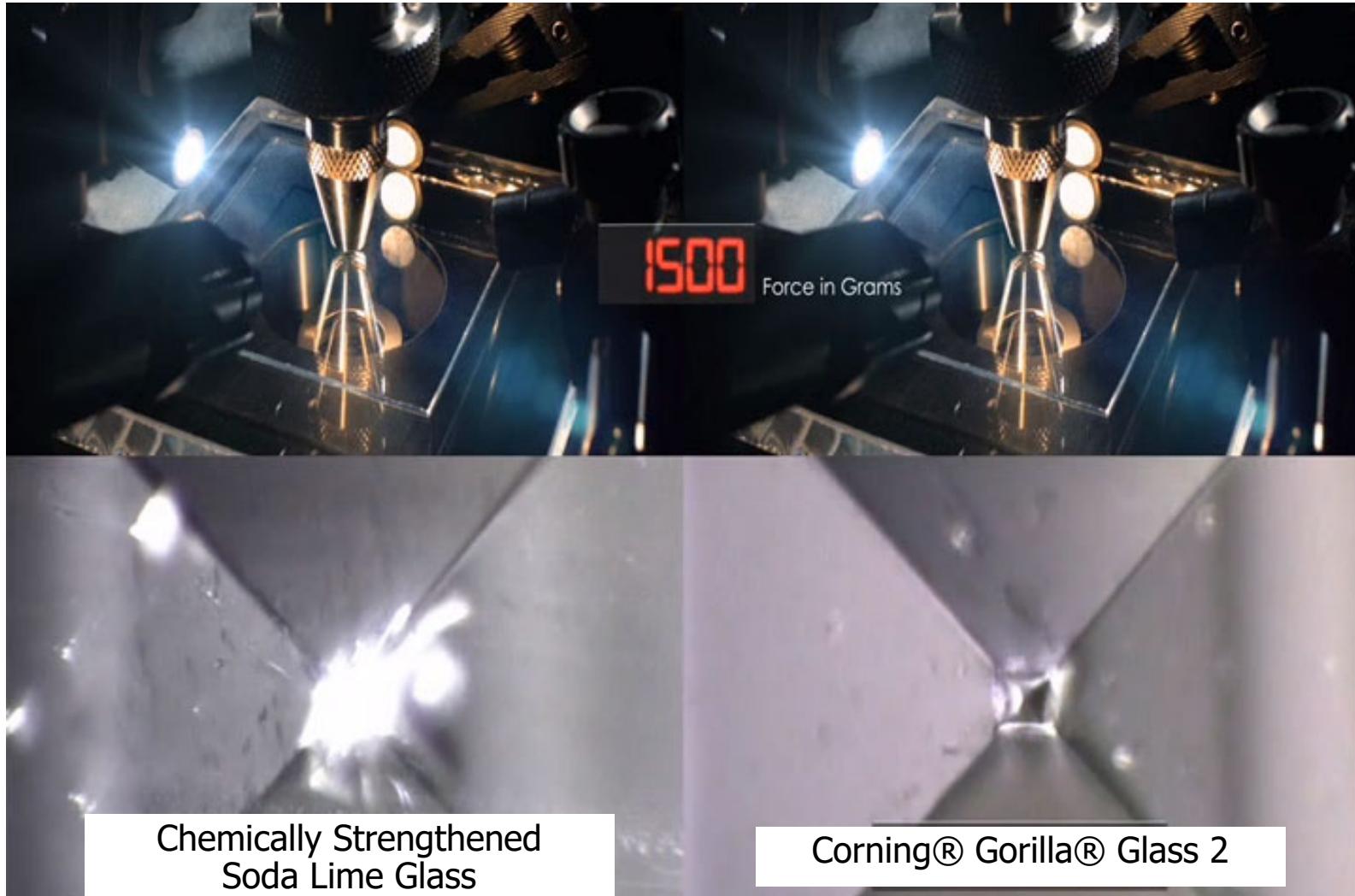


Outline

1. Why indentation ?
2. Some relations between composition and indentation fracture of glass
3. Indentation-induced densification in glass
4. How can we design tough glass ?

Why indentation ?

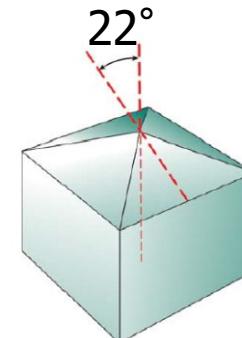
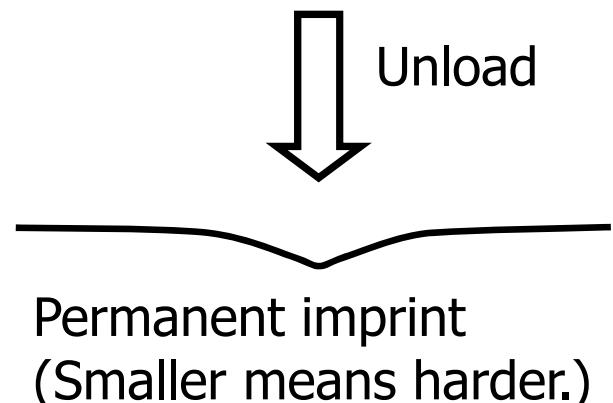
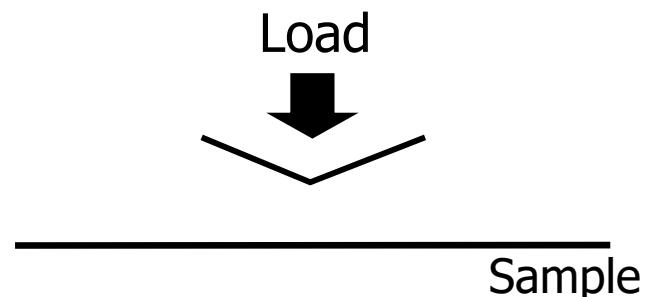
To evaluate fracture resistance against contact or scratch.



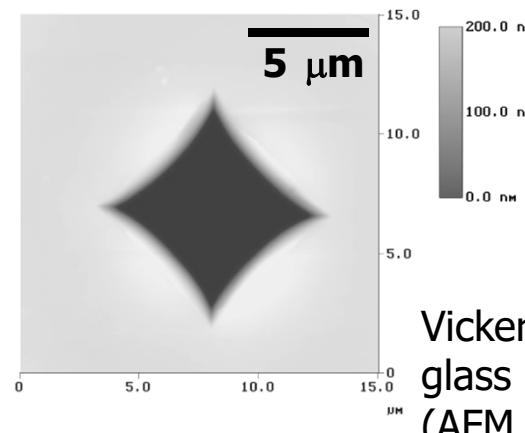
A snapshot of in-situ observation during indentation on glasses
(Corning Incorporated, <http://www.corninggorillaglass.com/>)

Why indentation ?

One of the simplest methods to evaluate mechanical properties of materials



Vickers indenter (Diamond indenter)



Vickers imprint on SLS glass at 300 mN (AFM top view)

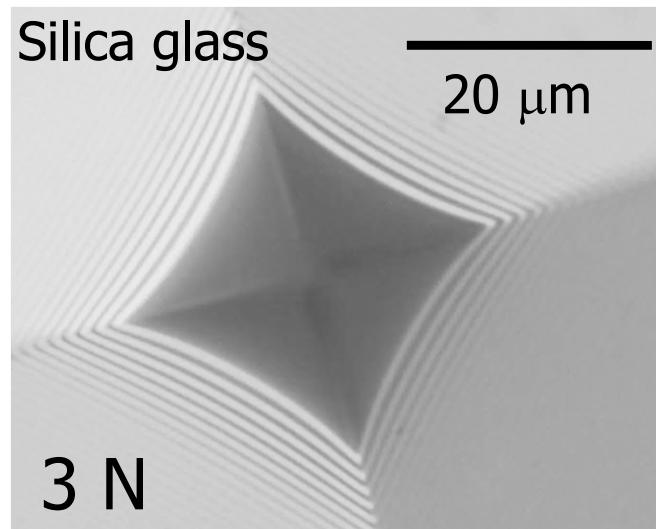
Resistance to **plastic deformation**

Why indentation ?

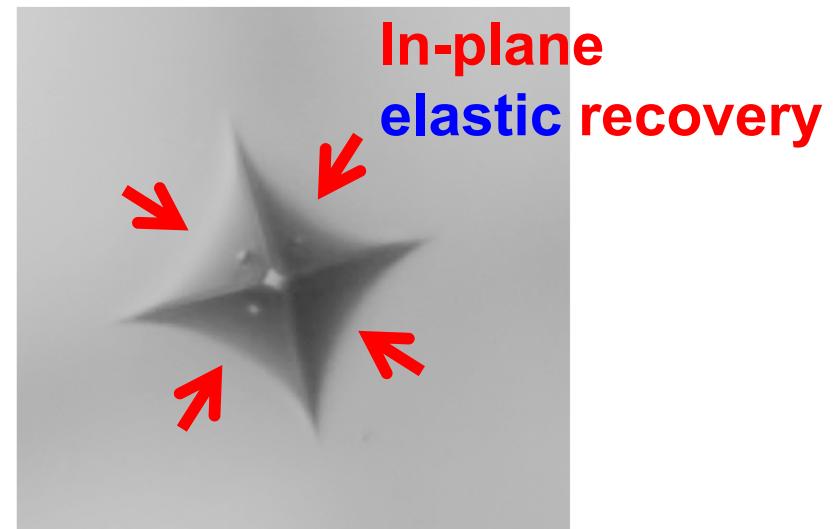
Not only plastic deformation ...

S. Yoshida *et al.*, *J. Mater. Res.* **30**(2015)2291.

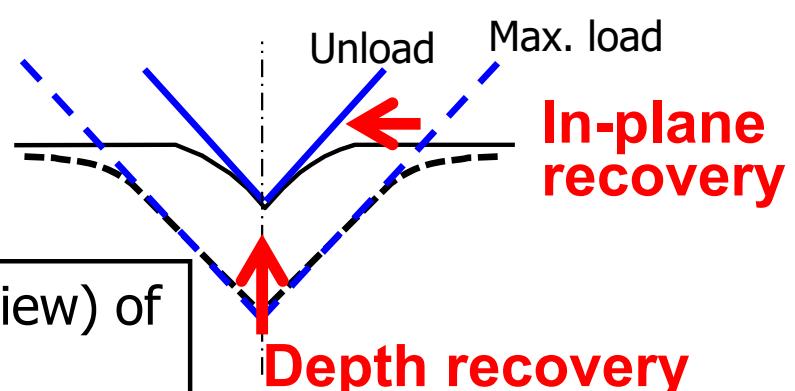
Resistance to elastic deformation (A large part of deformation can be recovered.)



Unload



In-situ observation
during indentation

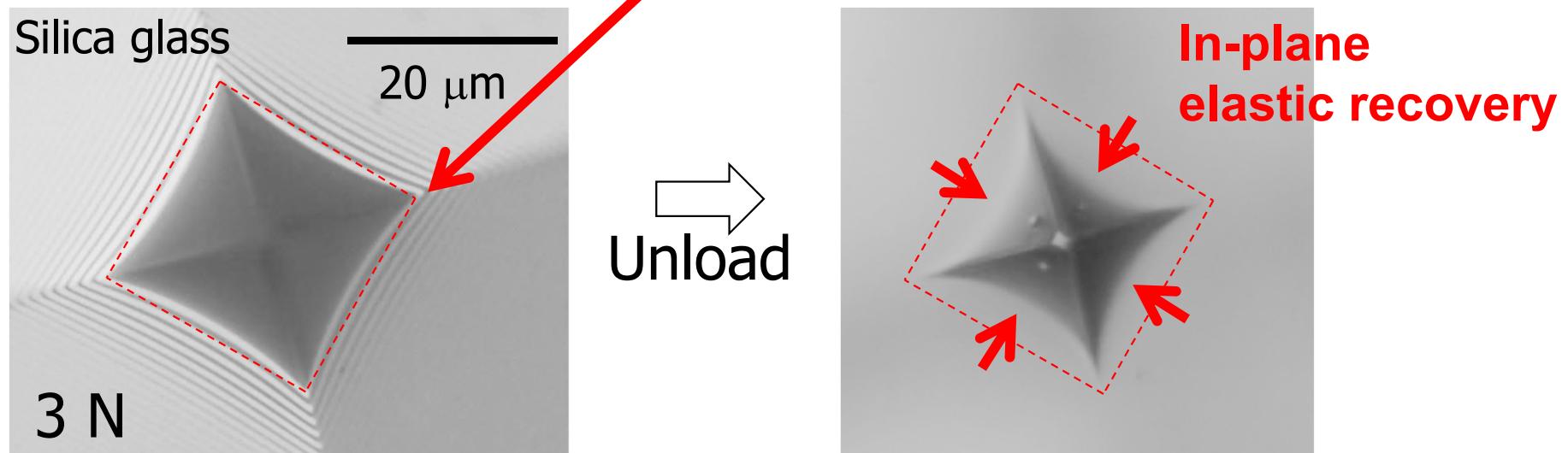


Schematic illustration (side-view) of
unloading process

Why indentation ?

Not only plastic deformation ...

Vickers hardness = (Load) / (Estimated contact area at a max. load)



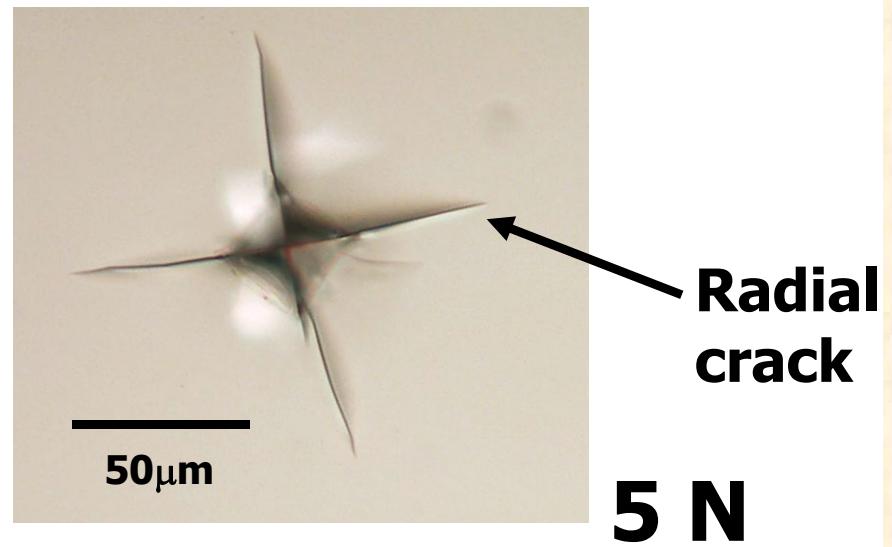
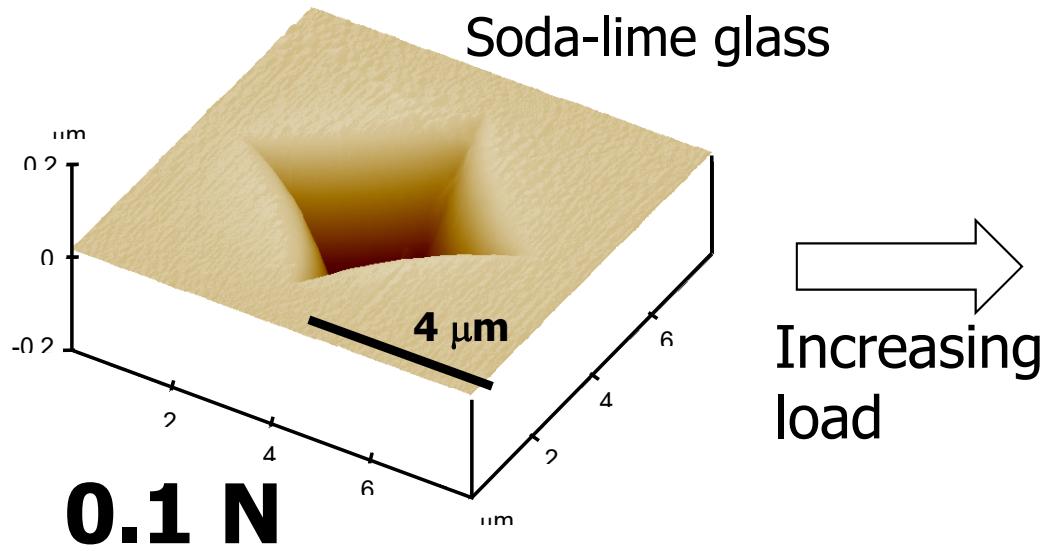
**Vickers hardness of glass is a measure of
Resistance to Elastic/Plastic deformation.**

M. Sakai, *J. Mater. Res.* **14**(1999)3630.

Why indentation ?

Not only elastic/plastic deformation ...

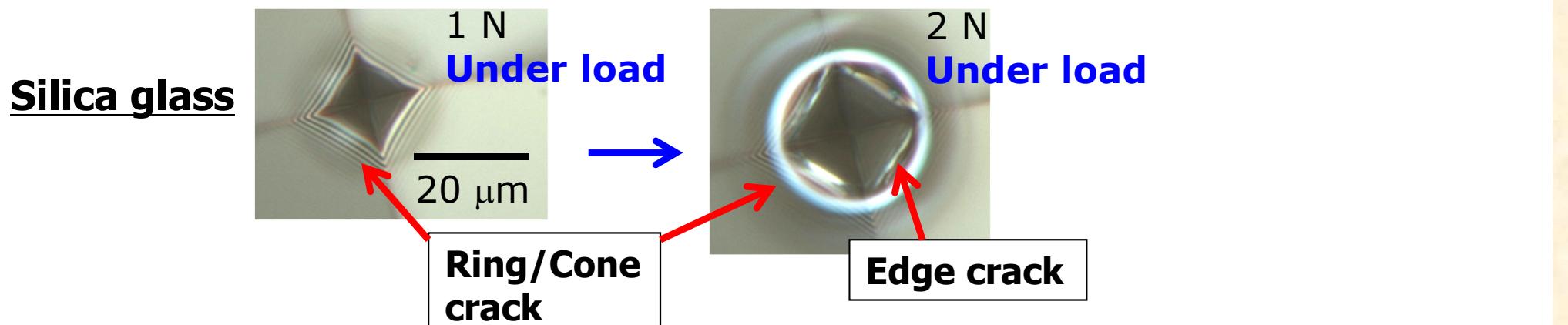
Resistance to **fracture**



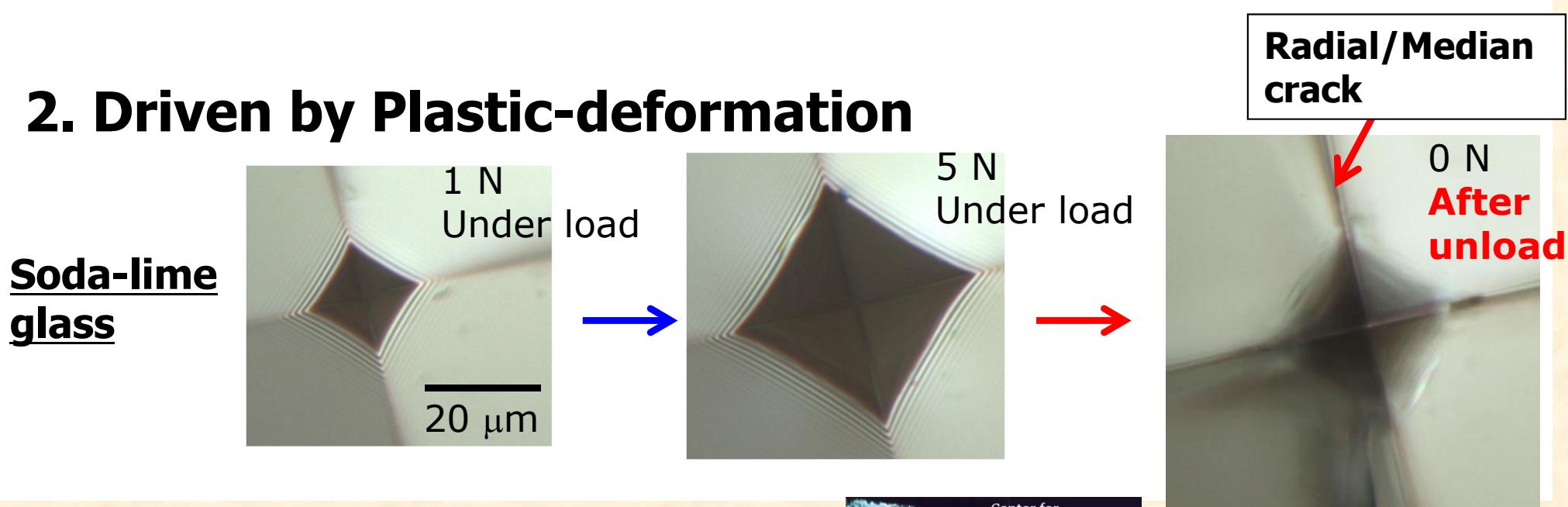
Indentation-induced fracture is affected both by elastic deformation and by plastic deformation.

Some types of indentation cracks

1. Driven by Elastic-deformation



2. Driven by Plastic-deformation

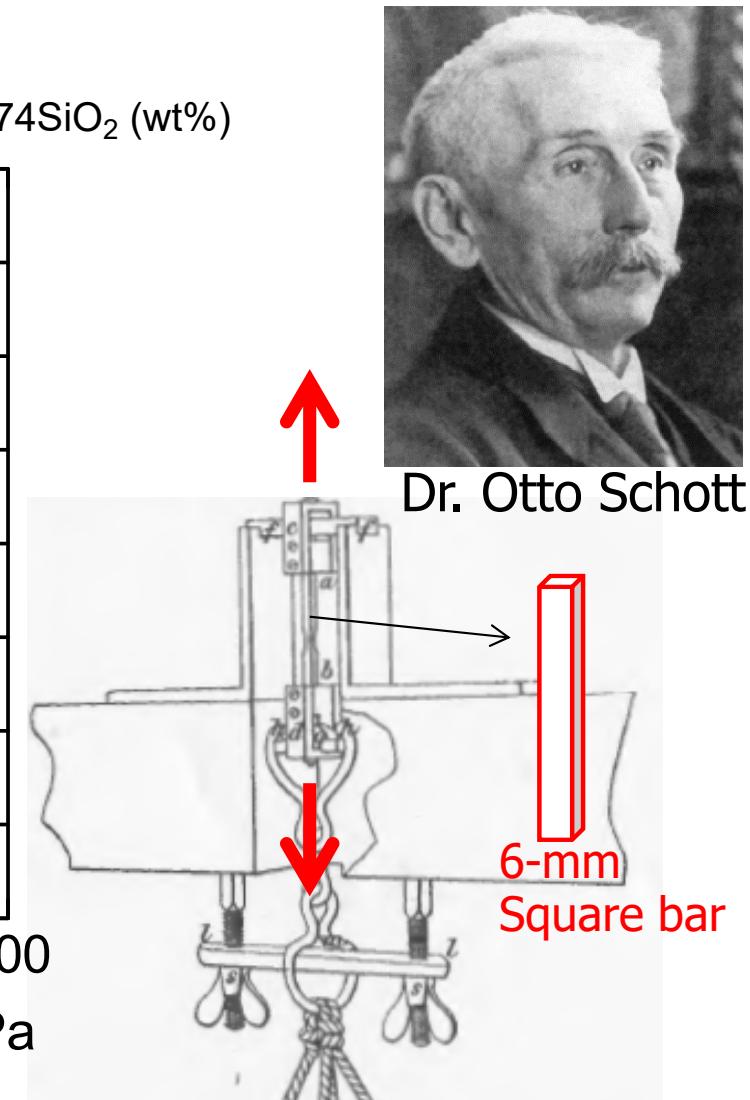
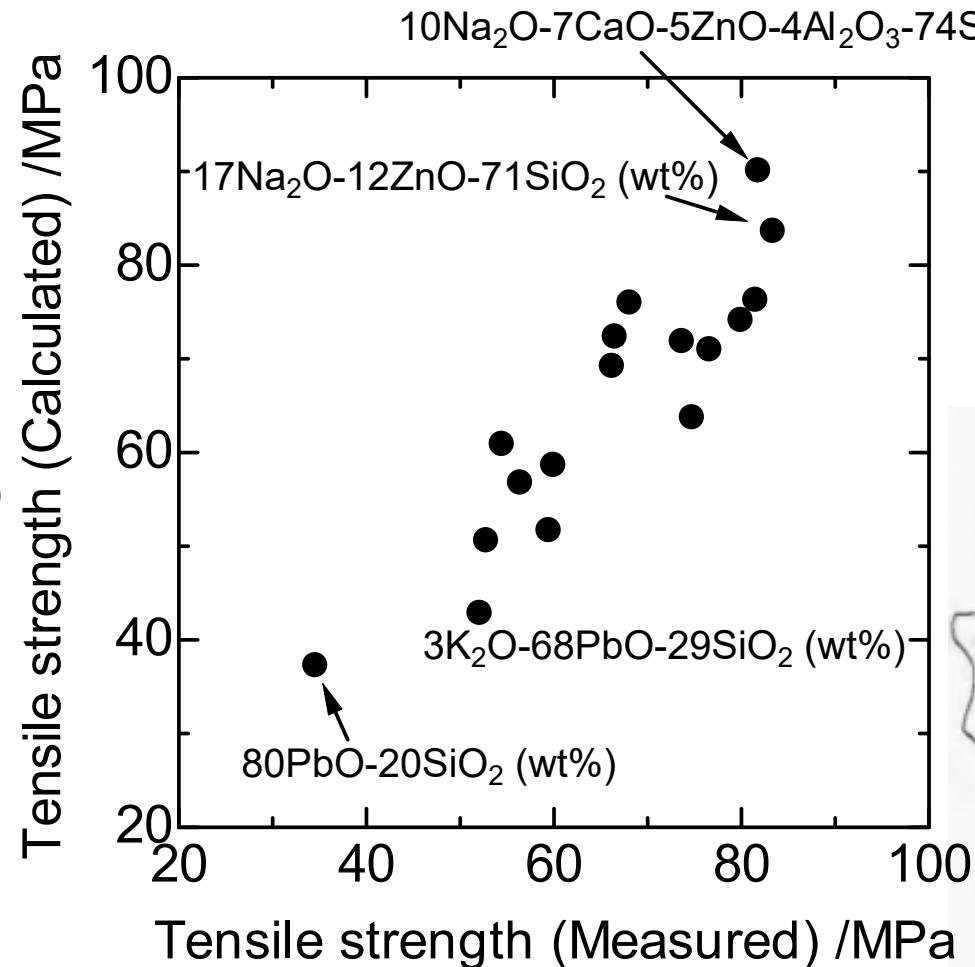


Some relations between glass composition and (indentation) fracture

The "classical" compositional variation of glass strength

C. Kurkjian & W.R. Prindle, *J. Am. Ceram. Soc.* **81**(1998)795.

From additivity
equation
→
like INTERGLAD
or SciGlass



A. Winkelmann & O. Schott, *Ann. Phys. Chem.* **51** (1894) 697.

Comp. dependence of indentation cracking

80 years from Otto-Schott

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

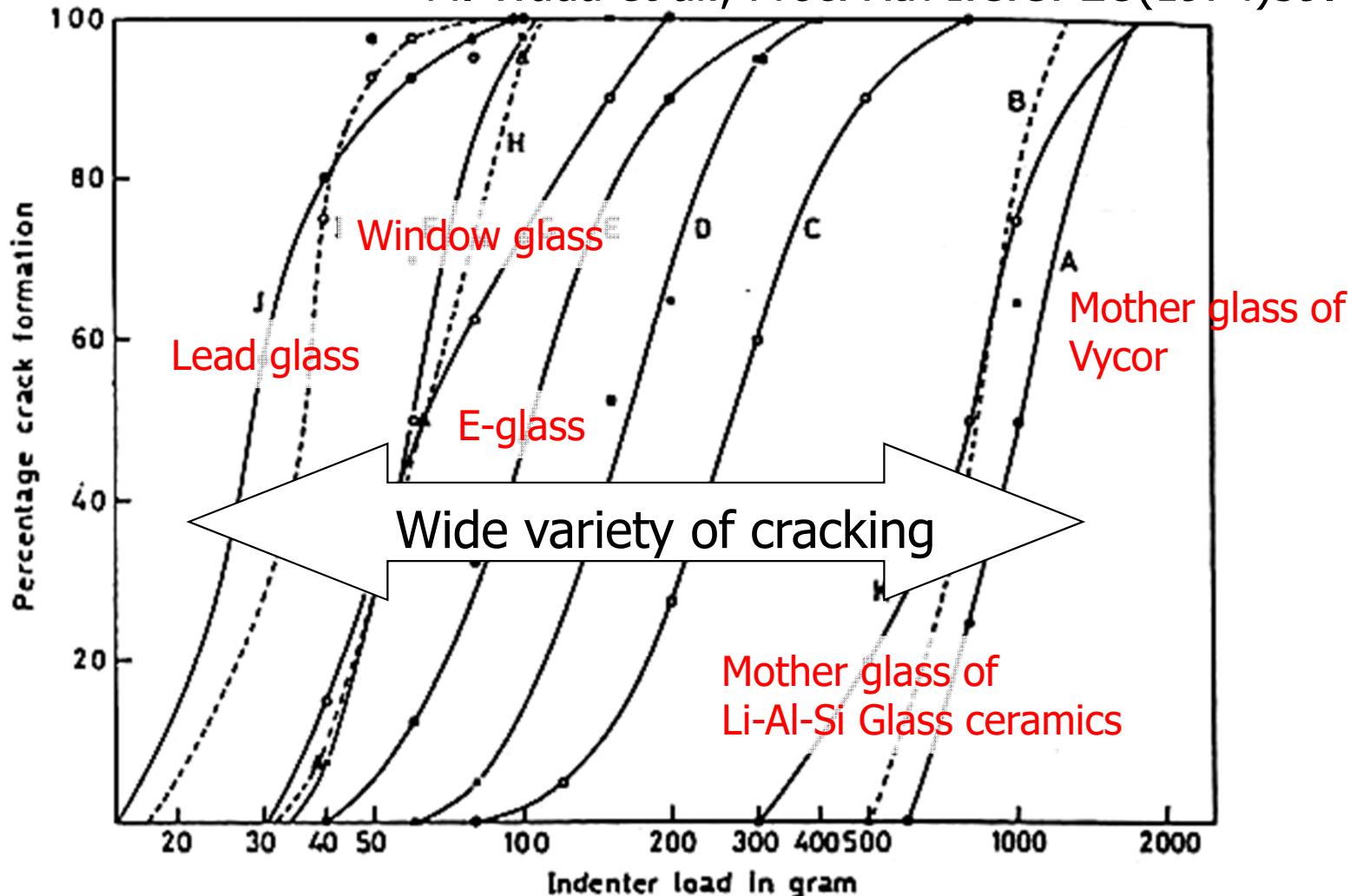
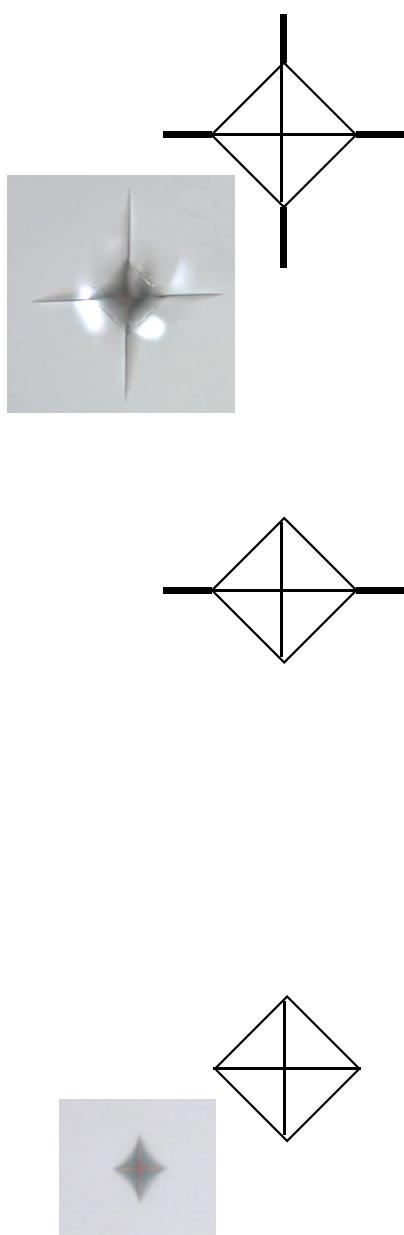


Figure 4. Crack resistances of various glasses.

Radial/Median

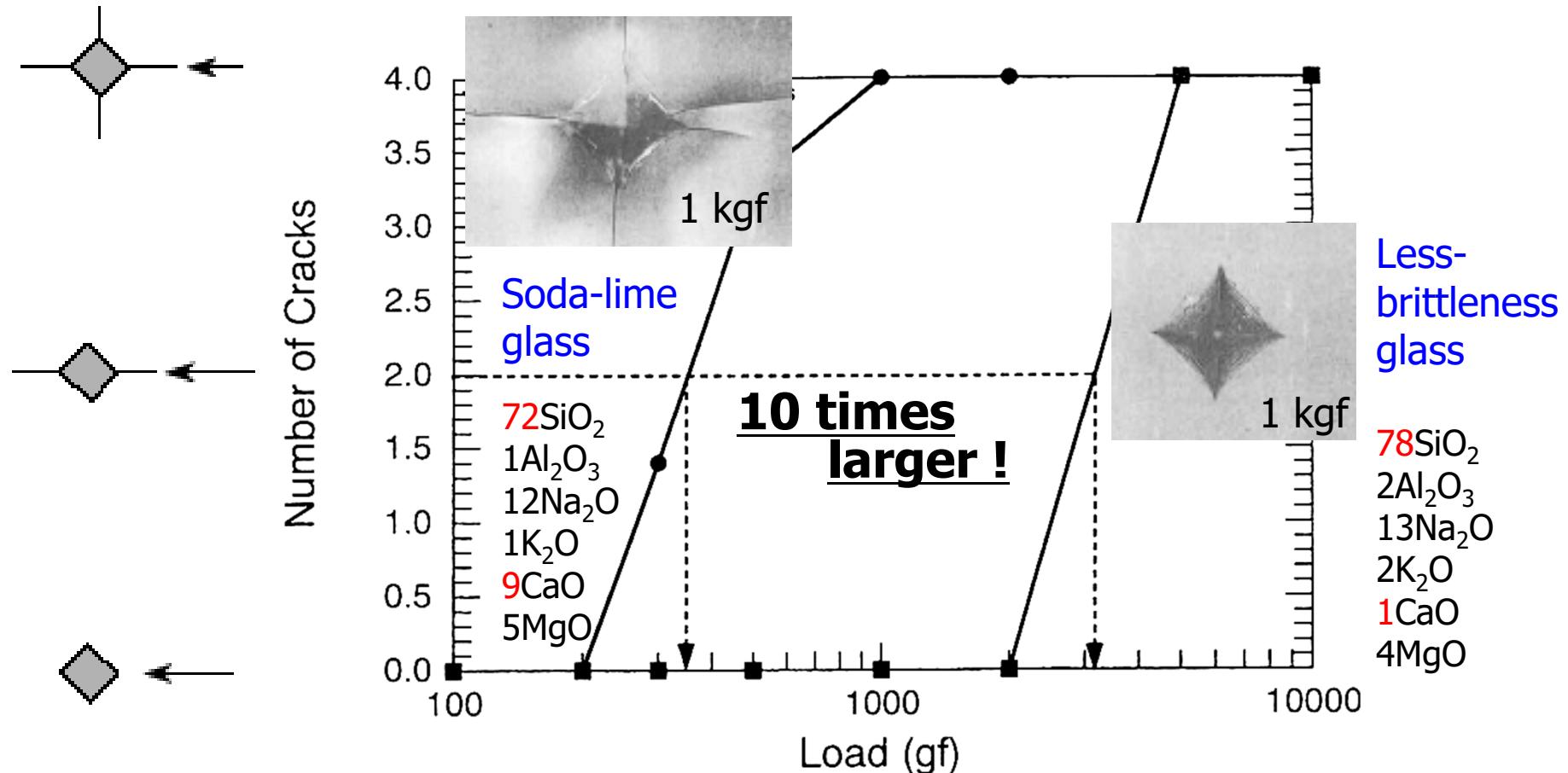


THE UNIVERSITY OF
SHIGA PREFECTURE

The University of Shiga Prefecture

Comp. dependence of indentation cracking

104 years from Otto-Schott



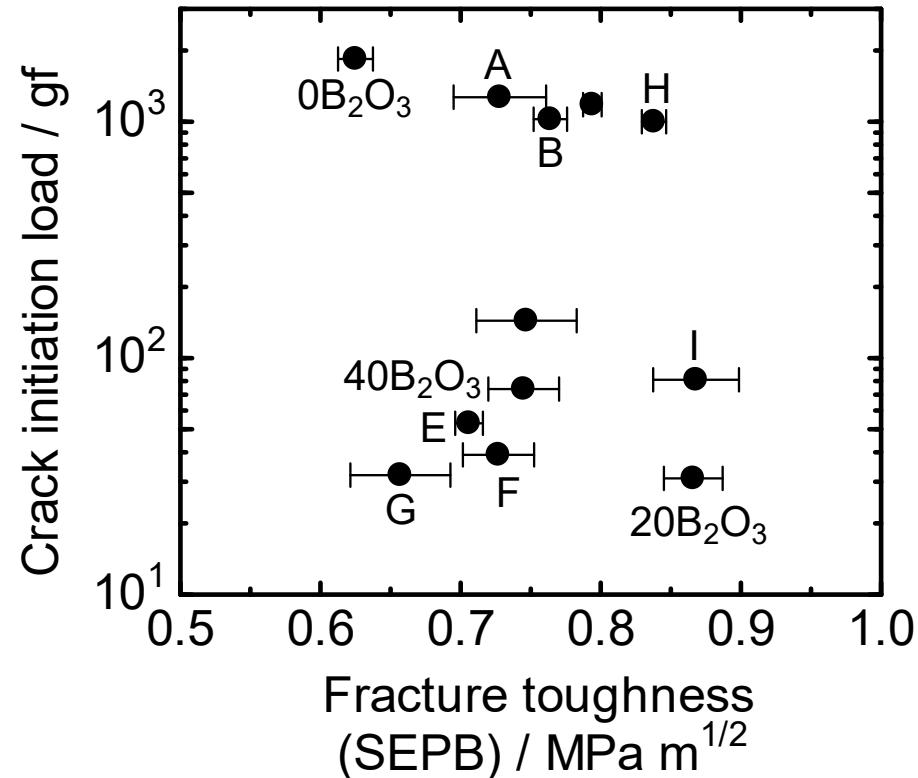
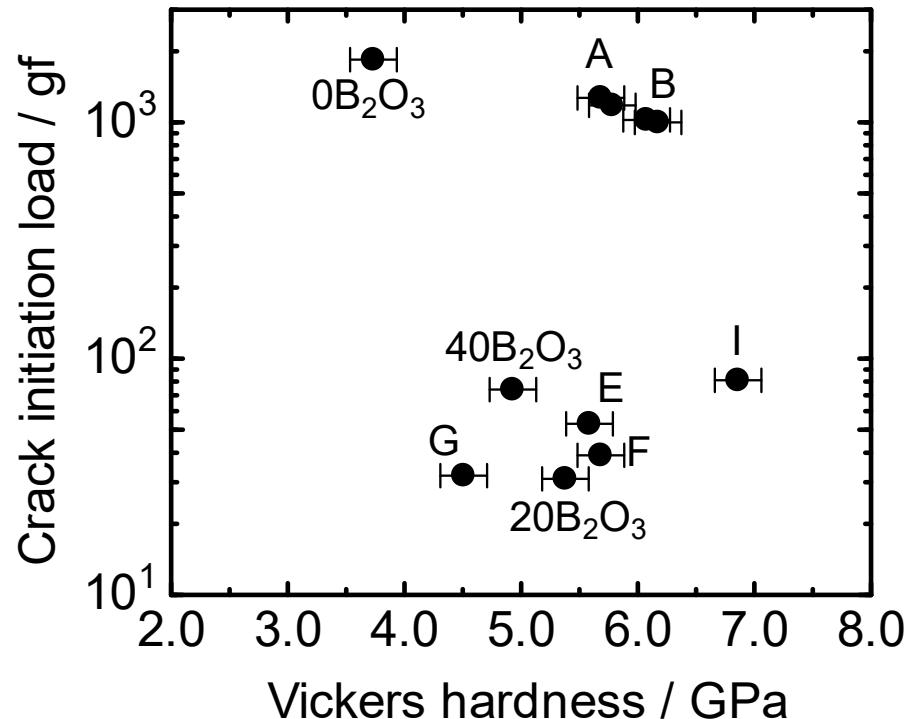
J. Sehgal & S. Ito, *J. Am. Ceram. Soc.* **81**(1998)2485.

What factors determine the crack initiation load?

No relation between crack initiation and other mechanical properties

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

Y. Kato, *JNCS* (2010)



A: SiO₂-B₂O₃-K₂O

D: SiO₂-CaO-Na₂O

G: SiO₂-B₂O₃-PbO

0B₂O₃, 20B₂O₃, 40B₂O₃: (80-x)SiO₂-x B₂O₃-20Na₂O

B: SiO₂-B₂O₃-Na₂O

E: SiO₂-SrO-Na₂O

H: SiO₂-Al₂O₃-Li₂O

C: SiO₂-Al₂O₃-B₂O₃

F: SiO₂-SrO-K₂O

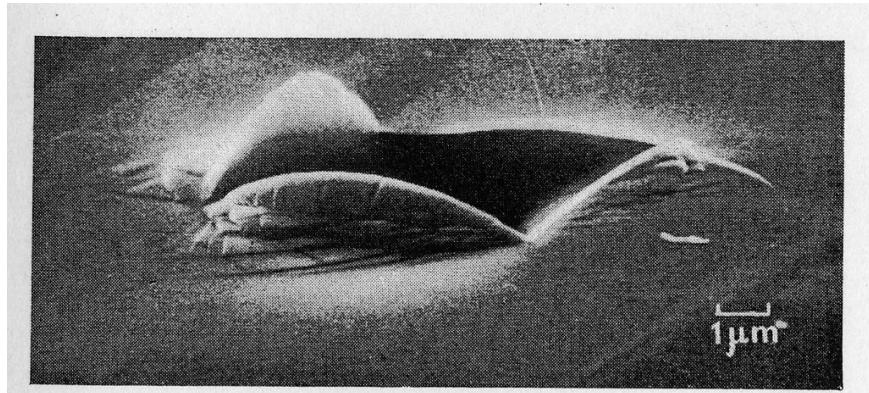
I: Li-Al-Si Glass-ceramics

Indentation-induced permanent densification

Indentation-induced flow and densification

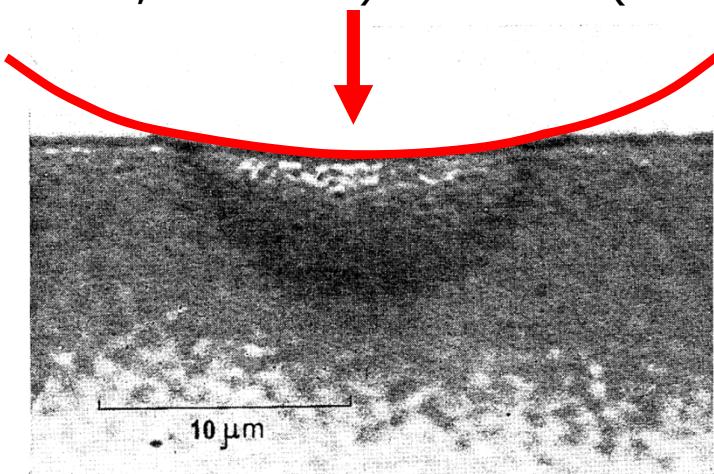
Plastic flow and/or Densification

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 = $20 \mu\text{m}$, Load = 100 gf)

Blunt indenter
Densification !

Indentation-induced Residual Stress

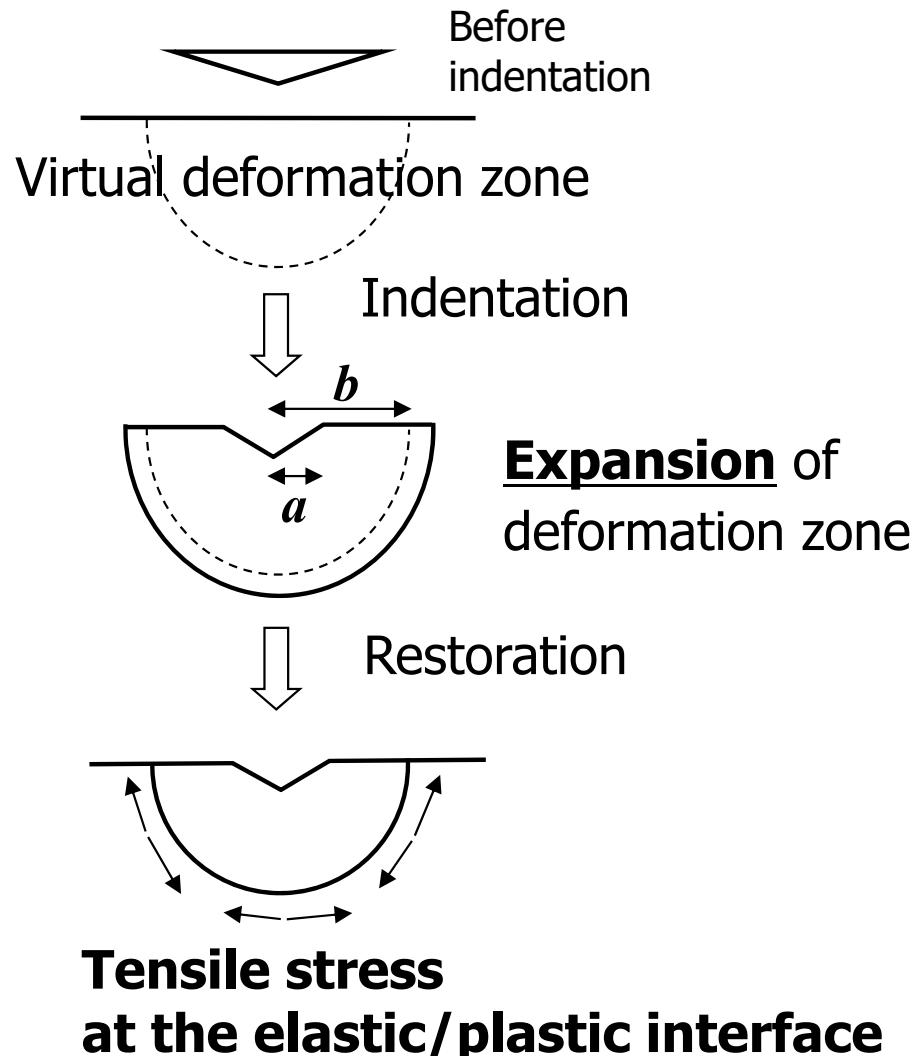
B.R. Lawn, A.G. Evans, D.B. Marshall, *J. Am. Ceram. Soc.* **63**(1980)574.

Residual stress =
Bulk modulus \times Volume strain

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

$\Delta V \propto a^3$,
Lost volume $V \propto b^3$
 Plastic-zone
 volume

K : Bulk modulus



Indentation on glass results in

1. Shear flow (Volume conservative)

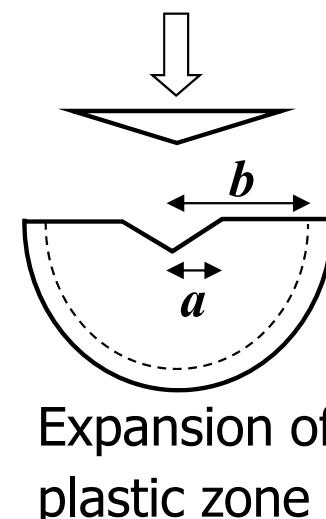
and

2. Permanent densification (Shrinkage)

Densification does not contribute to expansion of plastic zone.

Expansion of plastic zone

No expansion

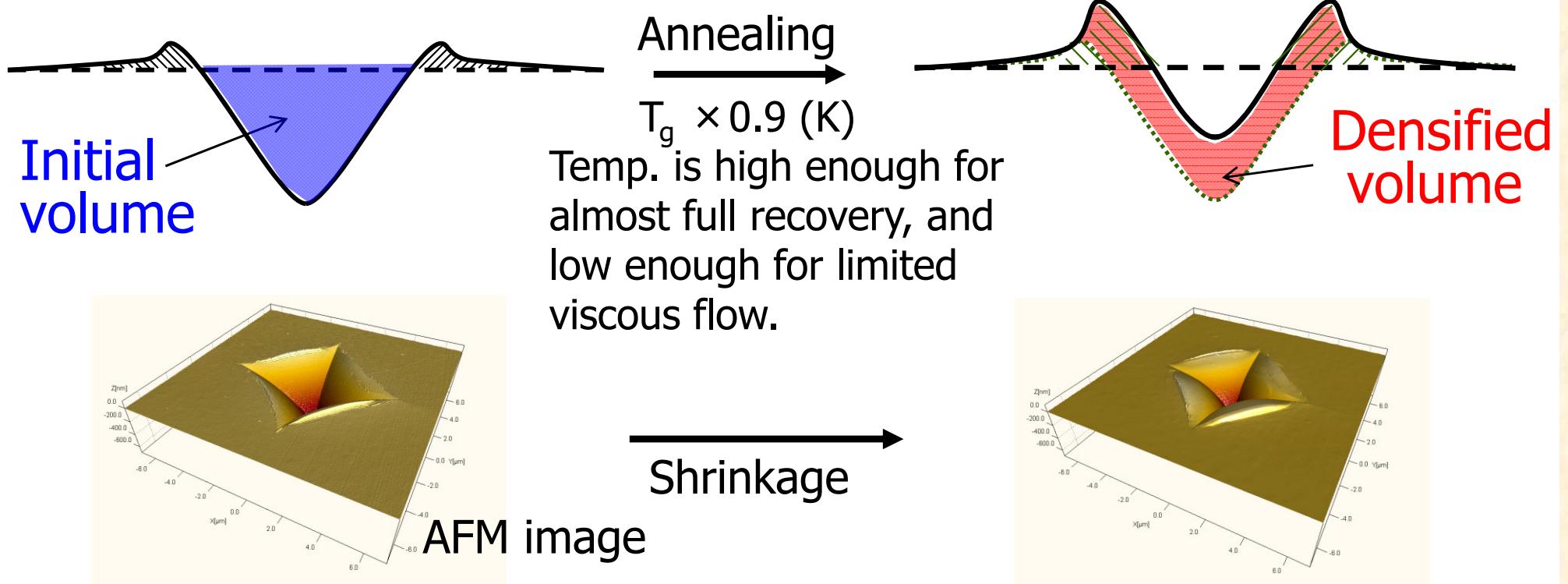


Estimation of densification contribution to total indentation deformation

Discrimination of the densified volume

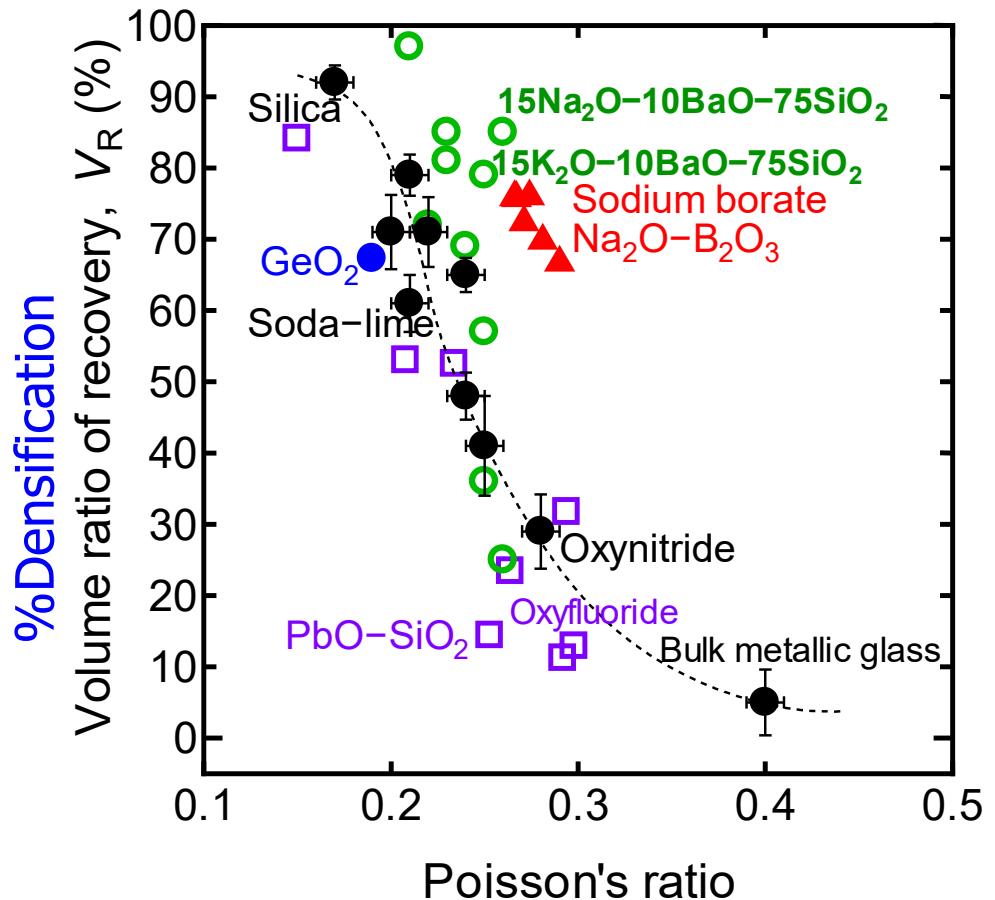
Densified region can be relaxed by annealing at around T_g

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



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

Compositional variation of densification contribution



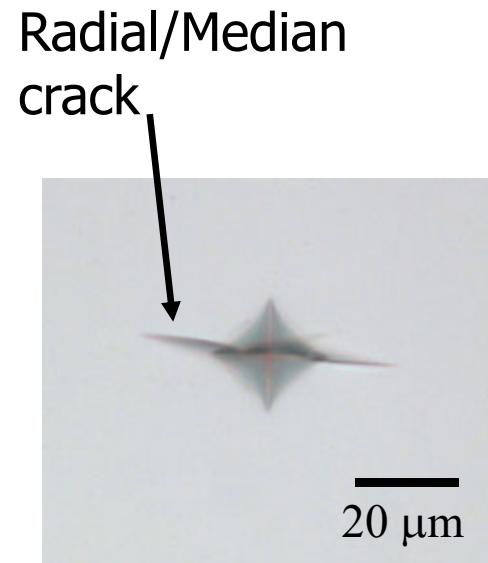
Lower Poisson's ratio results in higher %densification.

Elastic property (Poisson's ratio) controls Plastic property (%densification).

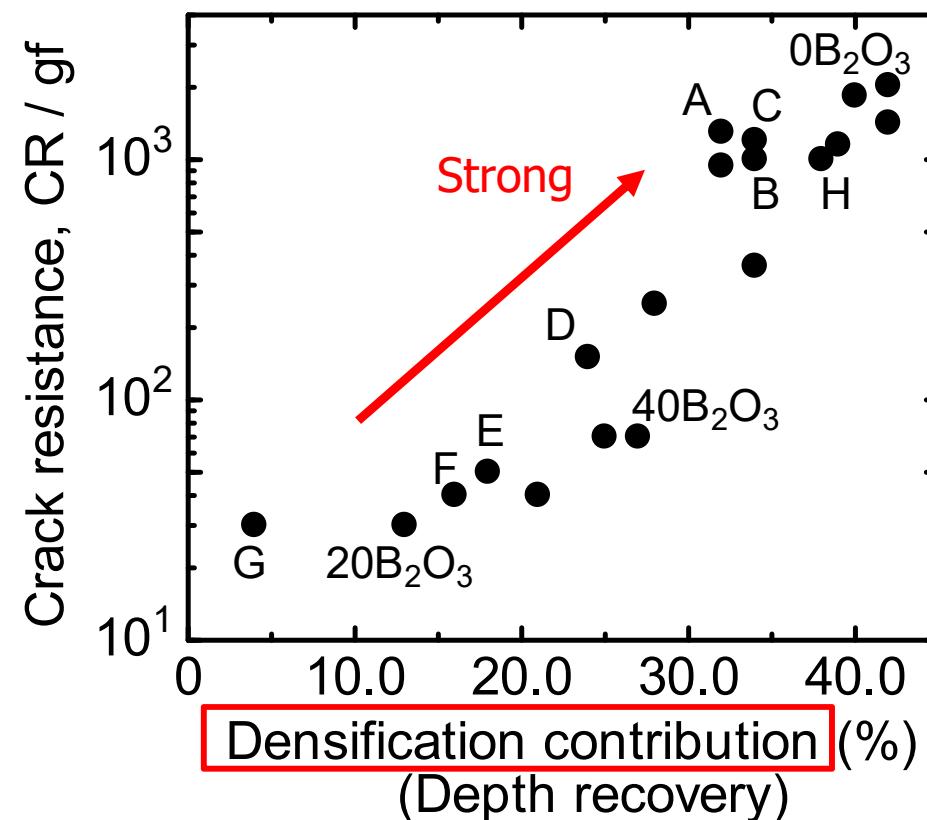
- C. Hermansen *et al.*, *J. Non-Cryst. Solids* **364** (2013) 40.
- P. Sellappan *et al.*, *Acta Materialia* **61** (2013) 5949.
- ▲ S. Yoshida *et al.*, *Phys. Chem. Glasses* **50** (2009) 63.
- S. Yoshida *et al.*, *J. Mater. Res.* **20** (2005) 3404.
- Unpublished data

Densification improves cracking behaviors

, because permanent densification reduces the residual stress.

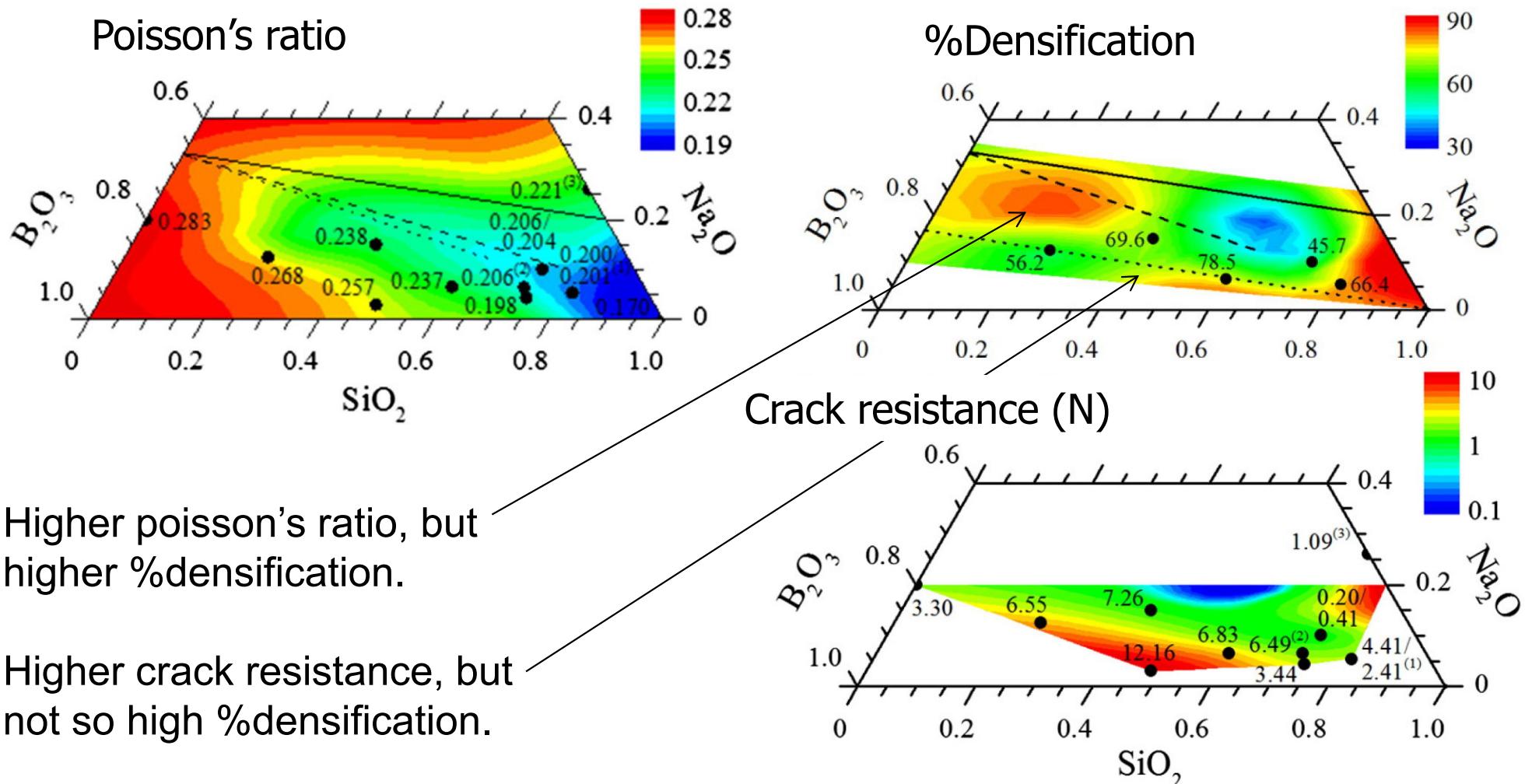


Crack Resistance is the load where the percentage of crack formation is 50 % (2/4 corners).



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

Not only %densification controls fracture.



Phase separation, change in coordination number, and others should be considered.

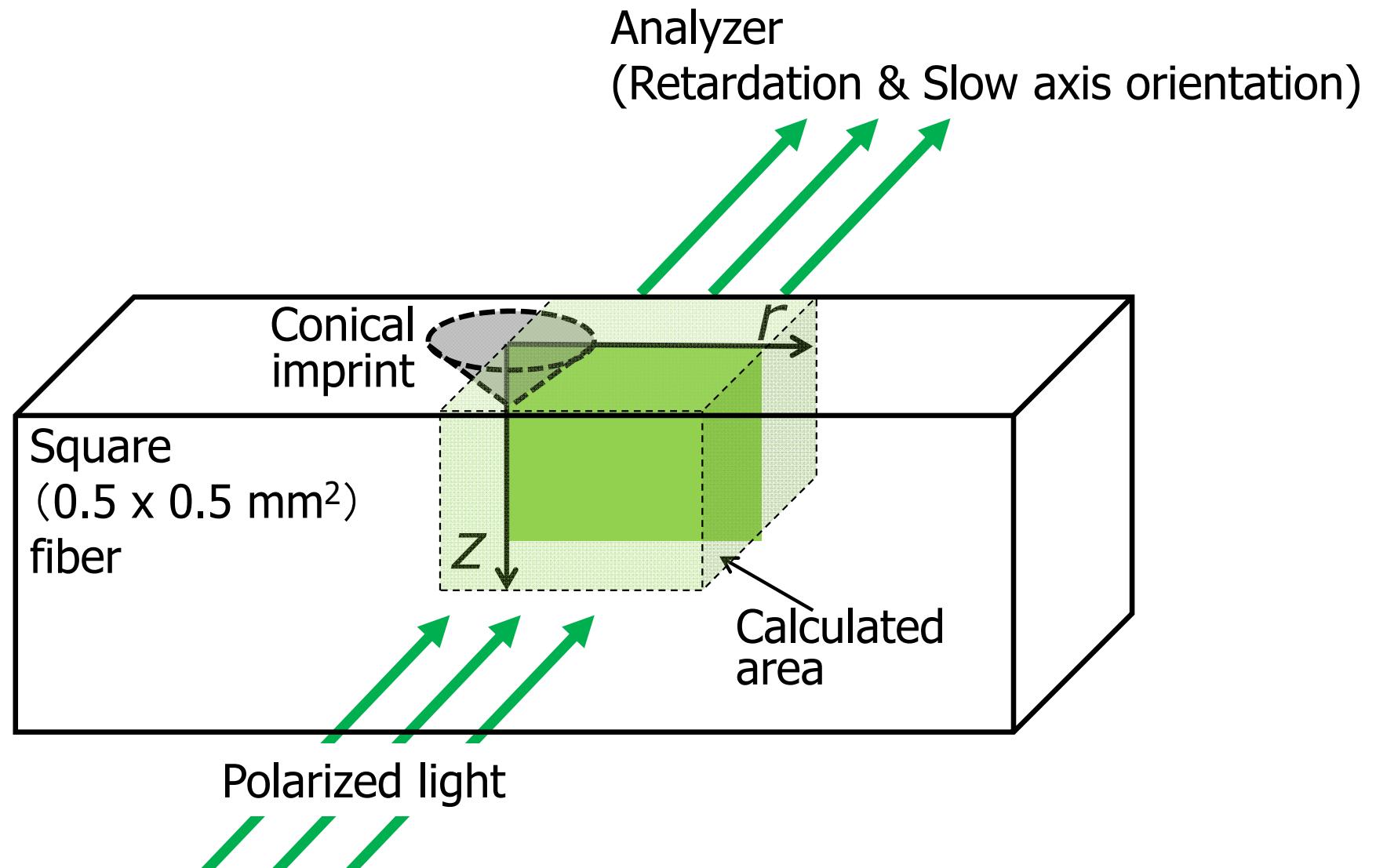
R. Limbach *et al.*, *J. Non-Cryst. Solids* **417-418**(2015)15.

Experimental determination of Residual stress around an indentation imprint

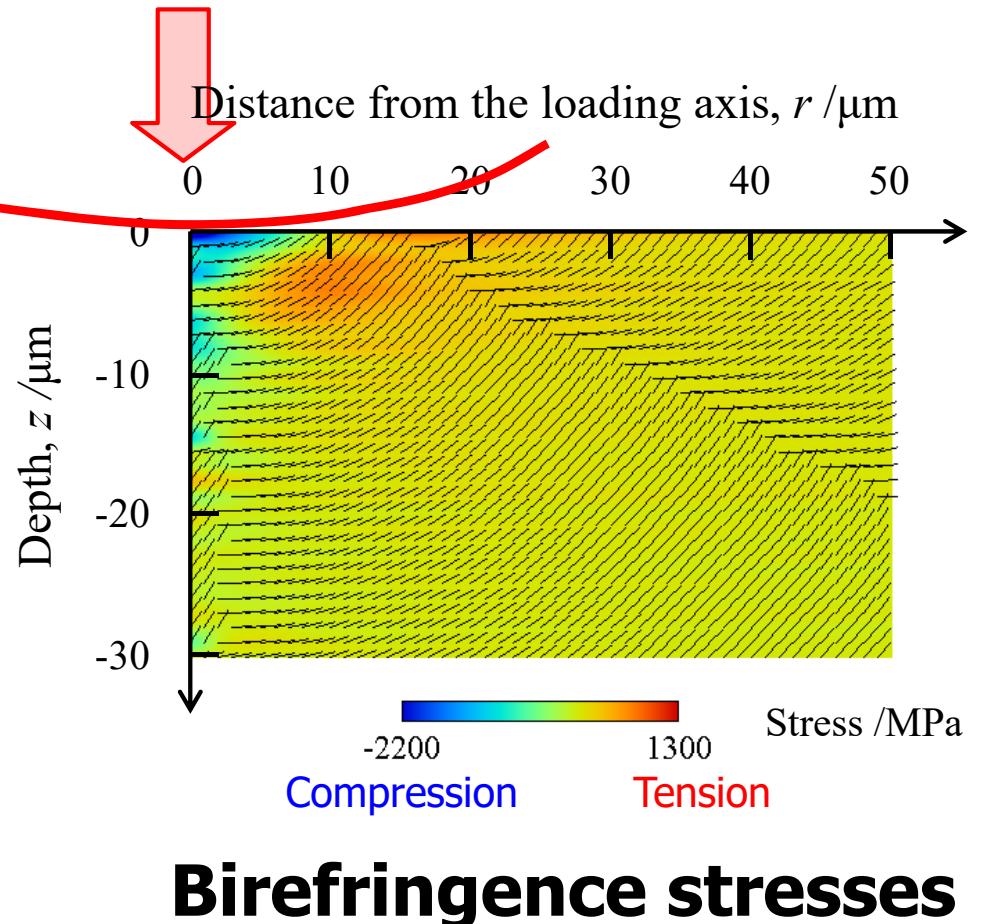
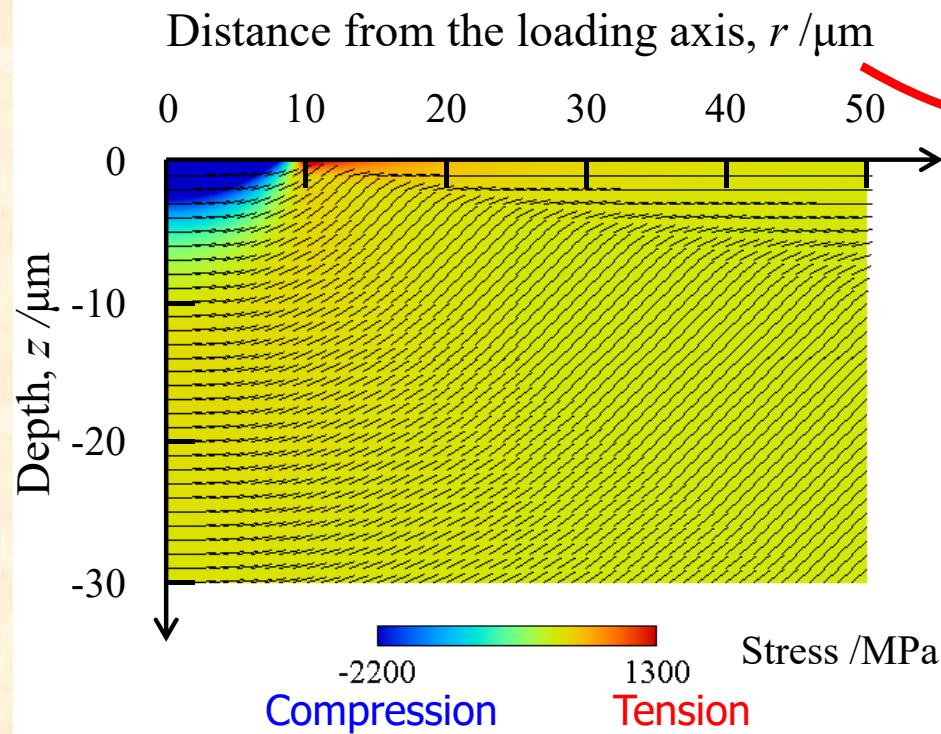
w/ Dr. Chuck Kurkjian and Dr. Andrei Errapart
Dr. Etienne Barthel

Micro-birefringence measurement

to determine residual stresses around an imprint.



Comparison of birefringence stress (σ_1) with Hertz stress

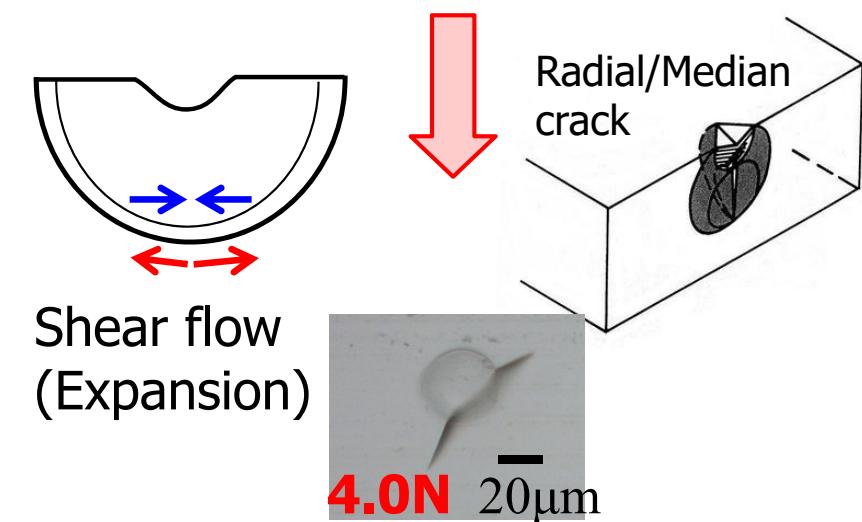
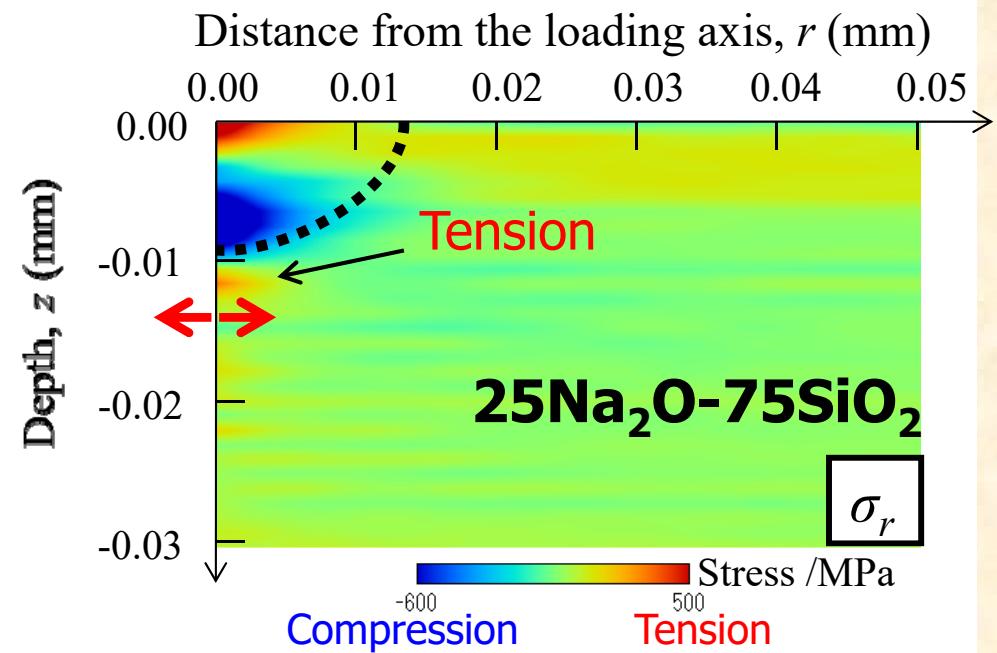
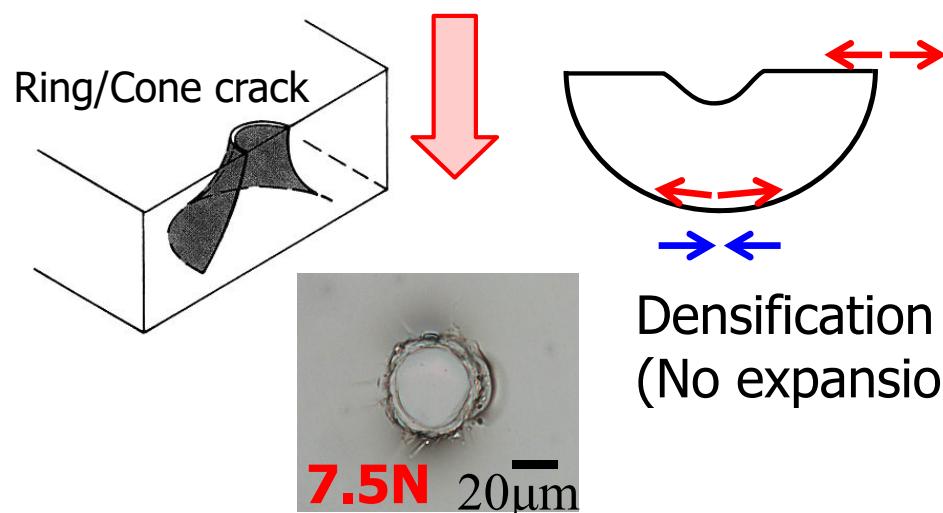
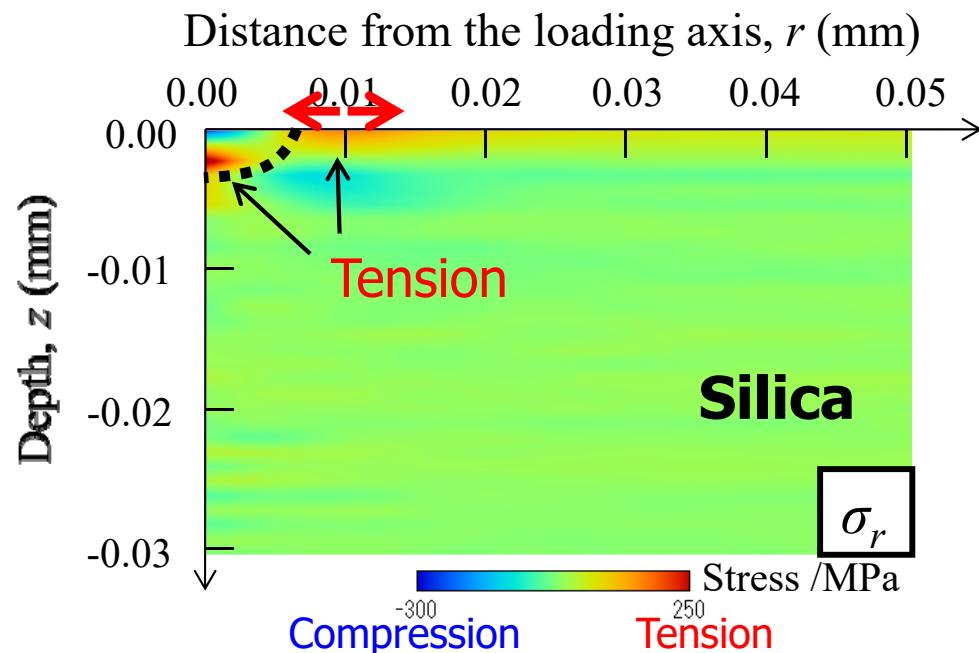


Silica glass under a 0.05R diamond ball (Load = 1.5 N)

Residual stress (σ_r) around imprint

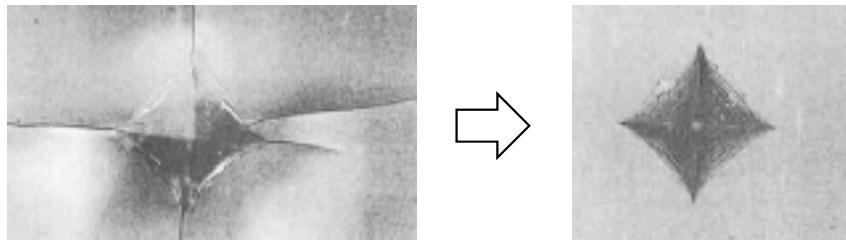
Ball (R=0.05mm)

Max. load = 3.0 N

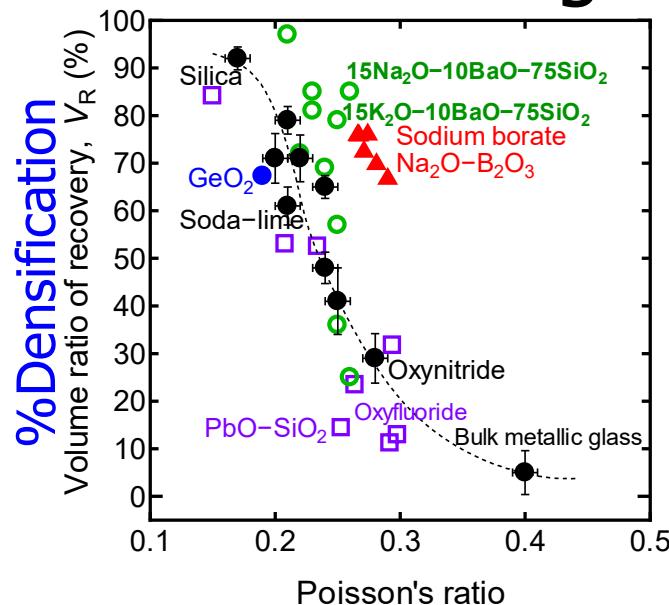


Indentation-induced permanent densification of glass

1. increases Crack Resistance.



2. increases with decreasing Poisson's ratio.



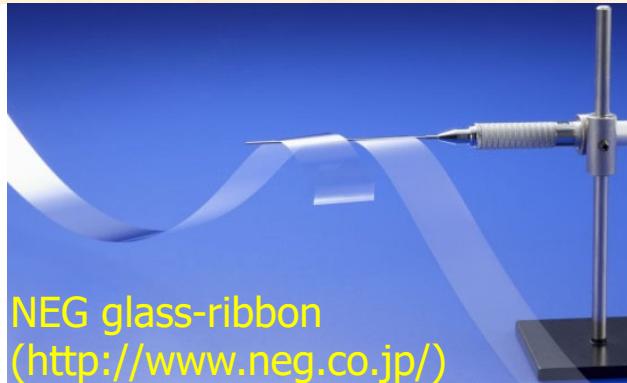
3. is important, but it does not control everything.

How can we design tough glass?

In terms of Plastic deformation in glass, (Elastic deformation and Surface condition are also important.) there are some clues to the problem.
More experimental works and modeling will be required.

1. Densification reduces the residual stress by contact/scratch.
2. Lower Poisson's ratio results in higher %densification.
(Less densely packing) ... but higher elasticity causes easy Hertzian cracking.
3. We should know much more about shear flow of glass.
Change in chemistry, Change in property (density or ..), etc.
Easy flow under a load should also relax the stress.
4. We should know more about structural change of glass under a high stress.
Irreversible coordination change etc.





NEG glass-ribbon
(<http://www.neg.co.jp/>)



Schott AG Xensation®
(<http://www.schott.com/>)



Corning® Willow™
(<http://www.corning.com/>)

Thank you very much for your kind attention.

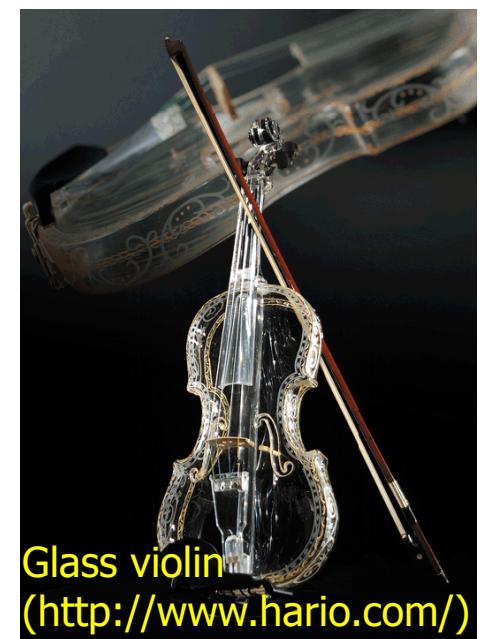
Your flesh ideas are indispensable to design
super-tough glass.



AGC Spool
(<http://www.agc.com/>)



Pilkington Optiwhite™



Glass violin
(<http://www.hario.com/>)

"A step into the space"
(<http://www.compagniedumontblanc.fr/>)