Reliability Issues

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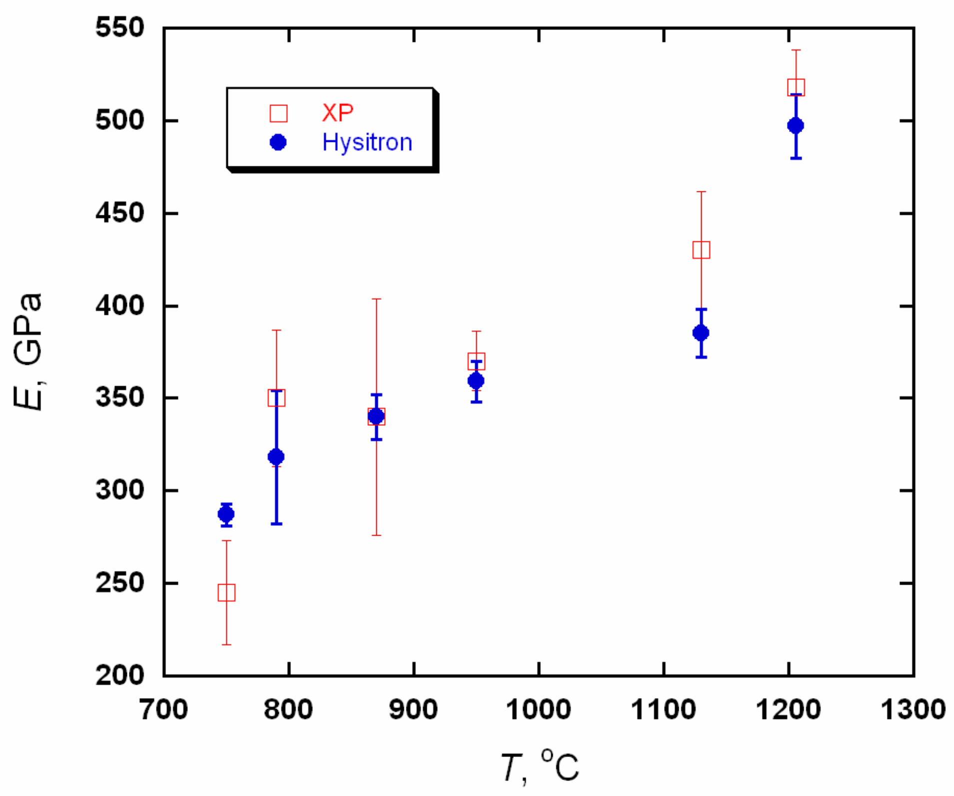
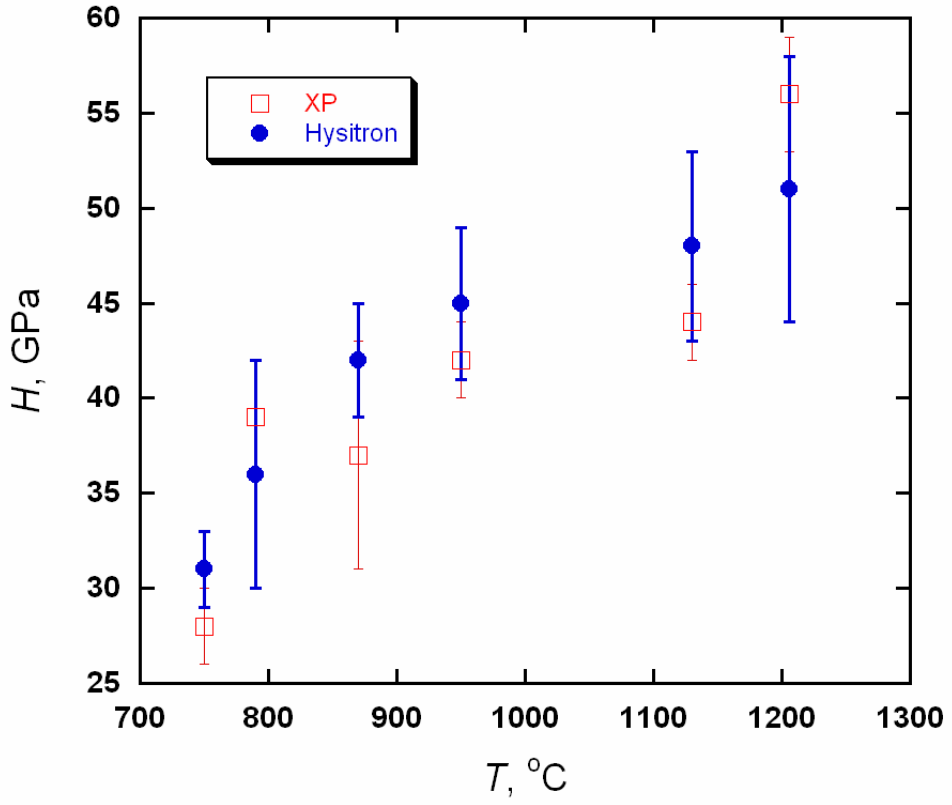
- State-of-the-Art
- Current Areas of Investigation
- Barriers to Accurate Measurement (or understanding)
  - Size Effect
  - Yield Points
  - Adhesion
  - Work Hardening
- Road Map

State-of-the-Art Measurements

Thermal Plasma CVD SiC





Error bars = 1σ

MTS XP Continuous Stiffness ~ average of 3-5 runs (500 pts. Each)

Hysitron TriboIndenter ~ average of 12 runs

From Fall 2004 MRS

Symposia R: Nanoindentation and Nanomechanics
V: Size Effects in Plasticity

What are the hot topics addressing:
"Mechanical Behavior Aspects in Design at the Nanoscale"?

S:	Size Effects	:	32 papers
T:	Temp. or Viscoelasticity	:	15 papers
M:	Multiscale Modeling	:	14 papers
A:	Adhesion or Surface Energy	:	11 papers

(also highlighted at the Nanomechanics Workshop in Asilomar)

What Barriers do these represent to accurate nanoscale measures?

S: Size Effects; T: Temperature or Viscoelasticity; A: Adhesion or Surface Energy

Polymers	Metals & Semiconductors	Ceramics
$S(\gamma_s, W_A, p_o) \rightarrow E, \sigma_{ys}, K_{IC}$	$S(\gamma_s, \perp, \Sigma\perp, p_o) \rightarrow E, \sigma_{ys}, K_{IC}$	S(?)
$T(W_A, t_c) \rightarrow E, \sigma_{ys}, K_{IC}$, da/dt	$T(\perp, \Sigma\perp, t_c) \rightarrow \sigma_{ys}, K_{IC}$, da/dt	$T(\perp, t_c) \rightarrow K_{IC}$, da/dt
ΔT(drift) → (all)	ΔT(drift) → (all)	ΔT(drift) → (all)

Nomenclature: Properties

E, σ_{ys}, K_{IC} are relatively time-independent

da/dt represents time dependent creep, fatigue, stress corrosion

Fundamental Variables

γ_s – surface energy; W_A – work of adhesion

p_o – pressure; $\perp$ - dislocation nucleation or image force

$\Sigma\perp$ - work hardening or strain gradient plasticity

t_c – characteristic time for diffusion, cross-slip, corrosion

Challenges: Small Length Scales

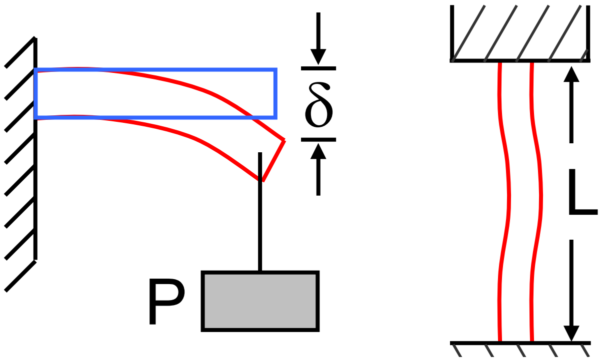
- Importance
- Indentation Size Effect
  - Metals
  - Polymers?
- Film Fracture
- Nanoparticles

top down

Length Scales for Property Prediction

Rigidity

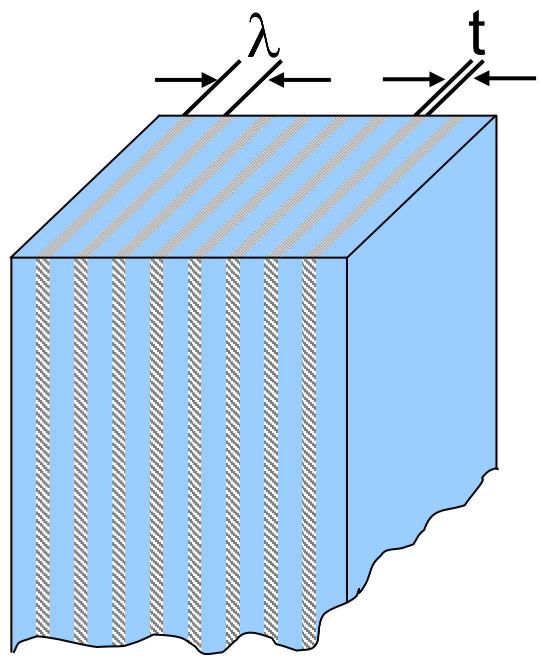
Macro



$$P \sim \alpha_i \left(\frac{\delta}{L^3} \right) \sim \alpha_i \left(\frac{1}{L^2} \right)$$

α_i has $N \cdot m^2$ units

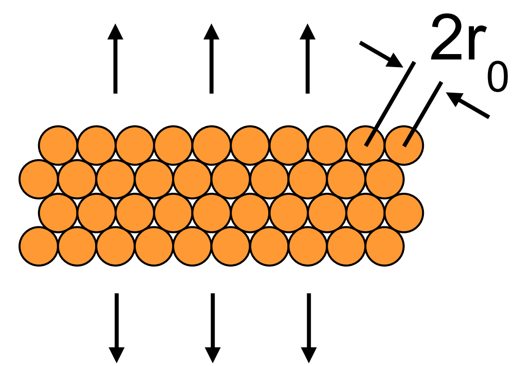
Micro



$$\sim \alpha_i \left(\frac{1}{\lambda \cdot L} \right)$$

$\lambda =$ laminate spacing

Atomistic



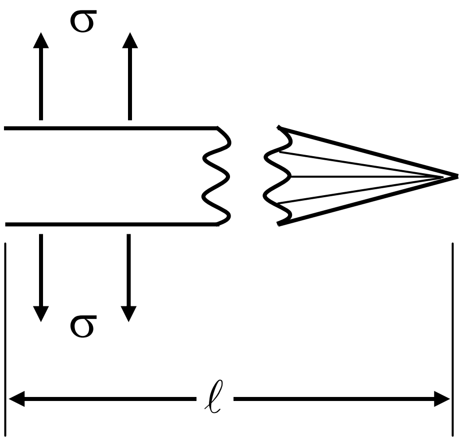
$$\sim \alpha_i \left(\frac{A}{r_0^4} \right) \cdot \left(\frac{\delta}{L} \right)$$

$A =$ cross $-$ section

Length Scales for Property Prediction

Fracture Energy, G

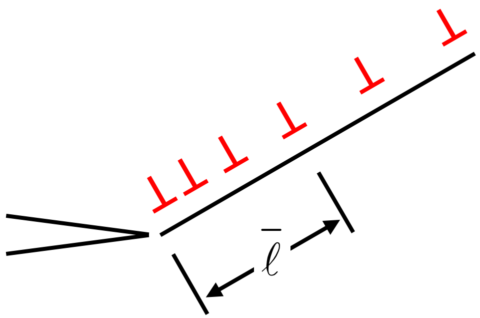
Macro	Micro	Atomistic



$$G \cong \frac{\sigma^2 \ell}{E}$$

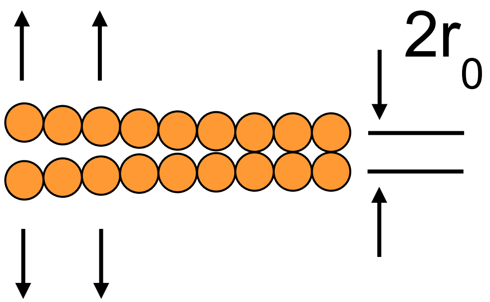
ℓ = crack

E = modulus



$$\sim \frac{Eb^2}{\bar{\ell}}$$

λ = average dislocation spacing



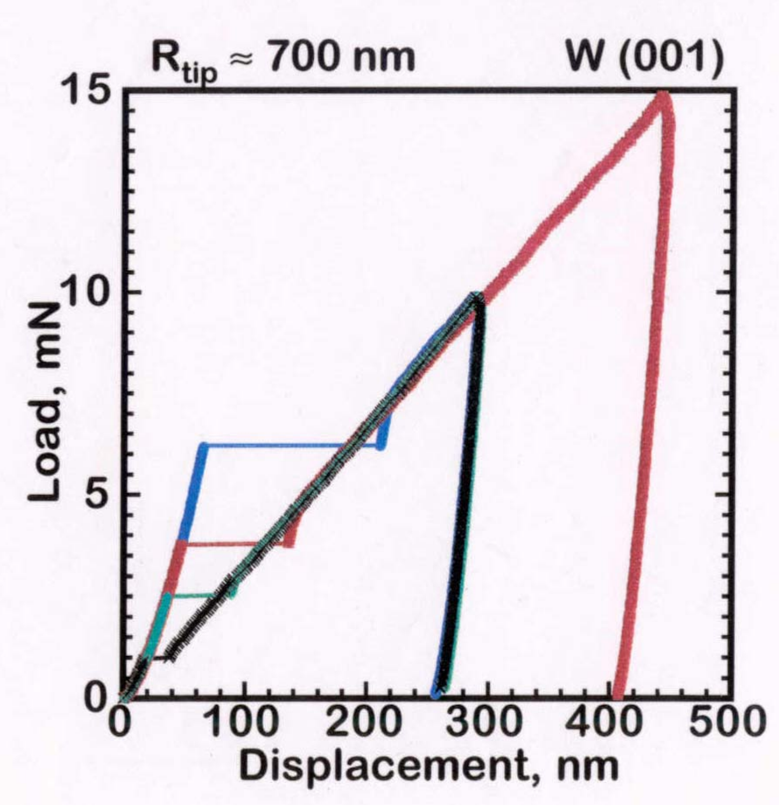
$$\sim 2\gamma_s \sim \frac{\alpha_i}{r_0^3}$$

γ_s = surface energy

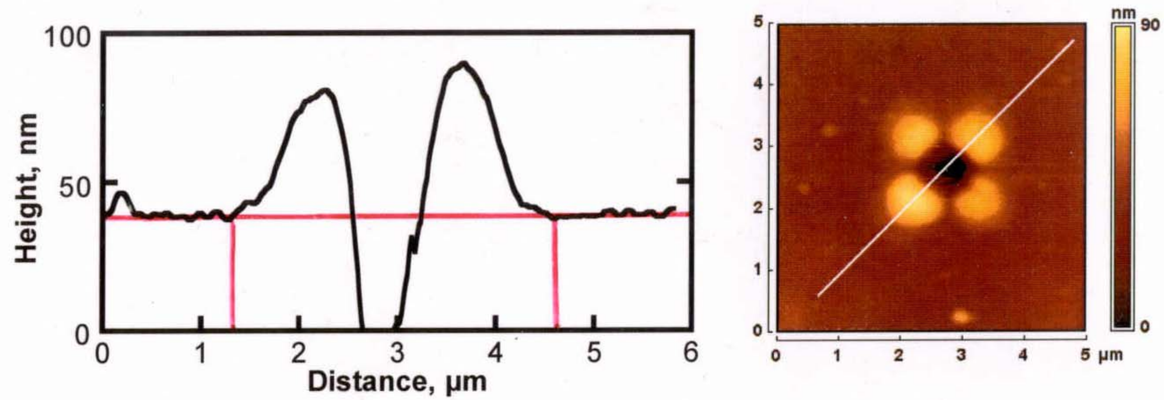
r_0 = atomic radius

Single Crystal Indentation : Master Curve

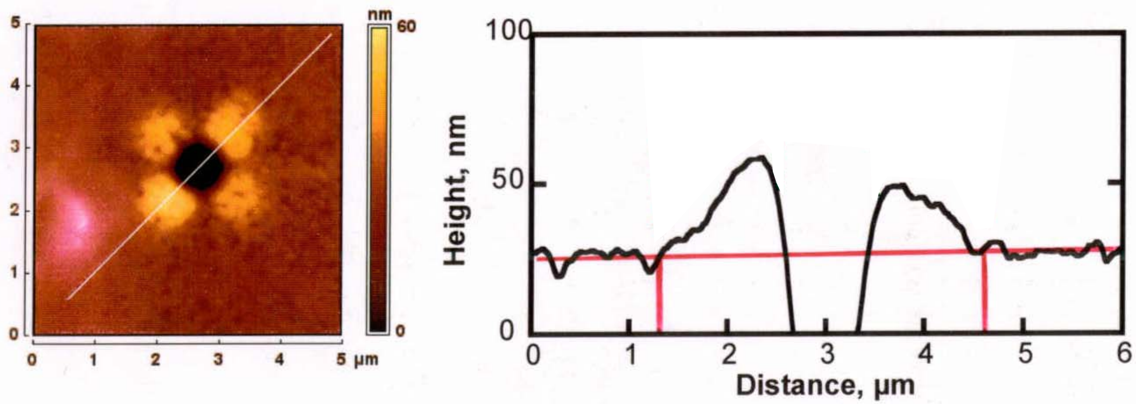
- Excursion displacement scales with critical load.
  - Initial displacement follows from elastic Hertzian contact
  - Final displacement determined by elastic/plastic master curve



Plastic Zone Around <100> Fe-3%Si Before and After Thermal Oxide Treatment

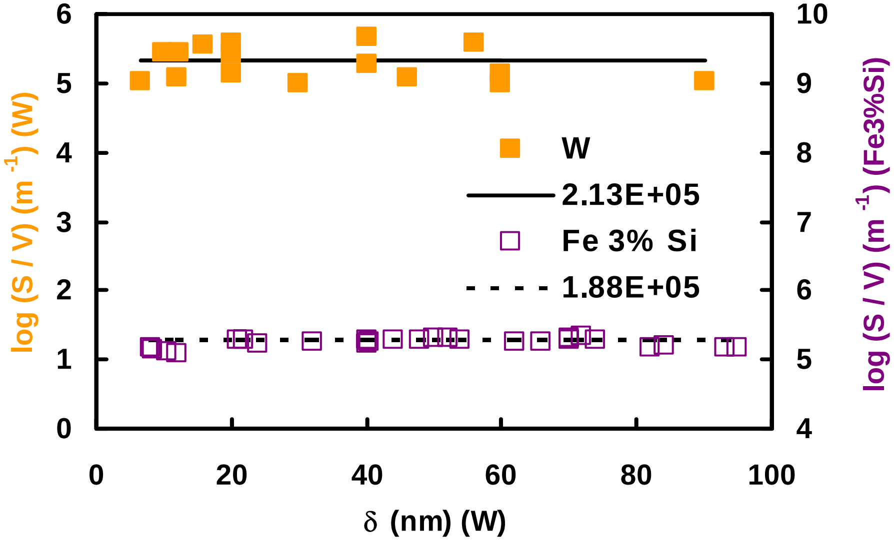


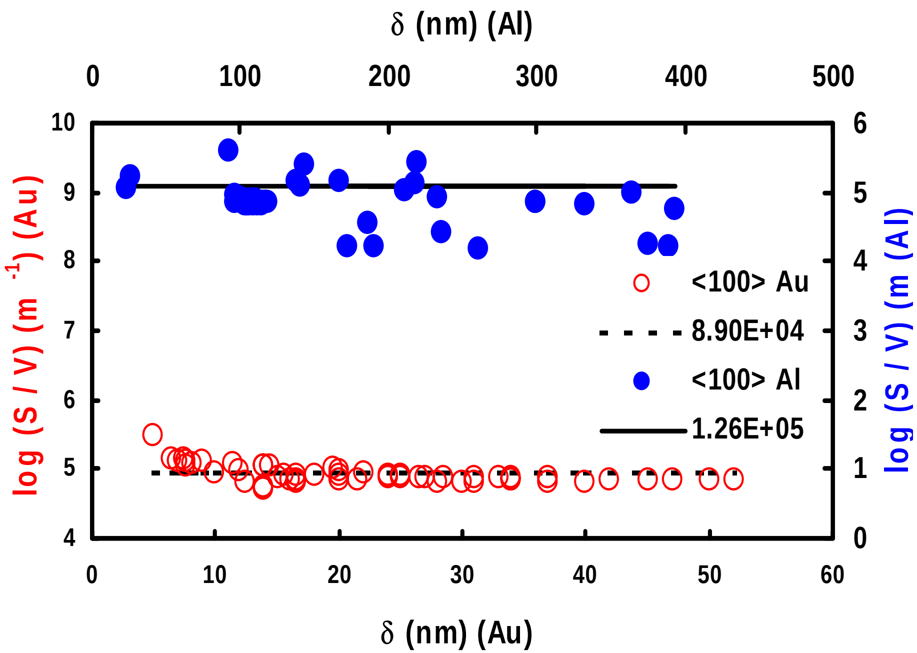
Before Oxide Treatment



After Oxide Treatment

Volume-to-Surface Ratio as a Length Scale





Gerberich, W.W., *et al.* , *J. Appl. Mech.*, **69**, 433-442, (2002)

Continuum Pile-up

(1) $$h(r) \cong (1-\upsilon)\frac{\sigma_{ys}}{E}\left[\frac{c^3}{r^3}\right]\left[\frac{\pi r}{2}\right]$$

$h(r) =$ height as a function of distance

$\sigma_{ys}/E =$ yield to modulus ratio

$c =$ plastic zone radius

(2) $$h(r = a) \cong nb$$

where n ⊥'s are released to form pile up from (1) + (2)

(3) $$n_{CR} = \frac{\pi(1-\upsilon)\sigma_{ys}}{2bE}\cdot\frac{c^3}{a^2}$$

since $\ell_s = V/S = 2c^3/3a^2$, (3) becomes

(4) $$n_{CR} = \frac{3\pi\ell_s(1-\upsilon)\tau_{ys}}{4b\mu}$$

$$\frac{\tau_{ys}}{\mu} \sim \frac{\sigma_{ys}}{E}$$

--Eshelby (1951) --

(5) $$n_{pile-up} = \frac{\pi\ell_s(1-\upsilon)\tau_{ys}}{2b\mu}$$

ISE – Volume-to-Surface Ratio

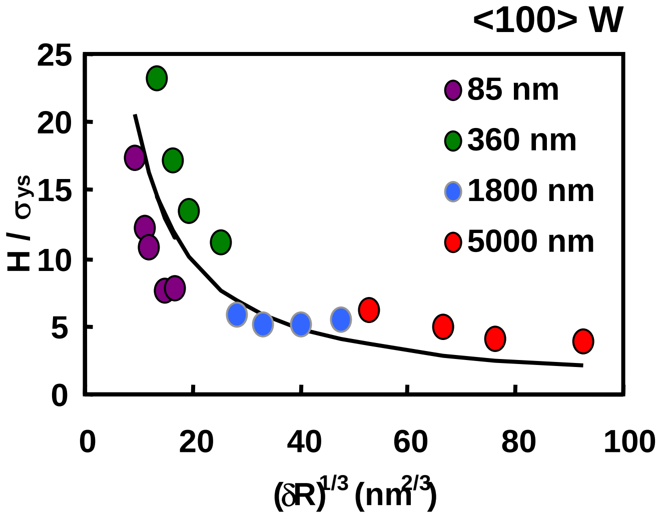
Observed a relationship between the volume of deformation and generated surface area

Combined with plastic zone model

$$H = \sigma_{ys}\left(\frac{V}{S}\right)^{2/3}\frac{1}{(3\delta R)^{1/3}}$$

$$\frac{V}{S} \longrightarrow \textit{length scale}$$

W



H : Hardness

R : Indenter tip radius

σ_{ys} : Yield stress

δ : Tip displacement

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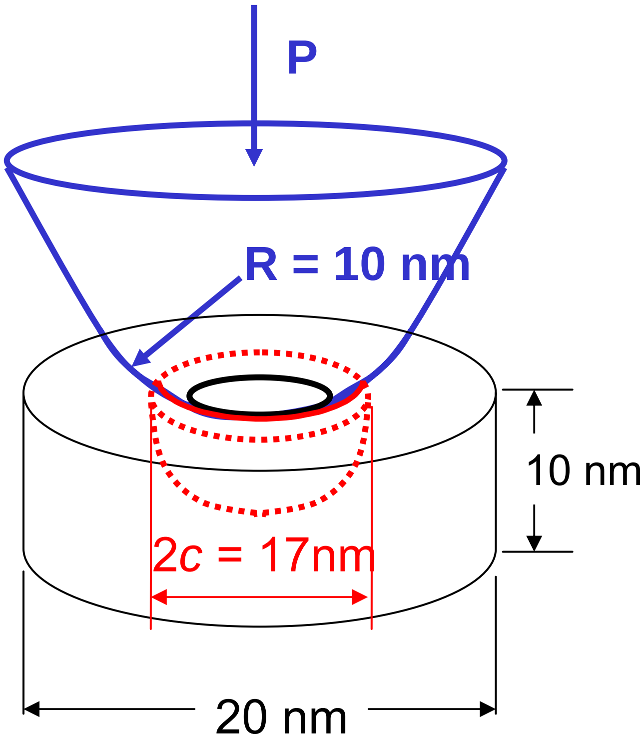
Consider a Cu Feature {Boundary Constraint}



Assume: 1 nanodot = 10 nm thick, 20 nm diameter

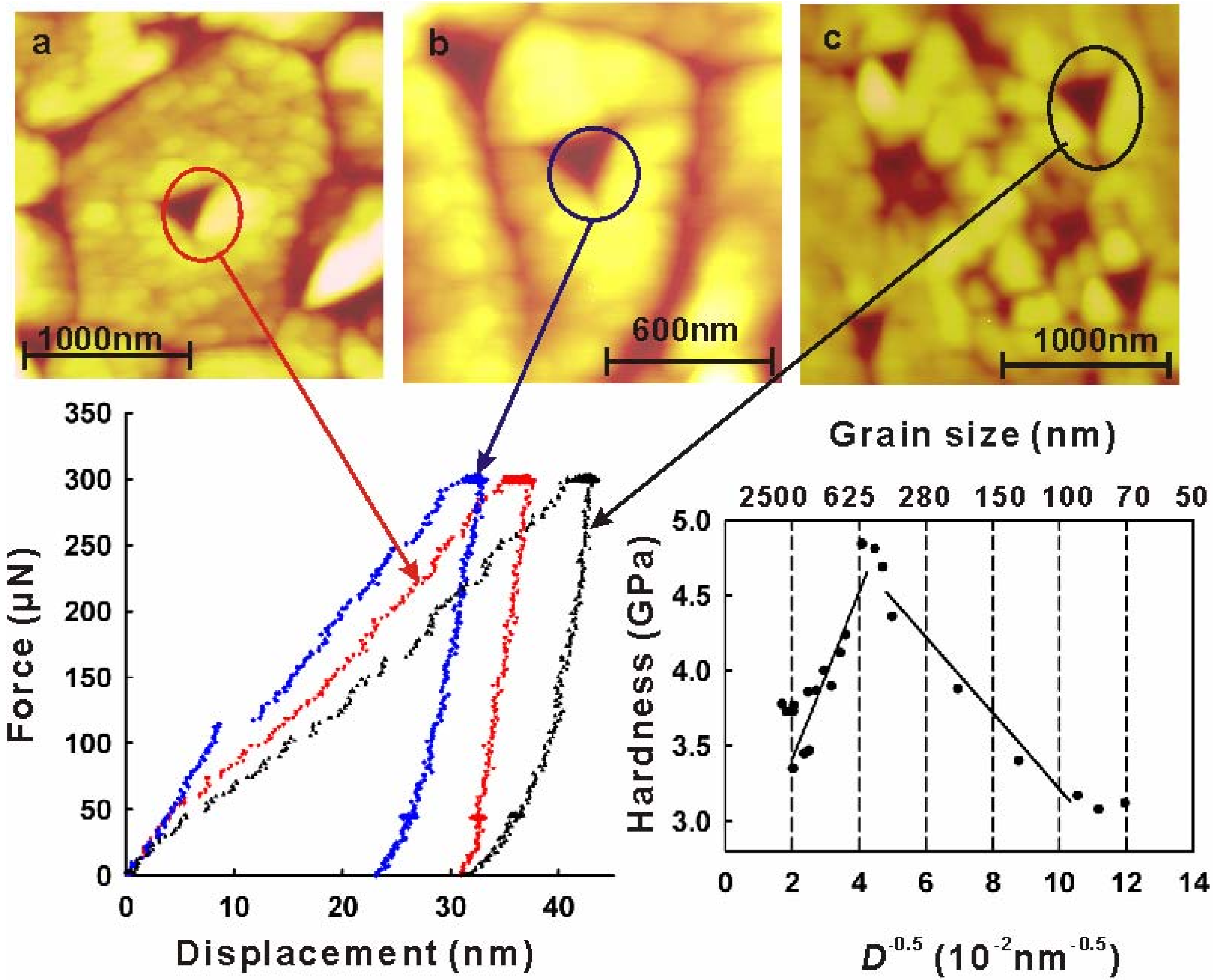
$\sigma_{ys}^{cu} \sim 1.4$ GPa
$\delta \sim 1$ nm
R ~ 10 nm
$a \sim 4$ nm

P = 4.2GPa{5×10^{-17}m^2} = 210 nN
(H)

If (??) continuum, plastic zone, *c*

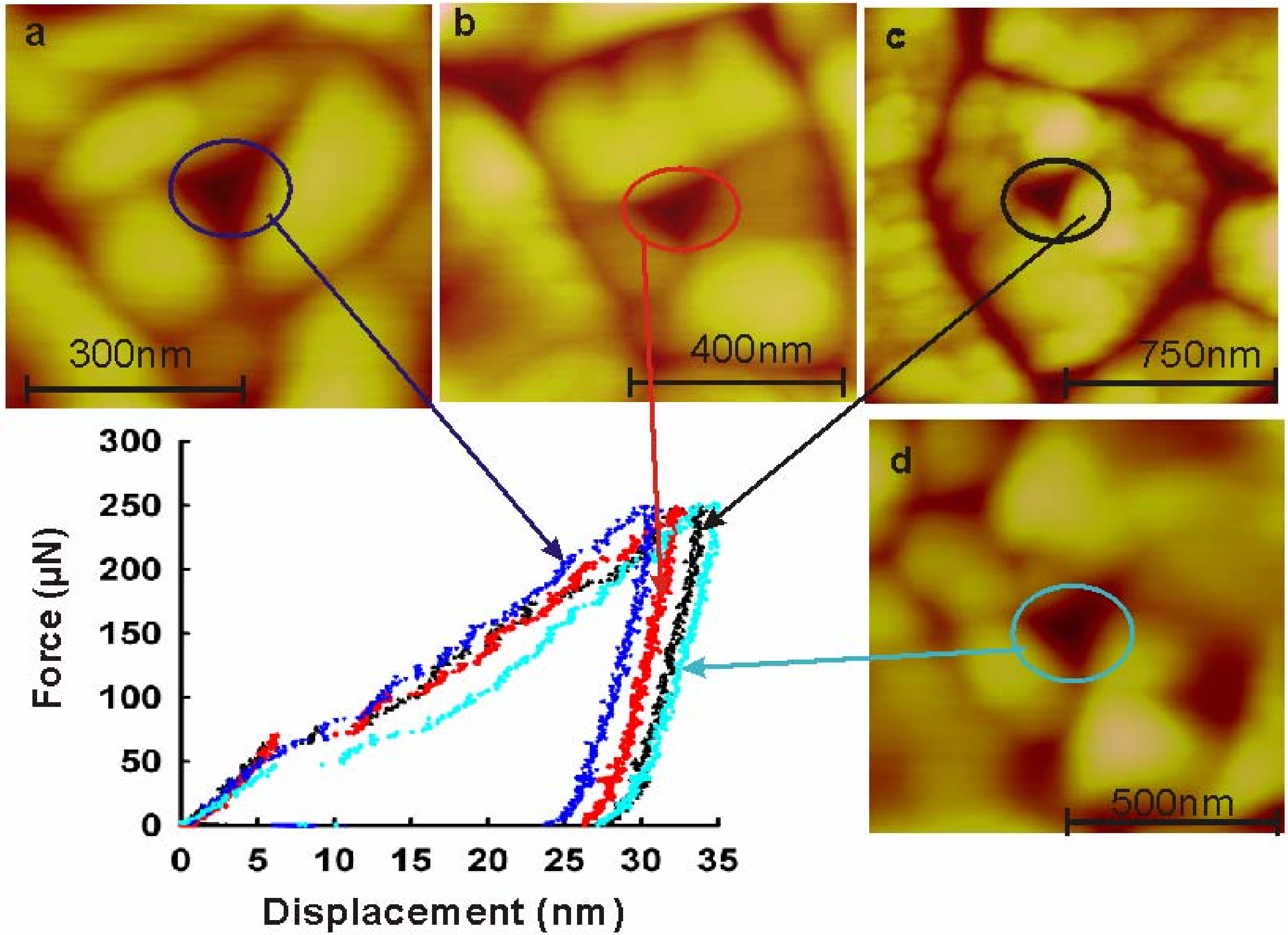
$$c \sim \sqrt{\frac{3P}{2\pi\sigma_{ys}}} = 8.5 \text{ nm}$$

Note $c = 2.12a$



Nanoindentation with peak force 300 μN

Bo Yang and H. Vehoff: Z. Metallkd. 95 (2004) 6

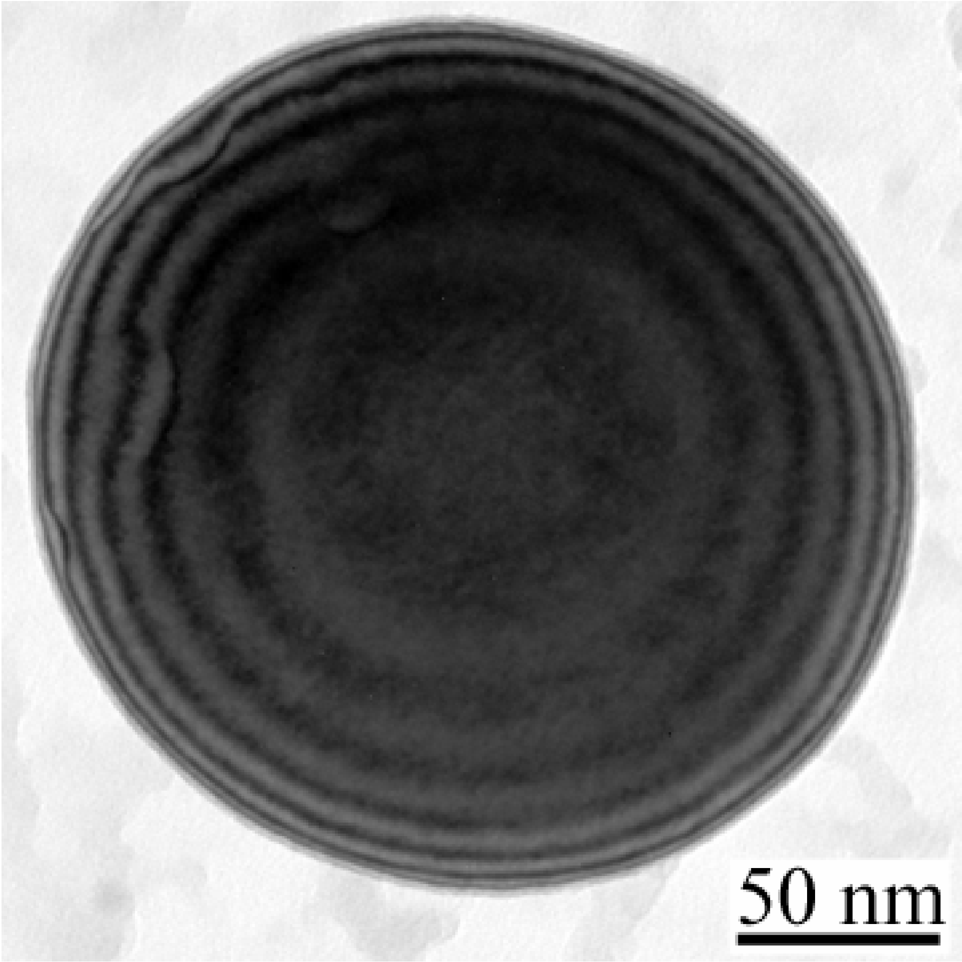


Nanoindentation with peak force 250 µN

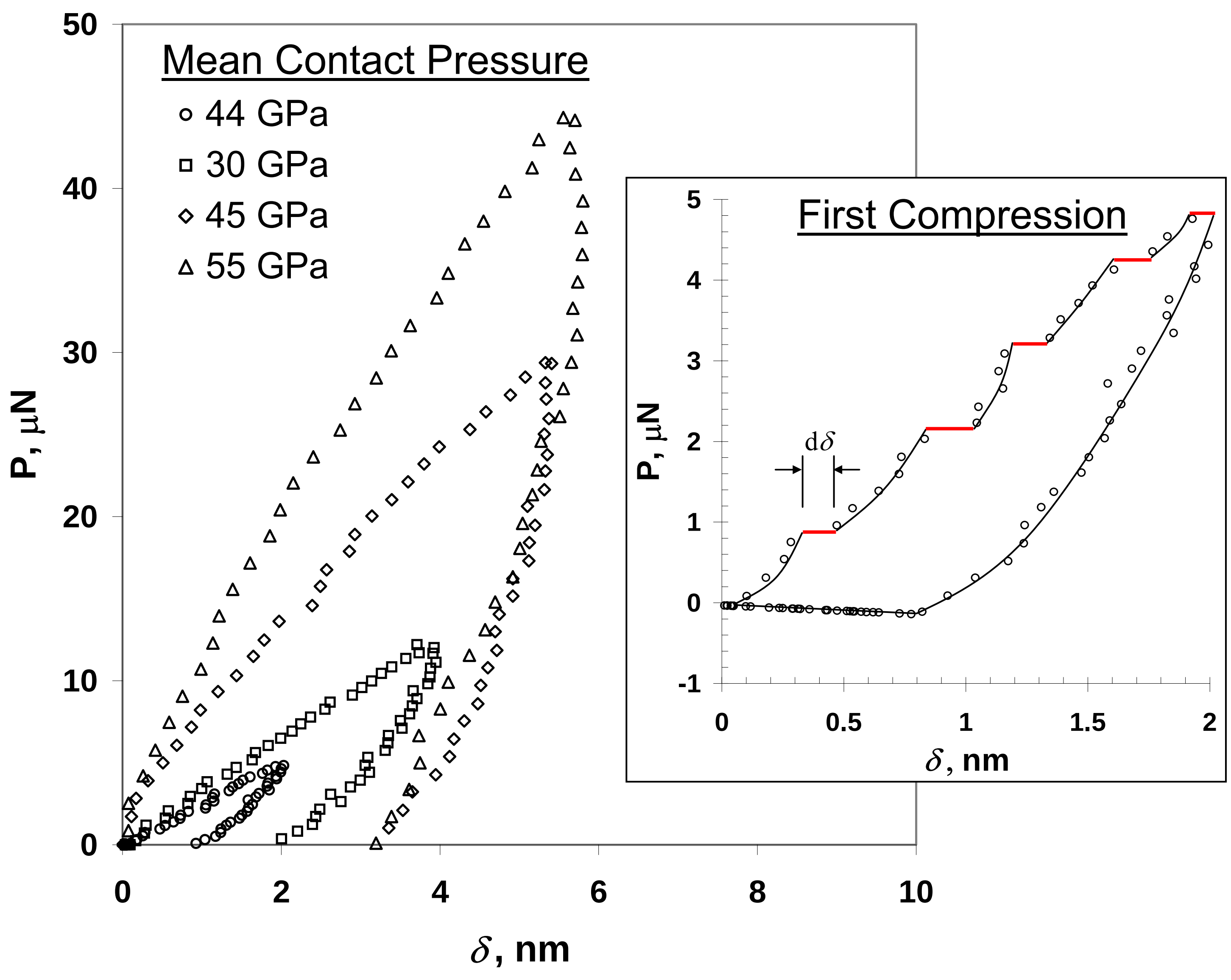
Bo Yang and H. Vehoff: Z. Metallkd. 95 (2004) 6

Silicon Nanoparticles

TEM of non-equilibrium particle

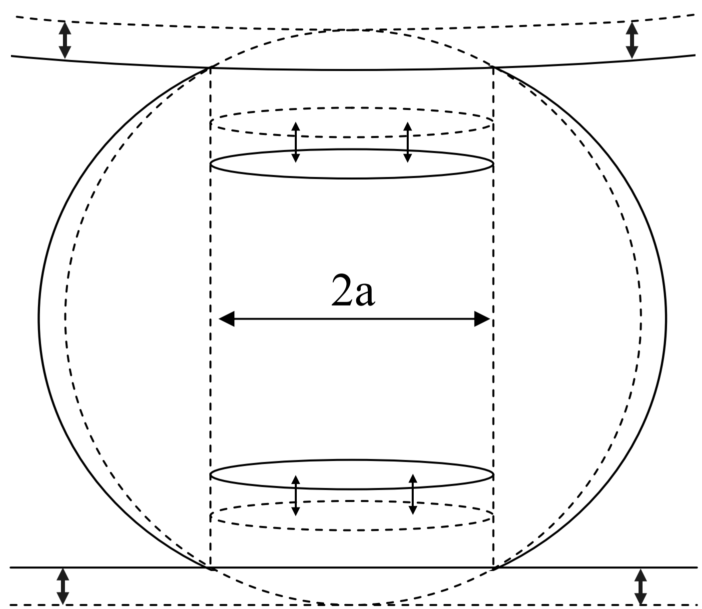


Silicon nanoparticle: 39 nm diameter



Idealized Compression of a Nanosphere

$$\ell_s = \frac{\frac{4}{3}\pi r^2}{2\pi a^2} = \frac{2r^3}{3a^2}$$



Equilibrium Arrest Position

Zhou and Thomson's Analysis for a Plastically Polarizable Material (Crack Problem ➔ Indent)

$$\sigma_f = \frac{p_o}{2}\left[\frac{1+\nu}{1-\frac{z_i}{a}\tan^{-1}\left(\frac{a}{z_i}\right)+\frac{1}{2}\left(1+\frac{z_i^2}{a^2}\right)} - \frac{a^2}{a^2+z_i^2}\right] - \frac{\mu b_i}{4\pi(1-\nu)z_i} + \sum_{\substack{j \\ j\neq i}} \frac{\mu b_j}{2\pi(1-\nu)}\frac{1}{z_i - z_j}$$

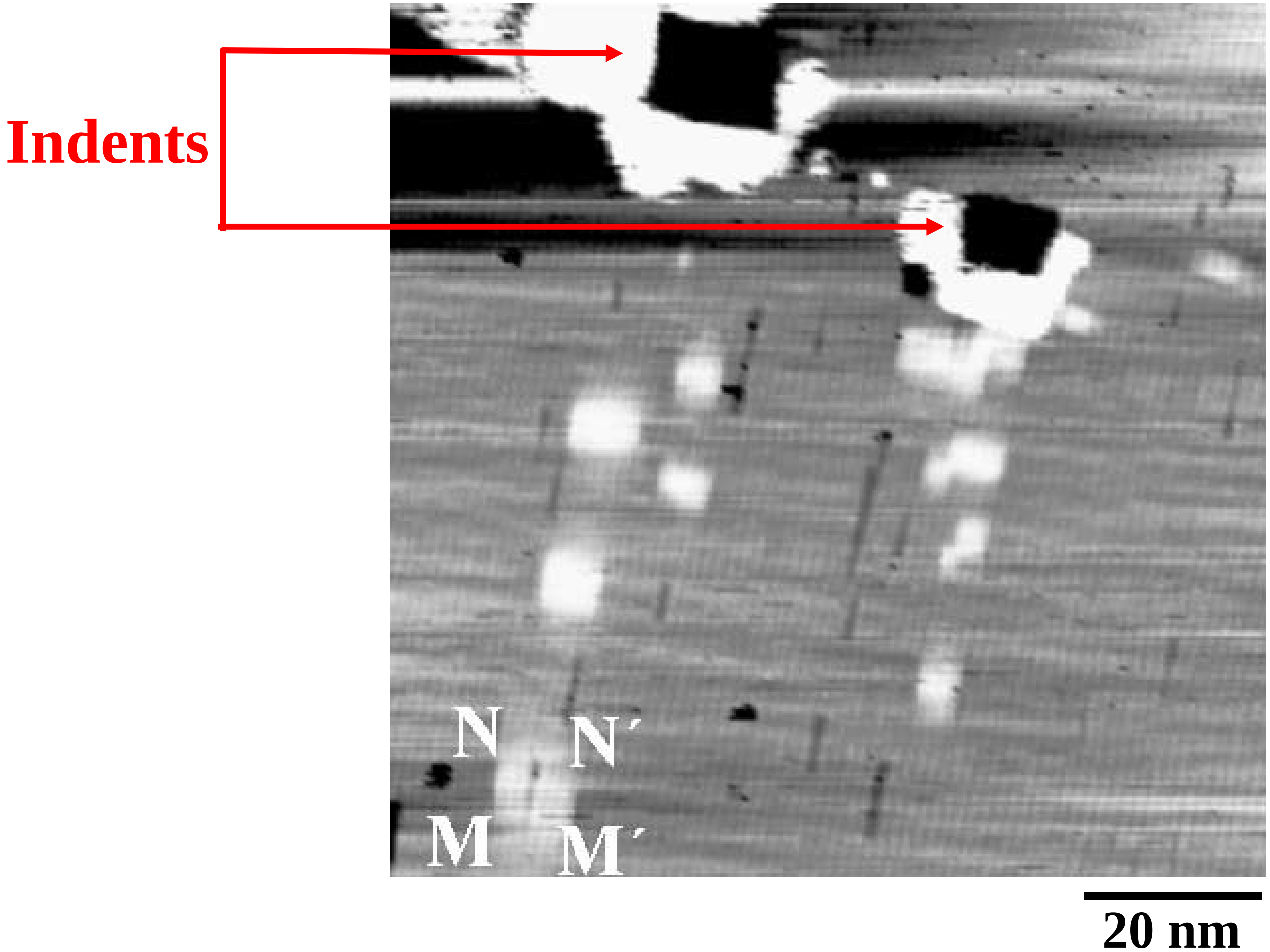
σ_f = friction stress at arrest

p_o = maximum pressure = $\dfrac{3P}{2\pi a^2}$

z = distance below contact

a = contact radius

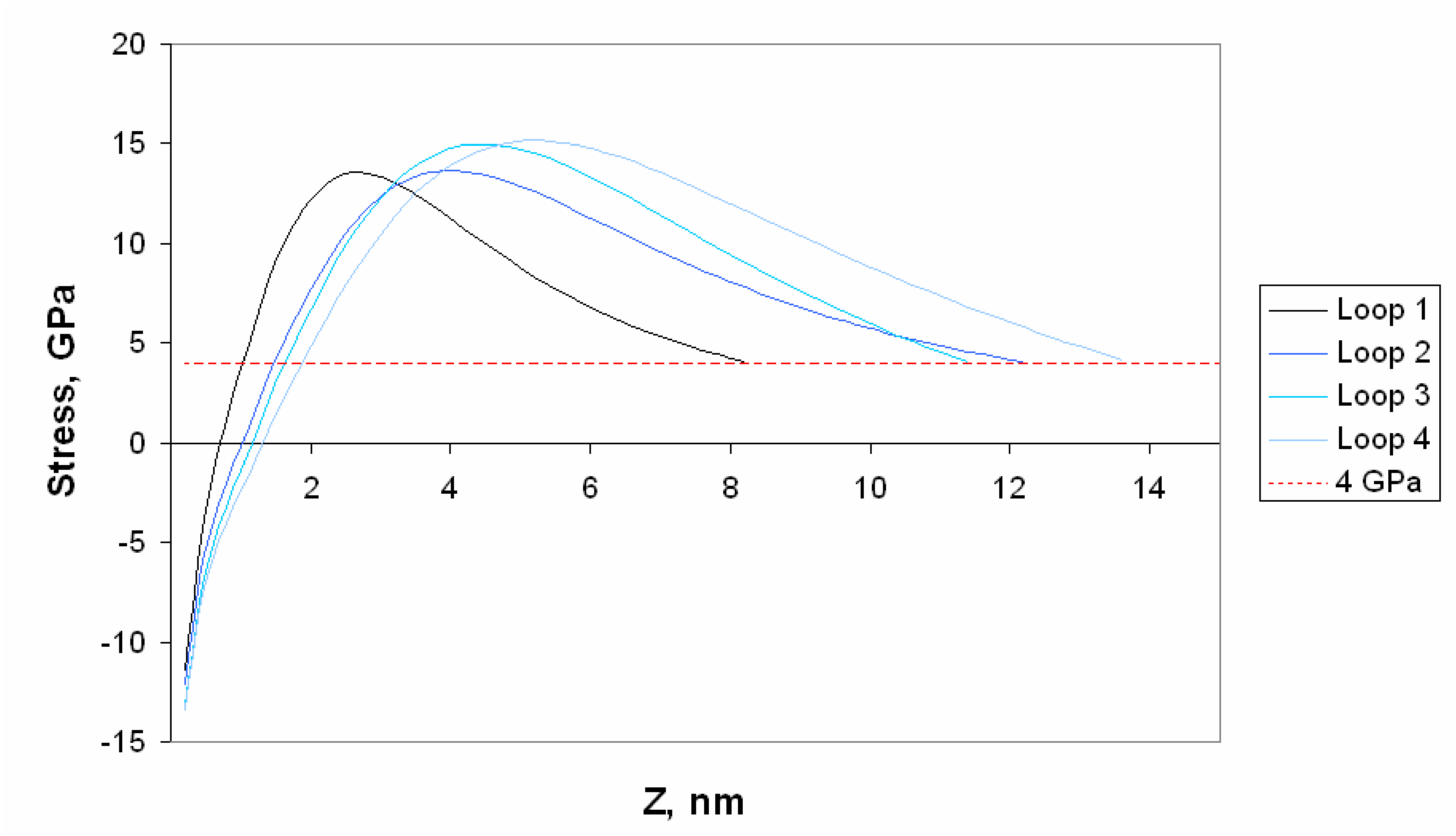
STM image of nanoindentations in the Au(001) surface



Rows of hillocks stemming from the nanoindentation points and following a <110> direction are visible.

Courtesy of O.R. de la Fuente, et al., Phys. Rev. Let., 88(3), 2002, 036101.

Silicon nanoparticle: 39 nm diameter Dislocation Glide Depth



Work to move a dislocation below the surface for a sphere of Silicon

1st Excursion: $P = 0.92\ \mu\text{N}, \quad \delta_{before} = 0.26\ \text{nm}, \quad \delta_{after} = 0.41\ \text{nm}$

$$W_E = Pd\delta = \boxed{1.43\text{x}10^{-16}\ \text{N}\cdot\text{m}} = \Delta S + \Delta W_\perp + \Delta U_\perp$$

Surface Work: $a_{before} = 2.22\ \text{nm}, \quad a_{after} = 2.81\ \text{nm}$

$$\Delta S = \gamma_s A_{after} - \gamma_s A_{before} = 2.91\text{x}10^{-17}\ \text{N}\cdot\text{m}$$

Dislocation Work: $\Delta W_\perp = \tau_{ys} b \cdot 2\pi a \cdot x = 10.5\text{x}10^{-17}\ \text{N}\cdot\text{m}$

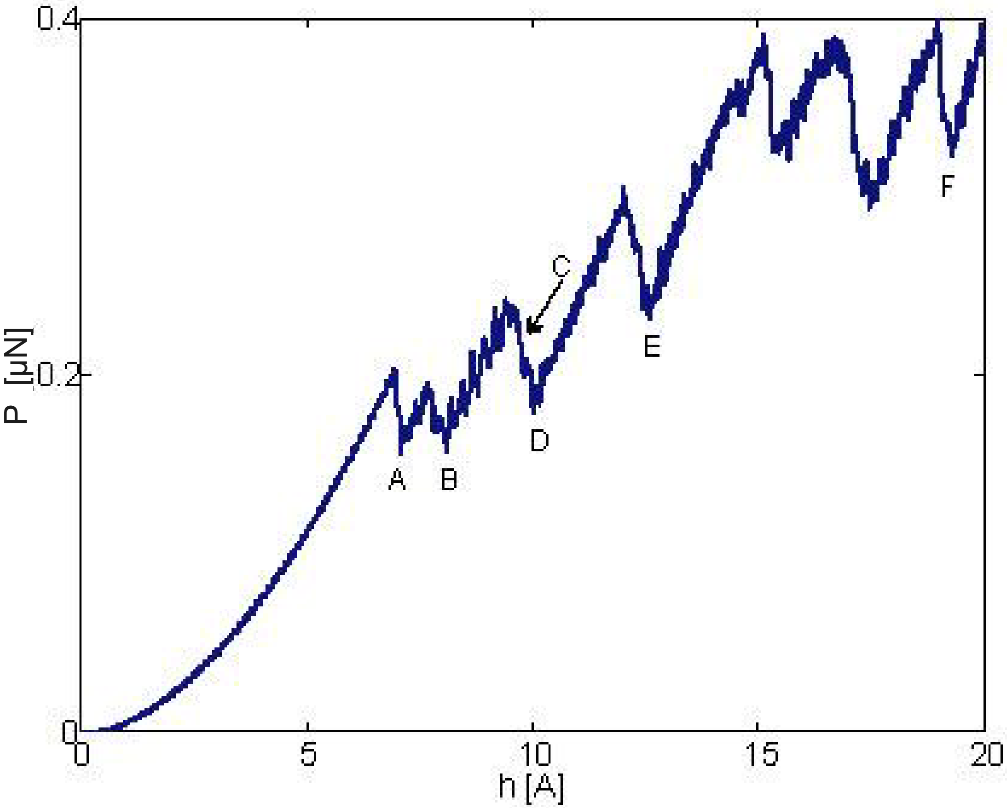
Stored Elastic Energy: $\Delta U_\perp = \pi a\left[1 + \frac{1}{1-\nu}\right]\left[\frac{\mu b^2}{4\pi}\ln\frac{ae^2}{b}\right] = 2.65x10^{-17}\ \text{N}\cdot\text{m}$

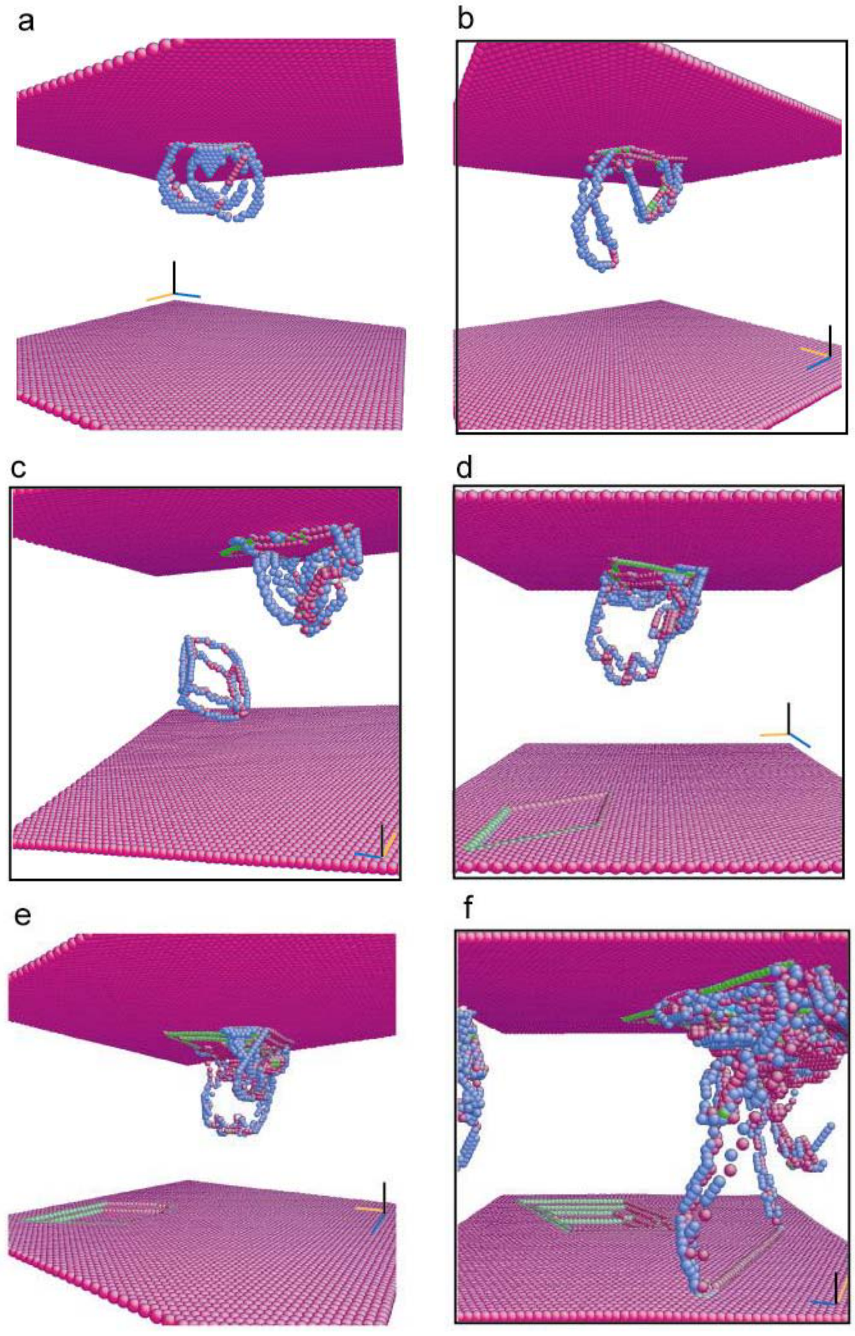
(Surface Work + Dislocation Work + Stored Elastic Energy) $= \boxed{1.61\text{x}10^{-16}\ \text{N}\cdot\text{m}}$

113% Accounted for

4 such excursions : 93 ± 20%

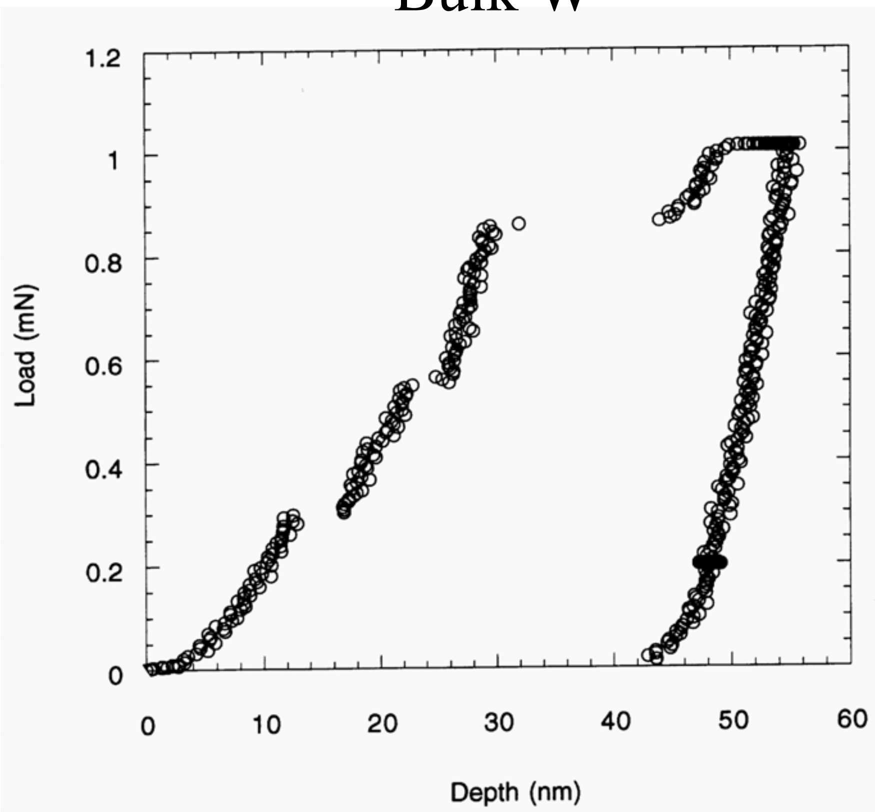
Courtesy, S. Yip, K van Vliet, MIT



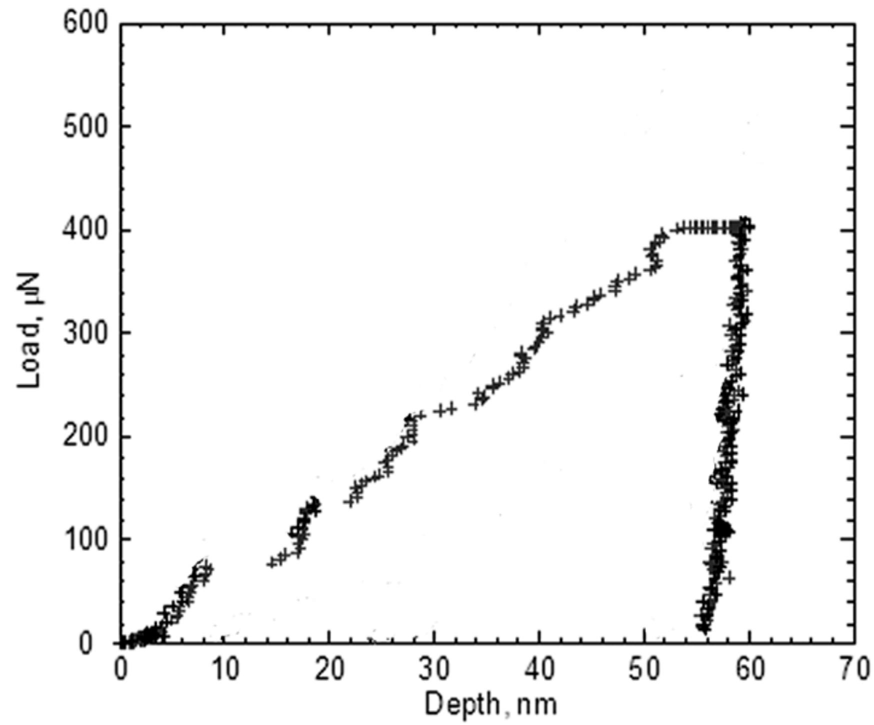


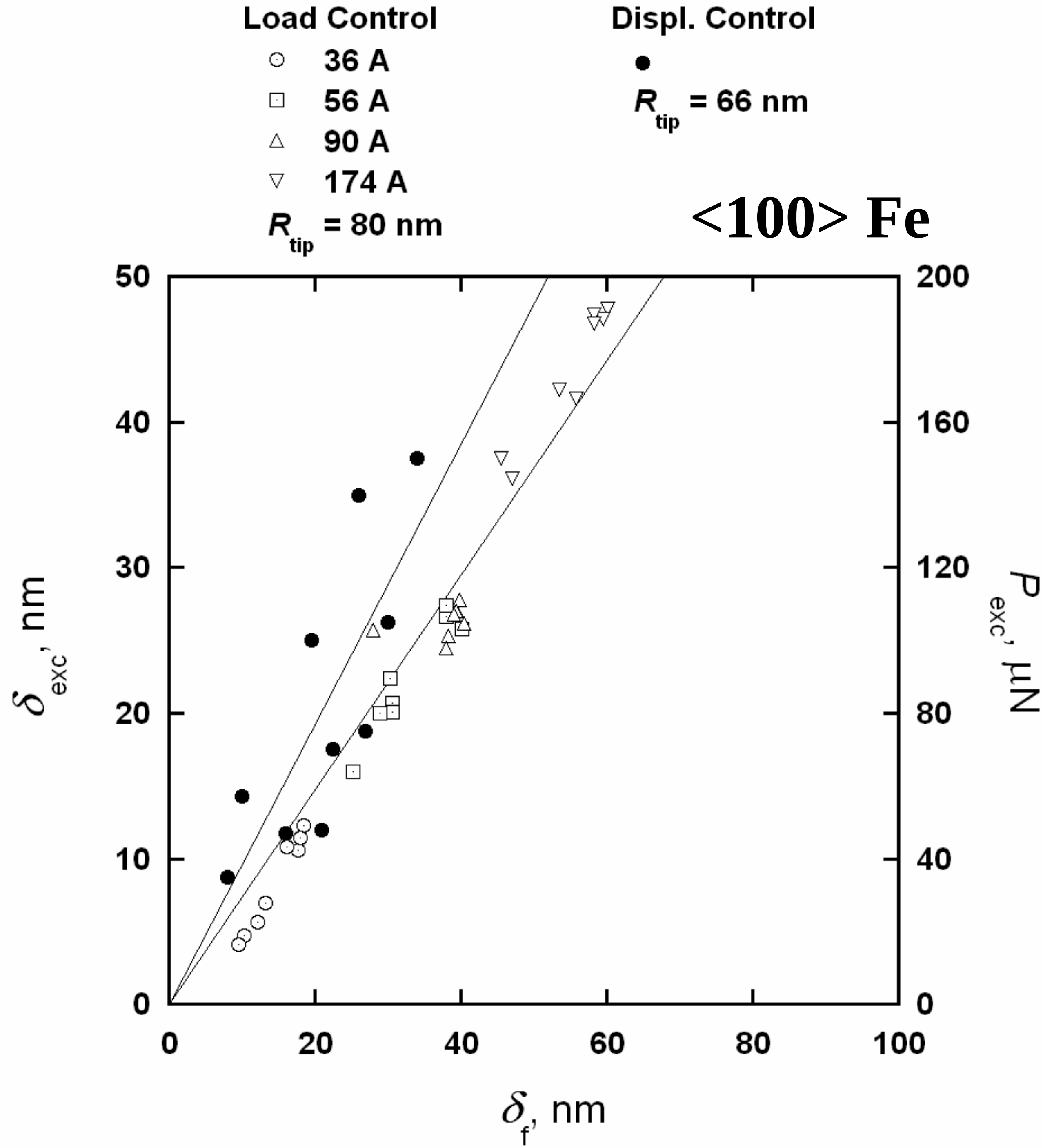
Staircase Yielding

Bulk W

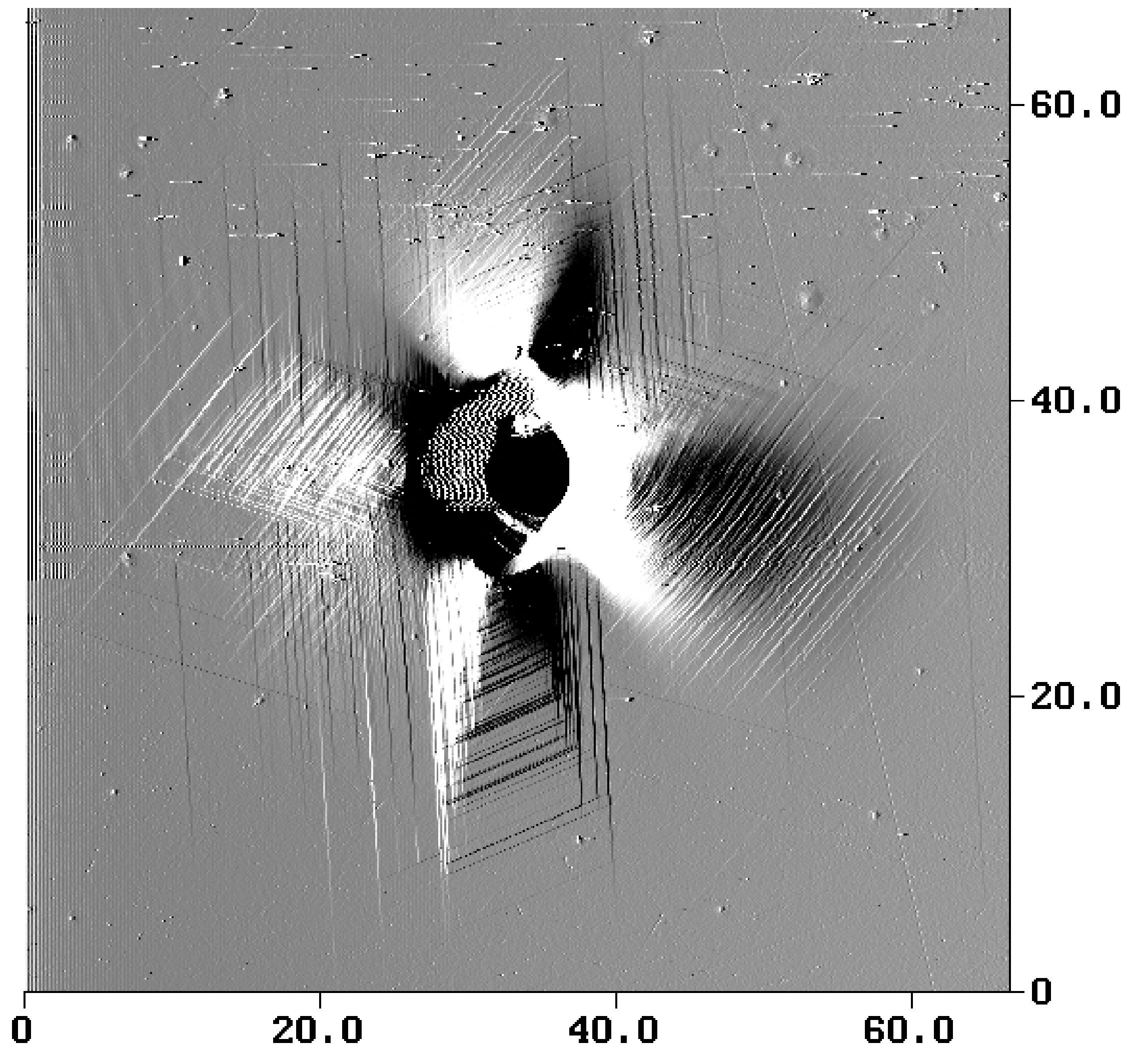


Thin film Al





Load Control
36 A
56 A
90 A
174 A
R_{tip} = 80 nm
Displ. Control
R_{tip} = 66 nm
<100> Fe
δ_{exc}, nm
P_{exc}, μN
δ_f, nm
0
10
20
30
40
50
0
40
80
120
160
200
0
20
40
60
80
100



courtesy K. Nibur, D. Bahr, Washington State

Energy Balance Approach to Yield Excursions

Released: $U_E = -P\left(\frac{a_1^{\ 2}}{2R} - \frac{a_0^{\ 2}}{2R}\right)$: a = contact radius, R = tip radius, P = load

Absorbed: $W_P = \frac{4}{9}\frac{Pa^2}{R}$: plastic energy from prismatic punched loops of radius a

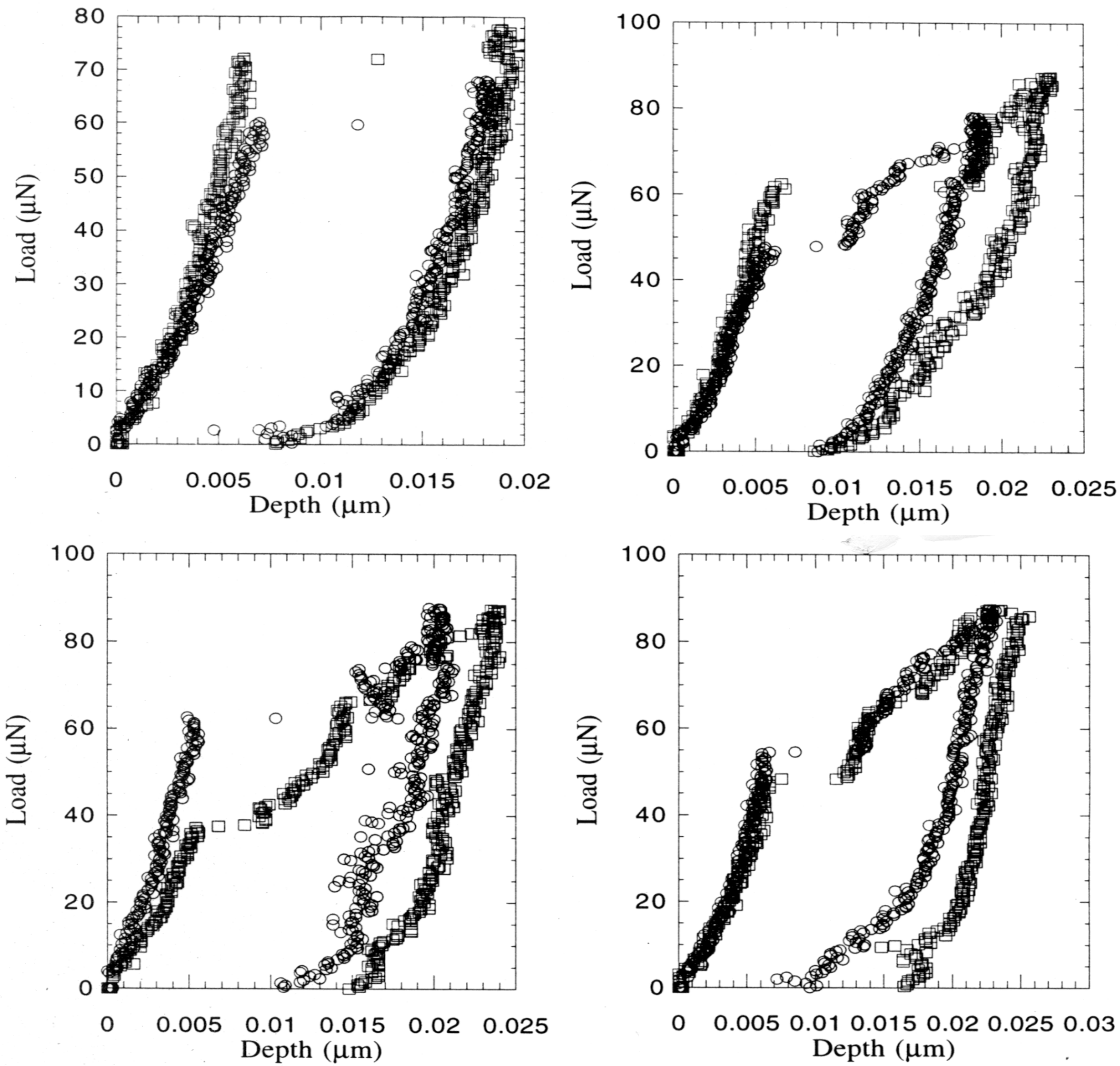
Absorbed: $\alpha\gamma_s A = \alpha\pi\frac{a^3}{R}$: energy from new area created, γ_s = surface energy

$$U_E + W_P + \alpha\gamma_s A = -P\delta_{exc} + \frac{4}{9}\frac{Pa^2}{R} + 2\sqrt{2}\pi\alpha\delta_f^{\ 3/2}R^{1/2}\gamma_s$$

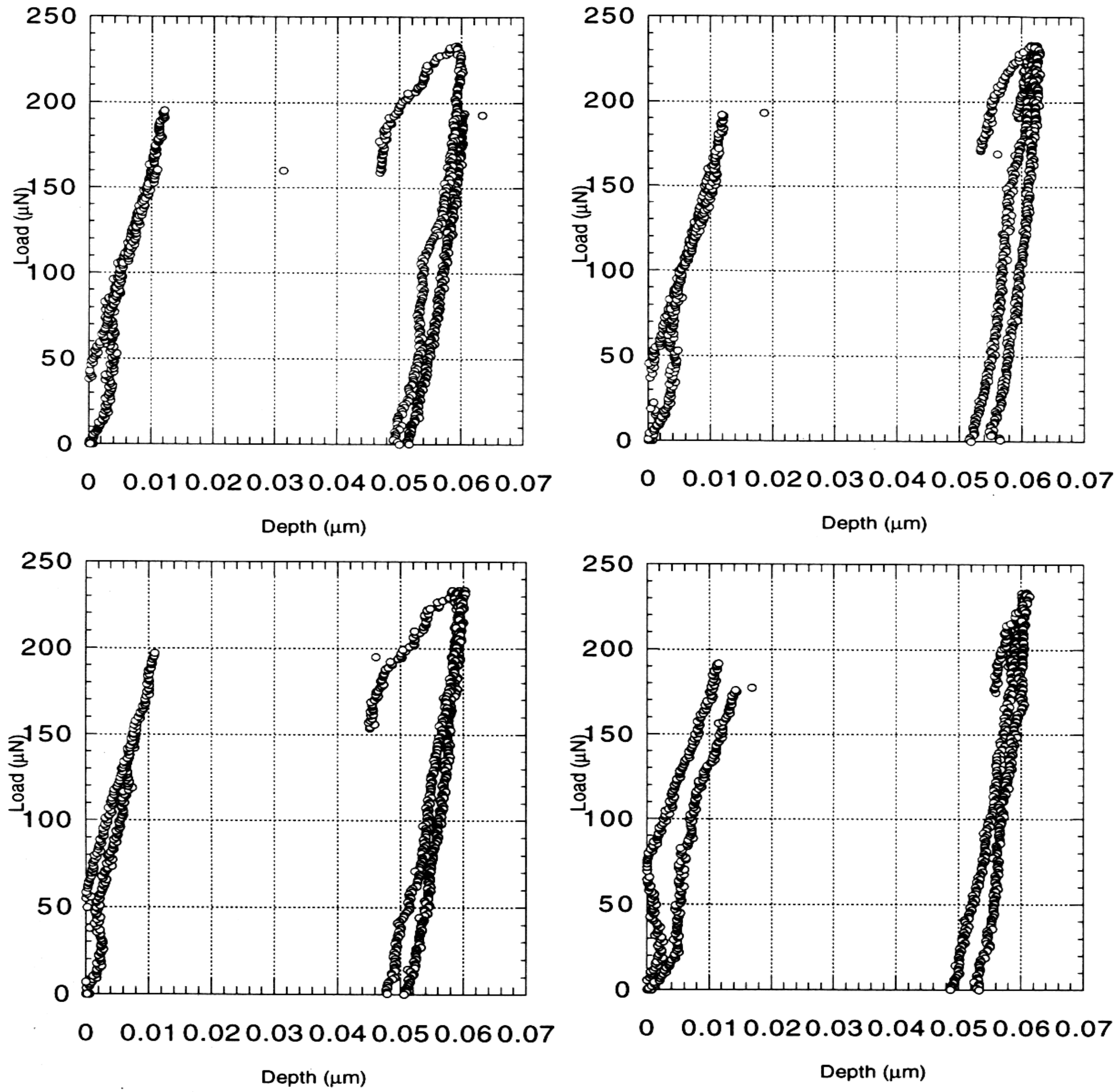
$$\boxed{\delta_{exc} = \frac{const.\ \alpha\gamma_s R^{1/2}\delta_f^{\ 3/2}}{P}}$$

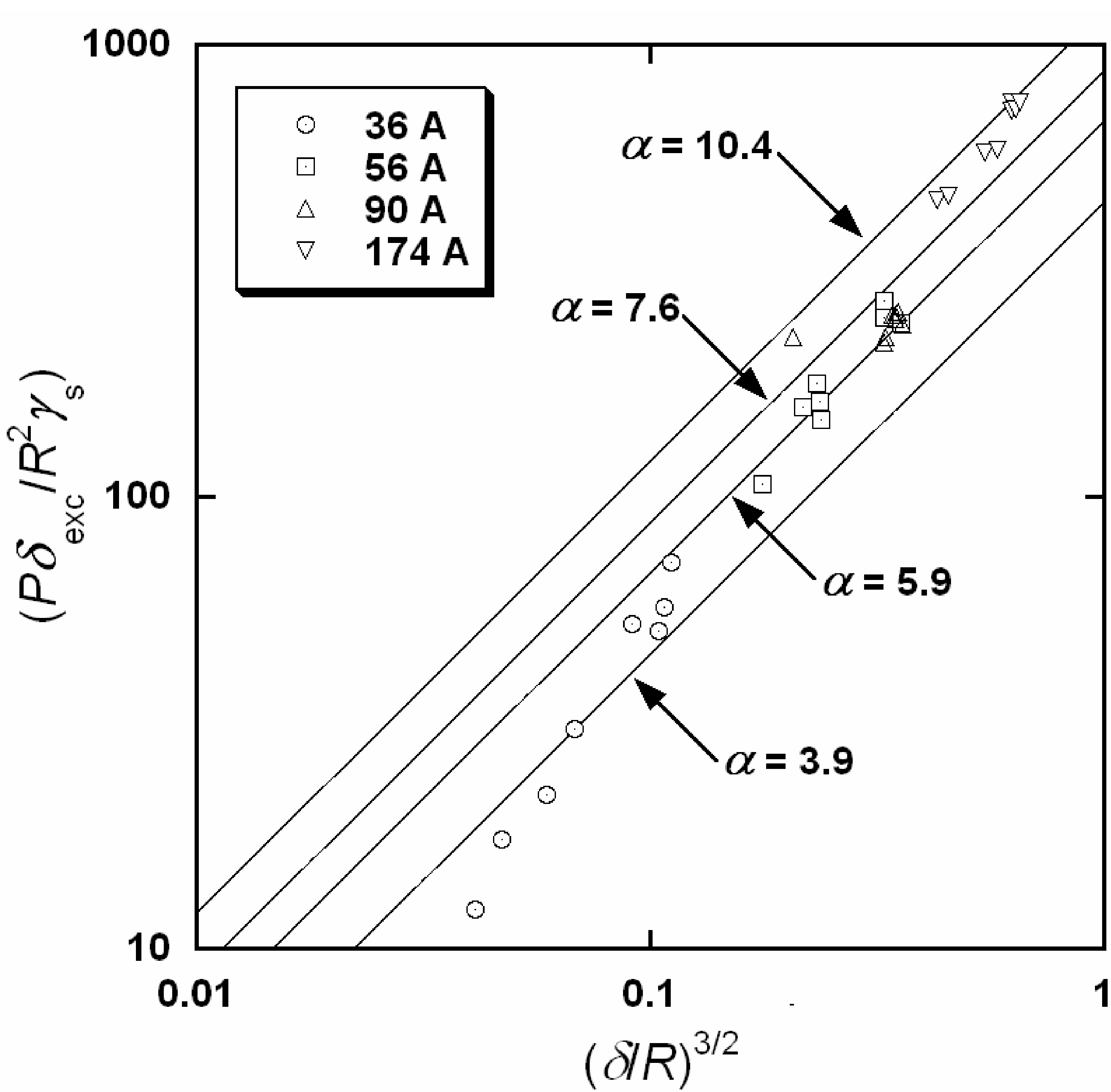
: δ_f = displacement at end of excursion, α = mat'l specific constant

Fe 3% Si: 36 Å native oxide grown in air



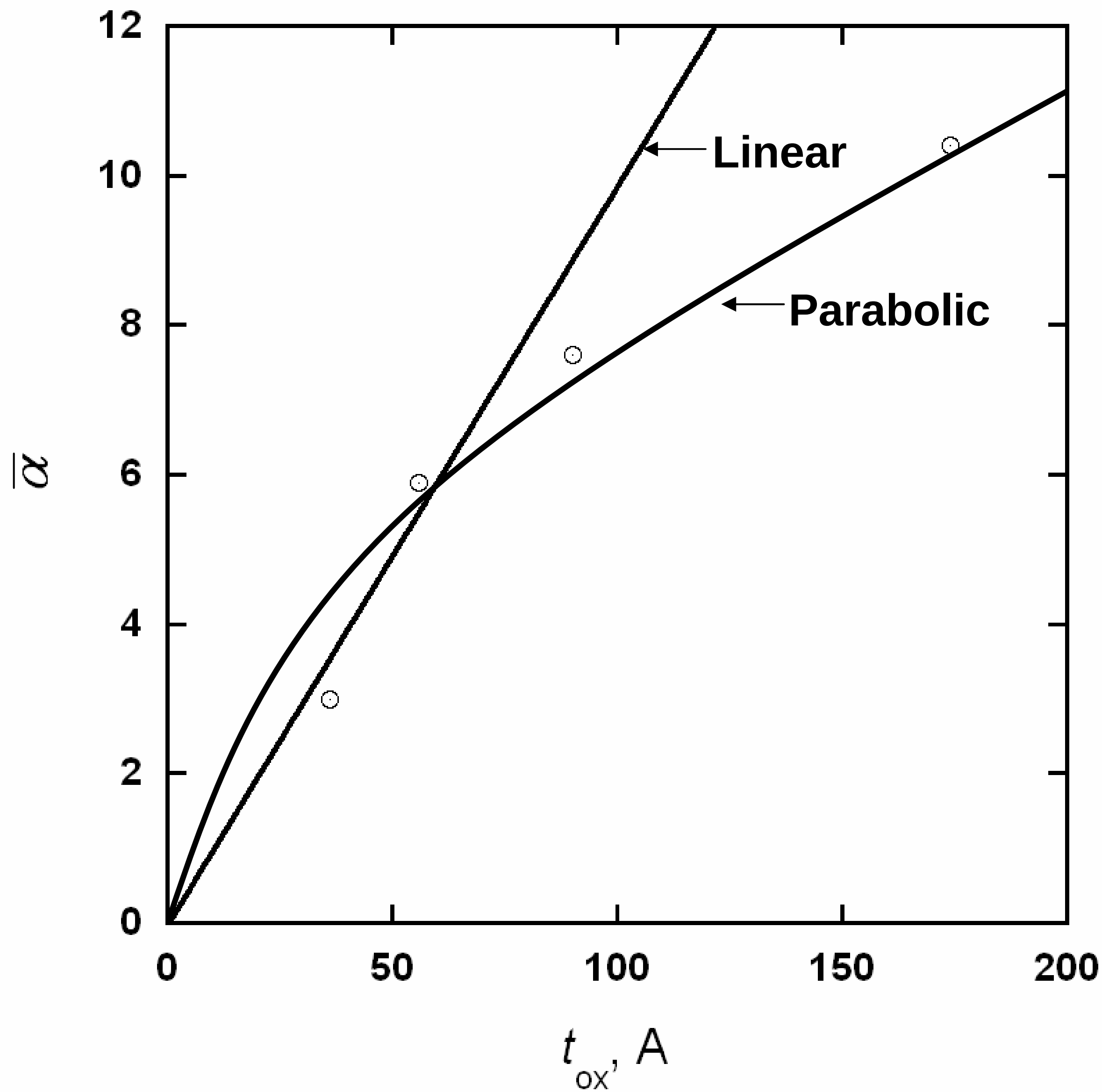
Fe 3% Si: 174 Å thermal oxide grown at 300 °C



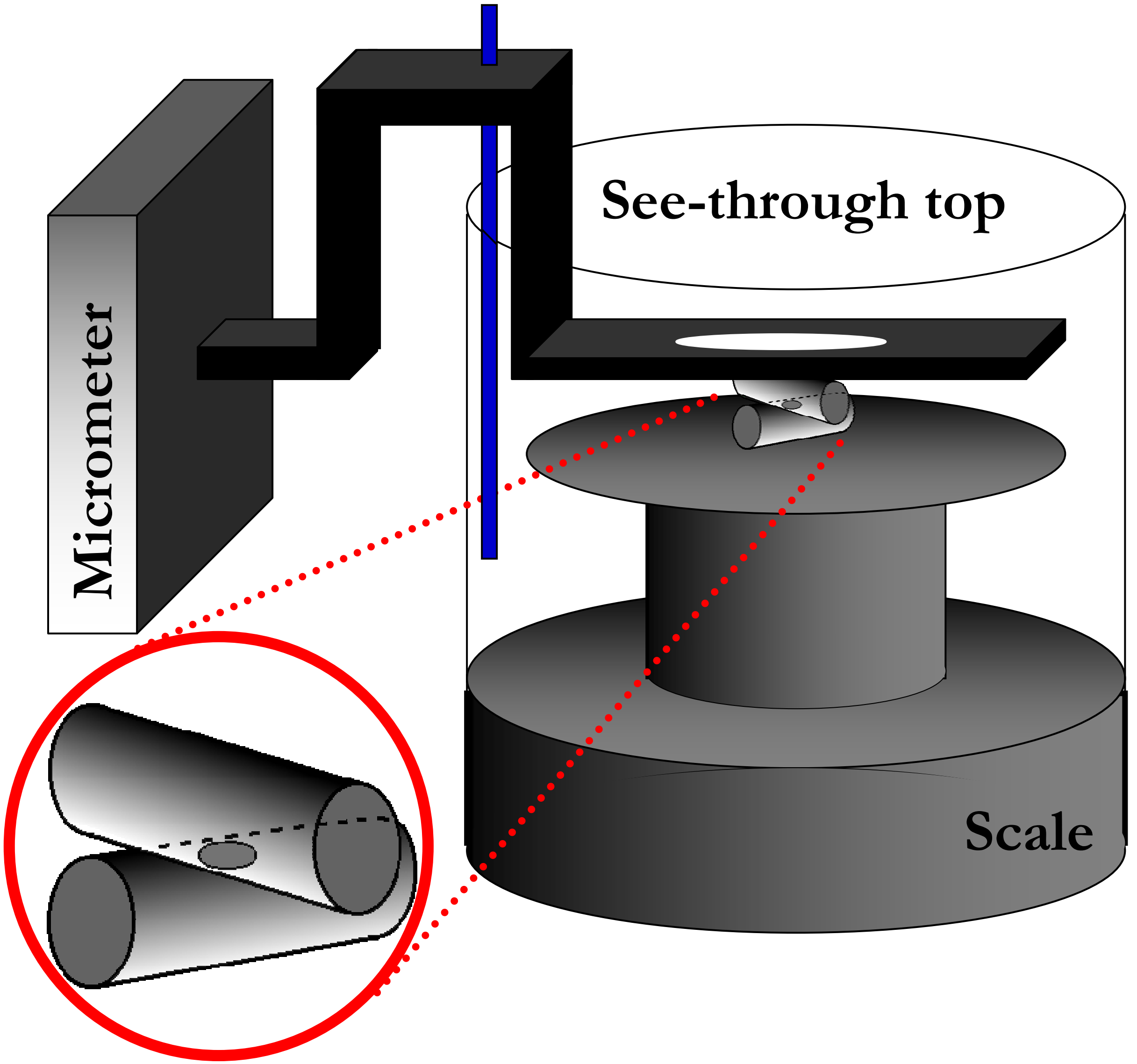


36 A
56 A
90 A
174 A
α = 10.4
α = 7.6
α = 5.9
α = 3.9
1000
100
10
0.01
0.1
1
$(P\delta_{exc}/R^2\gamma_s)$
$(\delta/R)^{3/2}$

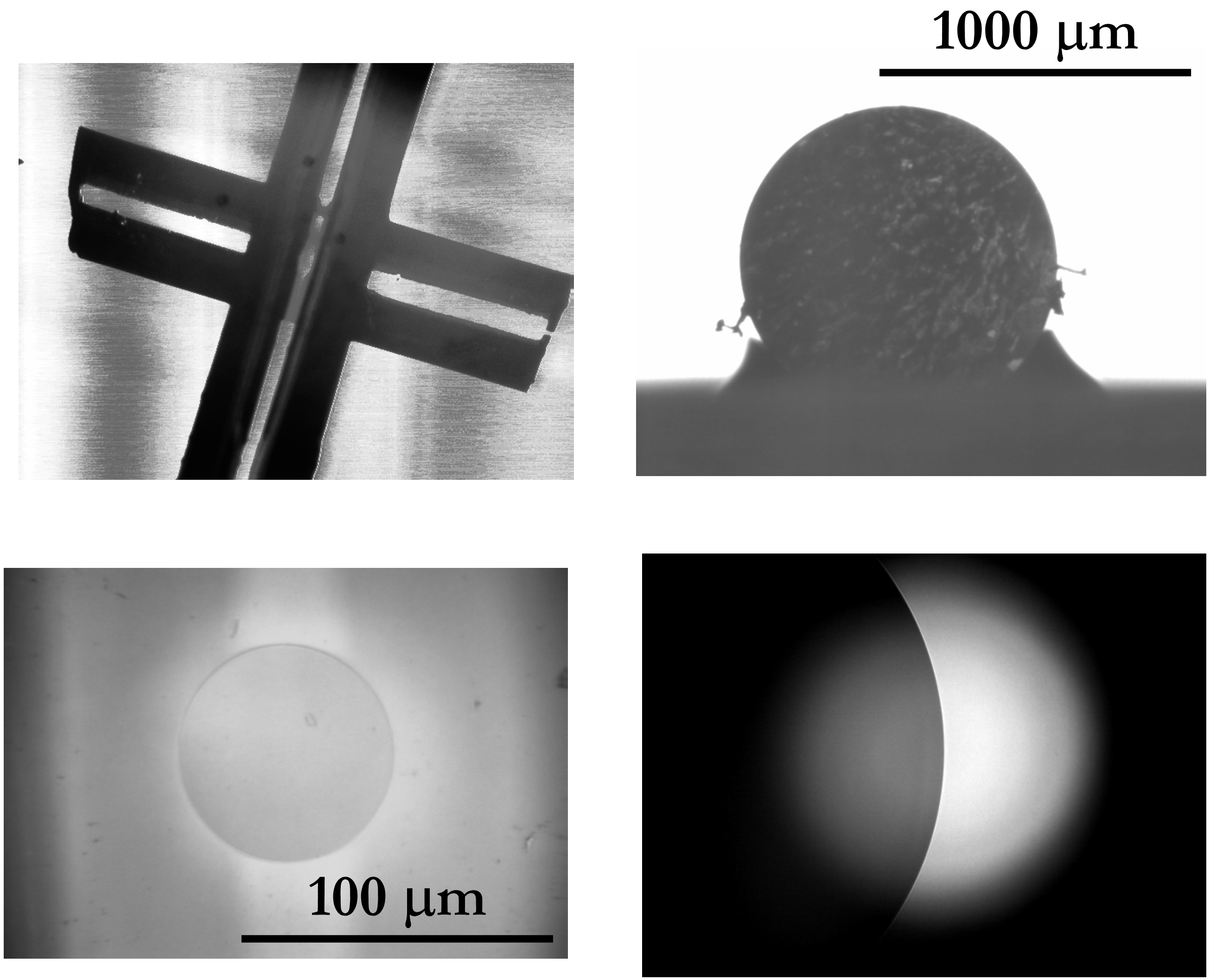
Oxide films on <100> Fe



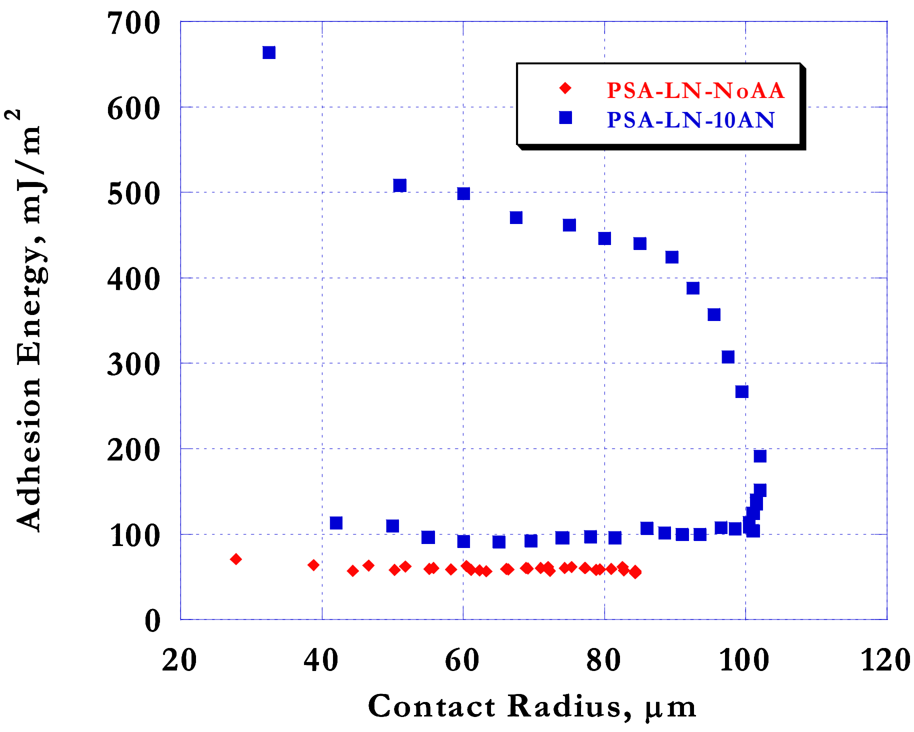
Normal Contact Adhesion Effects

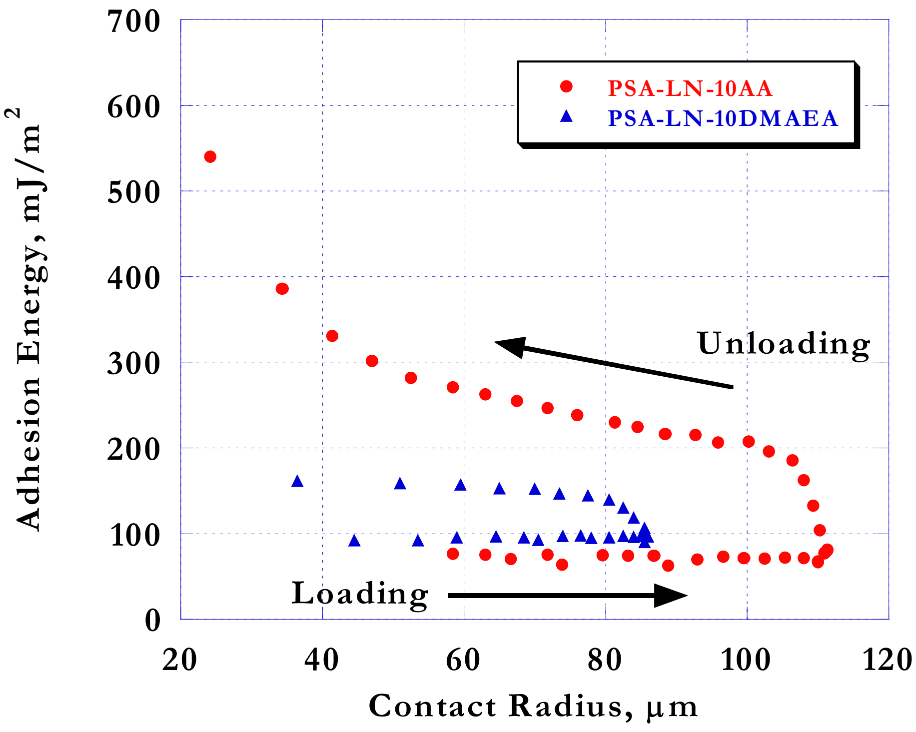


Optical Images

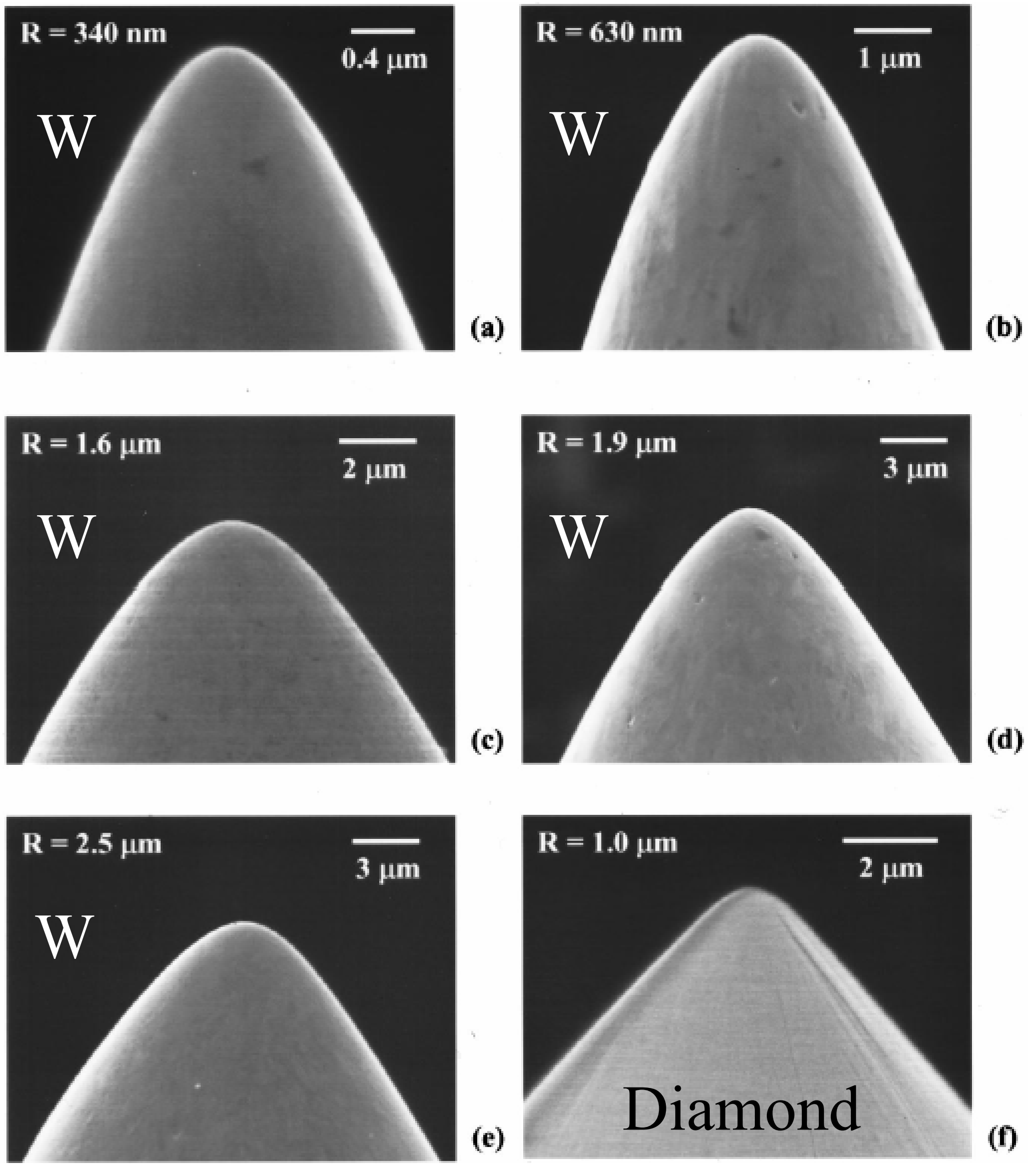


Adhesion Hystereses



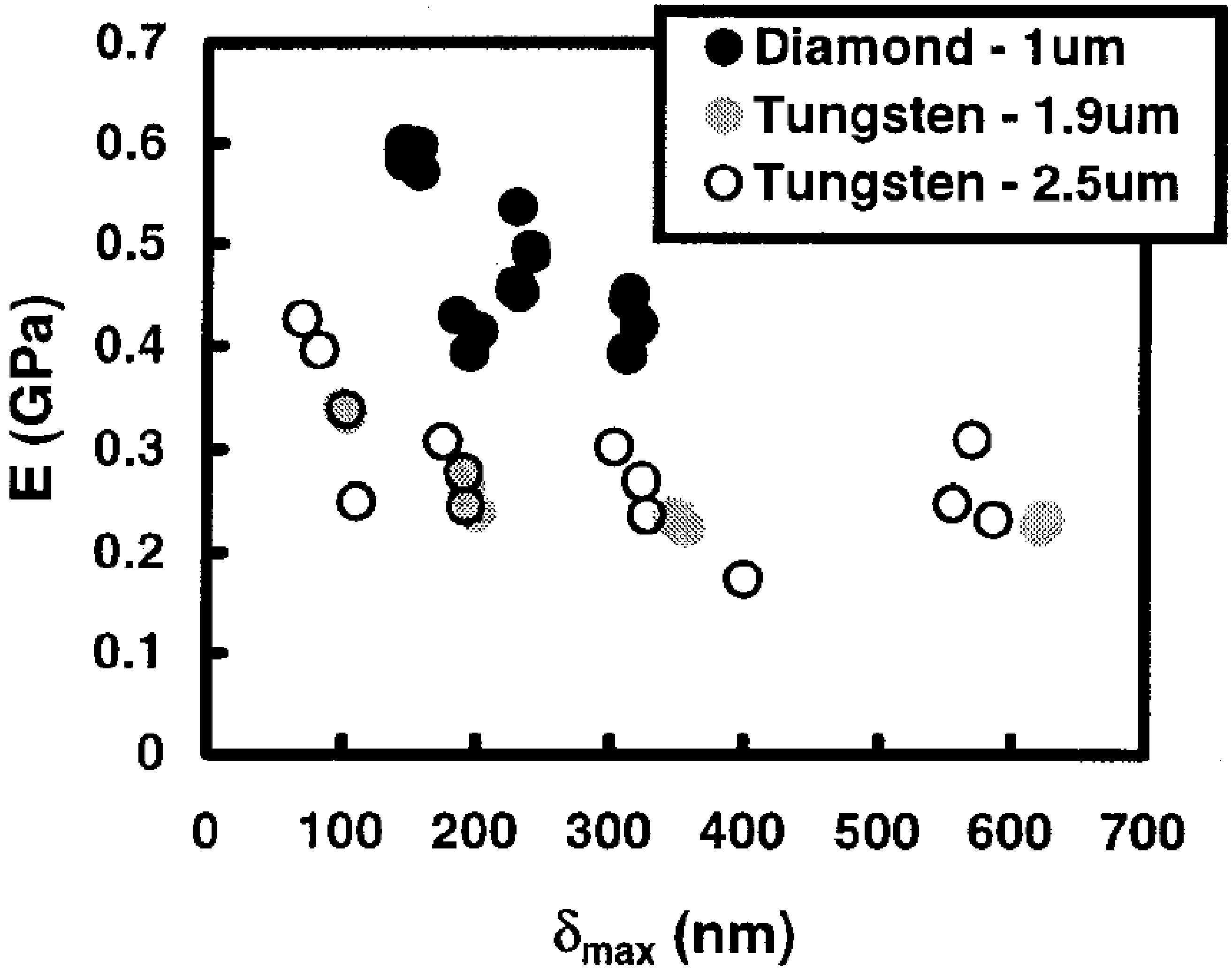


SEM of W indenter tips



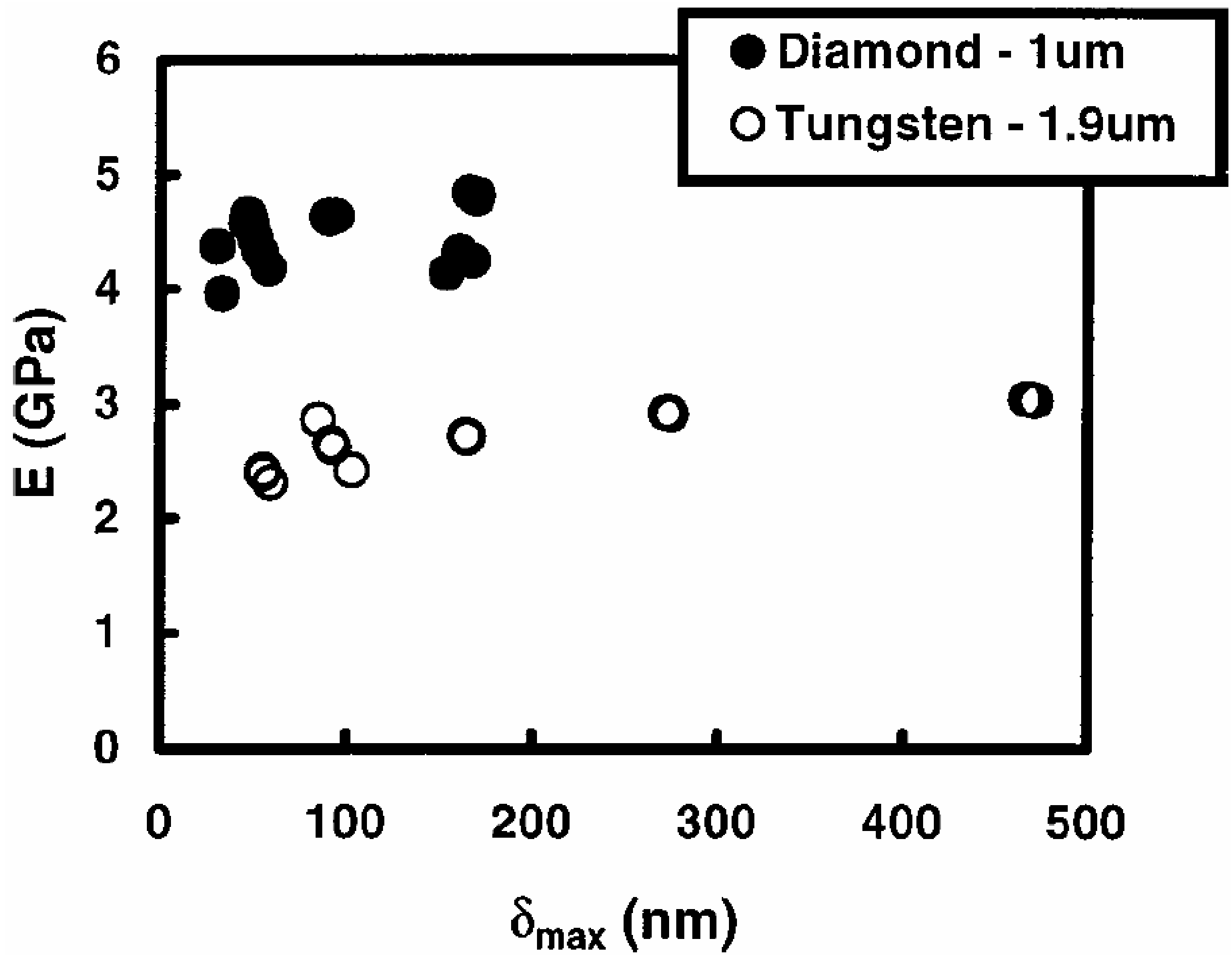
Rev. Sci. Instruments, Grunlan, *et al*. (2001)

Indentation into bulk LDPE



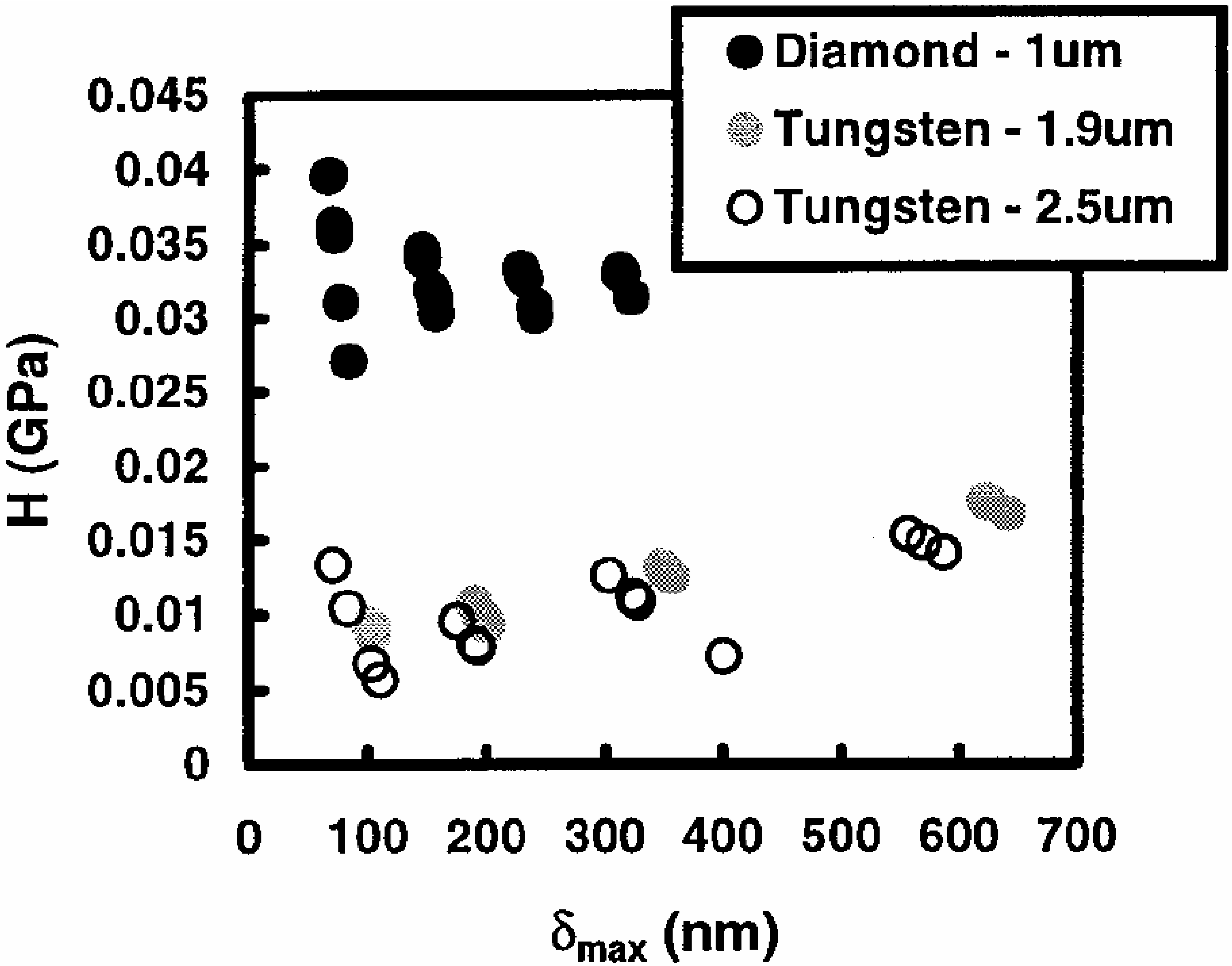
Rev. Sci. Instruments, Grunlan, *et al*. (2001)

Indentation into bulk Polystyrene



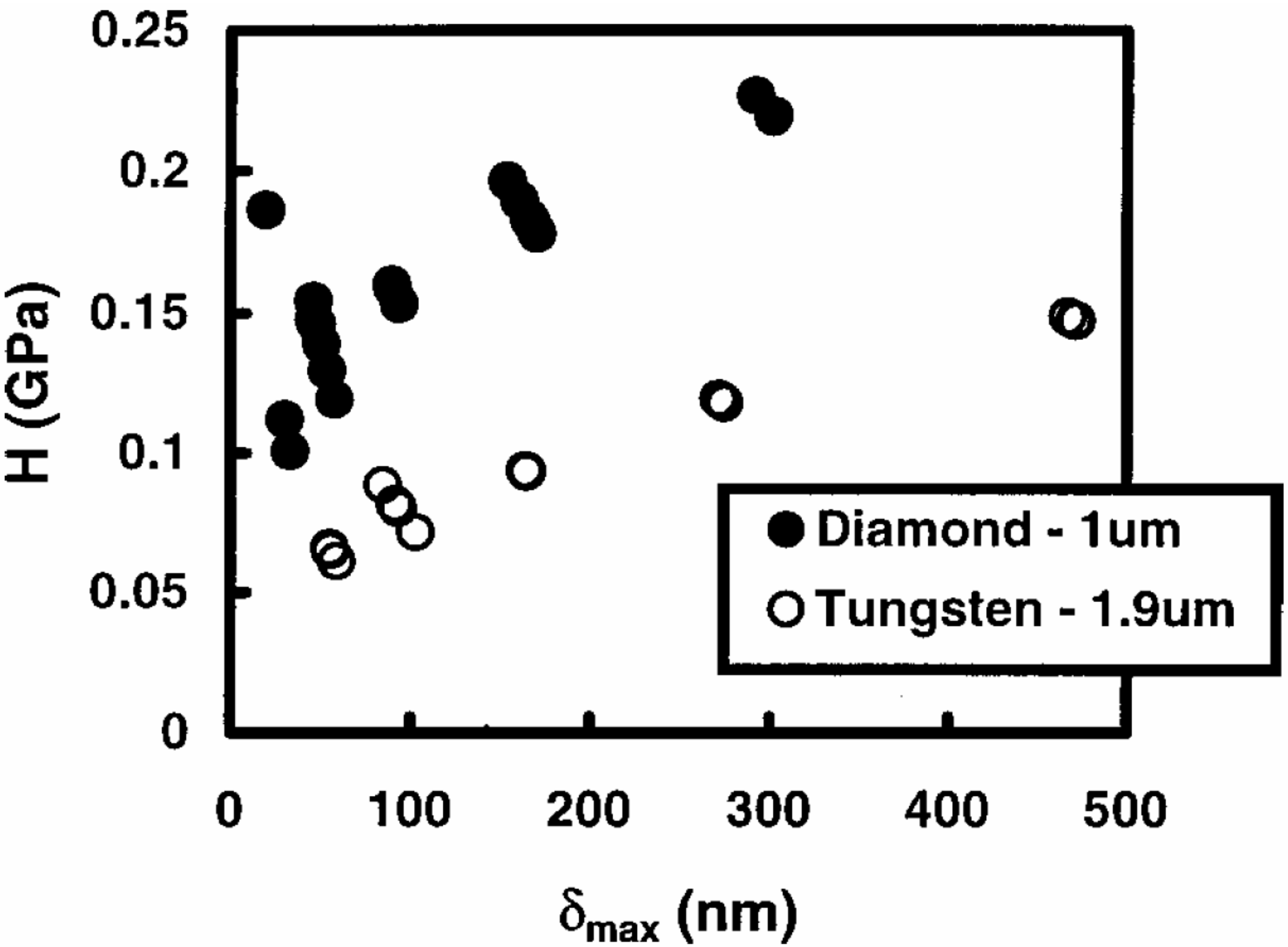
Rev. Sci. Instruments, Grunlan, *et al*. (2001)

Indentation into bulk LDPE



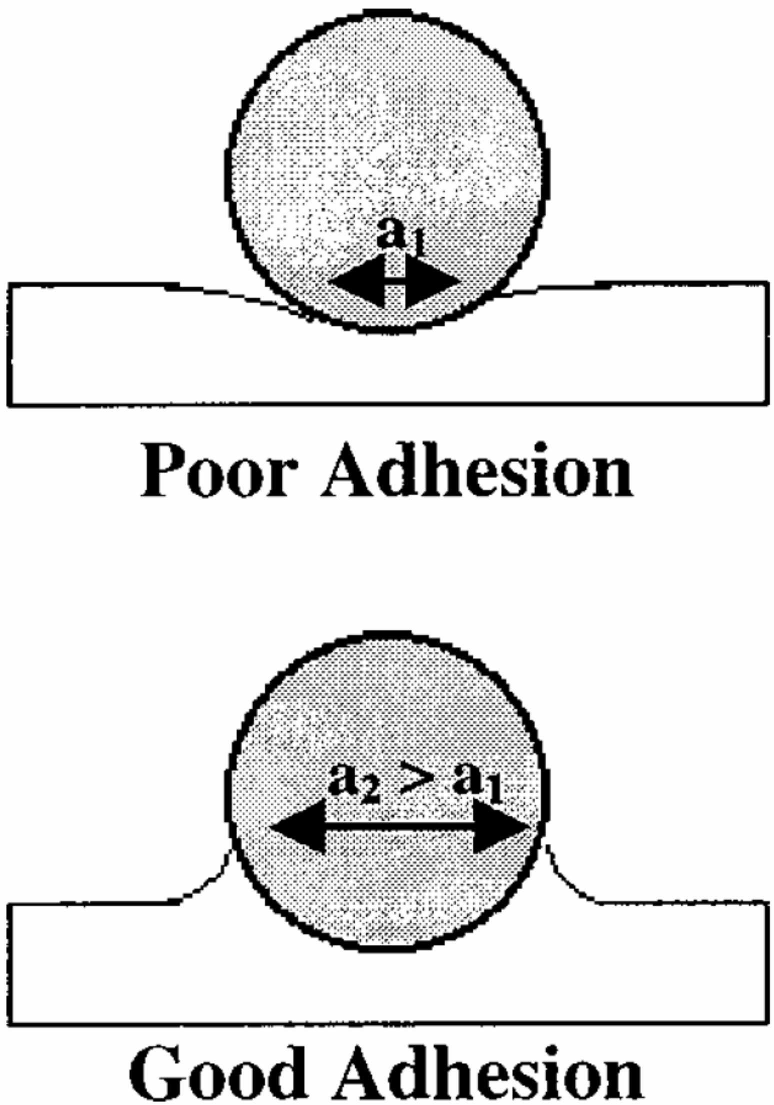
Rev. Sci. Instruments, Grunlan, *et al*. (2001)

Indentation into bulk Polystyrene



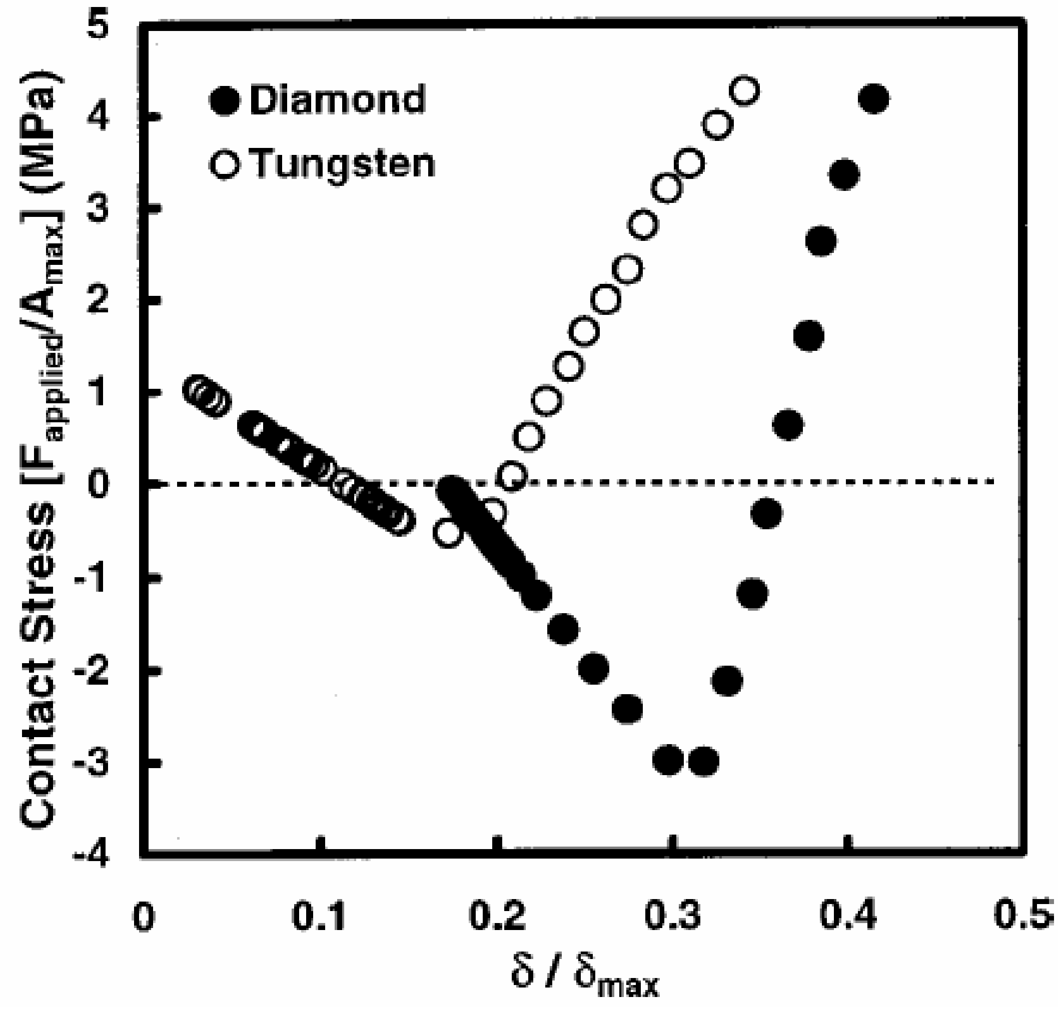
Rev. Sci. Instruments, Grunlan, *et al*. (2001)

Contact Size Variation



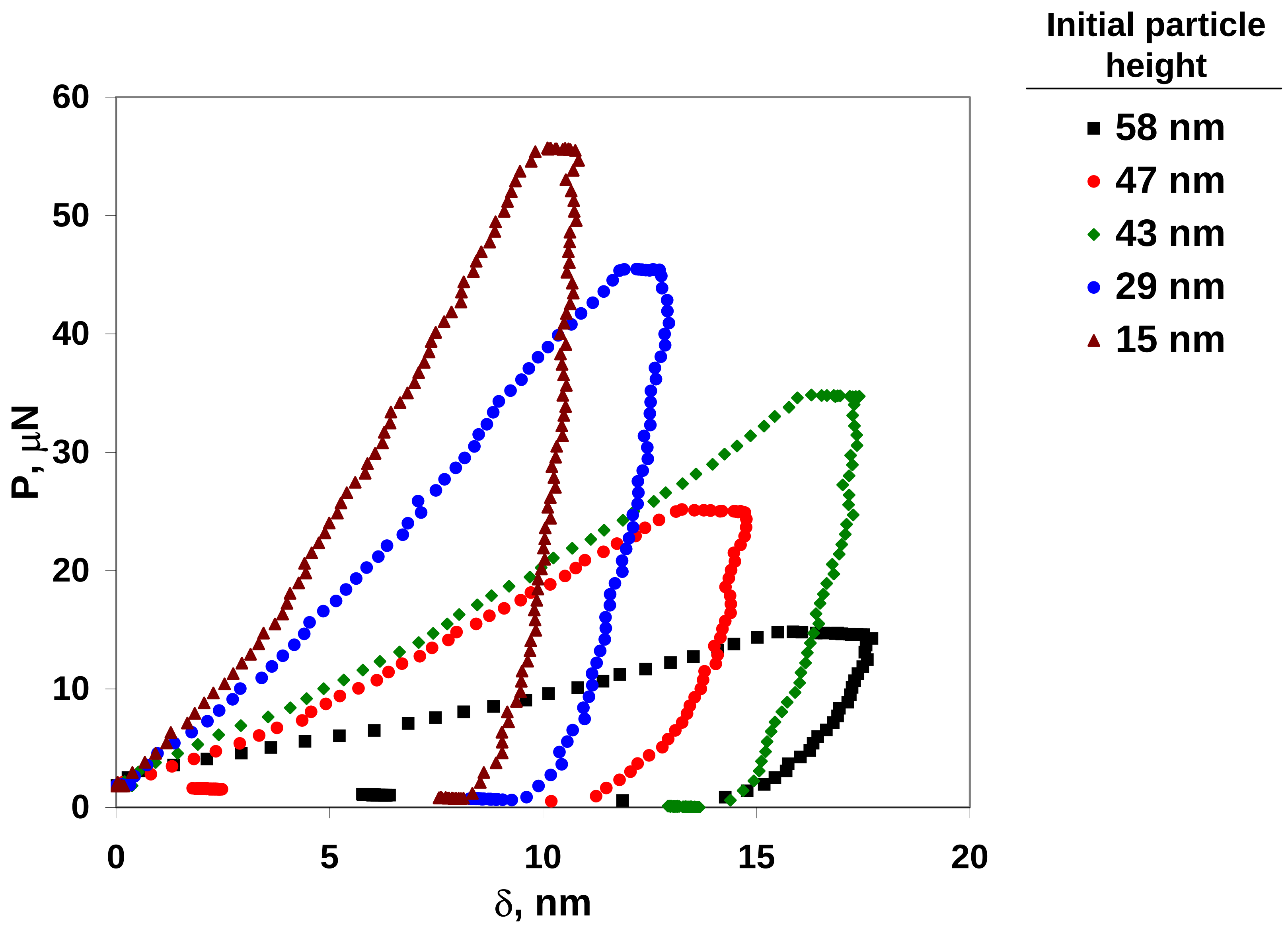
<u>Rev. Sci. Instruments</u>, Grunlan, *et al*. (2001)

Pull-off from Indentation into Polystyrene

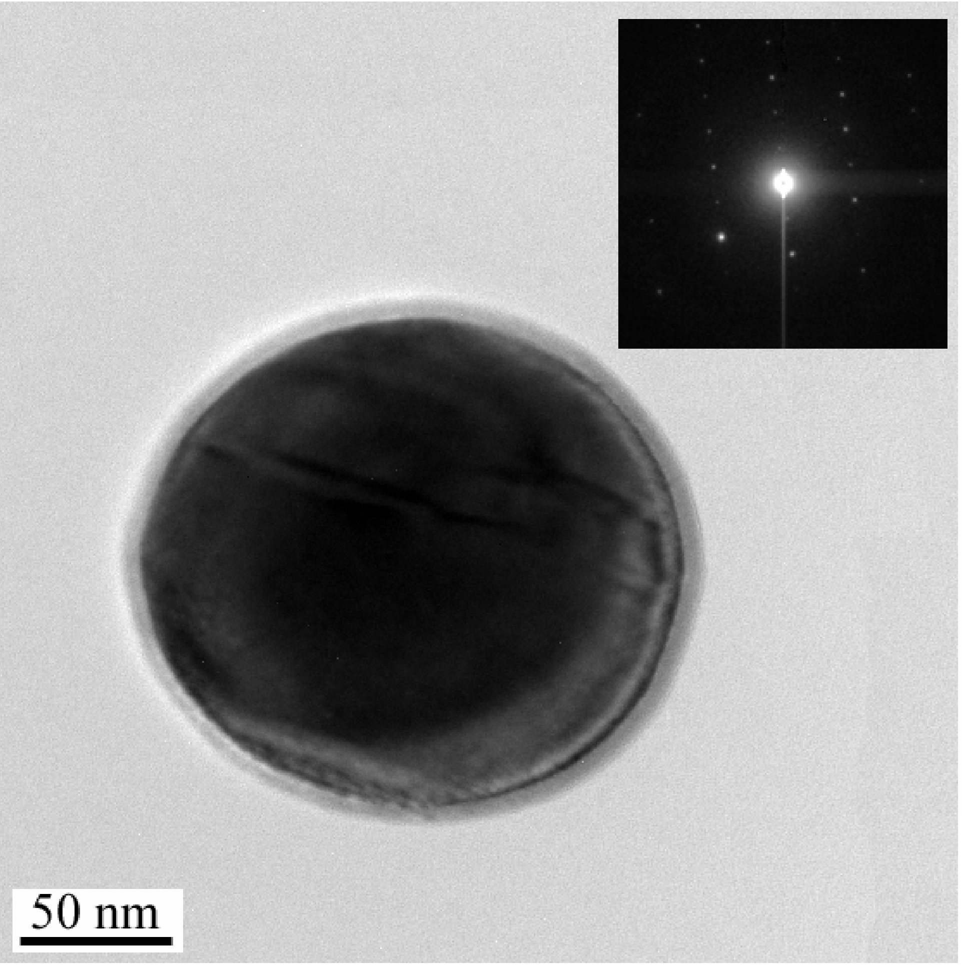


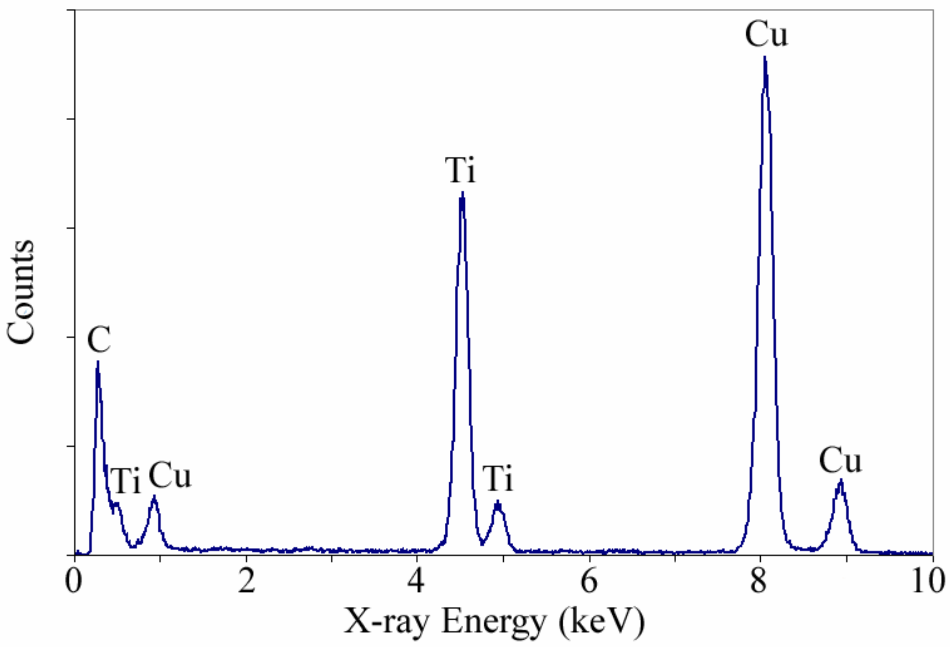
<u>Rev. Sci. Instruments</u>, Grunlan, *et al*. (2001)

Ti Nanoparticle, h = 58 nm



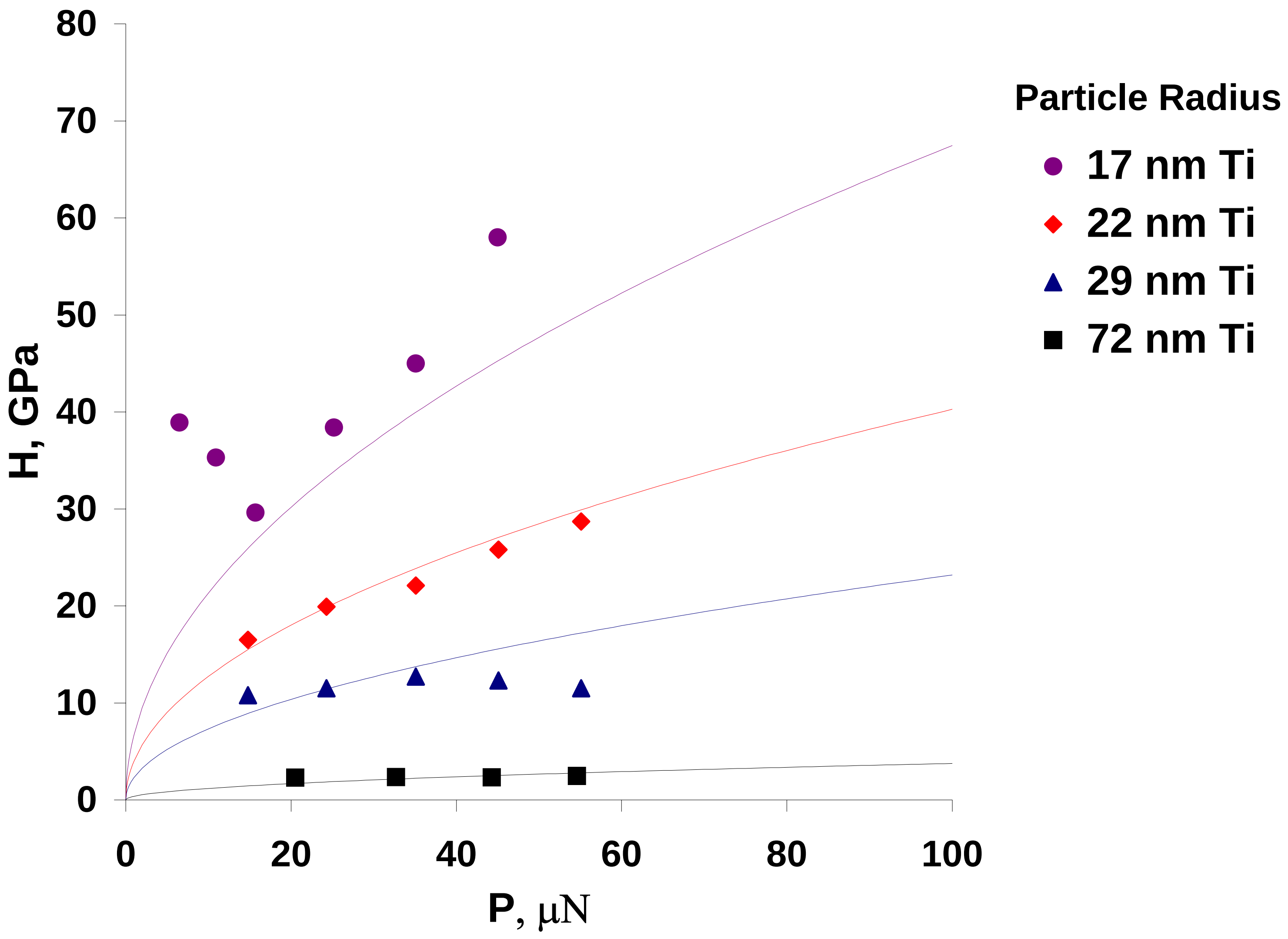
TEM of Ti Nanoparticle





Courtesy of C.R. Perrey and C.B. Carter, University of Minnesota

Ti Nanosphere Contact Pressure

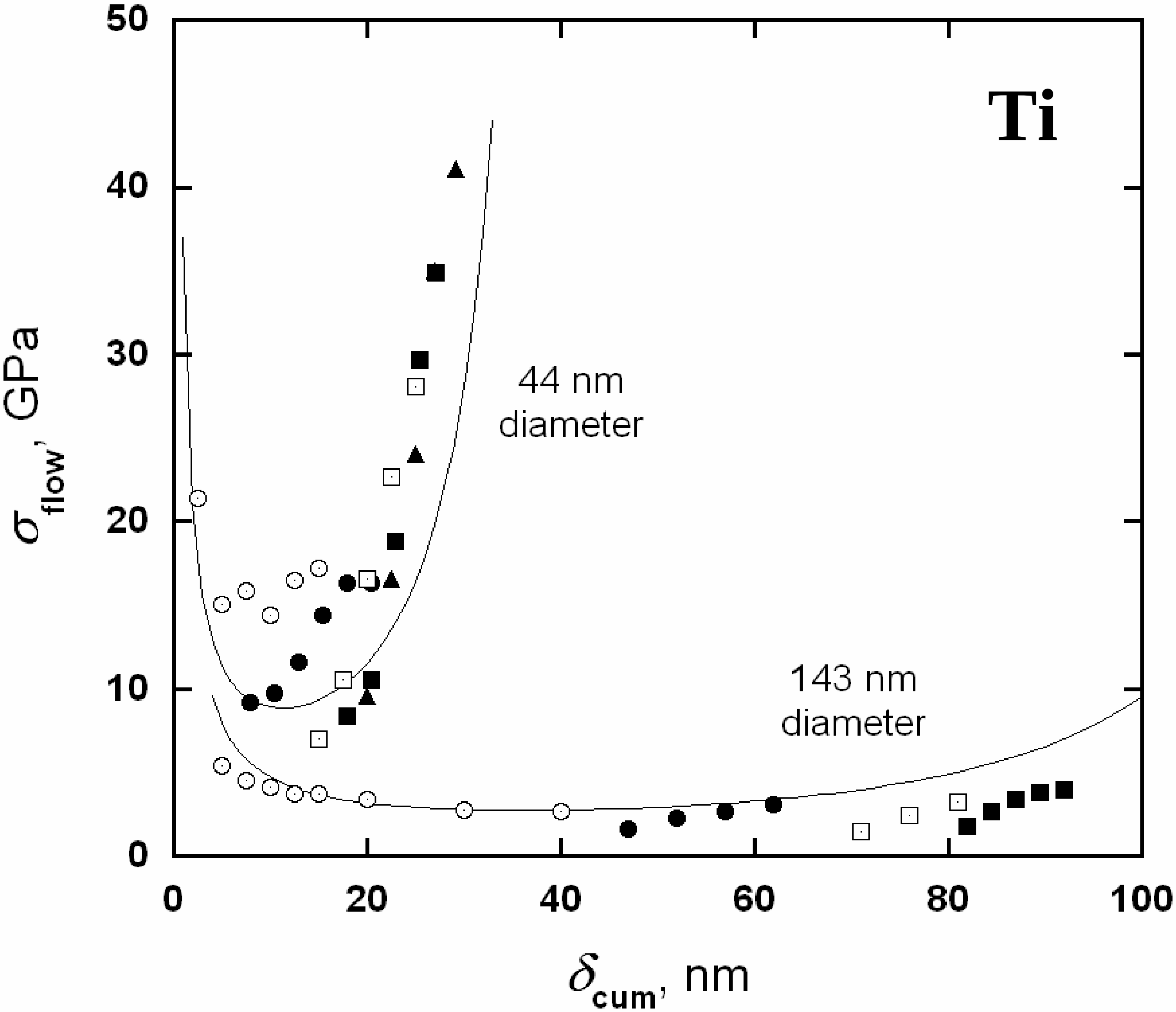


Hardness vs. Displacement

Using length scale $\frac{V}{S} = \frac{2r^3}{3a^2}$, one can show

$$H = \underbrace{\frac{2}{3}\sigma_{ys}\left(\frac{r}{\delta}\right)}_{\text{Surface Dominant}} + \underbrace{\frac{3\mu}{2\pi(1-\nu)}\left(\frac{\delta}{r}\right)^2}_{\text{Plastic Pile-up Dominant}}$$

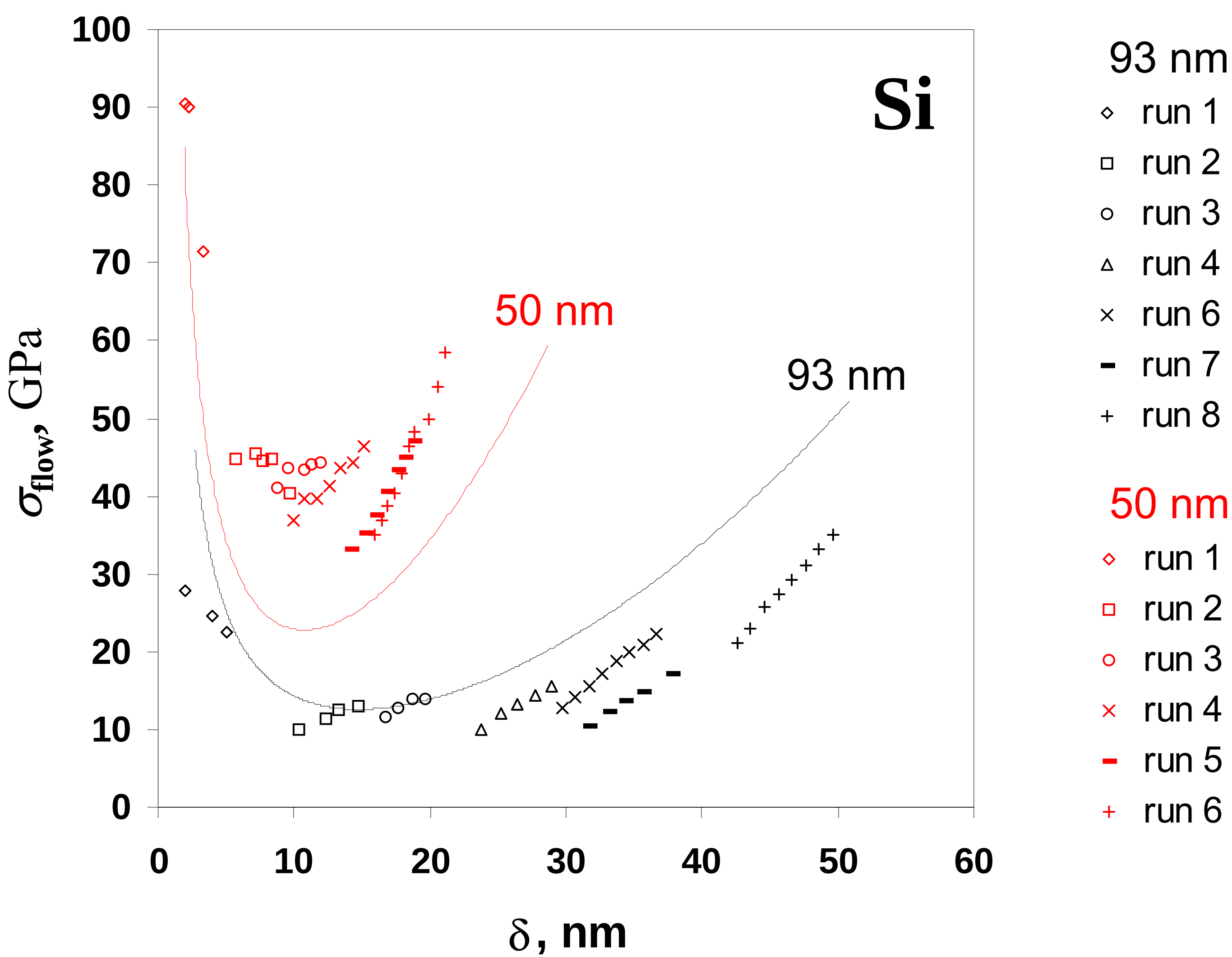
σ_{ys} is the only variable which can be back-calculated from the minima.



Ti
44 nm
diameter
143 nm
diameter
σ_{flow}, GPa
δ_{cum}, nm
0
10
20
30
40
50
0
20
40
60
80
100

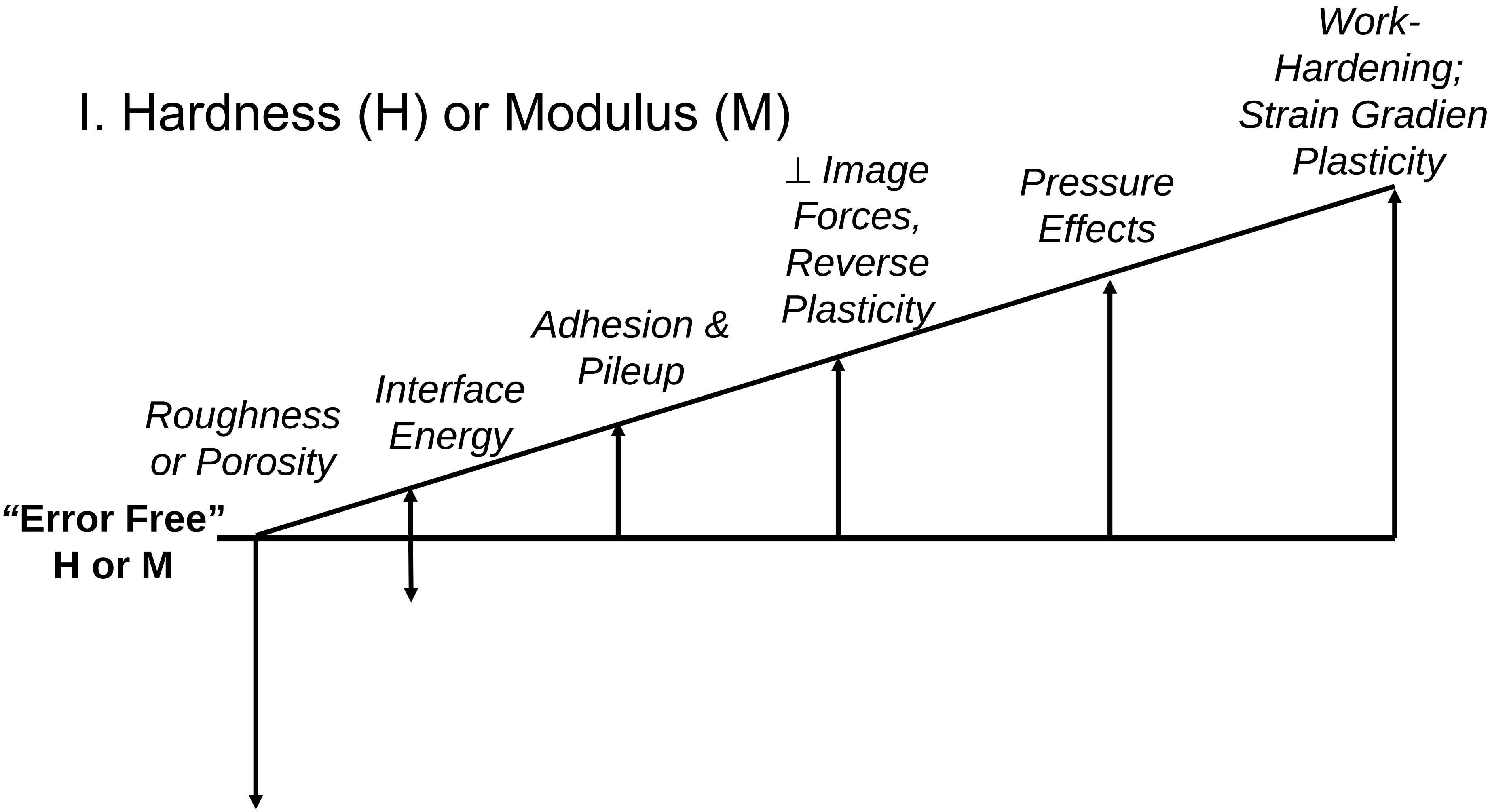
• Simple Superposition :

$$\sigma_{flow} = \frac{2}{3}\sigma_{ys}\left(\frac{r}{\delta}\right) + \frac{3\mu}{2\pi(1-\nu)}\left(\frac{\delta}{r}\right)^2$$



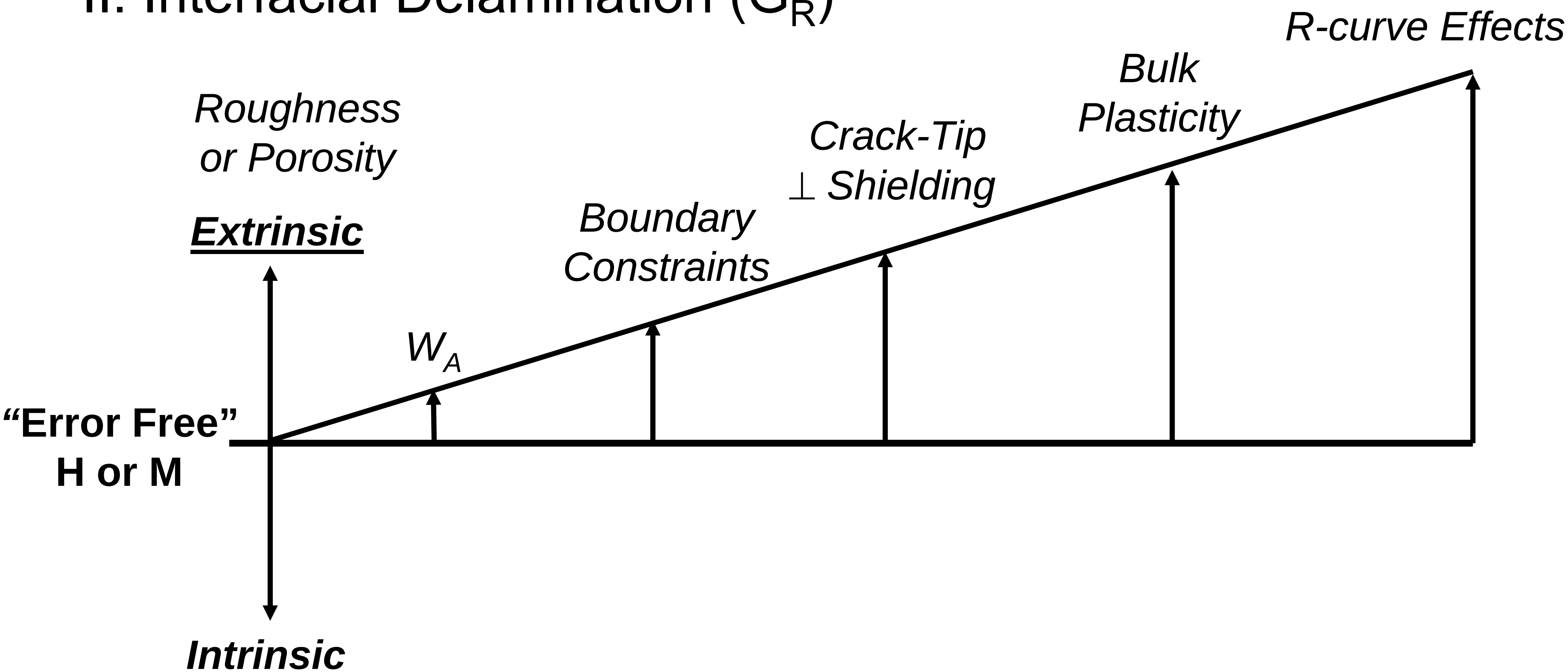
Measurement Road Maps: Nanoindentation

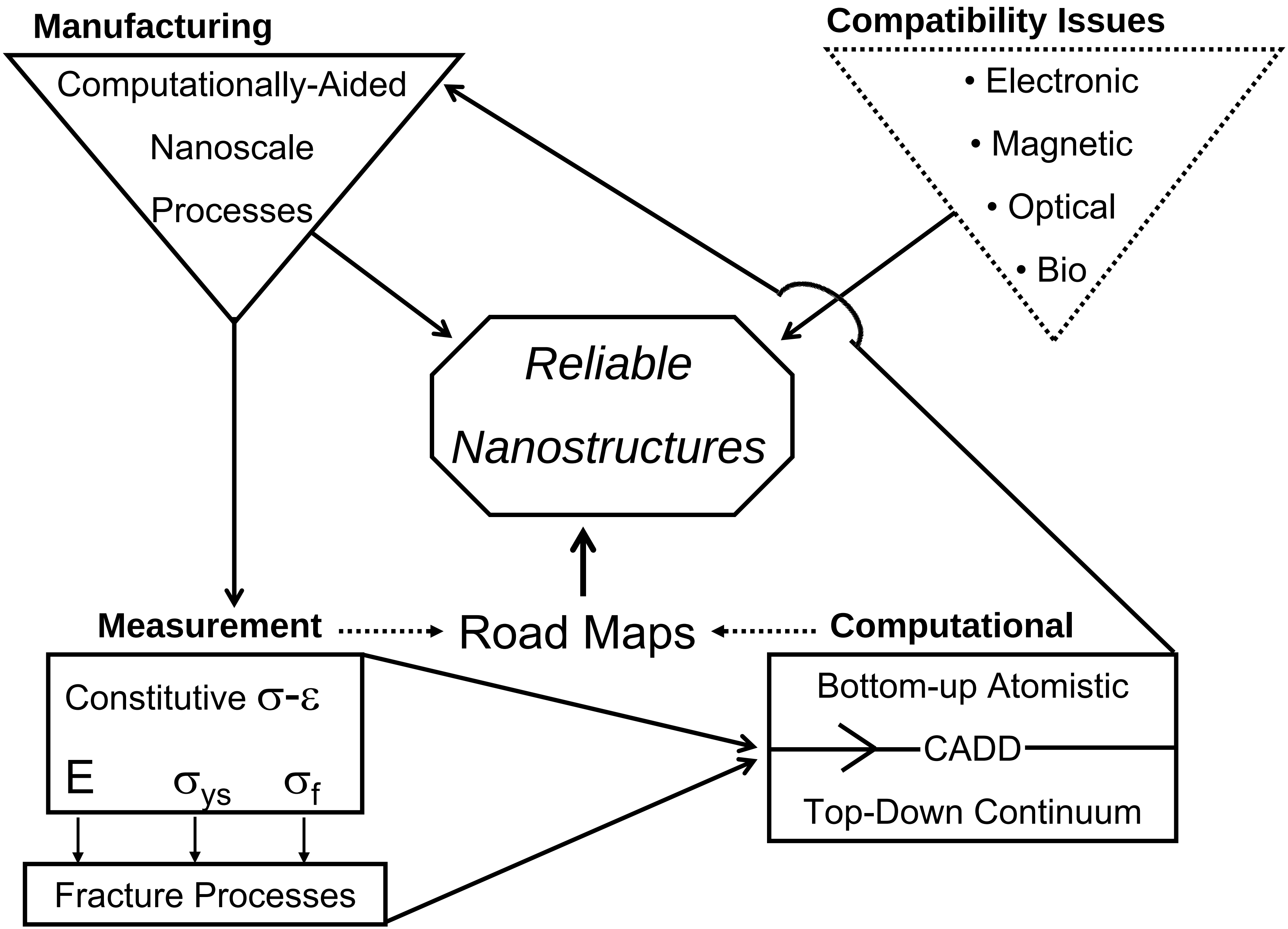
I. Hardness (H) or Modulus (M)



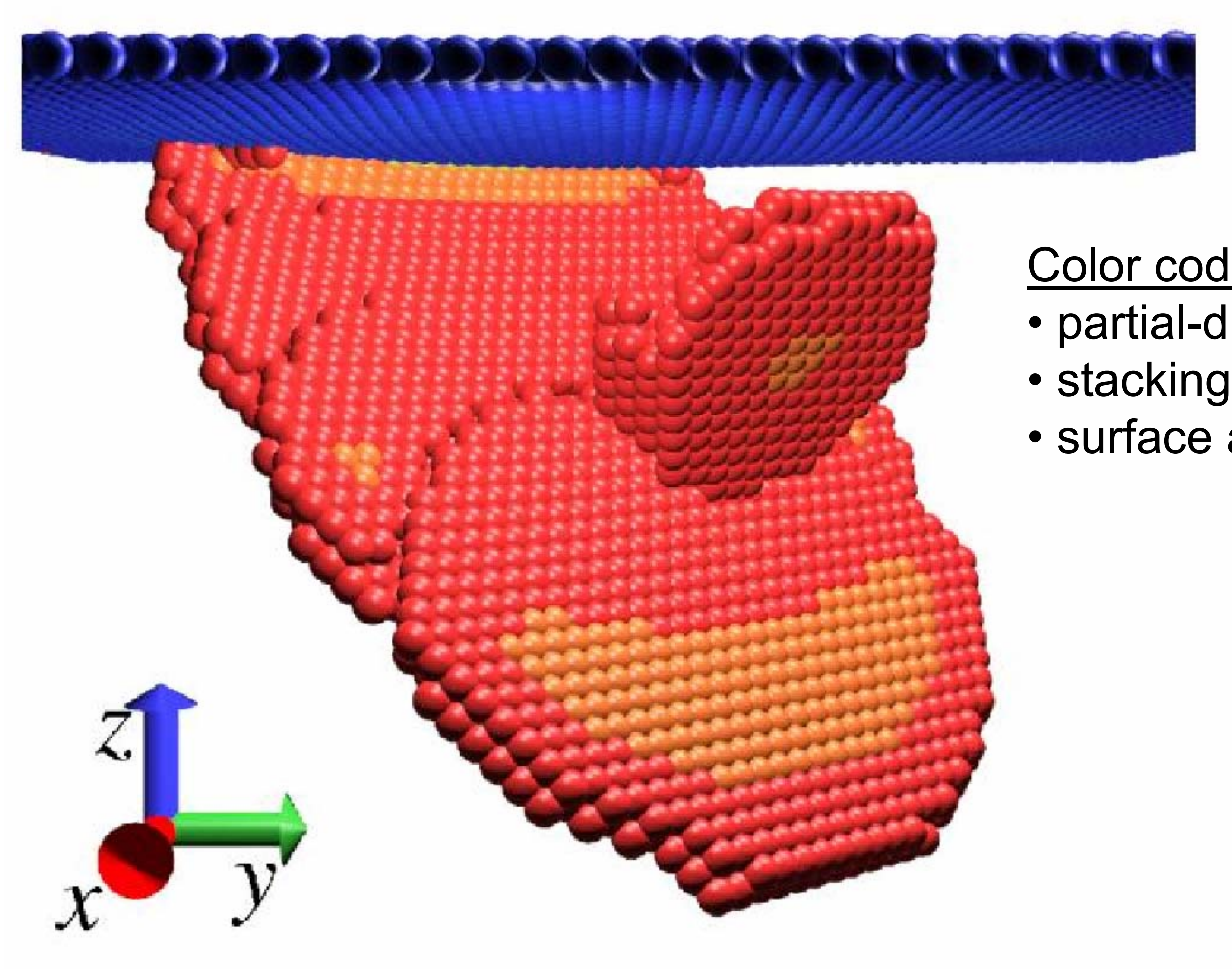
Measurement Road Maps: Nanoindentation

II. Interfacial Delamination (G_R)




Manufacturing
Computationally-Aided
Nanoscale
Processes
Compatibility Issues
• Electronic
• Magnetic
• Optical
• Bio
Reliable
Nanostructures
Measurement
Road Maps
Computational
Constitutive σ-ε
E
σ_{ys}
σ_f
Fracture Processes
Bottom-up Atomistic
CADD
Top-Down Continuum

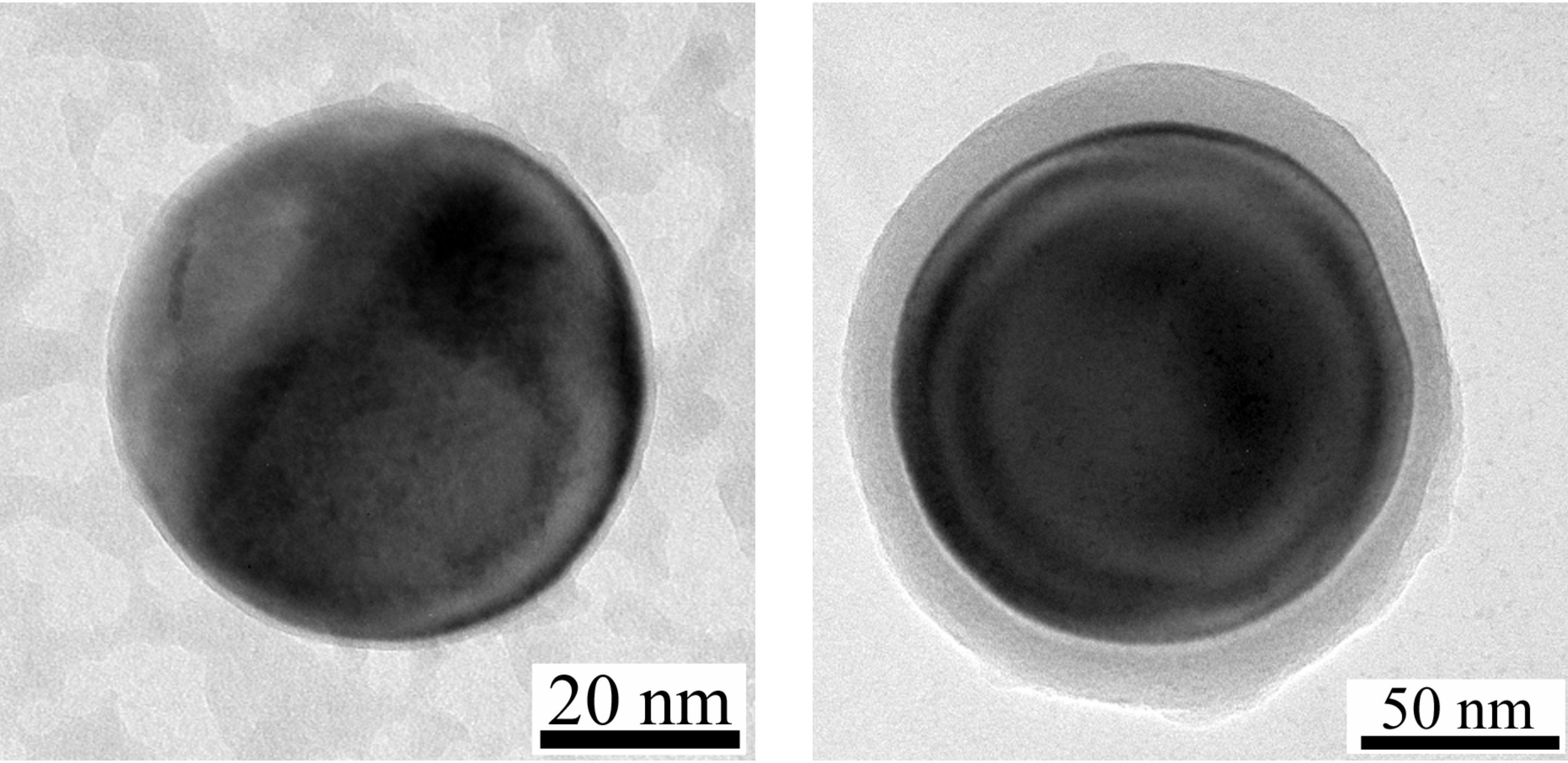
Dislocation pattern resulting from indentation into (001) gold



Color coding
- partial-dislocation core atoms (red)
- stacking-fault atoms (yellow)
- surface atoms (blue)

M. Ortiz, A.M. Cuitino, J. Knap and M. Koslowski, *MRS Bulletin,* **26** (3) 216-221 (2001)

Si Nanospheres: Variation of Oxide Thickness



Courtesy of C. Perrey and C.B. Carter, U. of Minnesota