NANOINDENTATION IN MATERIALS RESEARCH: PAST, PRESENT, AND FUTURE

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OUTLINE

A Brief History

- *the instrument (WCO)*
- *the method for H&E measurement (GMP)*
- *the obvious applications (GMP)*

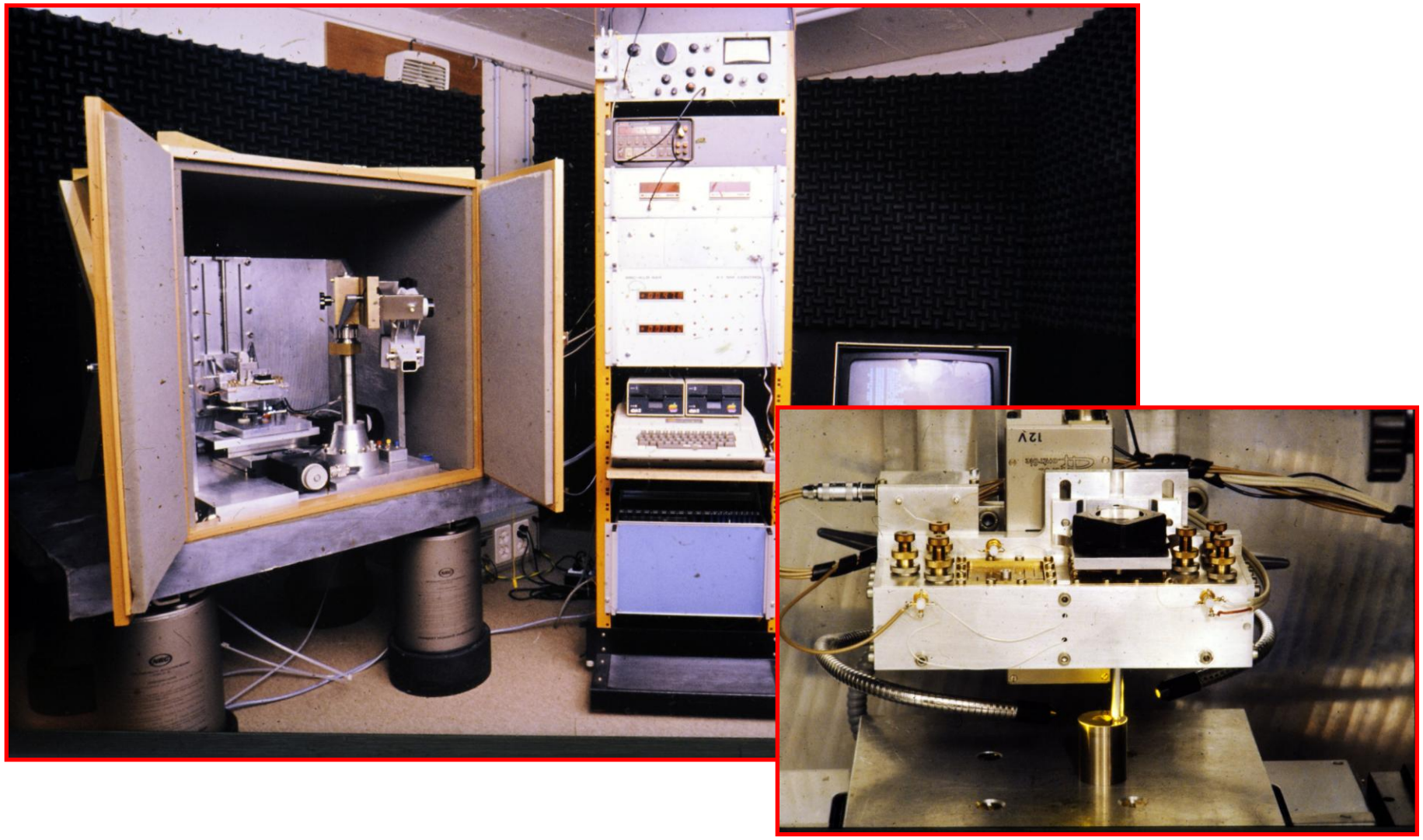
Some Applications That Weren't So Obvious

- *geology & planetary materials (WCO)*
- *polymers & viscoelastic materials (WCO)*
- *the continuum to atomistic bridge (GMP)*
- *biology & medical science (WCO)*
- *some unusual applications (GMP)*

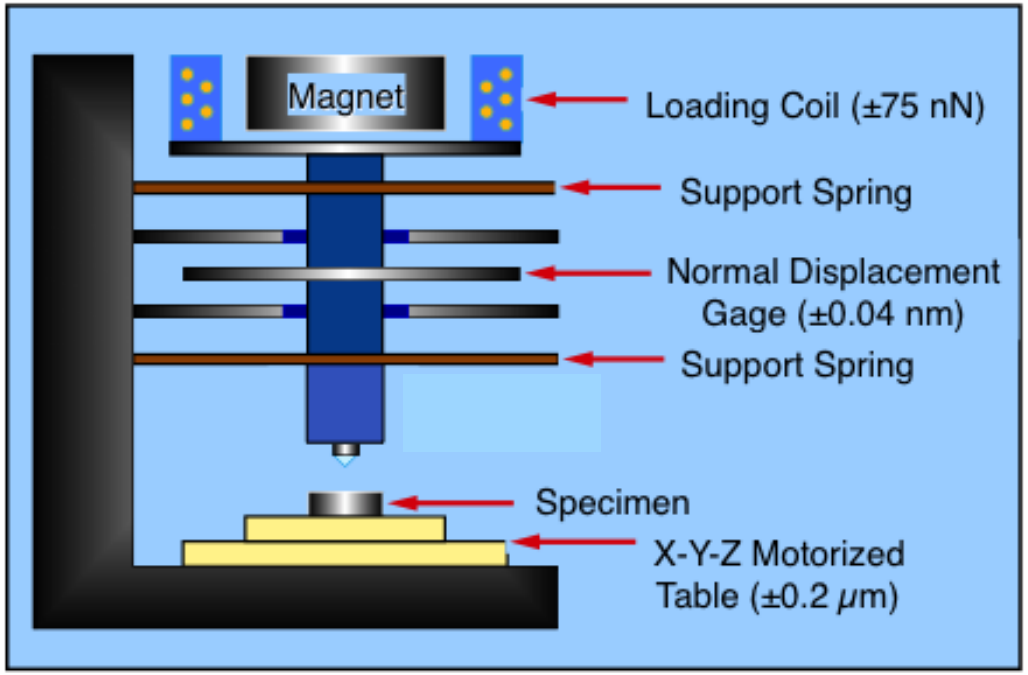
THE ORIGINAL NANOINDENTER

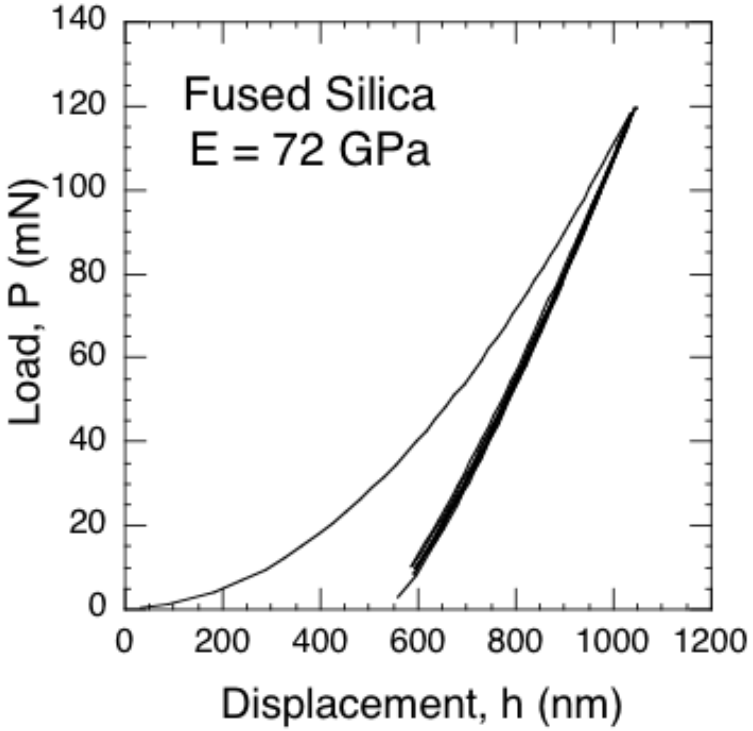
THE ORIGINAL NANOINDENTER

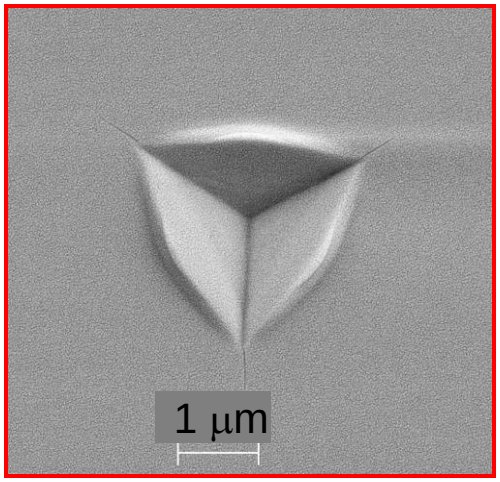
- Pethica, Hutchings, and Oliver, *Phil Mag A***48**, 593(1983)

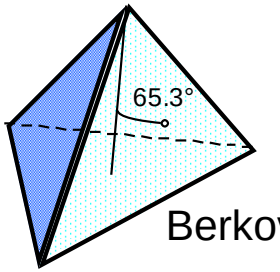


BASIC MEASUREMENTS





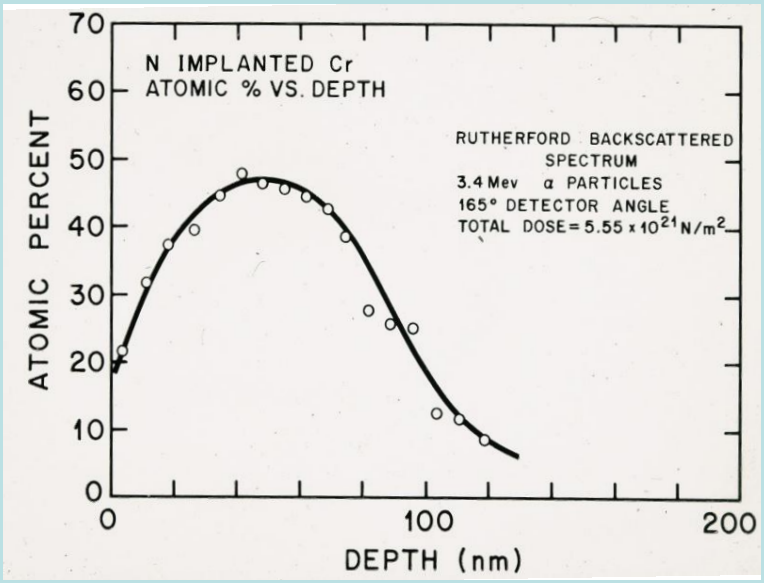




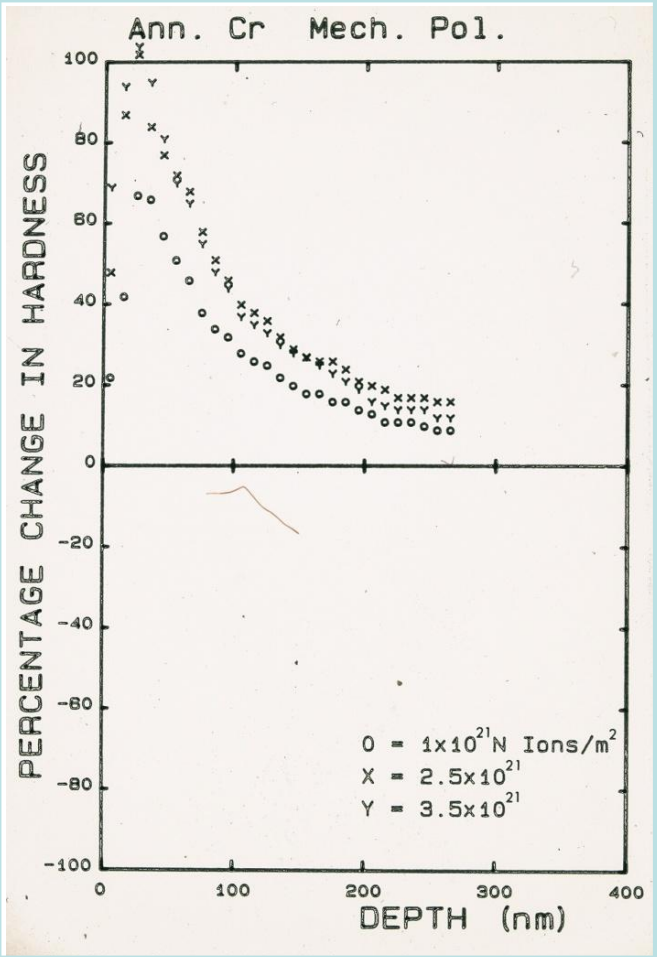
Berkovich indenter

THE ORIGINAL APPLICATION

- *Hardness of ion-implanted metals*



- Pethica, Hutchings and Oliver, *Nuclear Instruments and Methods*, 209/210 (1983)



THE METHOD: A BRIEF OVERVIEW

KEY PRIOR WORK

N.A. Stillwell & D. Tabor

"Elastic recovery of conical indentations"

Proc. Phys. Soc. London **78**, 169 (1961)

I.N. Sneddon

"The relation between load and penetration in the axisymmetric Boussinesq problem for a punch of arbitrary profile"

Int. J. Engng. Sci. **3**, 47 (1965)

S.I. Bulychev et al.

"Determination of Young's modulus according to indentation diagram"

Zavod. Lab. **41**, 1137 (1975)

J.L. Loubet et al.

"Vickers indentation curves of magnesium oxide (MgO)"

J. Tribology **106,** 43 (1984)

M.F. Doerner & W.D. Nix

"A method for interpreting the data from depth-sensing indentation instruments"

JMR **1**, 601 (1986)

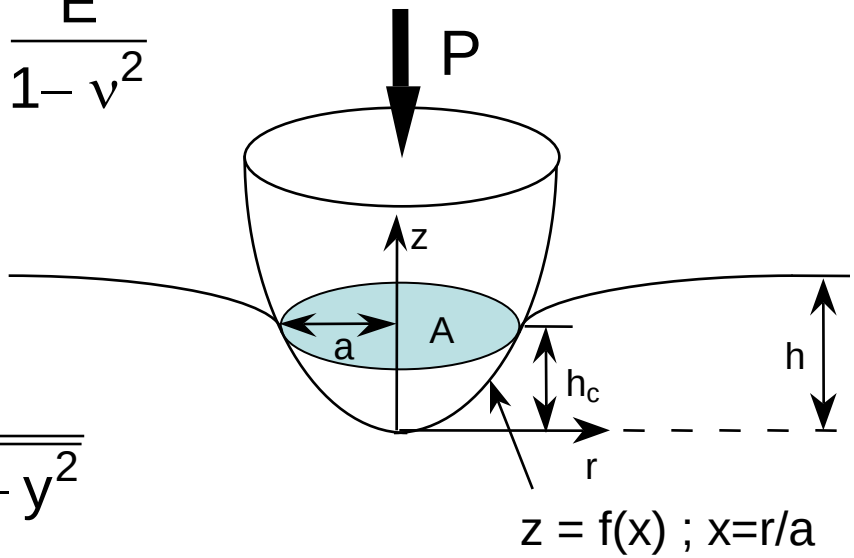
THE FUNDAMENTAL EQUATION

- Pharr, Brotzen, & Oliver, *J Mater Res* **7**, 613 (1992)

$$P(a) = 2E_{eff}\, a \int_0^1 \frac{x^2 f'(x)\,dx}{\sqrt{1-x^2}}; \quad E_{eff} = \frac{E}{1-\nu^2}$$

$$h(a) = \int_0^1 \frac{f'(x)\,dx}{\sqrt{1-x^2}}$$

$$h_c = \lim_{x \to 1} \frac{2}{\pi}\sqrt{x^2-1} \int_0^1 \frac{y f(y)\,dy}{(y^2-x^2)\sqrt{1-y^2}}$$



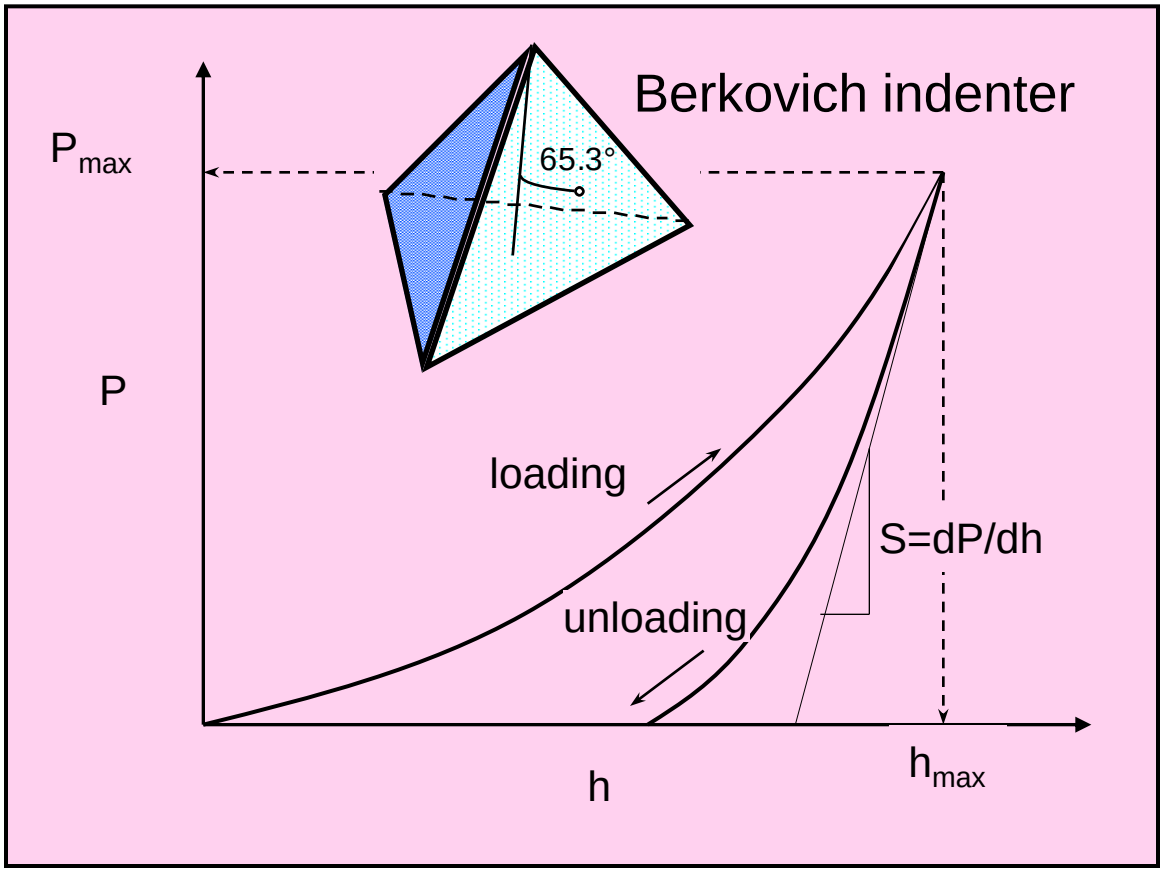
z = f(x) ; x=r/a

Sneddon, *Int J Engrg Sci* **3**, 47 (1965)

$$S = \frac{dP}{dh} = 2E_{eff}\, a = \frac{2}{\sqrt{\pi}} E_{eff} \sqrt{A}$$

HARDNESS & MODULUS MEASUREMENT

- Oliver & Pharr, *J Mater Res* **7**, 1564 (1992)

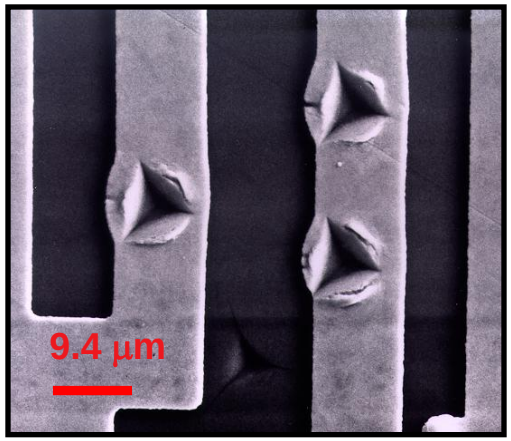


$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S}$$

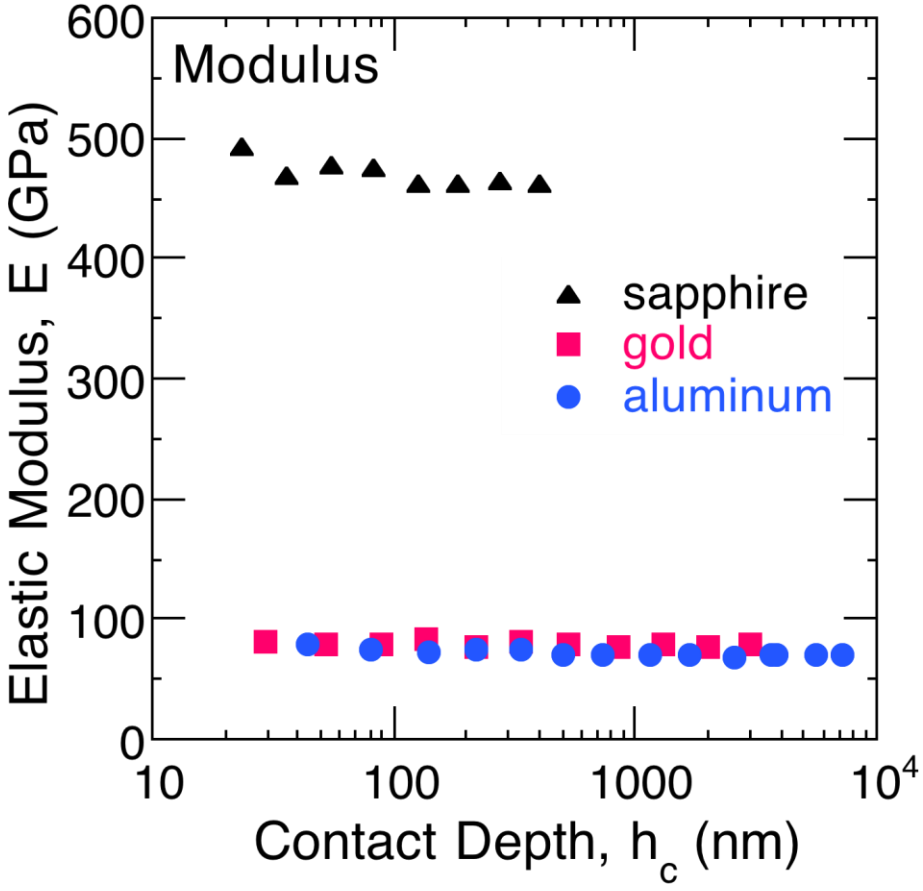
$$A = A(h_c) \quad \text{(area function)}$$

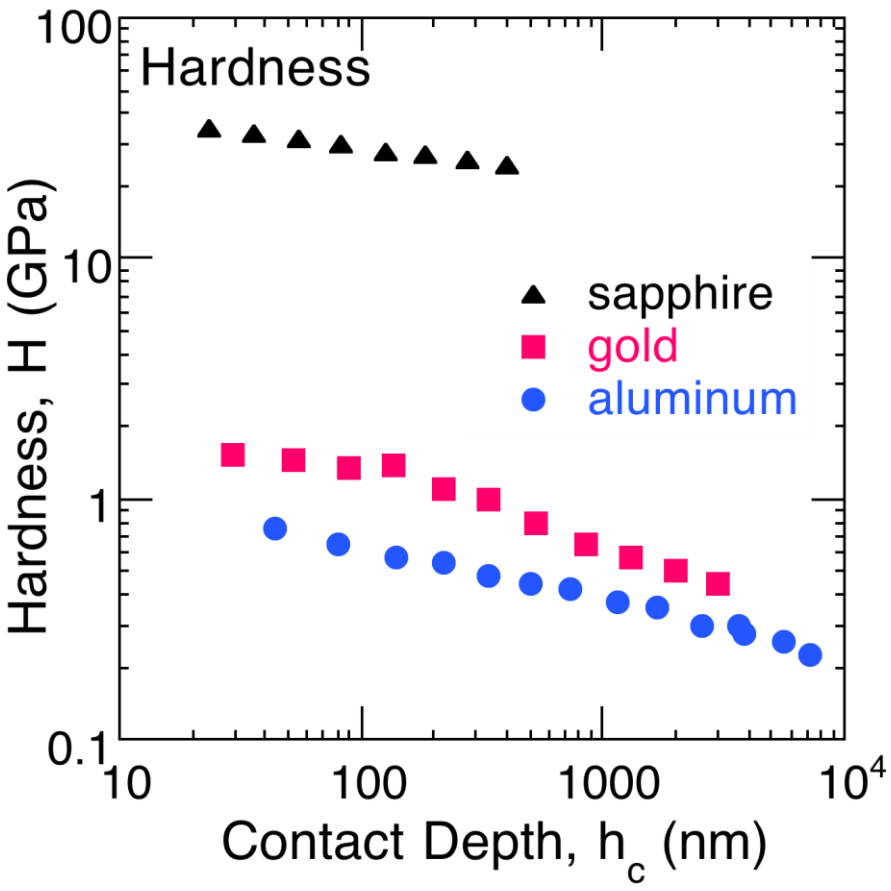
$$H = \frac{P}{A}$$

$$E_{eff} = \frac{E}{1-\nu^2} = \frac{S}{2a} = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}}$$

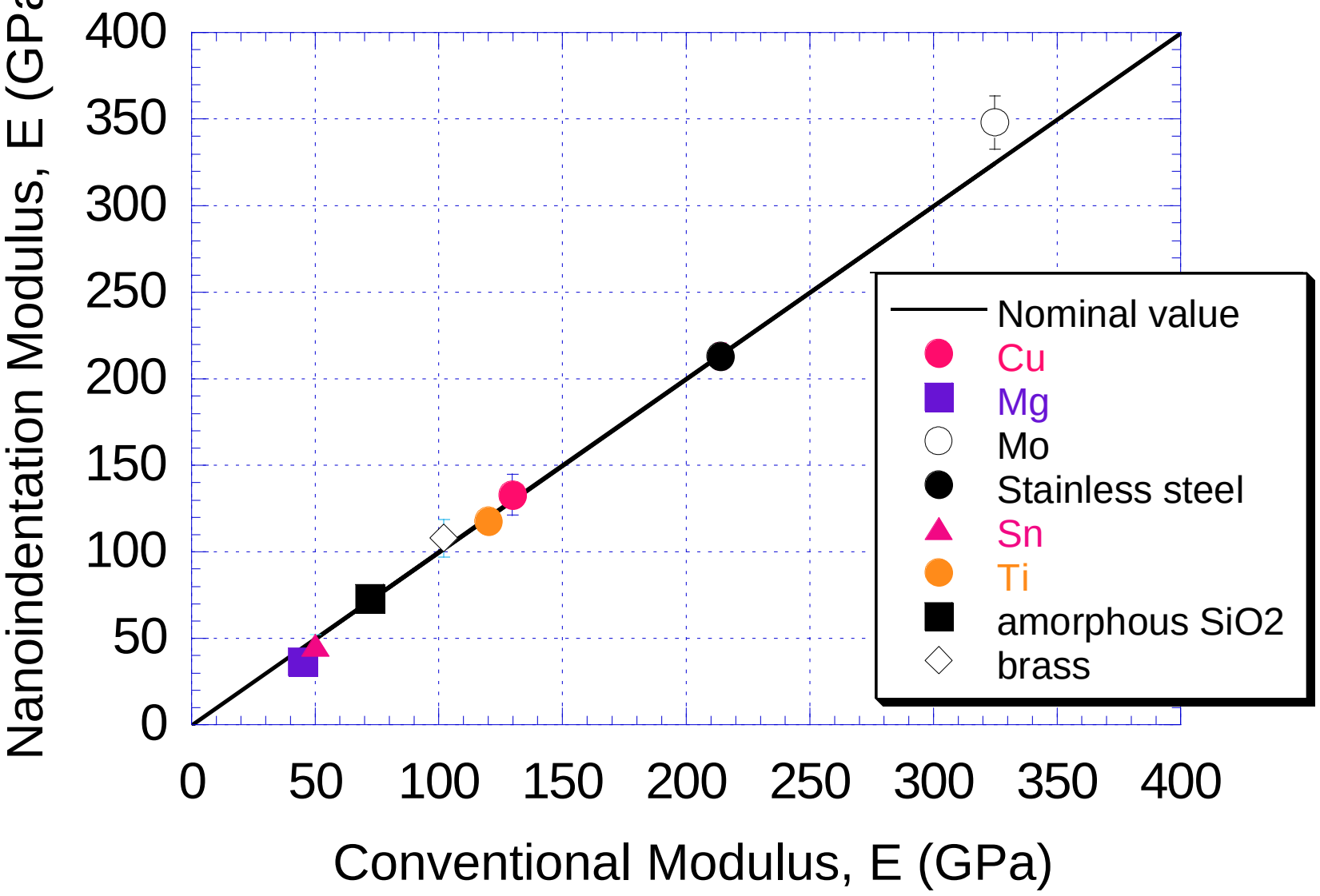


MONOLITHIC MATERIALS



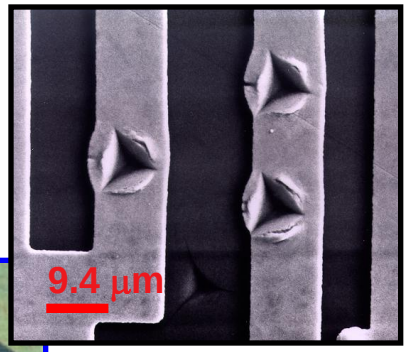


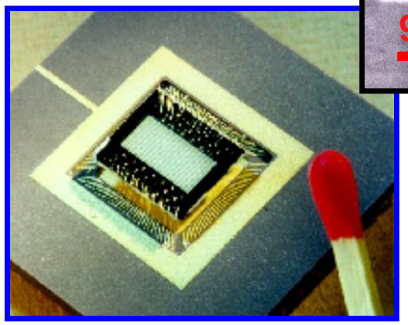
ASSESSMENT OF METHOD

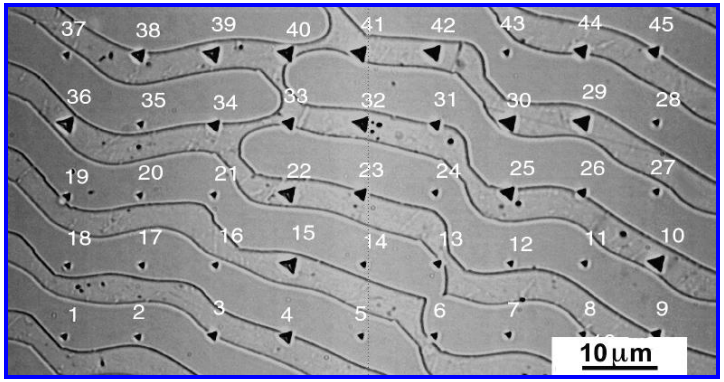


OBVIOUS APPLICATIONS

- *Surface modified materials*
  - *ion implanted*
  - *laser treated*
  - *radiation damaged*
- *Thin films*
  - *semiconductor*
  - *magnetic storage*
  - *optical coatings*
- *Materials of limited size*
  - *powders*
  - *small crystals*
- *Composite & multiphase materials*
- *Hard coatings*
  - *machine tool*
  - *thermal spray*
  - *diamond-like carbon (DLC)*
- *Weldments & joints*

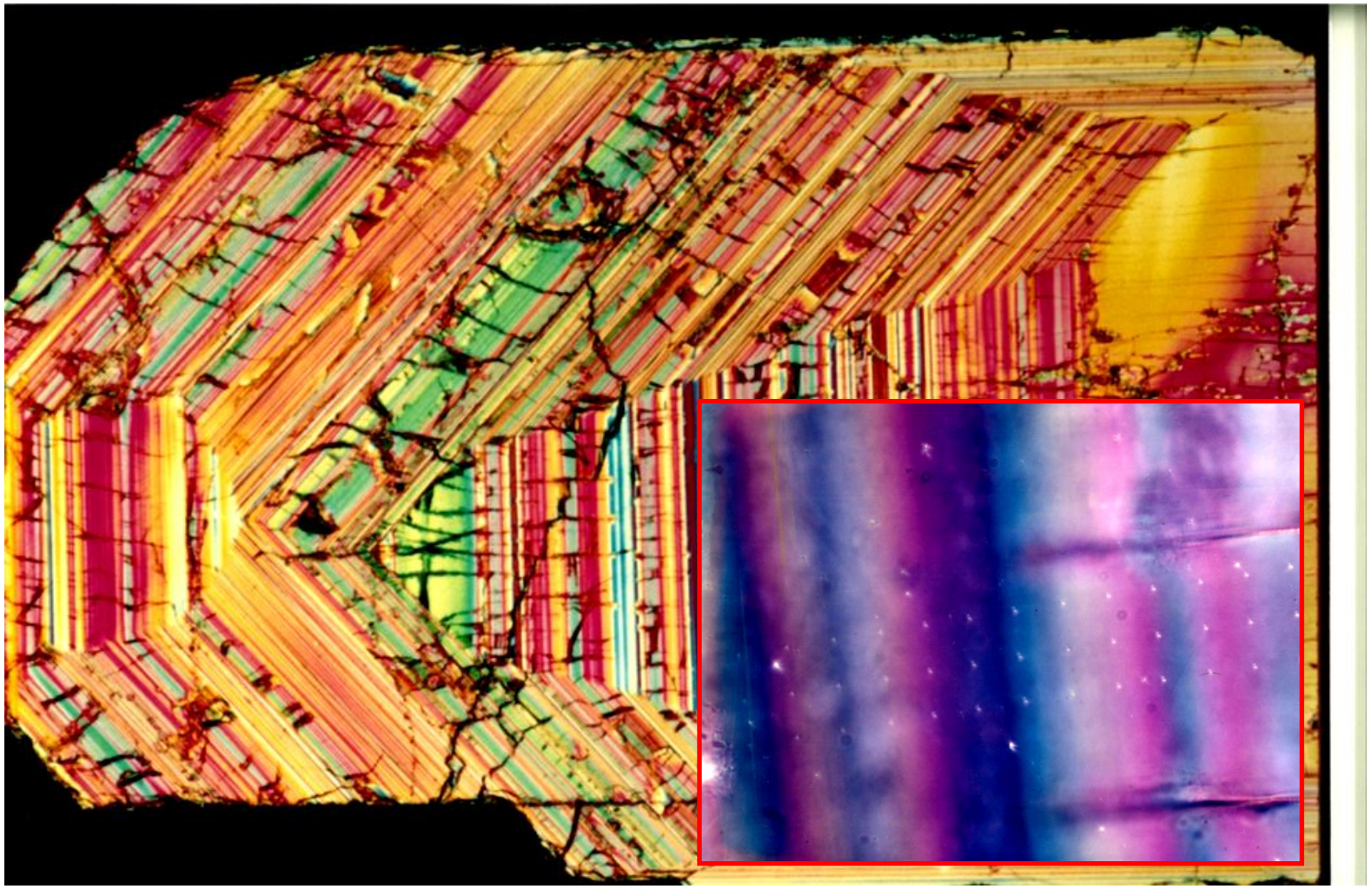






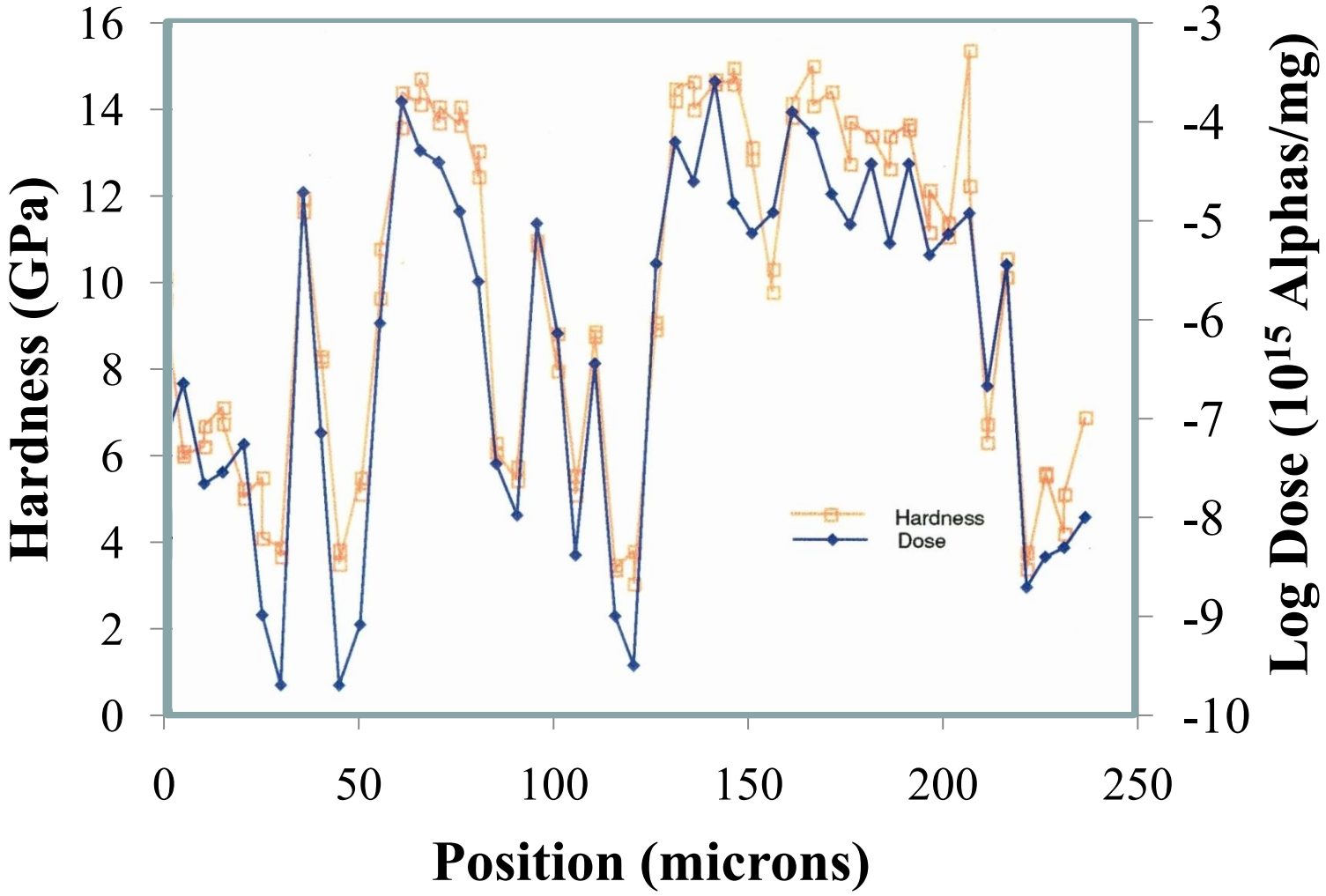
GEOLOGY & PLANETARY MATERIALS

LONG TERM RADIATION DAMAGE



- Chakoumakos, Oliver, Lumpkin and Ewing, *Radiation Effects and Defects in Solids*, 118 (1991)

RADIATION DAMAGED ZIRCON

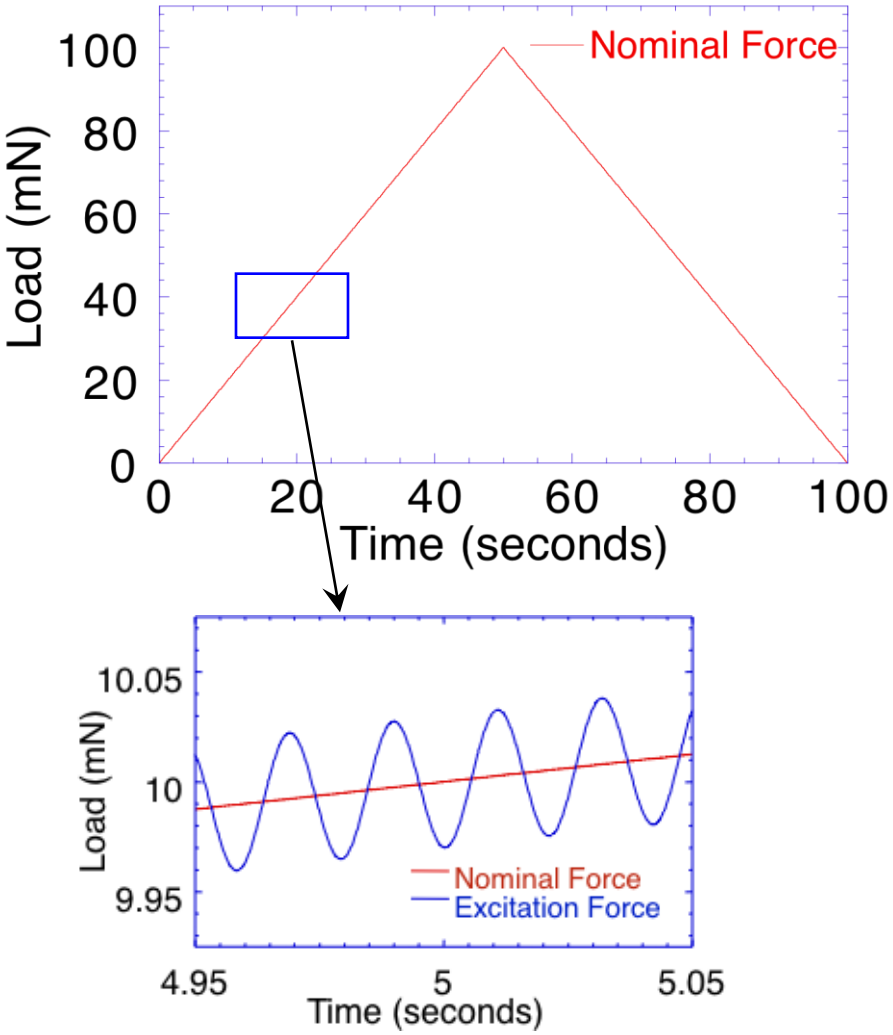


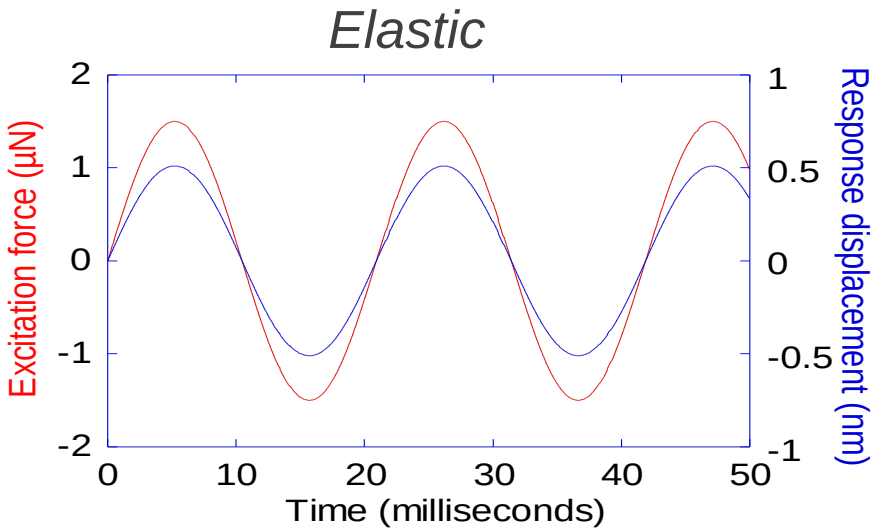
Chakoumakos, Oliver, Lumpkin and Ewing, *Radiation Effects and Defects in Solids*, 118 (1991)

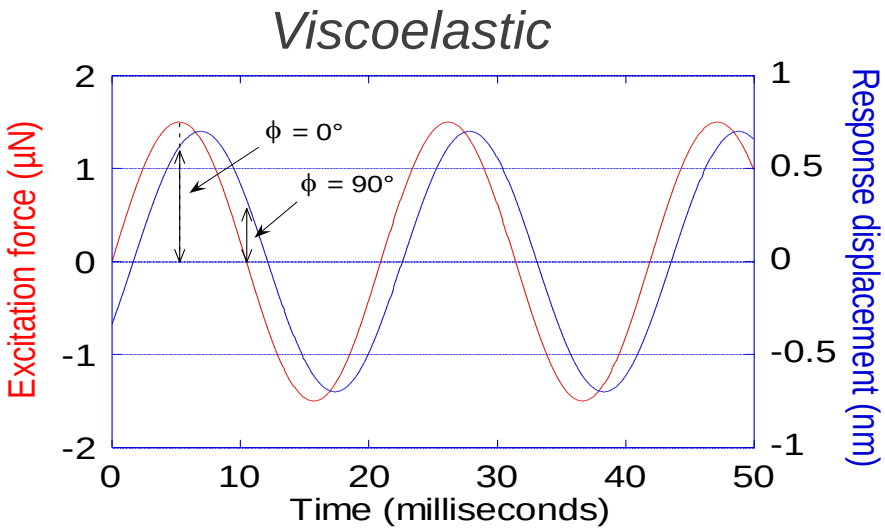
POLYMERS & VISCOELASTIC MATERIALS

CONTIUOUS STIFFNESS MEASUREMENT

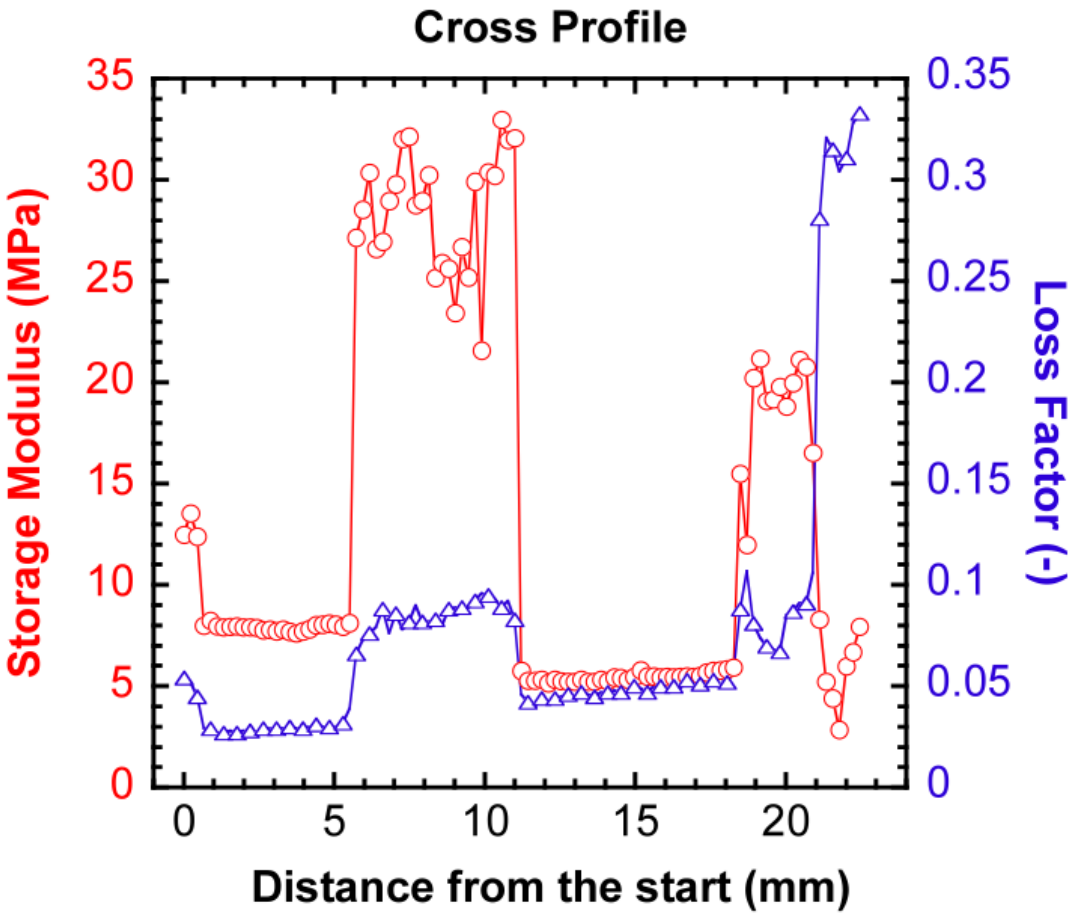
- Oliver & Pethica, *US Patent No. 4,848,141, July 1989*



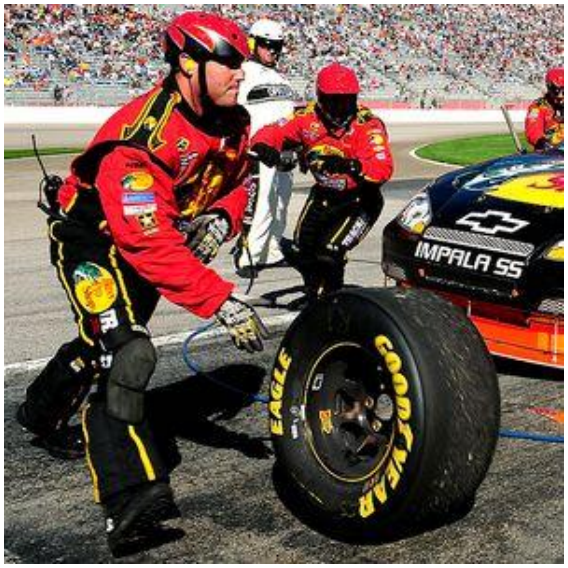


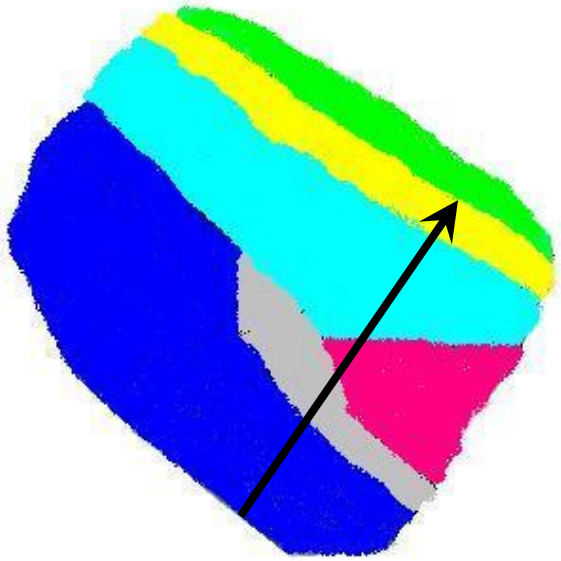


AUTOMOTIVE TIRES



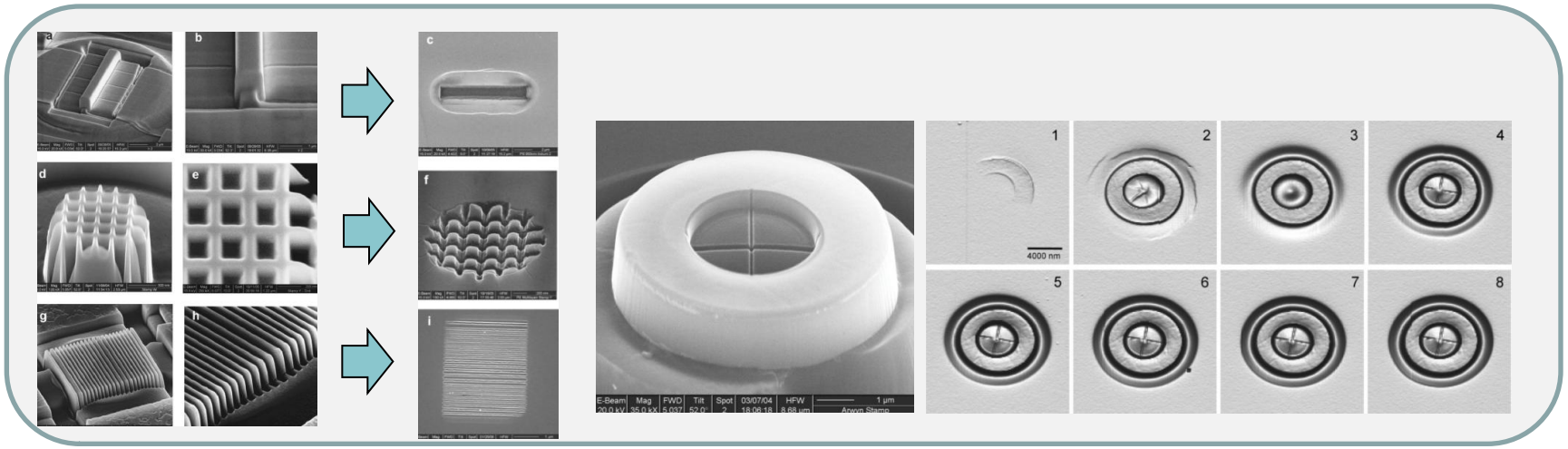
45 µm dia. flat punch; 10 Hz

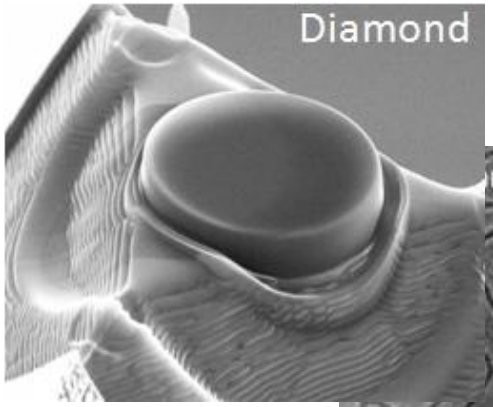




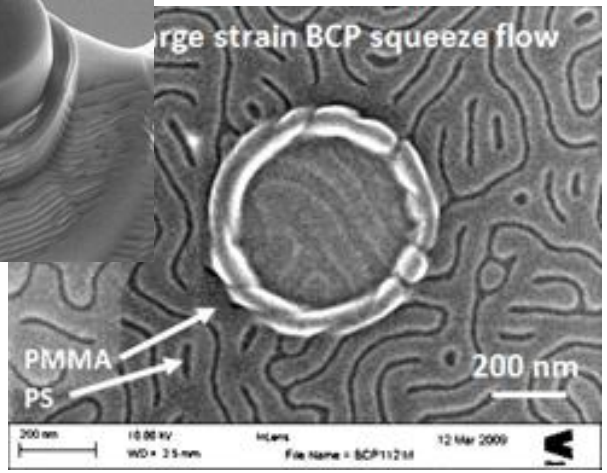
Courtesy of Tom Fleischman and Remi Granier, Goodyear Tire & Rubber Co.

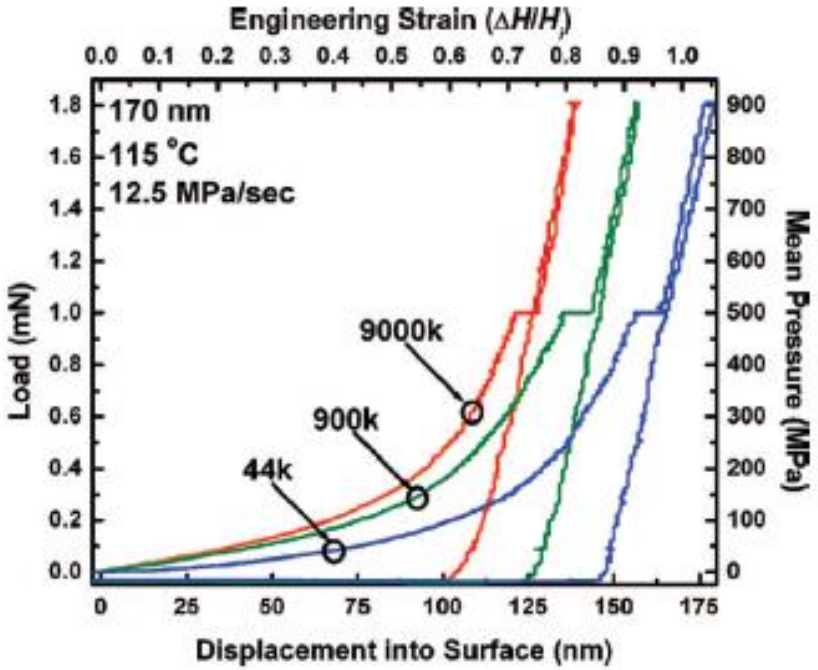
NANOIMPRINT FORMING





Nanometer gap polymer squeeze flow, 115 ° C

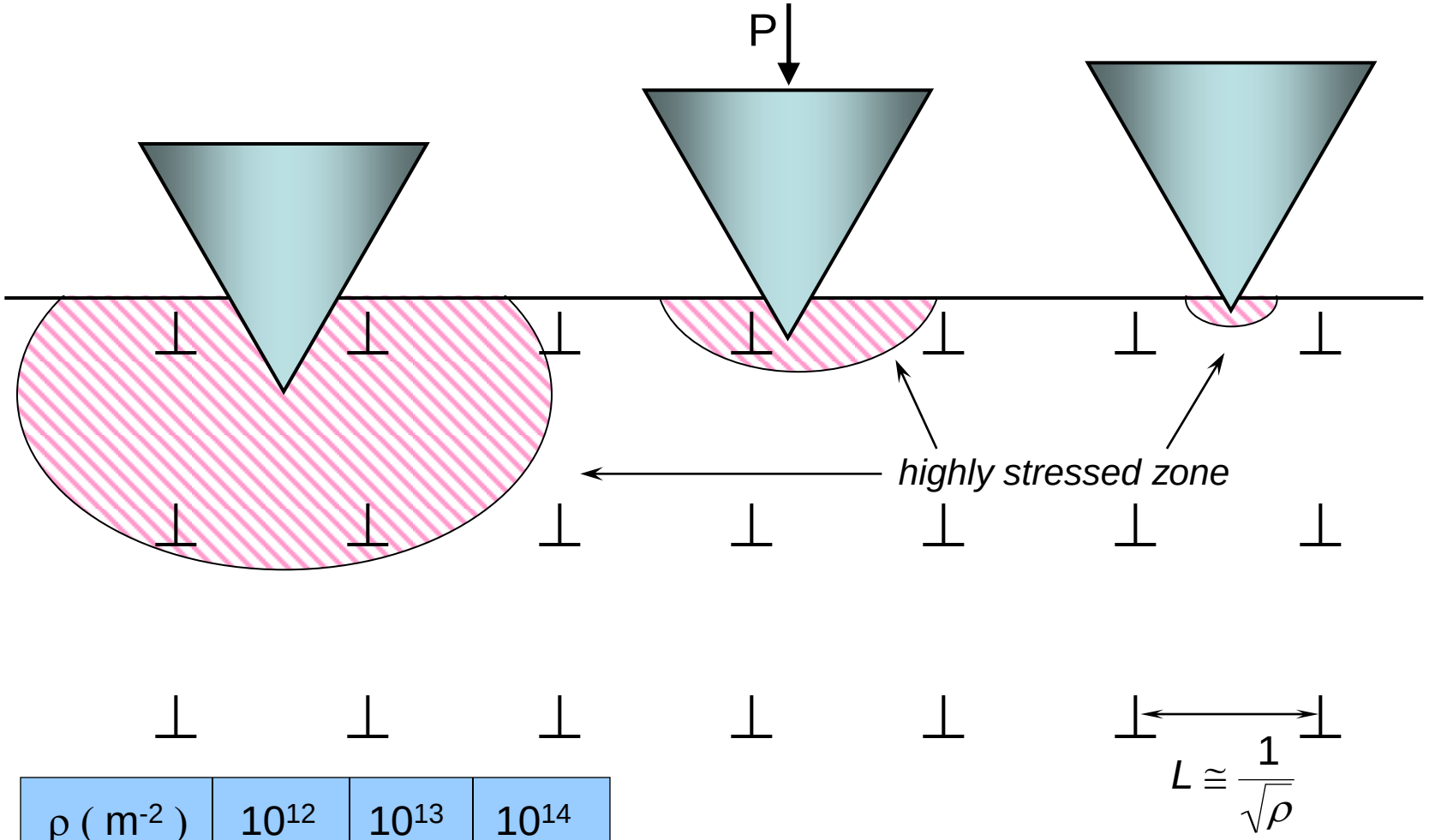




- *Courtesy of Graham Cross, CRANN, Trinity College Dublin*

THE CONTINUUM TO ATOMISTIC BRIDGE

GETTING "BETWEEN" THE DISLOCATIONS

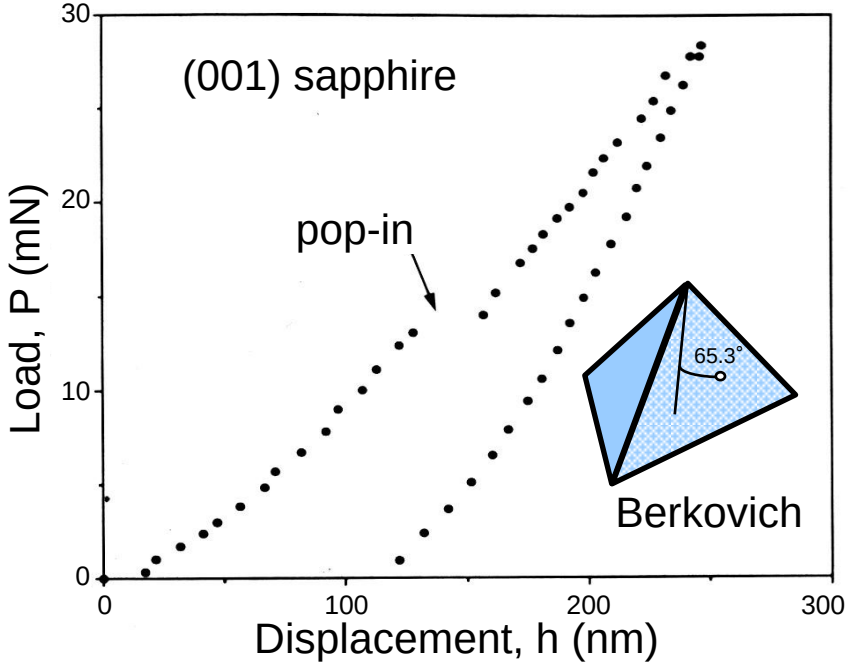


ρ (m^{-2})	10^{12}	10^{13}	10^{14}
L (μm)	1.0	0.3	0.1

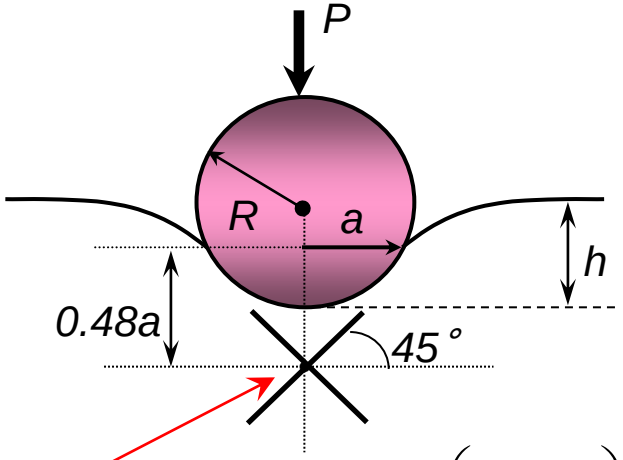
- Shim et al, *Scripta Mater* **59**, 1095 (2008)

POP-IN & THE THEORETICAL STRENGTH

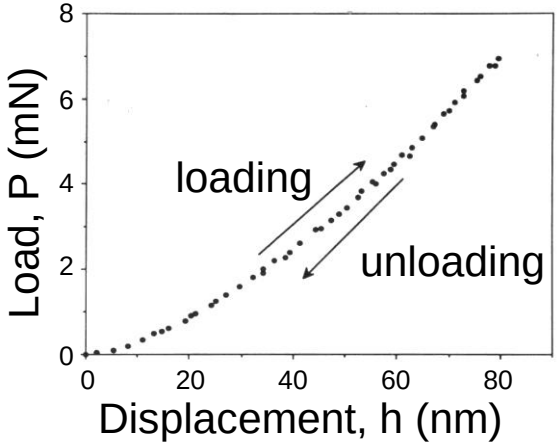
- Page, Oliver, and McHargue *J Mater Res* **7**, 450 (1992)

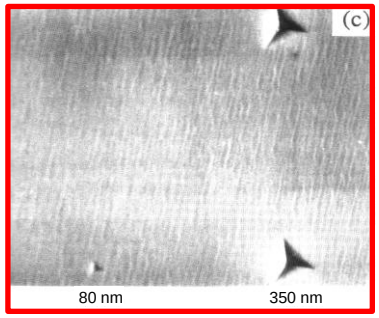


Hertzian Analysis



$$\tau_{max} = 0.31 p_m = 0.31 \left(\frac{6PE_r^2}{\pi^3 R^2} \right)^{1/3}$$



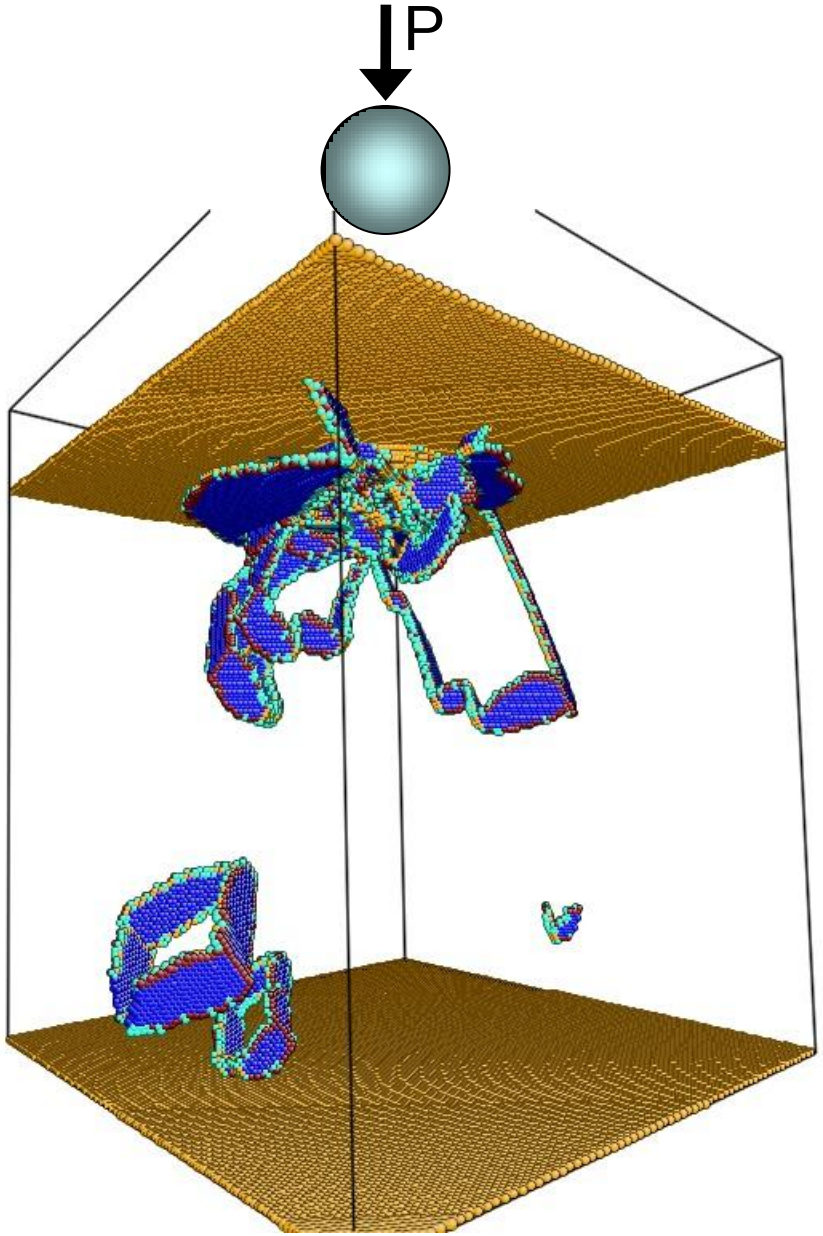


At pop-in:

$\tau_{max} \cong \tau_{theo} \cong G/30 - G/5$

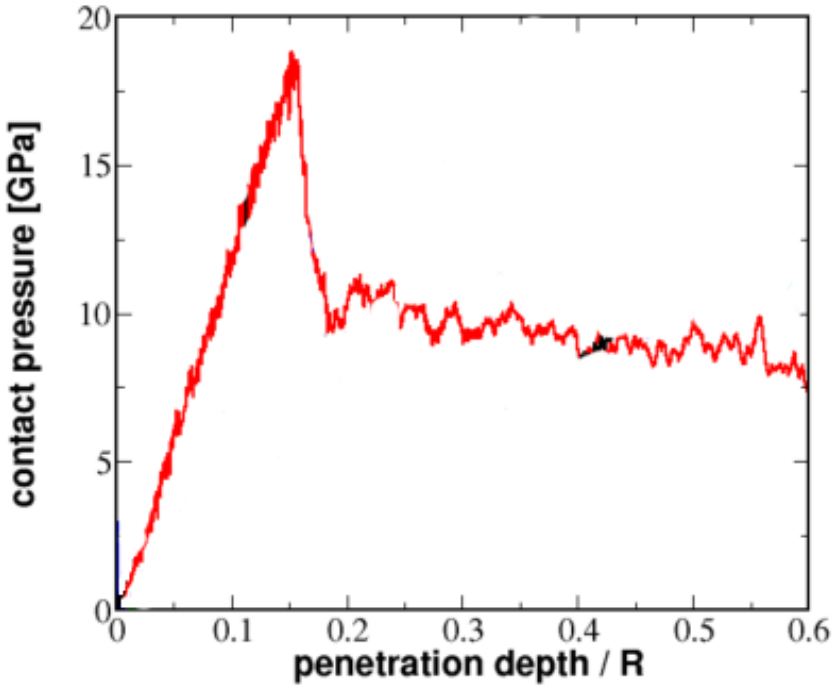
e.g.: Al_2O_3, W, Ni_3Al, Au, Cu, Al, Ni, Mo_5SiB_2, CaF_2, etc.

ATOMISTIC MODELING



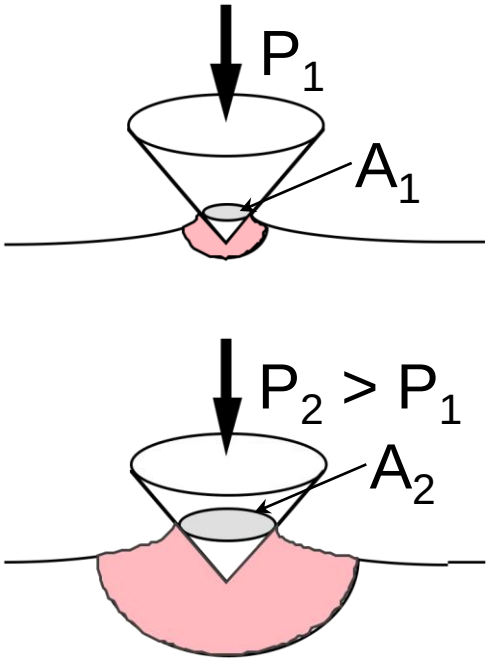
Molecular Dynamics Simulation

(111) copper single crystal
2.5×10^6 atoms; $(30\ nm)^3$
R = 8 nm; h = 2 nm



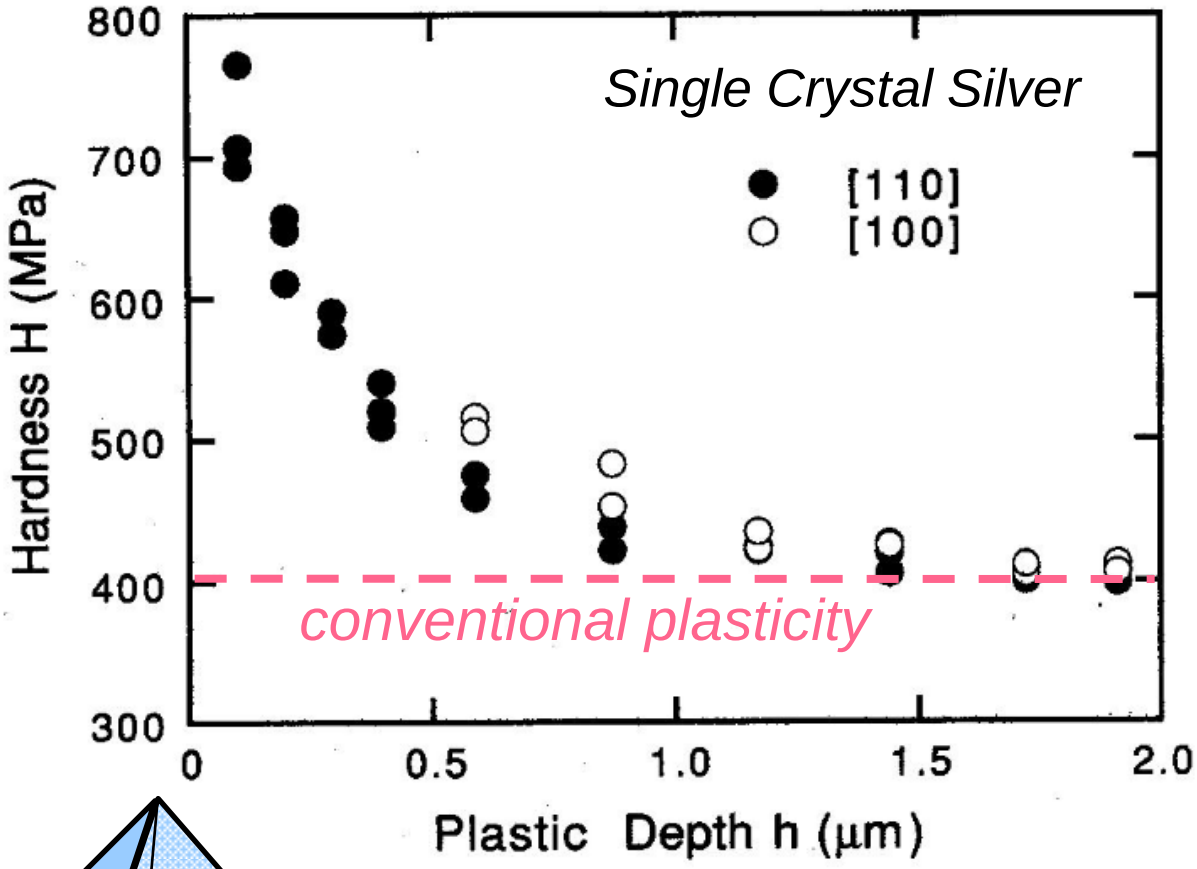
- *courtesy Prof. A. Hartmaier, Ruhr University Bochum*

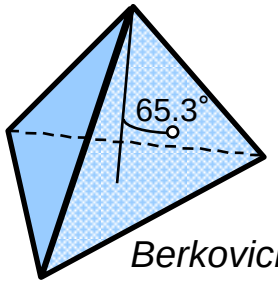
THE INDENTATION SIZE EFFECT



Hardness: $H = \frac{P}{A}$

Ma & Clarke, *J Mater Res.* **10**, 853 (1995)



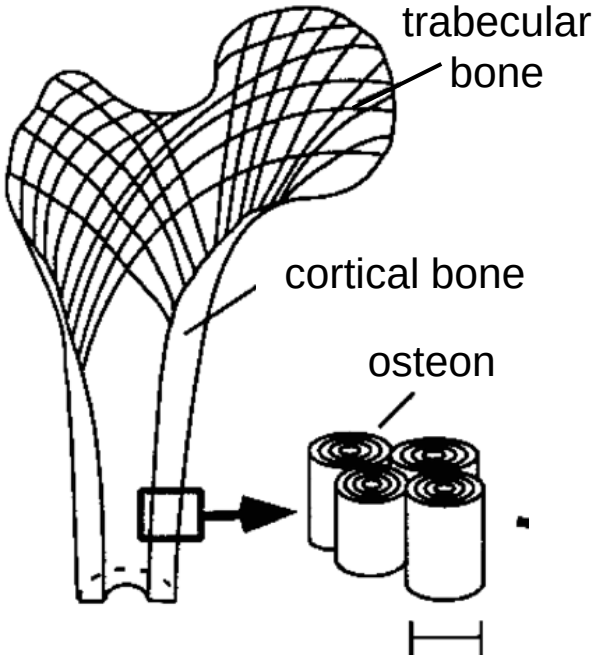


Smaller is stronger !

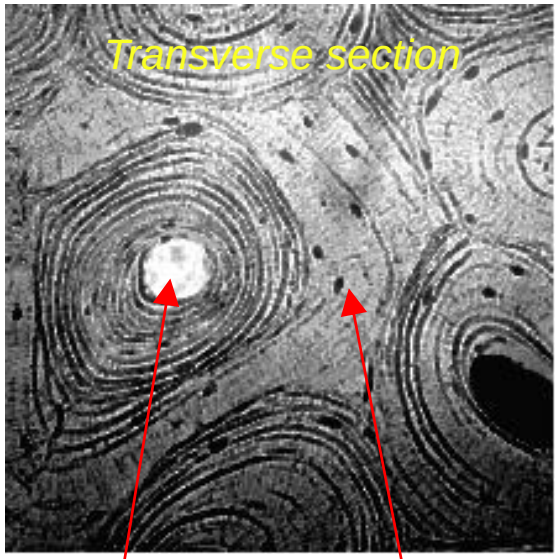
BIOLOGY & MEDICAL SCIENCE

CORTICAL vs. TRABECULAR BONE

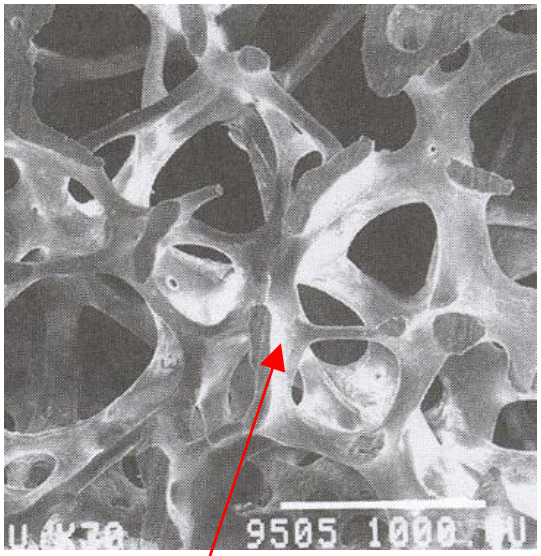
- Turner et al, *J Biomech* **32**, 437 (1999)



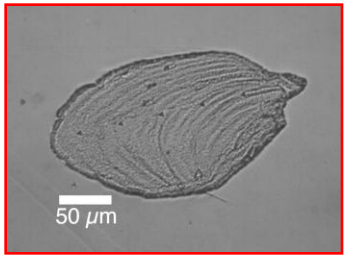
Cortical *(dense, compact)*



Trabecular *(spongy, cancellous)*



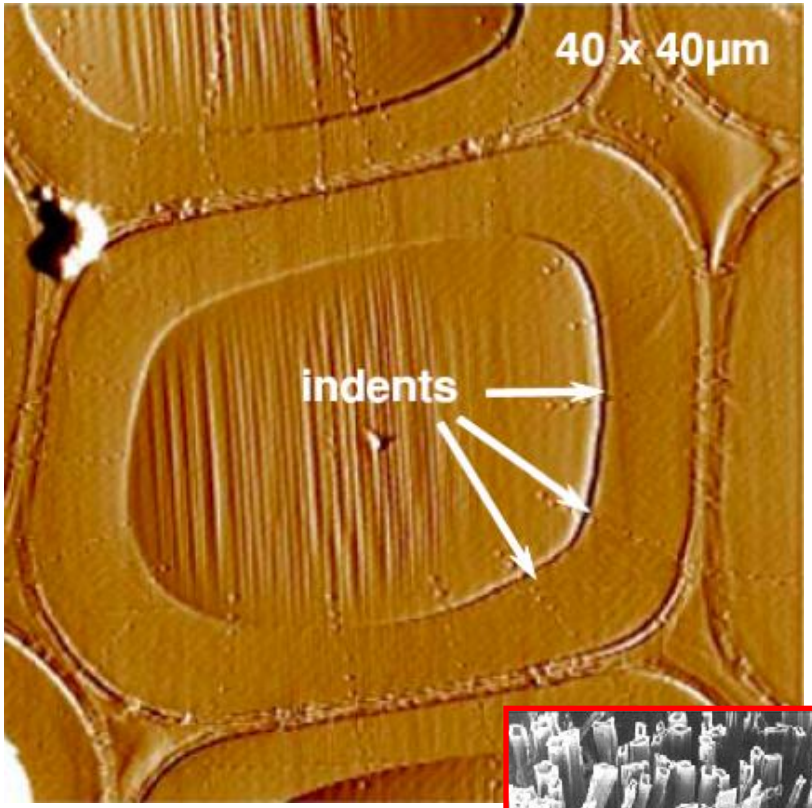
Specimen	E (GPa) Acoustic	E (GPa) Nanoindentation
trabecular	17.2	18.2 ± 0.5
cortical (transverse)	15.1	16.6 ± 0.3
cortical (longitudinal)	20.9	23.4 ± 0.6

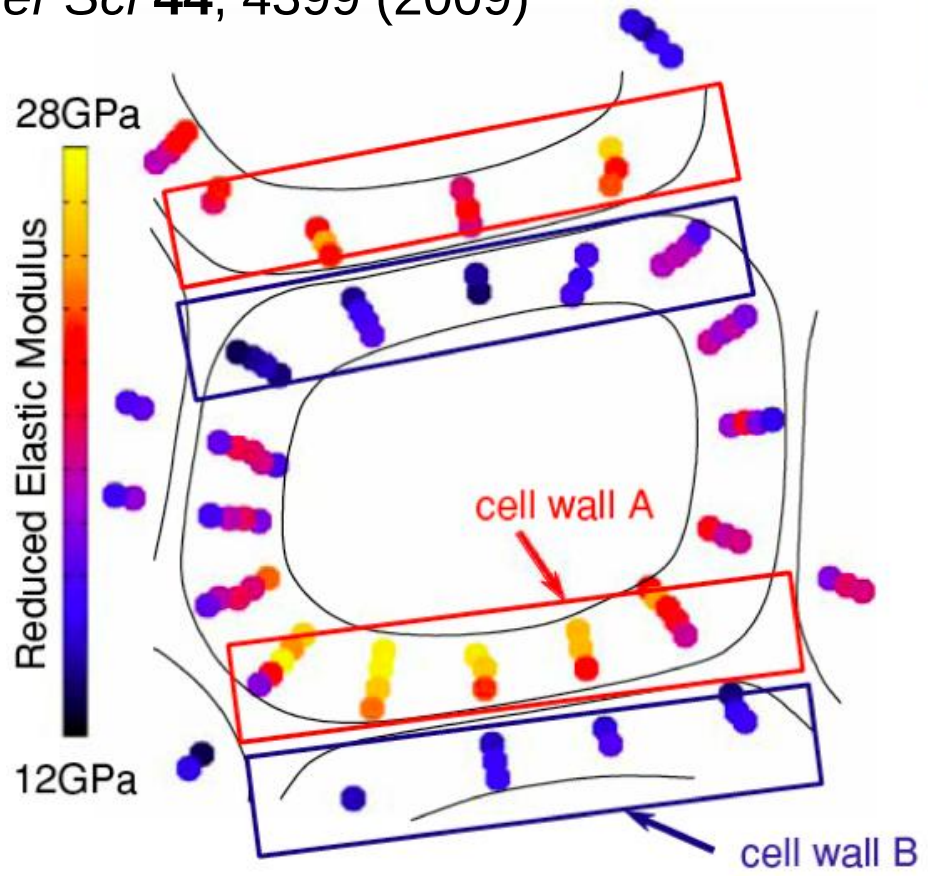


transverse section

WOOD CELL WALLS

• Konnerth et al, *J Mater Sci* **44**, 4399 (2009)





cellulosic microfibrils

SOME UNUSUAL APPLICATIONS

HYDROTHERMAL VENT GASTROPODS

“Protection mechanisms of the iron-plated armor of a deep-sea hydrothermal vent gastropod”

- Yao et al, *PNAS 107, 987 (2010)*

HYDROTHERMAL VENT GASTROPODS

“Protection mechanisms of the iron-plated armor of a deep-sea hydrothermal vent gastropod”

C. Squamiferum (2001)

• Yao et al, *PNAS 107, 987 (2010)*

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Vent crab

- Yao et al, *PNAS 107, 987 (2010)*

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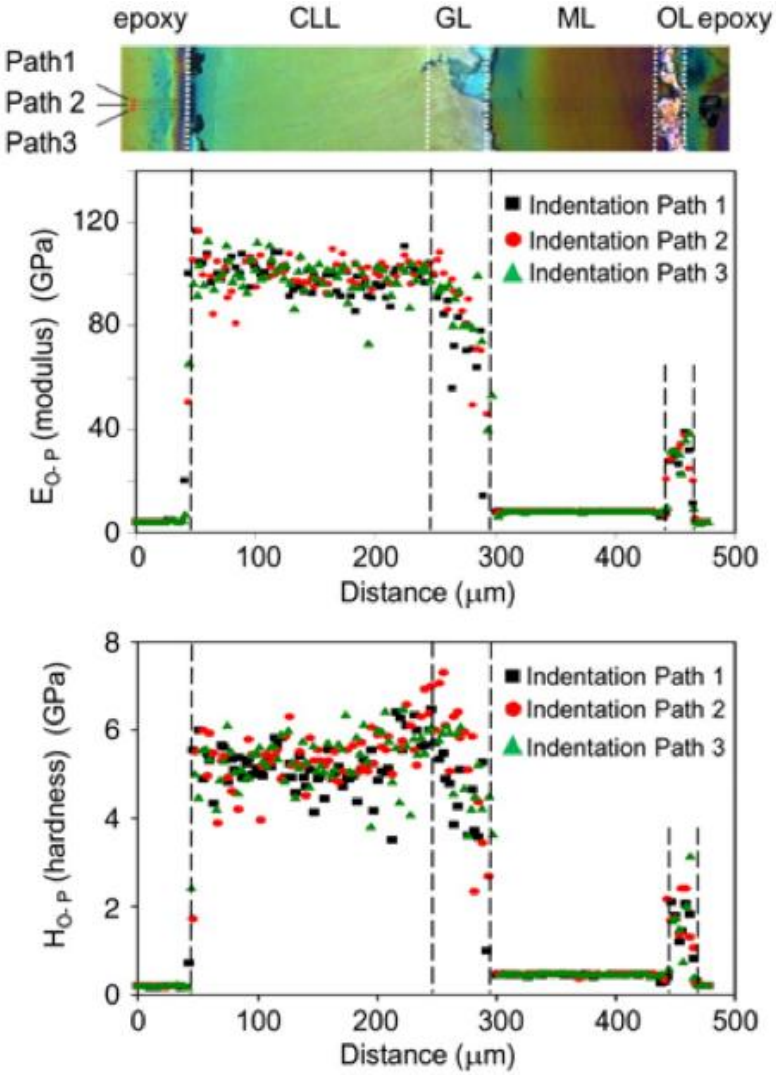

- Yao et al, *PNAS 107, 987 (2010)*

HYDROTHERMAL VENT GASTROPODS

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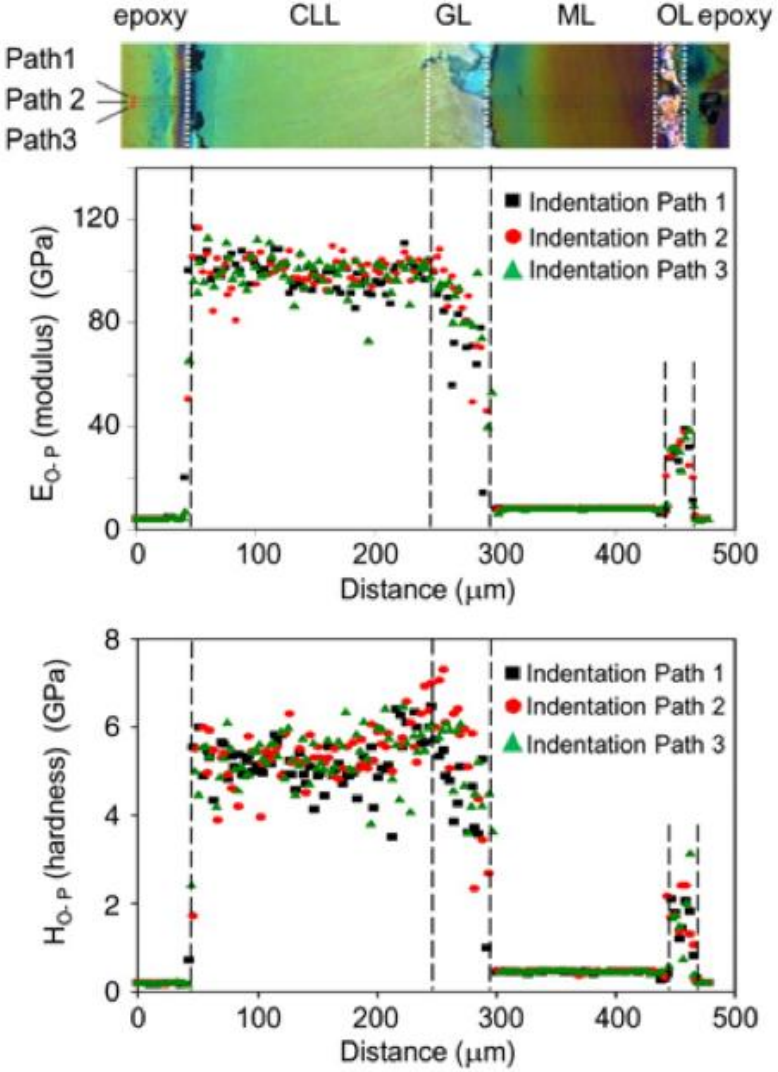
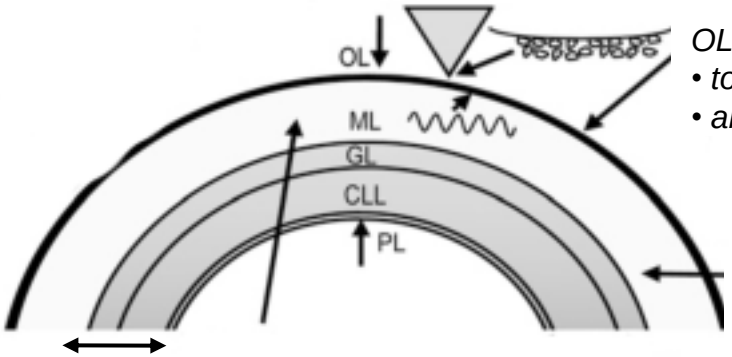
C. Squamiferum (2001)

Vent crab



Armor Mechanics (FEM)



OL: Fe-based granular
- *tough*
- *abrades penetrator*

ML: organic
- *compliant*
- *arrests cracks*
- *protects brittle IL*

Il: calcified shell
- *structural support*
- *fatigue/bendiing resistance*

- Yao et al, *PNAS 107, 987 (2010)*

THE LAACHER SEE HYPOTHESIS

“Testing the ‘Laacher See hypothesis’: tephra as dental abrasive”

- Riede & Wheeler, *J Archaeological Sci* **36**, *2384 (2009)*

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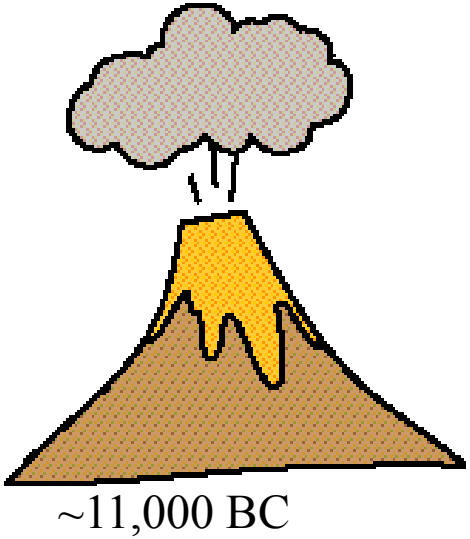

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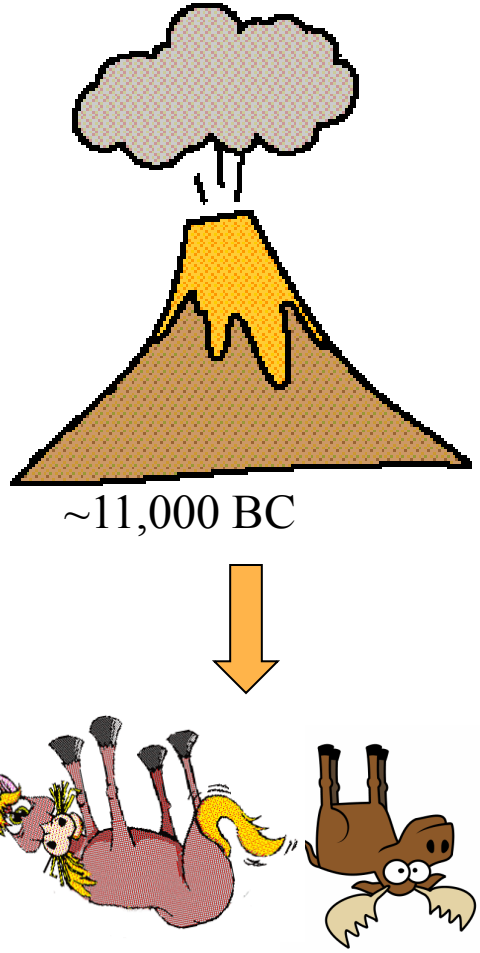

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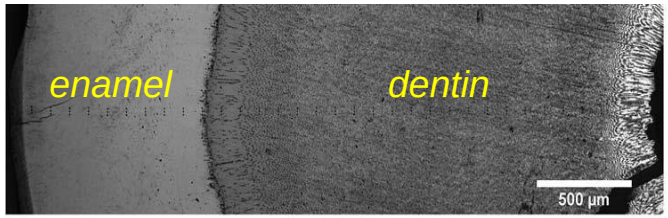
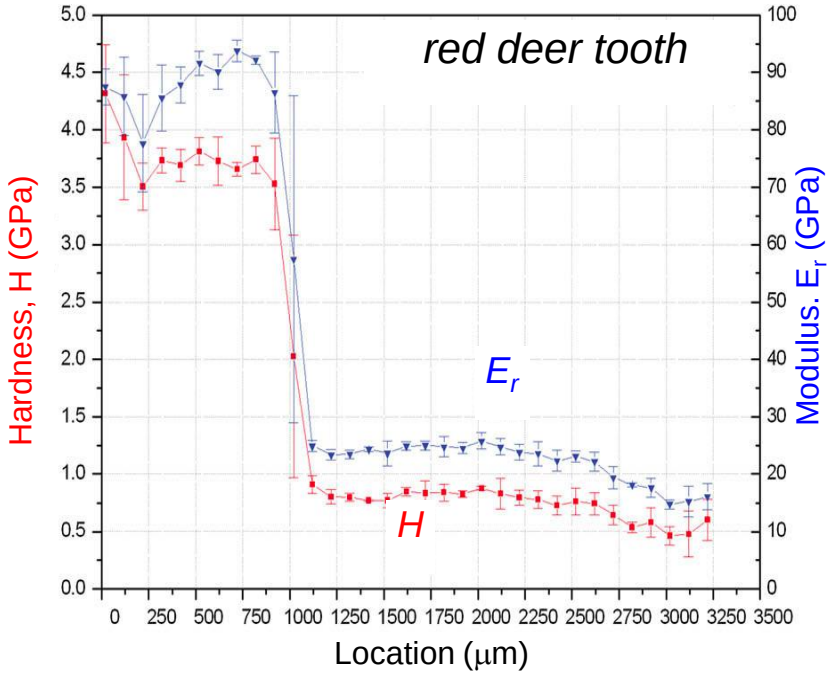
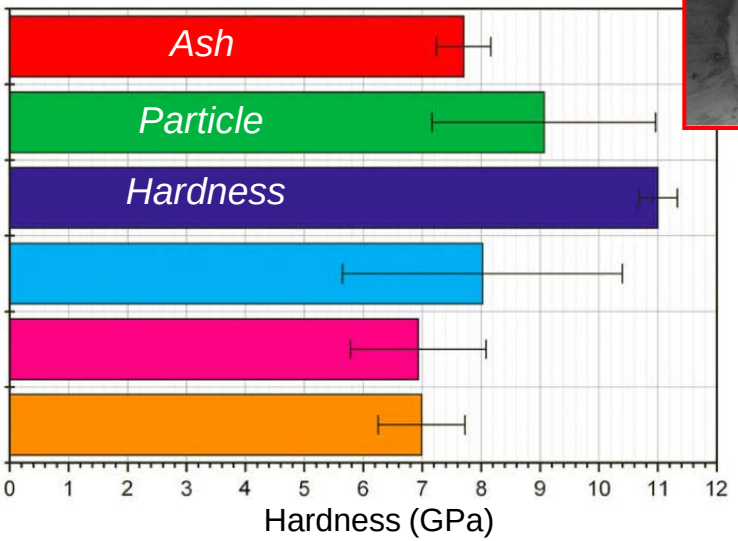
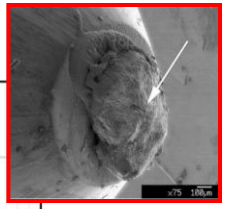
TESTING THE HYPOTHESIS

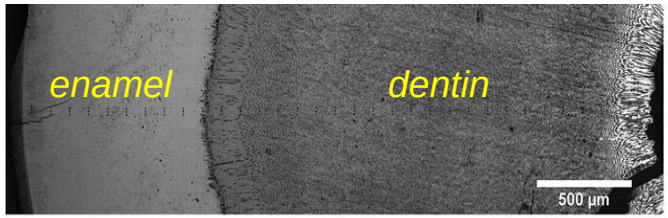


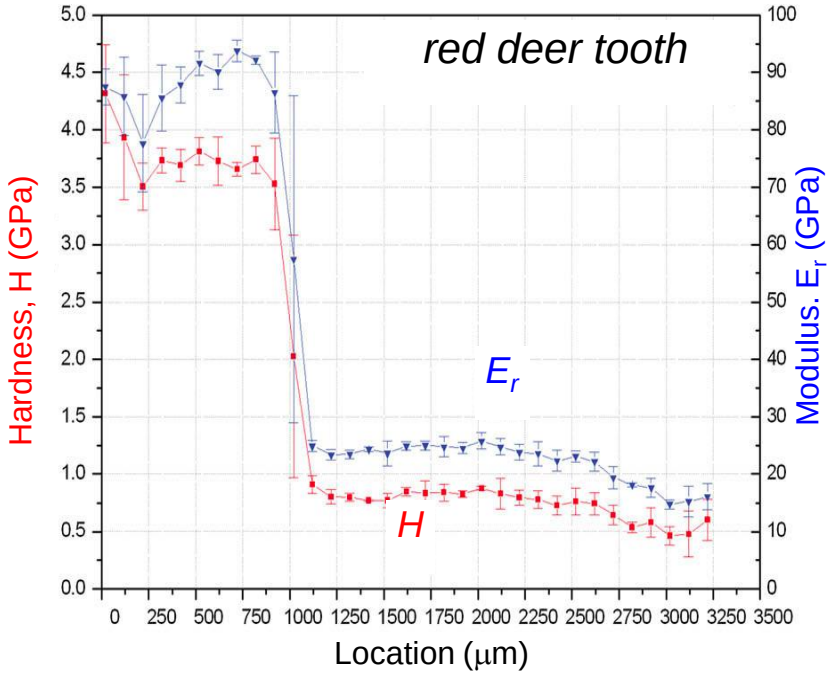


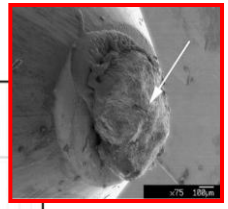


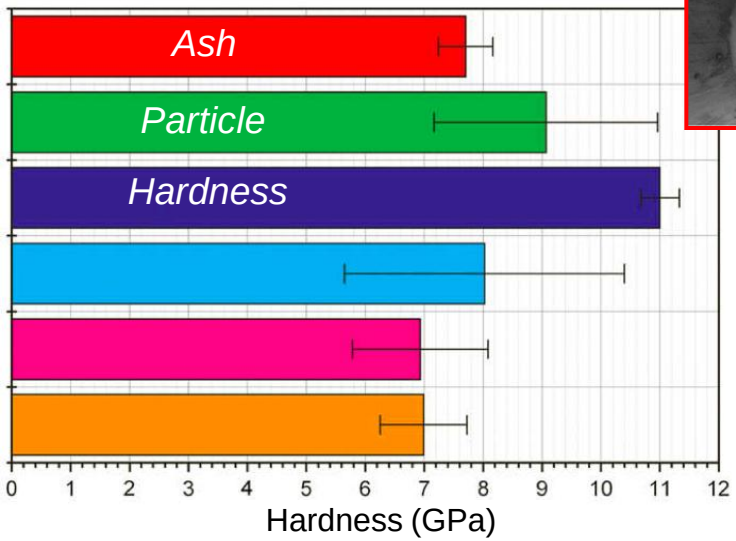


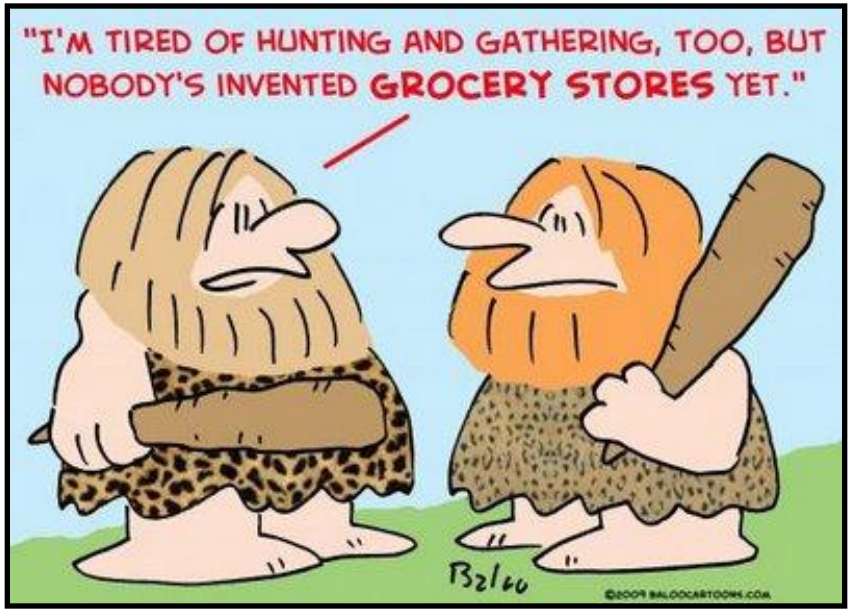
TESTING THE HYPOTHESIS











Thanks for your attention !!