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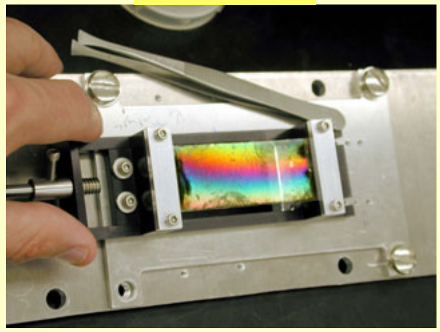
Strain Induced Elastomer Buckling Instability for Mechanical Measurements

A non-contact technique to measure the mechanical properties of thin films

- Mechanical measurements obtained from periodicity of corrugations on thin silicone sheet.
- Applicable to academic systems, *e.g.* polystyrene films, as well as formulations.

With Strain



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Mechanical Properties of Thin Films: The Need

There is a need for a simple, robust, and flexible measurement technique for the mechanical properties of thin polymer films.

Who Would Benefit?

- The COATINGS industry, which tailors its dispersions or optical layers to be of designated mechanical properties.
- The SEMICONDUCTOR industry, which depends upon thin films of photoresist to be mechanically stable.
- The OPTICAL ADHESIVES industry, which sell glues with SPECIFIED mechanical properties and used in THIN FILMS.

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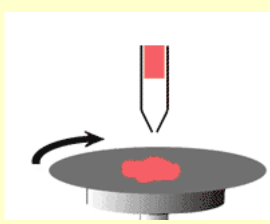
Conventional Techniques

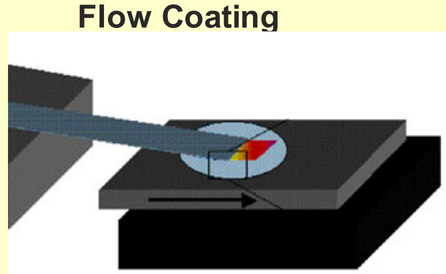
- The properties of the coating are measured in bulk, using A Dynamic Mechanical Analysis (DMA) or Instron-like device **OR**
- Depth Sensing Indentation instruments, or Mechanical Property Microprobes Measure are employed, to **MEASURE**
  - Young's Modulus
  - Yield Stress - creep
  - Visco-elastic recovery
  - Cracking thresholds
  - Adhesive/interfacial energy

Thin Film Fabrication Techniques

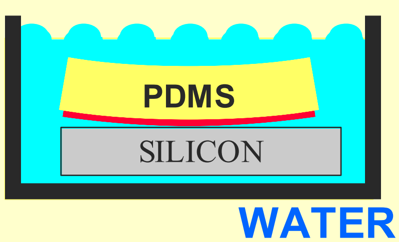
Spin Coating

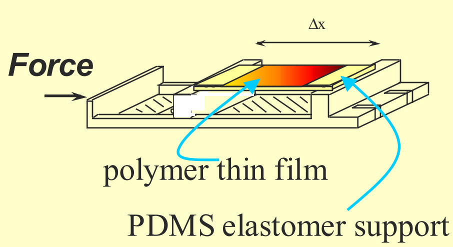


Flow Coating



Lift-Off onto PDMS





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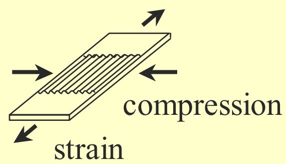
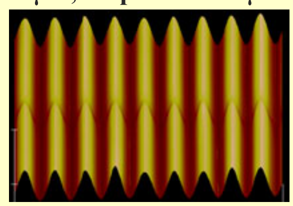
SALS of Buckled Film

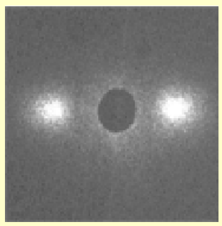
a) Film buckling on strained PDMS



b) AFM image of film (wavelength = 6 µm, amplitude 0.3 µm.

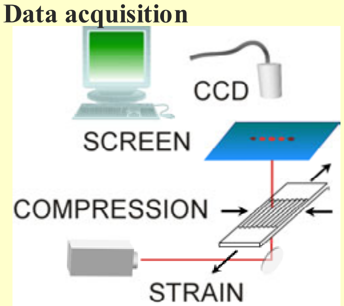


c) Light scattering



$$q = \frac{4\pi}{\lambda} \sin\frac{\theta}{2}$$

d) Data acquisition



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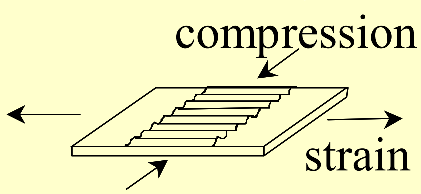
Strain Induced Compression Triggers Mechanical Instability



$$d \sim h\left(\frac{E_p}{E_m}\right)^{1/3}$$

The buckling wavelength, *d*, is directly determined by the film thickness *h*, and moduli of the silicone and polymer film.

- SIEBIMM directly measures the Young's modulus **WITHOUT** material-dependent modeling.
- As SIEBIMM is a LOCAL measurement, multiple samples can be placed on one silicone sheet for independent analysis.

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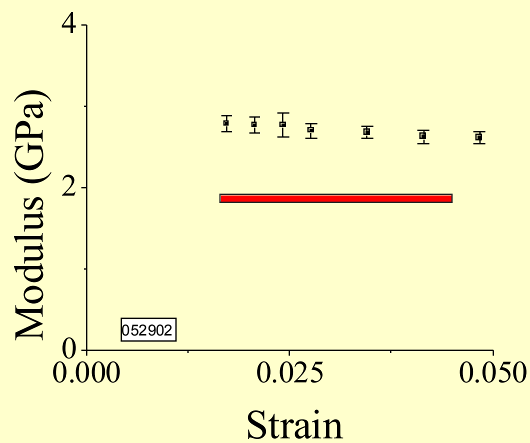
1. Insensitivity of Measured Modulus to Strain

- Even for glassy/brittle polystyrene, modulus measurement relatively insensitive to strain (for strain < 5%).
- Measurements typically made at lowest strain that triggers instability.



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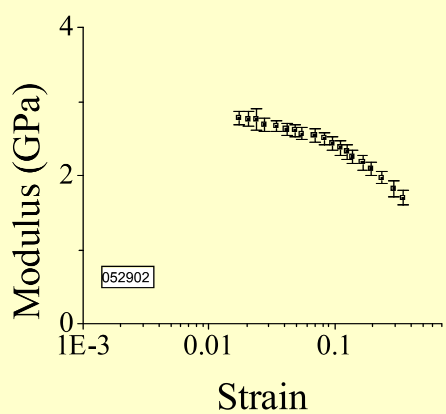
2. Sensitivity of Measured Modulus to Strain

- Large strains (>5%) will crack polymer film, altering its properties and hence the measured modulus.
- Critical strain depends upon both support and film moduli; we tune the support modulus to minimize the critical strain.



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Thickness Gradient Demonstration

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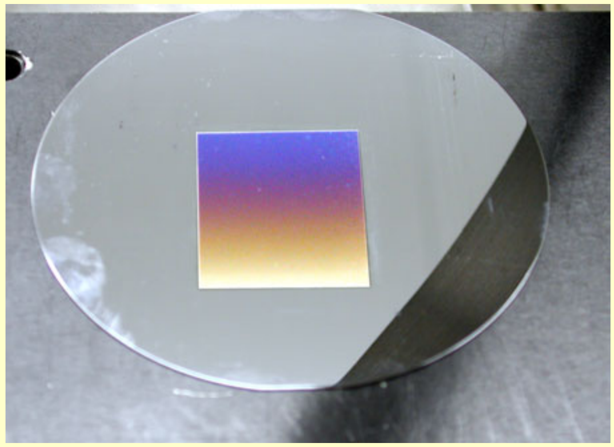

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Effect of Thickness on Buckling Wavelength

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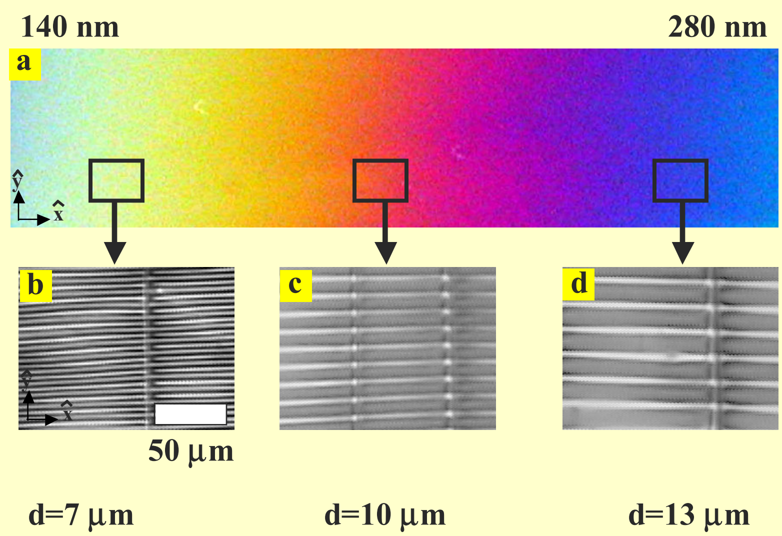

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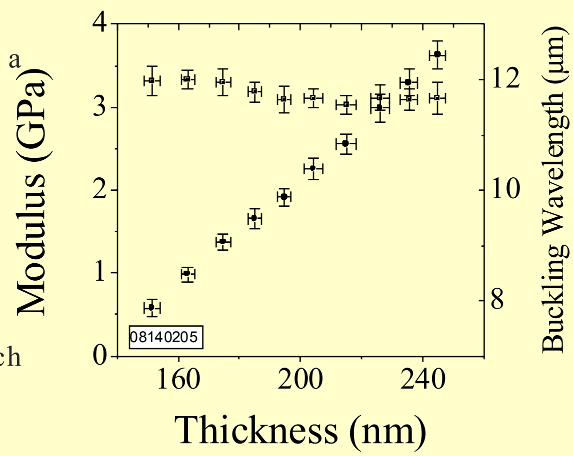
Measurements of Polystyrene Films

- First test: We examined a thin PS film with a thickness gradient. We found that the buckling wavelength *d* increased varied linearly with the thickness.
- Observations in full agreement with sandwich theory.



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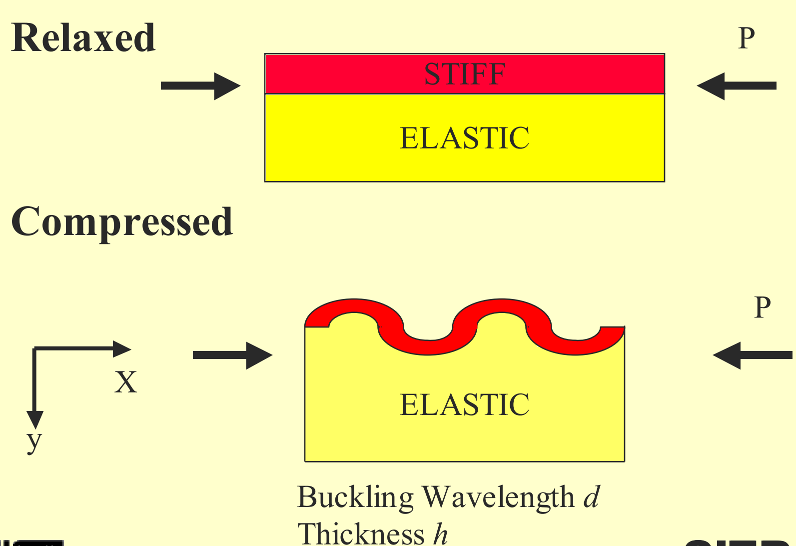
Compression Initiates Mechanical Instability



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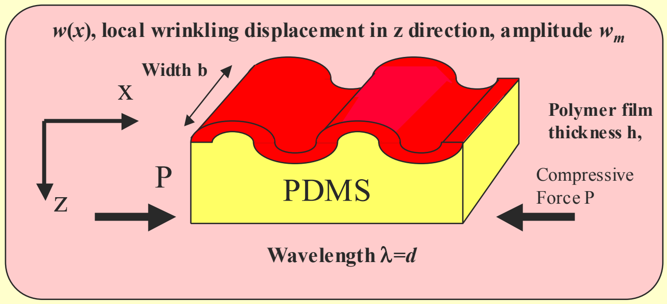
Bending of a Beam:Differential Equation

$$D\frac{\partial^4 w}{\partial x^4}+P\frac{\partial^2 w}{\partial x^2}=b\sigma_z$$



w is the distance the plate is deflected from neutral plane
D is the flexural rigidity of the plate
P is the tensile force in the beam
$b\sigma_z$ is the shear stress force

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Analysis of A Buckled Laminate

Suppose the Strut Buckles into Sinusoidal Waves – plug in:

$$w=w_m\sin\frac{2\pi}{d}x$$

Suppose perfect adhesion, and that the necessary stress is:

$$\sigma_z=-\frac{2a}{d}w_m\sin\frac{2\pi}{d}x$$

Where $$a=\frac{2\pi E_c}{(3-\nu_c)(1+\nu_c)}$$

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Analysis of A Structural Sandwich: Mechanical Instability

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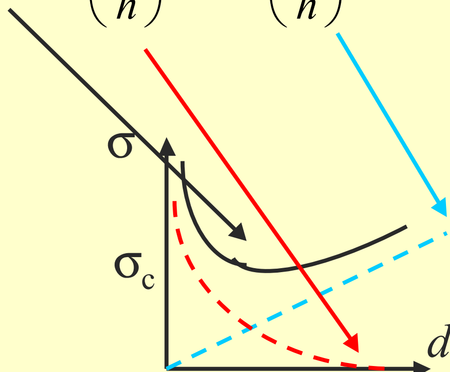
We rearrange, and solve for the critical LATERAL stress σ

$$\sigma = \alpha\left(\frac{d}{h}\right)^{-2} + \beta\left(\frac{d}{h}\right)^{1}$$

So now we ask: what h/l value minimizes the lateral stess? This is somewhat of an energy minimization condition



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Analysis of A Structural Sandwich: Mechanical Instability

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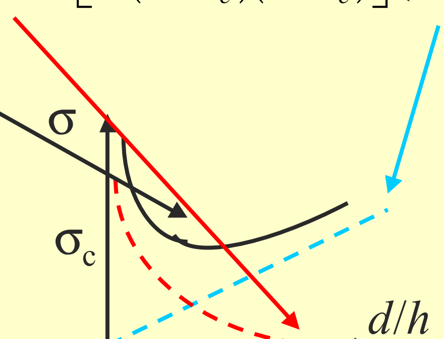
We rearrange, and solve for the critical LATERAL stress σ

$$\sigma = \frac{\pi^2 E_p}{3}\left(\frac{d}{h}\right)^{-2} + \left[\frac{E_c}{\pi(3-\upsilon_c)(1+\upsilon_c)}\right]\left(\frac{d}{h}\right)^{1}$$

So now we ask: what h/l value minimizes the lateral stess? This is somewhat of an energy minimization condition



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Analysis of A Structural Sandwich: Mechanical Instability

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We solve for the critical value of the lateral stress, σ, by the usual minimization condition.

$$\frac{\partial \sigma}{\partial (d/l)} = 0 \qquad d \sim h\left(\frac{E_p}{E_m}\right)^{1/3}$$

Buckling wavelength independent of strain and stress!

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SIEBIMM and PS/Plasticizer

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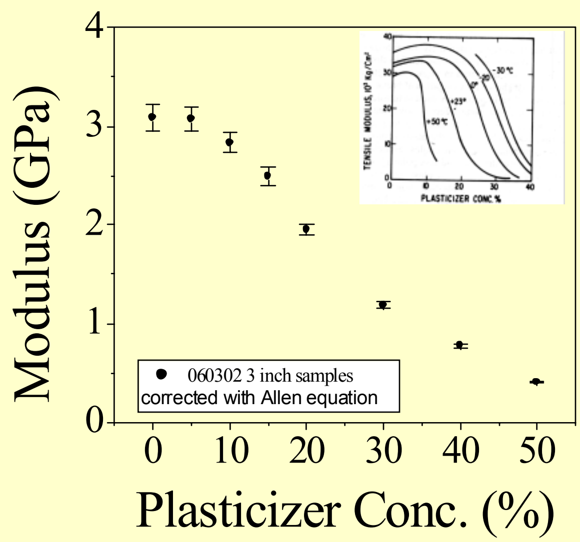
- Modulus measurement of plasticized polystyrene (dioctyl phthalate blend).
- I-BIMM successfully follows decrease in modulus with plasticizer.



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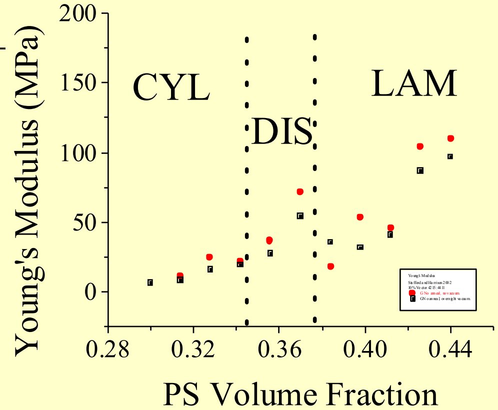
SIEBIMM and Vector S-I-S

- Young's modulus of the film is tuned by the ratio of two S-I-S polystyrene-polyisoprene triblock copolymers: Vector 4215 (30% PS, more rubbery) to Vector 4411 (44% PS, more glassy)
- Optically clear, miscible system - morphological transition has been observed by SAXS. AFM?



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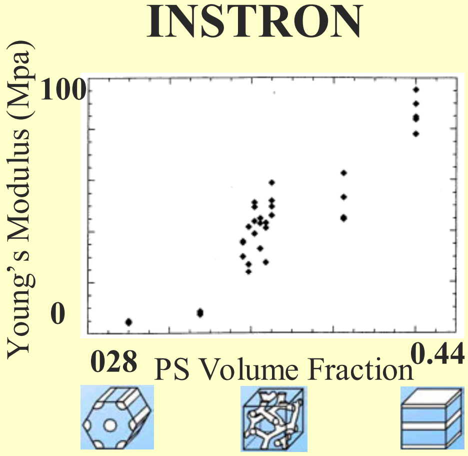
Comparison of SIEBIMM to Instron

INSTRON



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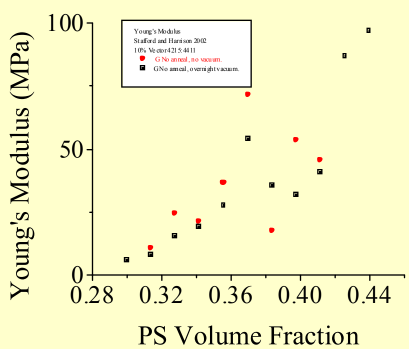

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Challenge: Releasing Polymer Films

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Polymer films often adhere to substrates, making their transfer to PDMS difficult. However , there are some solutions:

- Use of salt plate as a substrate
- Silicon wafer substrate: PRE-coating wafer with a thin layer of soap aids in sample release upon aqueous immersion.
- Use of a non-sticky sacrificial layer, such as teflon or gold.

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Gold Sacrificial Layer for Releasing Annealed Films

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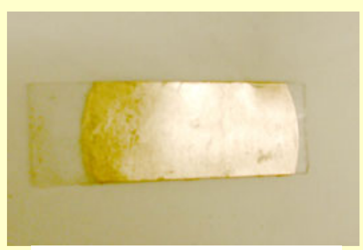
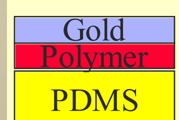
Gold on Si Wafer



PDMS/Gold/Polymer



Gold Etchant

Light Scattering

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Annealed Films: Crystallinity Gradient

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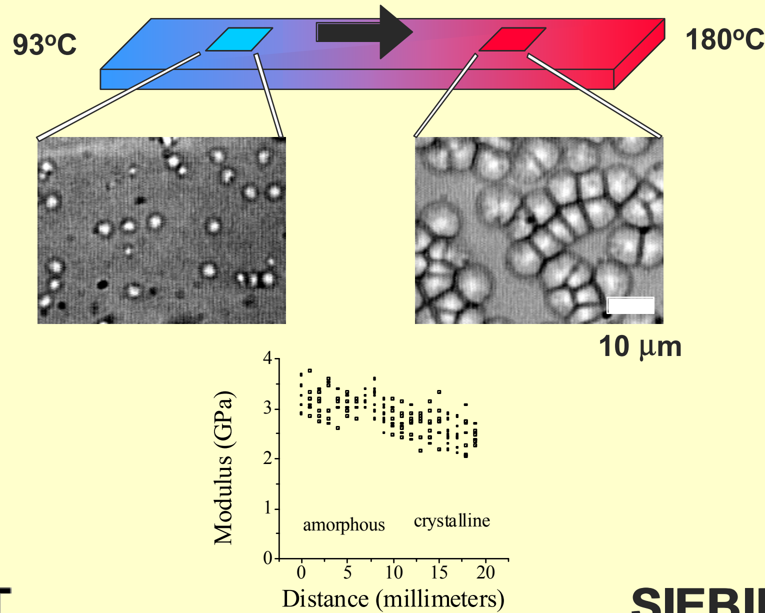

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Effect of Strain on Buckling Amplitude

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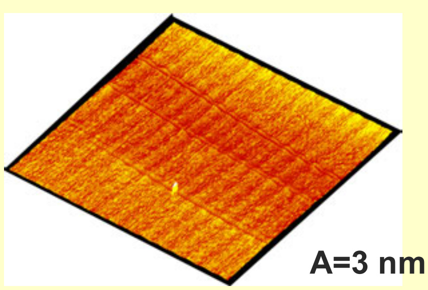

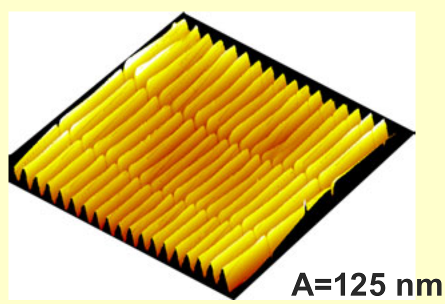

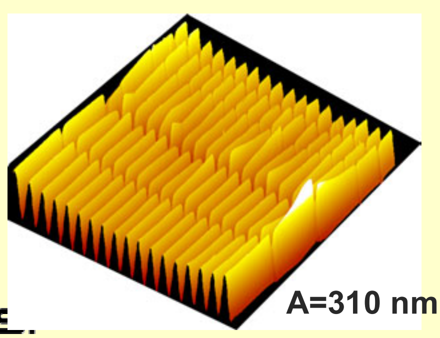

- Buckling imaged by contact mode AFM, 100 μm scans.
- Strain increases buckling amplitude A without strongly changing periodicity.

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Effect of Strain on Diffraction Pattern

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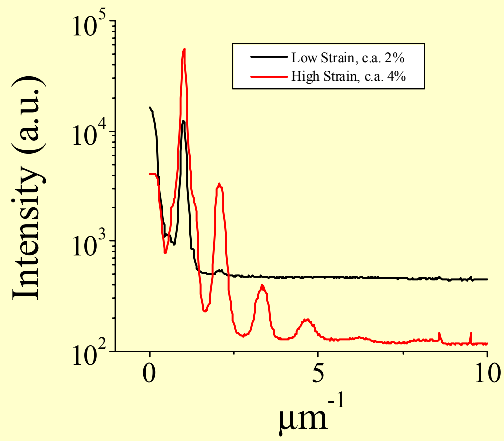
- Buckling measured by small angle light scattering.
- Scattering intensity increases with strain, higher order peaks emerge.
- Little shift in peak position with strain.



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Diffraction Gratings: Classic Approach

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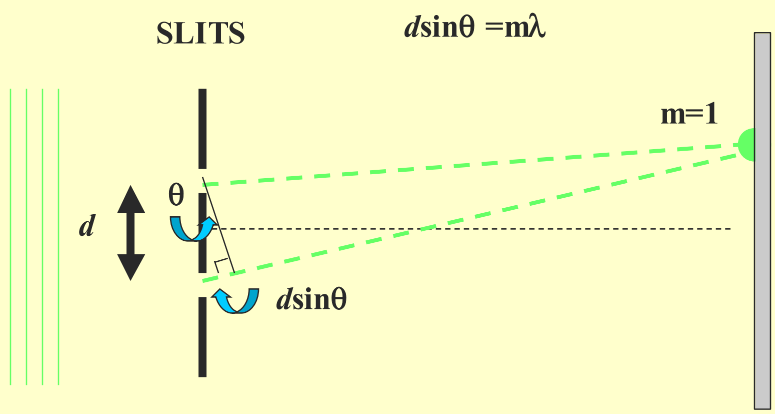

- Illumination of grating by coherent illumination produces diffraction peaks
- Optical path difference for neighboring sources differs by multiple of λ

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Page 1055 HRW must be coherent source

Light Scattering by Sinusoidal Phase Grating

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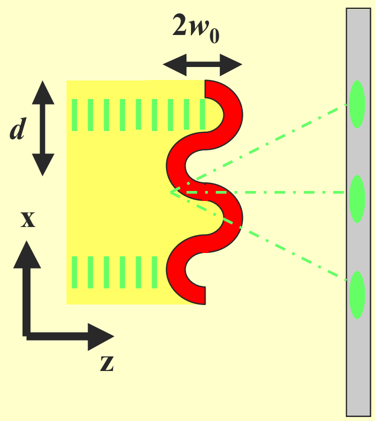
$$w = w_0 \hat{z} \sin\left(\frac{2\pi}{d} x\right)$$

- Phase shift imparted on coherent light wave varies sinusoidally with position..
- Relative phase shift is $\Phi = +/- 2\pi(nw_0/\lambda)$



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Effect of Strain on Buckling Amplitude

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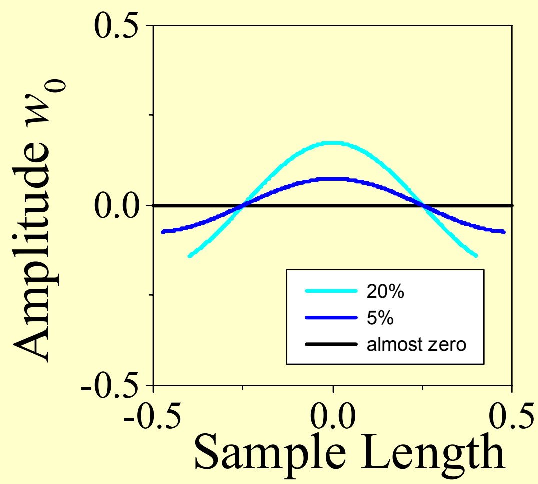
- Amplitude w_0 depends non-linearly on lateral strain
- Wavenumber fixed
- Reversible, tunable amplitude



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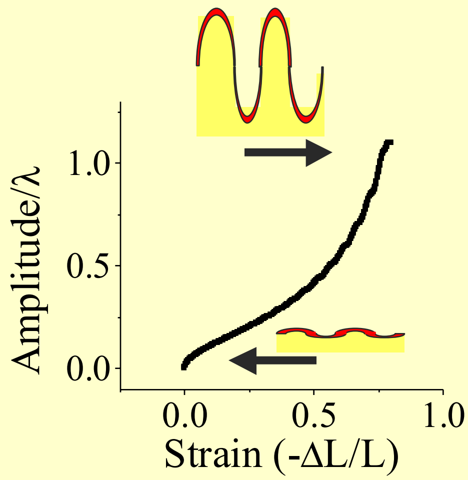
Buckling Amplitude as a Function of Strain

- Buckling amplitude depends non-linearly on strain.
- Calculation: numerical integration assuming pathlength is constant.



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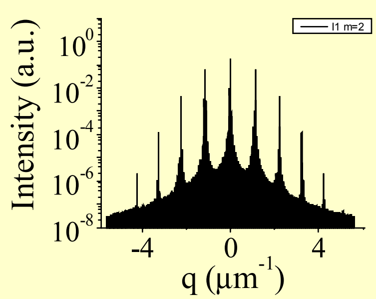
Diffraction Pattern of Sinusoidal Phase Grating

In the Fraunhofer limit we are very, very far away, typically *one kilometer*. However, it is pedagogically useful. The calculated diffraction intensity is:

$$I(x,y)=\left(\frac{A}{\lambda z}\right)^2\sum_{q=-\infty}^{q=\infty}J_q^2\left(\frac{m}{2}\right)\mathrm{sinc}^2\left[\frac{2w}{\lambda z}(x-qf_0\lambda z)\right]\mathrm{sinc}^2\left[\frac{2wy}{\lambda z}\right]$$



Purely sinusoidal phase grating produces many higher order peaks

$m \sim w_0$

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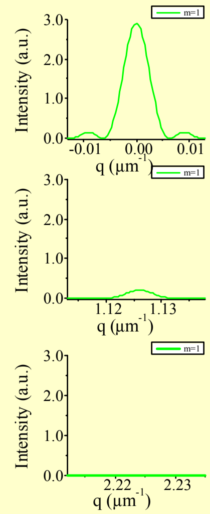
Understanding the Diffraction Pattern

$$I(x,y) = J_0^2\left(\frac{m}{2}\right)\mathrm{sinc}^2\left[\frac{2w}{\lambda z}(x)\right]$$

$$+ J_1^2\left(\frac{m}{2}\right)\mathrm{sinc}^2\left[\frac{2w}{\lambda z}(x - f_0\lambda z)\right]$$

$$+ J_2^2\left(\frac{m}{2}\right)\mathrm{sinc}^2\left[\frac{2w}{\lambda z}(x - 2f_0\lambda z)\right]$$

$+ \ldots\ldots$ **m ~ amplitude w_0**



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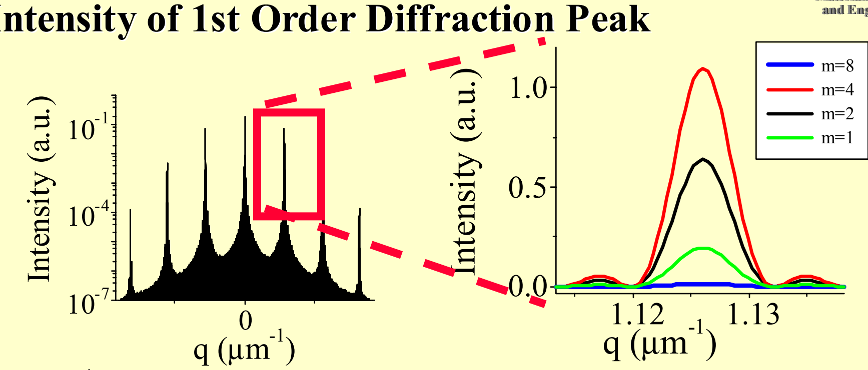
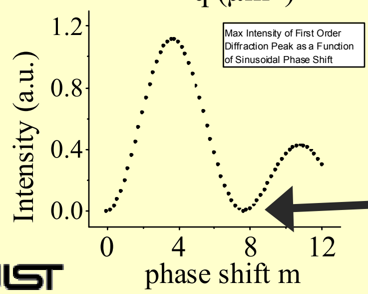
Intensity of 1st Order Diffraction Peak





- $$m = 4\pi \frac{nw_0}{\lambda}$$
- Condition met for w_0=200 nm, d=6 µm, Strain 1%.

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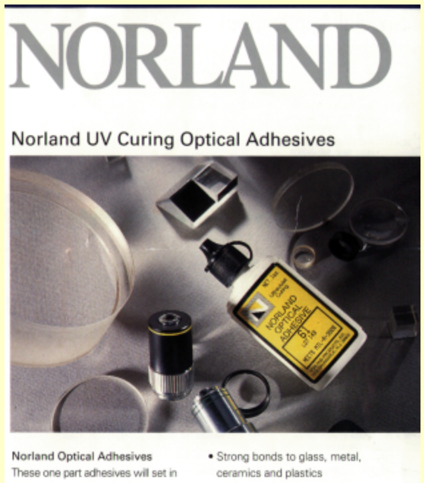
Formulation: Norland UV Curable Adhesive



Norland Optical Adhesive (NOA) and Special Applications

1 psi = 7142 Pa

Type	Description	Cure	Adhesion to: Glass	Metal	Plastic	Color	Typical Properties: Viscosity at 25 Degrees C	Refractive Index	Modulus PSI	Tensile PSI	Elongation at Failure
NOA 60	General purpose adhesive for bonding doublets, prisms or mounting components.	UV	Good	Good	Fair	Clear	300 CPS	1.56	135,000 1 GPa	2,800	35%
NOA 61	Preferred adhesive for military optics. Meets MIL-A-3920. Used for optics exposed to temperature extremes. Low shrinkage.	UV	Excellent	Excellent	Fair	Clear	300 CPS	1.56	150,000	3,000	38%
NOA 63	Cures well in thick sections. Use as fillet bond to hold lenses in place or for bonding where low fluorescence or good transmission in UV range is required.	UV	Good	Good	Fair	Clear	2,500 CPS	1.56	240,000	5,000	6%
NOA 65	Flexible adhesive suitable for low strain applications or for cold blocking of lenses.	UV	Good	Good	Fair	Clear	1,200 CPS	1.52	20,000	1,500	80%
NOA 68	Flexible adhesive for glass or plastics such as polycarbonate, CAB or acrylic.	UV	Excellent	Good	Good to Excellent	Clear	5,000 CPS	1.54	20,000	2,500	80%
NOA 71	Provides a strong bond to glass surfaces and has excellent clarity for light guides and other applications.	UV	Excellent	Excellent	Fair	Clear	200 CPS	1.56	55,000	1,300	43%
NOA 72	Low viscosity adhesive for bonding glass or plastics such as polycarbonate, CAB or acrylic. Cures with UV or visible light.	UV/VIS	Excellent	Good	Good to Excellent	Clear	175 CPS	1.56	2,400	500	34%
NOA 73	Flexible adhesive with low viscosity for bonding delicate parts.	UV	Excellent	Good	Fair	Clear	140 CPS	1.56	1,600	200	16%
NOA 76	High viscosity adhesive for bonding glass to plastic. Cures with UV or visible light.	UV/VIS	Excellent	Good	Excellent	Clear	4,500 CPS	1.51	970	450	47%
NOA 77	High viscosity adhesive for bonding plastic to plastic. Cures with UV or visible light.	UV/VIS	Good	Good	Excellent	Clear	5,000 CPS	1.51	900	570	63%
NOA 81	Fast curing adhesive for tacking or bonding. Excellent adhesion to glass or metal. Produces a hard, resilient bond.	UV	Excellent	Excellent	Fair	Clear	300 CPS	1.56	200,000	3,000	25%
NOA 83H	Fast curing adhesive that will cure with UV or heat for tacking or bonding UV opaque materials.	UV/HEAT	Excellent	Excellent	Fair	Clear	250 CPS	1.56	160,000	3,500	30%
NOA 88	Low outgassing adhesive for aerospace or electronic applications. Excellent transmission in UV range.	UV	Excellent	Excellent	Fair	Clear	250 CPS	1.56	112,000	1,800	40%
UVS 91	Screenable paste for bonding and sealing perimeters of liquid crystal displays.	UV	Excellent	Good	Good to Excellent	Translucent	Thixotropic Paste	N/A	45,000	2,900	58%
NBA 106	UV curing adhesive for cold blocking and temporary bonding of lenses to metal polishing mounts.	UV	Excellent	Excellent	Fair	Clear	350 CPS	N/A	N/A	N/A	N/A

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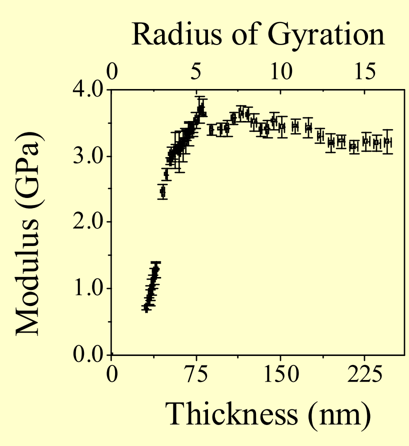
Thickness Effects: Preliminary Data

- Polymer film dimensions comparable to Rg are increasing important as lithographic features are decreased.
- Films below 5 Rg exhibit a lower modulus.



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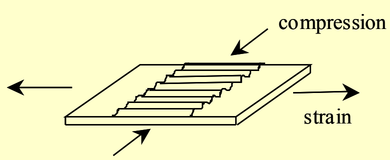
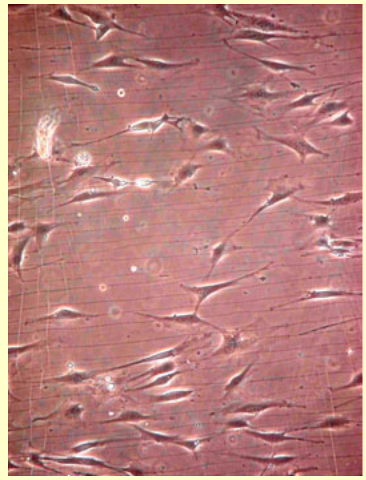
Applications to Biology

- Corrugations of polystyrene film induce a preferred direction in cells.
- Why care?
  We can introduce the corrugations in an ***oscillatory*** fashion
- Template for papillae between dermal layers





Amit Sehgal's Cells

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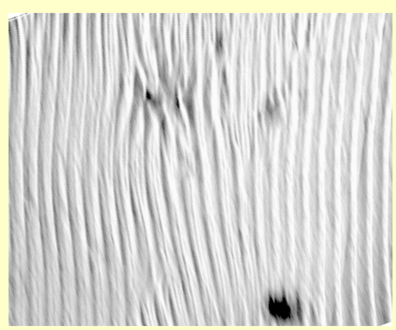
Application to Formulations.

- This technique applies to colloidal dispersions of polyurethane particles (Bayer Bayrol 123)
- Optical Image of Buckling on PDMS.



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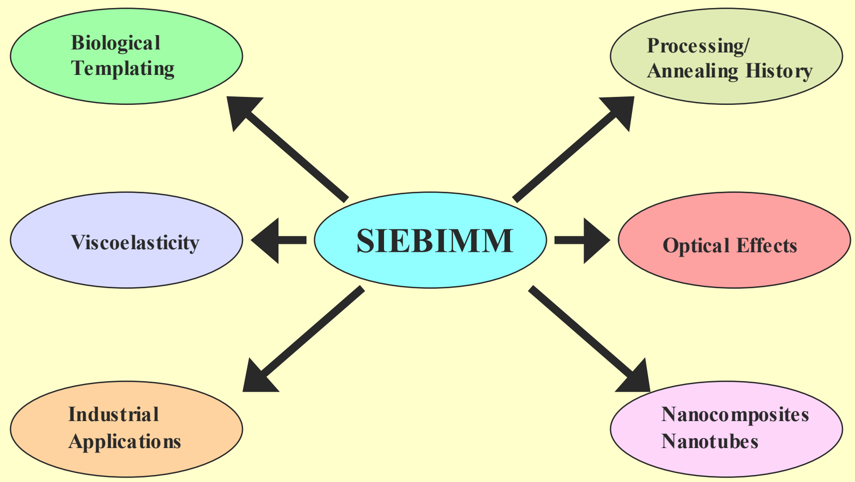
Future Directions



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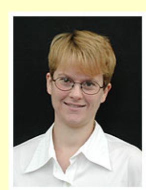
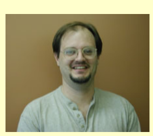
Participants: Team SIEBIMM

Christopher Stafford

Christopher Harrison



Kate Beers



Forrest Landis

Sammy Hong

Plus: Managers who have indulged our curiosity…

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