SAND2007-6001C

# Measuring Residual Stress in Glasses and Ceramics using Instrumented Indentation

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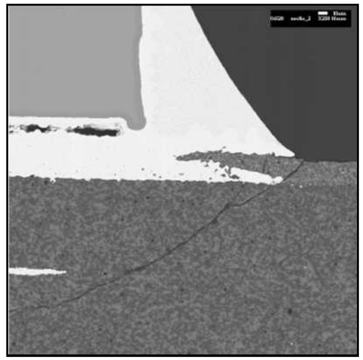
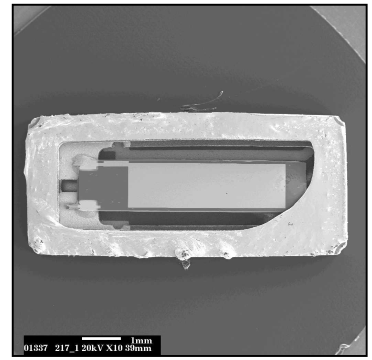
*Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-ACO4-94AL85000.*

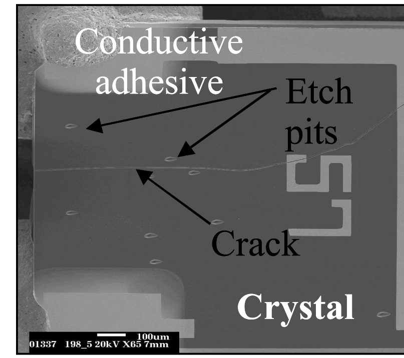
# Problem: Processing and thermally induced residual stresses lead to failure of ceramic-based microsystems components

Examples of cracks in LTCC ceramic substrates

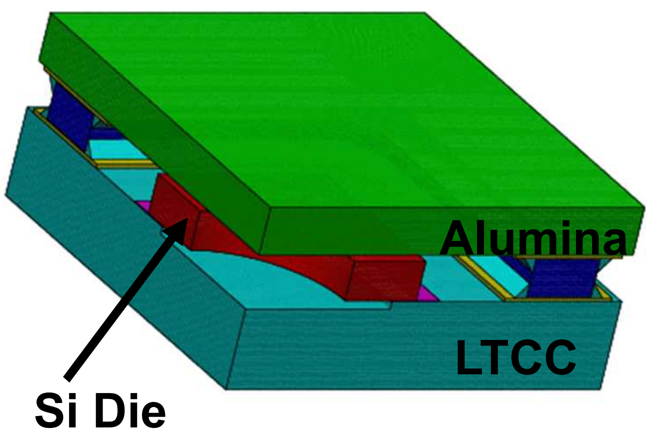


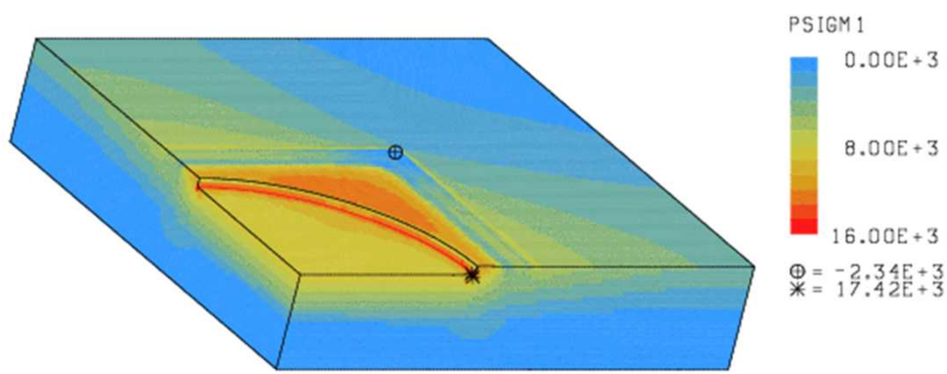
Quartz AT Strip SAW Device






FE simulation of Die Attached Microsystems assembly



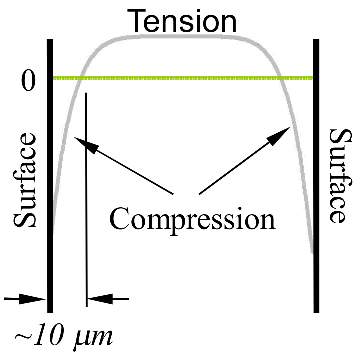



# Can nanoindentation be used as a method to measure residual stress in brittle materials?

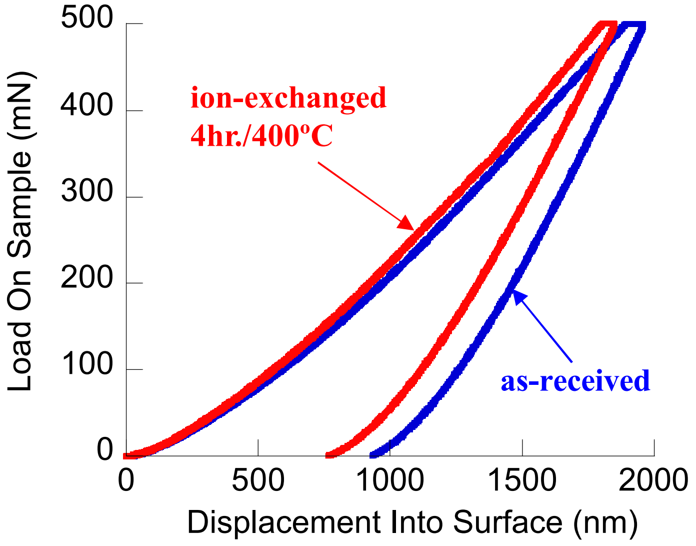
*An initial observation:*

- **A very noticeable and repeatable difference in measured indentation response between unstressed and stressed glass.**

**Stress Profile in Ion-Exchanged Glass**




**PPG Glass – 500 mN load spherical tip- 10 um nominal radius**



# Selected Previous Investigation of residual stress measurement using instrumented indentation

*All previous investigations rely on a measurable difference in the indentation load-displacement response between stressed and unstressed materials which are otherwise identical*

- Suresh, S. and Giannakopoulos, A.E., "A new method for estimating residual stress by instrumented sharp indentation", Acta mater., vol. 46, no. 16, p. 5755 (1998).
  - Attributes measurable differences in the indentation load-displacement response to the indentation contact area.
  - Attempts to elicit values of the residual stress from differences in load in depth-controlled experiments

- Xu, Z.H., and Li. X. "Influence of equi-biaxial residual stress on unloading behavior on nanoindentation", Acta Materialia, vol. 53, p. 1913 (2005).
  - empircally-based model using unloading portion of indentation load-displacement curve

- Swadener, J.G., Taljat, B., and Pharr, G.M., "Measurement of residual Stress by load and depth sensing indentation with spherical indenters", J. Mater. Res., vol. 16, no. 7, p. 2091 (2001).
  - Uses spherical cavity model to isolate elastic-plastic testing regime as optimal for sensing residual stress in substrate materials using instrumented indentation
  - Demonstrates that elastic residual stress cannot be sensed by instrumented indenation when “full” plasticity is achieved in the substrate during the experiment.

**$E/\sigma y \approx 25$ in glass, $E/\sigma y \approx 150$ or greater in most metals.**

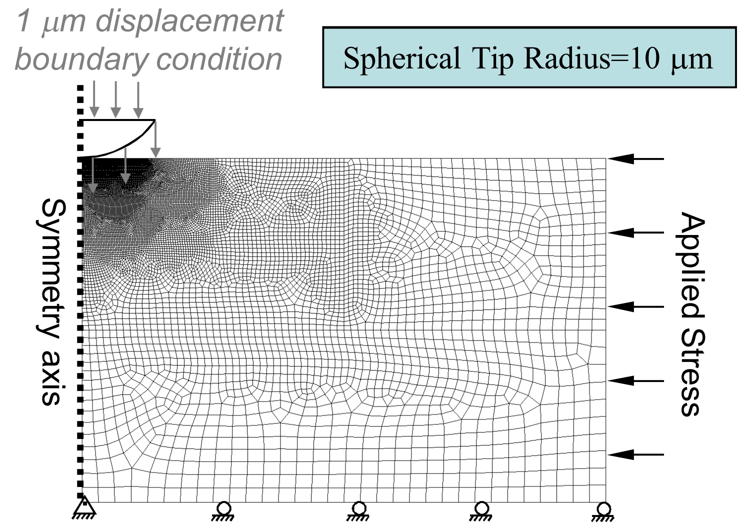
# A series of finite element simulations were performed to investigate the role residual stress on the indentation response

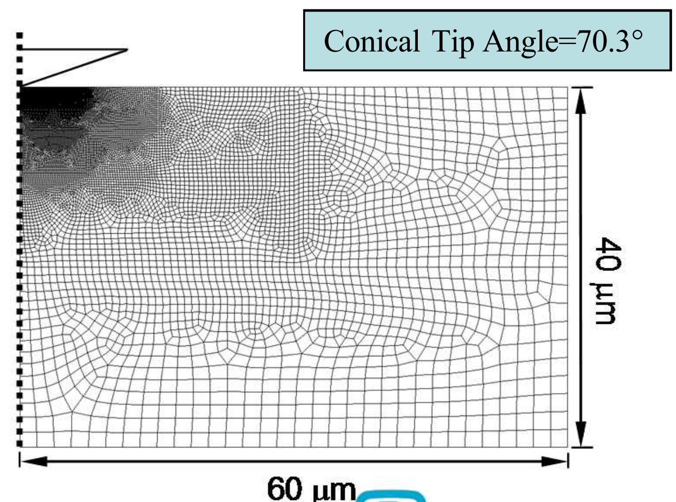
- 8 simulations- displacement control to 1 um depth

| material properties | tip geometry | residual stress |
|---|---|---|
| E=72 GPa<br>$\sigma_y$=3 GPa | spherical-<br>10 μm radius | 1) none<br>2) -500 MPa |
| E=72 GPa<br>$\sigma_y$=3 GPa | conical-<br>70.3º half-angle | 3) none<br>4) -500 MPa |
| E=72 GPa<br>$\sigma_y$=600 MPa | spherical-<br>10 μm radius | 5) none<br>6) -500MPa |
| E=72 GPa<br>$\sigma_y$=600 MPa | conical-<br>70.3º half-angle | 5) none<br>6) -500MPa |

**Simulation Details:**
- frictionless
- *rigid tip*
- substrate 30x40 μm –fixed
- approx. 20,000 elements
- axisymmetric elements

- FE meshes and boundary conditions



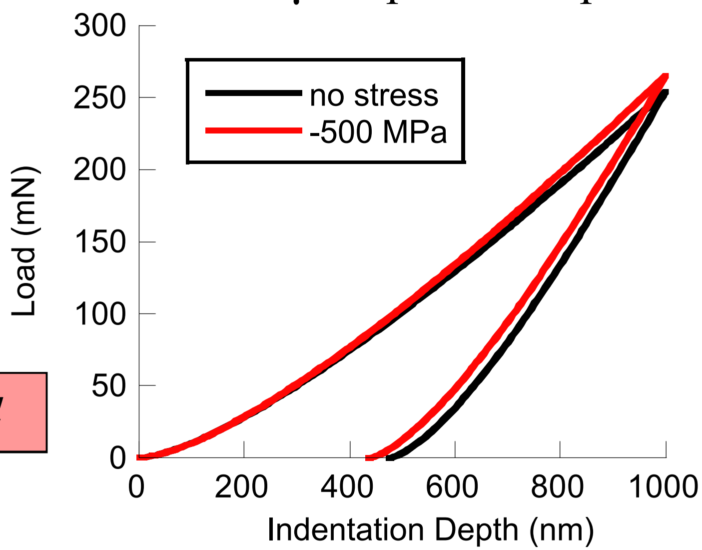
# Stressed substrates gave a significantly different load-displacement response in every simulated case

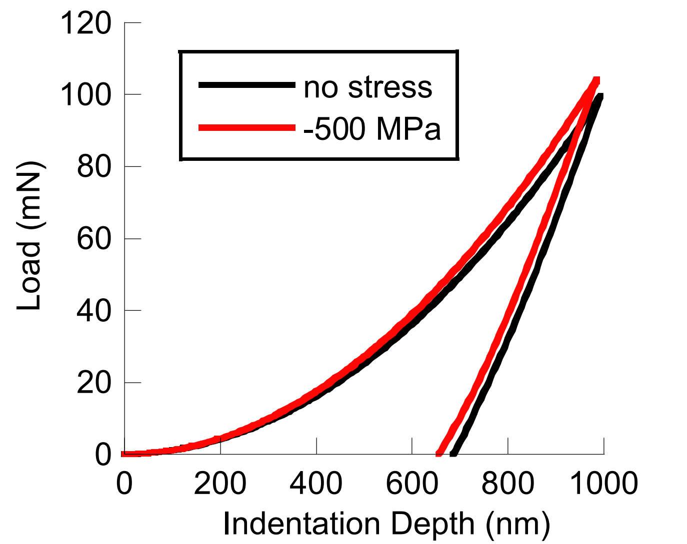
10 µm spherical tip

conical tip

E= 72 GPa
$\sigma_y$= 3 GPa

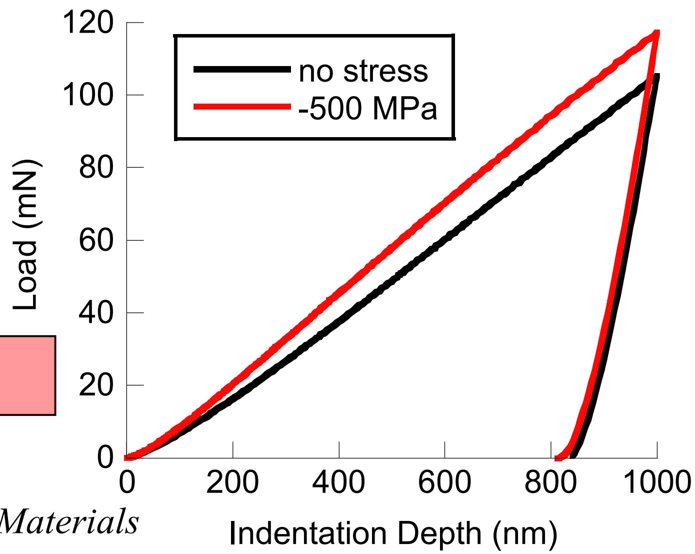
***E/$\sigma_y$= 24***

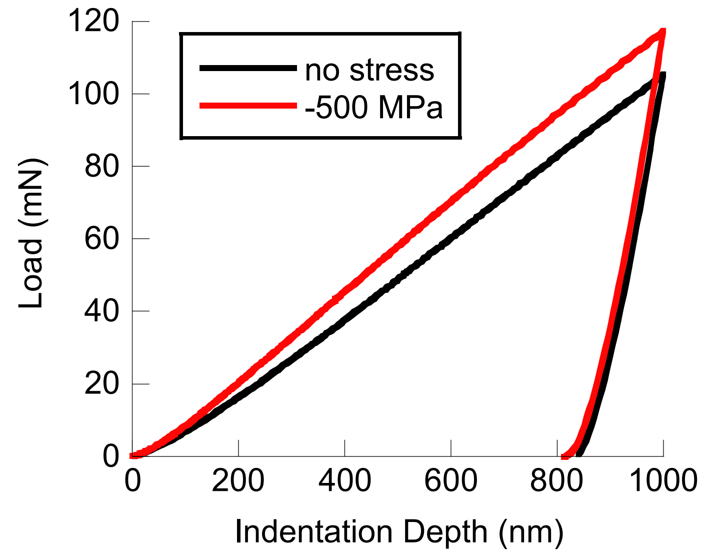



E= 72 GPa
$\sigma_y$= 600 MPa

***E/$\sigma_y$= 120***



# Tabular analysis of FE results begins to reveal trends:

| $E/\sigma_y$ Ratio (E=72 GPa) | Tip Geometry | Substrate Stress (MPa) | β*Modulus O-P method (GPa) | β*Modulus True $A_c$ (GPa) | Hardness O-P Method (GPa) | Hardness True $A_c$ (GPa) |
|---|---|---|---|---|---|---|
| 24 | Spherical | 0 | 83 | 82 | 6.04 | 6.04 |
| (υ=0.2) | ***10 μm rad.*** | -500 | 81 | 81 | 6.49 | 6.45 |
| 24 | Conical | 0 | 81 | 79 | 6.48 | 6.22 |
| (υ=0.2) | ***70.3º half-angle*** | -500 | 83 | 80 | 7.05 | 6.56 |
| 120 | Spherical | 0 | 93 | 84 | 1.97 | 1.59 |
| (υ=0.3) | ***10 μm rad.*** | -500 | 98 | 83 | 2.17 | 1.55 |
| 120 | Conical | 0 | 95 | 80 | 2.19 | 1.56 |
| (υ=0.3) | ***70.3º half-angle*** | -500 | 103 | 81 | 2.50 | 1.56 |

β is a correction factor dependent on tip geometry ≈ 1.04-1.1

- **O-P method relies on unloading portion of indentation load-displacement curve for computing contact stiffness, S, contact area, $A_c$, and ultimately hardness, H, and modulus, $E_r$, material properties**

***O-P method does not account for indentation pile-up***

Key Formulas →

$$S = \frac{2\beta}{\sqrt{\pi}} E_r \sqrt{A_c}$$

$$H = P / A$$

Oliver, W.C., and Pharr, G.C.,"An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments" J. Mater. Res., Vol. 7, No. 6, 1992, p 1564.

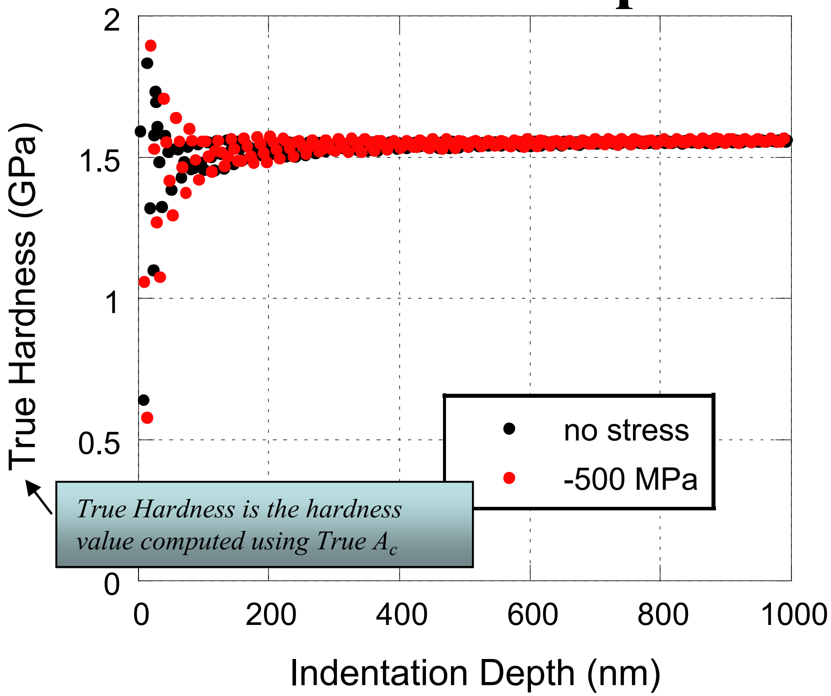
- **Properties determined using True Ac rely on the contact area between the indenter tip and substrate material determined by the finite element simulation**

***Using True Ac determined by FE accounts for indentation pile-up***

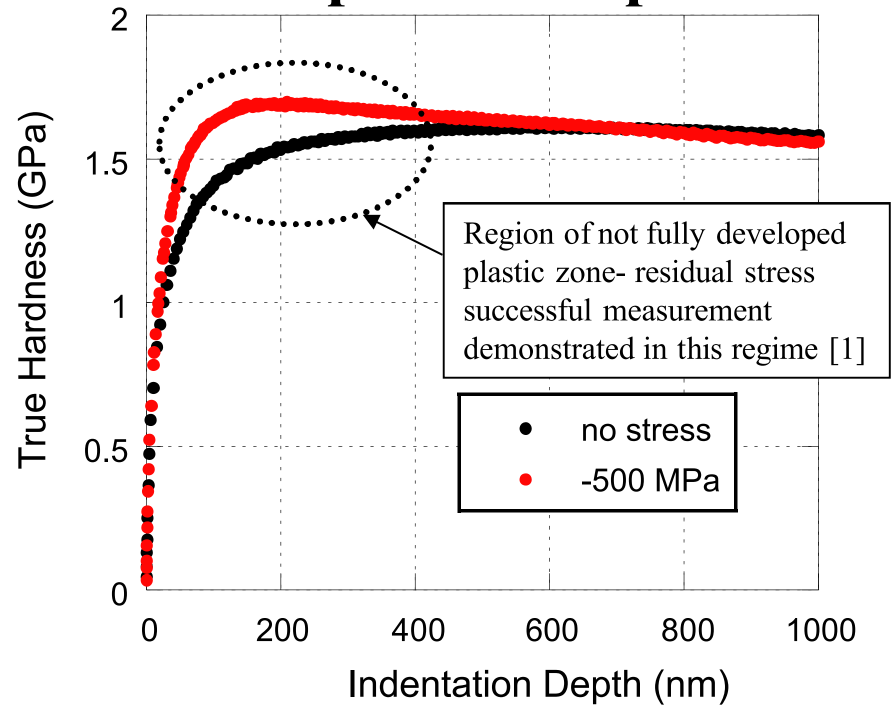
# Using true contact area in hardness determination removes the influence of indentation pile-up

$E/\sigma_y = 120$

## Conical Tip




## Spherical Tip




- **Compressive residual stress does not impact the true hardness when a material with a high $E/\sigma_y$ ratio is indented with a conical tip**

- **Compressive residual stress measurably impacts the true hardness when a material with a high E/σy ratio is indented with a spherical tip *only over a certain range of indentation depths***

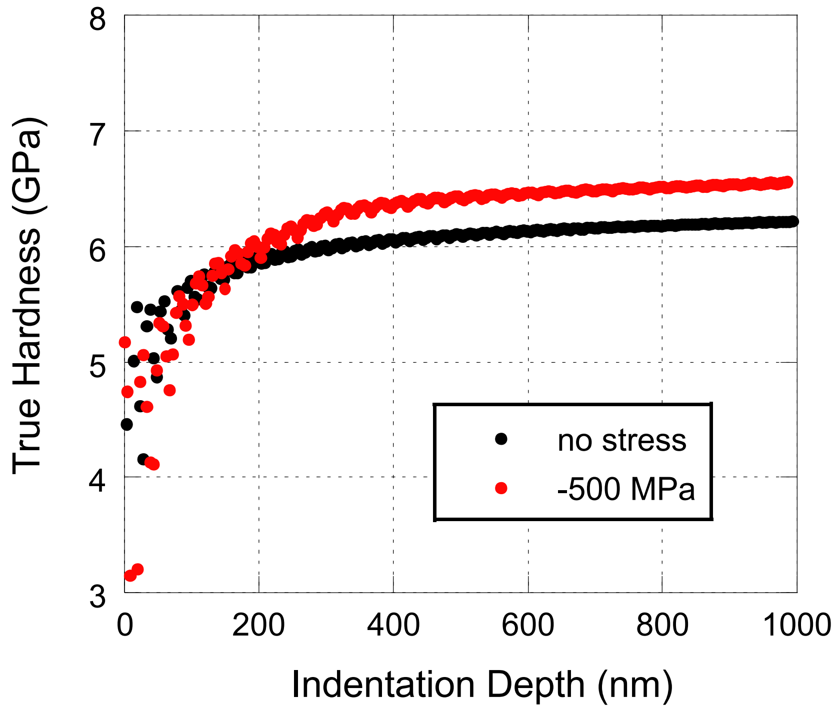


[1] Swadener, J.G., Taljit, B., Pharr, G.M. JMR, v.16,p.2091,2001

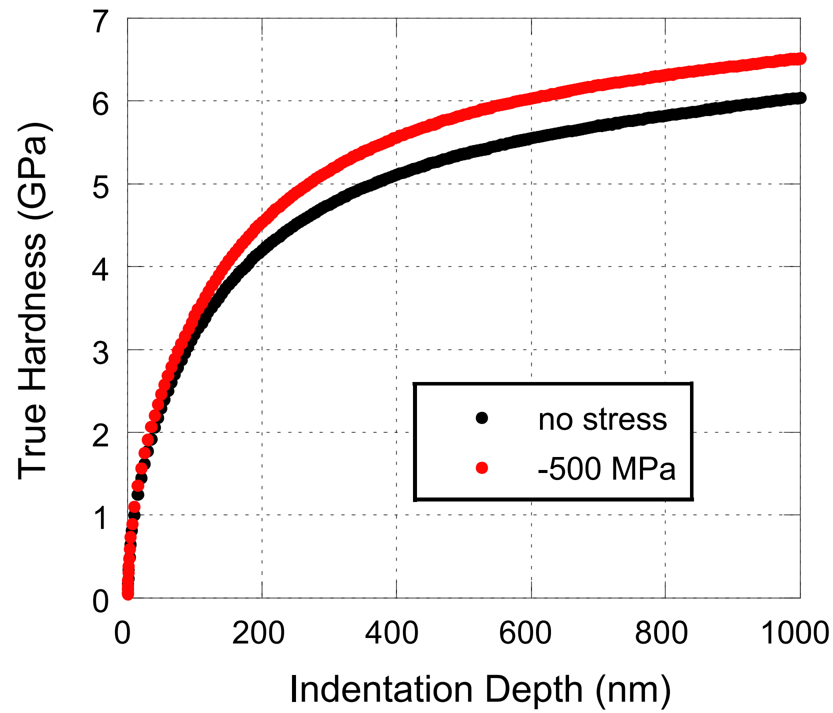
# Measurable difference in instrumented indentation response in materials with low $E/\sigma_y$ ratio

$E/\sigma_y = 24$

## Conical Tip

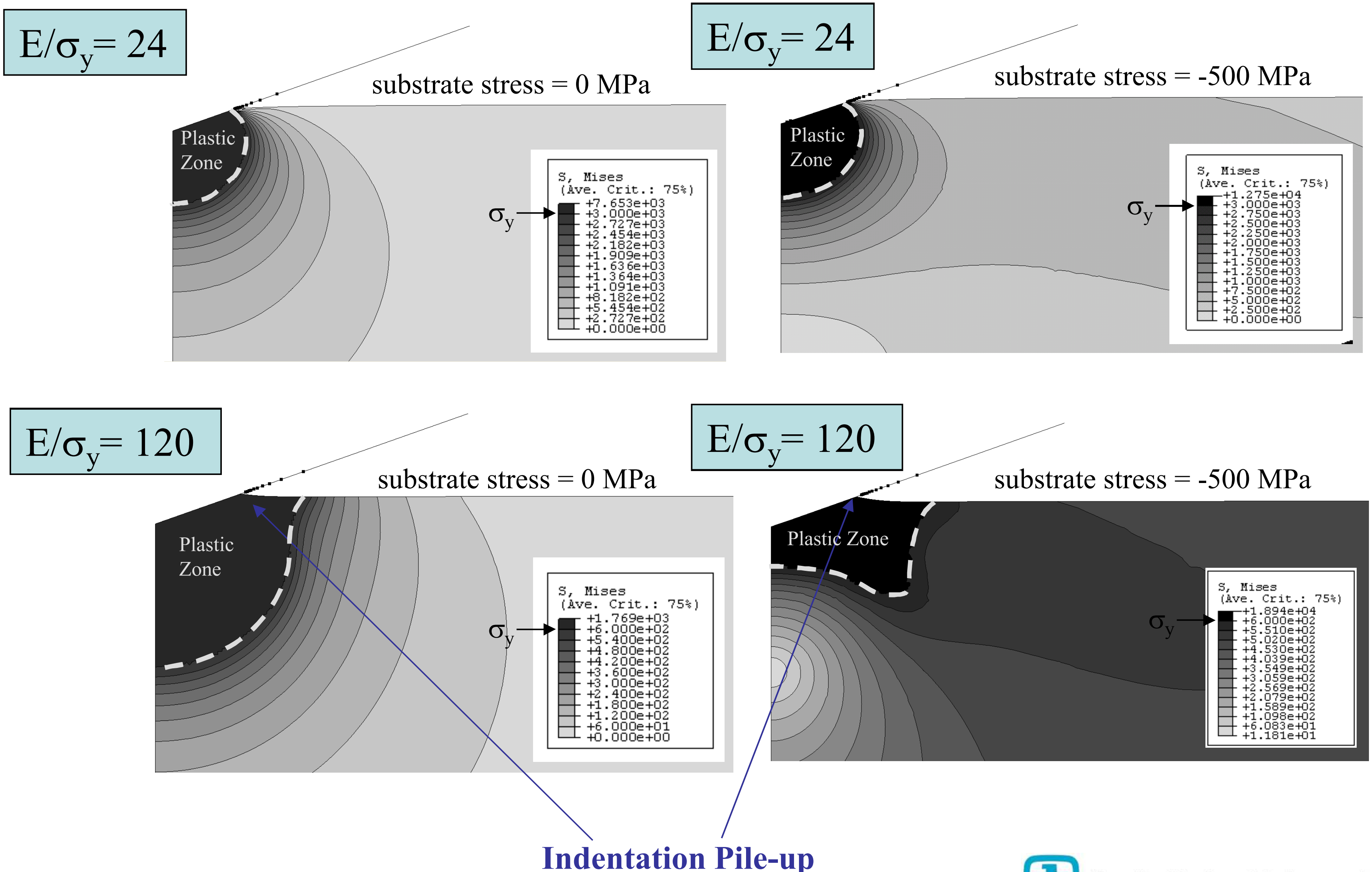



## Spherical Tip

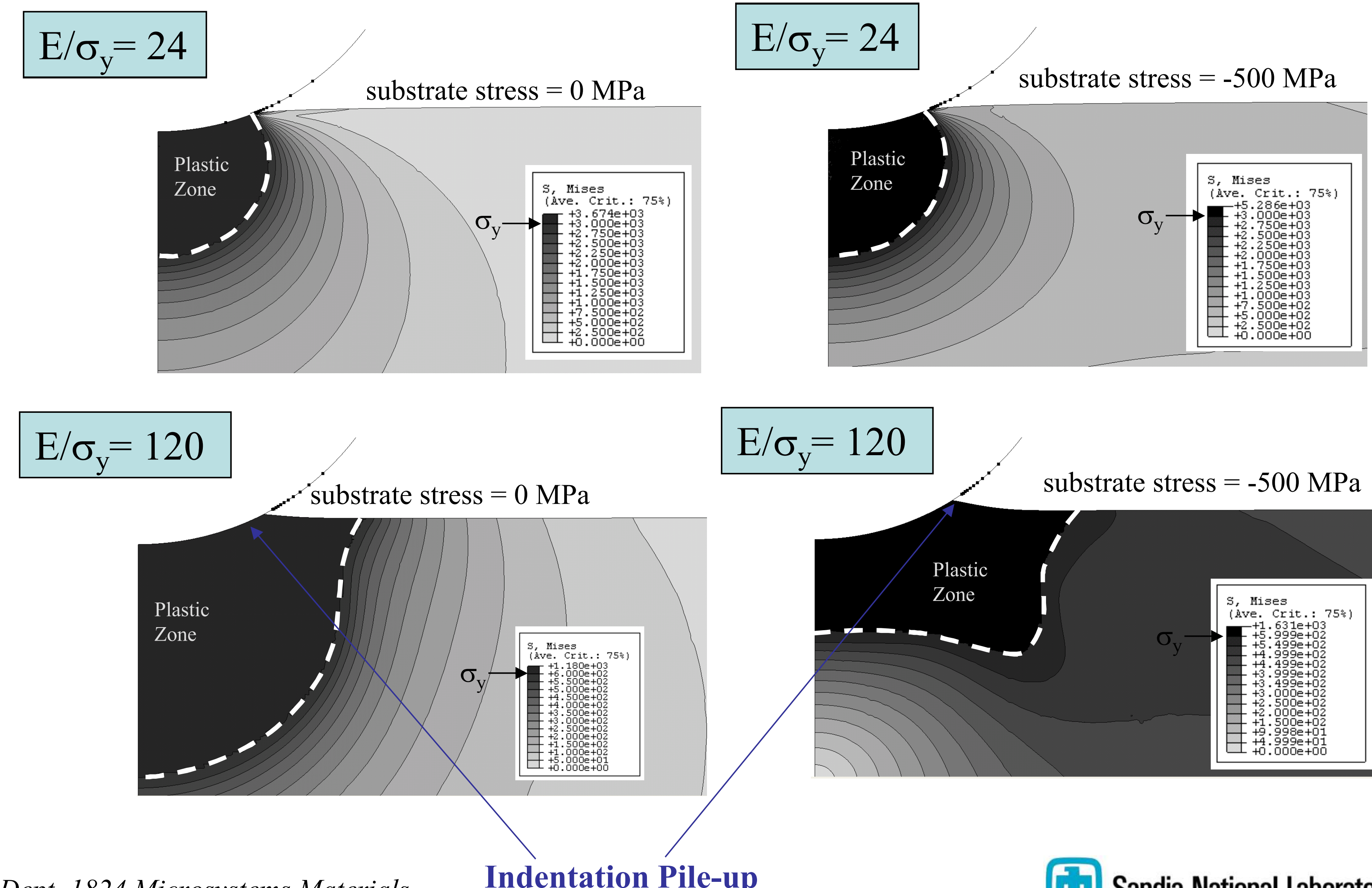



- **Compressive residual stress measurably impacts the true hardness when a material with a low E/σy ratio is indented with either a conical or spherical tip across a wide range of indentation depths, *why?***

# Stress distributions at 1μm indentation depth conical tip



# Stress distributions at 1μm indentation depth spherical tip



# Elastic-Plastic indentation described by the expanding cavity model

- When the yield point is first exceeded, the plastic zone is small and fully contained by material that remains elastic. In this circumstance , the material displaced by the indenter is accommodated by an elastic expansion of the surrounding solid. *K.L Johnson, "Contact Mechanics", Cambridge University Press, 1985.*

Conical tip:

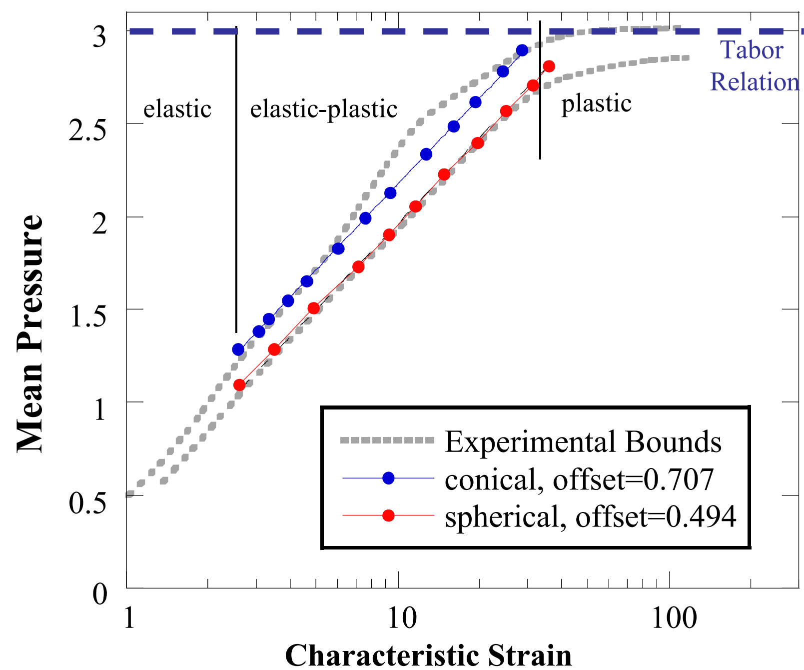
$$\frac{p_m}{\sigma_y} = \frac{2}{3}\left(1 + \ln\left(\frac{1}{3}\frac{E^* \tan\beta}{\sigma_y}\right)\right)$$

Spherical tip (tan β ≈ sin β ≈ β ≈ a/R) :

$$\frac{p_m}{\sigma_y} = \frac{2}{3}\left(1 + \ln\left(\frac{1}{3}\frac{E^* (a/R)}{\sigma_y}\right)\right)$$

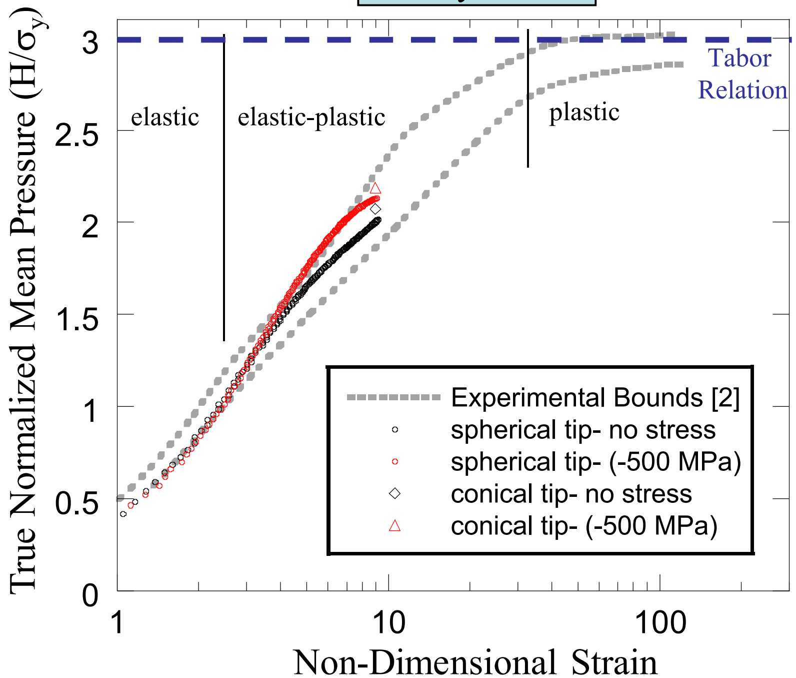
MEAN PRESSURE

CHARACTERISTIC STRAIN

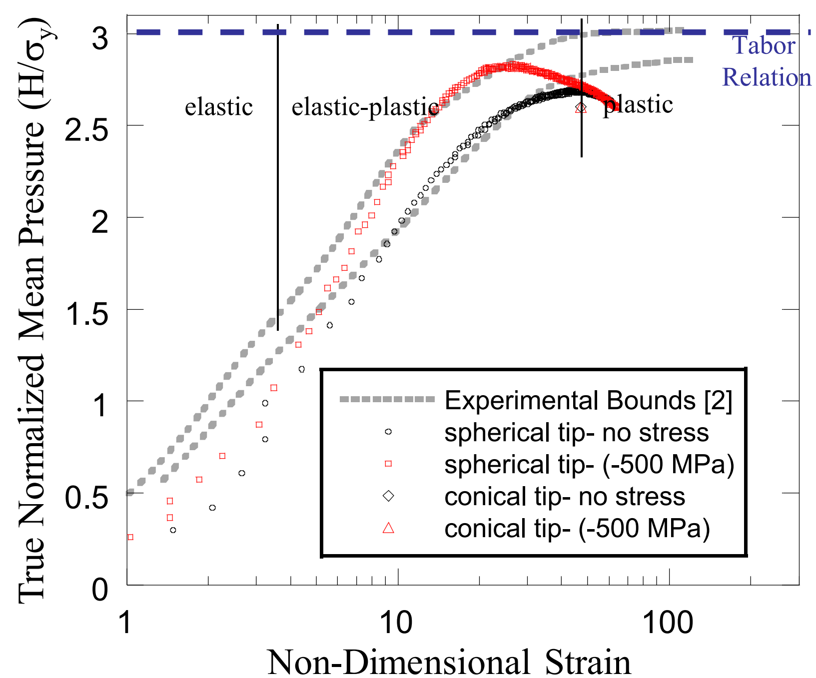


# When measuring residual stress, the expanding cavity model shows the benefit of performing experiments in the elastic-plastic regime

$E/\sigma_y = 24$




$E/\sigma_y = 120$




***Non-Dimensional Strain***

$\left(\frac{E_r a}{\sigma_y r}\right)$ for spherical tip, (a/r) is depth to tip radius ratio

$\left(\frac{E_r \tan\beta}{\sigma_y}\right)$ for conical tip

- plastic refers to "fully developed" plastic zone underneath tip. In the absence of indentation pile-up, this result demonstrates that distinguishing between stress and unstressed material using nanoindentation is not possible in the plastic region of the universal curve.
- A fully devloped plastic zone may never be achieved during indentation of a low $E/\sigma_y$ material

# Instrumented indentation experiments showing a measurable difference between stressed and unstressed glass

**50-100 MPa**

SLS Heat Strengthened

SLS Fully Tempered

**200 MPa**

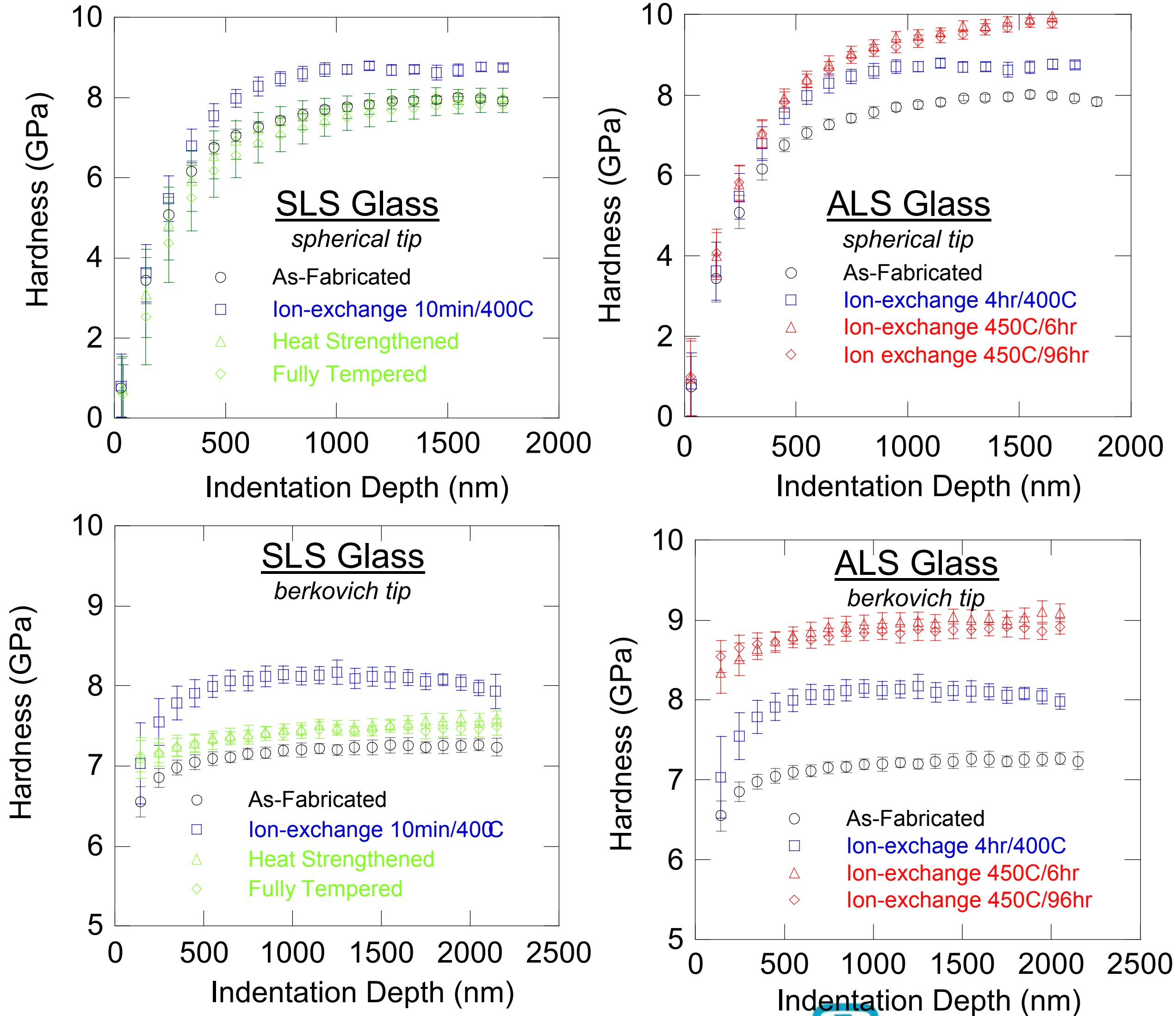
ALS Ion Exchanged 4hr/400C

SLS Ion Exchanged 10min/400C

**<300 MPa**

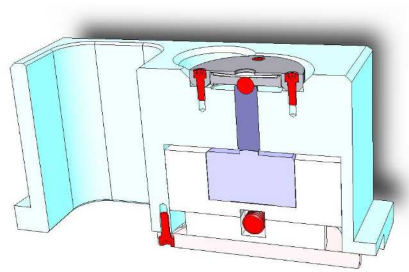
ALS Ion Exchanged 6hr/450C

ALS Ion-Exchanged 96hr/450C

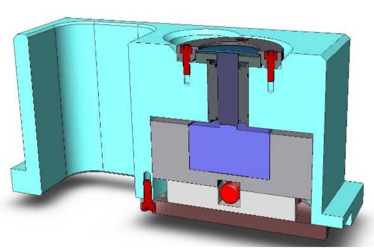


# Preliminary experiments using an in-situ stressing fixture support previous experimental and simulated observations

Picture of in-situ biaxial stressing fixture



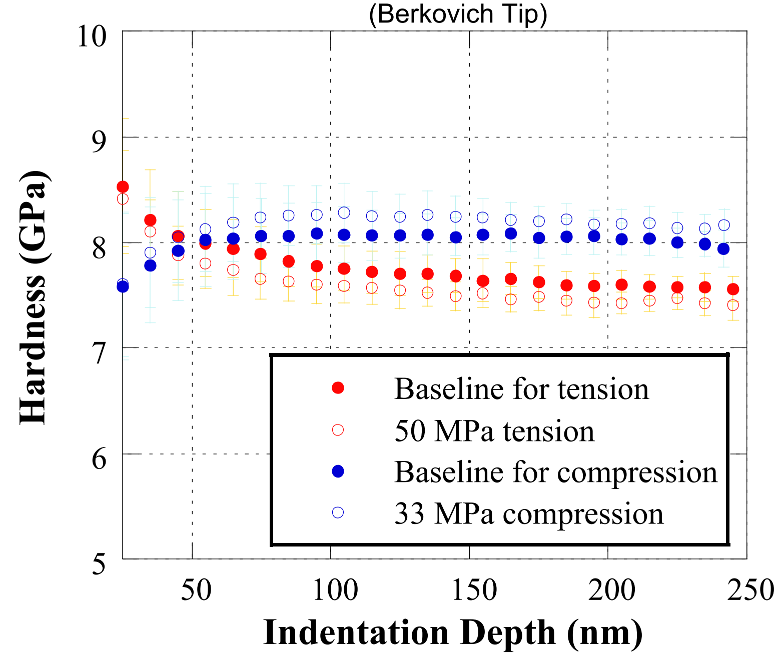
3-D section illustrating assembly for tension



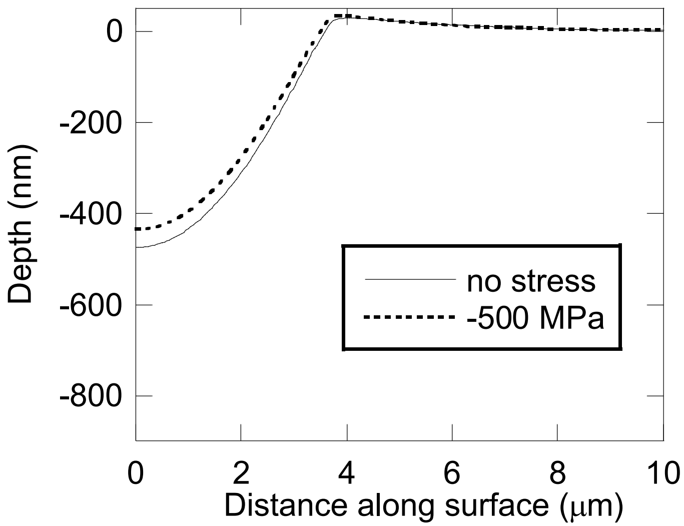
3-D section illustrating assembly for compression

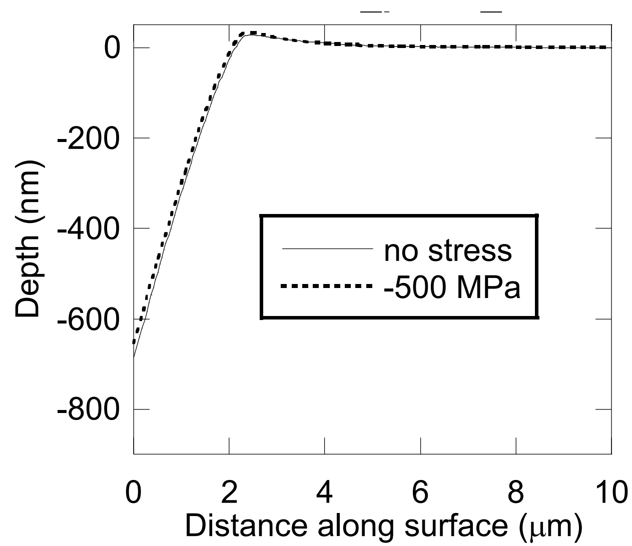
**Instrumented Indentation on Stressed Glass vs. Unstressed Glass**

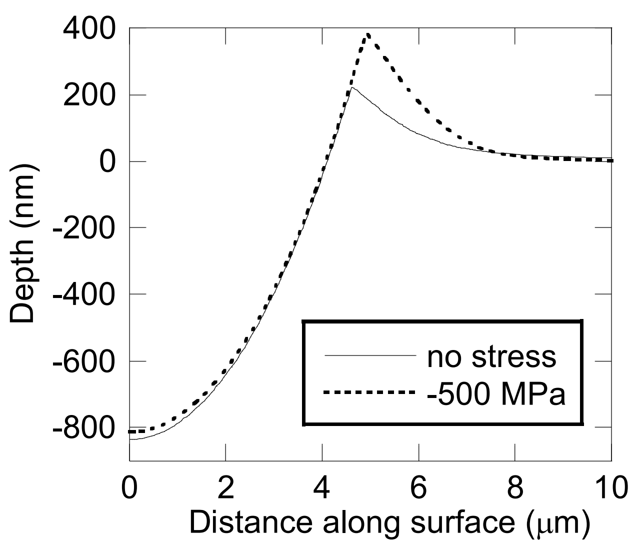
(Berkovich Tip)

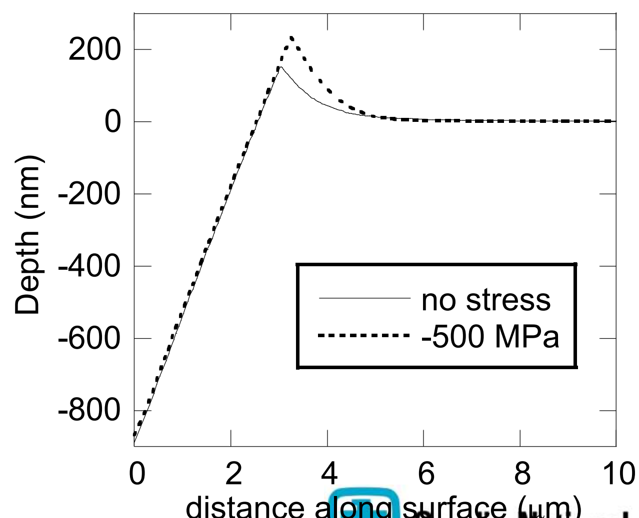



***Biaxial stress applied using in-situ fixture***


Depth (nm)
Distance along surface (µm)
no stress
-500 MPa


Depth (nm)
Distance along surface (µm)
no stress
-500 MPa


Depth (nm)
Distance along surface (µm)
no stress
-500 MPa


Depth (nm)
distance along surface (µm)
no stress
-500 MPa

# Observations and conclusions drawn from simulated results

- A plastic zone always constrained to region underneath tip, as demonstrated in the low $E/\sigma_y$ simulated results corresponds to a not fully developed plastic zone.

***These results showed a measurable difference in indentation response between stressed and unstressed substrates for both spherical and pyramidal tip geometries***

- A plastic zone that breaks out to the substrate surface, as demonstrated in the high $E/\sigma_y$ simulated results corresponds to a fully developed plastic zone.

***These results also showed a measurable difference in indentation response between stressed and unstressed substrates, that difference is completely attributable to "indentation pile-up" phenomenon***

An indentation experiment that creates a large elastic zone and a small confined plastic zone is most useful for measuring influence of residual stress using nanoindentation. Glasses and Cermanics are favorable materials for this type of measurement because of their high $E/\sigma_y$ ratio

[2] Johnson, K.L. Contact Mechanics, Cambridge University Press, pg. 171-179.