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

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Center for Glass Science and Technology
The University of Shiga Prefecture

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… Relation between cracking and indentation-induced densification

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A blunter indenter results in larger contribution of densification.

1. Background

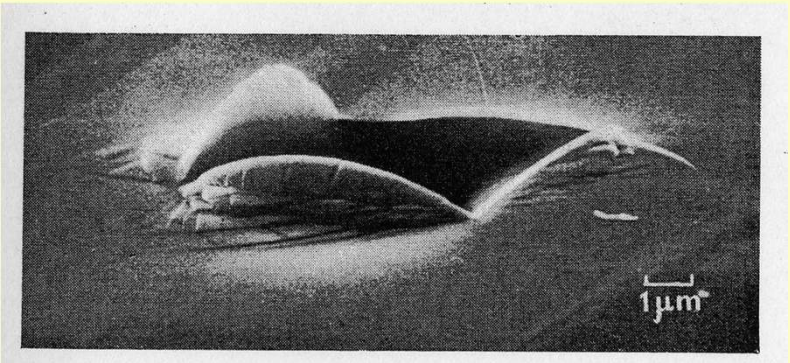
Indentation-induced densification of glass

2. *Experimental procedure*
3. *Results and Discussion*
4. *Summary*

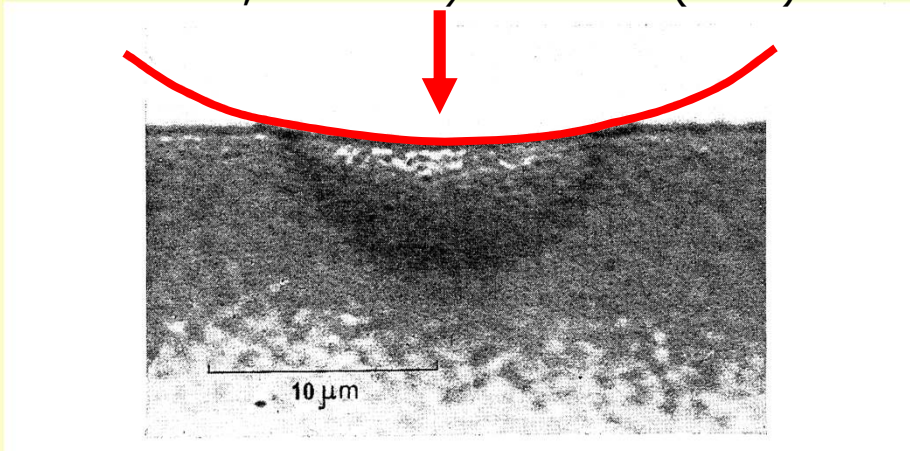
1. Background
Indentation impression on glass

Plastic flow and/or Densification ?

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)

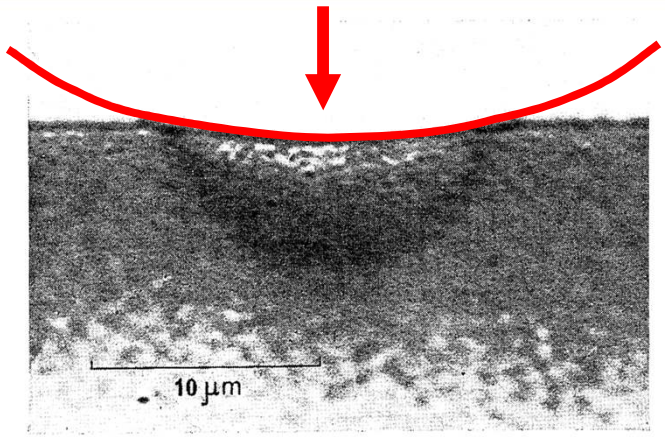
Sharp indenter
Piling-up ! (Shear flow)

Blunt indenter
Densification !

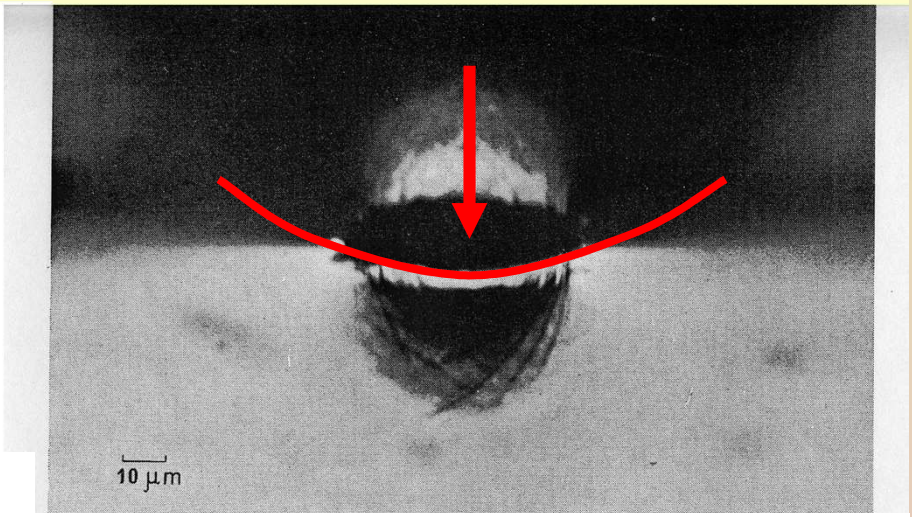
1. Background
Indentation impression on glass

Plastic flow and/or Densification ?

K.W. Peter, *J. Non-Cryst. Solids* 5(1970) 103.



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 !

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1. Background
Indentation impression on glass

Deformation properties by indentation

	Plate glass	SiO_2 74% Na_2O 14% CaO 10%	SiO_2 85% Na_2O 15%	SiO_2 100%
Densification	+	+	+	+
Piling-up	+	+	-	-
Slip(shear) lines below the indentation	+	+	-	-

K.W. Peter, *J. Non-Cryst. Solids* 5(1970) 103.

1. Background
Indentation impression on glass

Deformation properties by indentation

	Plate glass	SiO_2 74% Na_2O 14% CaO 10%	SiO_2 85% Na_2O 15%	SiO_2 100%
Densification				
Piling-up	+	+	-	-
Slip(shear) lines below the indentation	+	+	-	-

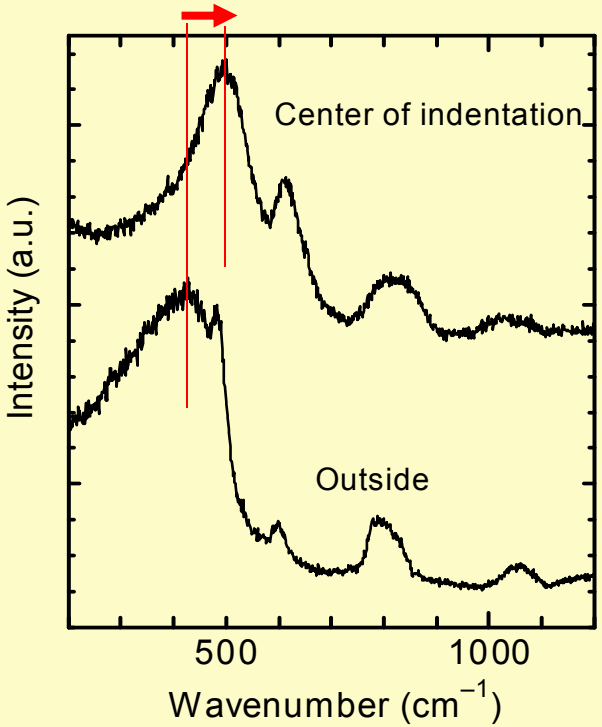
All Glasses can be compacted!

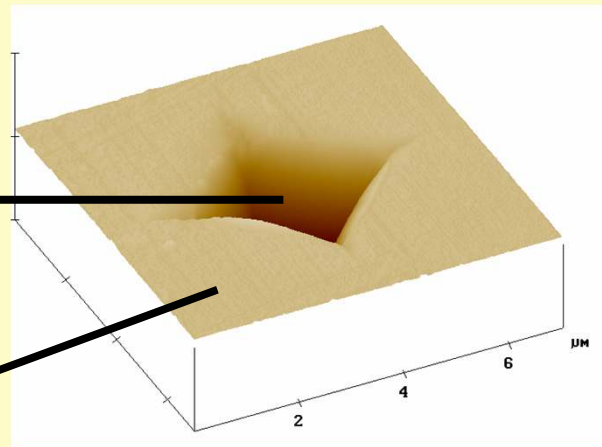
Flow properties are correlated to a minimum percentage of network modifiers.

) 103.

1. Background
Indentation-induced structural change

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





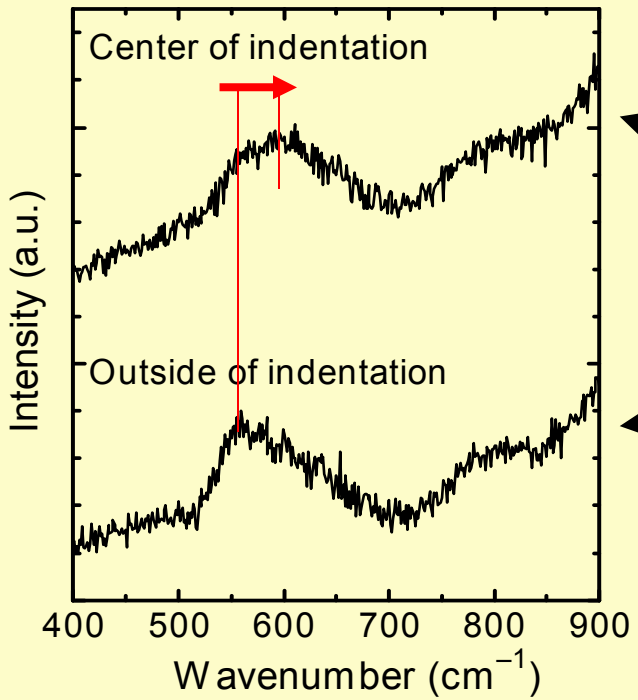
AFM 3D image of Vickers indentation

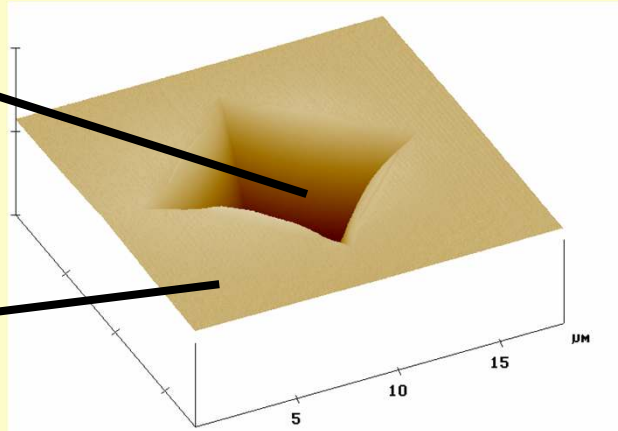
Raman spectra of silica glass

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.





AFM 3D image of Vickers indentation

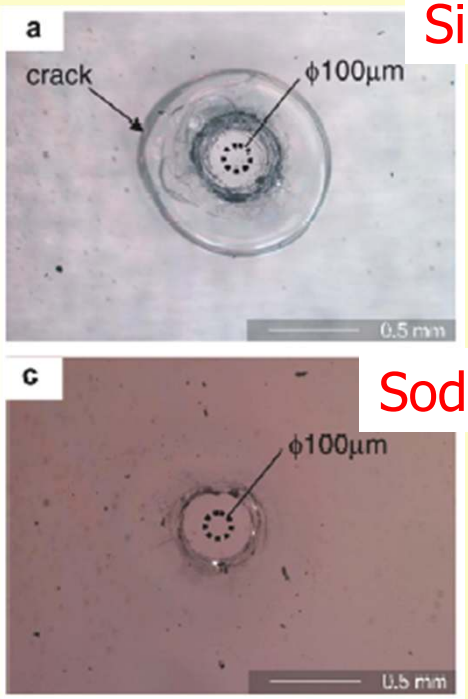
Raman spectra of soda-lime float glass

1. Background
Indentation-induced structural change

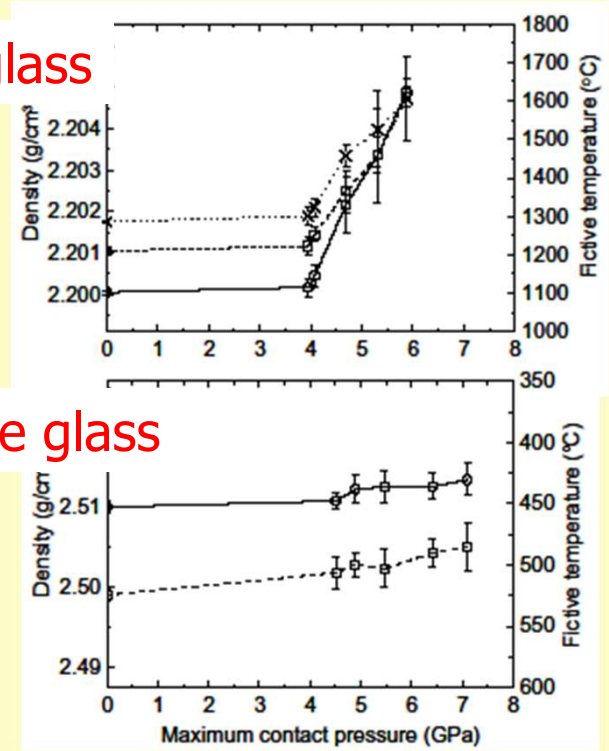
A structural change can be also detected by infrared spectroscopy.

A. Koike & M. Tomozawa, *J. Non-Cryst. Solids* 353(2007) 2318.

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



Photos of ball indentations



Contact pressure dependence of density

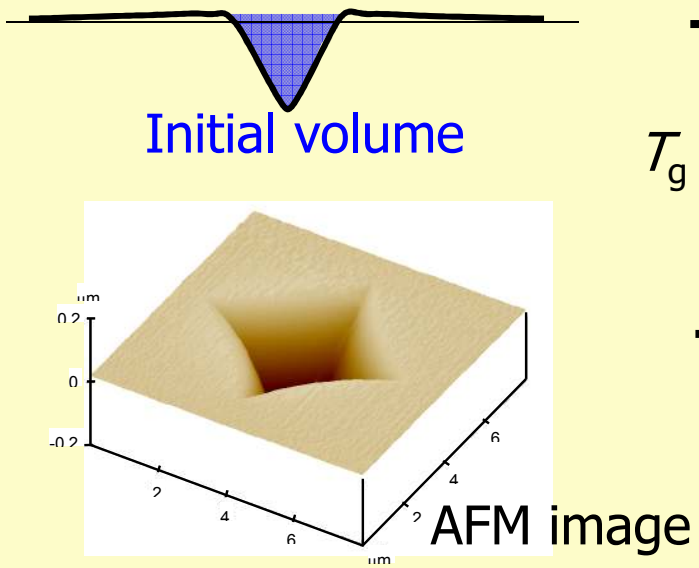
1. Background
Indentation impression on glass

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

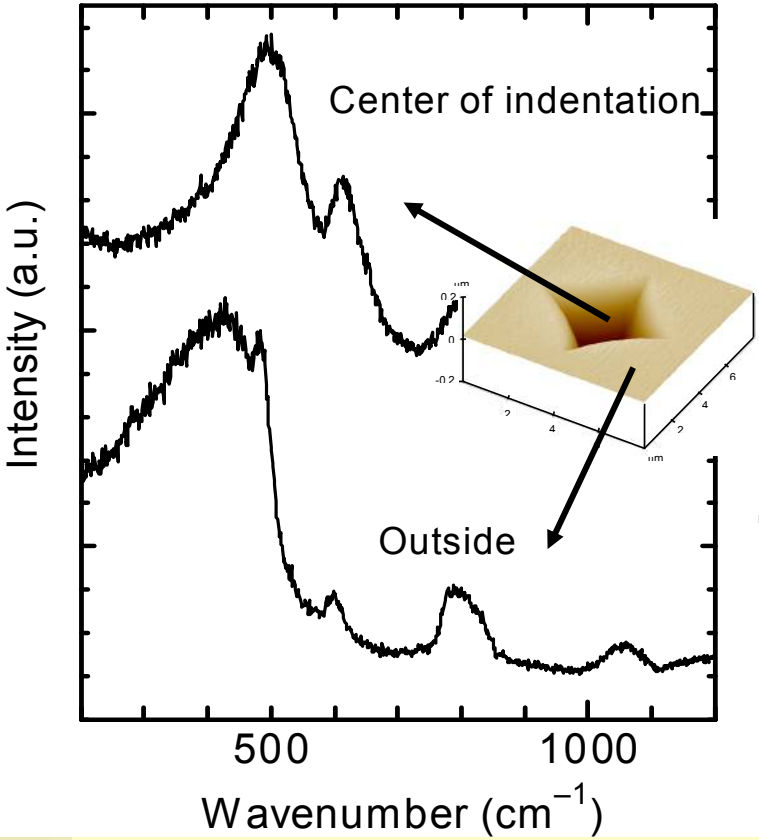
Mackenzie(1963), Neely & Mackenzie(1968), Yoshida (2001, 2005, 2007)



<u>Densification contribution</u> (%) = $\frac{\text{Densified volume}}{\text{Initial volume}}$

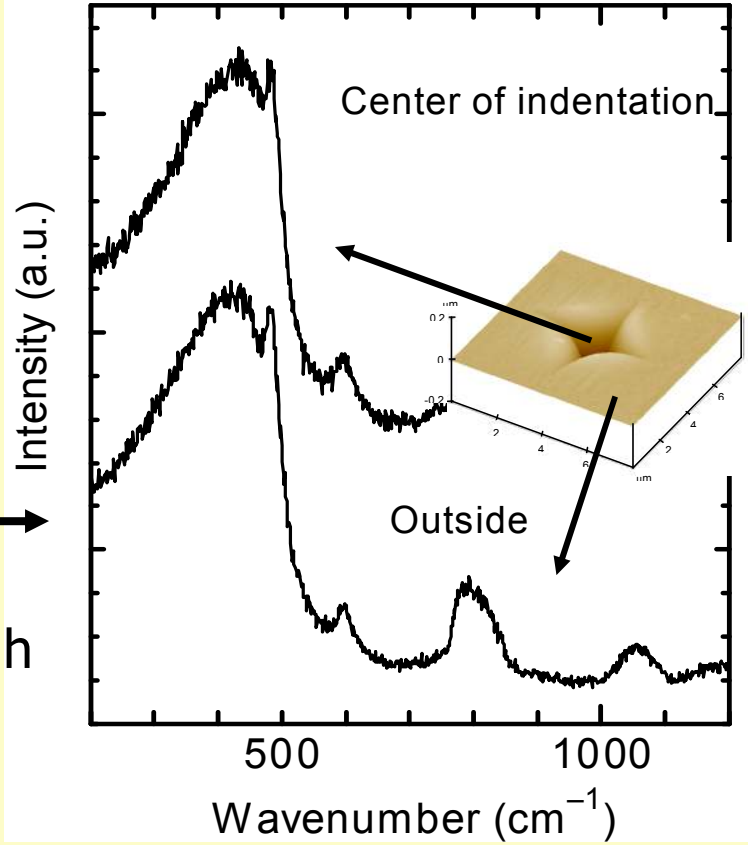
1. Background
Indentation impression on glass

Densified region is completely recovered by annealing.



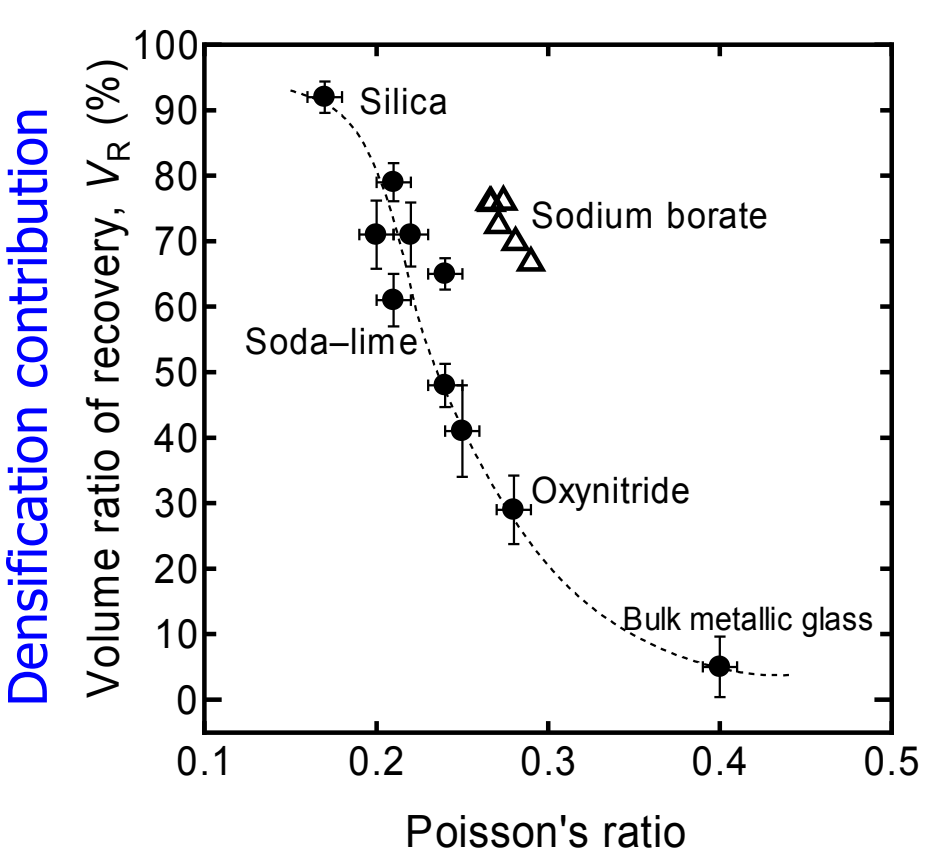
Annealing

T_g x 0.9, 2 h



1. Background
Indentation-induced densification

Densification is a general property for oxide glasses.

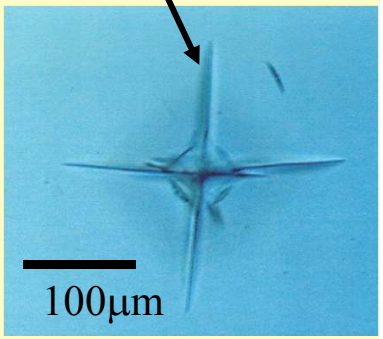


1. Various kinds of glasses experience densification <u>beneath an Vickers indenter.</u>
2. Volume recovery (densified volume) decreases with increasing Poisson's ratio.
3. Sodium borate series show different dependence on Poisson's ratio.

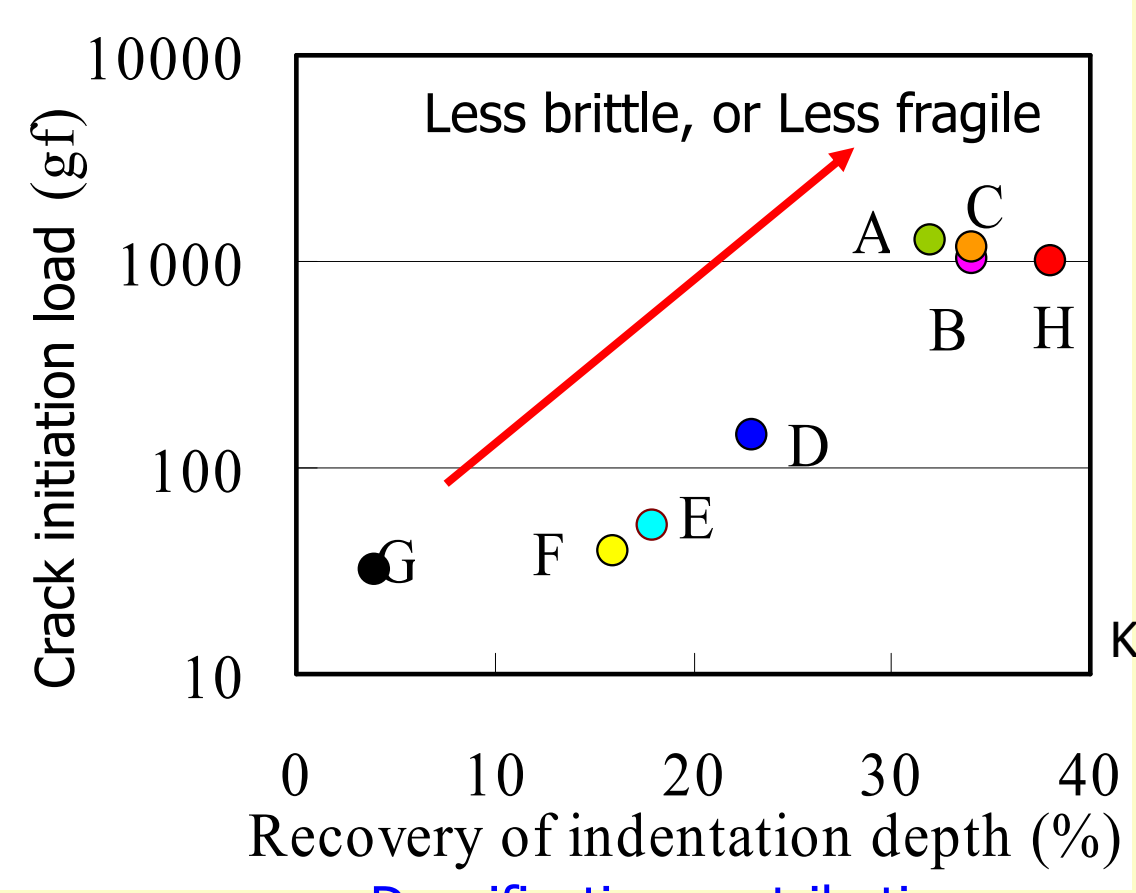
S. Yoshida *et al., J. Mater. Res.,* 20(2005) 3404.
S. Yoshida *et al., Phys. Chem. Glasses*, 50(2009)63.

1. Background
Indentation-induced densification

Densification affects radial cracking of some commercial glasses.



Aluminosilicate glass



Kato(2007)

Densification contribution

1. Background
Indentation impression on glass

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

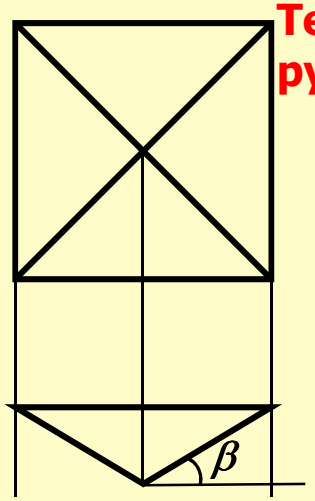
Sample :

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

Vickers indentation test :

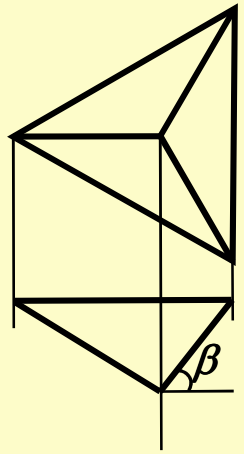
Load = 50 ~ 300 mN, Duration = 15 s, in air

Diamond indenter:



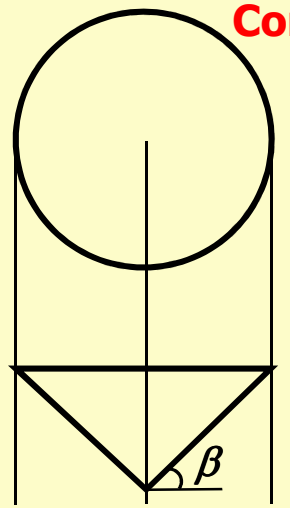
Tetrahedral pyramid

β = 22° (Vickers)



Trihedral pyramid

β = 10°
β = 25° (Berkovich)
β = 55° (Cube-corner)



Cone

β = 20°

2. Experimental procedure

Observation of indentation imprints:

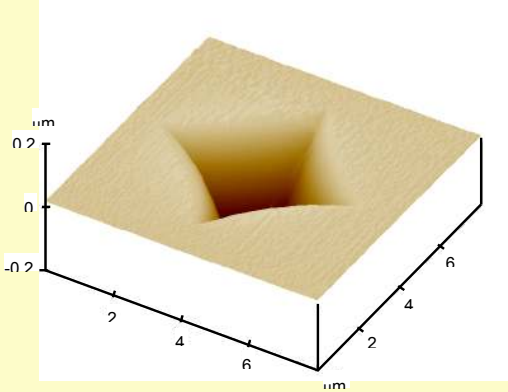
Atomic Force Microscope (AFM, Veeco Nanoscope E)

Contact mode

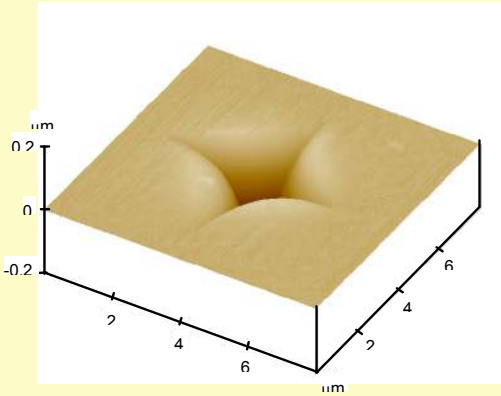
Scan area = ~ 15 μm^2

Annealing conditions :

Temp. = T_g x 0.9 (K), Time = 2h., in air

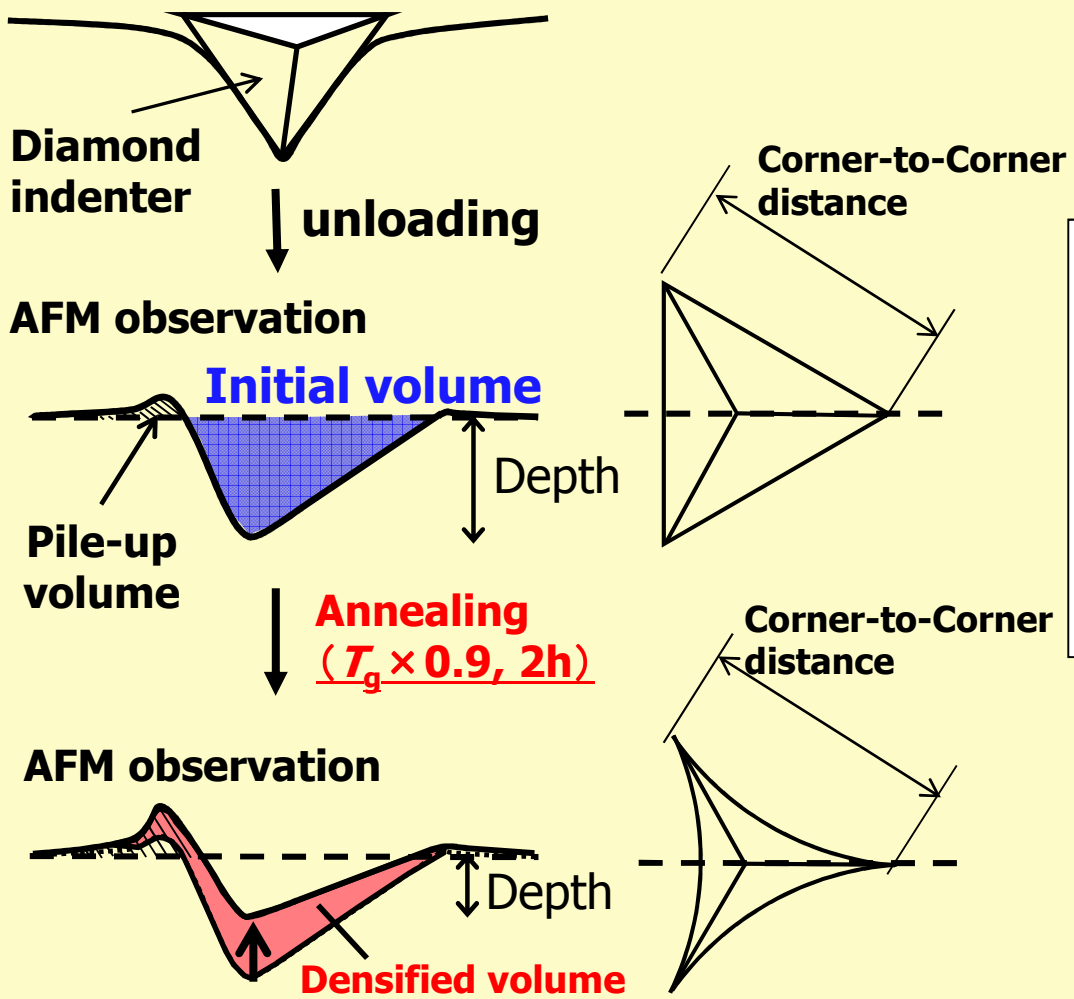


Annealing



2. Experimental procedure

AFM volume measurements before and after annealing



Densification contribution =

Densified volume / Initial volume

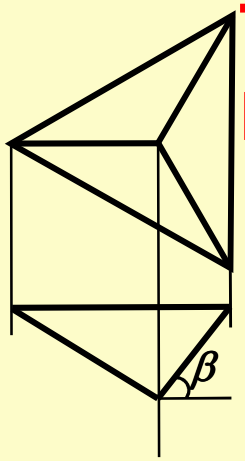
1. *Background*

2. *Experimental procedure*

3. Results and Discussion

4. *Summary*

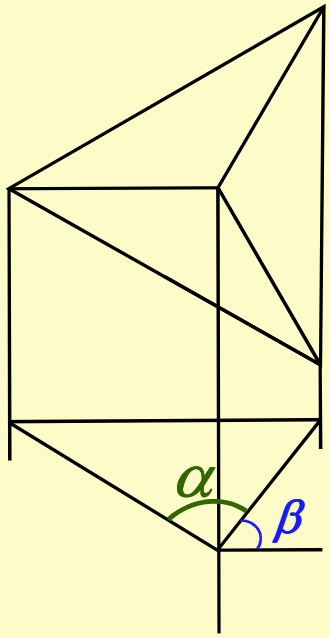
Three types of <u>trihedral</u> pyramids



Trihedral pyramid

β = 10°
β = 25° (Berkovich)
β = 55° (Cube-corner)

Trihedral pyramids



(a) 10 degree

α = **165 °**

β = 10 °

(b) Berkovich

α = **152 °**

β = 24.7 °

(c) Cube-corner

α = **90 °**

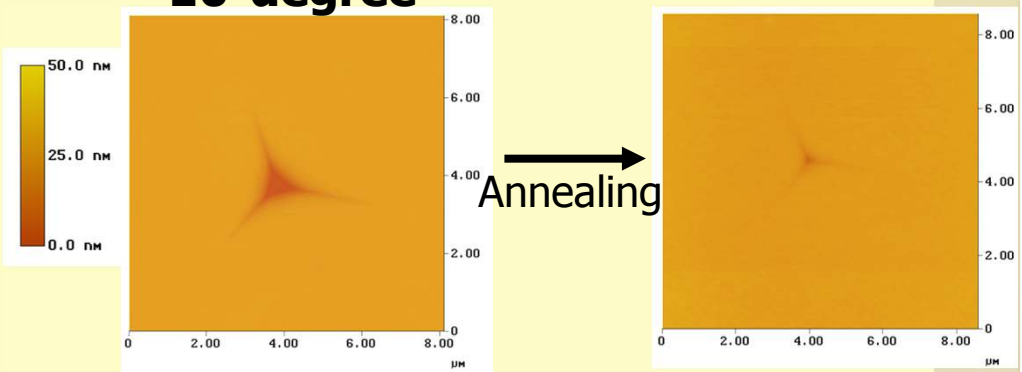
β = 54.7 °

Indentation load
= 50 mN

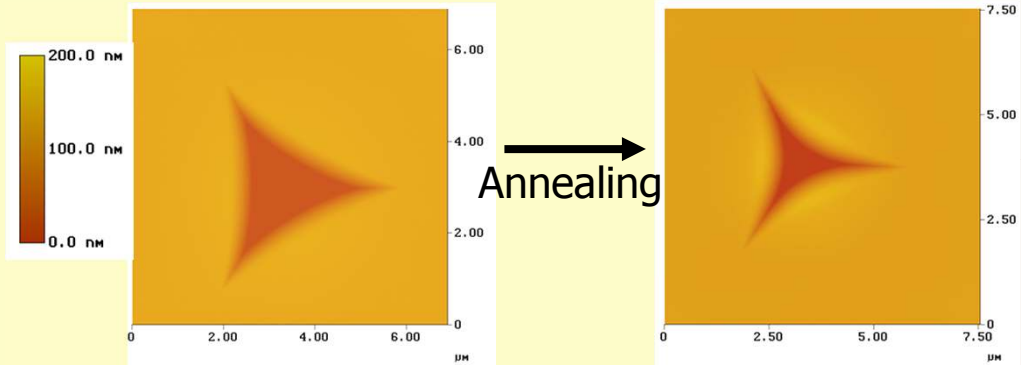
Blunt

Sharp

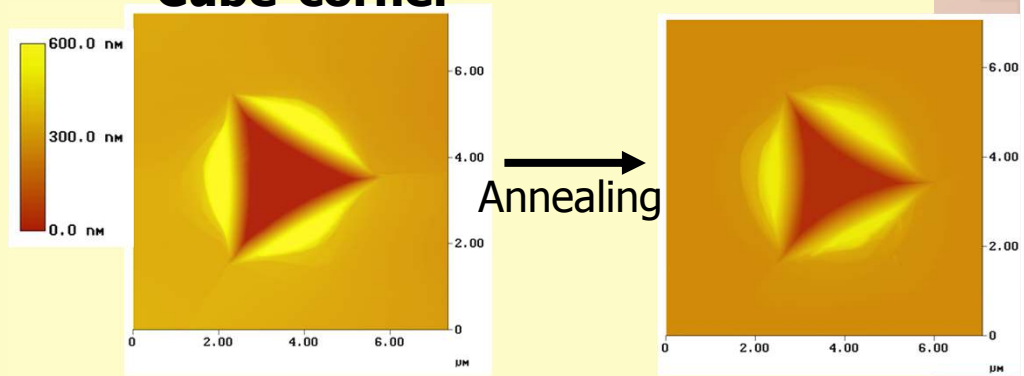
10-degree



Berkovich



Cube-corner

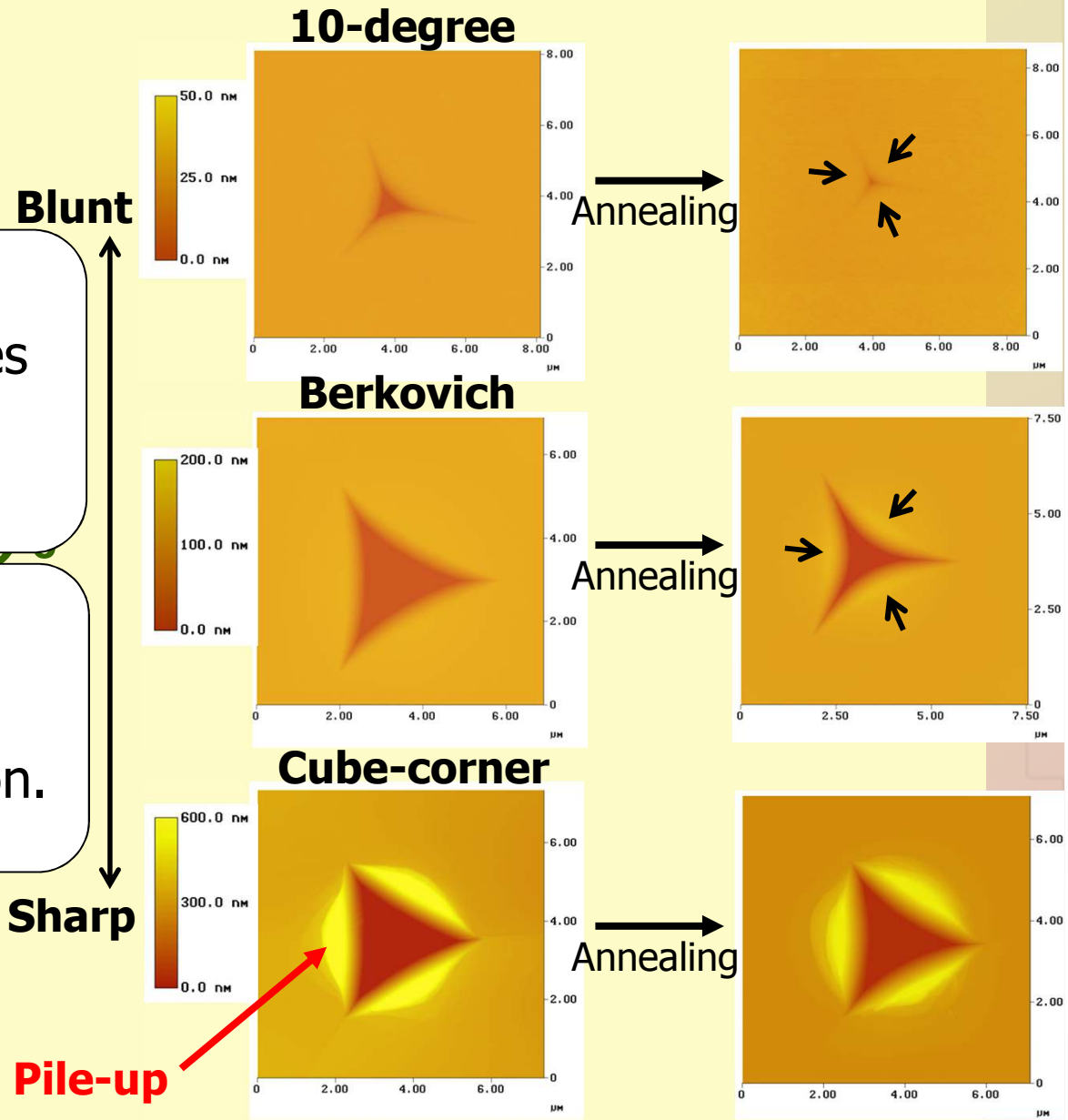


Trihedral pyramids

Annealing recovery is remarkable at the faces of 10-degree and Berkovich indentation.

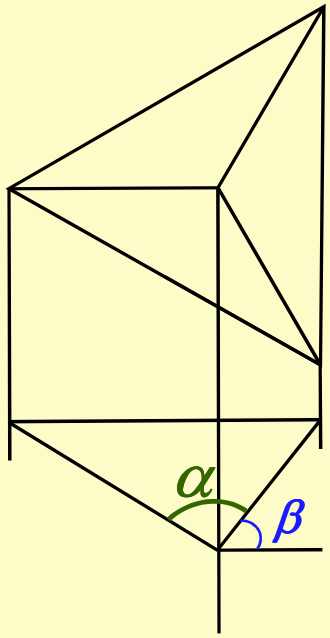
Distinct pile-up is observed around Cube-corner indentation.

Indentation load
= 50 mN



Trihedral pyramids

Trihedral pyramid



(a) 10 degree

α **= 165 º**

β = 10 º

(b) Berkovich

α **= 152 º**

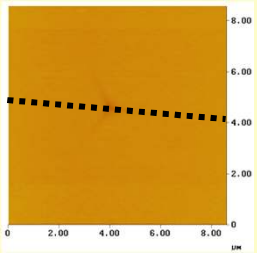
β = 24.7 º

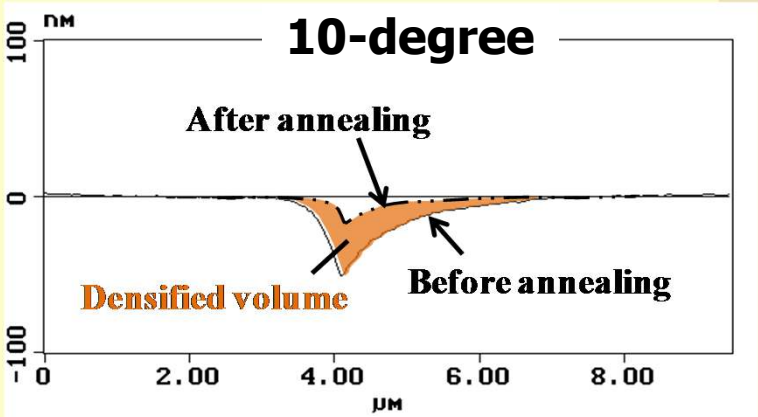
(c) Cube-corner

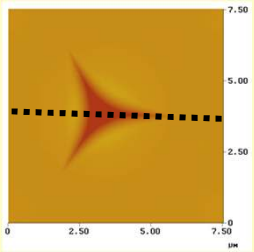
α **= 90 º**

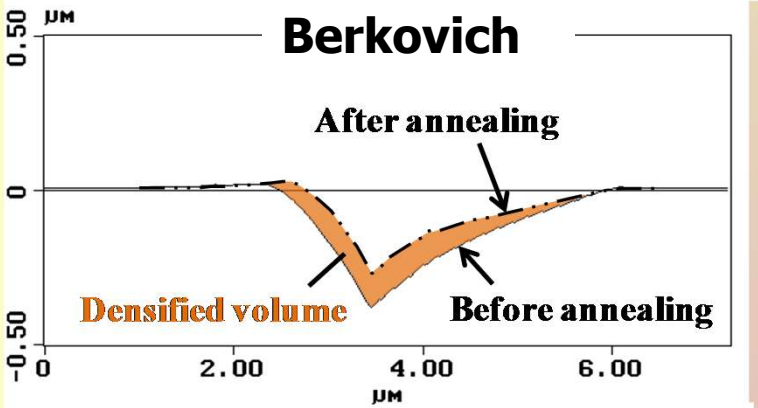
β = 54.7 º

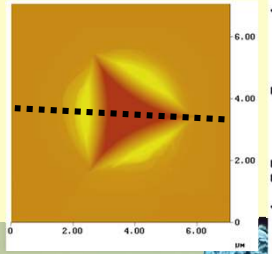
Indentation load
= 50 mN

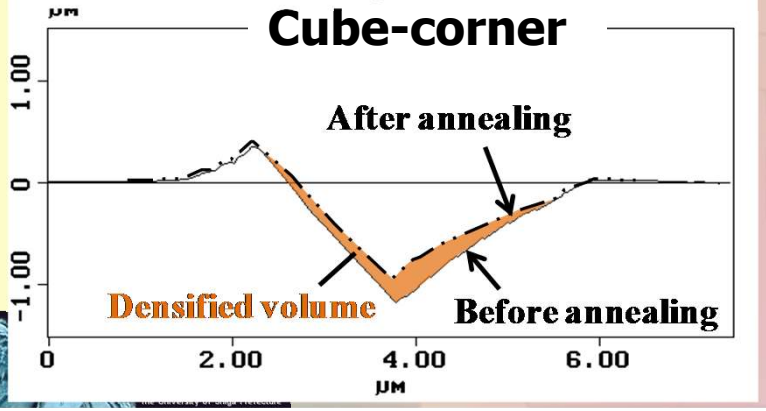






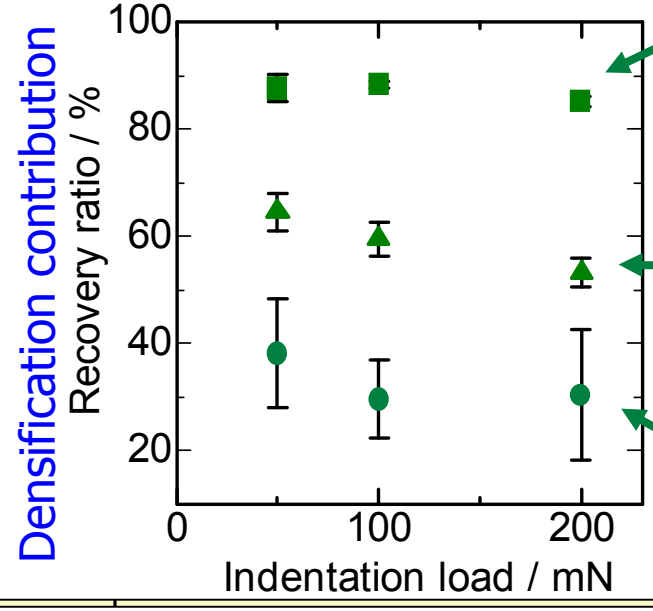


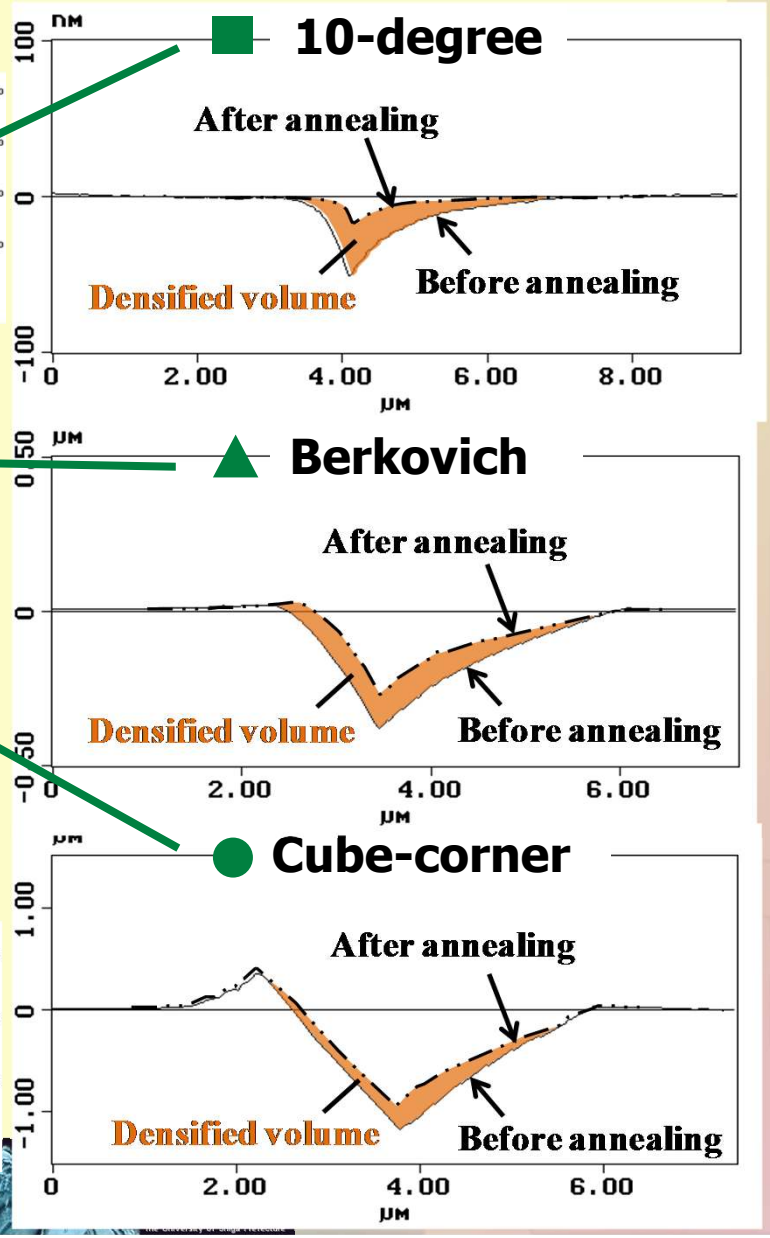




Trihedral pyramids

Load dependence of annealing recovery of volume





Densification contribution

10-degree > Berkovich > Cube-corner

No load dependence for 10-degree

Trihedral pyramid indenters

Change in indentation volume by annealing

Indentation load = 200 mN

	10-degree	Berkovich	Cube-corner
Initial volume (μm^3)	$0.20_{\pm 0.01}$	$4.53_{\pm 0.10}$	$16.05_{\pm 1.3}$
Densified volume (μm^3)	$0.17_{\pm 0.01}$	$2.41_{\pm 0.13}$	$4.86_{\pm 1.4}$
Densification contribution (%)	$85_{\pm 1.0}$	$53_{\pm 2.7}$	$30_{\pm 12}$

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

Densification contribution

10-degree > Berkovich > Cube-corner

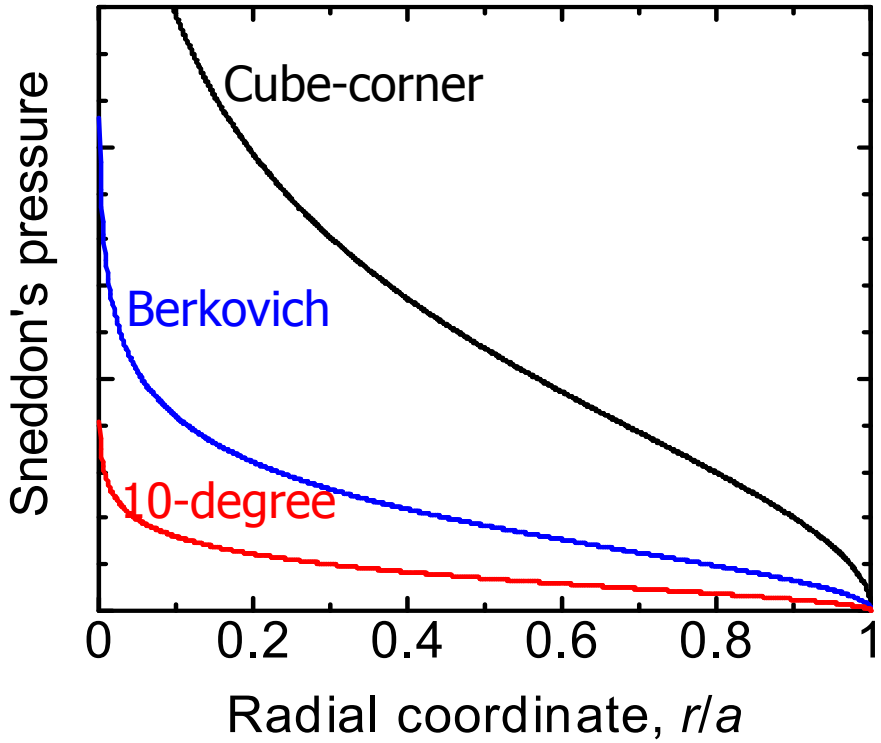
Trihedral pyramid indenters

Sneddon's pressure distribution for an equivalent rigid cone

$$p(r) = \frac{E}{2(1-\nu^2)} \frac{\cosh^{-1}(a/r)}{\tan\Psi} \qquad 0 \le r \le a$$

Sneddon(1965)

E : Young's modulus, ν : Poisson's ratio, a : contact radius,
r : radial coordinate in the surface, Ψ : half included angle



High pressure results in large indentation.

Trihedral pyramid indenters

Sneddon's pressure distribution for an equivalent rigid cone

$$p(r) = \frac{E}{2(1-\nu^2)} \frac{\cosh^{-1}(a/r)}{\tan\Psi} \qquad 0 \le r \le a$$

Sneddon(1965)

E : Young's modulus, ν : Poisson's ratio, a : contact radius,
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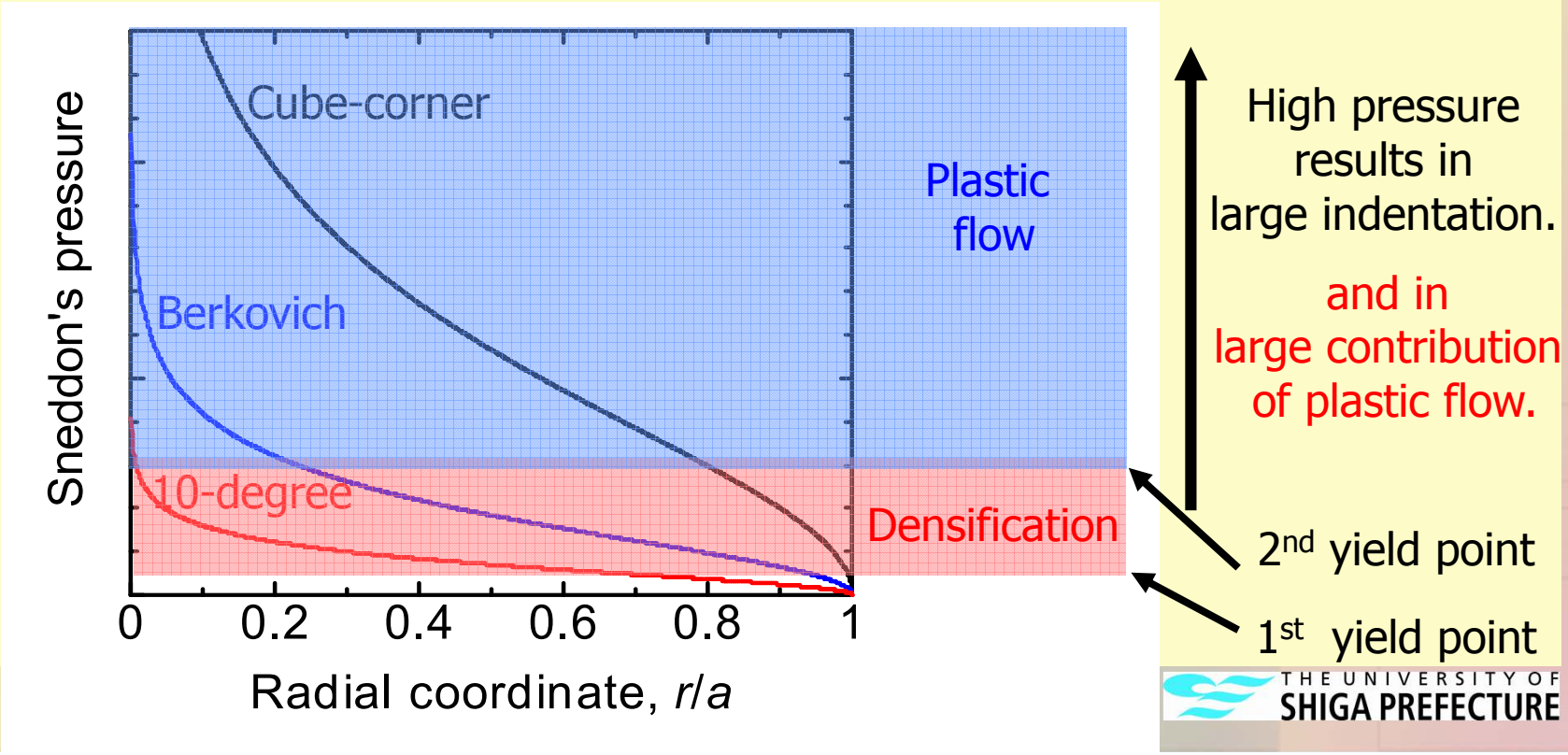

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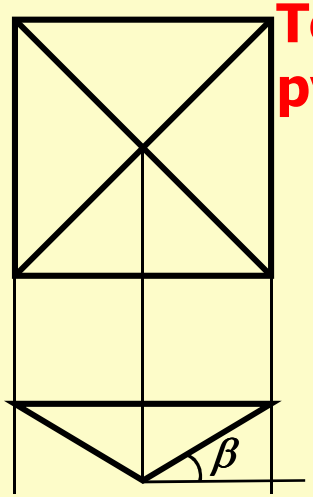
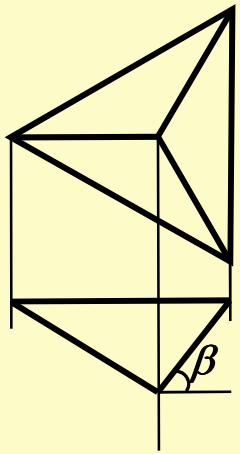
Three types of equivalent indenters

with different number of edges

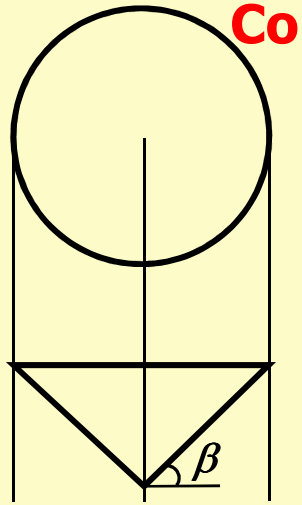
These indenters have the same projected area to depth ratio.



Tetrahedral pyramid



Trihedral pyramid



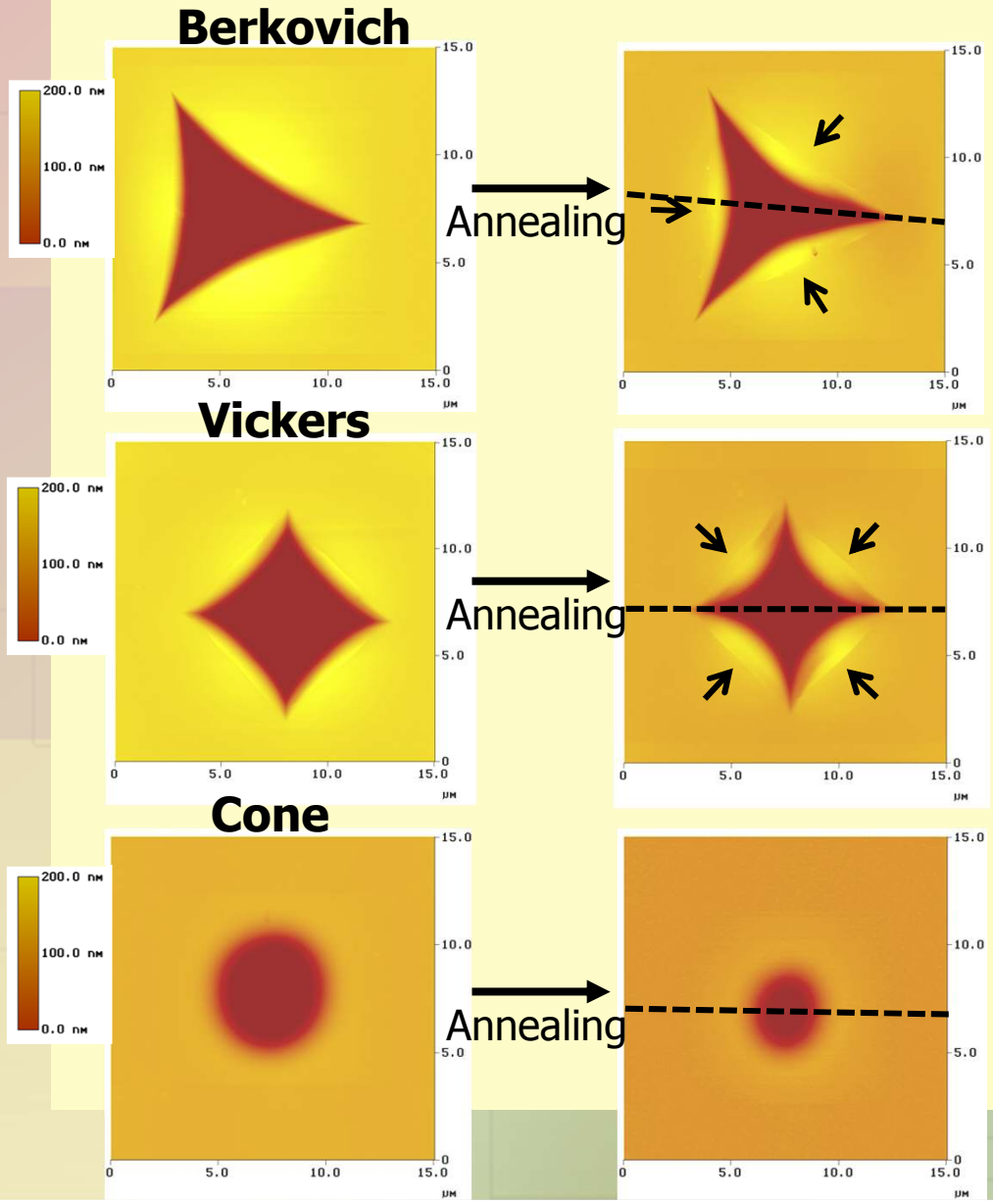
Cone

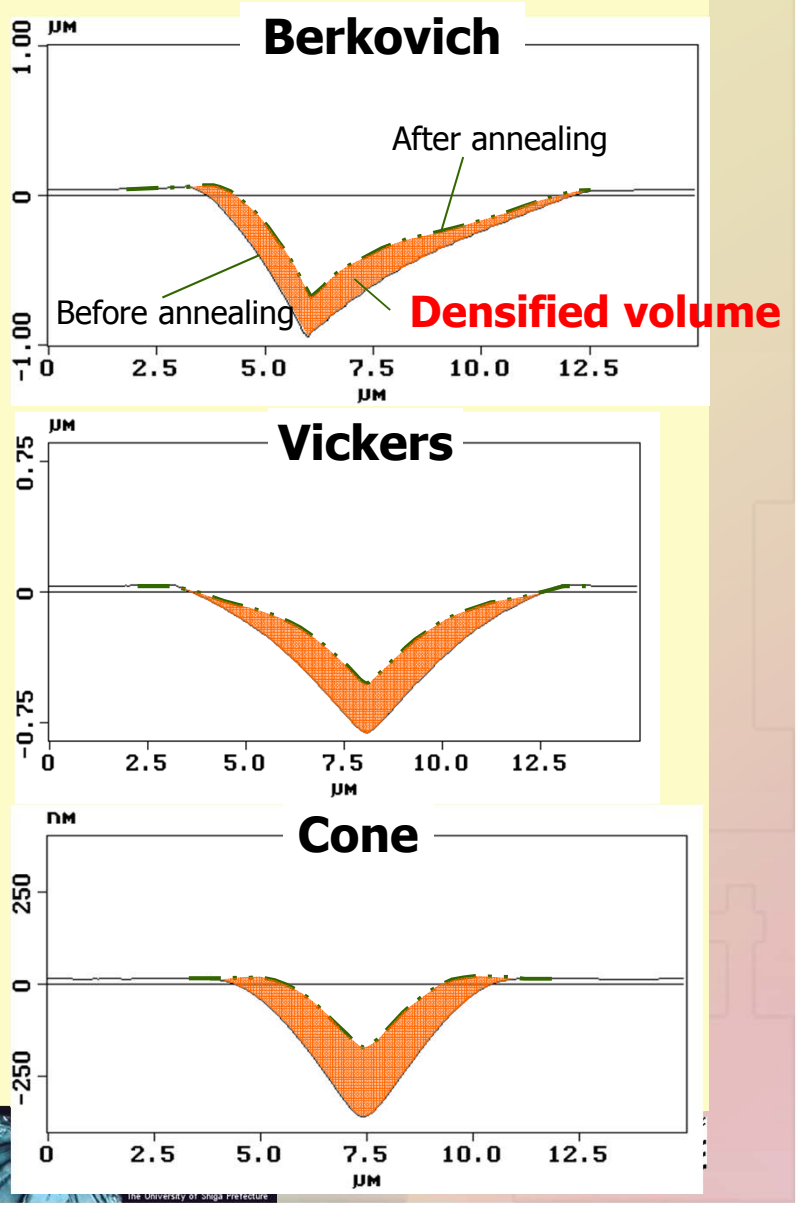
β = 22° (Vickers)

β = 25° (Berkovich)

β = 20°

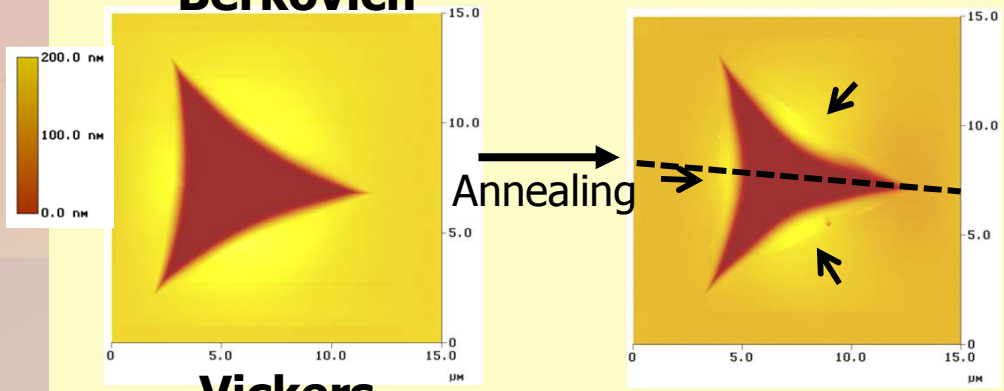
Equivalent indenters

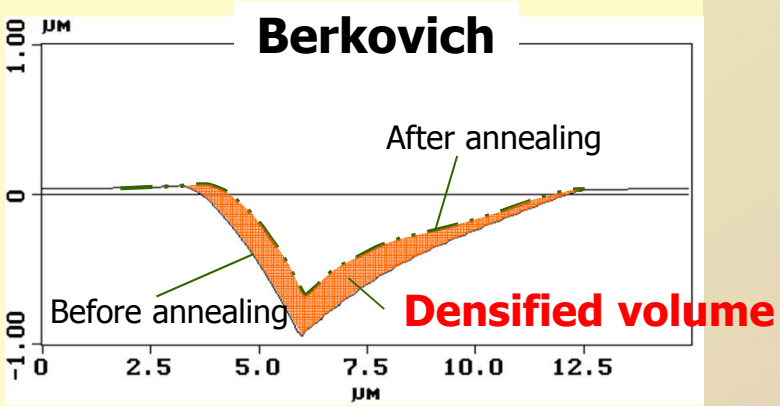




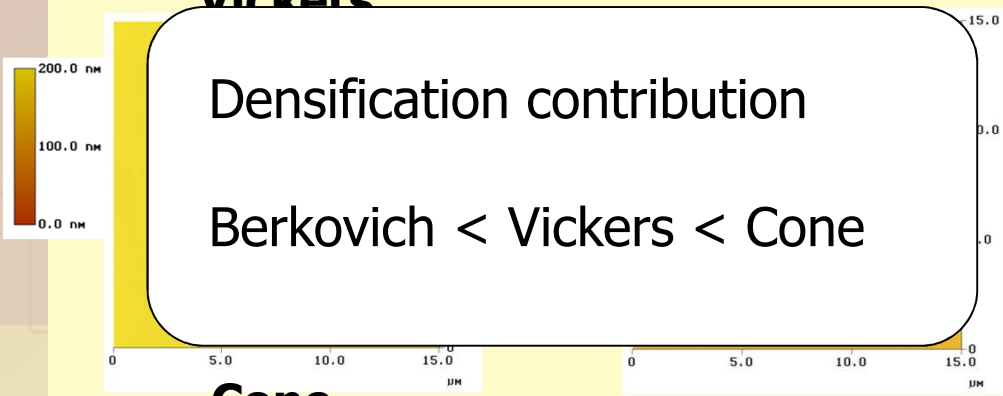
Equivalent indenters

Berkovich



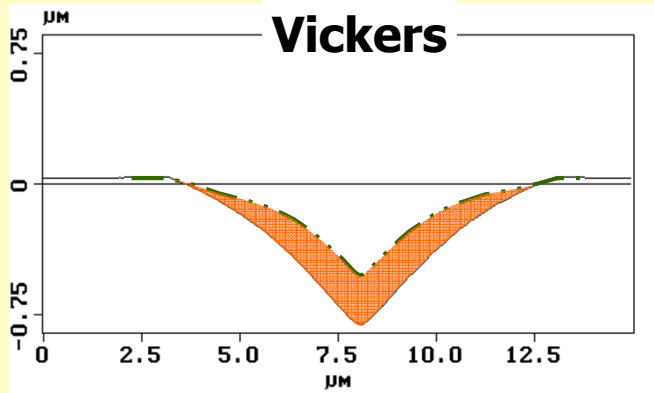


Vickers

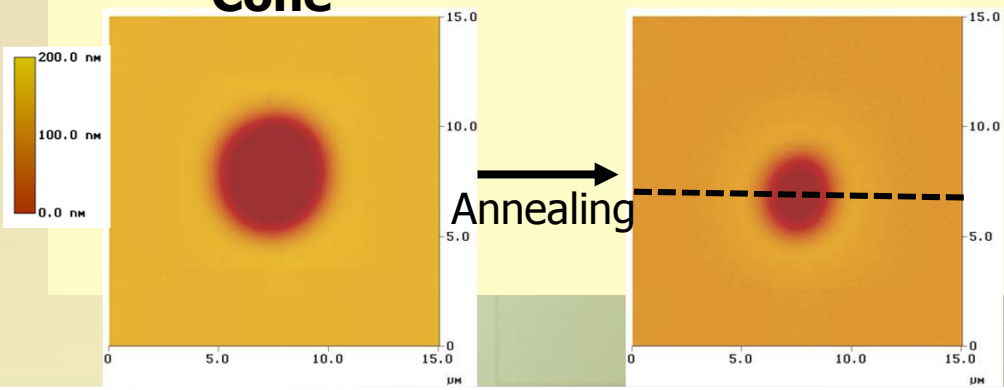


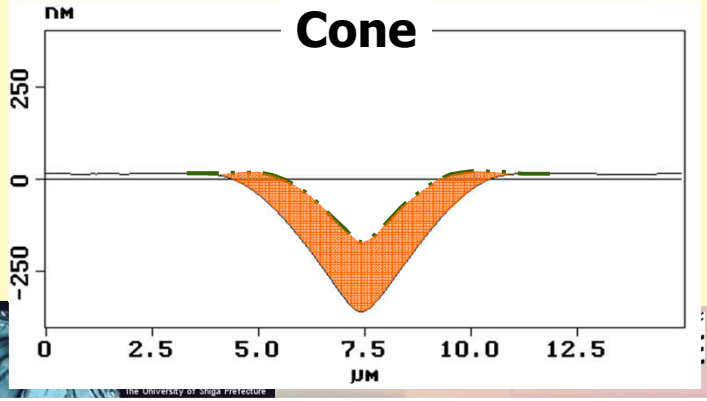
Densification contribution

Berkovich < Vickers < Cone



Cone





Equivalent indenters

Change in indentation volume by annealing

Indentation load = 300 mN

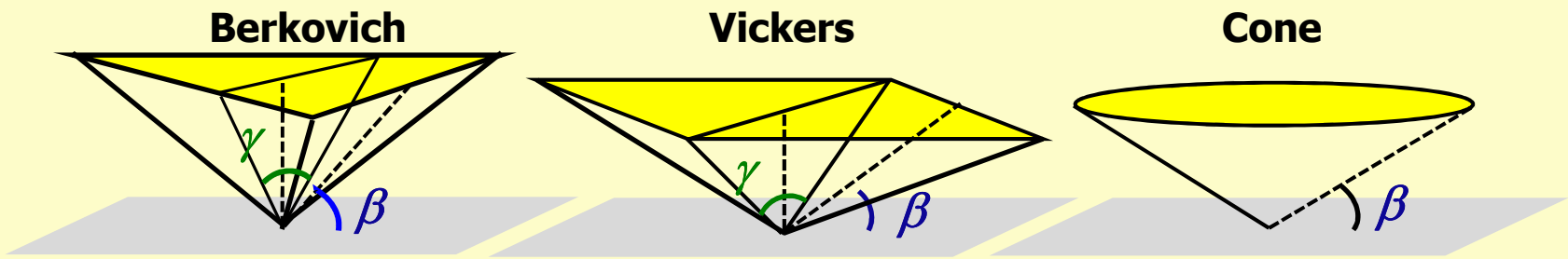
	Berkovich	Vickers	Cone
Initial volume (μm^3)	9.81±0.31	8.30±0. 11	3.77±0.15
Densified volume (μm^3)	5.29±0.54	5.28±0.15	3.05±0.18
Densification contribution (%)	54±2.8	64±1.4	81±1.6

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

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

Equivalent indenters

	Berkovich	Vickers	Cone
Initial volume (μm^3)	$9.81_{\pm 0.31}$	$8.30_{\pm 0.11}$	$3.77_{\pm 0.15}$
Densified volume (μm^3)	$5.29_{\pm 0.54}$	$5.28_{\pm 0.15}$	$3.05_{\pm 0.18}$
Densification contribution (%)	$54_{\pm 2.8}$	$64_{\pm 1.4}$	$81_{\pm 1.6}$



γ = 136° β = 25° γ = 148° β = 22° β = 20°

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.