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Measuring Residual Stress in Glasses and Ceramics using Instrumented Indentation

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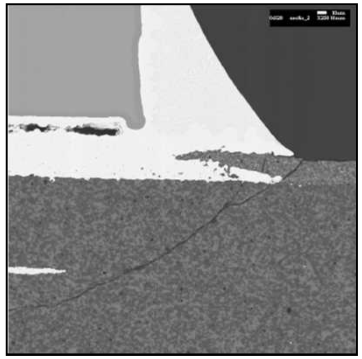
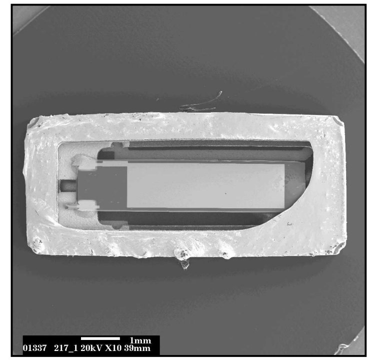
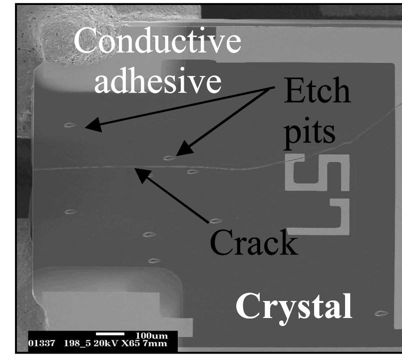
Problem: Processing and thermally induced residual stresses lead to failure of ceramic-based microsystems components

Examples of cracks in LTCC ceramic substrates

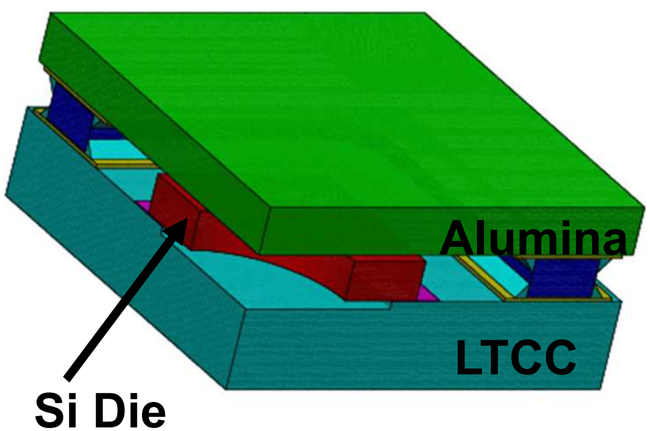


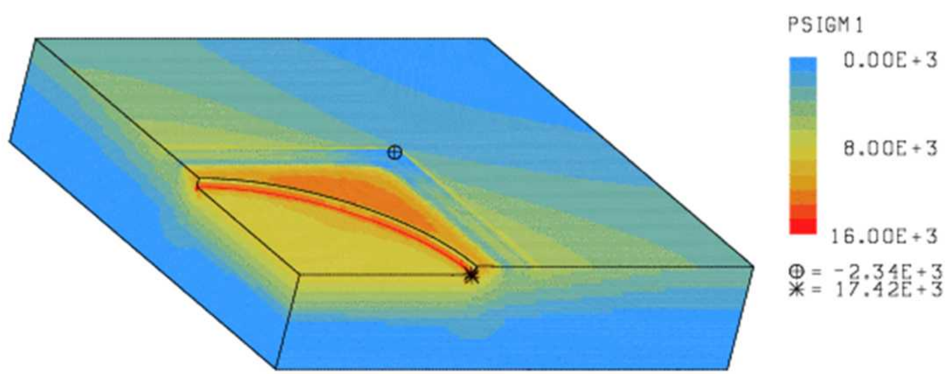
Quartz AT Strip SAW Device





FE simulation of Die Attached Microsystems assembly



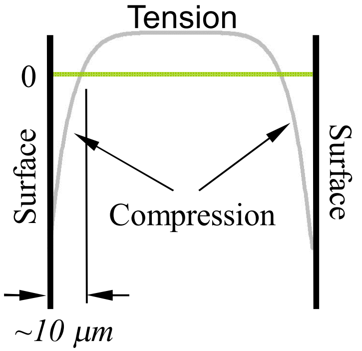


Can nanoindentation be used as a method to measure residual stress in brittle materials?

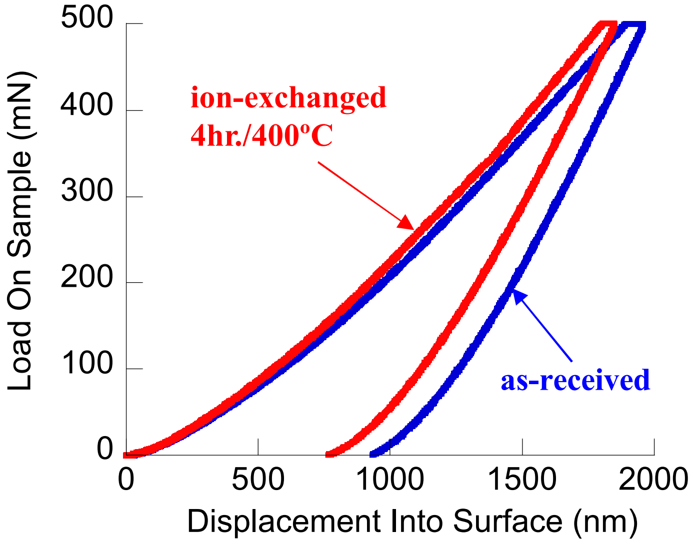
An initial observation:

- **A very noticeable and repeatable difference in measured indentation response between unstressed and stressed glass.**
- **$E/\sigma_y \approx 25$ in glass, $E/\sigma_y \approx 150$ or greater in most metals.**

Stress Profile in Ion-Exchanged Glass



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



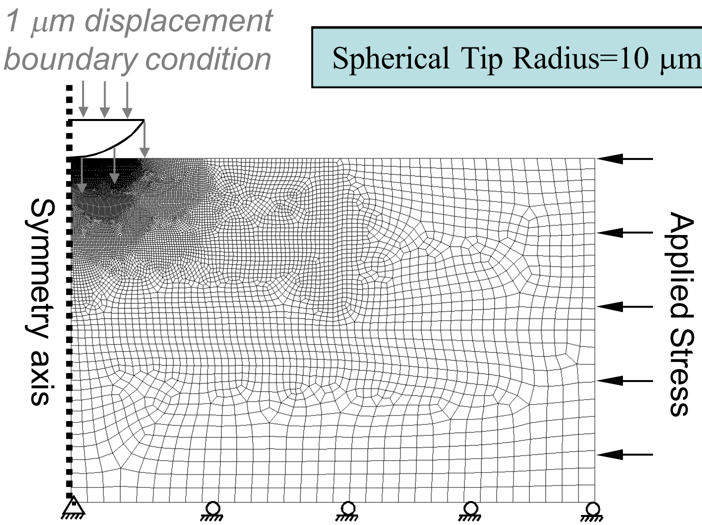
A series of finite element simulations were performed to investigate the role residual stress on the indentation response

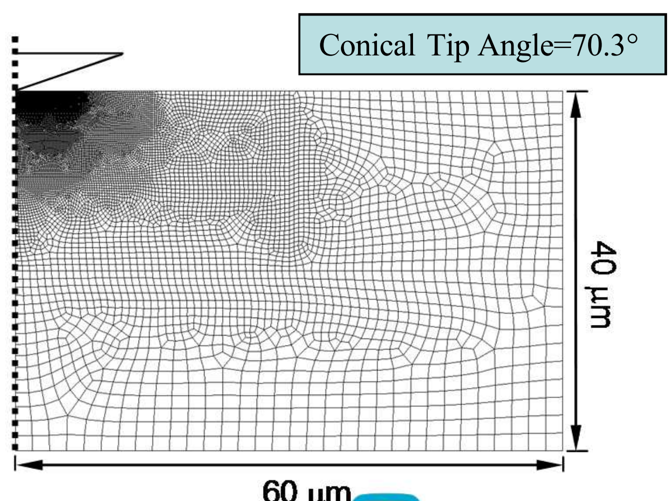
- 8 simulations- displacement control to 1 um depth

material properties	**tip geometry**	**residual stress**
E=72 GPa σ_y=3 GPa	spherical- 10 µm radius	1) none 2) -500 MPa
E=72 GPa σ_y=3 GPa	conical- 70.3° half-angle	3) none 4) -500 MPa
E=72 GPa σ_y=600 MPa	spherical- 10 µm radius	5) none 6) -500MPa
E=72 GPa σ_y=600 MPa	conical- 70.3° half-angle	5) none 6) -500MPa

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

- FE meshes and boundary conditions





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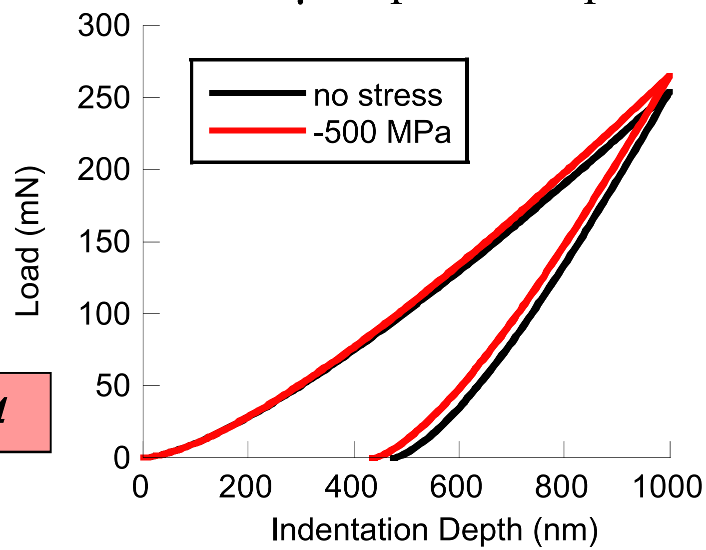
Stressed substrates gave a significantly different load-displacement response in every simulated case

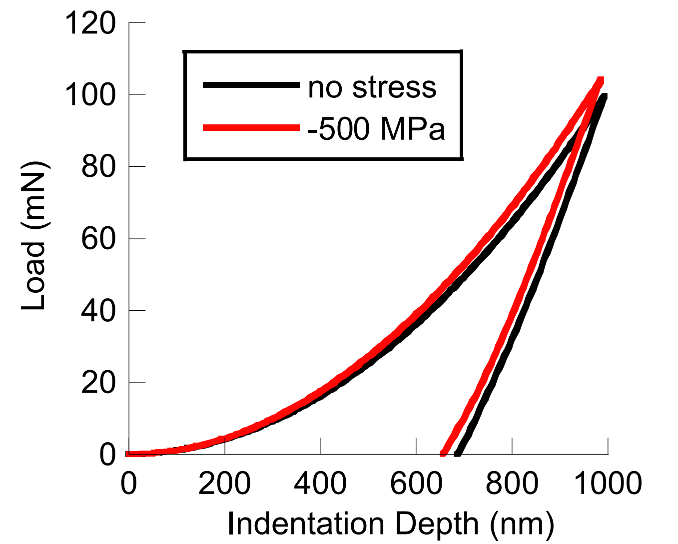
10 µm spherical tip

conical tip

E= 72 GPa
σ_y= 3 GPa

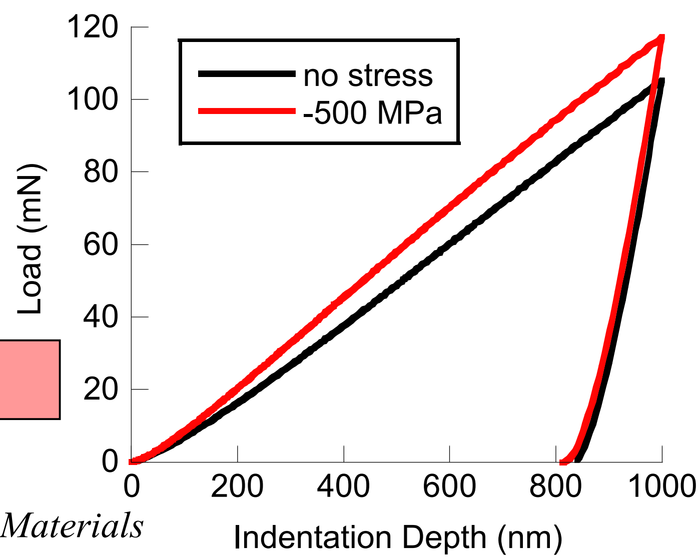
E/σ_y= 24

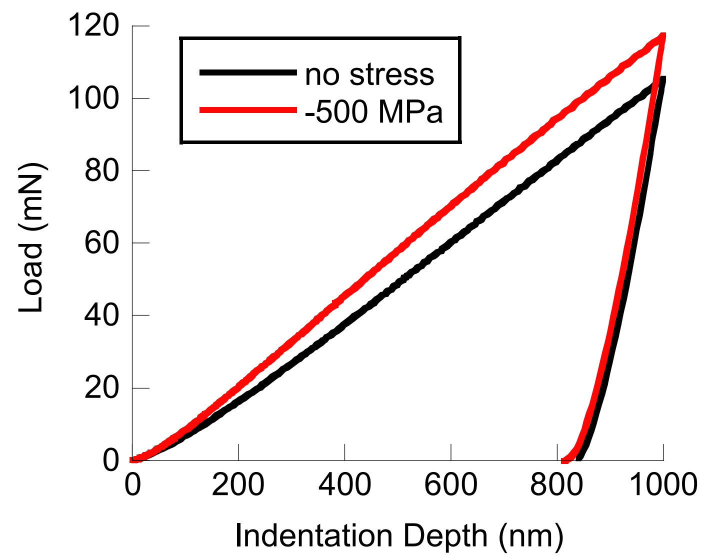




E= 72 GPa
σ_y= 600 MPa

E/σ_y= 120





Tabular analysis of FE results begins to reveal trends:

E/σ_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)	***70.3° 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)	***70.3° 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 →

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

$$H = P / A$$

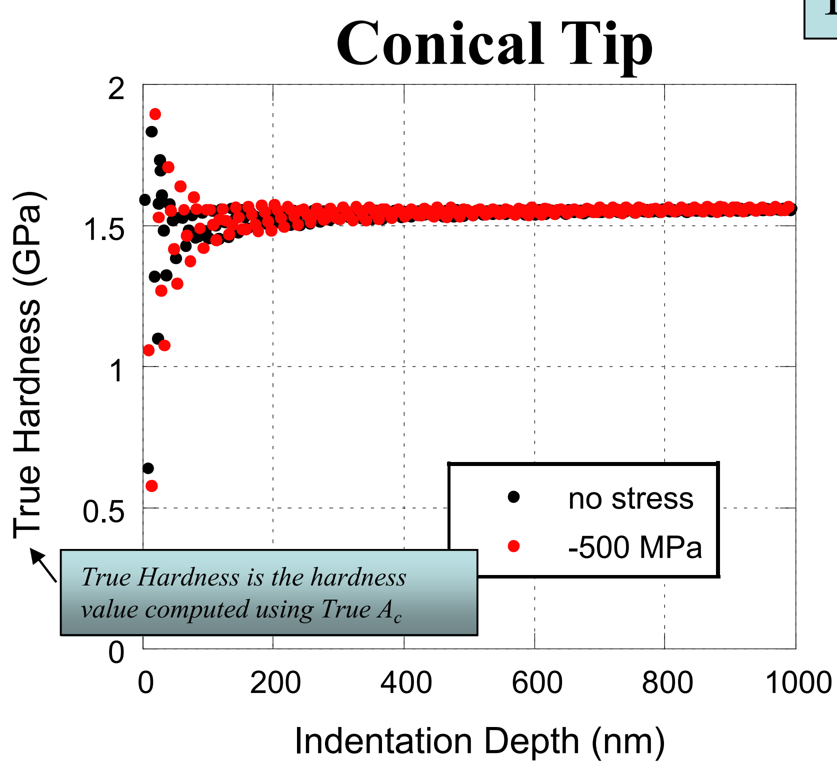
Oliver, W.C., and Pharr, G.C.,"An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments" J. Mater. Res., Vol. 7, No. 6, 1992, p 1564.

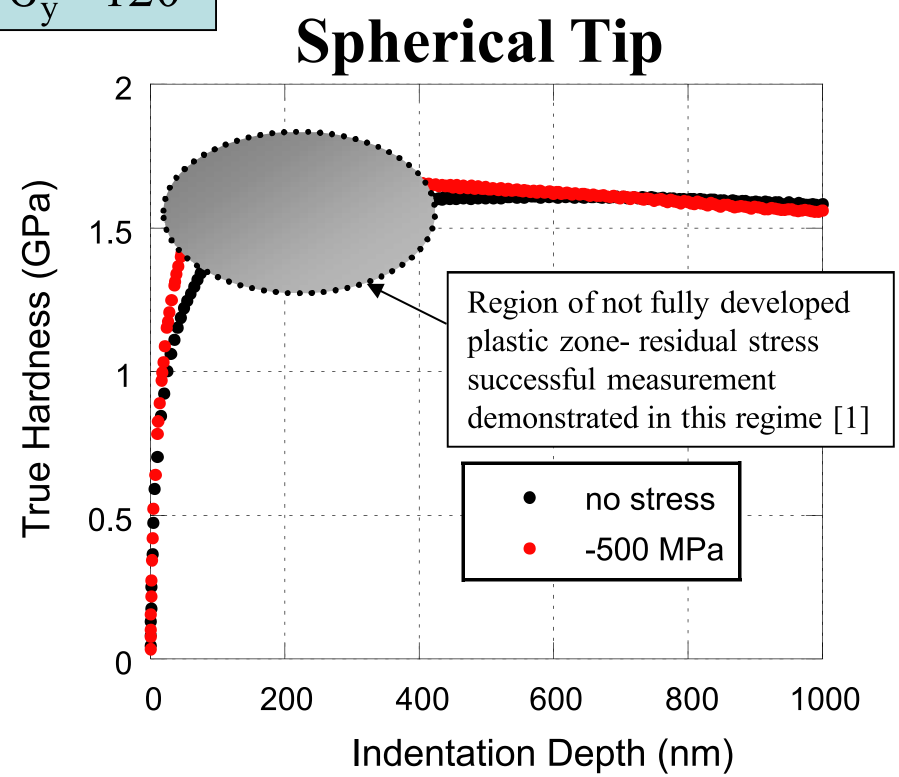
- **Properties determined using True Ac rely on the contact area between the indenter tip and substrate material determined by the finite element simulation**

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

$E/\sigma_y = 120$





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

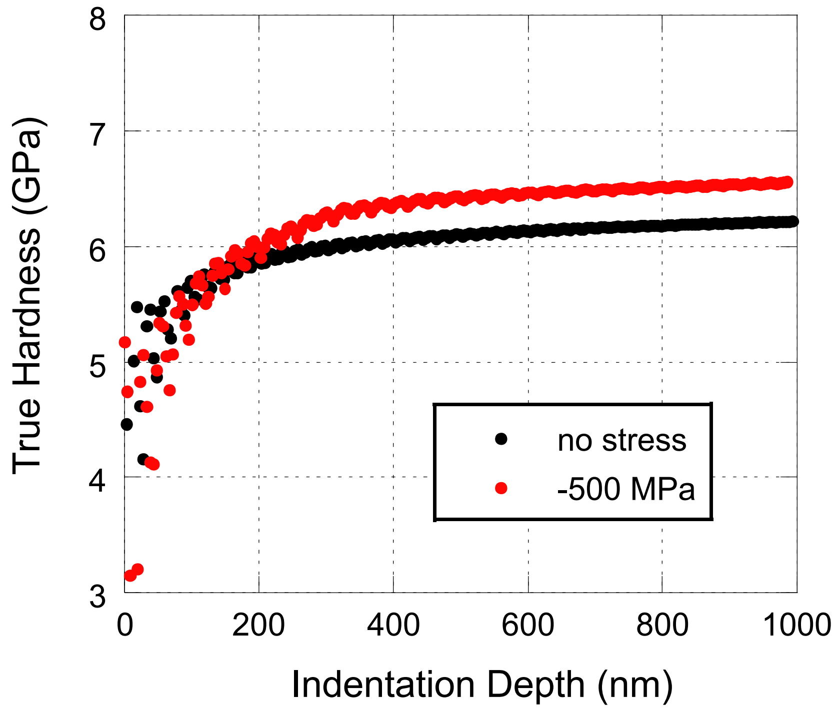
- **Compressive residual stress measurably impacts the true hardness when a material with a high E/σy ratio is indented with a spherical tip *only over a certain range of indentation depths***

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

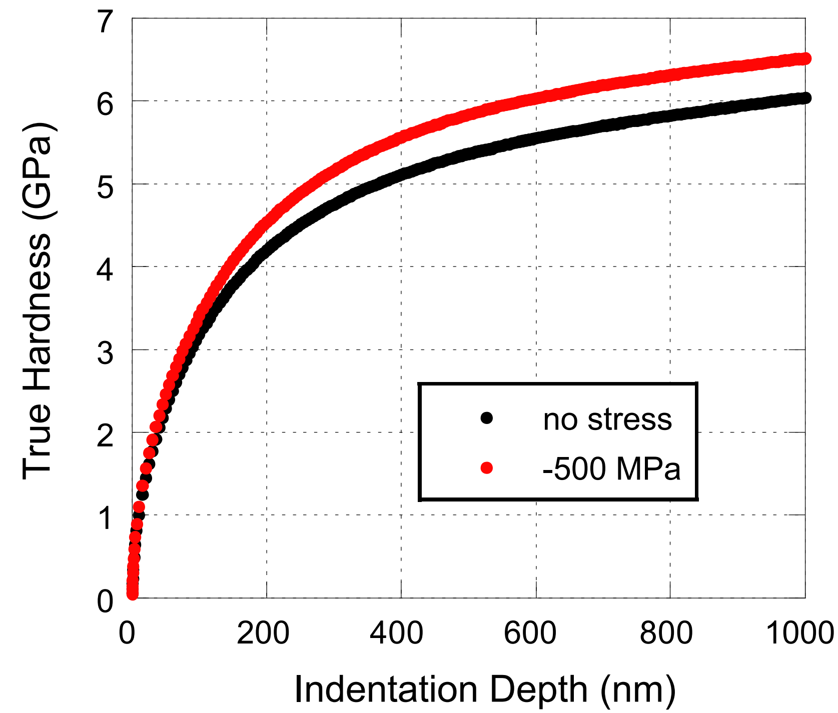
Measurable difference in instrumented indentation response in materials with low E/σ_y ratio

$E/\sigma_y = 24$

Conical Tip

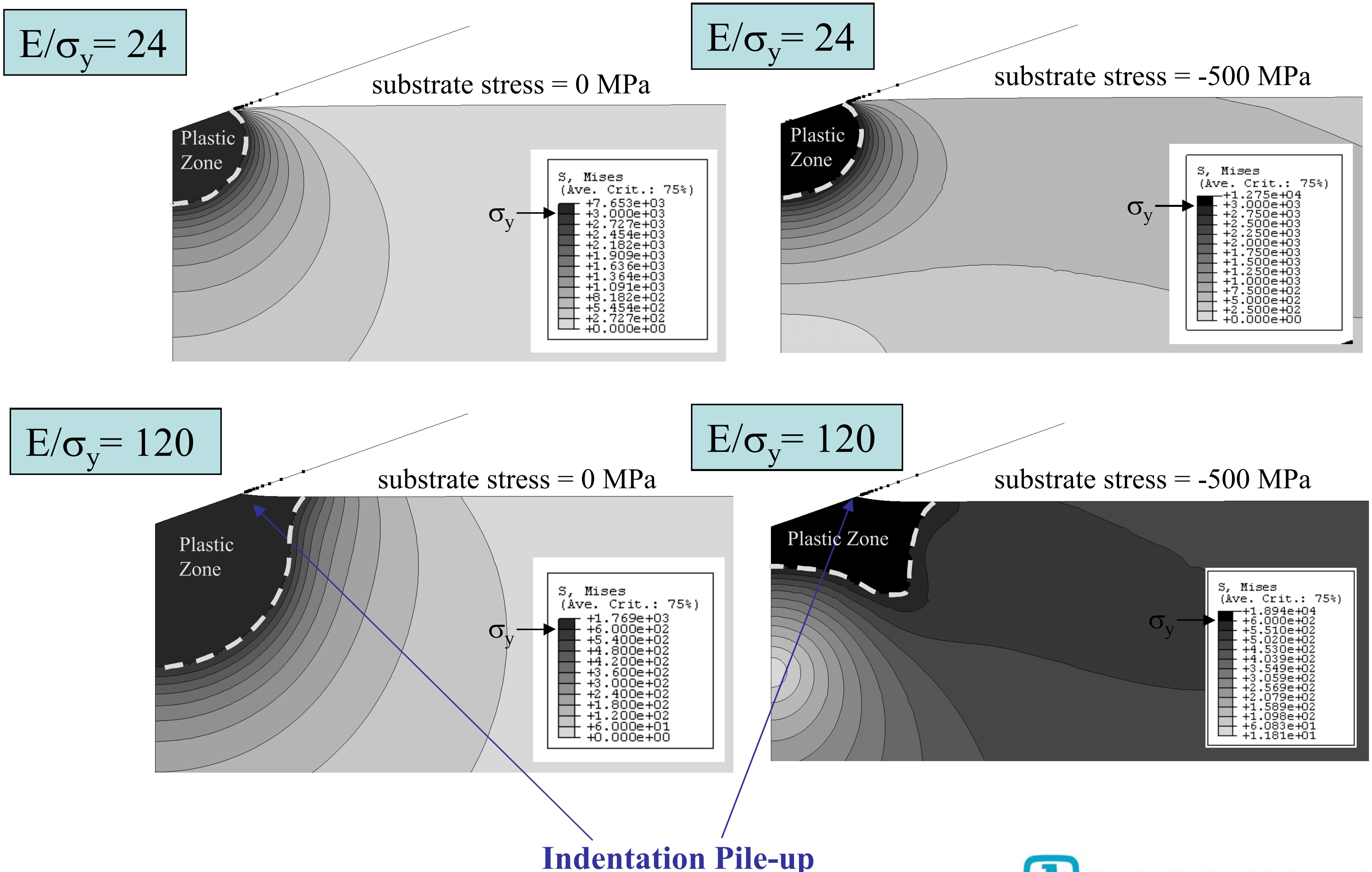


Spherical Tip

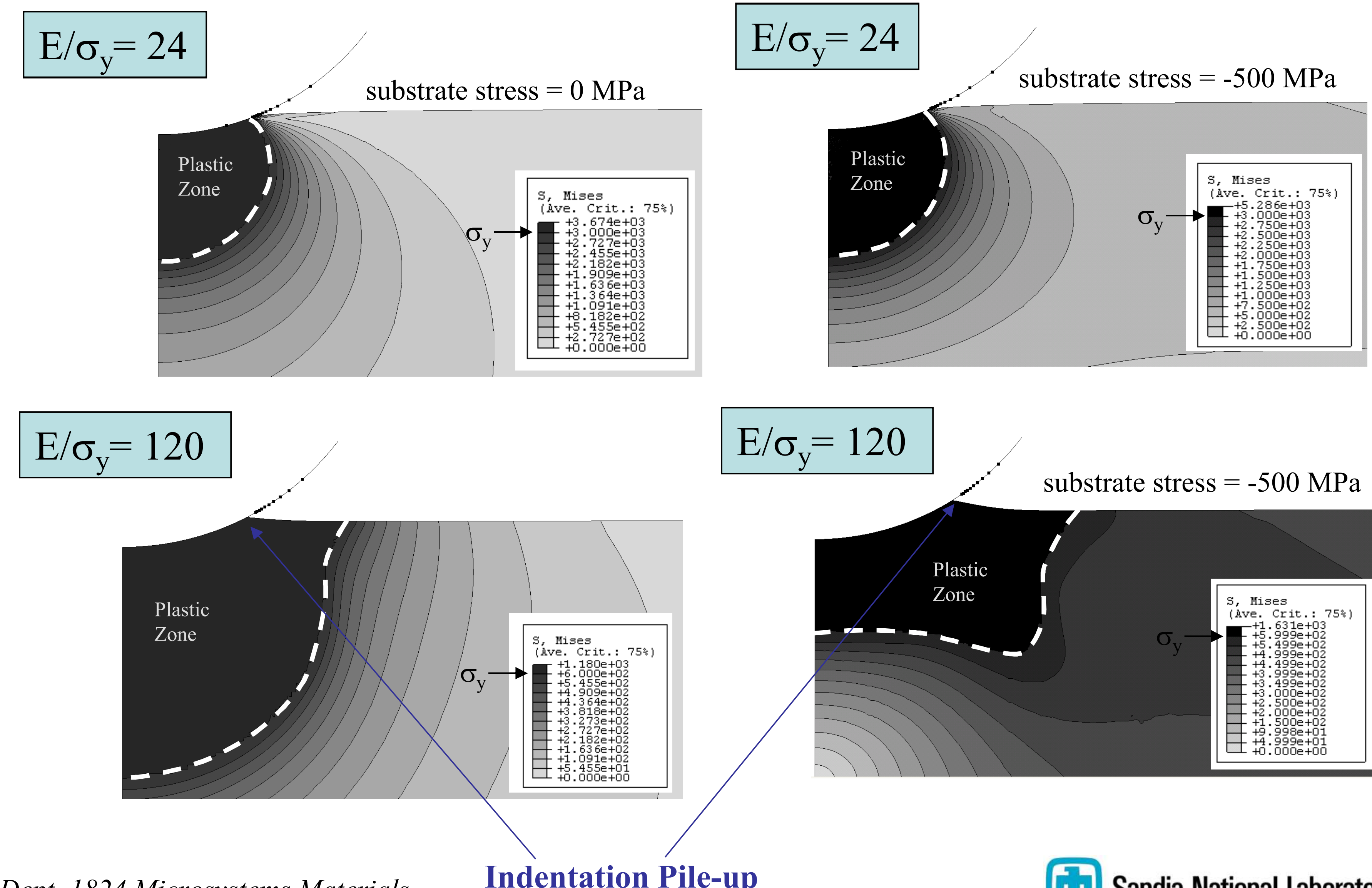


- **Compressive residual stress measurably impacts the true hardness when a material with a low E/σy ratio is indented with either a conical or spherical tip across a wide range of indentation depths, *why?***

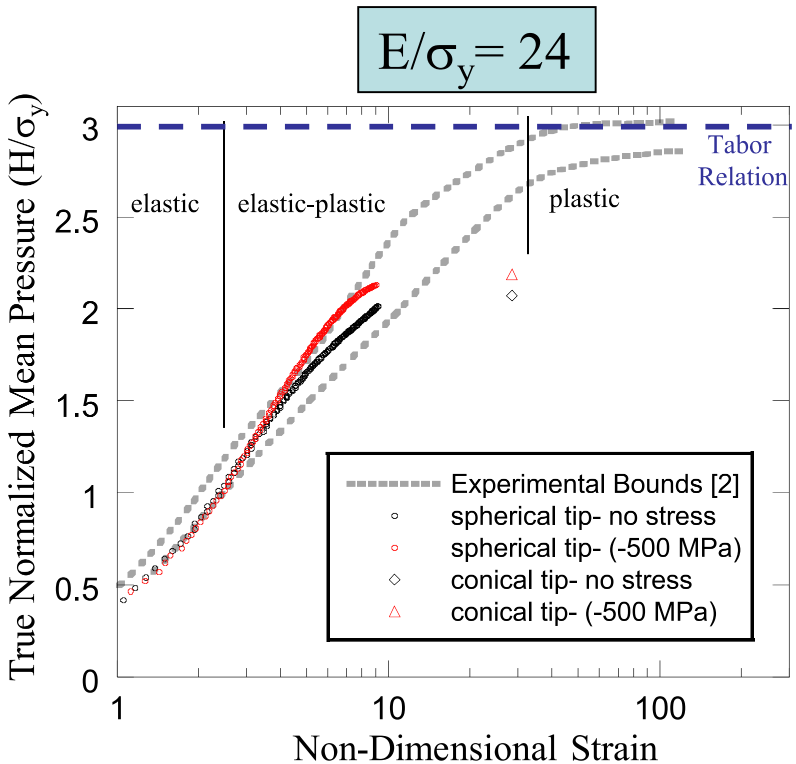
Stress distributions at 1μm indentation depth conical tip

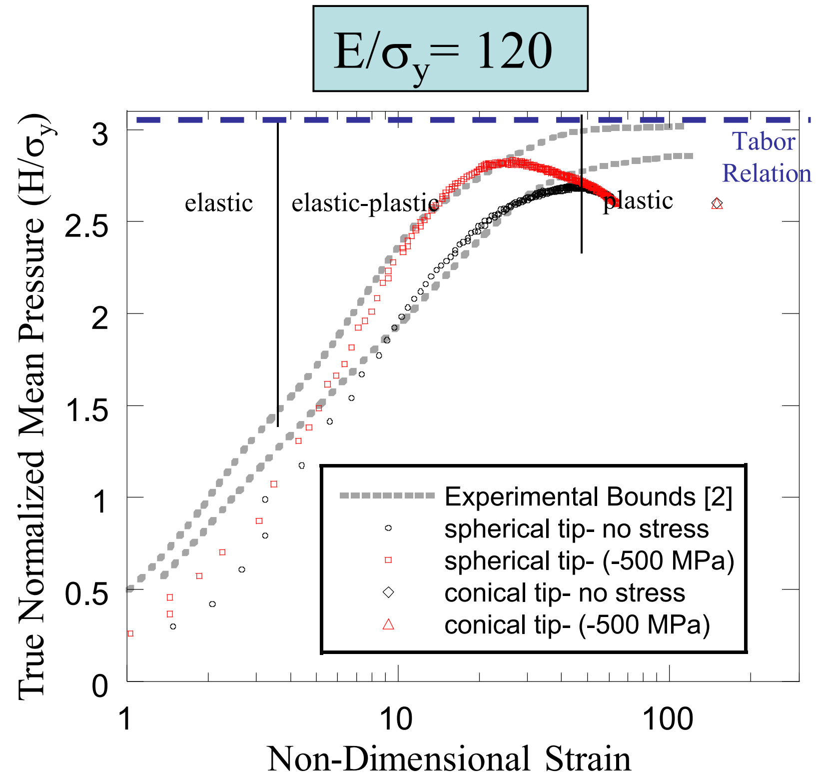


Stress distributions at 1μm indentation depth spherical tip



When measuring residual stress, the "Universal Curve" shows the benefit of performing experiments in the elastic-plastic regime





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/σ_y material

Observations and conclusions drawn from simulated results

- A plastic zone always constrained to region underneath tip, as demonstrated in the low E/σ_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/σ_y simulated results corresponds to a fully developed plastic zone.

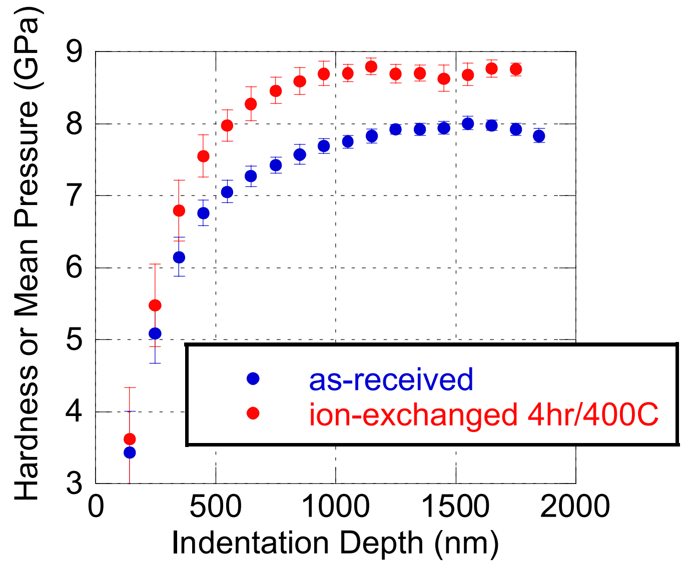
These results also showed a measurable difference in indentation response between stressed and unstressed substrates, that difference is completely attributable to "indentation pile-up" phenomenon

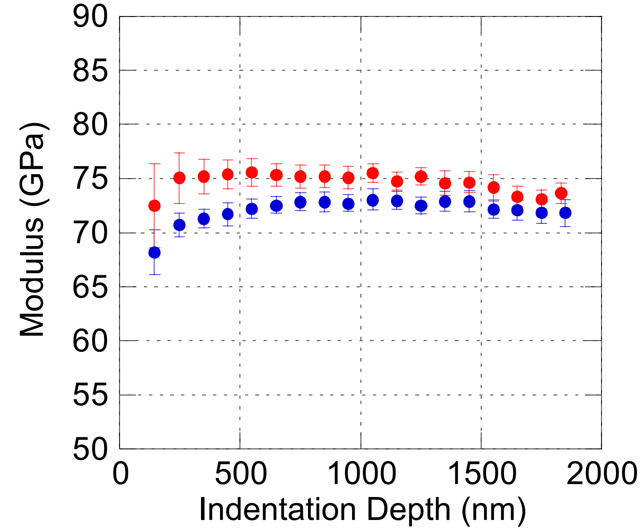
An indentation experiment that creates a large elastic zone and a small confined plastic zone is most useful for measuring influence of residual stress using nanoindentation. Glasses and Cermanics are favorable materials for this type of measurement because of their high E/σ_y ratio

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

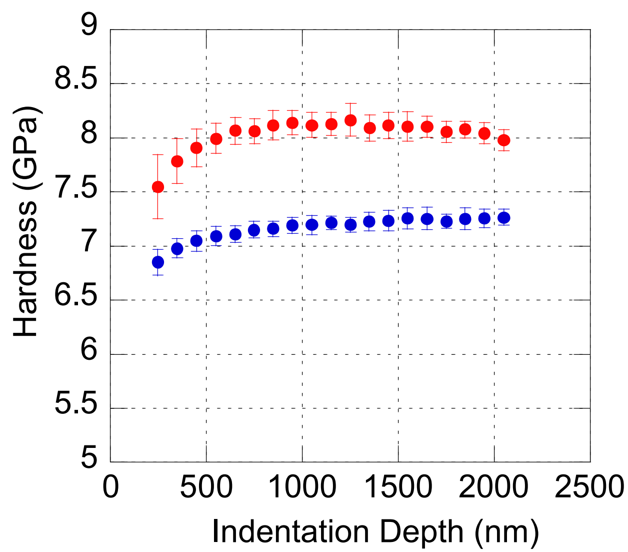
Nanoindentation results on PPG Glass-500 mN load

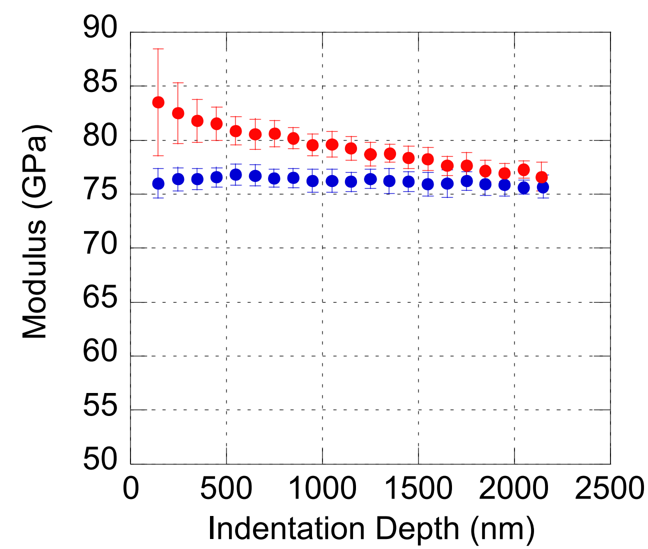
10 μm spherical tip →





berkovich tip →





Conclusions