Nanomechanical Characterization and Metrology for Low-k / ULK Materials

Ude Hangen

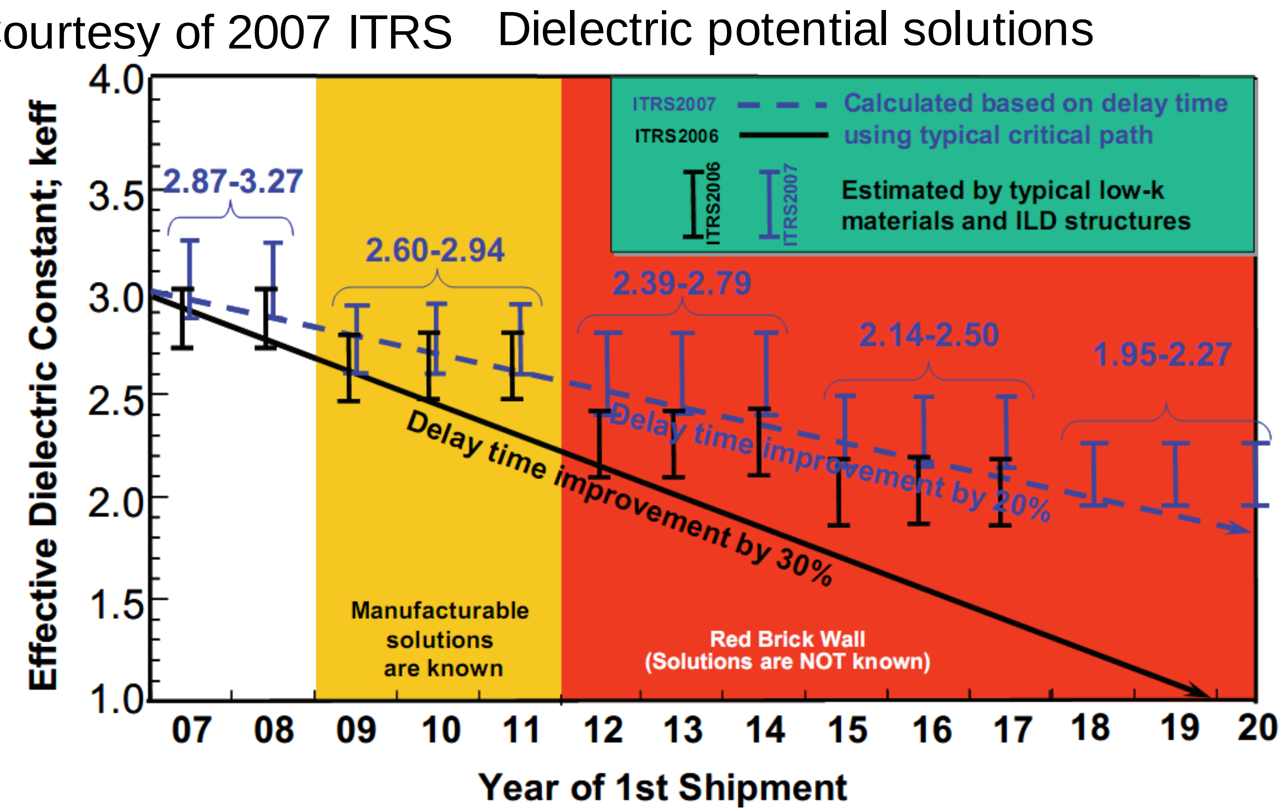
Hysitron Inc.

Outline

- Introduction
- Nanomechanical measurement methods
- Results
  - Nanoindentation
  - Creep
  - Nanoscratch to characterize interfacial Adhesion
  - Wedge indentation to characterize interfacial Adhesion
- Summary
- Concluding Remarks

Introduction

Courtesy of 2007 ITRS



Metrology Challenges

1. Mechanical Strength
   Modulus
   Hardness
   Fracture
2. Interfacial Adhesion
   Delamination
3. Pore size distribution

Low-k Materials	Continuous Improvement	Qualivication/Pre-Production	Development Underway
CVD-OSG	2.8≤k≤3.2	2.4≤k≤2.7	2.0≤k≤2.3
Spin-on Polymer	k=2.6	k=2.3	k=2.0
Spin-on MSQ	NA	2.4≤k≤2.7	2.0≤k≤2.3

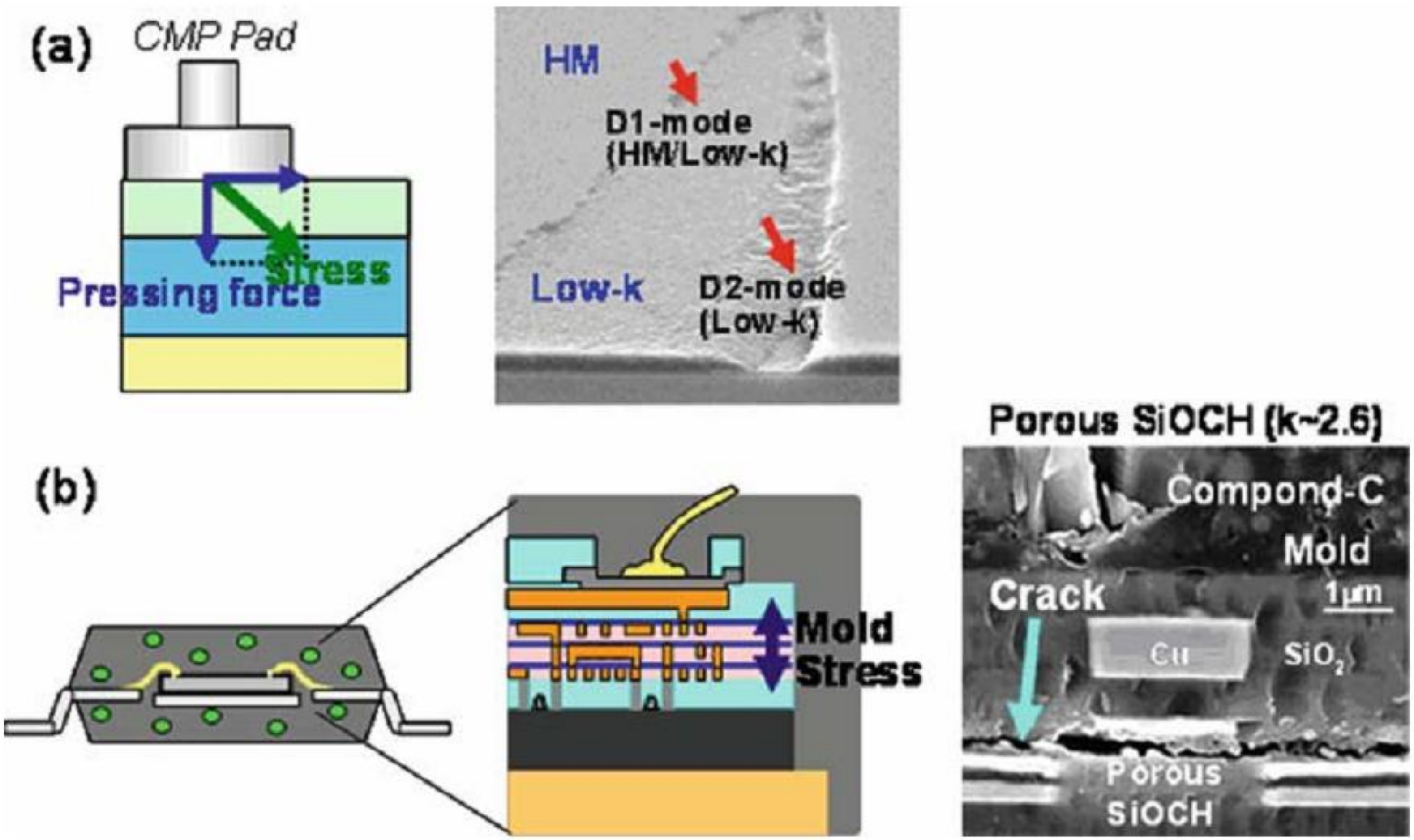
Introduction

HYSITRON

	Items	Method
1.	Dielectric Constant	Hg Probe
2.	**Young's Modulus**	**Nanoindentation**
3.	**Hardness**	**Nanoindentation**
4.	Structure	XPS, FT-IR
5.	Pore Size	Small Angle X-ray Scattering
6.	Thermal Stability	TDS, TG-DTA
7.	Moisture Absorption	TDS
8.	Thermal Conductivity	3ω method
9.	Leakage Current	Hg Probe, Al Dot
10.	Process Compatibility	Dielectric Constant, FT-IR, Damage
11.	**Adhesion to substrate**	**Scratch; Wedge Indentation**
	•Solvent, Etching, Ashing, CMP	

Introduction

CMP performance depends strongly on the mechanical property of the low-k material

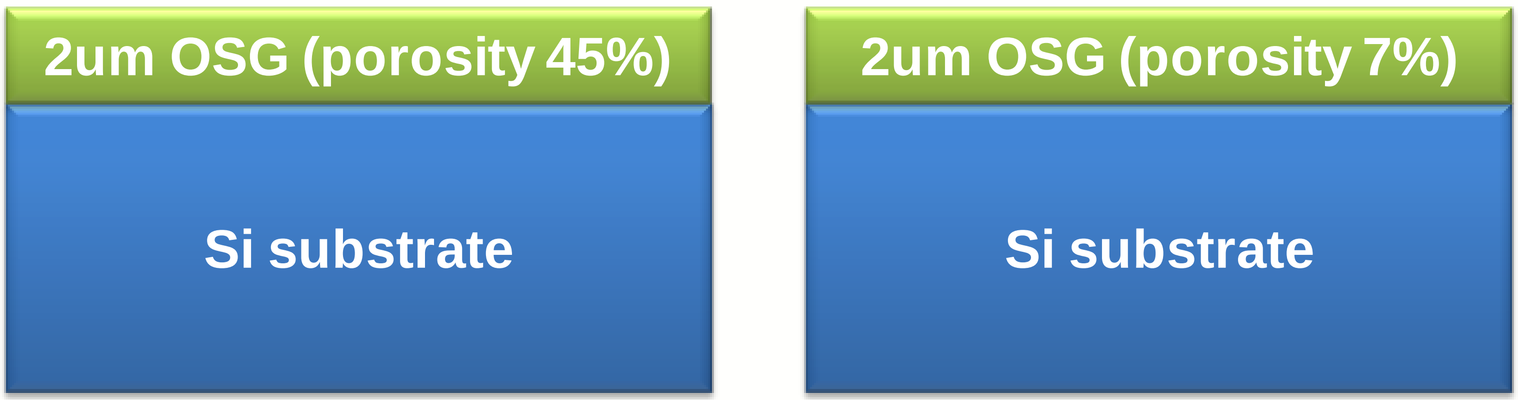


Courtesy of Tagami, M

Introduction

- What: Porous **Organosilicate glasses (OSG)** low-k materials containing nanometer-size pores prepared by PE-CVD using diethoxy-methylsilane DEMS as a precursor at 250°C with Helium and oxygen gas as carrier gas and oxidizer.
- Why: **Mechanical properties** are critical
  The pore introduction degrades the mechanical strength of the low-k films and results in serious damages to ULSI interconnects such as the film de-lamination during CMP and/or cracking due to the thermal stress from packaging mold resin.
- How: **Nanomechanical measurements** using Hysitron's Triboindenter to characterize the Hardness, Modulus, Fracture Toughness, Adhesion, Elevated Temperature Behavior, and Wear Behavior.

Low-k materials



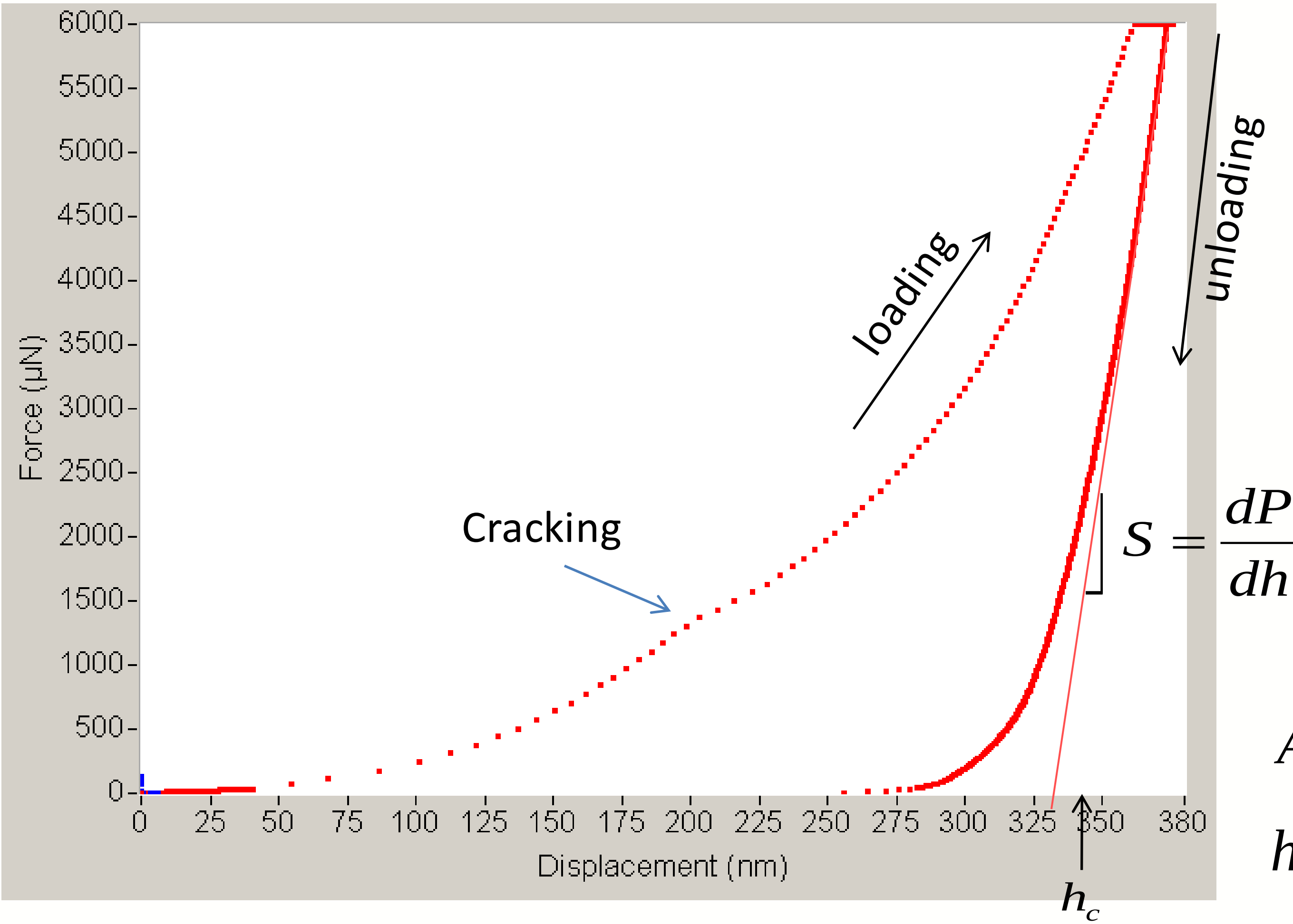
OSG45 **OSG7**

OSG #	Cap layer	Porosity	k
OSG45	No	45%	2.3
OSG7	No	7%	2.98

Hysitron TriboIndenter

- Berkovich, Conical probe
- Hardness, Reduced Modulus, and Fracture Toughness
- Elevated Temperature
- Creep
- nanoScratch
- nanoWear
- Acoustic Emission with a cube corner probe
- Conical probe Creep and Scratch
- nanoECR – Electrical Measurements

Nanoindentation



$$E_r = \frac{S\sqrt{\pi}}{2\sqrt{A}}$$

$$H = \frac{P_{max}}{A}$$

$$A = 24.5 h_c^2$$

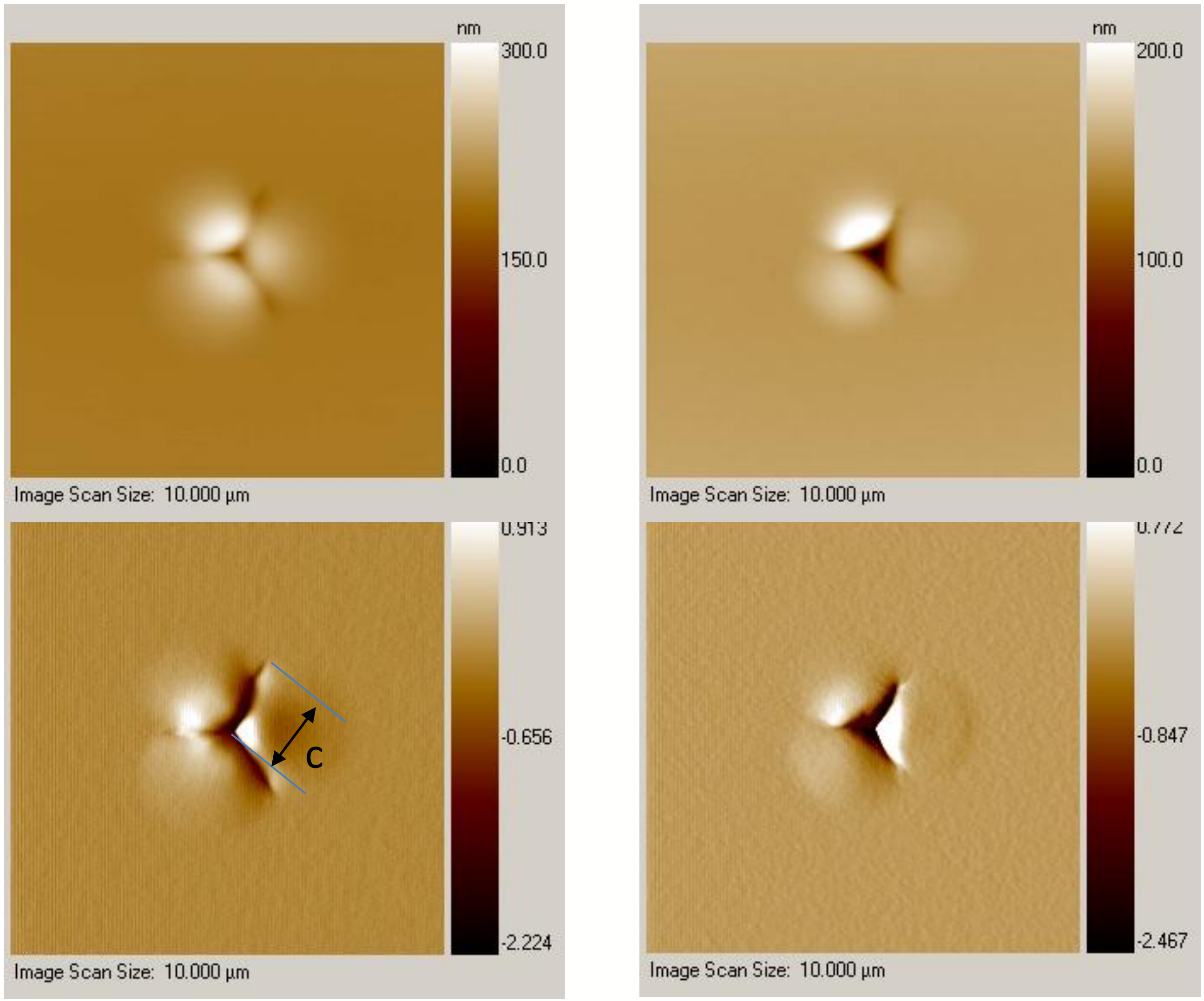
$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S}$$

OSG45

Nanoindentation

TOP

GRF



OSG45 **OSG7**

Nanoindentation

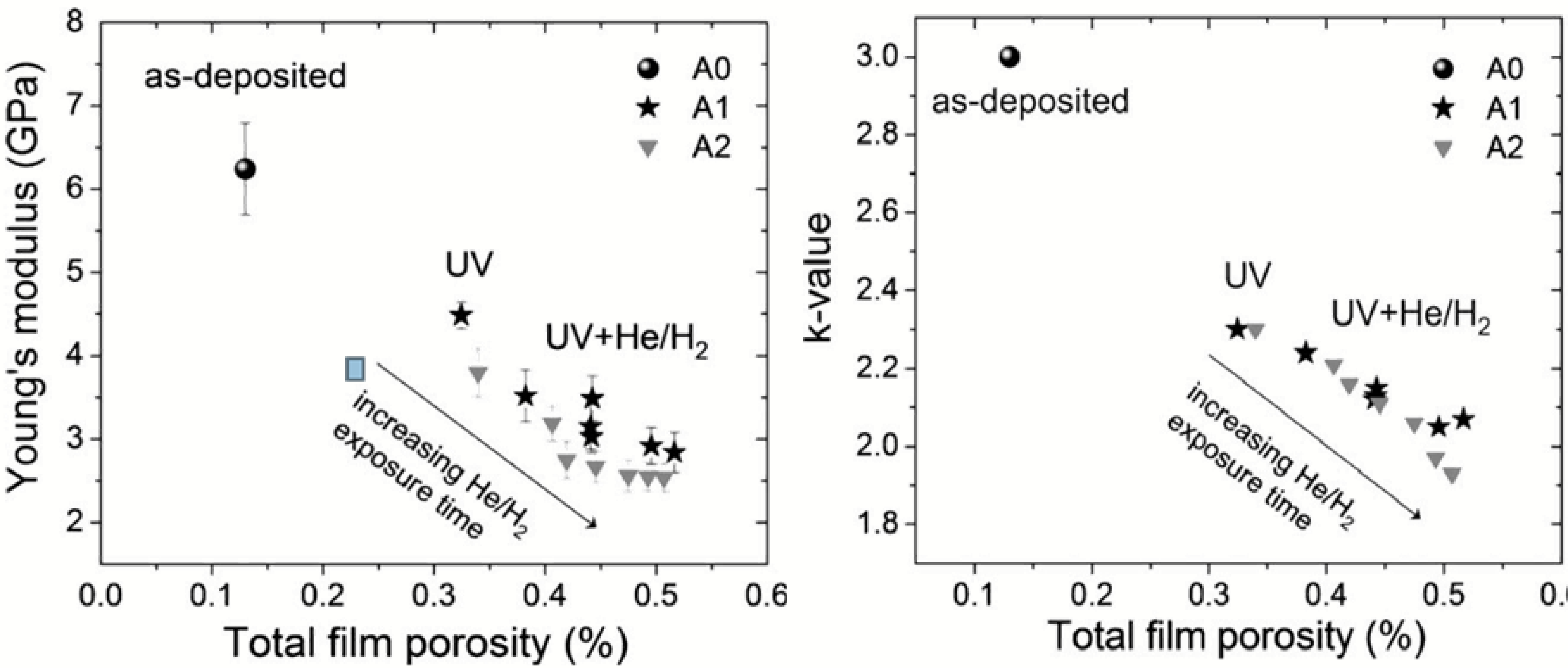
$$K_c = \alpha \left[\frac{E}{H}\right]^{\frac{1}{2}} \left[\frac{P}{c^{\frac{3}{2}}}\right]$$

Kc is the fracture toughness, E is the Young's Modulus, H is the hardness, P is the peak applied load, c is the length of the radial cracks, and α is an empirical constant taken as 0.016 for a Berkovich tip

	E (GPa)	H (GPa)	P (mN)	c (µm)	K_c ($MN/m^{3/2}$)
OSG45	7.1	0.34	6	1.78	0.14
OSG7	22	1.8	8	1.16	0.35

Modulus, k-value and Porosity

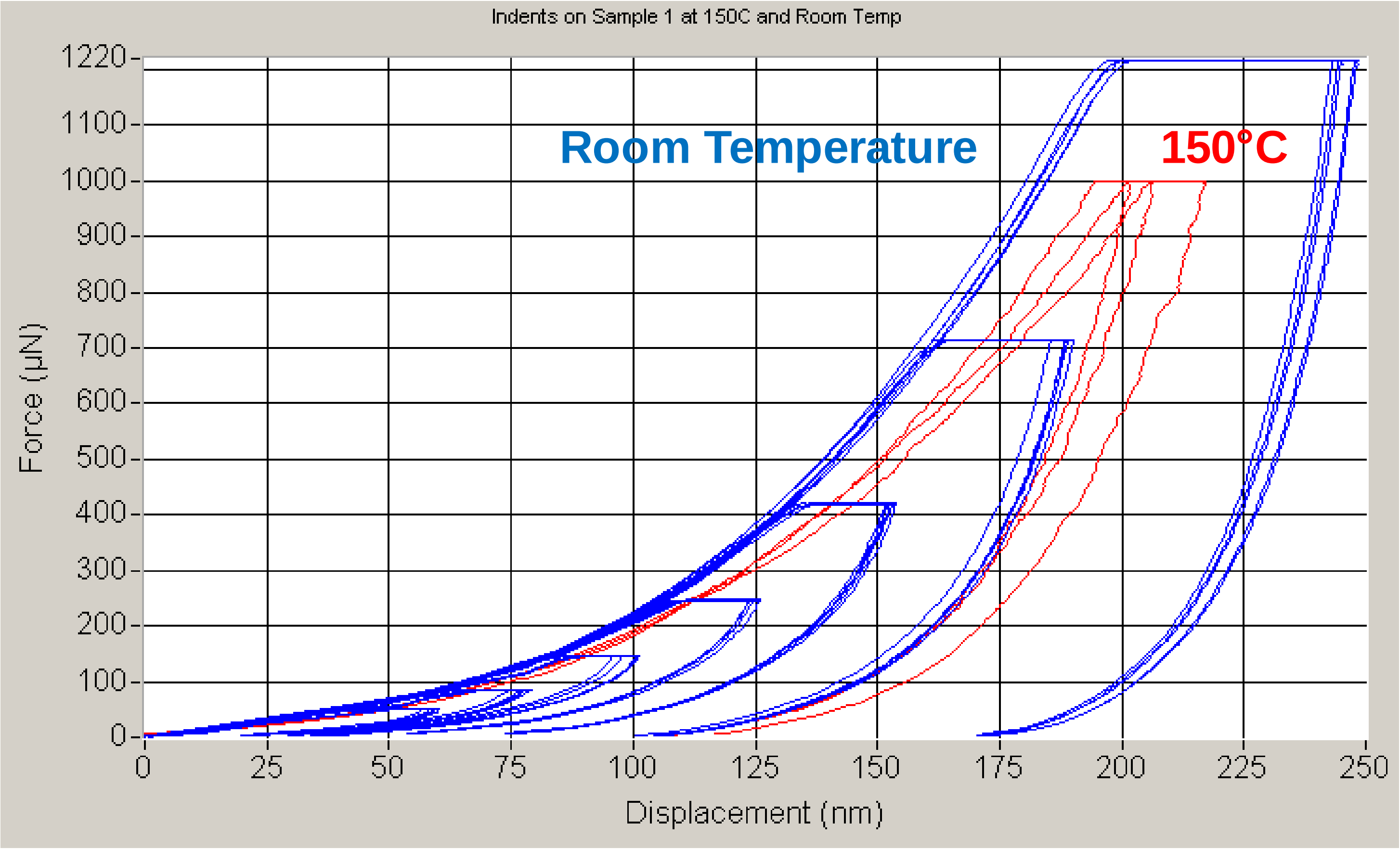
SiCOH – low –K exposed to H/He-plasma



K. Vanstreels and A. M. Urbanowicz
J. Vac. Sci. Technol. B, Vol. 28, No. 1, Jan/Feb 2010

=> For a known material Modulus would be enough to measure

Elevated Temperatures

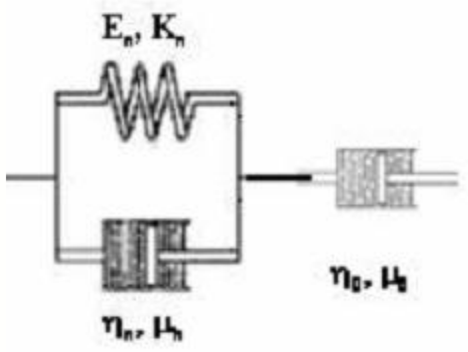


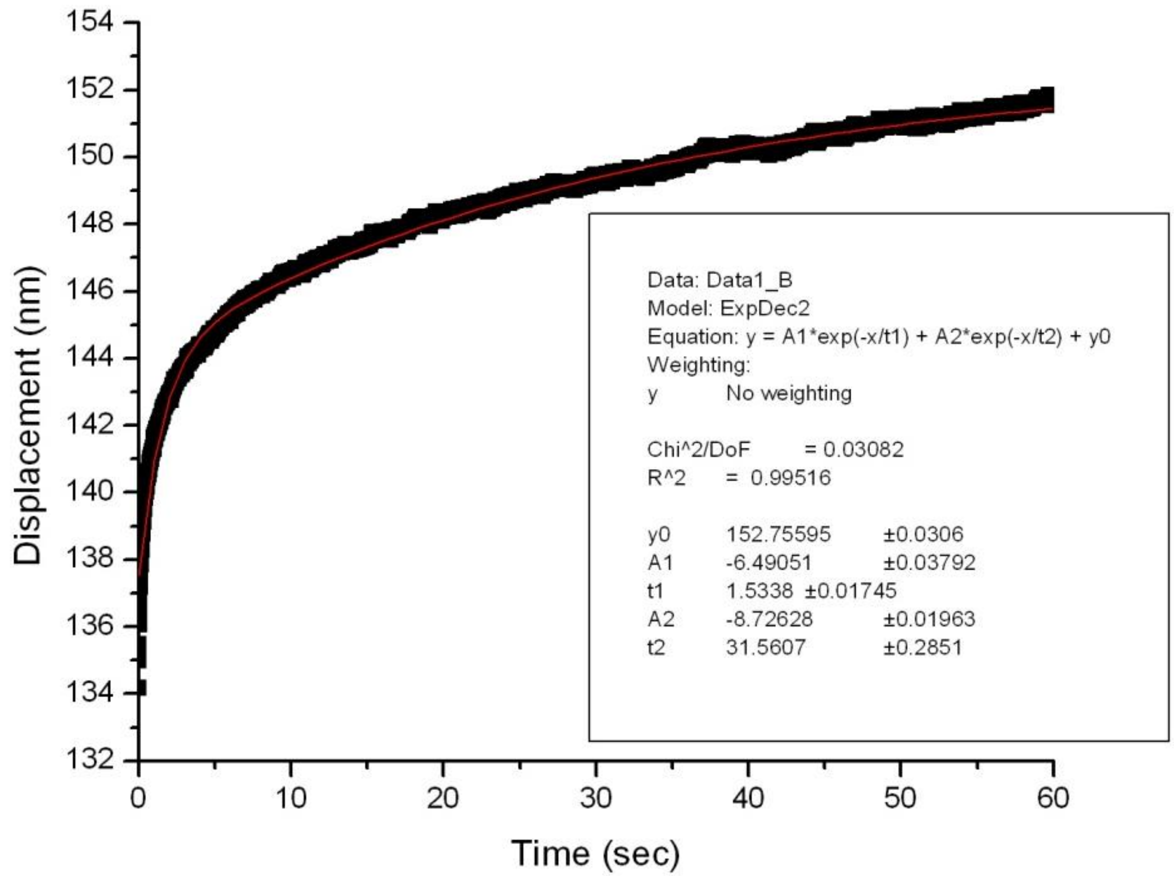
OSG45

Creep

$$h = h_{in}\frac{P_0}{E_0 A_0} + \sum_i^n \frac{P_0 h_{in}}{E_i A_0}\left(1 - e^{-E_i t/\eta_i}\right) + \frac{h_{in}}{\eta_0} t = h_e + \sum_i^n h_i\left(1 - e^{-t/\tau_i}\right) + t/\mu_0$$

Kelvin Model





OSG45, 420uN

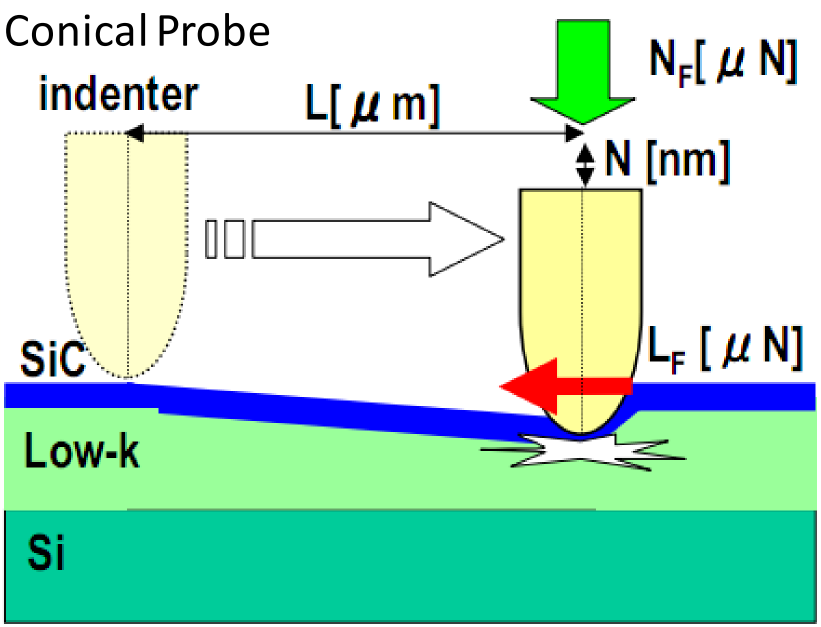
Creep

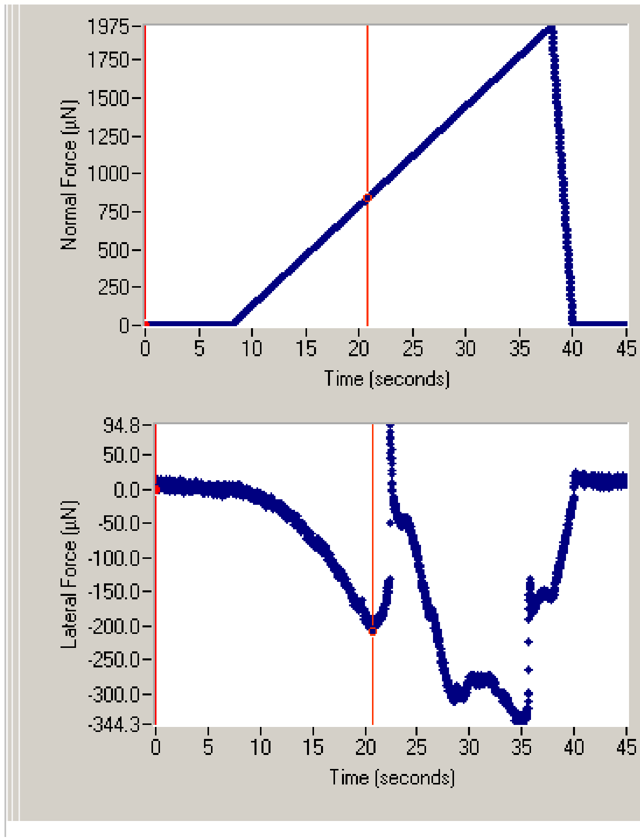
$$E_i = \frac{h_{in} \cdot P_i}{A_i} \boxed{\frac{1}{h_i}} \qquad \eta_i = t_i \cdot E_i = \frac{h_{in} \cdot P_i}{A_i} \boxed{\frac{t_i}{h_i}}$$

	h_0 (nm)	h_1 (nm)	h_2 (nm)	t_1 (s)	t_2 (s)	t_1/h_1 (s/nm)	t_2/h_2 (s/nm)	
OSG45	152.76	6.49	8.73	1.53	31.56	0.236	3.62	Lower viscosity
OSG7	135.36	1.38	2.37	0.66	14.23	0.478	6.00	Higher viscosity

Adhesion Measurement

Adhesion Measurement: nanoScratch

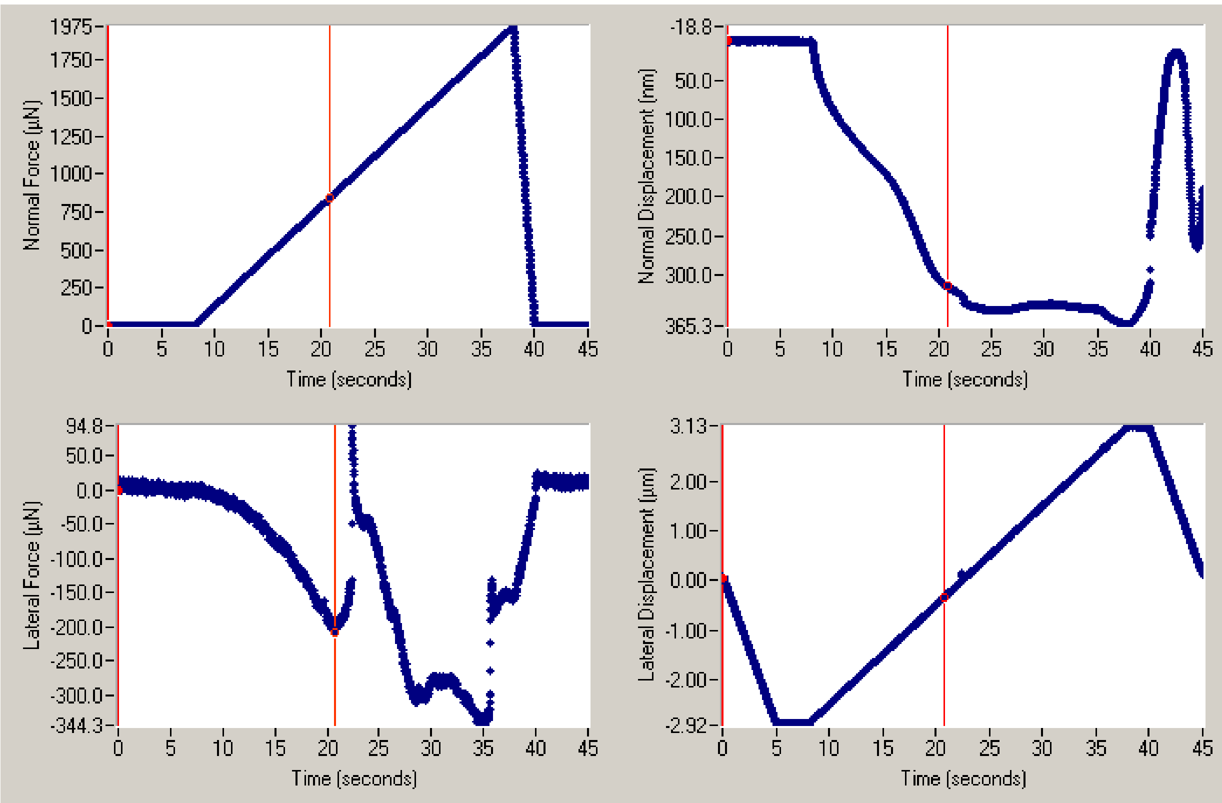


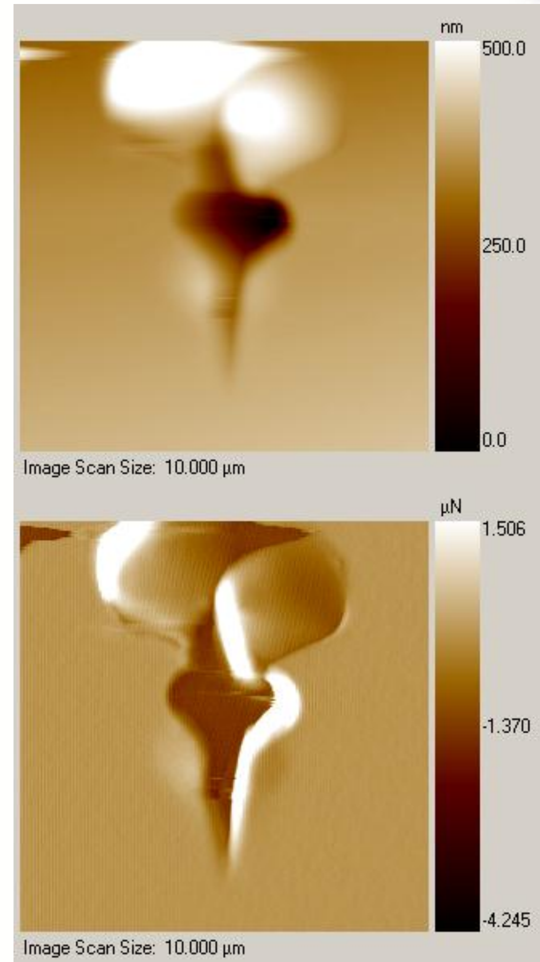


Normal Displacement : N [nm]
Normal Force : N_F[μN]
Lateral Displacement : L[μm]
Lateral Force : L_F[μN]

Friction μ ($=L_F/N_F$)

Scratch at Nanoscale

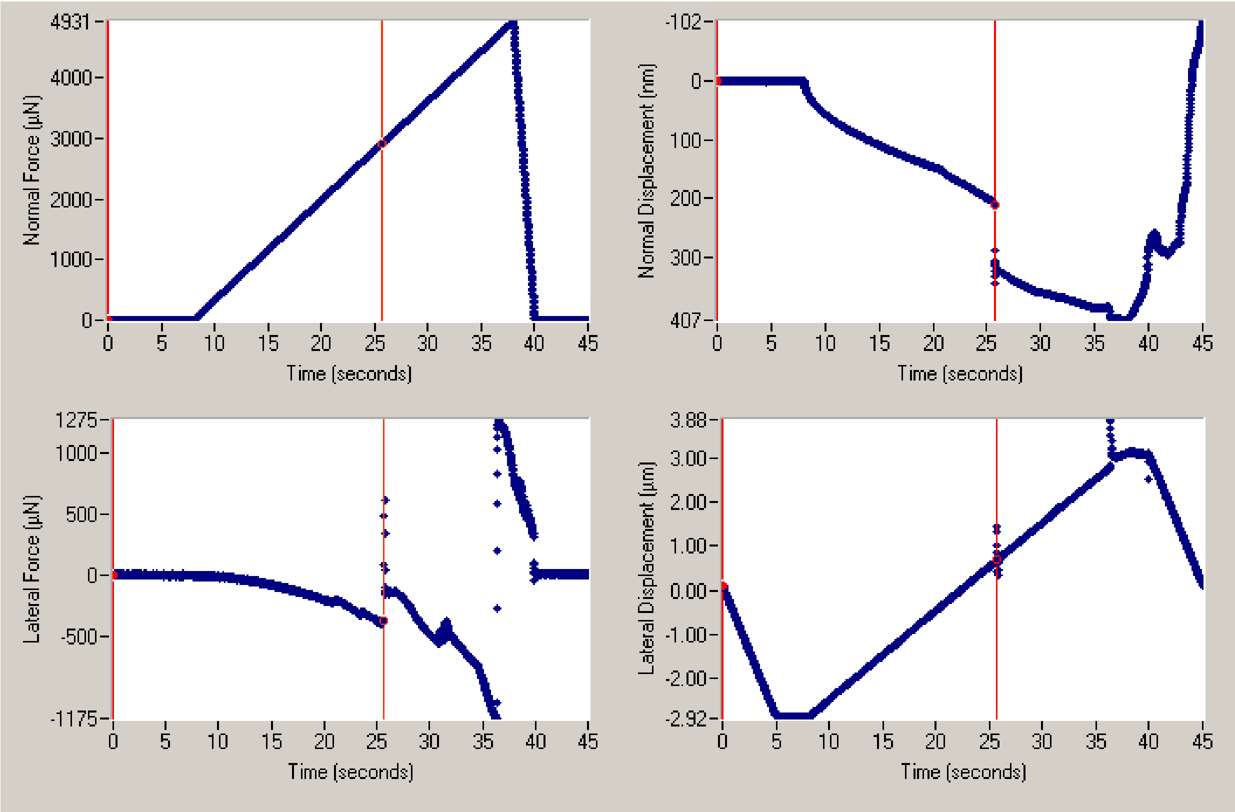


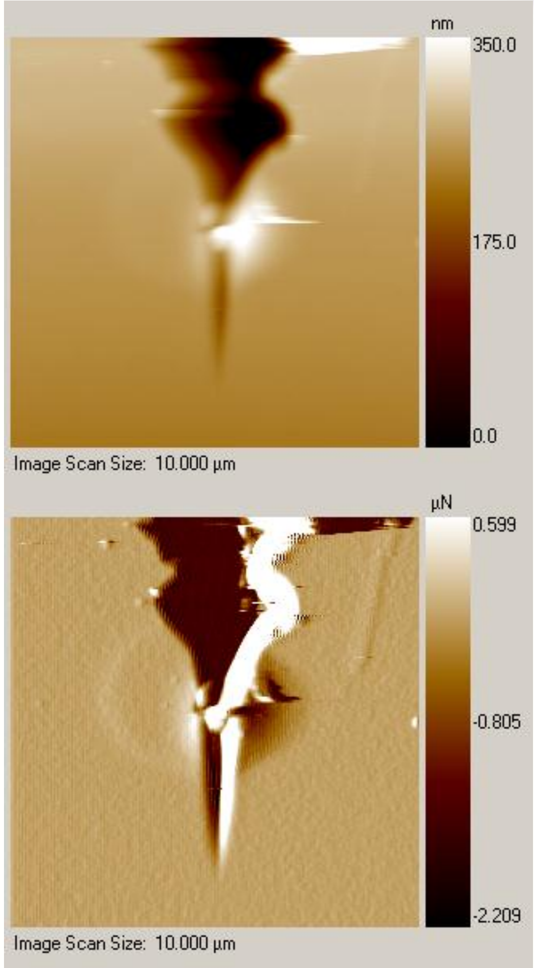


Friction Coefficient
LF/NF=200/750=**0.267**

OSG45

Scratch at Nanoscale

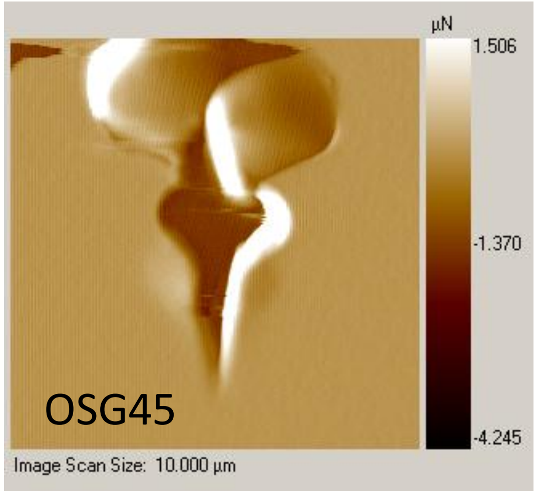


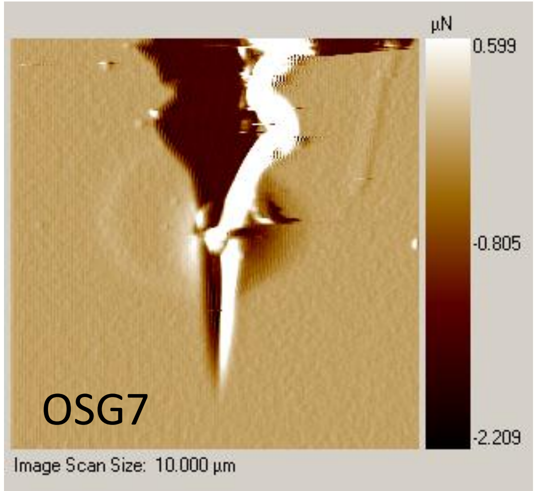


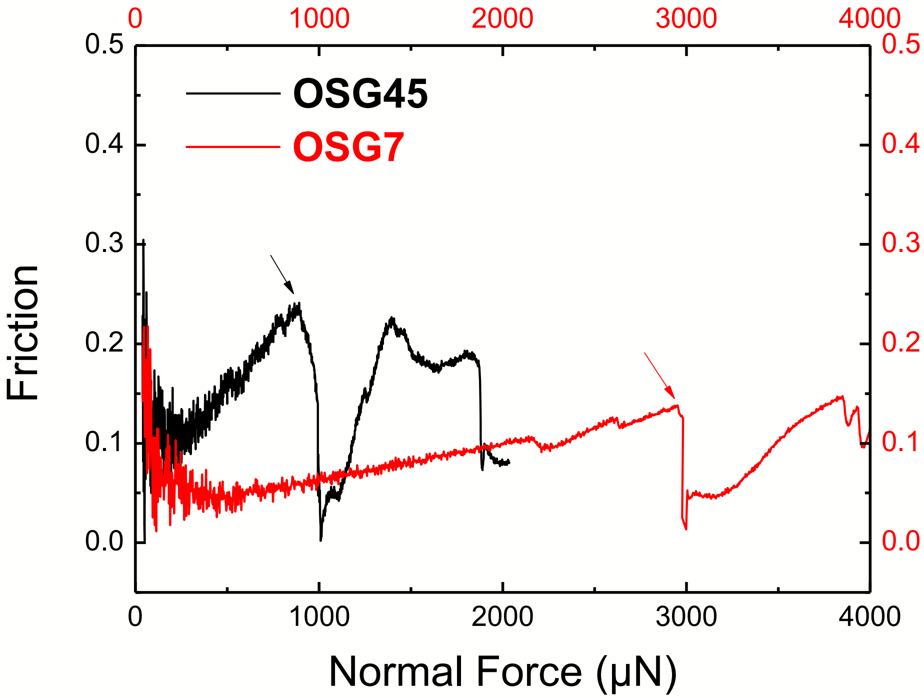
Friction Coefficient
LF/NF=400/3000=**0.133**

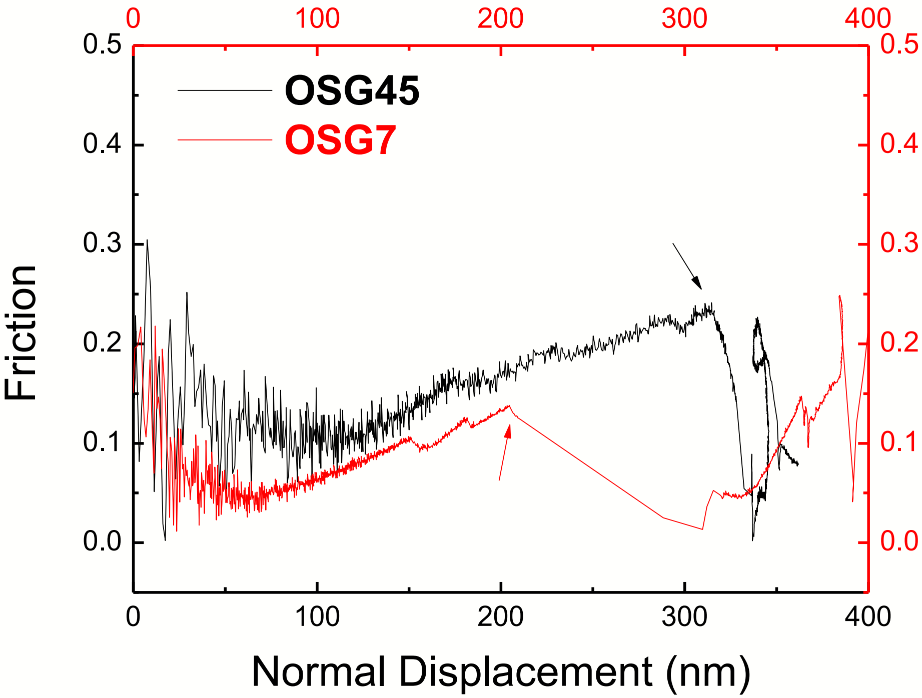
OSG7

Scratch at Nanoscale









Scratch at Nanoscale

HYSITRON

OSG45

The critical loads from the 2 mN ramping force scratch tests on Sample OSG45 using a 1 µm conical probe

Test #	Critical Load (µN)
1	705
2	843
3	894
4	738
Ave:	**795**
Std Dev:	**88**

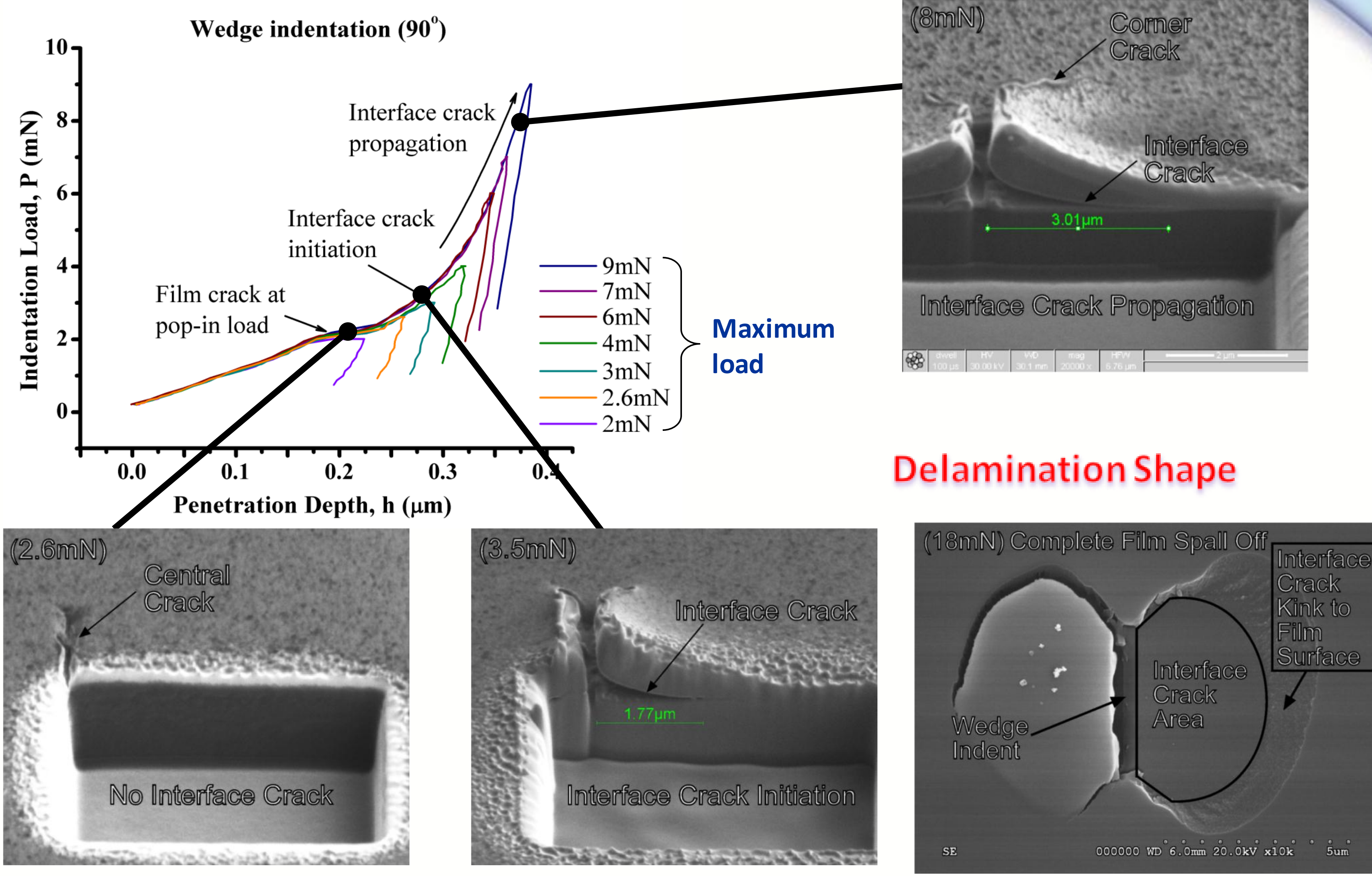
OSG7

The critical loads from the 5 mN ramping force scratch tests on Sample OSG7.4 using a 1 µm conical probe

Test #	Critical Load (µN)
1	2904
2	2917
3	2926
4	2967
Ave:	**2929**
Std Dev:	**27**

Reference load for CMP processing down force

Tip Independend Adhesion by Wedge-Indentation Induced Interfacial Fracture



Compressive Buckling and Crack Front Curvature

Buckling occurs when σ_o is larger than σ_c

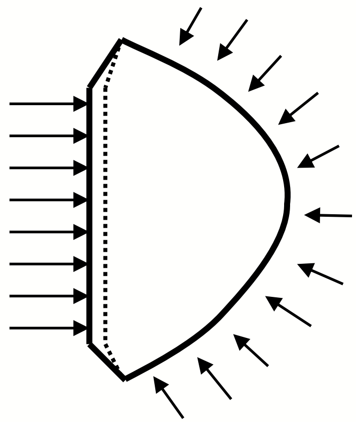
Interfacial toughness:

$$G_c = \frac{t(1-\nu_f^2)}{2E_f}\left[4\sigma_0\sigma_c - 3(\sigma_c)^2\right]$$

Critical Buckling Stress:

$$\sigma_c = \frac{Y\pi^2}{12}\left(\frac{E_f}{1-\nu_f^2}\right)\left(\frac{t}{a'}\right)^2$$

Indentation Induced Stress, σ_o

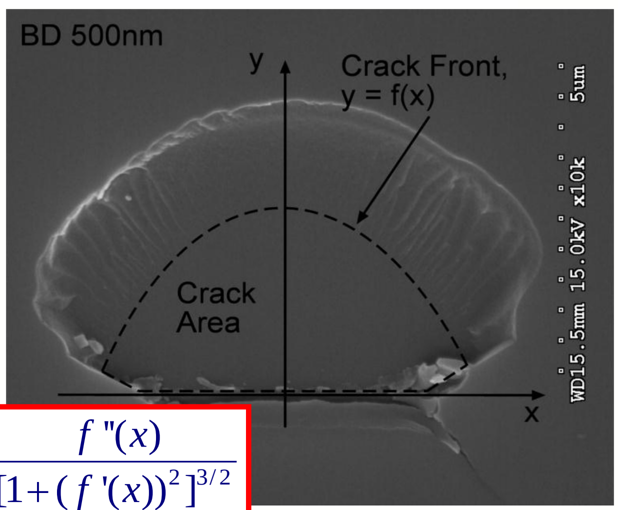


Dimensionless Constant, Y

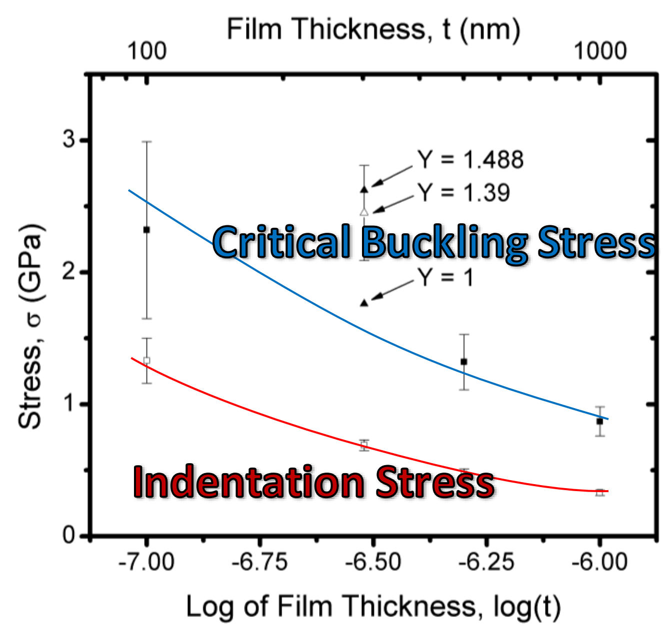
Y = 1 when $\kappa \rightarrow 0$, straight sided buckling

Y = 1.488 when $\kappa \geq 0.5\mu m^{-1}$, circular buckling

Crack front curvature:



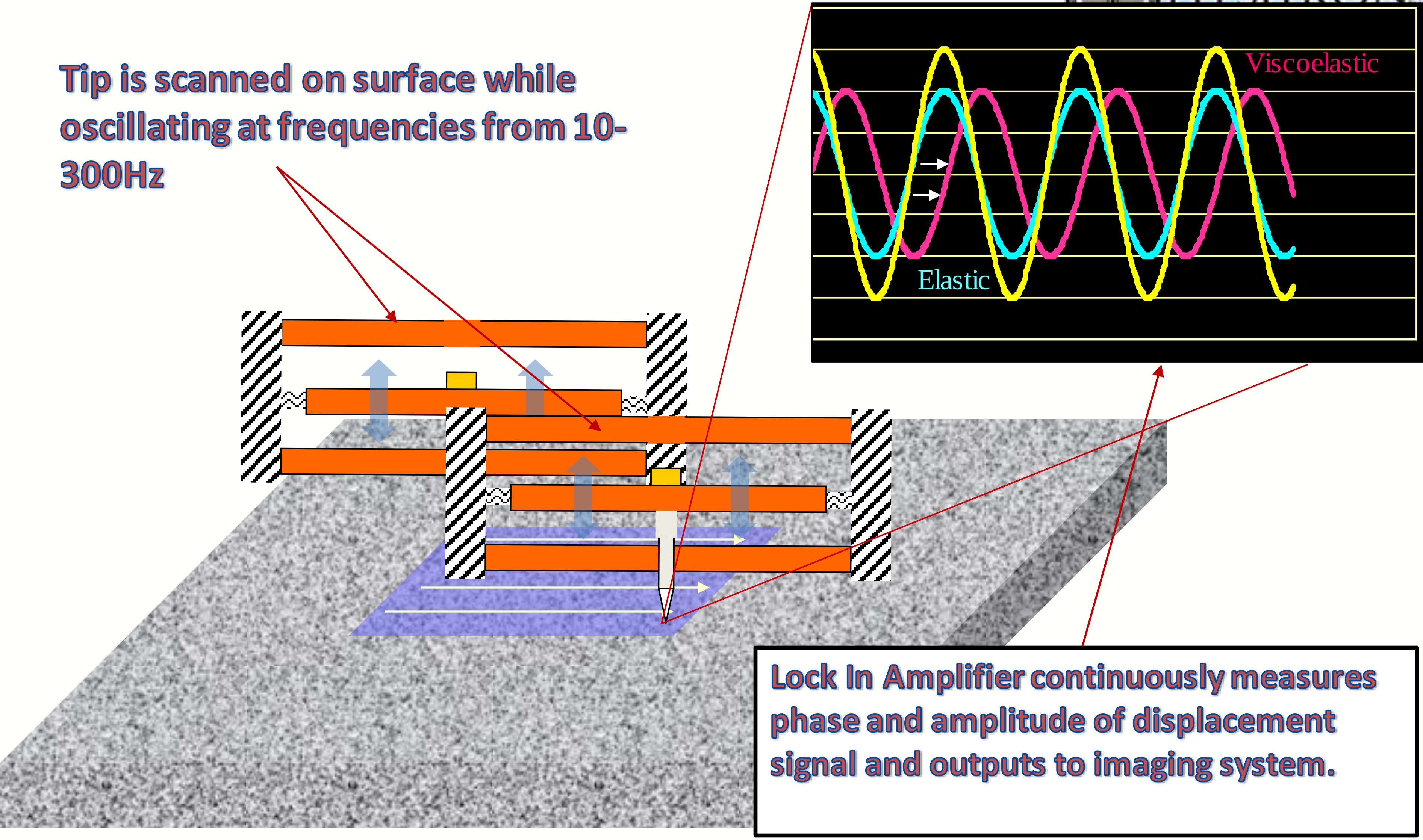
$$\kappa = \frac{f''(x)}{[1+(f'(x))^2]^{3/2}}$$



No Buckling!!

Modulus Mapping

Tip is scanned on surface while oscillating at frequencies from 10-300Hz



Lock In Amplifier continuously measures phase and amplitude of displacement signal and outputs to imaging system.

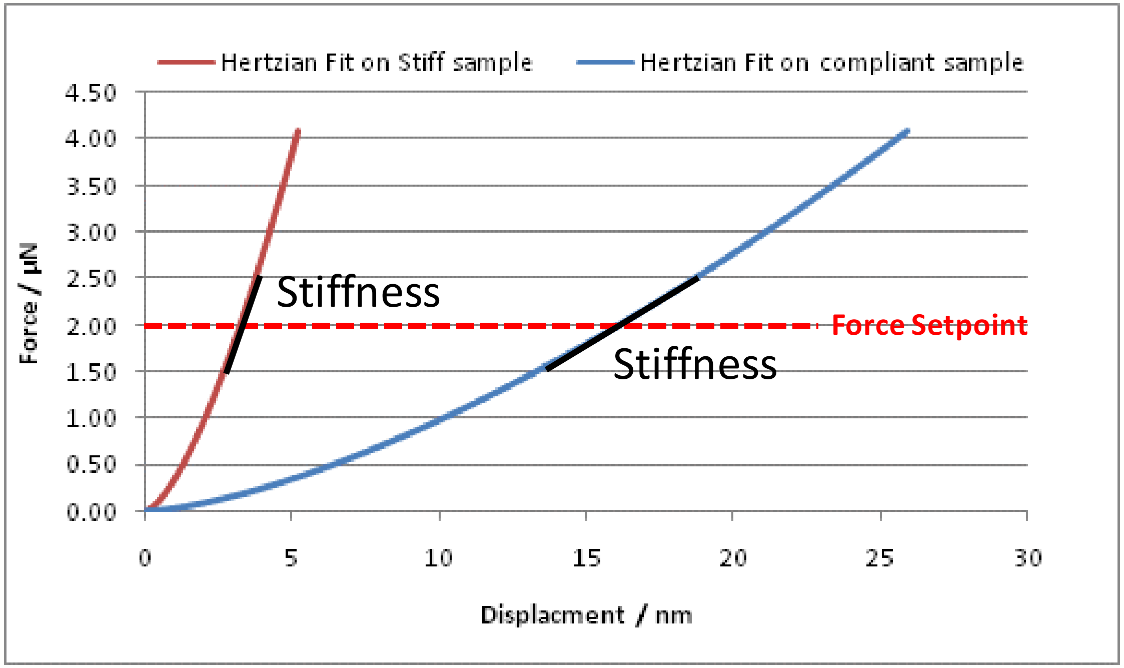
Hertzian Elastic Contact

The stiffness for two samples of different Reduced Modulus leads to a typical stiffness for a given force:

The contact force during imaging is constant; the tip radius is constant
The stiffness therefore relates directly to the Red. Modulus of the sample

The simple case: E´

$$E' = \sqrt{\frac{S'^3}{6 \cdot F_{setpoint} \cdot R}}$$



Indentation Analysis

HYSITRON

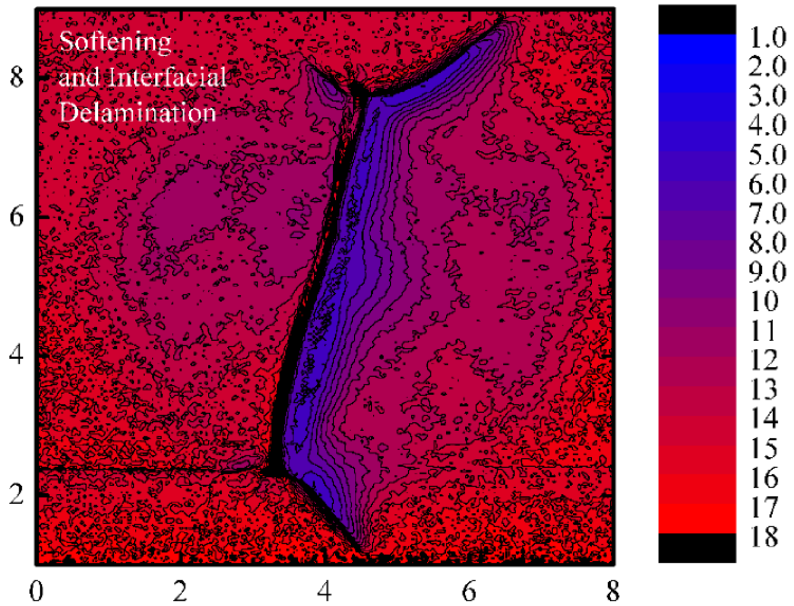
Average Stress

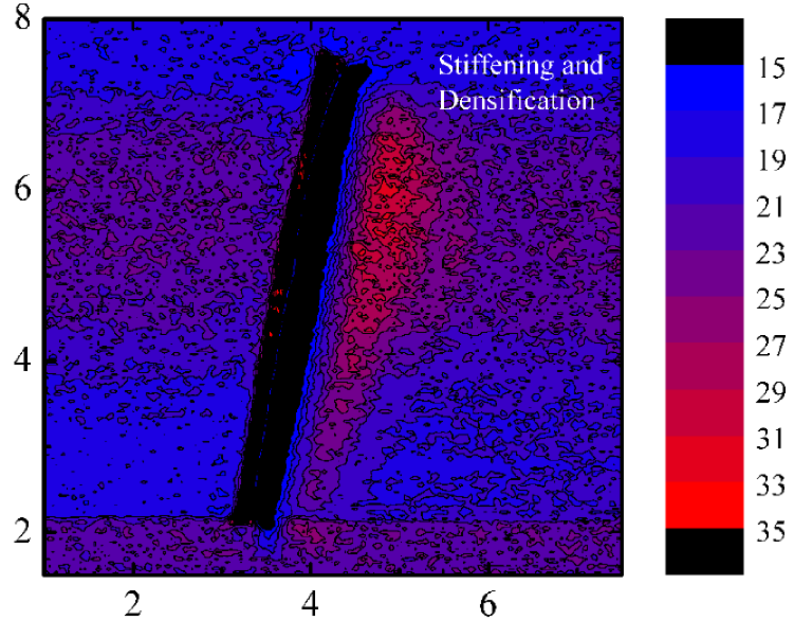
$$\sigma_o = E_f \frac{V_o}{V_c}$$

Interfacial Toughness

$$G_c = \frac{(1-\nu_f^2)t\sigma_o^2}{2E_f} = Z\frac{\sigma_o^2}{E}$$

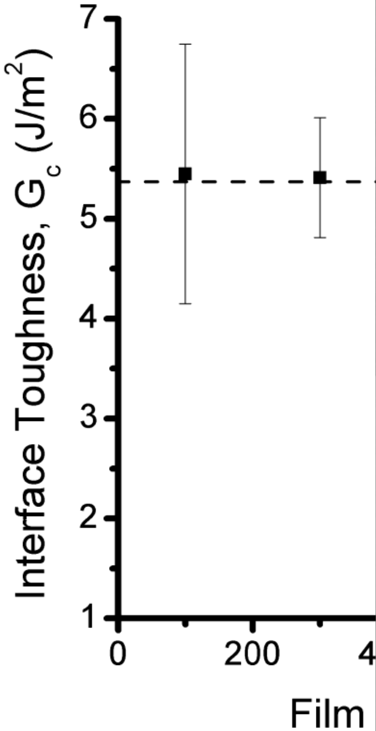
Modulus Mapping ('SEMless')

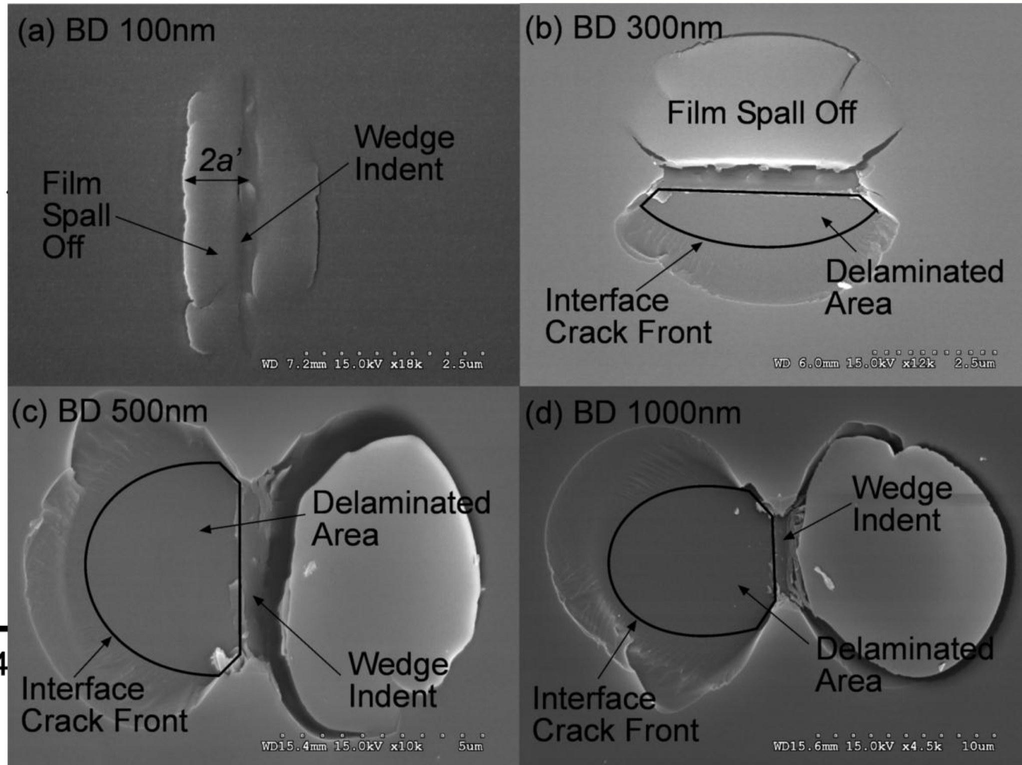




Results

HYSITRON





ess gives
on shape.

wedge
or self-

$/m^2$ for
ip; and
$/m^2$ for
tip

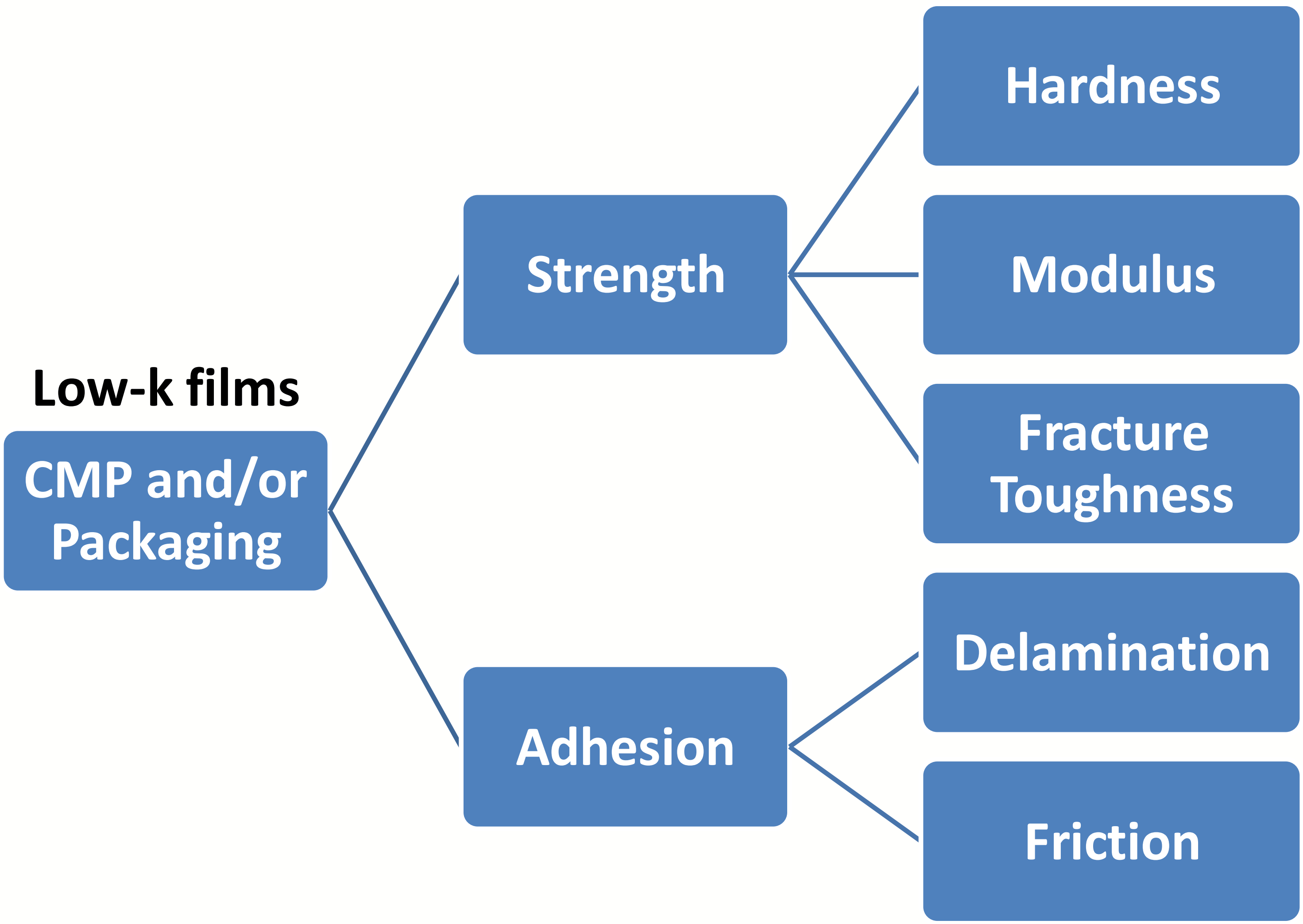
Results are consistent with those reported in literature 4PBT.

MSQ porosity less than 40%, G_c = 1.7 to 2.4 J/m^2

CVD deposited Organosilicate glass, G_c = 4.7 to 7 J/m^2

Guyer et al., JMR (2006); Lin et al., ActaMater (2007)

Summary



Low-k films
CMP and/or Packaging
Strength
Adhesion
Hardness
Modulus
Fracture Toughness
Delamination
Friction

Outlook

nanoIndentation & nanoScratch

Design &FEM Modeling:
Reduced Modulus
Hardness (Yield Strength)
T-dependence
Creep & Relaxation
Adhesion

Process Control:
Indentation:
Porosity
Densifications
Collapsing Pores
Sensitive to Film Thickness
Scratch:
Adhesion
Wedge Indentation:
Adhesion

…=> Lot of information that is easy to measure.

Concluding Remarks

- Nanomechanical measurements provided by Hysitron's Triboindenter can quantitatively evaluate various properties of low-k films.
- Test can be performed with no sample preparation, high throughput and a controlled particle generation
- Mechanical tests are reproducible.
- The measured properties can be related to core functional properties: H&M↔Porosity↔k-value; Critical force during Scratch↔interfacial adhesion; Wedge indentation: Balance of Energy release rate ↔adhesion.
- Nanomechanical tests can be performed on narrow structures – scribe lines or test structures

HYSITRON

THANK YOU!

Acknowledgements:

1. Han Li and Joost Vlassak; Havard University; ULK film samples
2. S.X. Song, Ryan Stromberg; Hysitron Inc.; Measurements and Analysis of Low-K
3. Kong Boon Yeap and Ehrenfried Zschech; Fraunhofer IZFP-Dresden; Wedge indentation