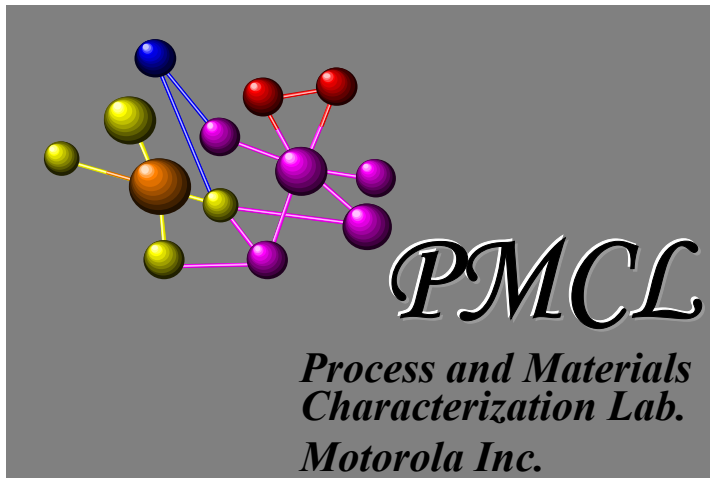


# Critical Issues for Interconnects: Mechanical Strength and Adhesion based on Nanoindentation

**Indira Adhietty**

**Joe Vella**

**Alex Volinsky**

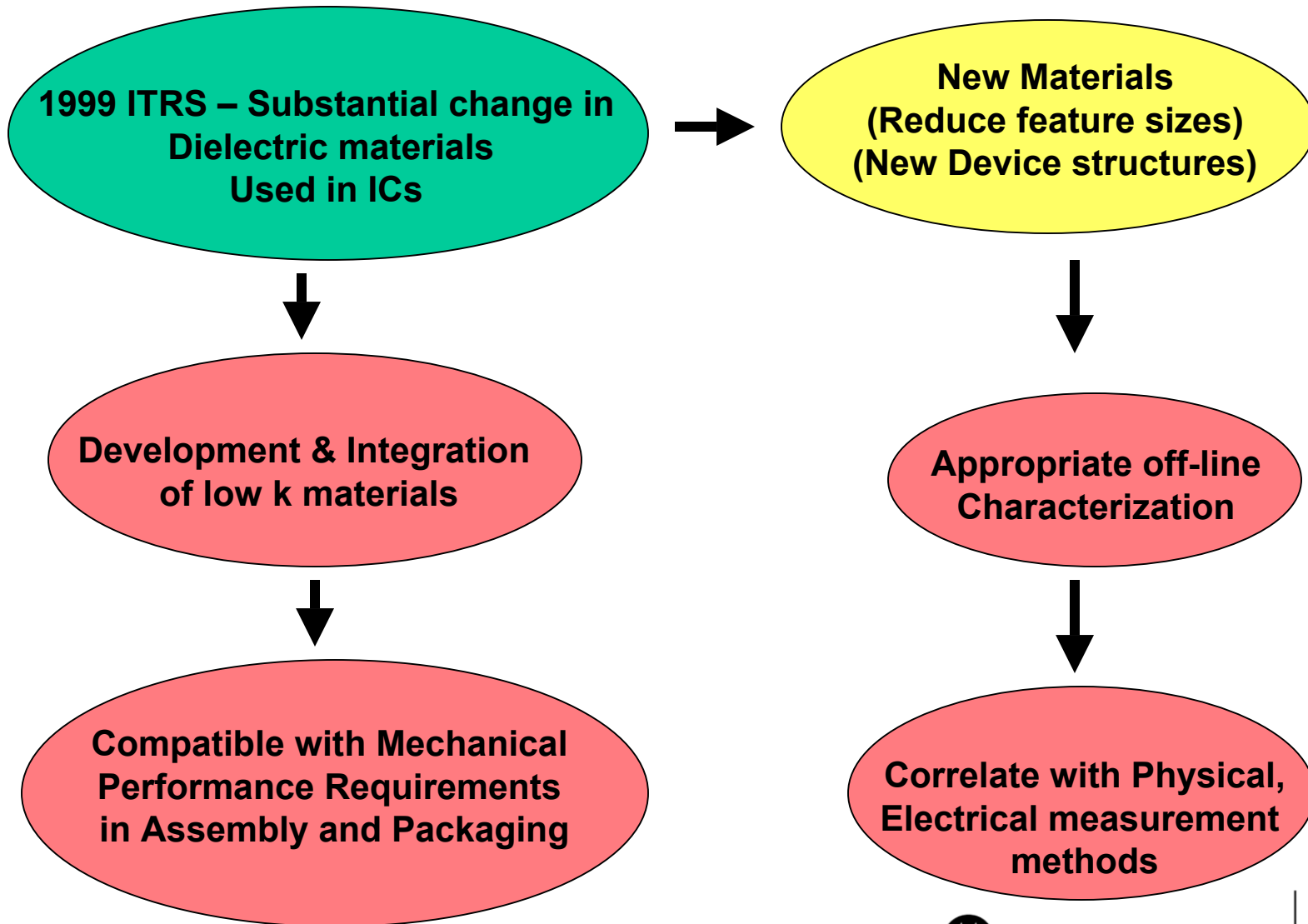


**Acknowledgements: Bob Carpenter,  
Wentao Qin, Ginger Edwards, Bruce Xie,  
Anotoli Korkin, PMCL, MOS13 and APRDL  
low k Teams**

# Outline

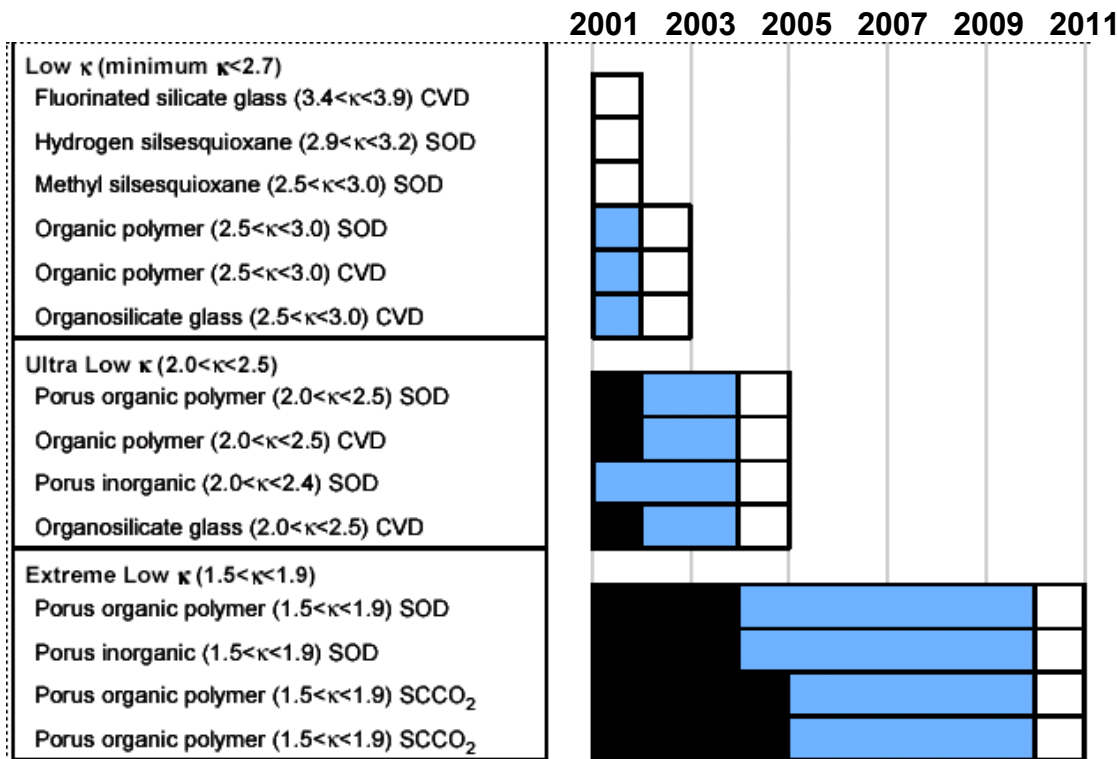
- I. Why nano-Indentation ?**
- II. Overview of OSG Structure and Properties**
- III. NanoIndentation Metrology of Low-k Materials**
  - A. Utility of Hardness and Modulus**
  - B. Mechanical Properties Give Insight to Porosity  
-Percolation Theory**
- IV. Adhesion and Fracture Toughness Measurements**
  - A. Description of Methodology and Limitations**
  - B. NanoIndentation induced fracture calculations**
- V. Conclusions**

# Why Nano-Indentation ?



# Development & Integration of low k materials

## Dielectric Potential Solutions



## Metrology Challenges

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

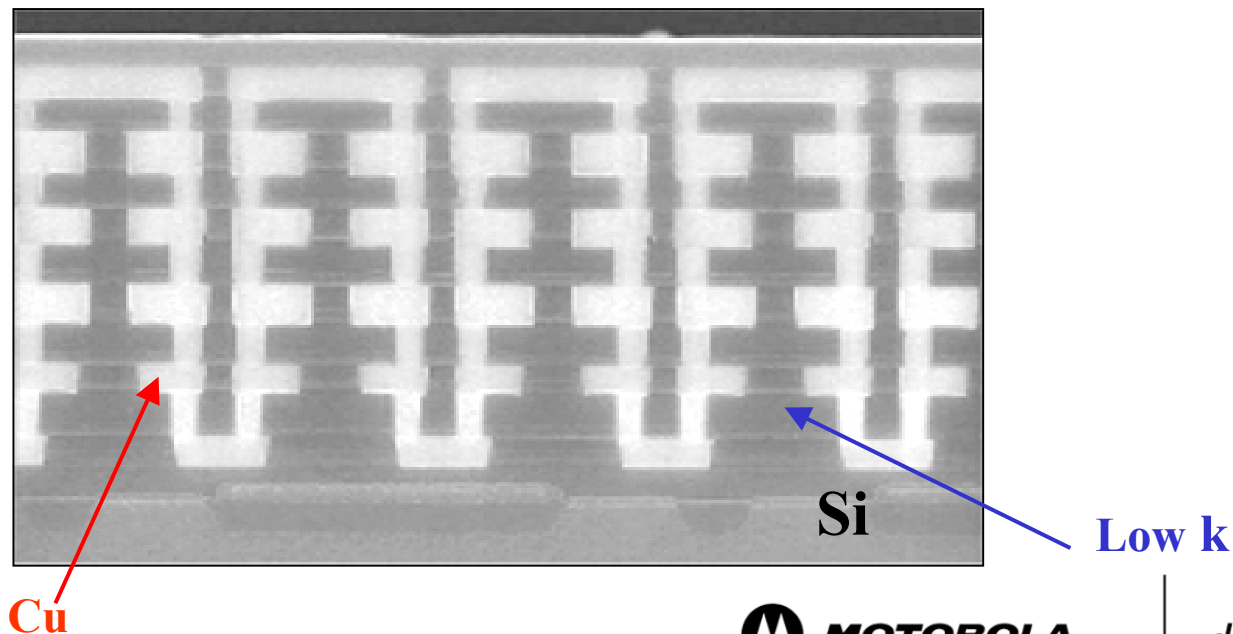


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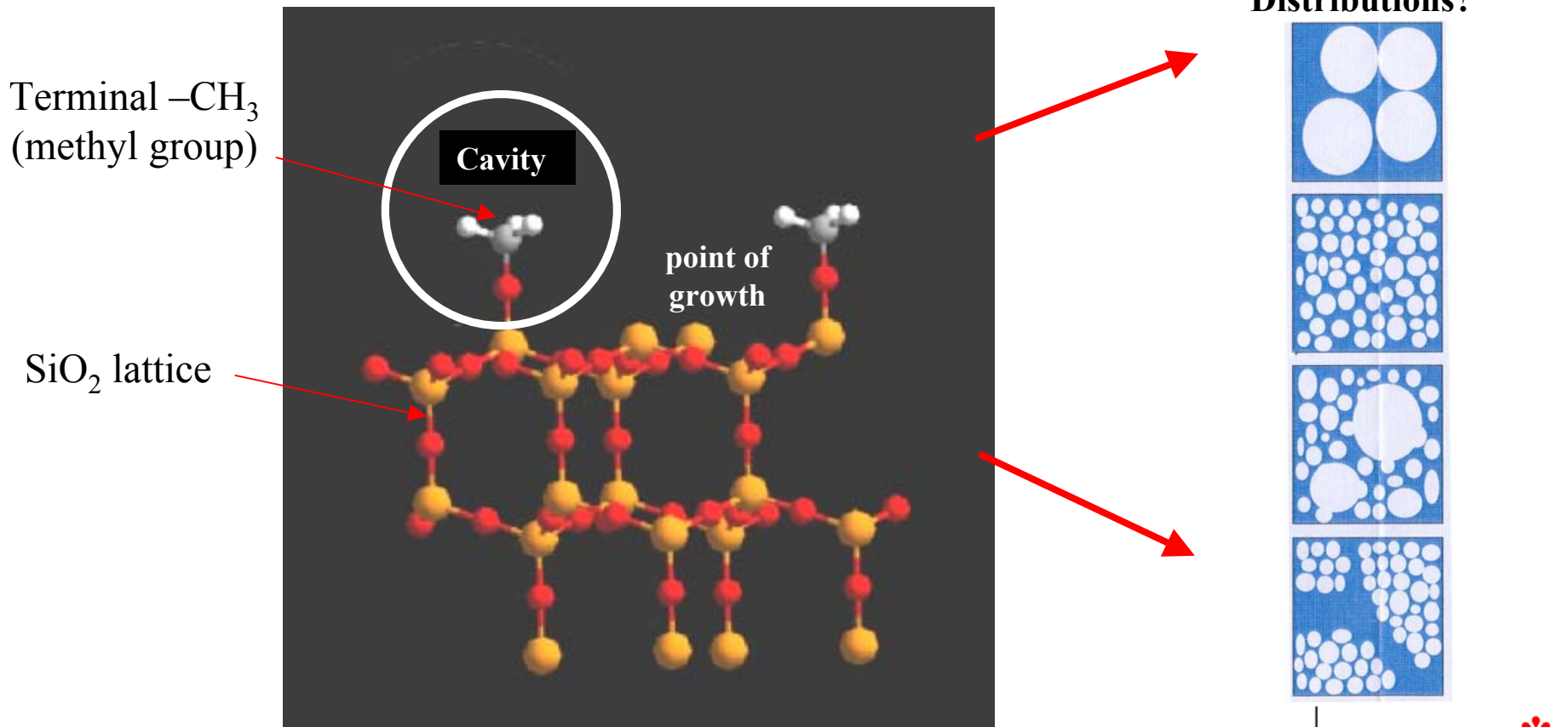
# Organo-silicate Glass (OSG) Low-k Integration at Motorola

- Dielectric properties of fully dense silica ( $k \sim 4.1$ ) can be extended by
  - reducing the density
  - increasing the porosity,
  - utilizing the dielectric constant of free space ( $k=1$ ) and organic inclusions.
- Electrical properties provide a smooth transition from traditional silica to next generation IC's
- Mechanical reliability properties require extensive study in thin film form. Often these films cannot be grown to thicknesses greater than  $3\mu\text{m}$ .
- Nanoindentation offers unique capabilities in classifying these types of materials.

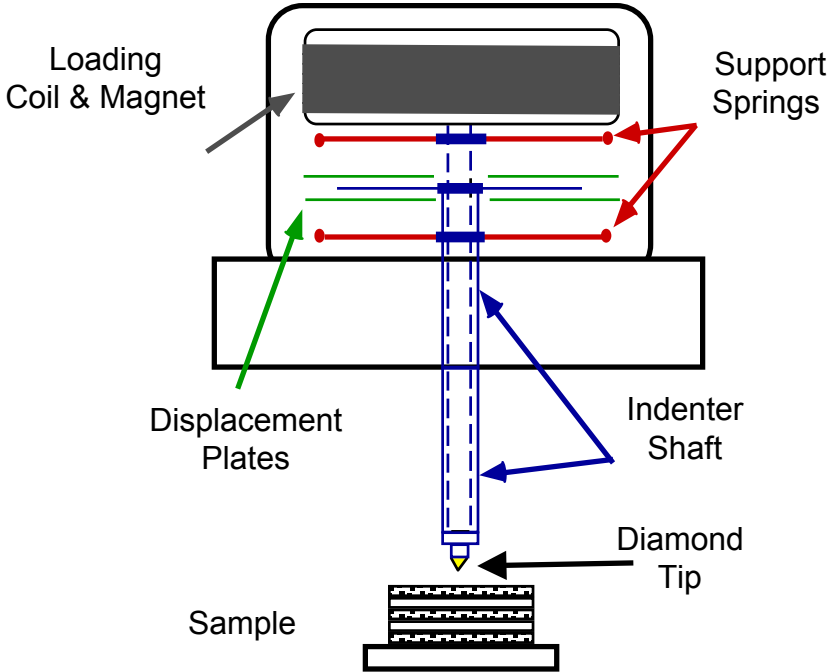


# OSG Pore Distribution

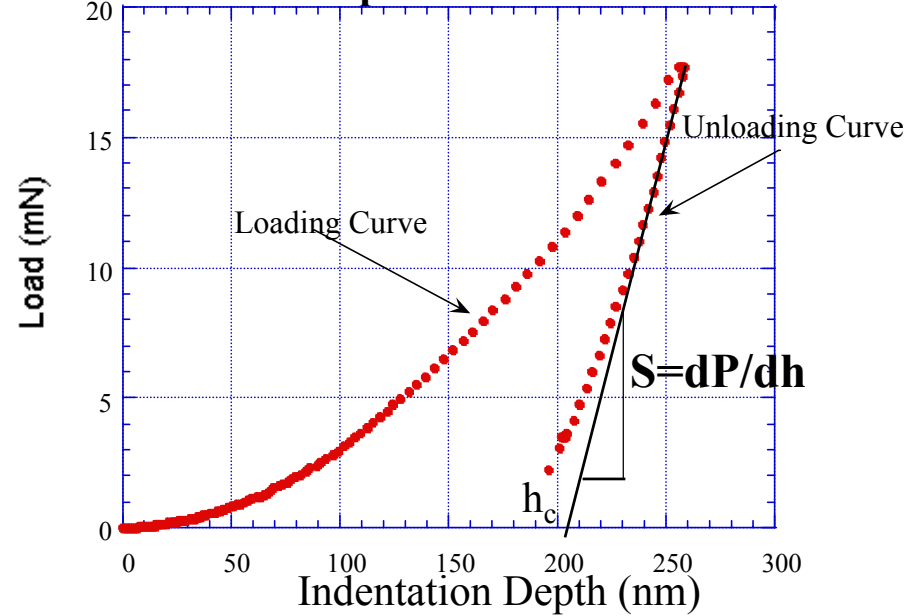
- Pore is created by the introduction of CH<sub>x</sub> group that retards Si-O bonding network.
- Small pore size (1nm) therefore is largely occupied by the dangling methyl group



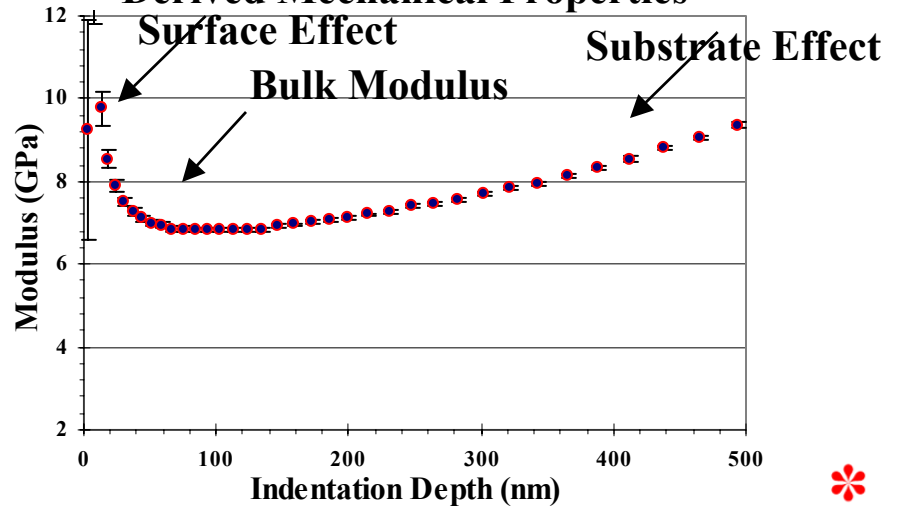
# Mechanical Property Measurements



## Mechanical Response to Indentation



## Derived Mechanical Properties



## Load Measurement

Current is applied through a coil in a magnetic field generating a force

## Displacement Measurement

Movement of the Capacitor plate

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

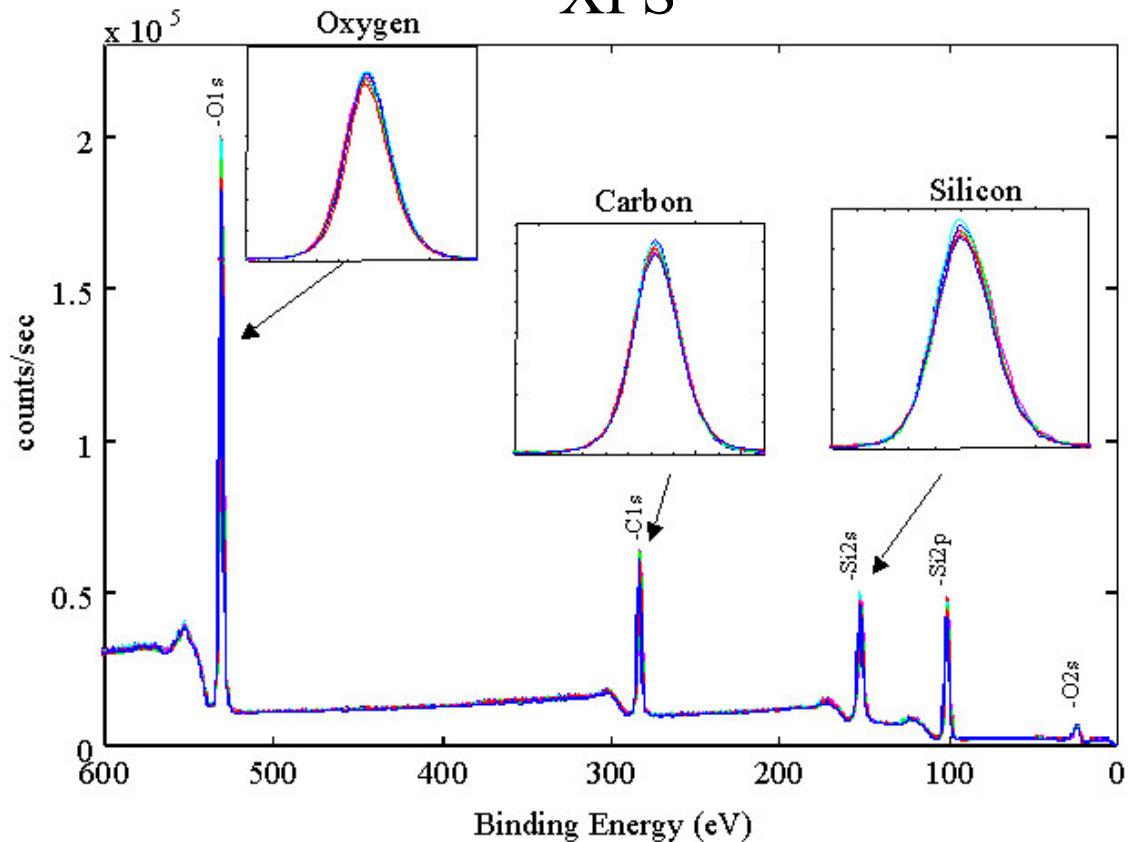
$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A}}$$



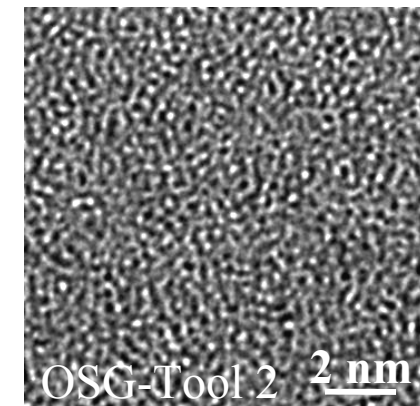
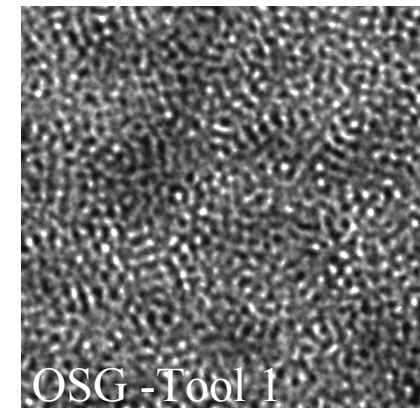
# Low-K Film Metrology – New Tool Installation

- X-Ray Photoelectron Spectroscopy shows that 7 OSG of varying process parameters are ostensibly chemically identical.
- TEM shows amorphous and possibly porous structure however no discernible discrepancies.

## XPS



## TEM

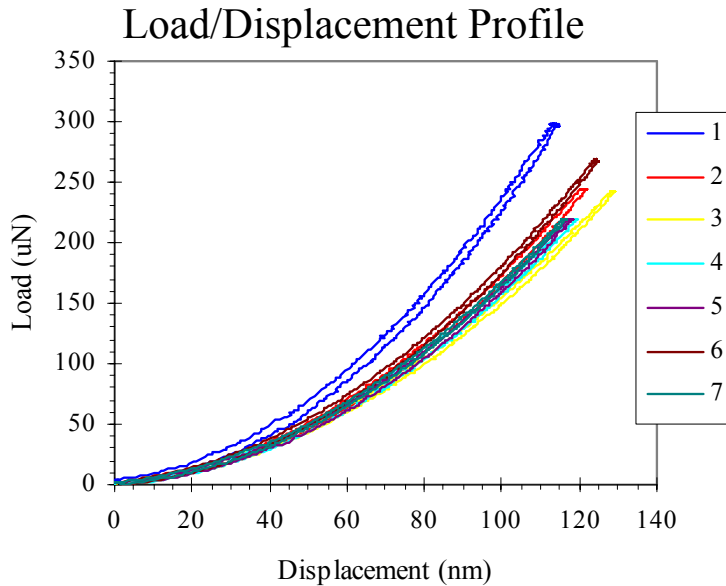


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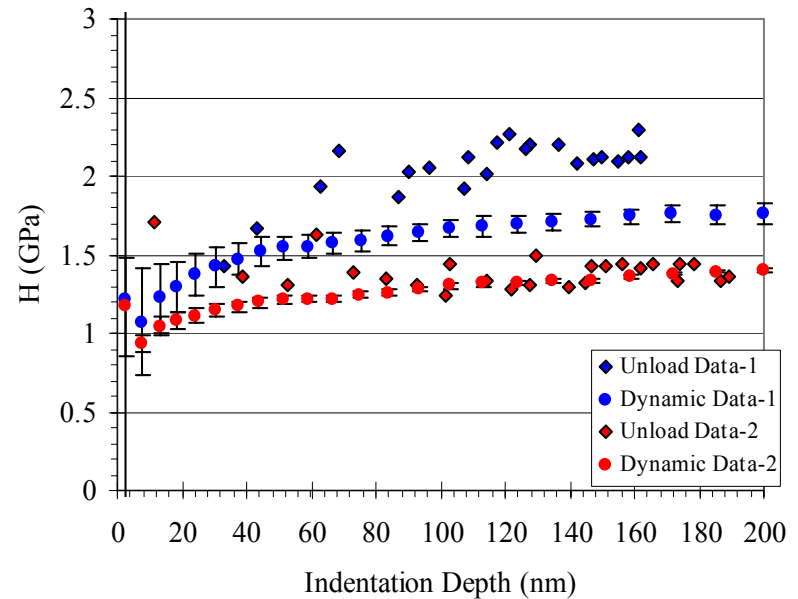
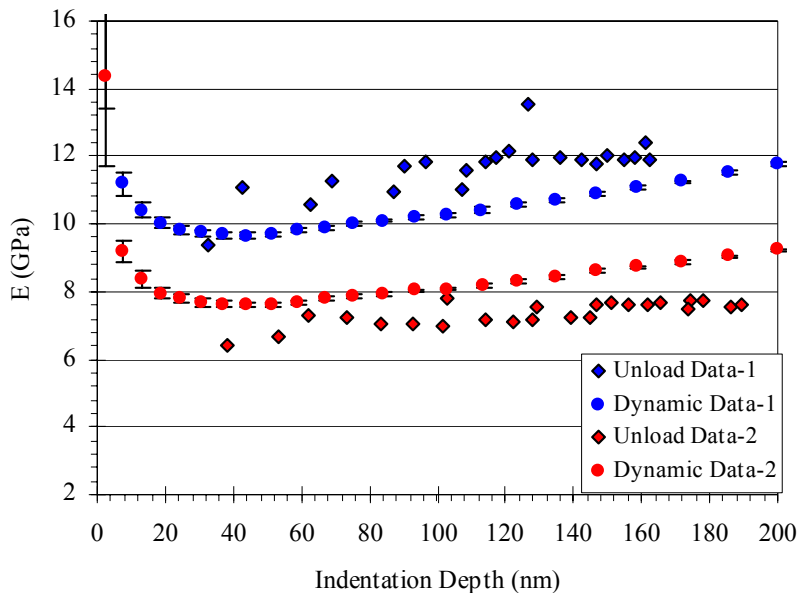
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# NanoIndentation results of OSG series



- Significant mechanical properties difference observed among the two tools
- Load/displacement profile shows entirely elastic contact to depths of 120nm



# Does Percolation Theory Hold Up on the Nanoscale?

## Percolation Theory

$$\frac{E^*}{E_0} = \left(1 - \frac{p^*}{p_c}\right)^f$$

FILM 1	Open Pore Porosity (%)	Closed Pore Porosity (%)
Modulus Data Calc	59.7	76.8
Hardness Data Calc	58.9	70.2

## Gibson and Ashby Predictions

### Open Cell Pores

Modulus

$$\frac{E^*}{E_0} = \left(\frac{\rho^*}{\rho_0}\right)^2$$

Hardness

$$\frac{H^*}{H_0} = .23 \left(\frac{\rho^*}{\rho_0}\right)^{3/2} \left(1 + \left(\frac{\rho^*}{\rho_0}\right)^{1/2}\right)$$

Fracture Toughness

$$\frac{K_{Ic}^*}{K_{Ic0}} = .65 \left(\frac{\rho^*}{\rho_0}\right)^{3/2}$$

### Closed Cell Pores

$$\frac{E^*}{E_0} = \phi^2 \left(\frac{\rho^*}{\rho_0}\right)^2 + (1 - \phi) \left(\frac{\rho^*}{\rho_0}\right)$$

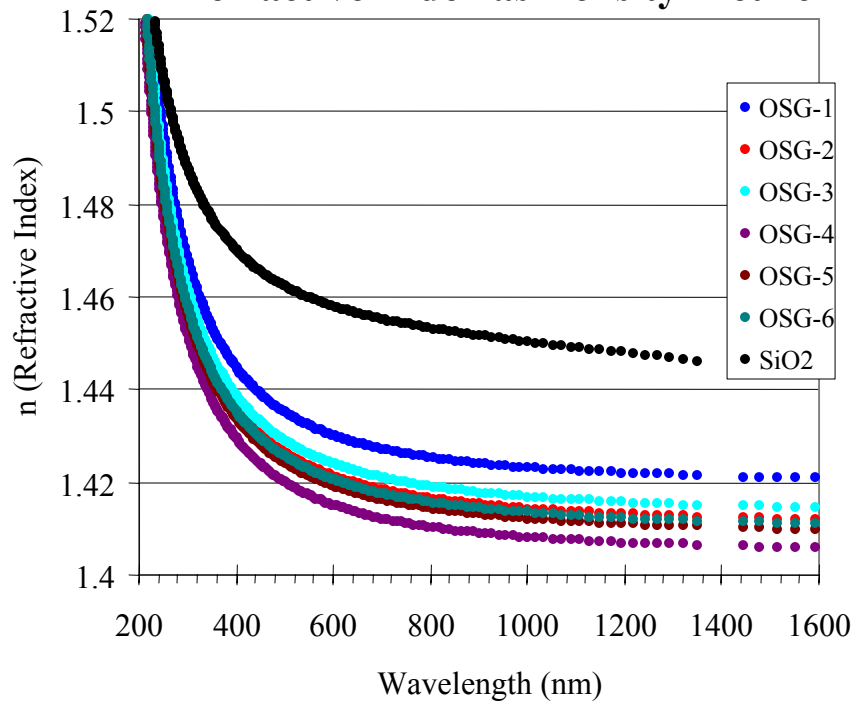
$$\frac{H^*}{H_0} = .3 \left(\phi \frac{\rho^*}{\rho_0}\right)^{3/2} + .4(1 - \phi) \left(\frac{\rho^*}{\rho_0}\right)$$

$$\frac{K_{Ic}^*}{K_{Ic0}} = \left(\frac{\rho^*}{\rho_0}\right)^{3/2}$$

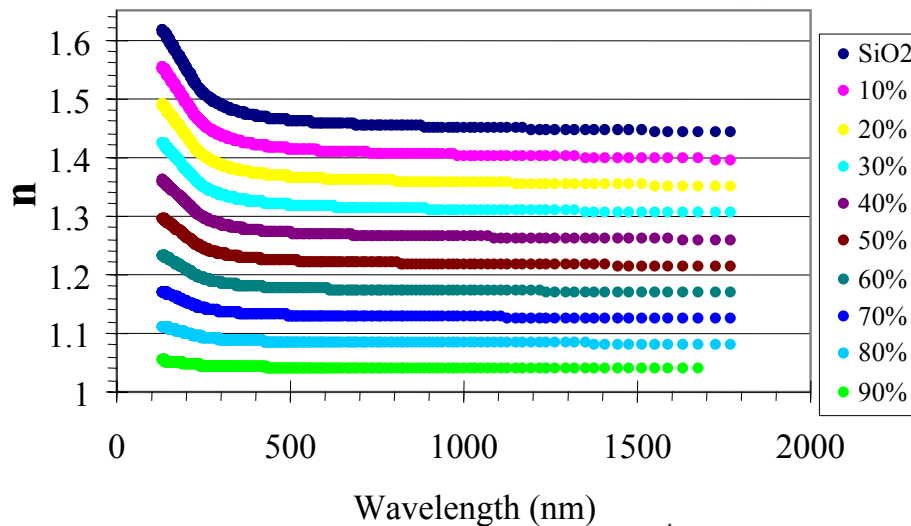
E. O. Shaffer II, K. E. Howard, M. E. Mills, P. H. Townshend, *Mat Res. Soc. Symp. Proc.*, Vol. **612**, (2000)  
 L. Gibson and M. Ashby, *Cellular Solids*, 2<sup>nd</sup> Ed. (1997).

# Spectroscopic Ellipsometry Shows Difference in Density

## Refractive Index as Density Metric

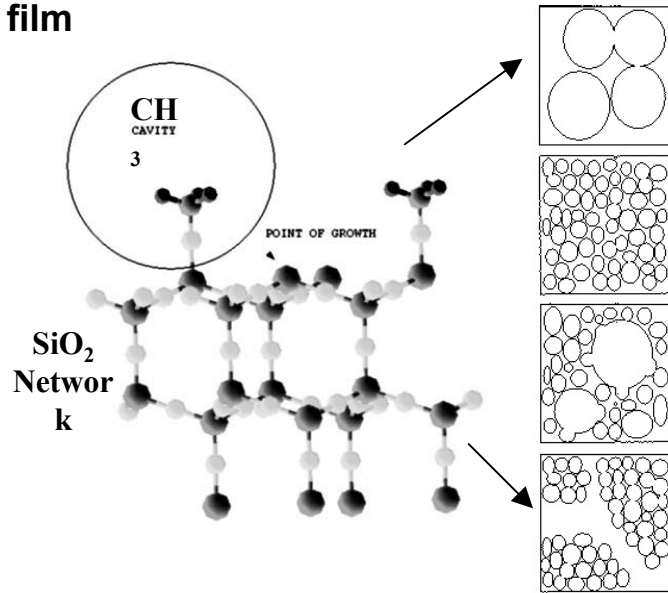


## Porous Silica Refractive Index (n)

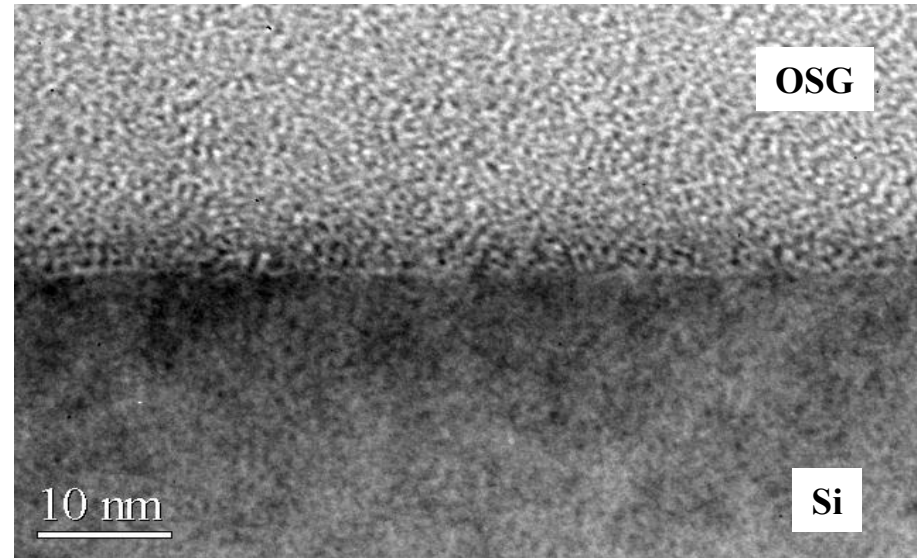


# Evaluation of Porosity

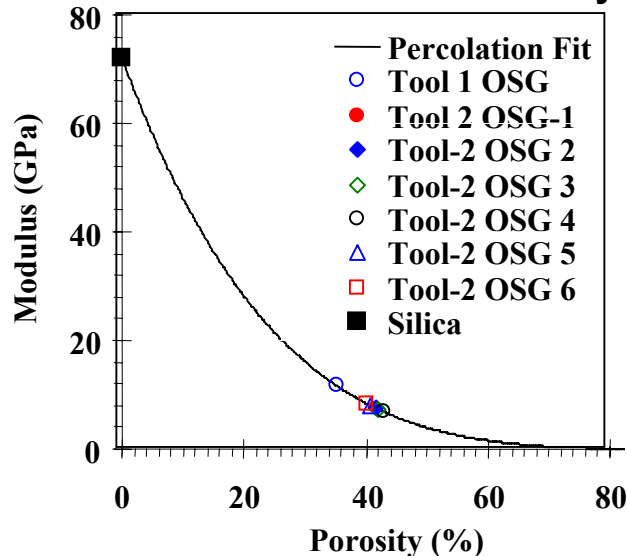
## Porosity formation process in a low- k dielectric thin film



## TEM cross section of a low- k dielectric film



## Indirect measurement of Porosity through Modulus



	Modulus (GPa)	Percolation Porosity (%)
Tool 1 OSG	11.7	35.1
Tool 2 OSG-1	7.60	41.4
Tool 2 OSG-2	7.49	41.6
Tool 2 OSG-3	7.24	42.1
Tool 2 OSG-4	6.90	42.8
Tool 2 OSG-5	7.92	40.9
Tool 2 OSG-6	8.32	40.1
Silica	72.00	0.0

- Pores are created by methyl inclusion in a molecular scale.
- NI is able to determine the porous and dielectric nature of the films.

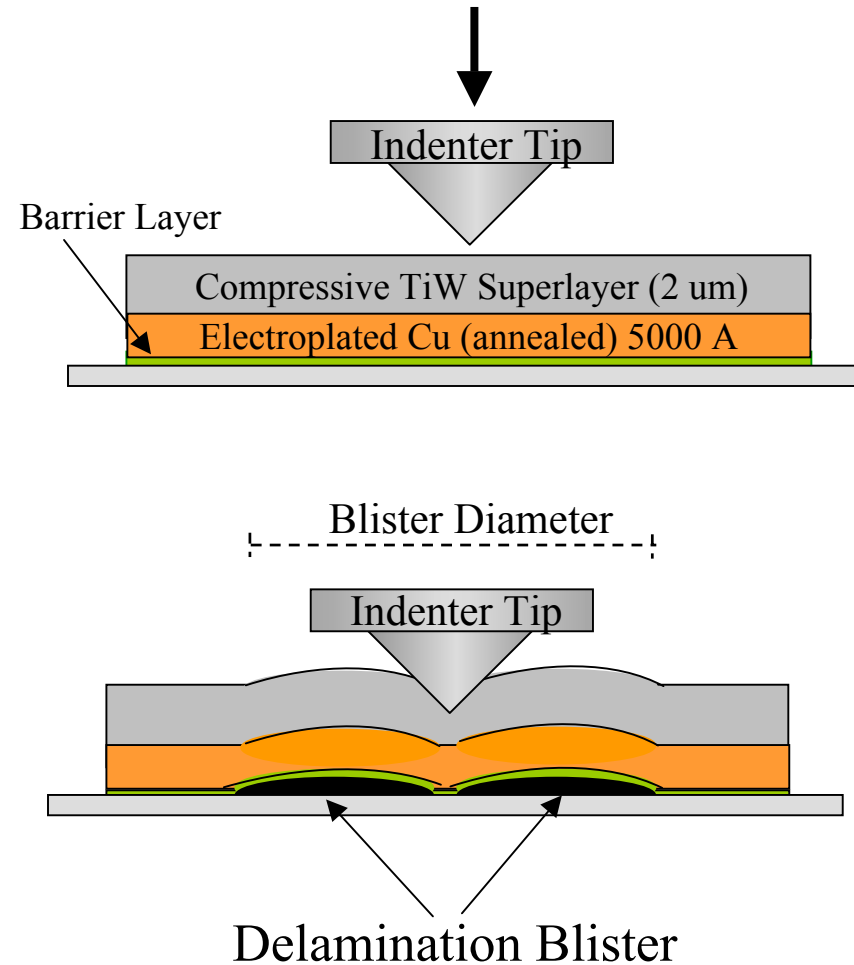


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# Adhesion Measurements

- Thin film delamination is induced by depositing a highly compressive superlayer capable of storing elastic energy
- Interfacial fracture is initiated by Nanoindentation at loads (25-700mN) and further driven by superlayer
- Delamination blister size is used to calculate Practical Work of Adhesion ( $J/m^2$ ) (Smaller blister = Better adhesion)
- Material and residual stress properties of superlayer and underlying film must be known for calculation

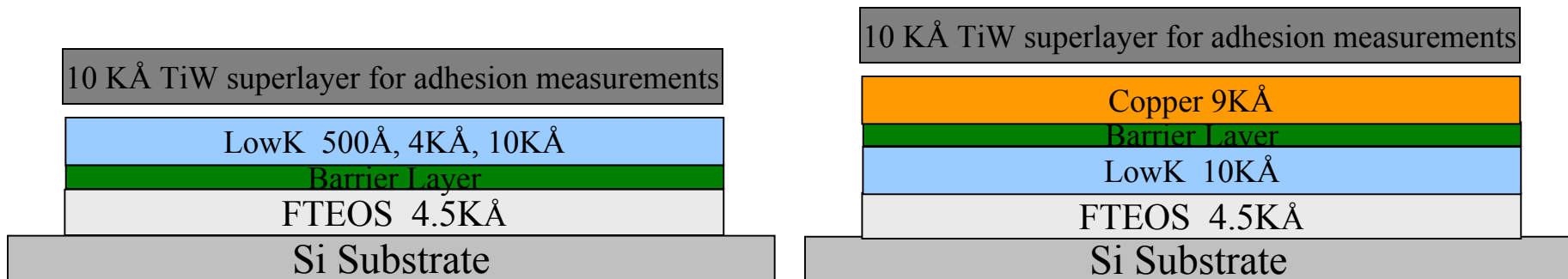


Nanoindent Adhesion Test  
(Delamination blister)

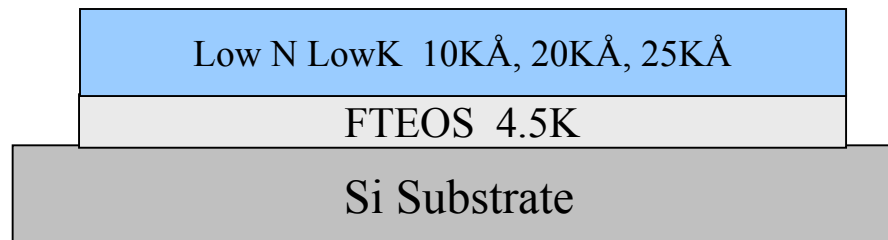
# Low-k Test Structures

## CVD Organic Glass Low-k

### Adhesion



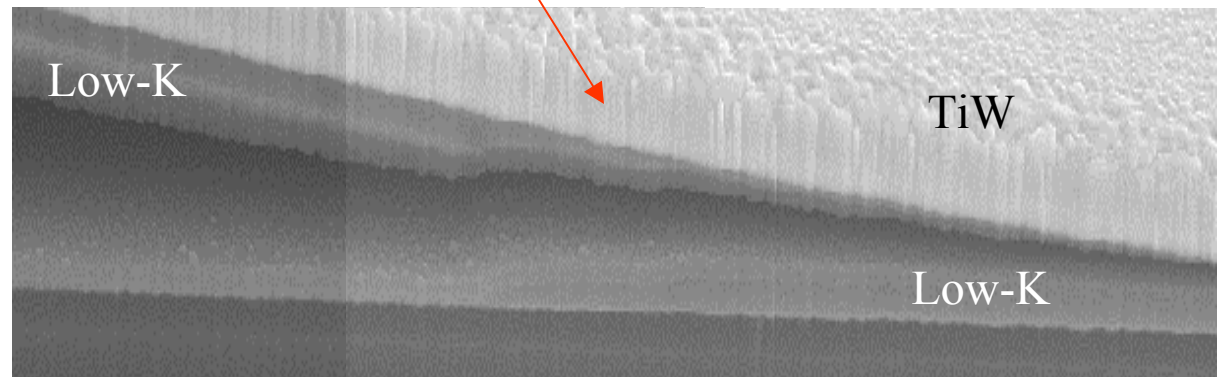
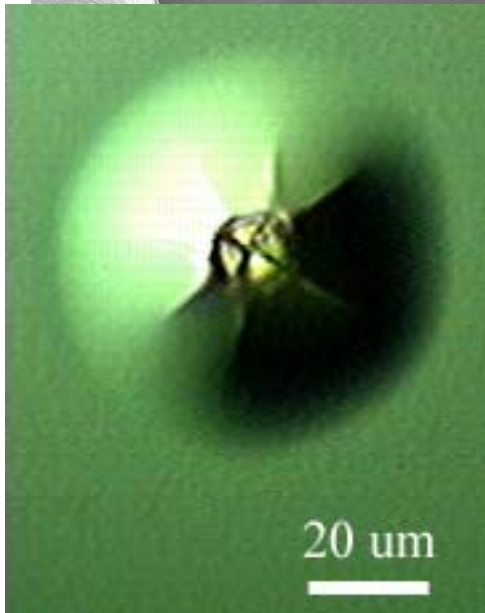
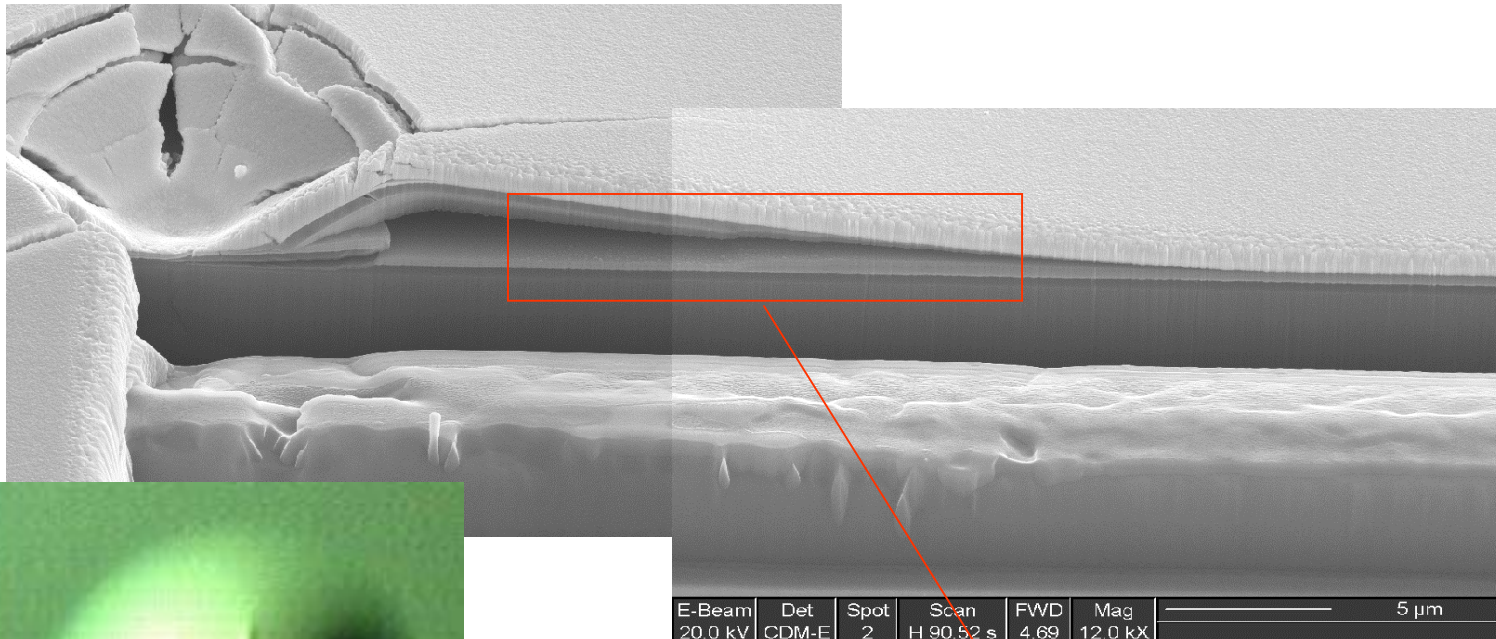
### Fracture toughness



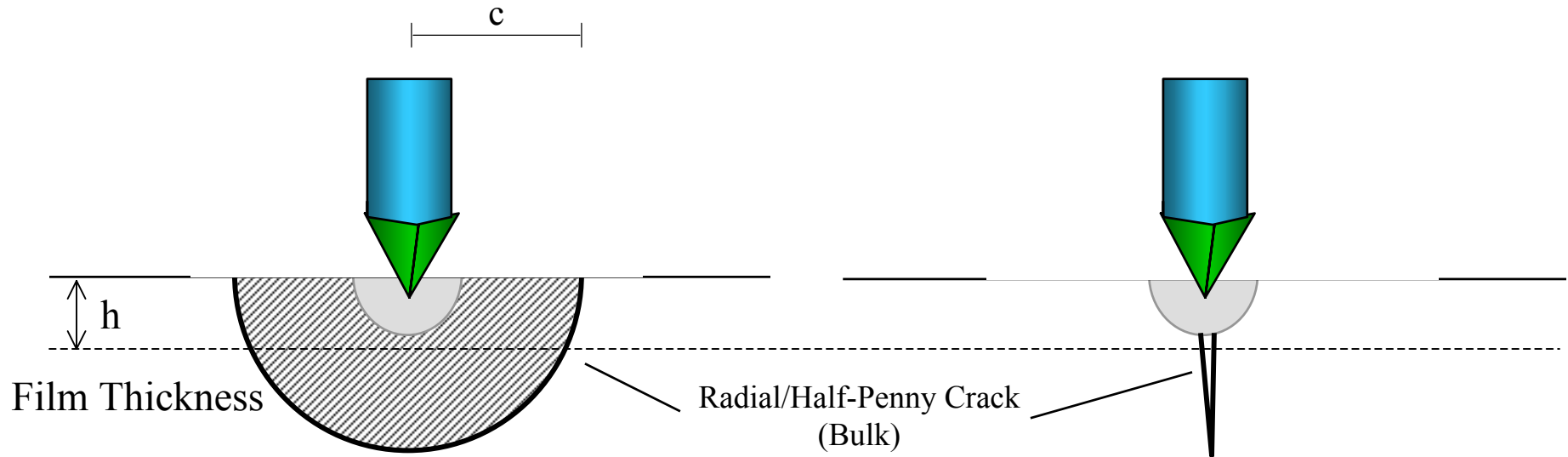
$E \sim 10 \text{ GPa}$

$H \sim 1.5 \text{ GPa}$

# 4 kÅ Low-k



# Fracture Toughness Measurement



$$K_{c \text{ Bulk}} = \alpha \left( \frac{E}{H} \right)^{1/2} \left( \frac{P_{\max}}{c^{3/2}} \right) \quad 40\% \text{ Accuracy}$$

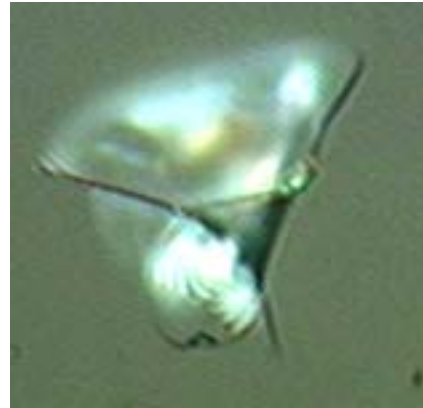
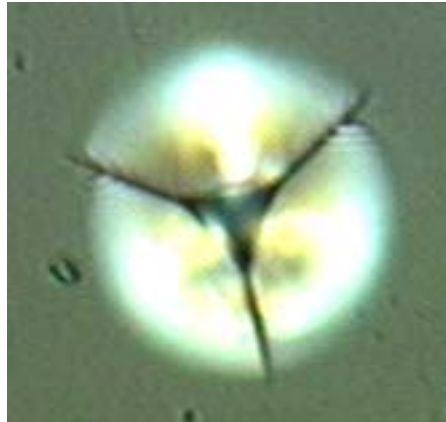
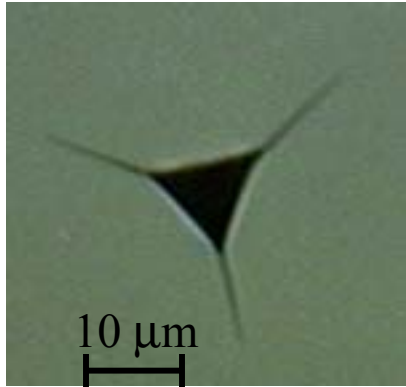
$$K_{c \text{ Thin Film}}(h, H, E) = ?$$

Lawn, B.R., Evans, A.G., and Marshall, D.B. (1980) 'Elastic/Plastic Indentation Damage in Ceramics: The Median/Radial Crack System', *J. Amer. Cer. Soc.* 63, 574-581.

Pharr, G.M., Harding, D.S., and Oliver, W.C., Mechanical Properties and Deformation Behavior of Materials Having Ultra-Fine Microstructures, 449-461. M Nastasi et al. (eds.), Kluwer, Netherlands, 1993.



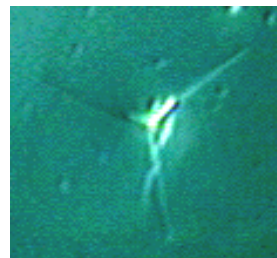
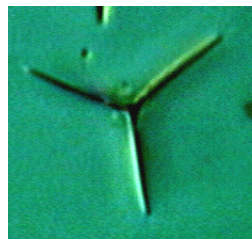
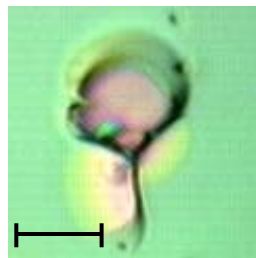
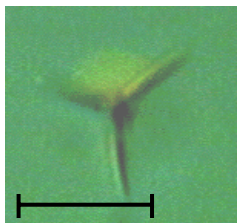
# Cube Corner Indents



Silica:  
 $K_c \sim 0.5 \text{ MPa}\cdot\text{m}^{1/2}$   
vs. literature:  
 $K_c \sim 0.75 \text{ MPa}\cdot\text{m}^{1/2}$

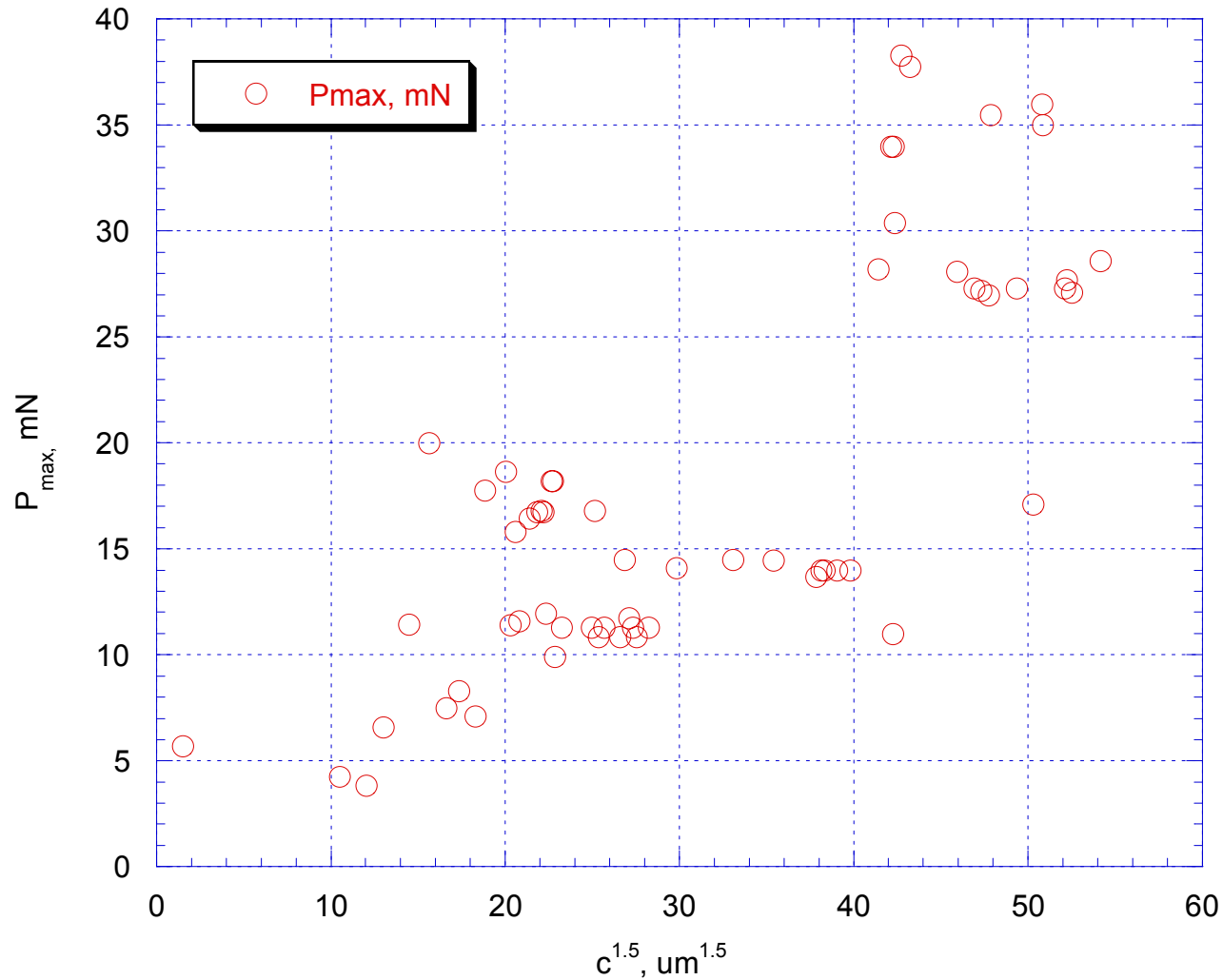
1 um LowK

2 um LowK

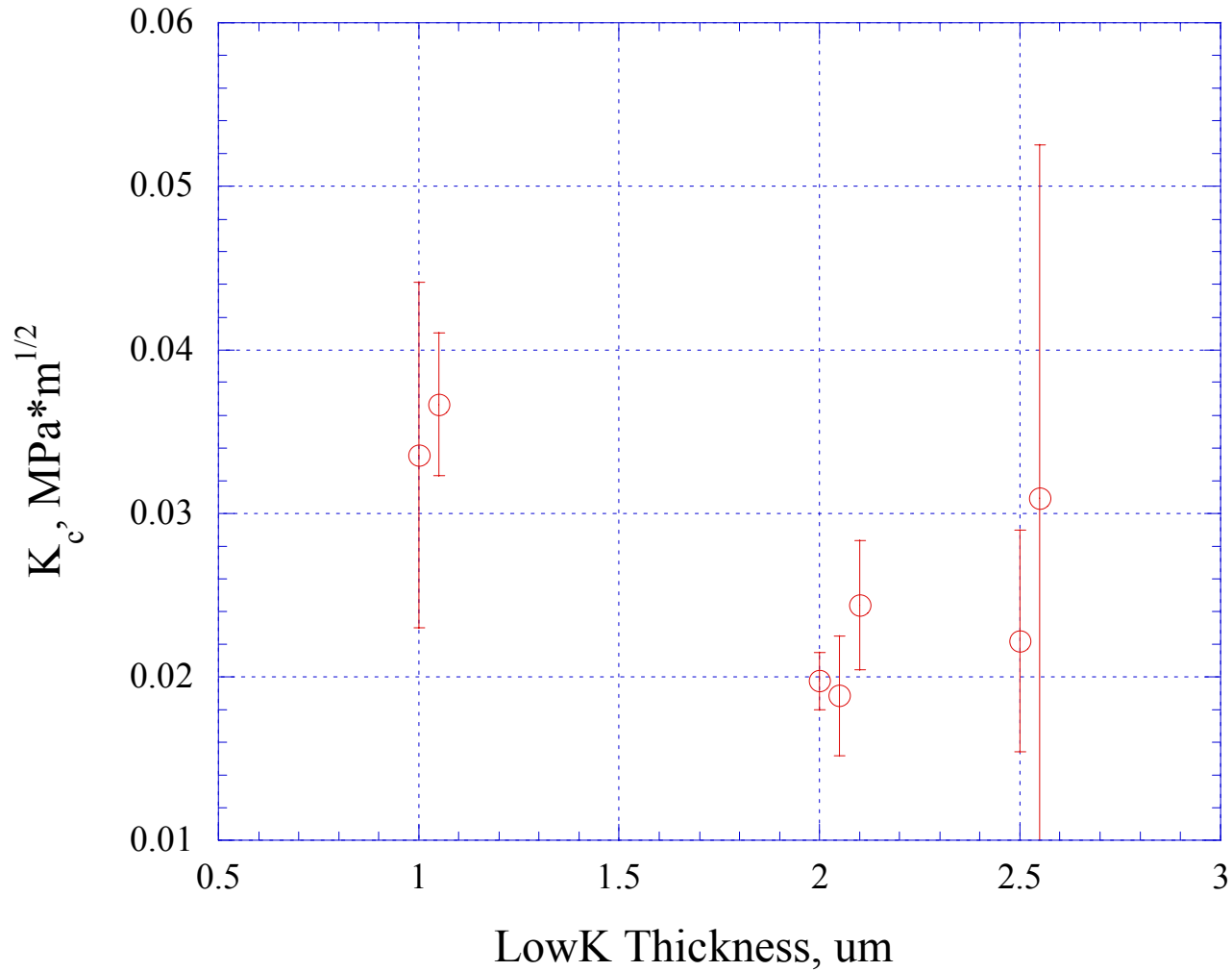


$K_c \sim 0.01\text{-}0.05 \text{ MPa}\cdot\text{m}^{1/2}$

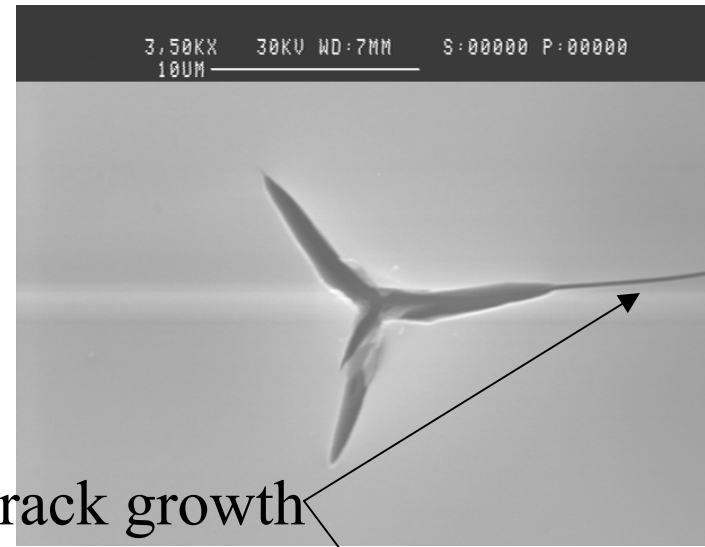
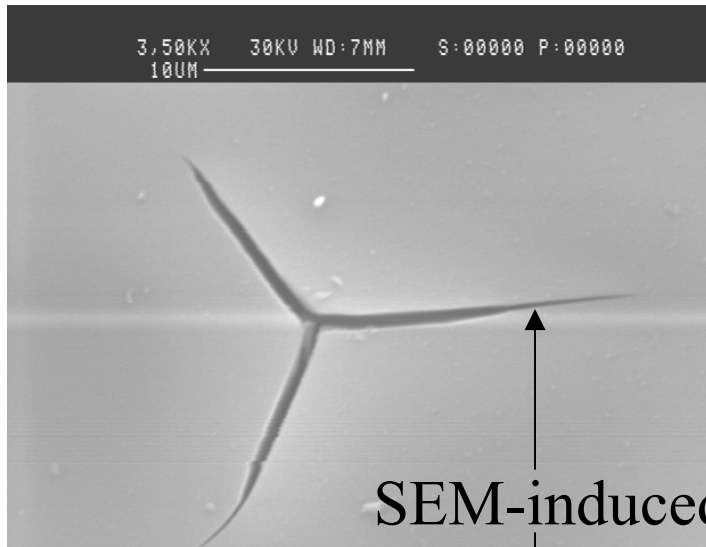
# Linear relationship for $P_{\max}$ vs. $c^{3/2}$



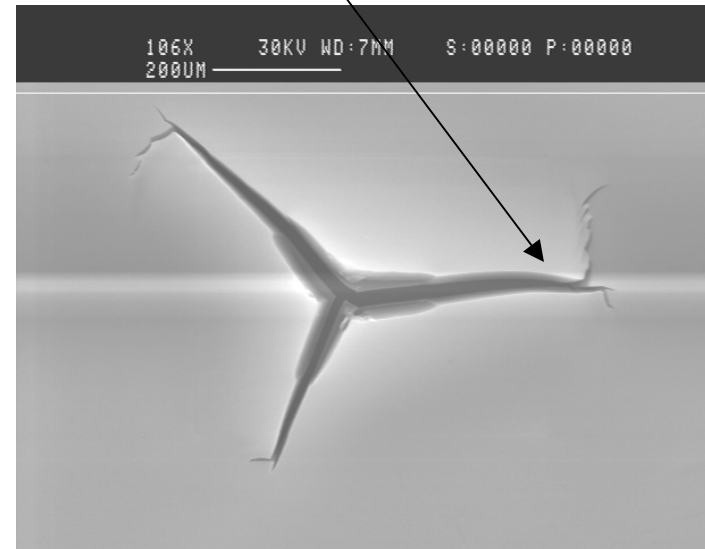
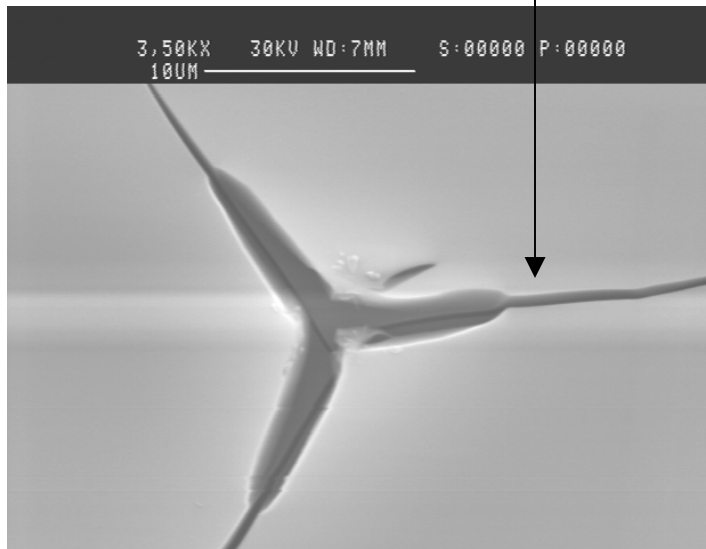
# Low-k Fracture Toughness



# Si/20KÅ LowK Cracking in SEM



SEM-induced crack growth



# Summary

**Nano-Indentation was able to successfully address some of the metrology challenges in development and integration of low k materials**

**Technique is also capable of identifying adhesion issues and provide selection for optimum process conditions**

**Currently investigating the compatibility of low k materials during assembly and packaging using Nano-Indentation**