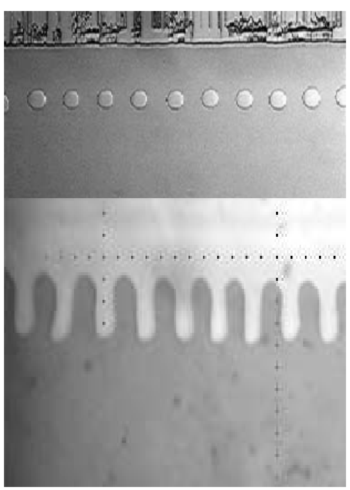
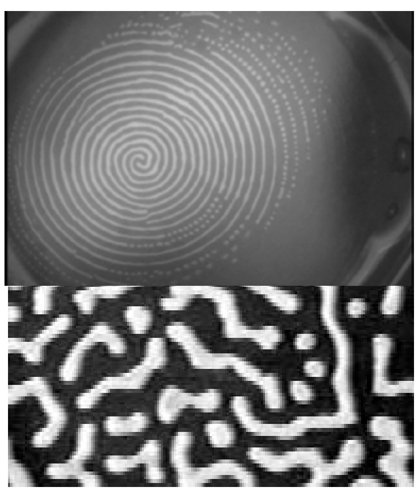
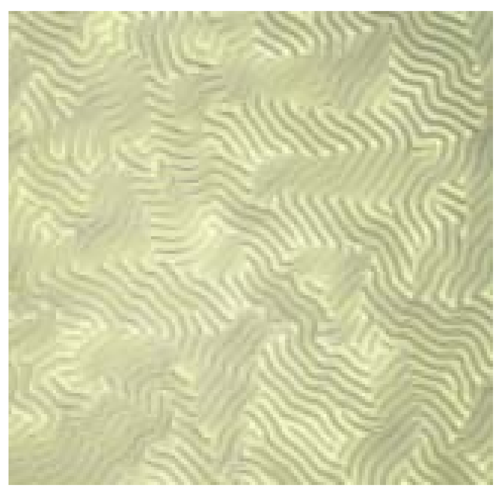
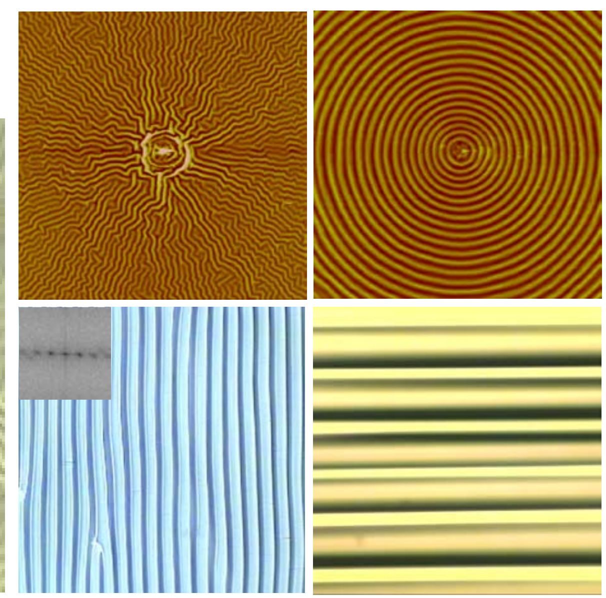
Surface Wrinkling: A Powerful Tool for Measuring Material Properties

Jun Young Chung

Polymers Division
National Institute of Standards and Technology

(NCMC 14 – November 7, 2008)

Instabilities & Self-Patterning by Stress Engineering

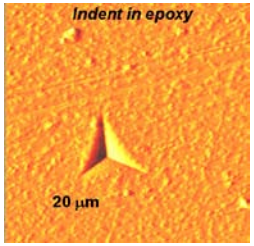
Motivation

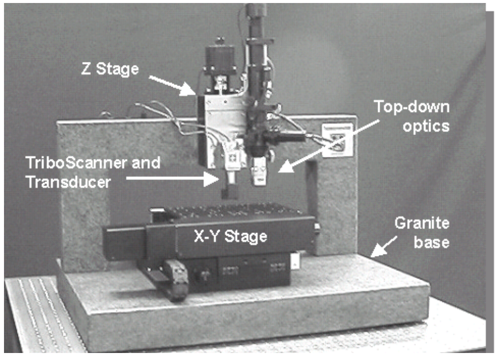
- Mechanical properties (e.g., modulus, Poisson's ratio, CTE) are critical in many applications.
  - performance and reliability
  - predictive modeling of complex systems

- Measuring mechanical properties of sub-micron (nano) films remains difficult.

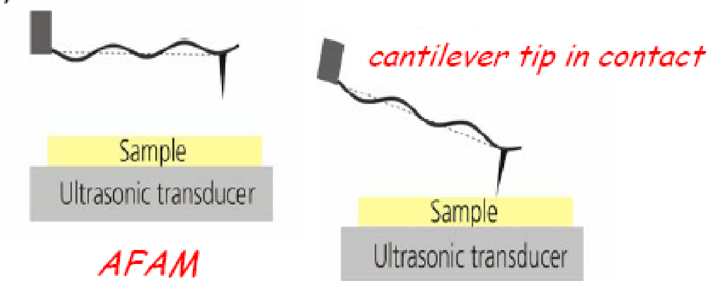
Nanomechanics:

Indentation



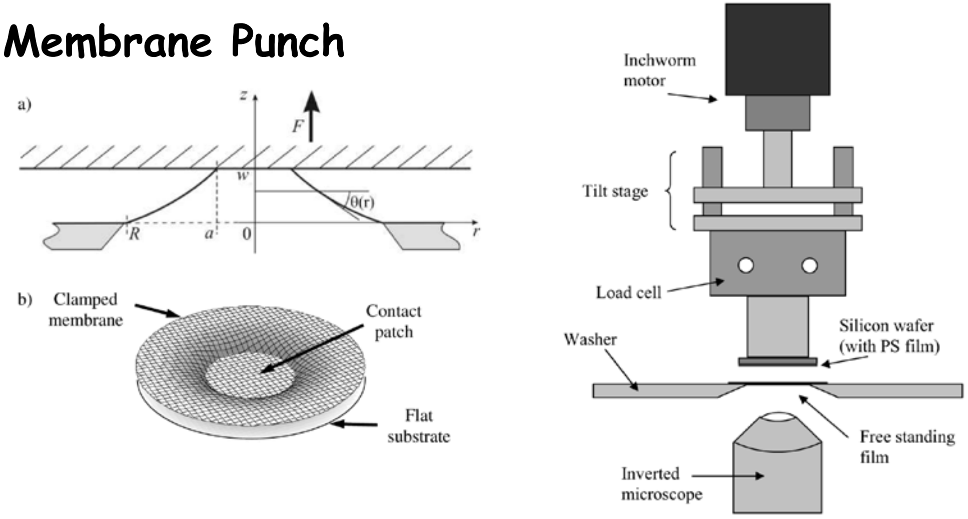


clamped-free AFM cantilever



Rabe et al. *J Vac Sci Technol B* **15** 1506 (1997)
Hurley et al. *J Appl Phys* **94** 2347 (2003)

Membrane Punch



Raegan et al. *Eur. Phys. J. E* **19**, 453-459 (2006)

Challenged by thin (soft) polymers
- substrate effects!

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Wrinkling-Based Metrology

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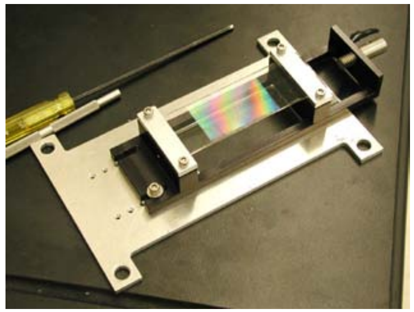
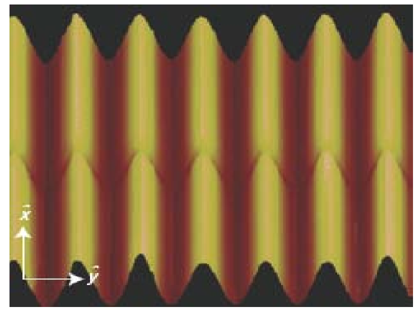
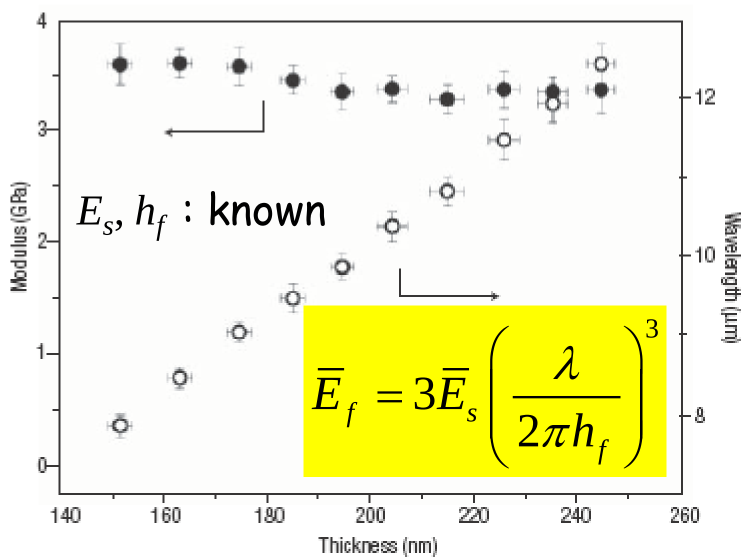
Technology Administration, U.S. Department of Commerce

Young's Modulus Measurements:





PS Film / PDMS



Stafford et al., *Nature Mater* (2004)

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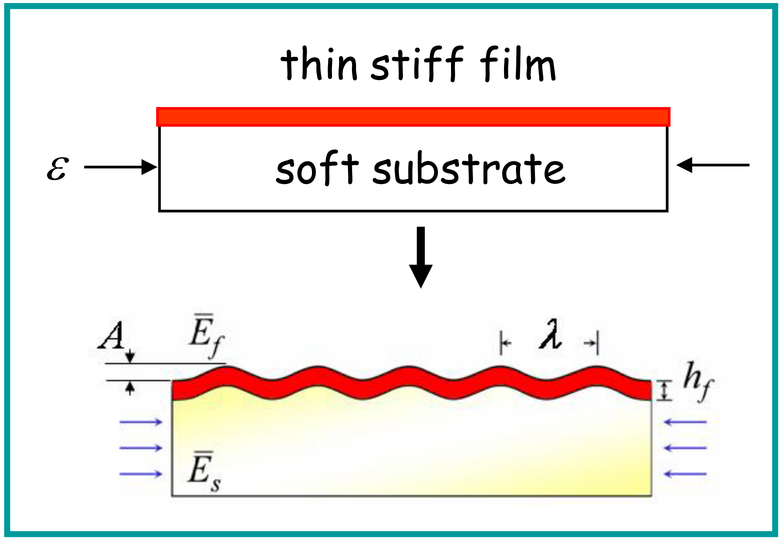
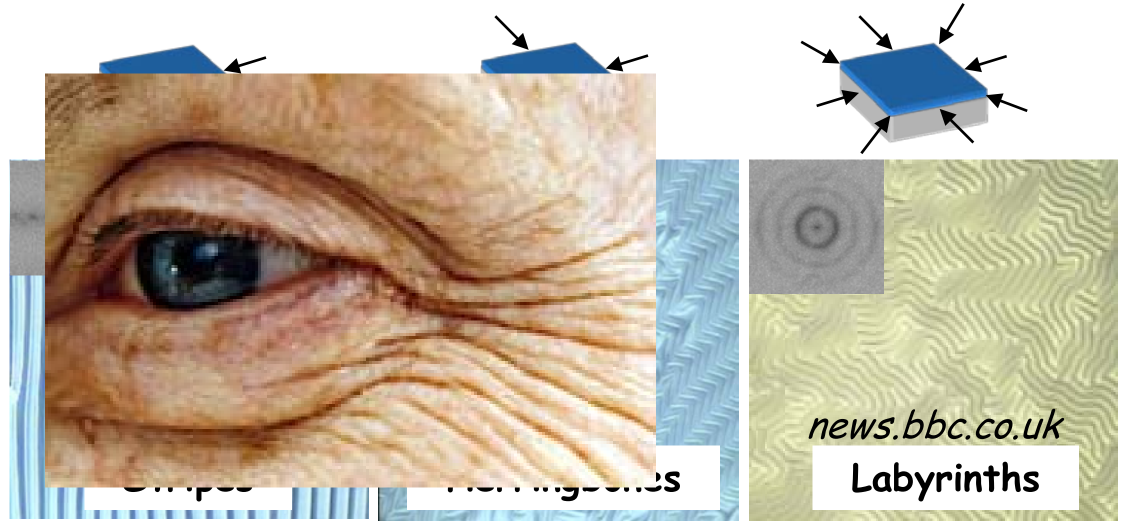
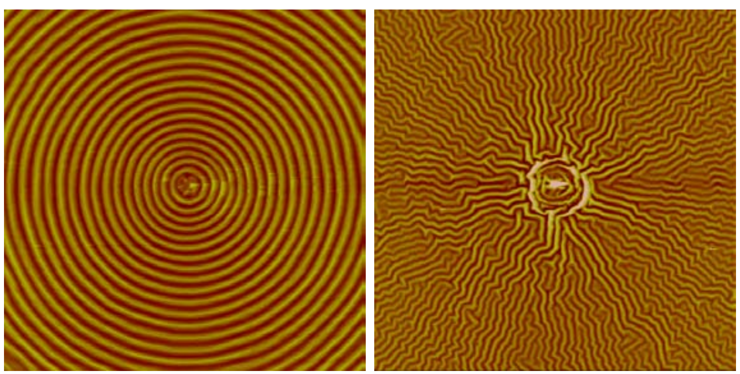
National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

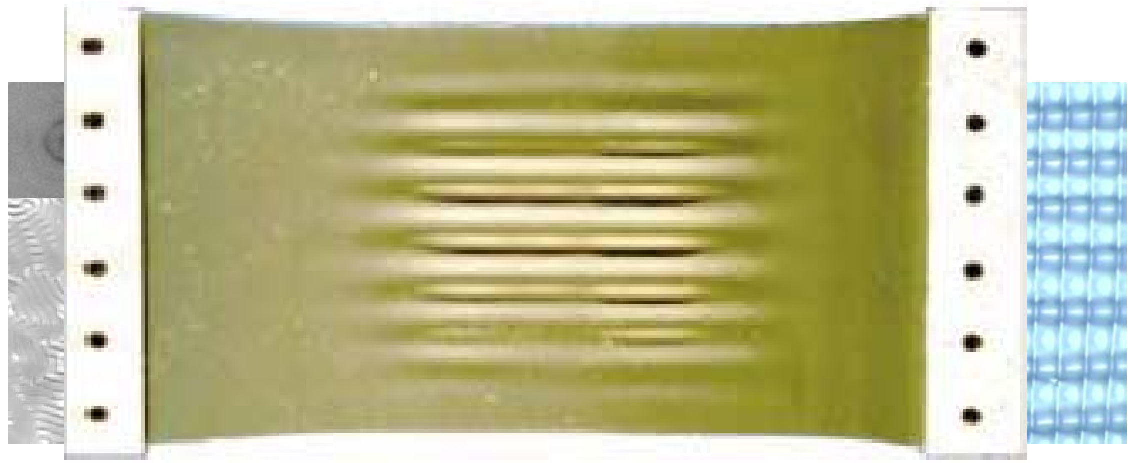
Surface Wrinkling





Mechanical Compression





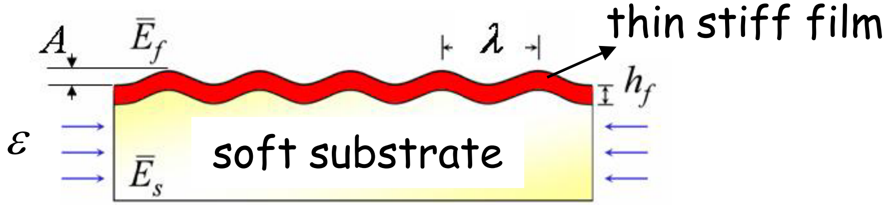
Solvent Swelling through Initial Defects

Thermal Contraction

Mechanical + Thermal

On Pre-patterned Substrates

Surface Wrinkling - Mechanics



Governing Equations:

$$\lambda = 2\pi h_f \left(\frac{\bar{E}_f}{3\bar{E}_s} \right)^{1/3} \qquad A = h_f \left(\frac{\varepsilon}{\varepsilon_c} - 1 \right)^{1/2}$$

$$\varepsilon_c = -\frac{1}{4} \left(\frac{3\bar{E}_s}{\bar{E}_f} \right)^{2/3}$$

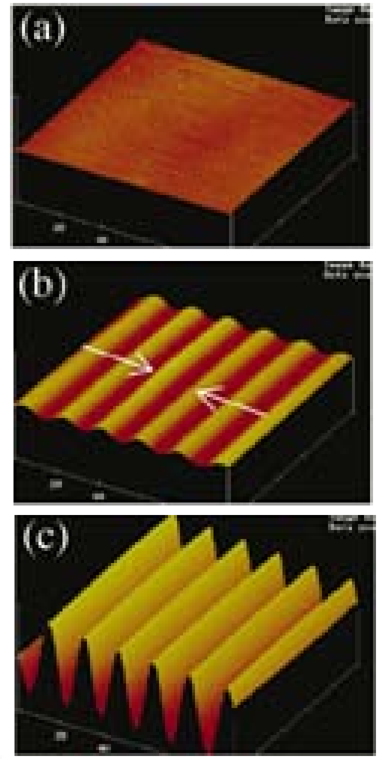
$$\bar{E} = E/(1-\nu^2)$$
$$E_s << E_f$$
$$h_s >> h_f$$

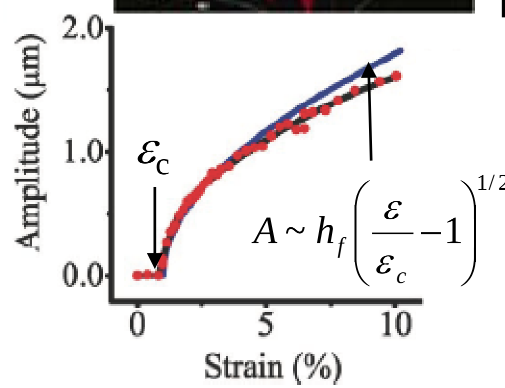
- interface must be well-bonded
- materials behave elastically

Volynskii et al., *J Mater Sci* (2000), Groenewold, *Phys A* (2001)*, Huang, *J Mech Phys Solids* (2005)

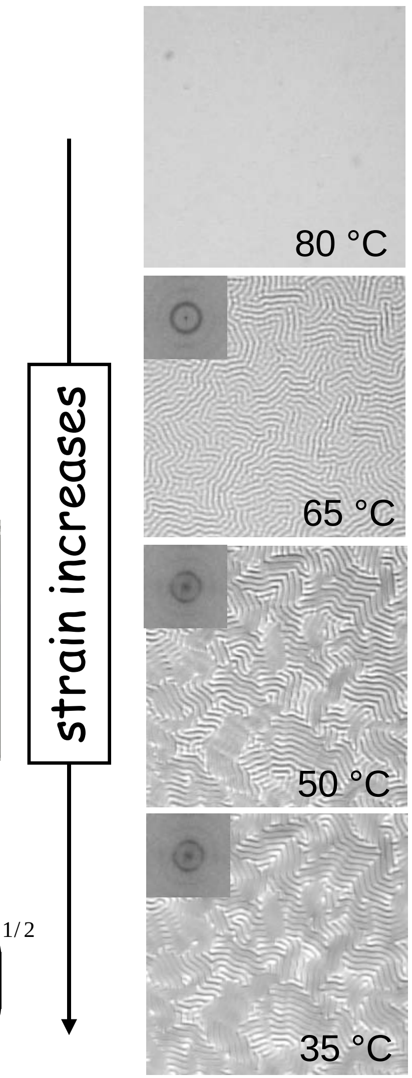
Wrinkling wavelengths are insensitive to strain for low strain levels and wrinkles are reversible.

Mechanical (PS Film / PDMS) **Thermal**





Harrison et al., *APL* (2004)



Mei, Chung, Stafford, Yu & Huang, *APL* (2007)

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Wrinkling-Based Metrology

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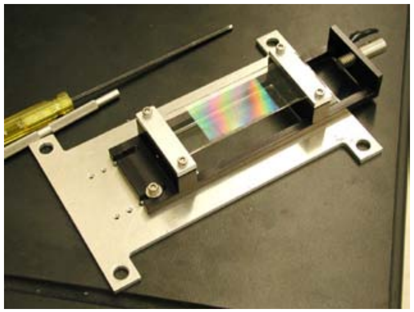
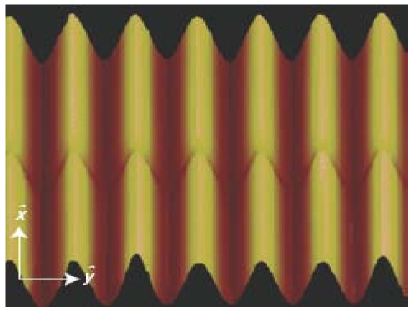
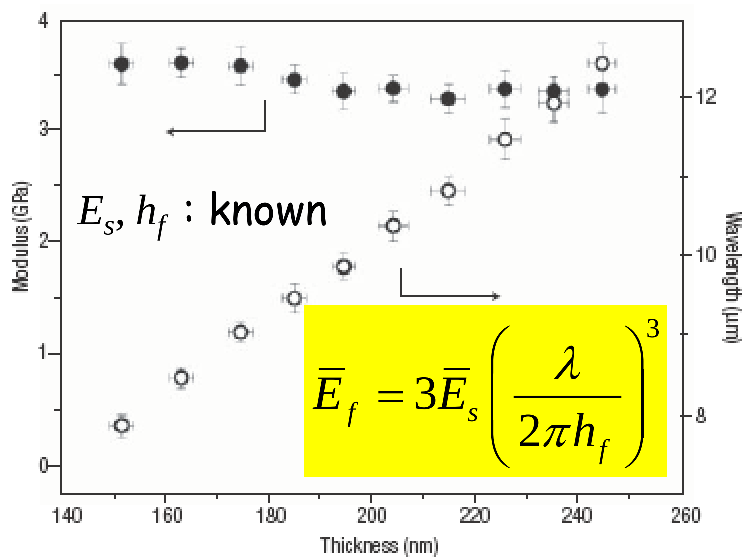
Technology Administration, U.S. Department of Commerce

Young's Modulus Measurements:





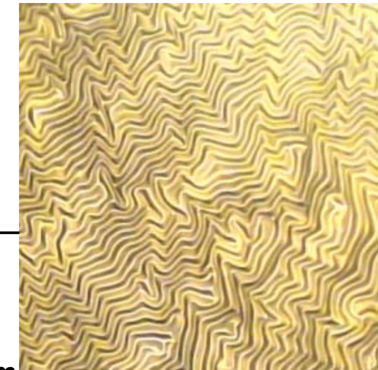
PS Film / PDMS

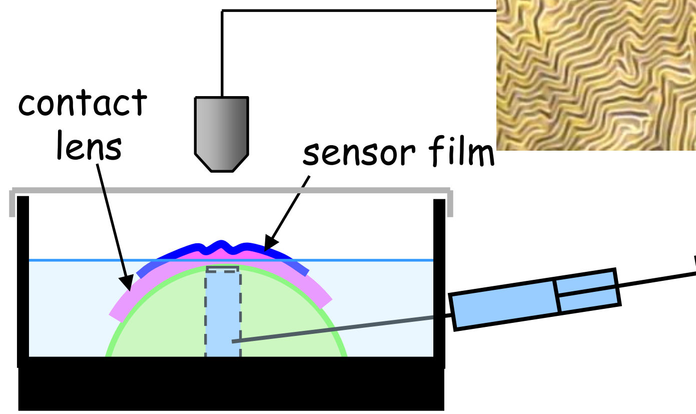


$$\bar{E}_f = 3\bar{E}_s\left(\frac{\lambda}{2\pi h_f}\right)^3$$

Stafford et al., *Nature Mater* (2004)

PMMA Film / Contact Lens





Reverse metrology *– Employ a 'sensor' film of known modulus and thickness to report back the substrate modulus:*

$$\bar{E}_s = \frac{\bar{E}_f}{3}\left(\frac{\lambda}{2\pi h_f}\right)^{-3}$$

E_f, h_f : known

NCMC Focus Project

Vistakon Division
of Johnson & Johnson Vision Care, Inc.

Wilder et al., *Macromolecules* (2006)
Chastek, Chung, Hopson, Fasolka & Stafford (in progress)

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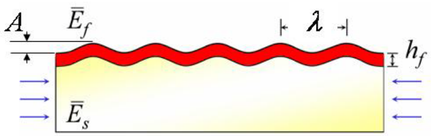
Leveraging Wrinkling Approaches (I)

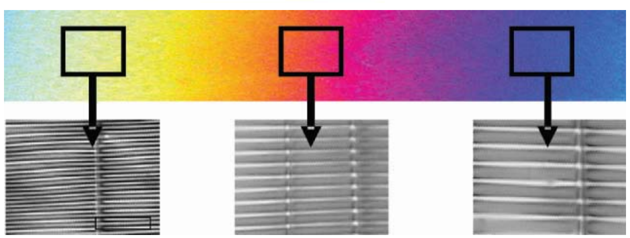
NIST National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

Taking on the challenge of difficult systems:

Wrinkling Approach: Mechanical Modulus across Gradient Polymer Film Libraries



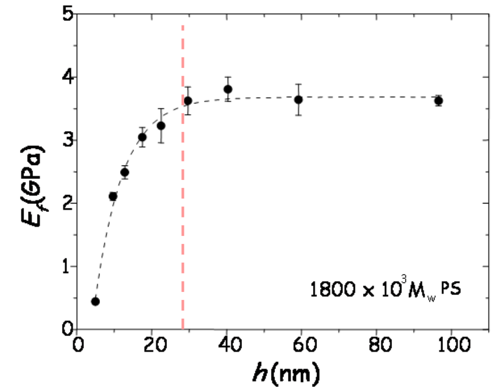
$$\bar{E}_f = 3\bar{E}_s\left(\frac{\lambda}{2\pi h_f}\right)^3$$



Stafford et al., *Nat Mater* (2004)
Stafford et al., *Rev Sci Instrum* (2005)

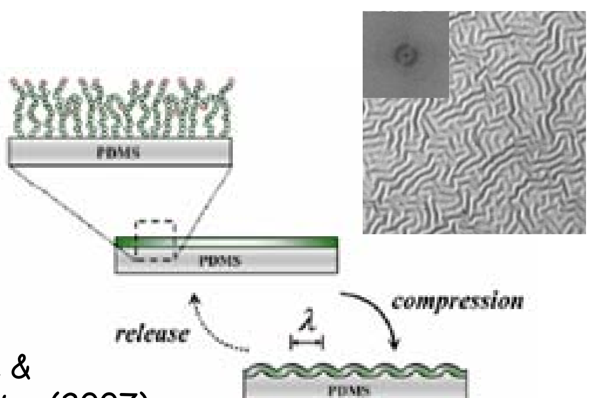
Extend Limit

Modulus of ultrathin films (< 100 nm!)



Stafford et al., *Macromolecules* (2006), Huang et al. *J Aerospace Eng* (2007)

Polymer Brush

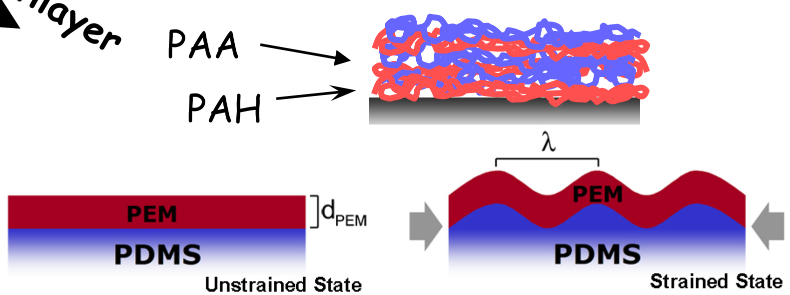


Polymer brush layers tethered on PDMS

Huang, Chung, Nolte & Stafford, *Chem Mater* (2007)

Polyelectrolyte Multilayer

LbL assembly of polyelectrolytes directly on PDMS



Nolte et al., *Macromolecules* (2005) and (2006)
Nolte, Chung, Walker & Stafford (submitted)

Leveraging Wrinkling Approaches (II)

Taking on the challenge to perform difficult measurements:

- Thermo-mechanical properties

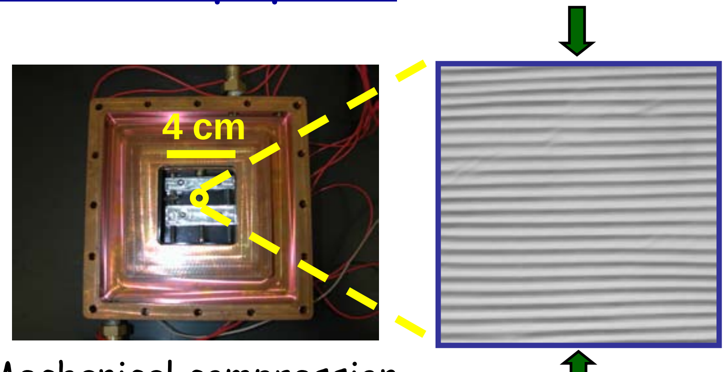
$$\overline{E}_f(T) = 3\overline{E}_s\left(\frac{\lambda(T)}{2\pi h_f}\right)^3$$

- Residual stress

$$\varepsilon_{Theory} = -\frac{1}{4}\left(\frac{3\overline{E}_s}{\overline{E}_f}\right)^{2/3}$$

Access to thermo-mechanical properties

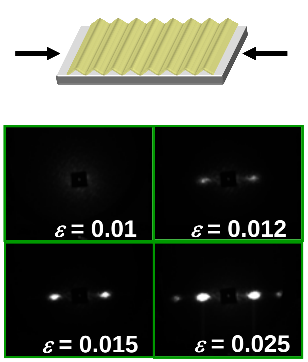
Temperature-Dependent Modulus

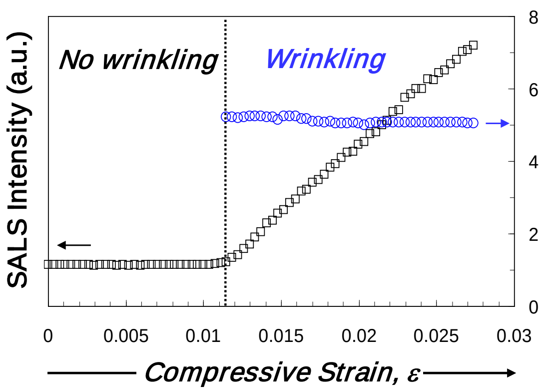


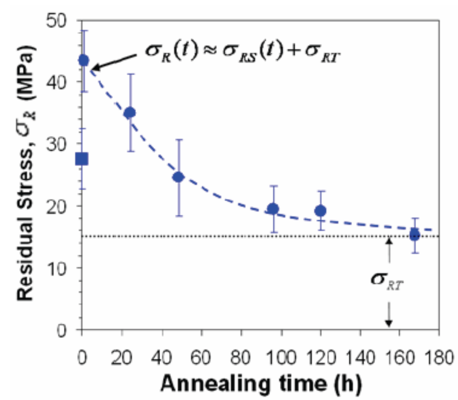
Mechanical compression at an elevated temp.

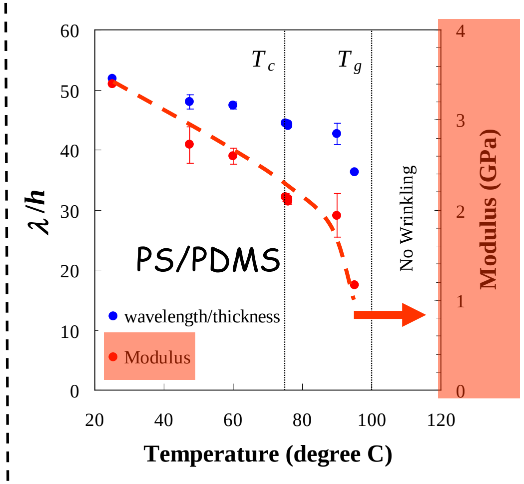
Refine Analysis

Chung, Chastek, Fasolka, Ro & Stafford (submitted)









Chung & Stafford (in progress)

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Poisson's Ratio Measurement

NIST
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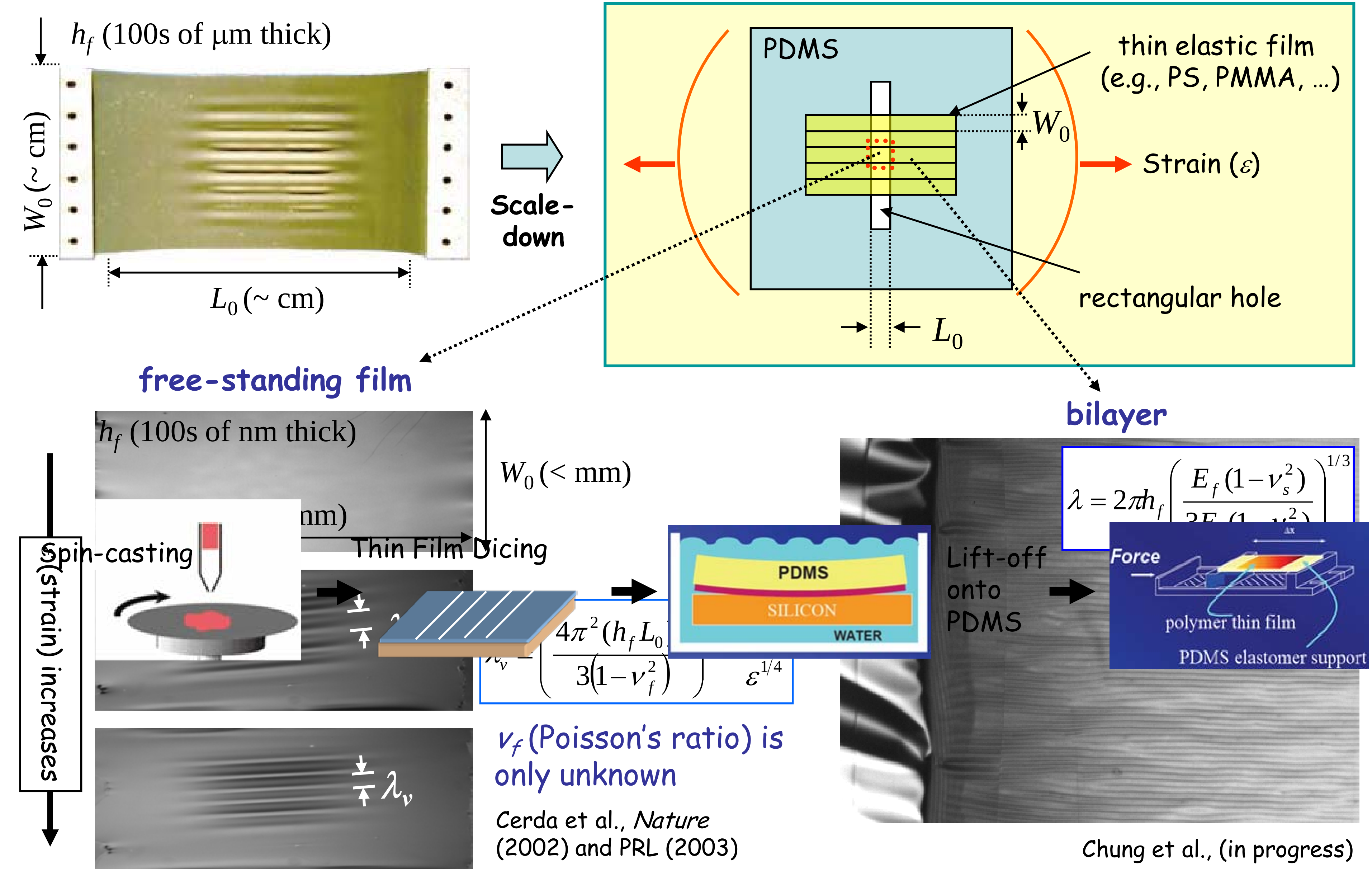
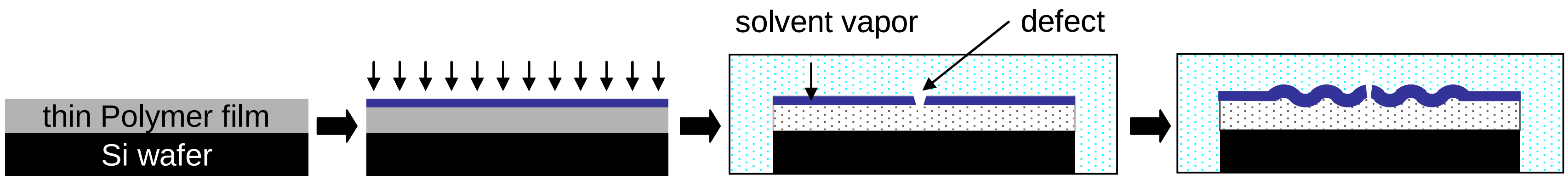
Technology Administration, U.S. Department of Commerce

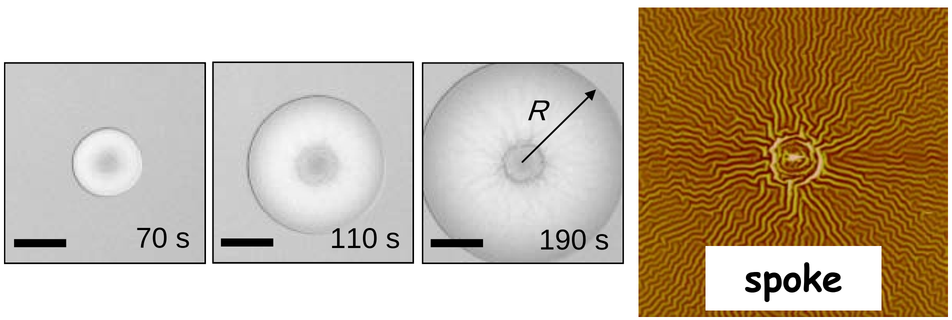


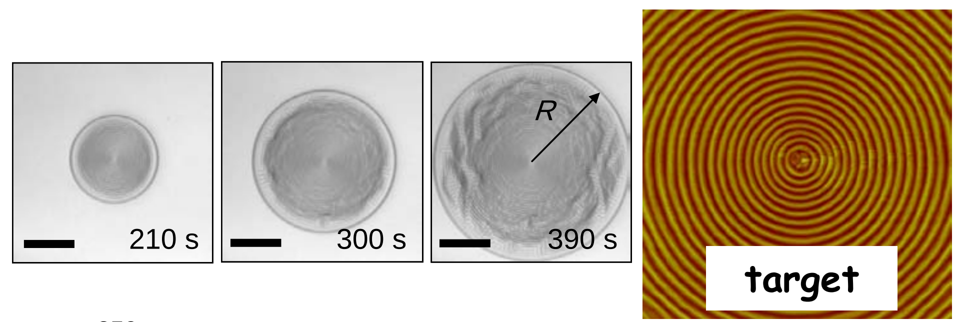
Diffusion Coefficient Measurement

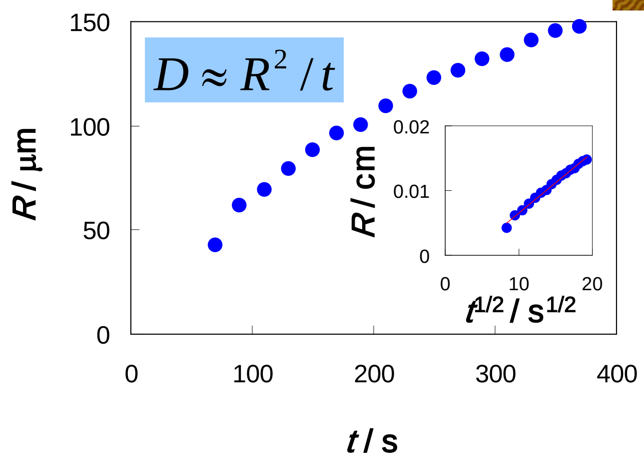
Differential swelling (Defect-mediated wrinkling nucleation and growth)

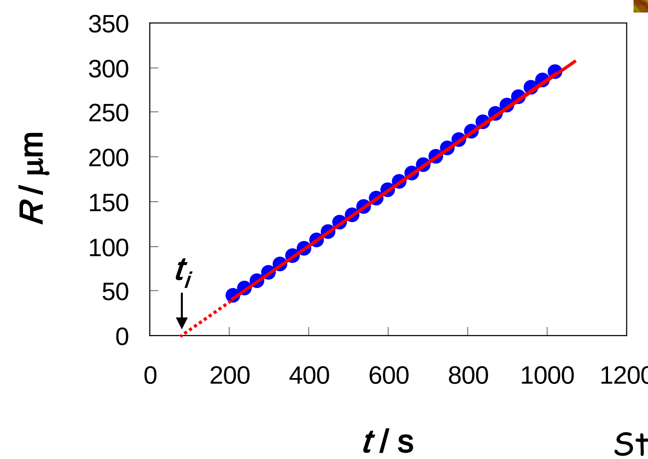


Growth kinetics of spoke and target patterns









Chung, Nolte & Stafford (submitted)

Surface Wrinkling: A Powerful Tool for Measuring Material Properties

- Elastic Modulus of Thin Films: glassy polymers (PS and PMMA)
  hydrated polymer gels (contact lenses)
  polymer brush layers
  polyelectrolyte multilayers

- Themo-Mechanical Properties: temperature-dependent modulus
  coefficient of thermal expansion

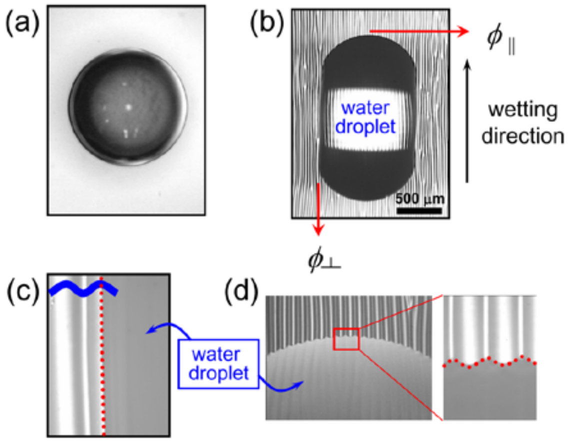
- Residual Stress: nanoscale thin spin-cast polymer films

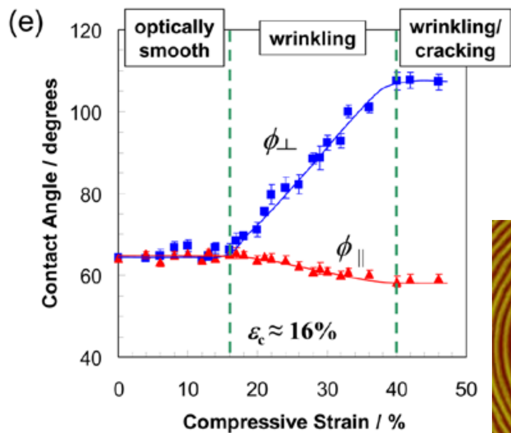
- Poisson's Ratio: glassy polymers
  apply to low-k dielectric materials (collaboration w/ IBM)

- Diffusion Coefficient: glassy polymers

Surface Wrinkling - Technology

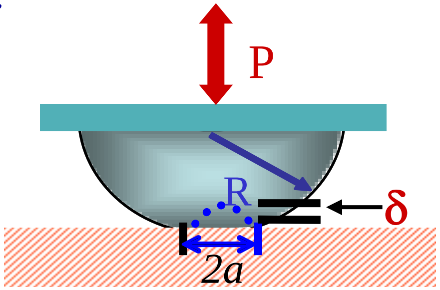
Controlled topography: wettability & adhesion studies, …

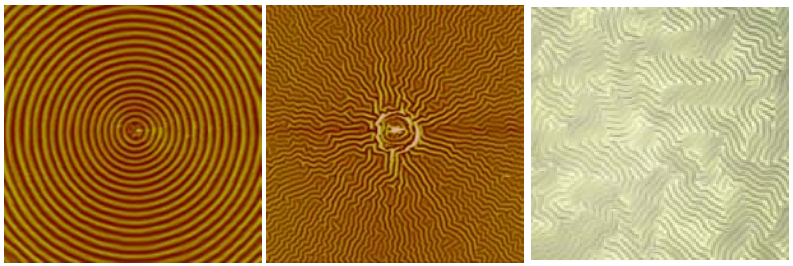




Chung, Youngblood & Stafford, *Soft Matter* (2007)

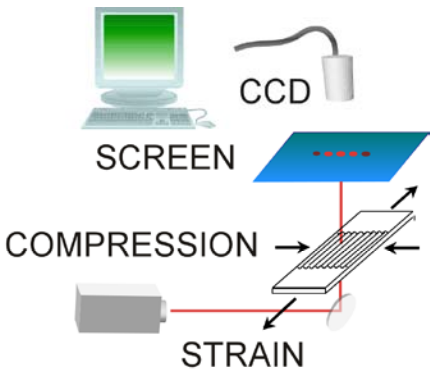
spherical probe

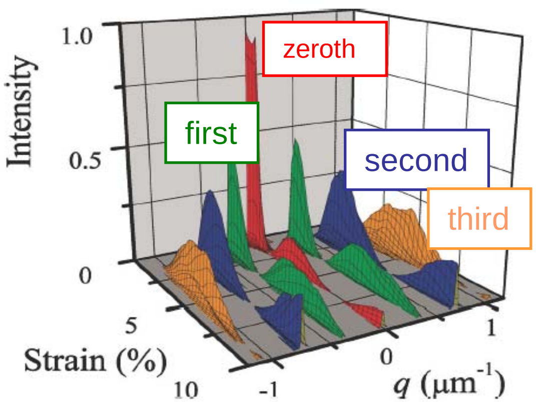




patterned substrate

Periodic nature of wrinkling: tunable phase grating, microfluidics,…





Harrison, Stafford, Zhang & Karim, *APL* (2004)

Acknowledgements

Mentors

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Dr. Michael J. Fasolka (NIST)
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Prof. Robert Y. Lochhead (U. Southern Mississippi)

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Prof. Rui Huang (U. Texas - Austin) for wrinkling mechanics
Prof. Jeffrey P. Youngblood (Purdue U.) for wettability
Dr. Peyton L. Hopson (Vistakon) for contact lenses
Dr. Ho-Cheol Kim (IBM) for low-k materials

NIST

Dr. Thomas Q. Chastek for residual stress measurement
Dr. Adam J. Nolte for brush/LbL wrinkling & diffusivity measurement
Dr. Thuy T. Chastek for reverse buckling
Dr. Hyun-Wook Ro for x-ray reflectivity & thin film stress measurement
Dr. Hae-Jeong Lee for low-k materials
Dr. Peter Johnson
Dr. Edwin P. Chan

Questions?