

Low modulus standards for nanoindentation and their application to testing low-k materials

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Select requirements of low modulus standards

Standards in the range of 1 GPa to 10 GPa

Bulk, homogenous solid

Stability over time in a normal environment (*on the order of years*)

Ability to characterize the elastic modulus through alternate means

Minimal room temperature time dependence



Two common polymers that fit this set of requirements

Polycarbonate

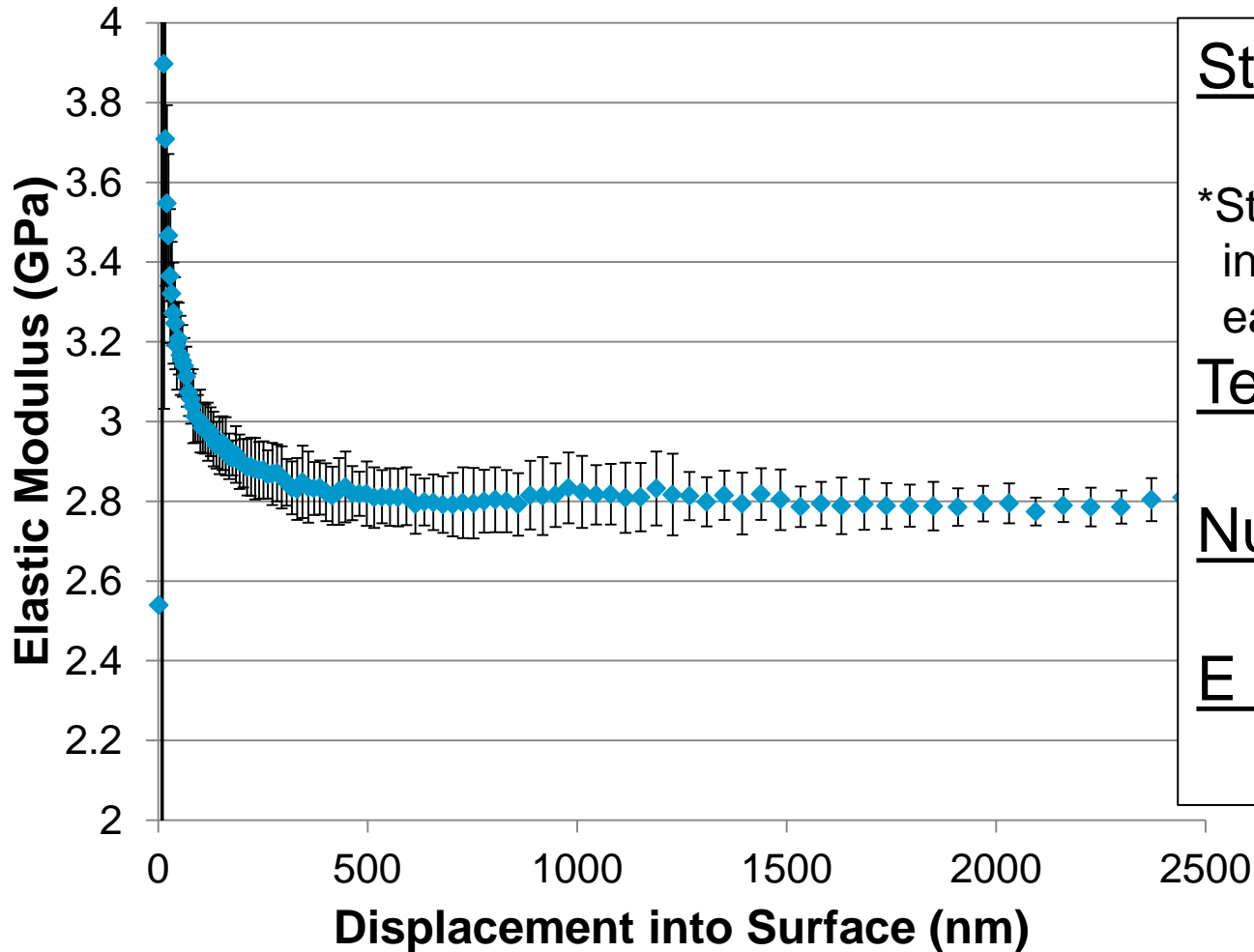
- Amorphous, $E = 2.3 - 2.4$ GPa, $E/Y_s = 42$, $\nu = 0.37$, $t_g = 150^\circ\text{C}$

PMMA

- Amorphous, $E = 2.4 - 3.3$ GPa, $E/Y_s = 30$, $\nu = 0.35 - 0.4$, $t_g = 80^\circ\text{C}$

The much higher t_g of PC makes it more desirable for operating conditions over a normal range of room temperatures.

Nanoindentation Results on Polycarbonate



Strain Rate Range*

0.05/s to 2.0/s

*Strain rate was increased by 30% for each successive test.

Temperature

26°C

Number of Tests

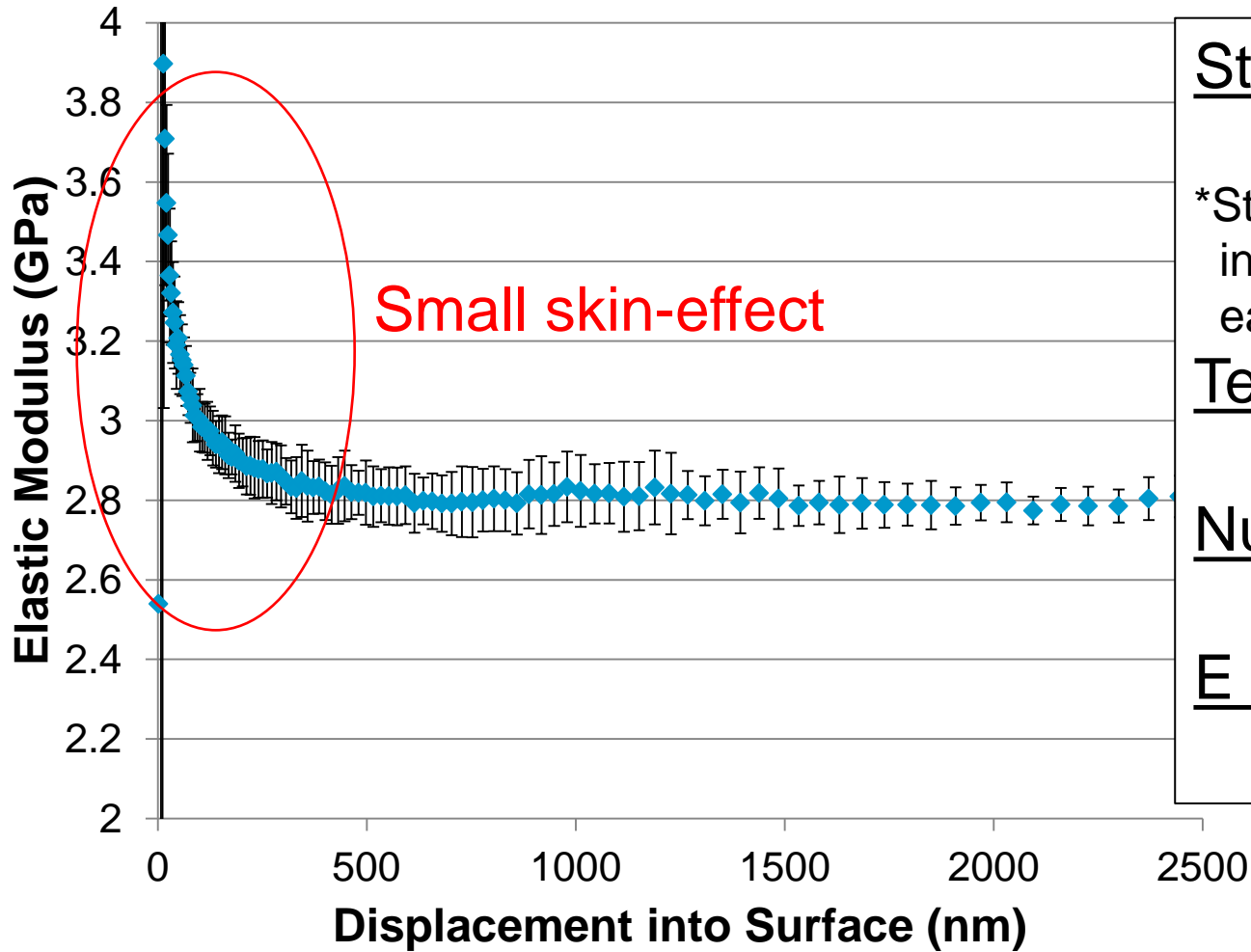
15

E *ultra-sound*

2.83 GPa



Nanoindentation Results on Polycarbonate



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0.05/s to 2.0/s

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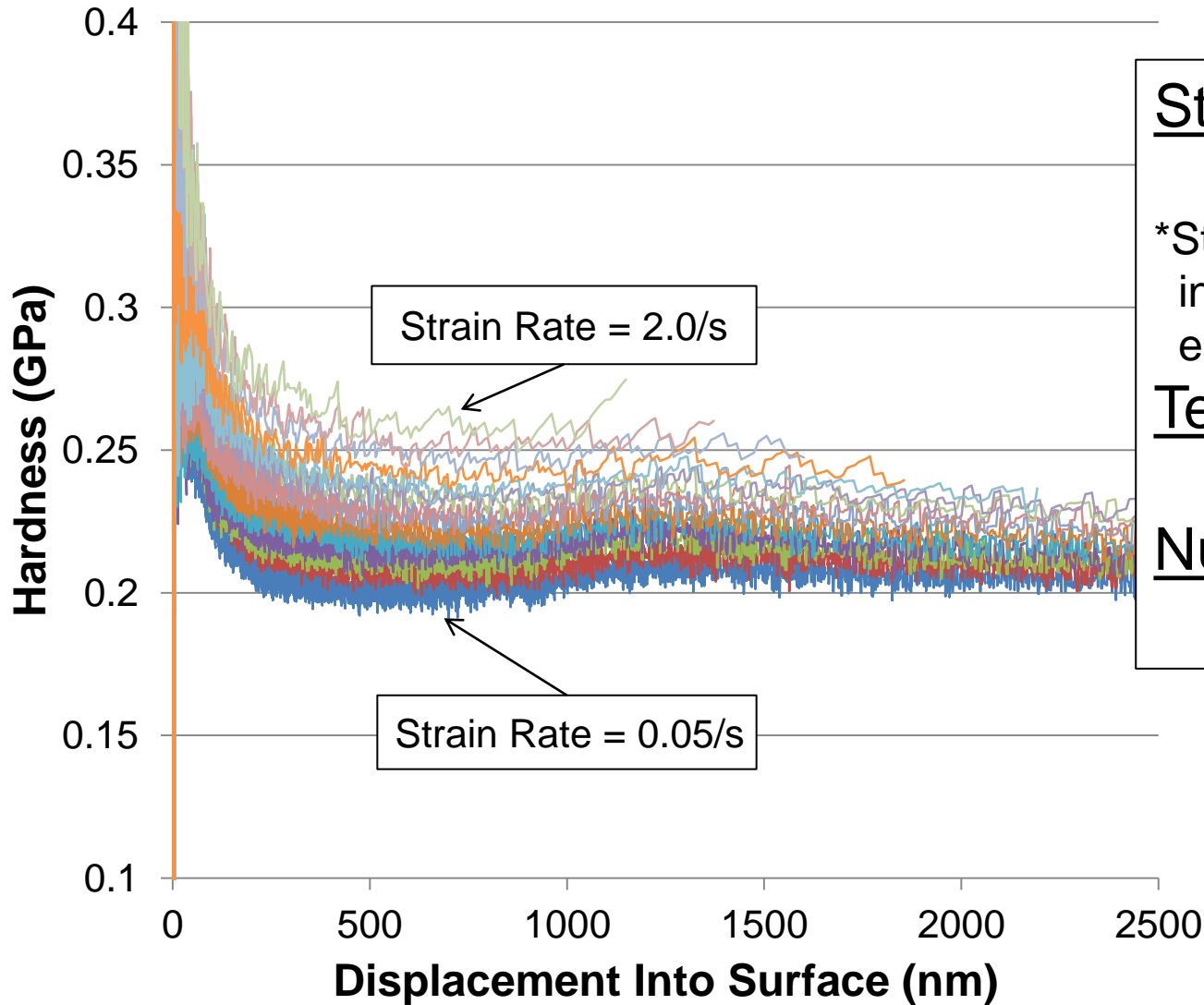
Number of Tests

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Nanoindentation Results on Polycarbonate



Strain Rate Range*

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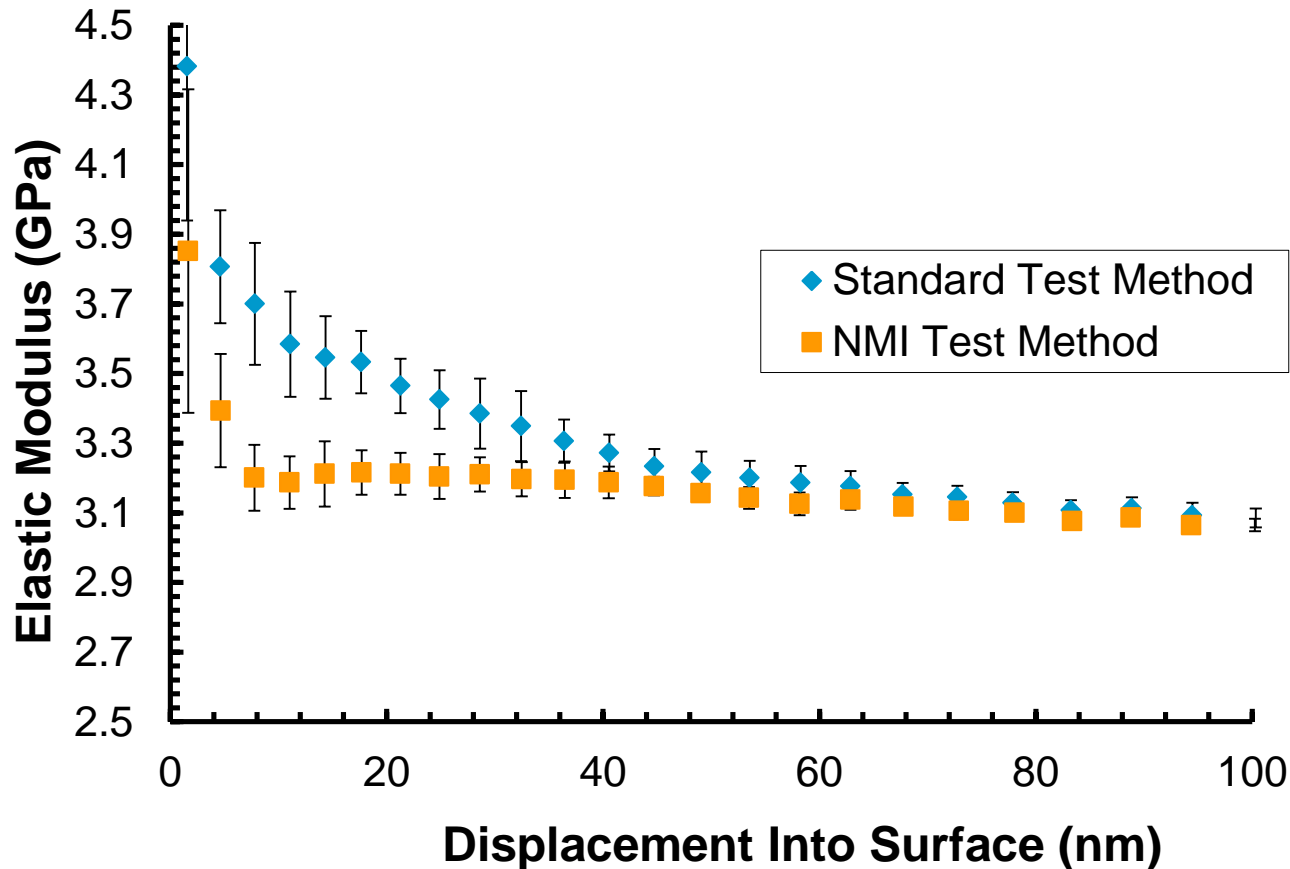
26°C

Number of Tests

15



Identification of test discrepancies using low modulus reference materials



Strain Rate

0.05/s

Number of Tests

10/each

Same Sample

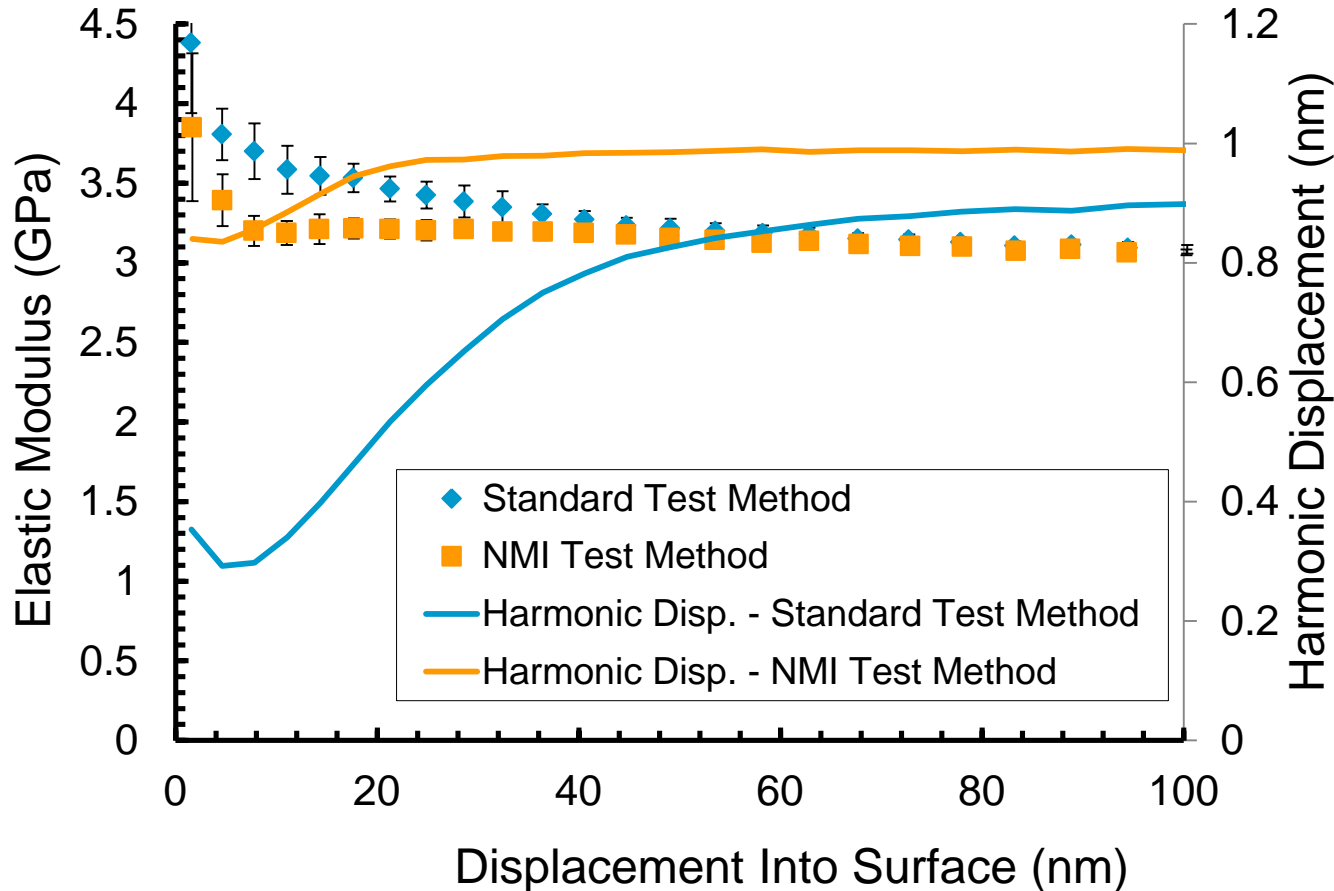
Same Tip

Same Day

Same Temp.

Initial Observations

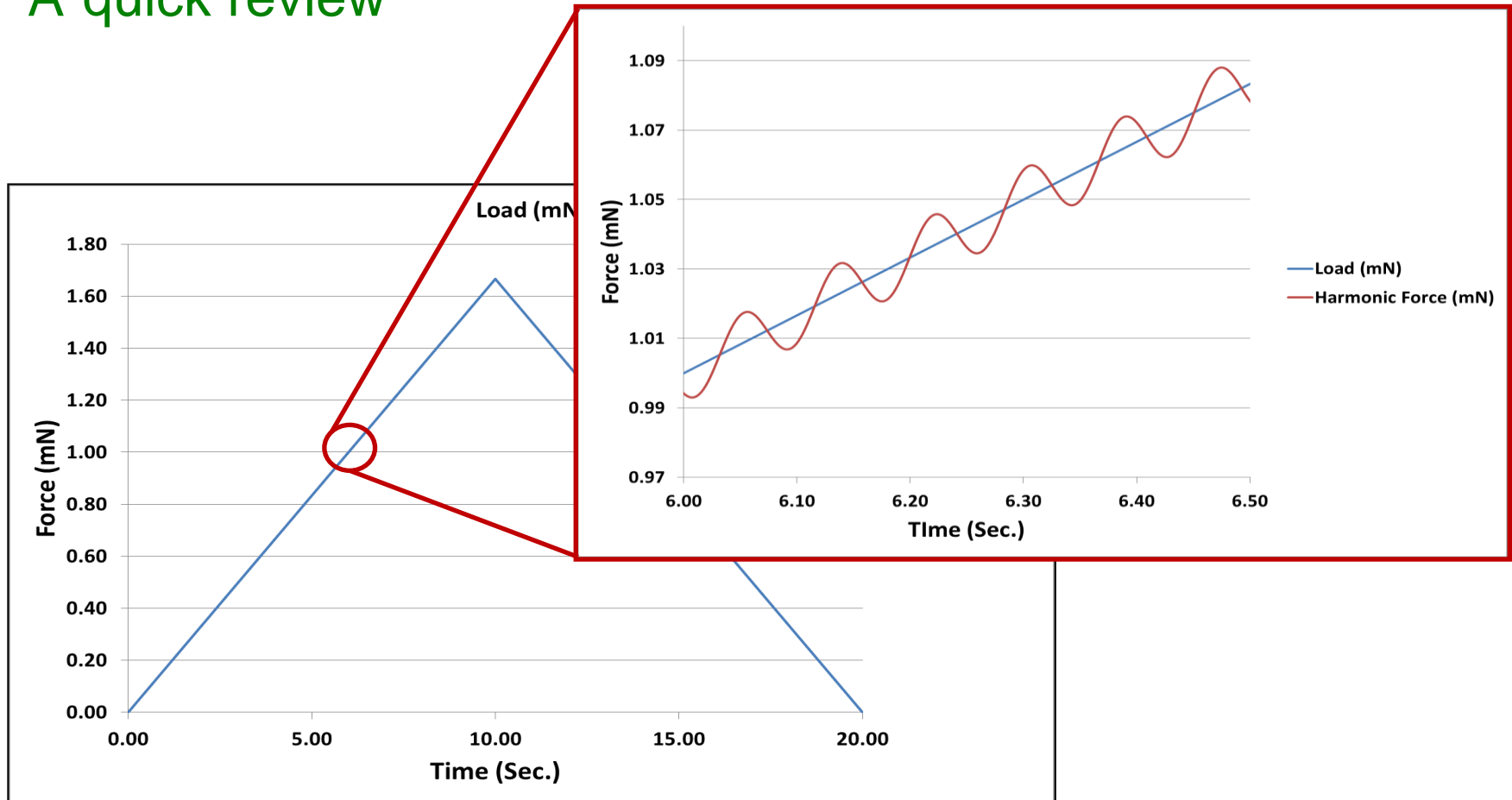
Harmonic displacement is not at a steady-state value



It was assumed that non-steady state harmonic displacement would cause errors in the stiffness measurement due to time constants.

Continuous Stiffness Measurement (CSM)

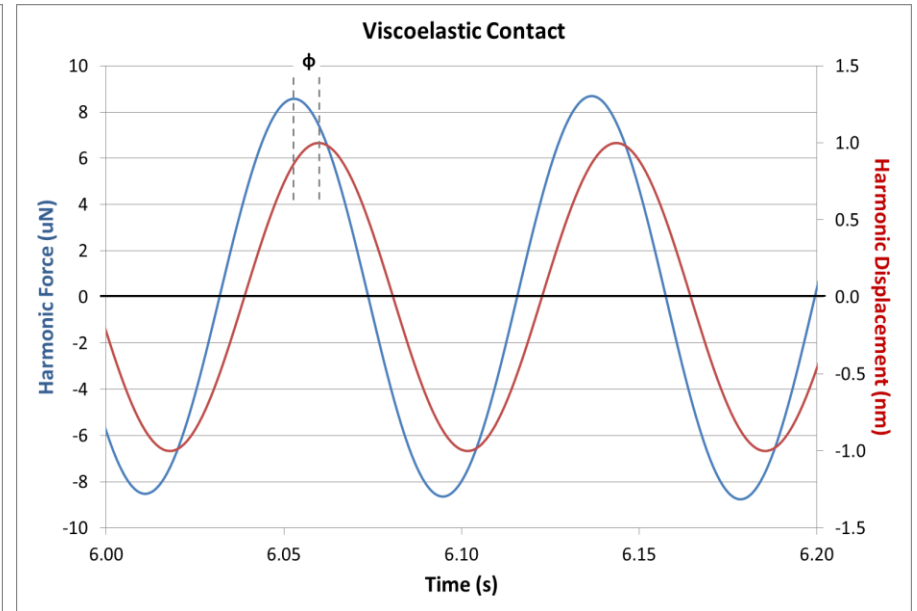
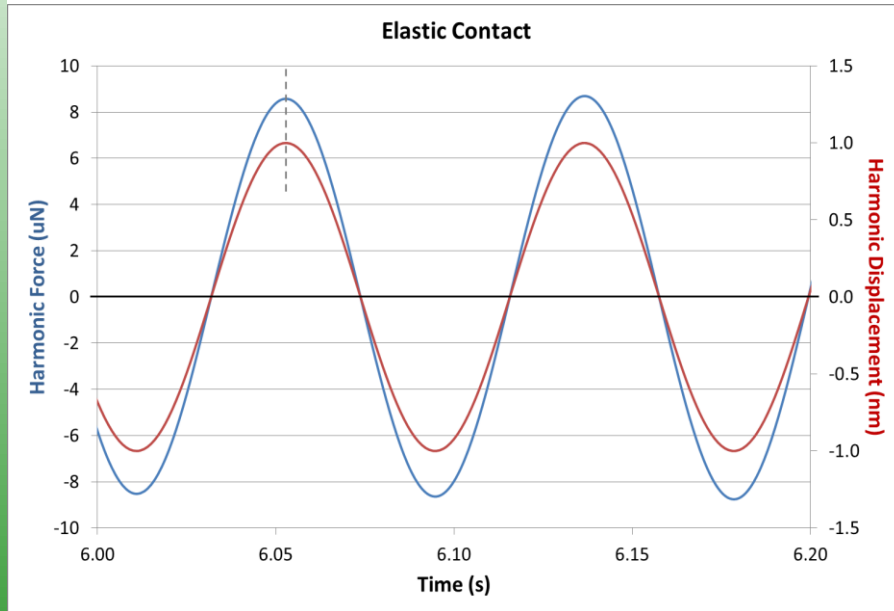
A quick review



Courtesy of Nanomechanics, Inc.
www.nanomechanicsinc.com

CSM - Linear Elastic Materials

A quick review



The phase and amplitude of the material's response to the force oscillations are characteristic of the stiffness of the contact as well as the damping of the material.

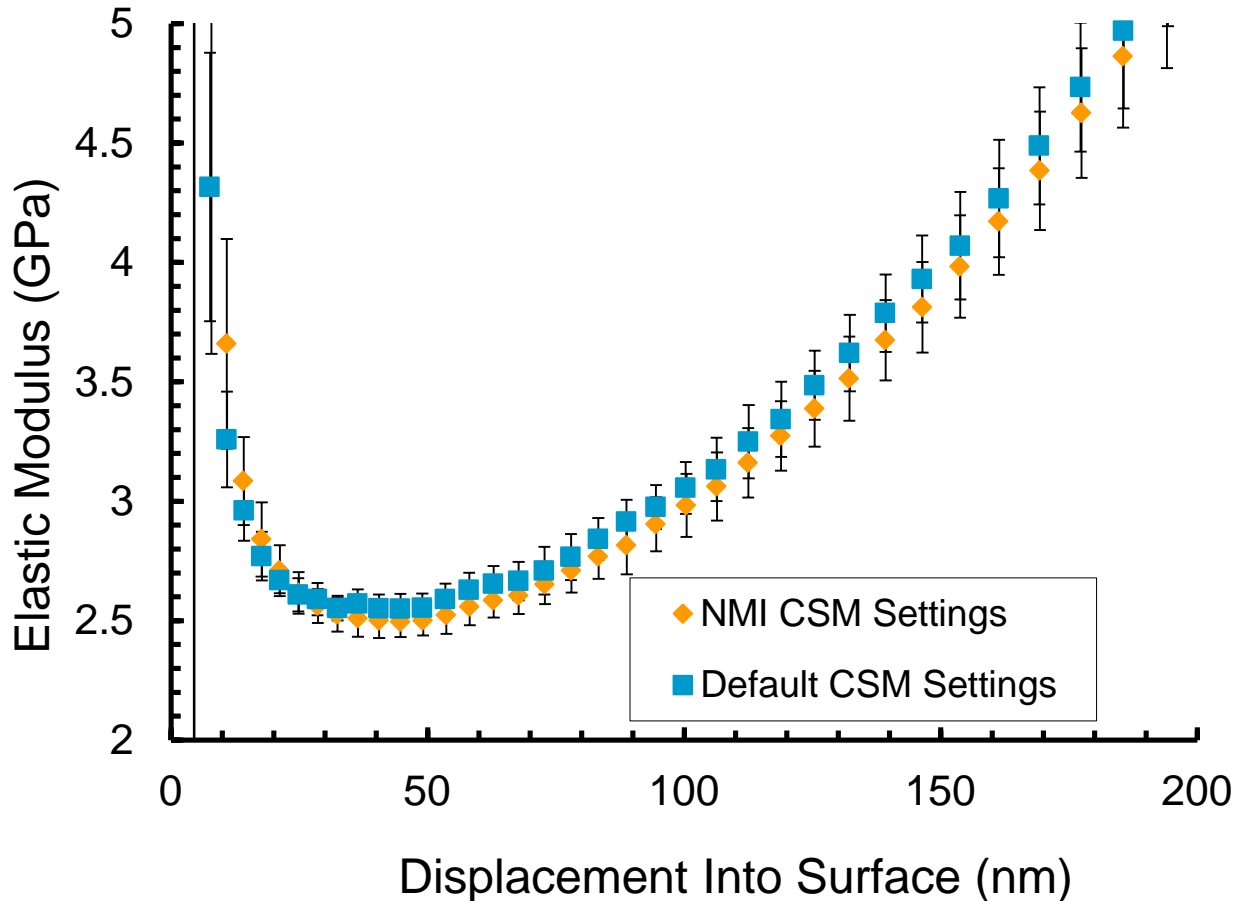
$$S = \frac{F}{h} \cos \phi$$

Courtesy of Nanomechanics, Inc.
www.nanomechanicsinc.com

$$C\omega = \frac{F}{h} \sin \phi$$

The Check

Same test method with different CSM controls = same result



Sample

Low-K film

Film Thickness

495 nm

Strain Rate

Default CSM tests

0.02/s to 0.8/s

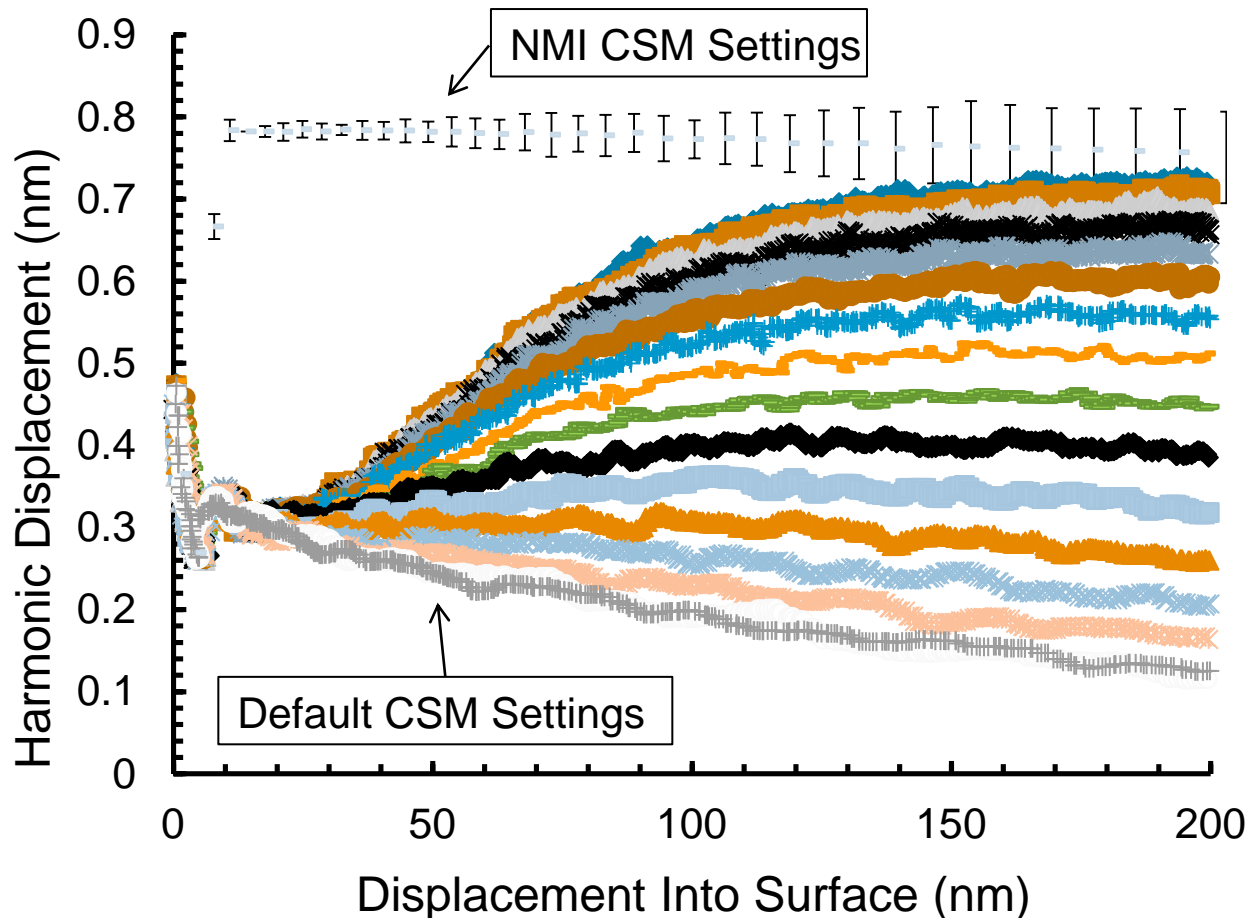
NMI CSM tests

0.02/s to 1.4/s

*At these rates, full CSM tests are being completed in less than 10 seconds.

The Twist

Harmonic Displacement – not the problem



The same results were obtained despite the huge difference in harmonic displacement response.

Therefore, the harmonic stability was not the culprit for the difference in the skin-effect.

The phase lock amplifier for the CSM unit is doing a fantastic job of measuring stiffness even under difficult situations.

Closer Examination Reveals

Surface contact error of approximately 1.5 nm

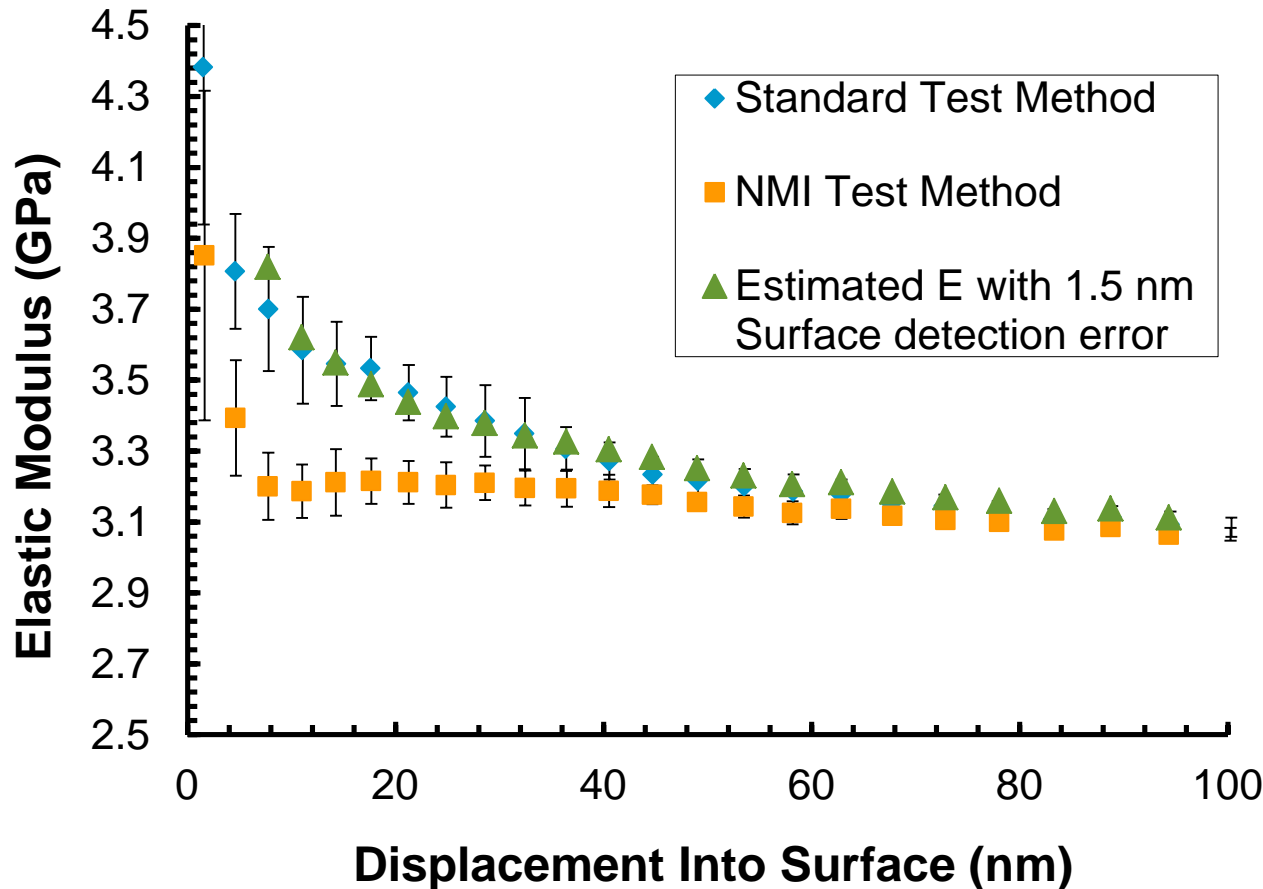
Standard Test Method

Default data rate: 5 Hz

Default Approach Rate:
10 nm/s

The contact error in the standard test method is due to too slow of a default data rate as the tip approaches the surface.

*The data rate does not cause errors in fused silica because the small force change between data points does not cause a significant displacement change when the tip initially contacts the surface.



Conclusions

A low modulus reference material has been shown to increase consistency in measurements between test techniques.

The default settings of the Continuous Stiffness Measurement technique show excellent performance up to a strain rate of 0.8/s on low-k materials; with the NMI modifications, strain rates of 1.4/s can be accurately achieved in testing low-k materials.

Make sure that data acquisition is sufficient for at least 0.5 nm resolution in the surface approach data.

*Polycarbonate reference materials will be available through Nanomechanics' Analytical Services Laboratory starting in early spring.

