Carbon Deposition by Film Delamination and X-ray Mirror Curvature Control

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Current IRES $150K (with overhead)

3-4 summers, 4-5 students per summer ($2,000/months + airline ticket)

Started in 2010. Grant supports travel and allowance

What Mechanical Engineering students skills are helpful?

- CAD/Design
- Data acquisition
- Mechanics of Materials
- FEM
- Thermodynamics/Heat transfer
- Fluid Mechanics
- Vibrations
- Robotics

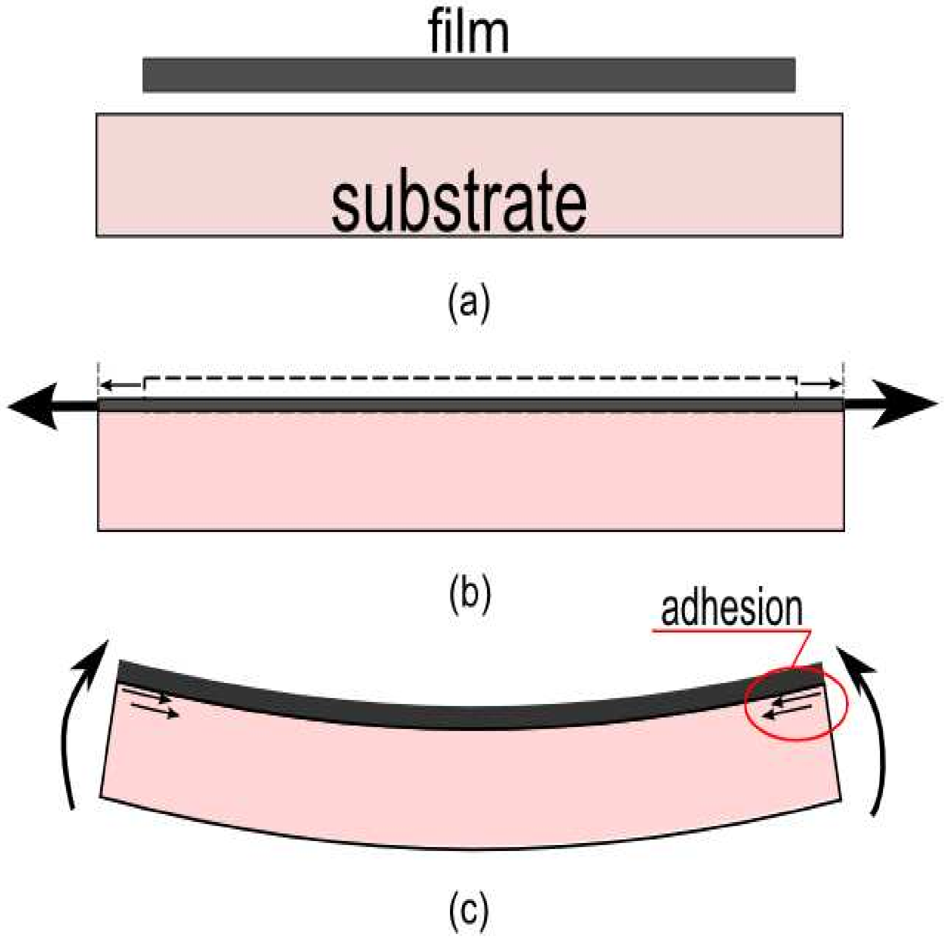
Outline

- X-ray optics stability and control, temperature and strain distribution simulations
- Stress relief effects in thin films and multilayers
- Electrowetting experiments

Thin film residual stress

- ➢ Thermal
- ➢ Intrinsic
- ➢ Epitaxial

} Total residual stress



<u>Consequences</u>

- New equilibrium state
- Failure
- Promotes diffusion

<u>Length scales of stress</u>

- Microscopic
- Macroscopic

Measuring residual stress

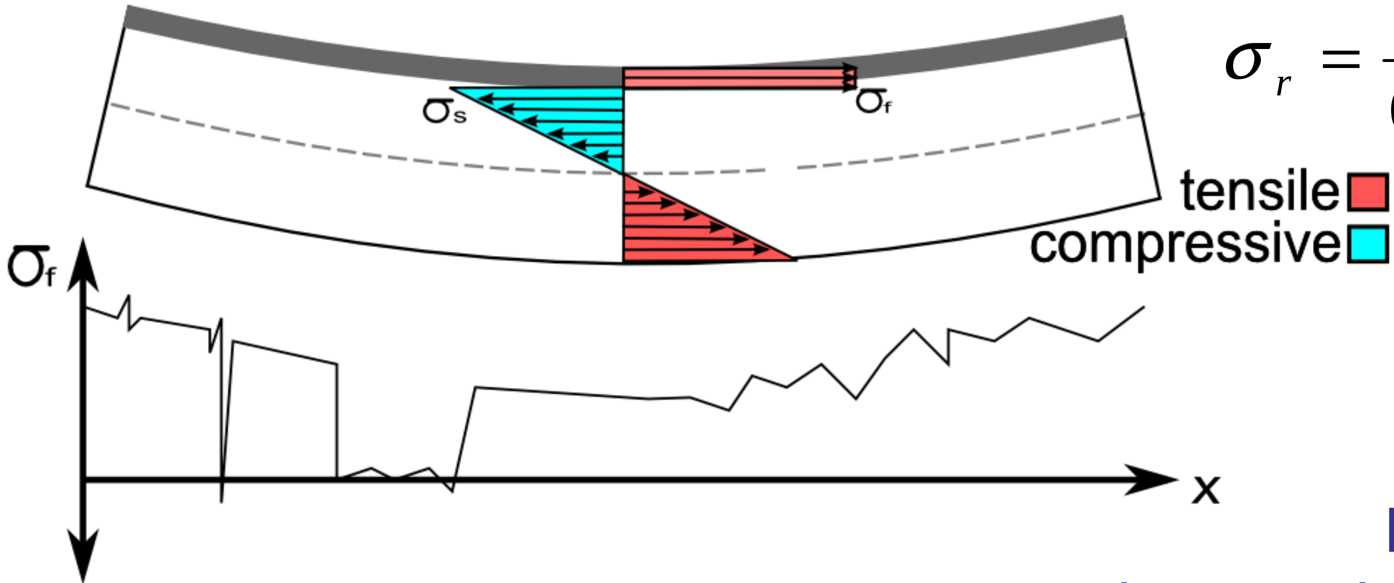
Film material strain

- X-ray Diffraction
  - Bragg's law
- Raman spectroscopy

Substrate curvature

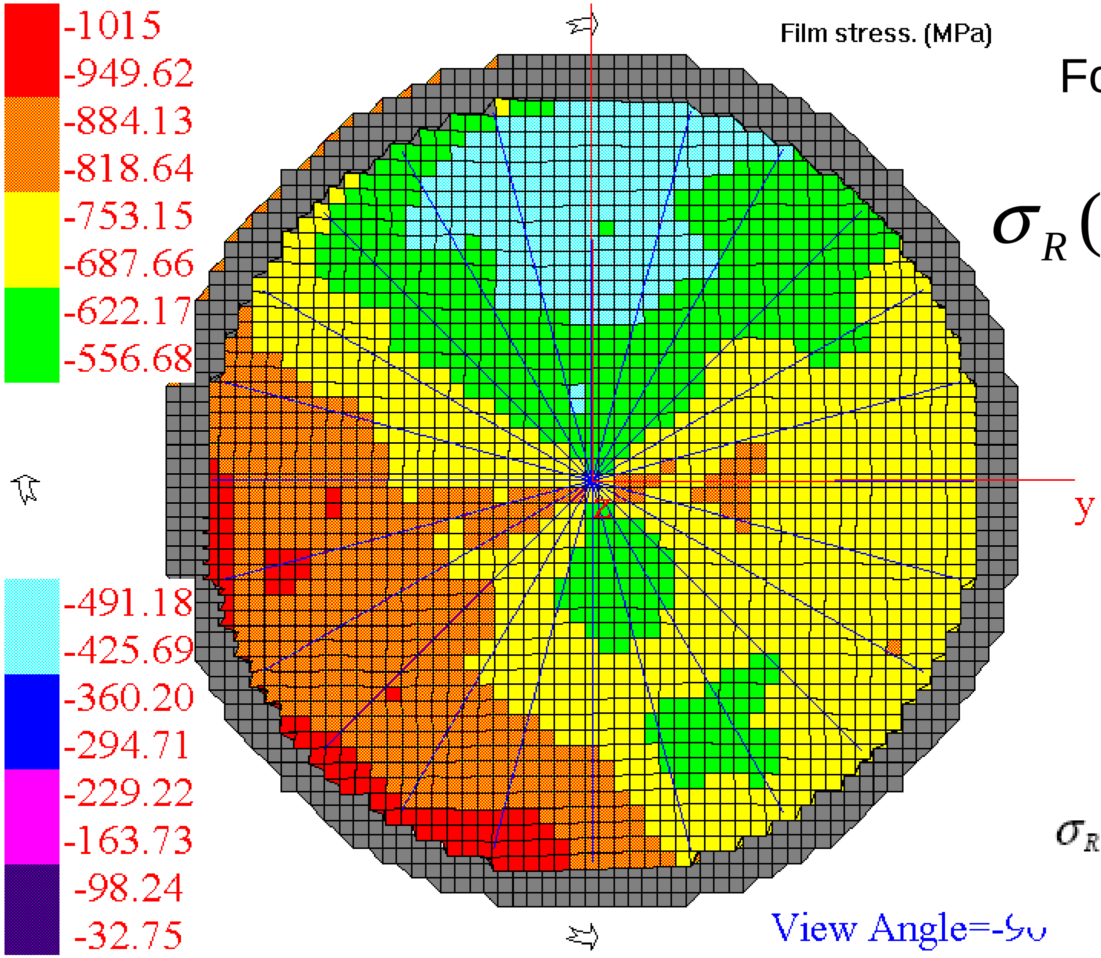
- Optical interferometry
- Optical profiling
- Mechanical profiling

$$\sigma_r = \frac{E_s \cdot t_s^{\,2} \cdot \Delta k}{6 \cdot (1 - \nu_s)^2 \cdot t_f}$$



Macroscopic vs.
Microscopic measurements

Modified Stoney Formulas



TiWN film on 6" Si wafer

For non-uniform film thickness

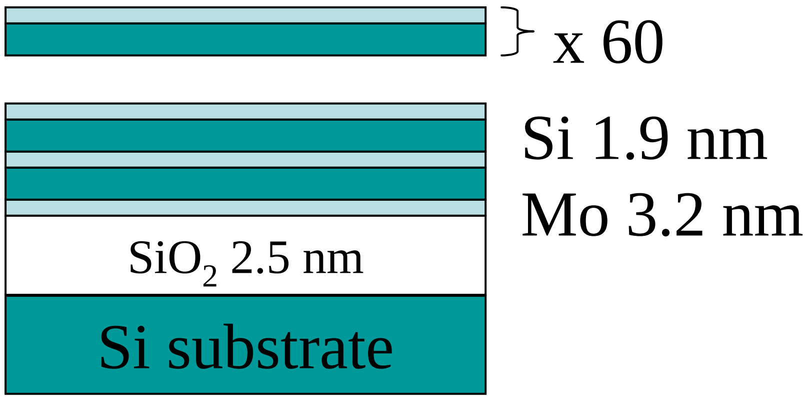
$$\sigma_R(r) = \frac{E_s}{1-\nu_s}\frac{\Delta k(r) h_s^2}{6h_f(r)}c(r)$$

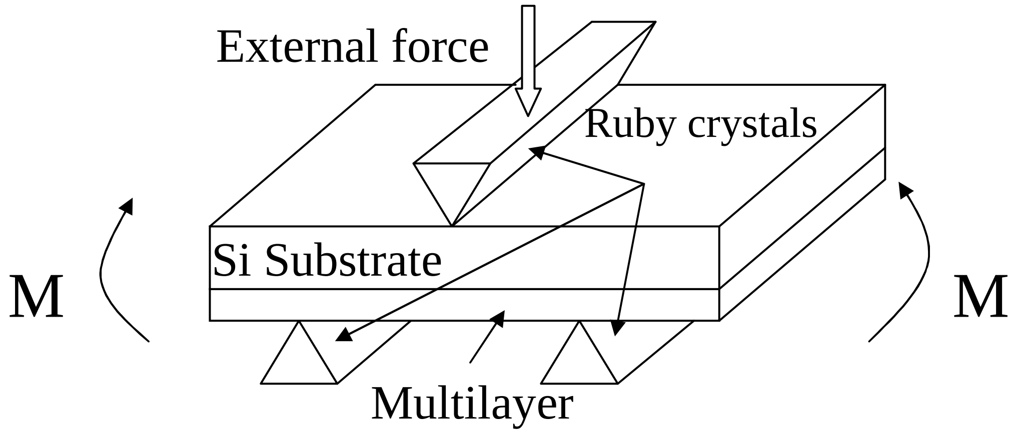
Residual Stress in CVD-grown 3C-SiC Films on Si Substrates, A.A. Volinsky, G. Kravchenko, P. Waters, J. Deva Reddy, C. Locke, C. Frewin, S.E. Saddow, Mat. Res. Soc. Symp. Proc. Vol. 1069, D3.5, 2008

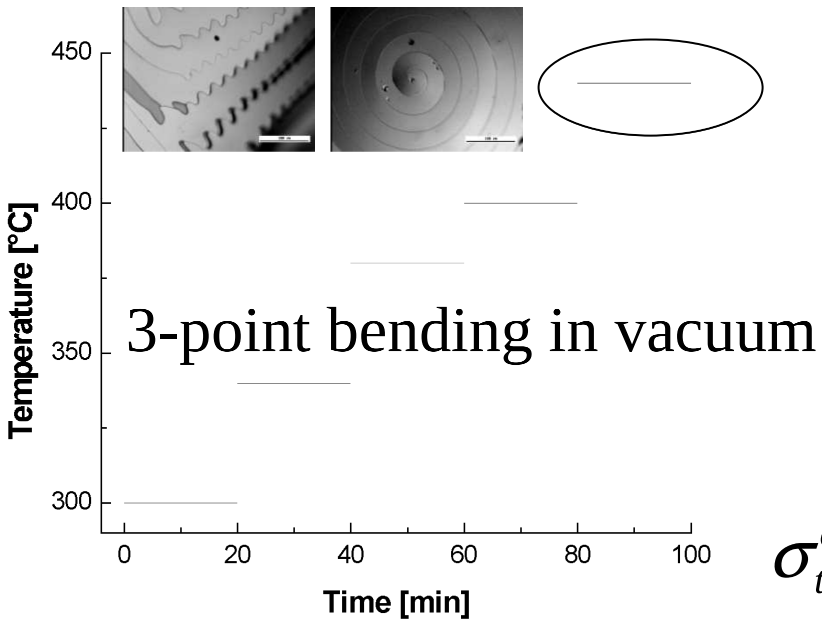
2D case:

$$\sigma_R = \frac{E_s}{1-\nu_s}\frac{h_s^2}{6h_f R_1}\cdot\left[1+\left[\frac{\nu}{1+\nu}\right]\cdot\left[\frac{R_1}{R_2}-1\right]\right]$$

Mo/Si Mirror Bending Experiments







$$\sigma_r^{comp.} \approx 100\,MPa$$

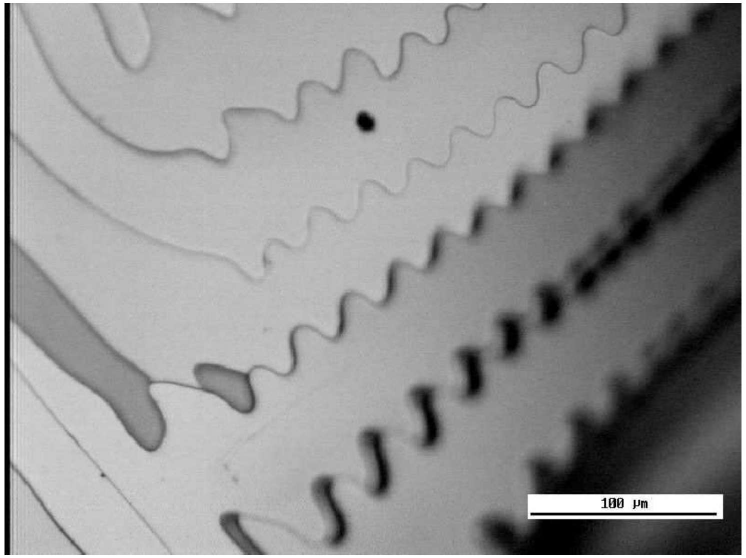
$$\sigma_T^{comp.} = \frac{\Delta\alpha\Delta T E_f}{1-\nu_f} = 460\,MPa$$

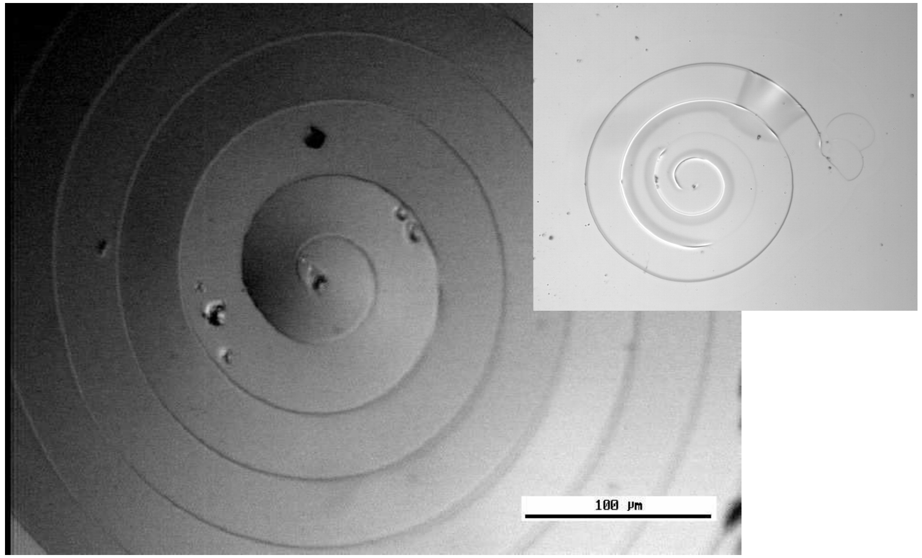
$$\sigma_{bend}^{tens.} = \frac{Mt}{2I} = 225\,MPa$$

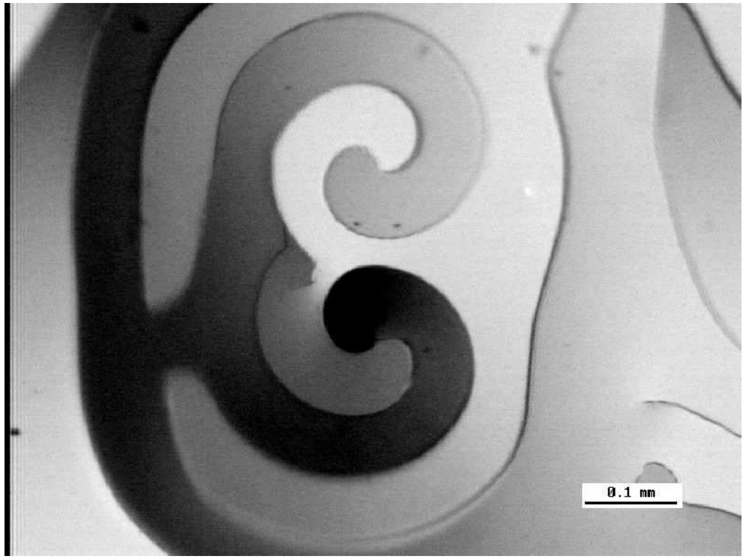
$$\sigma_{total}^{comp.} = \sigma_{residual} + \sigma_{bending} + \sigma_T = 335\,MPa$$

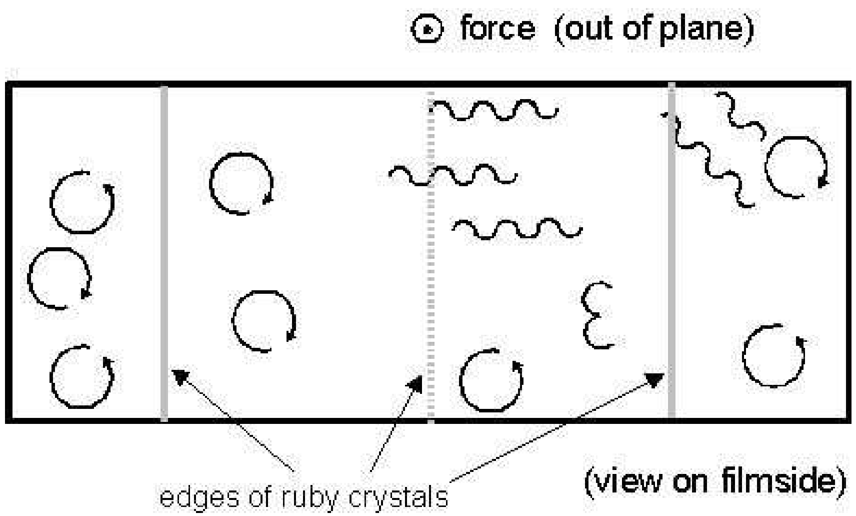
D.C. Meyer, T. Leisegang, A.A. Levin, P. Paufler, A.A. Volinsky, Appl. Phys. A 78, pp. 303-305, 2004

Tensile Crack Patterns: Mo/Si

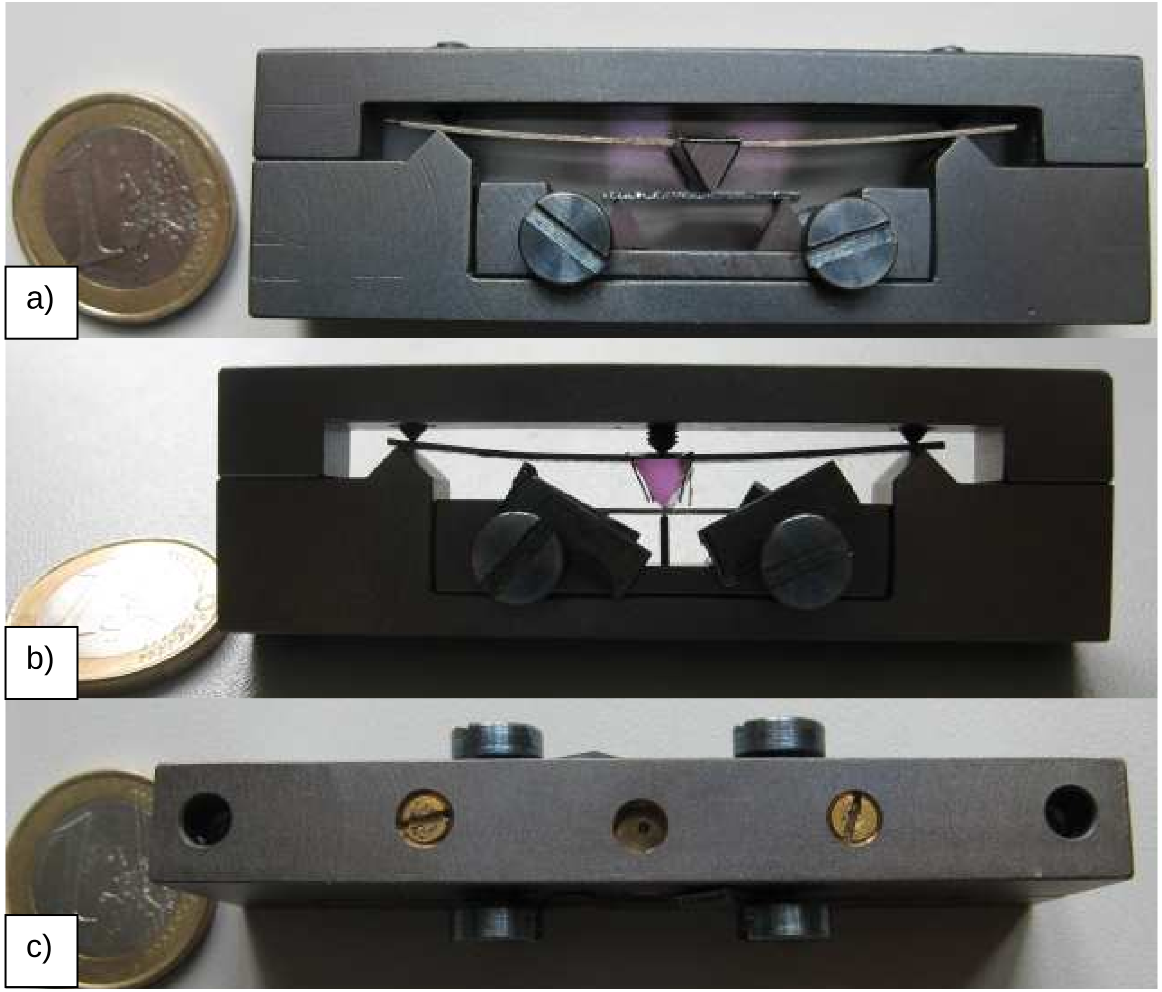








3-point Bending Fixture Improvement



Before modification

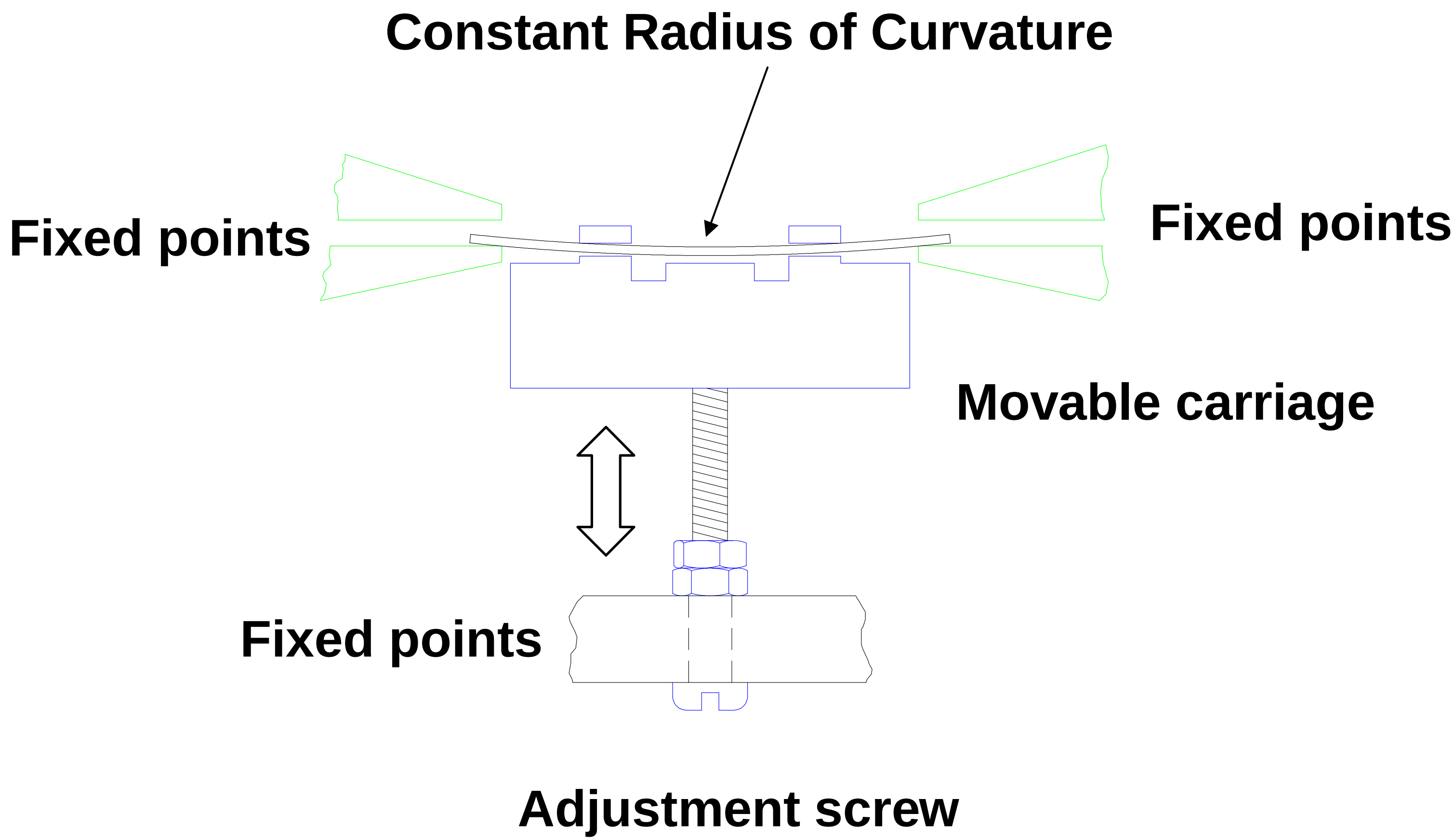
Modified fixture

80-100 μm beam displacement,

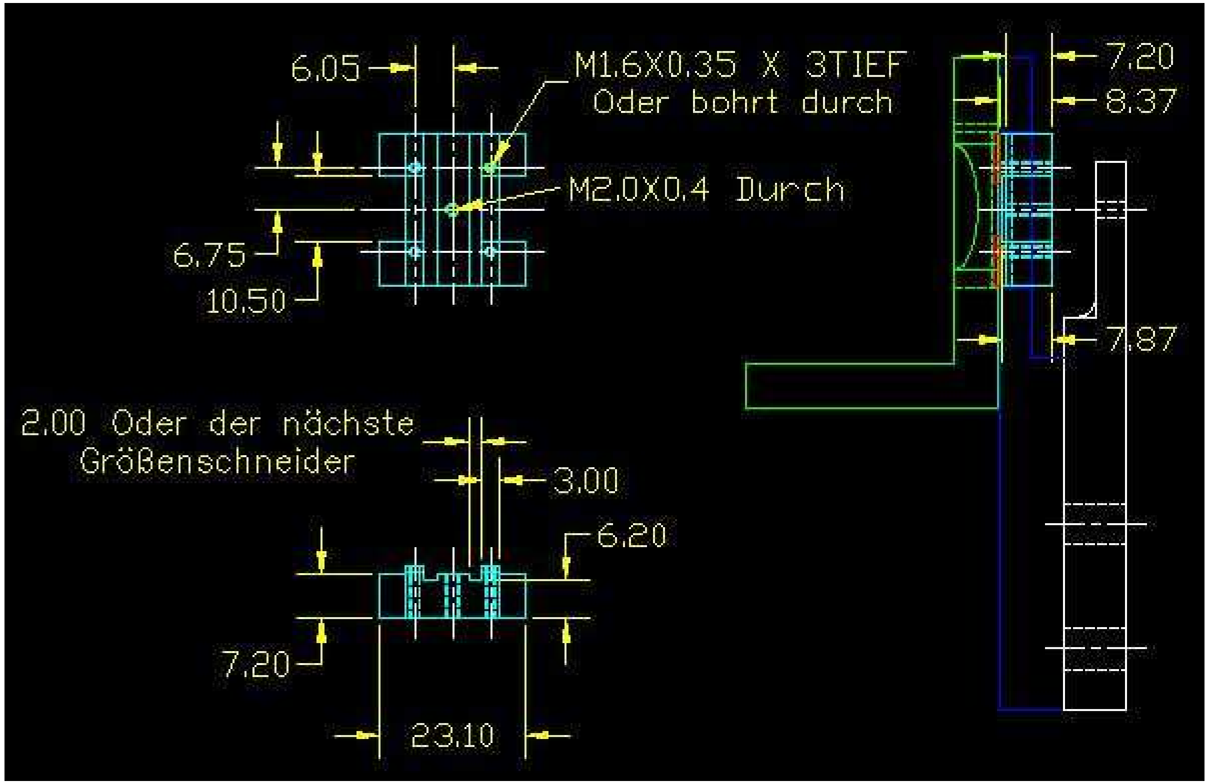
15-19 N Force,

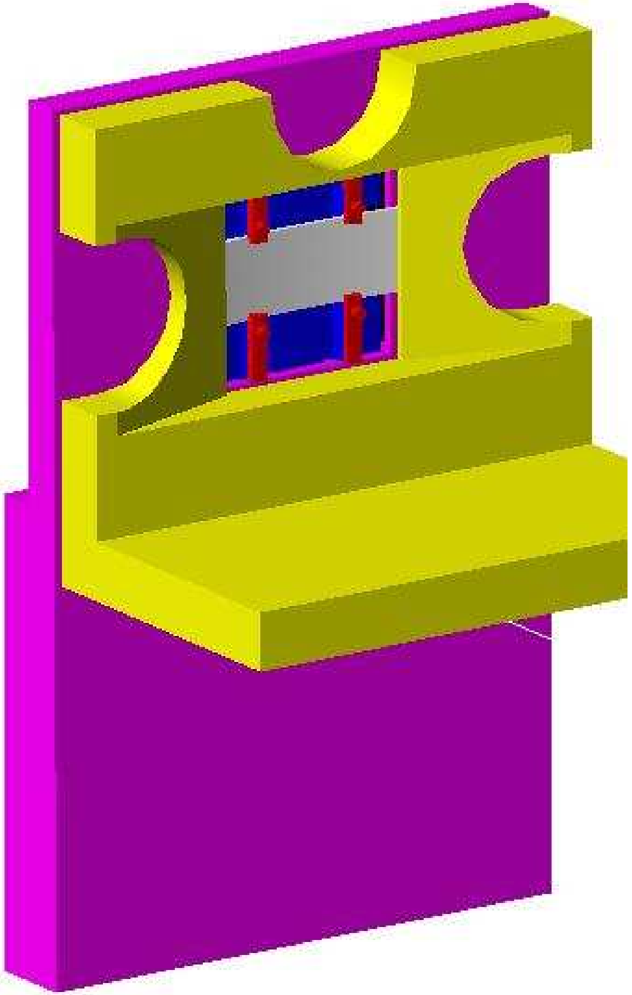
250-350 MPa max. normal stress due to bending

4-Point Bending Fixture



In-Situ 4-point Bending Fixture. Tension-Compression

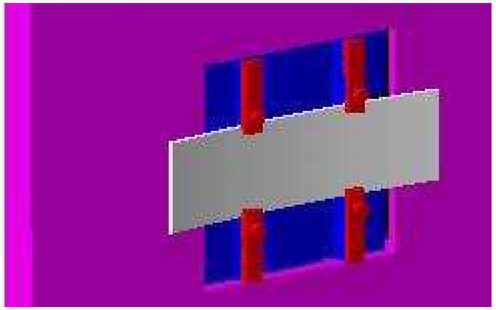




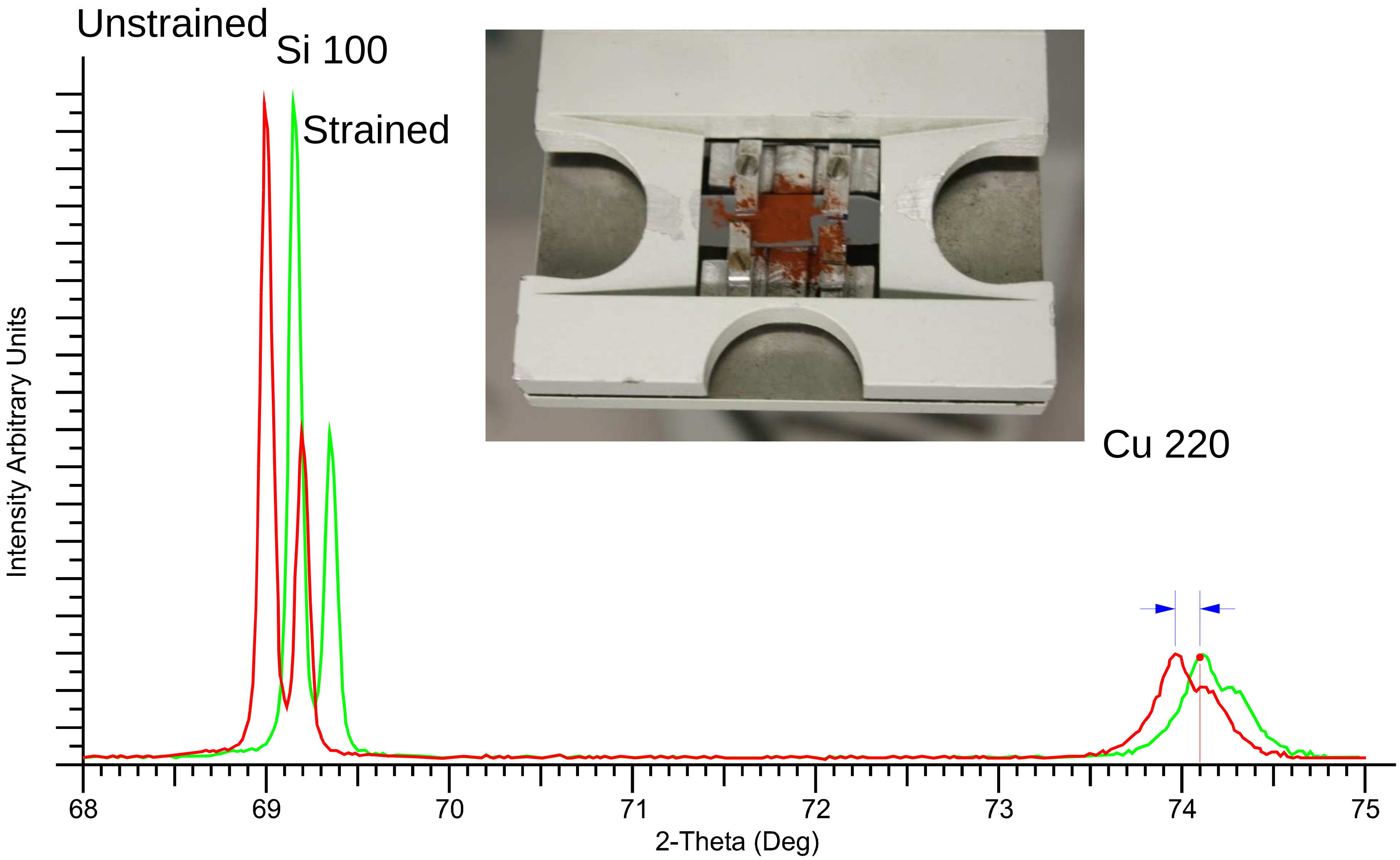
James Rachwal

5 days of AutoCAD work

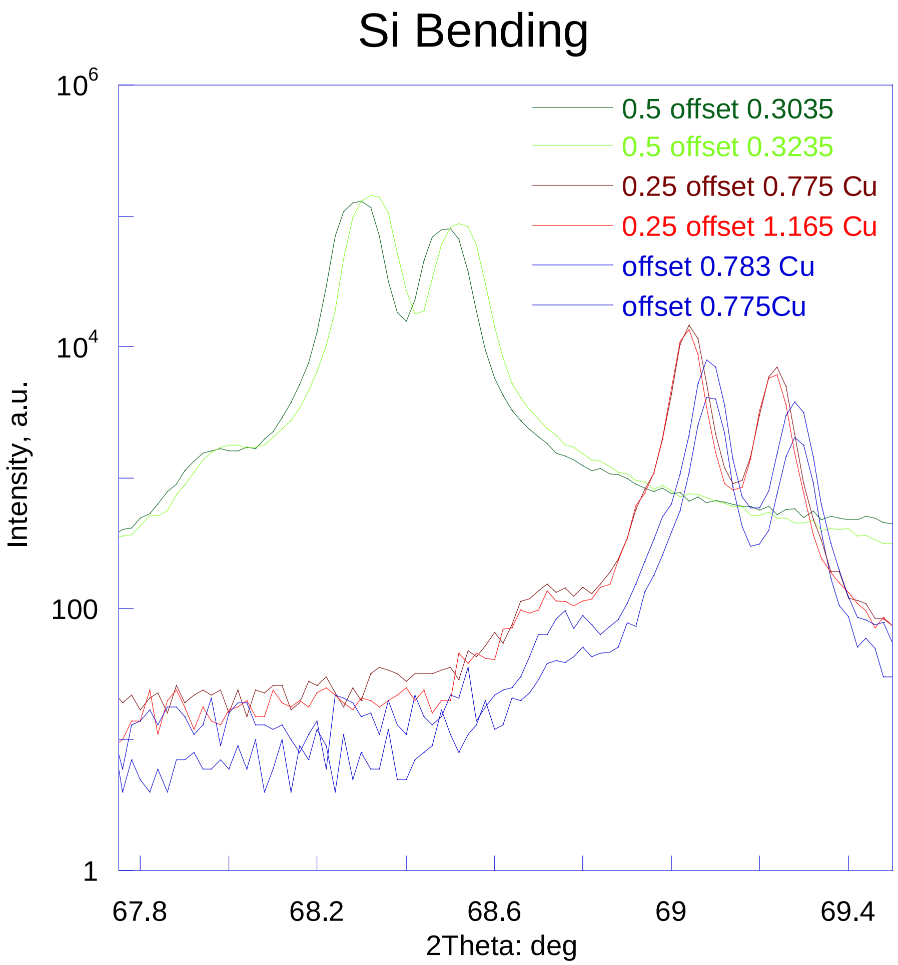
3 weeks machining

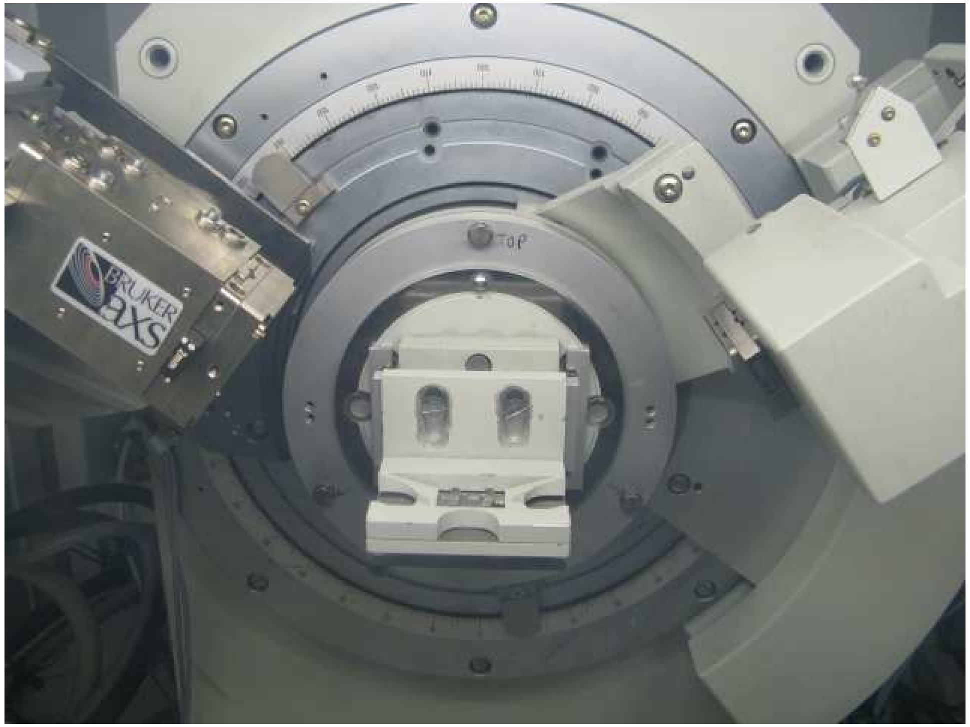


Copper Powder Corrections



Bending Fixture Preliminary Data

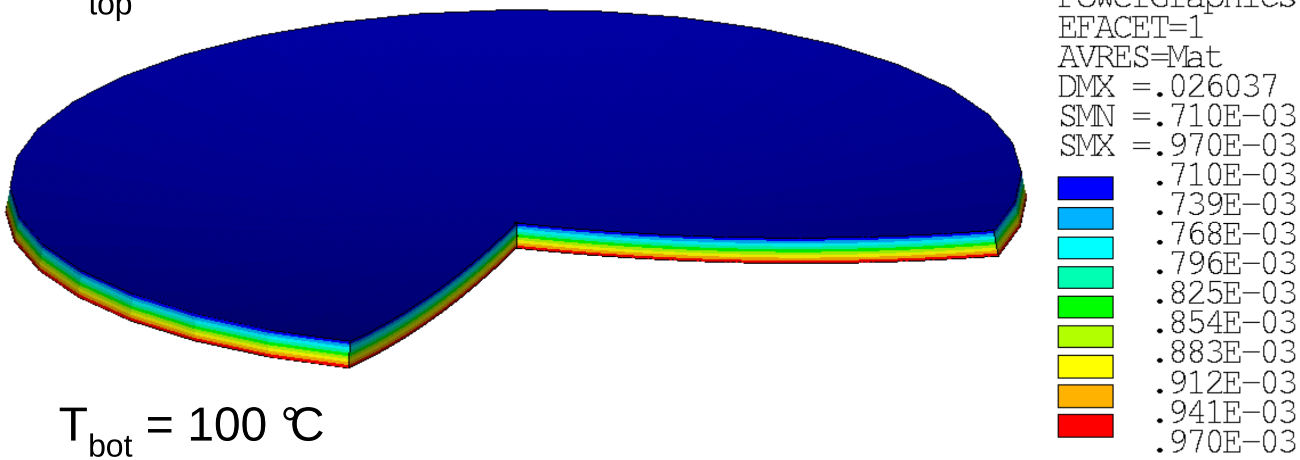




Curvature due to temperature gradient T_z

- Axisymmetric model of 0.5 mm Si substrate
- Steady-state
- No films, bare Si

T_{top} = 0 ℃



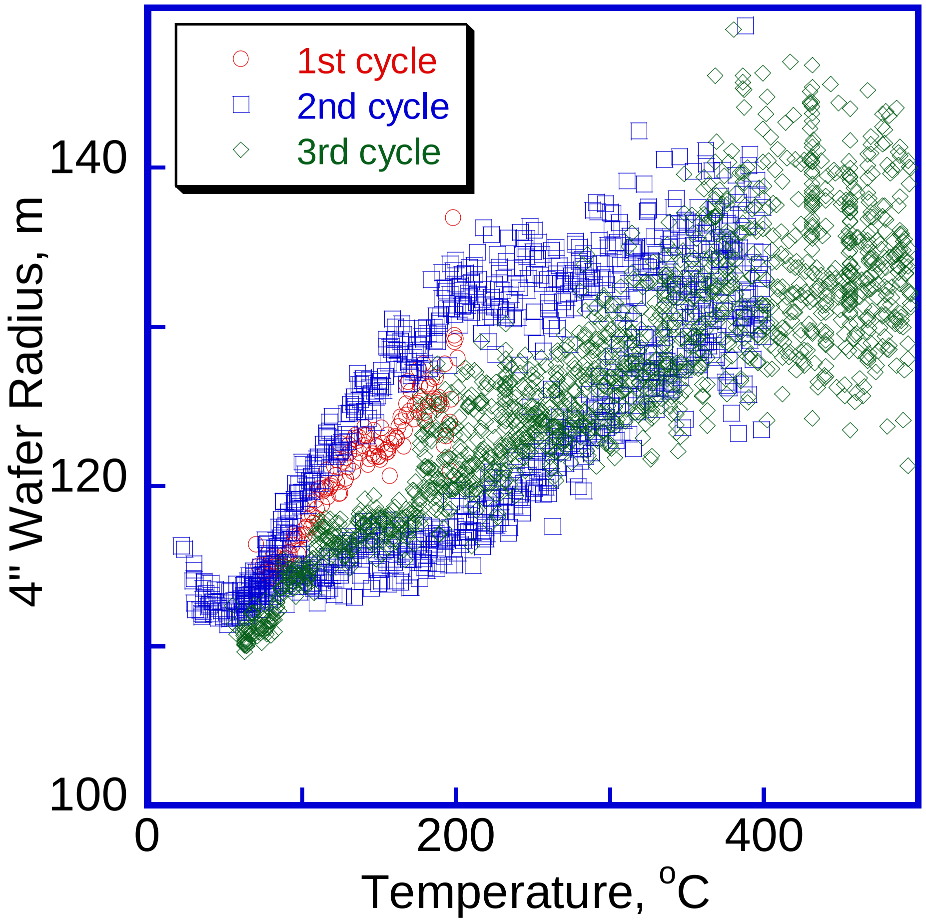
T_{bot} = 100 ℃

- Radius of curvature: R = 2 m

Grygoriy Kravchenko

Modeling GIGO problem

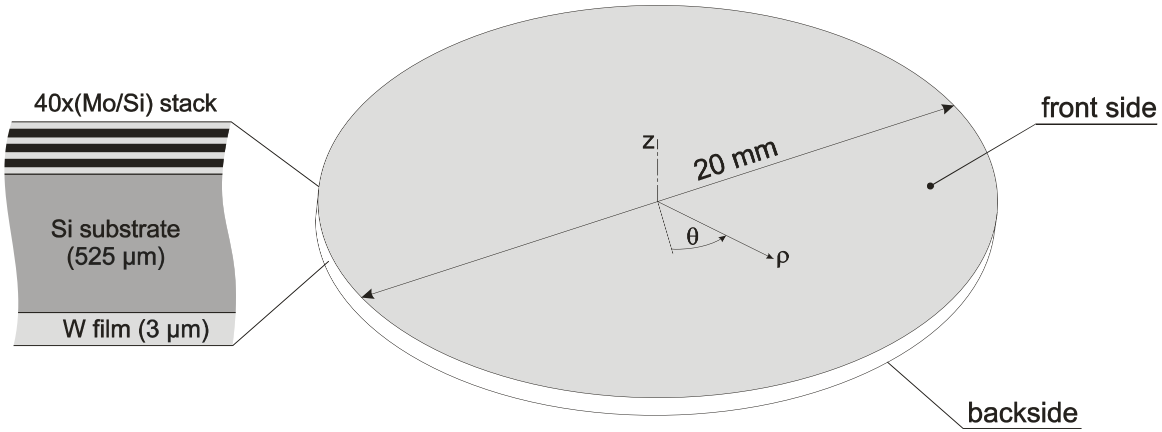
4” Si Wafer Uniform Heating



Uniform slow heating, wafer becomes flatter with T due to thin SiO_2 layer,

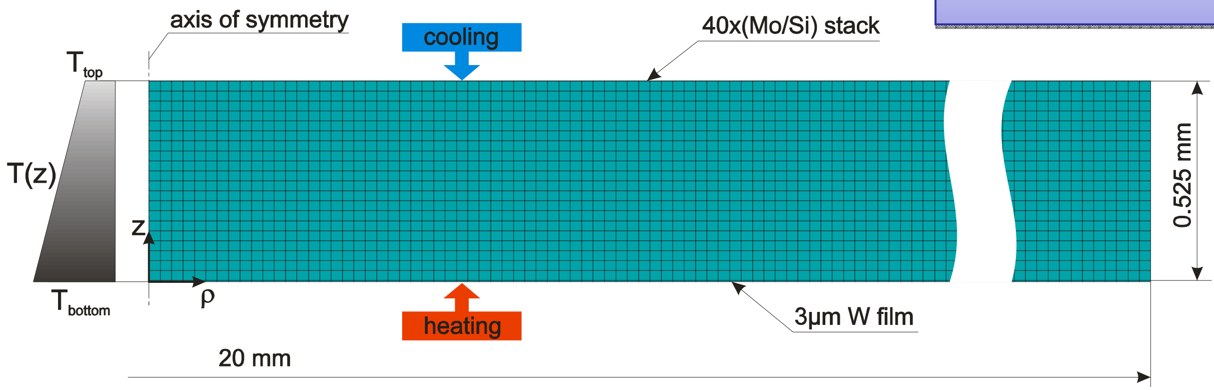
Similar to the simulation results

Temperature gradient with films



- Target radius of curvature:
  R = 10 m
- T_{upper} = 100 ℃

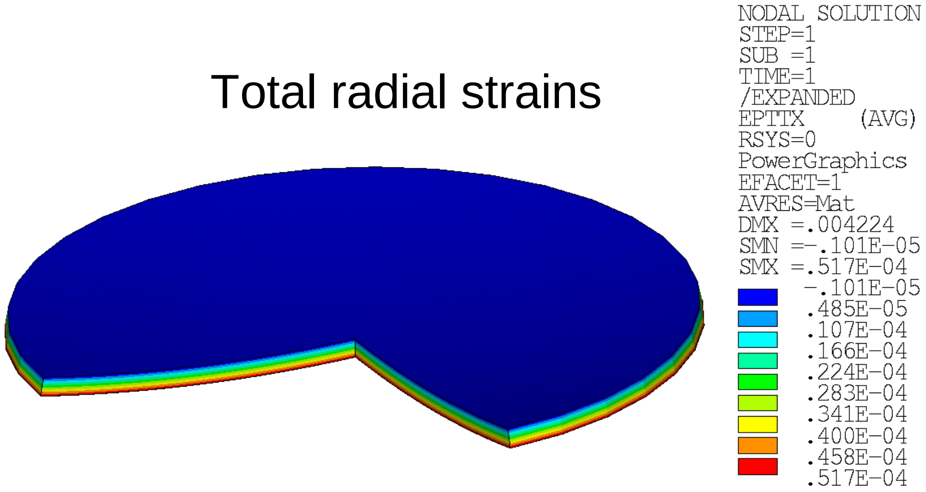
Thermally-induced curvature control?



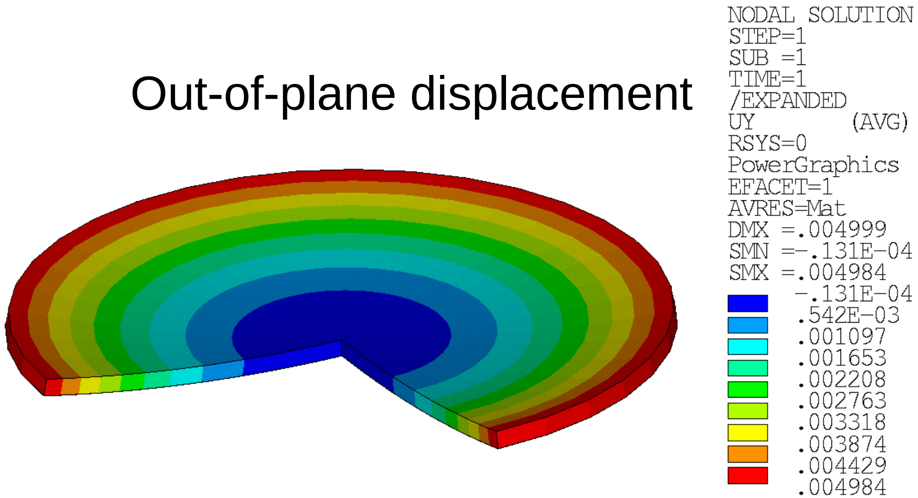
Axisymmetric finite element model

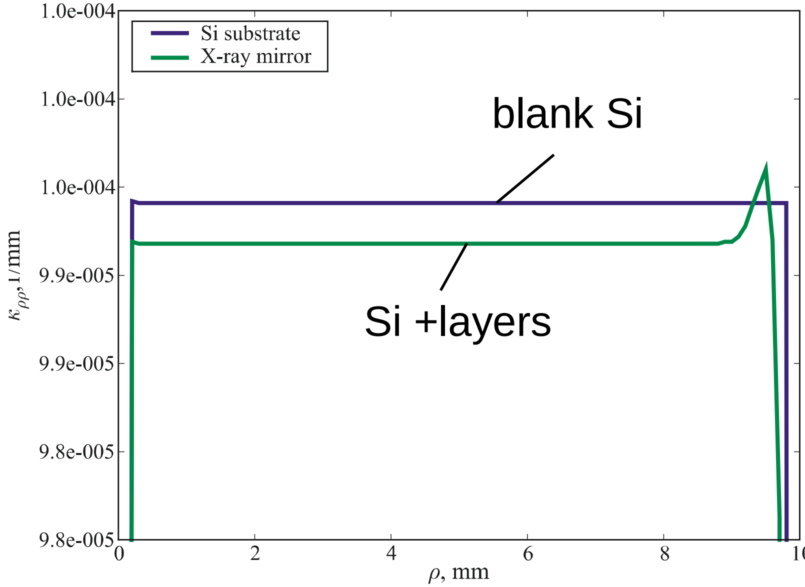
Temperature gradient ΔT_z = 20 ℃

Total radial strains



Out-of-plane displacement





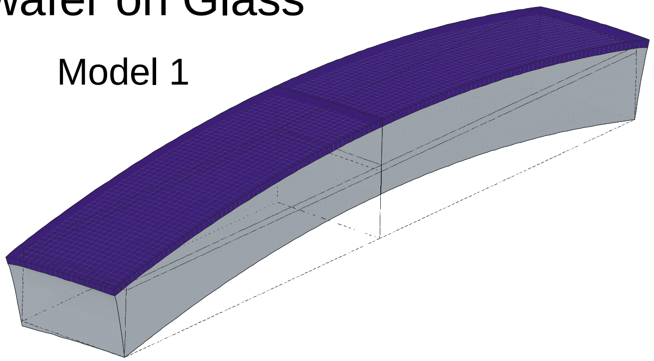
Curvature vs. radial position

- Curvature is uniform - spherical surface (small deformations)
- Influence of Mo/Si and W layers is small (thermal mismatch is negligible)

X-ray mirror deformed shape

Si wafer on Glass

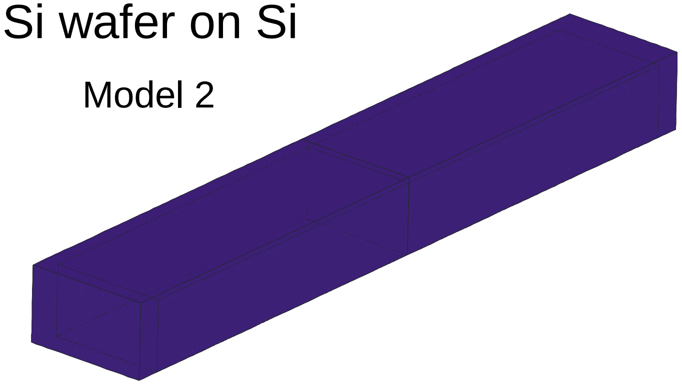
Model 1



largest thermal expansion mismatch

Si wafer on Si

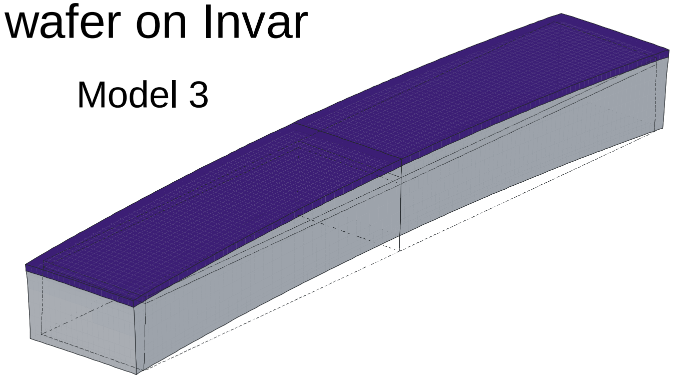
Model 2



no thermal expansion mismatch

Si wafer on Invar

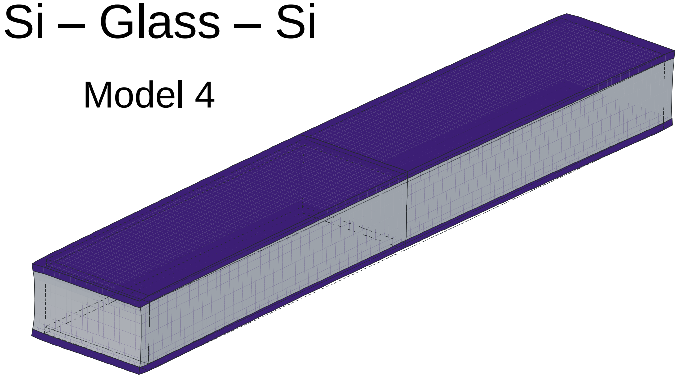
Model 3



smallest thermal expansion mismatch

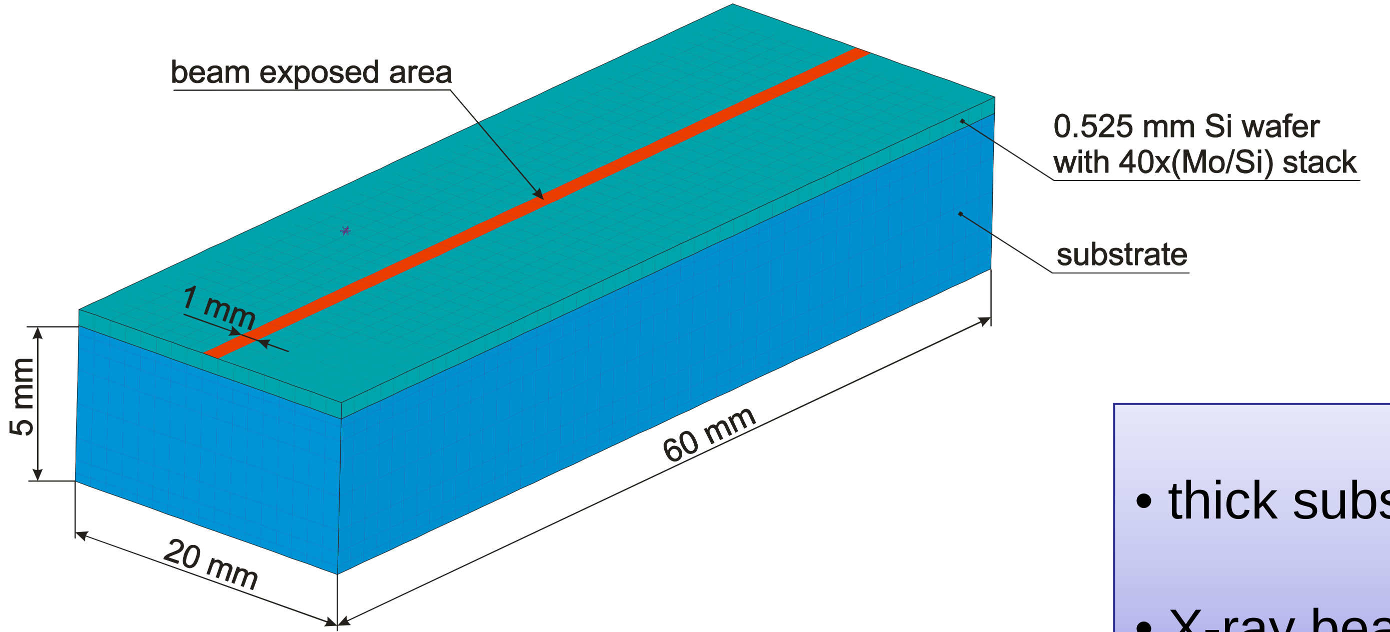
Si – Glass – Si

Model 4



compensated thermal expansion mismatch

X-ray mirror thermal deformations



- thick substrate
- X-ray beam: 1 W/cm^2

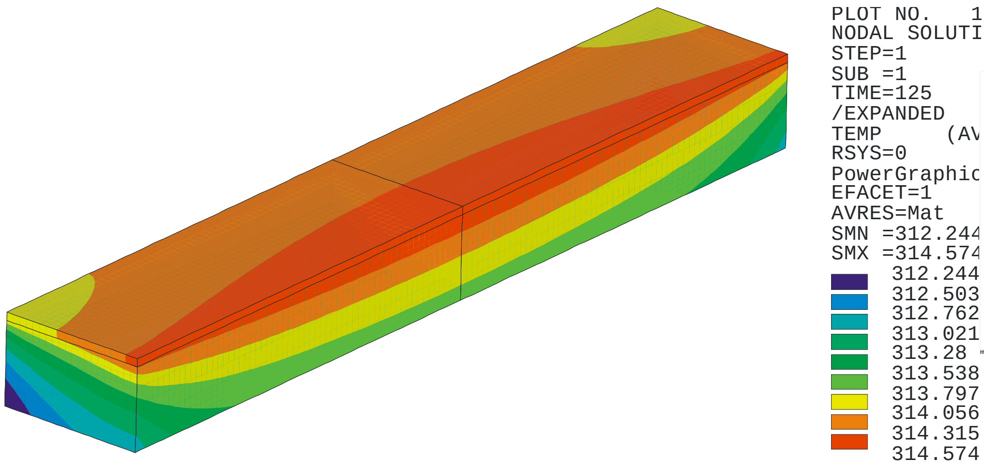
1. Solve the thermal problem
2. Solve the structural problem

Identify materials/geometry influence

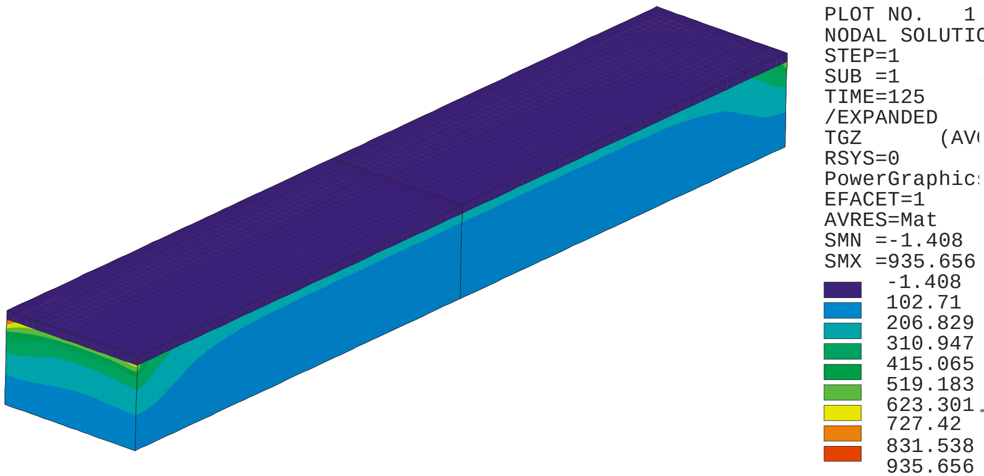
Model variations:

1. Si wafer on Glass
2. Si wafer on Si
3. Si wafer on Invar
4. Si wafer – Glass – Si wafer

Thermal results (Model 1)



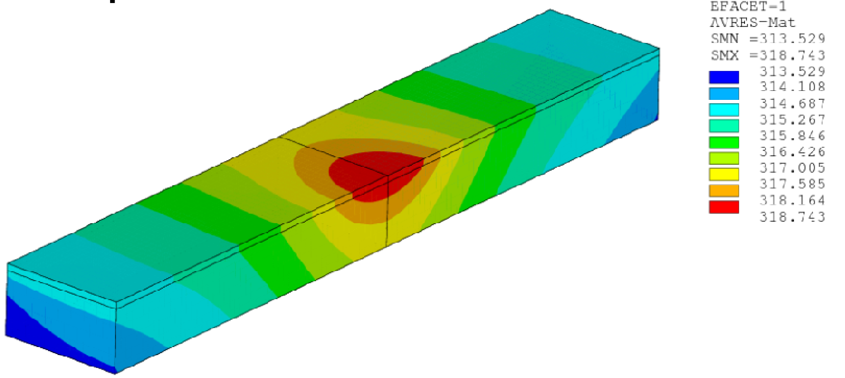
- temperature increases up to 15 ℃
- almost uniform temperature distribution



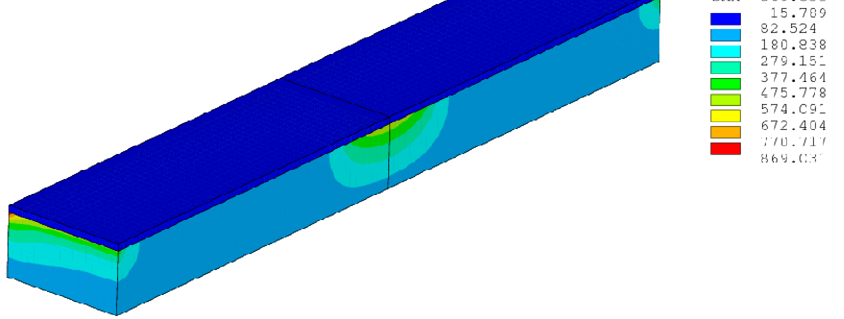
- very small temp. gradient ∇T_z in Si
- small ∇T_z in glass (<0.1 ℃/mm) (away from the material corners)

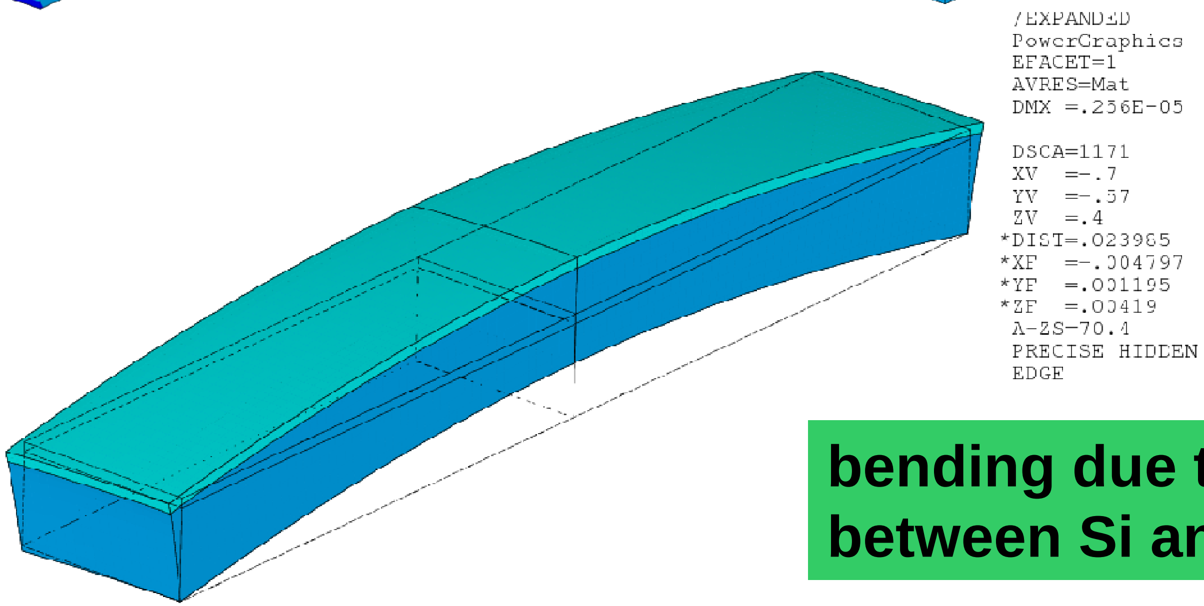
Si on Glass substrate, 1 W/cm^2

temperature field



temperature gradient, T_z

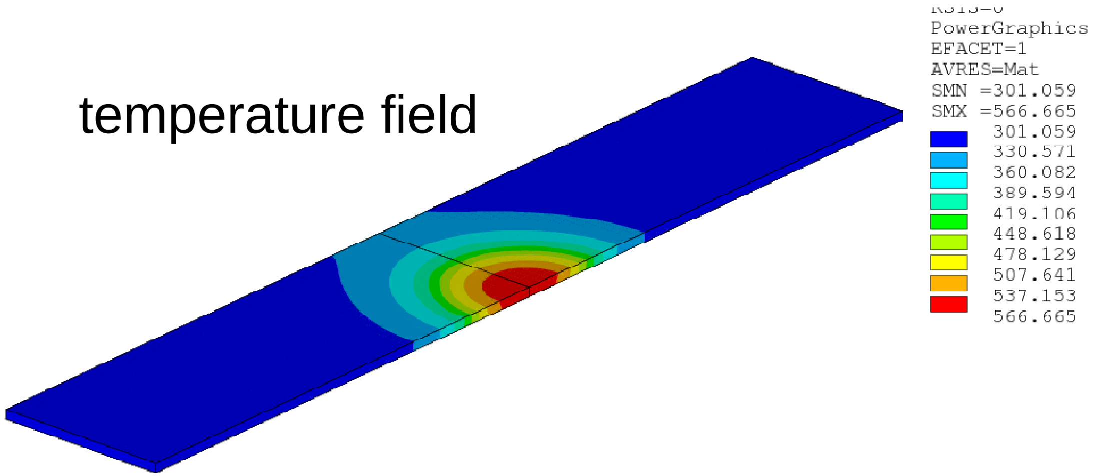


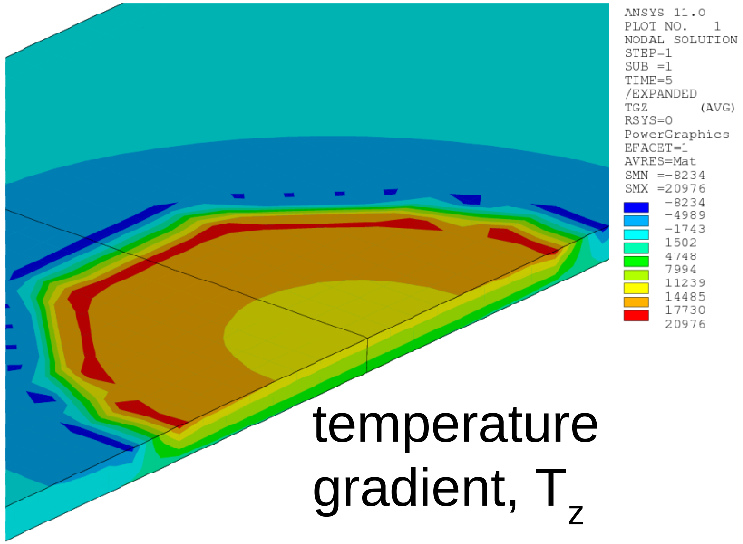


bending due to ΔCTE between Si and glass substrate

Si, 300 W/cm^2 (BESSY)

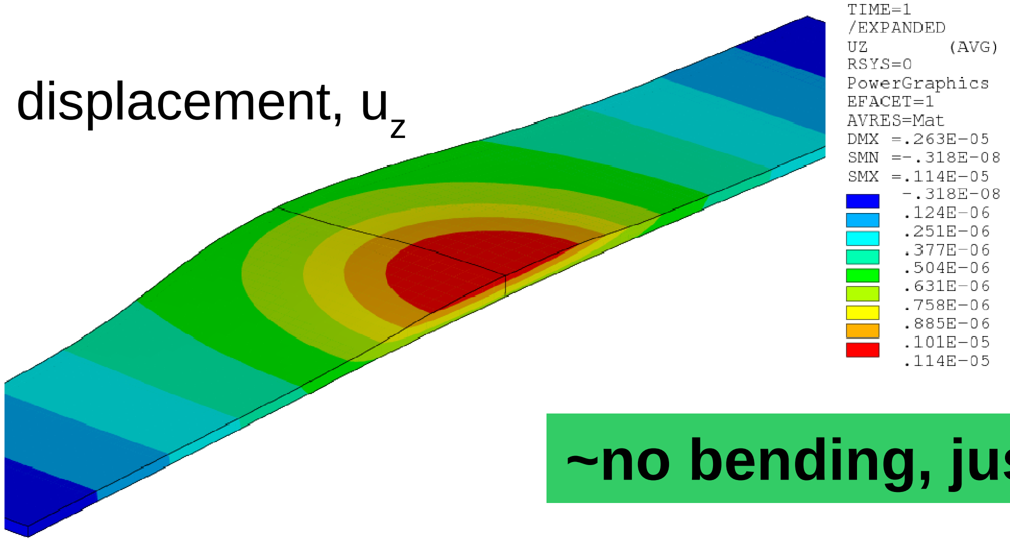
temperature field





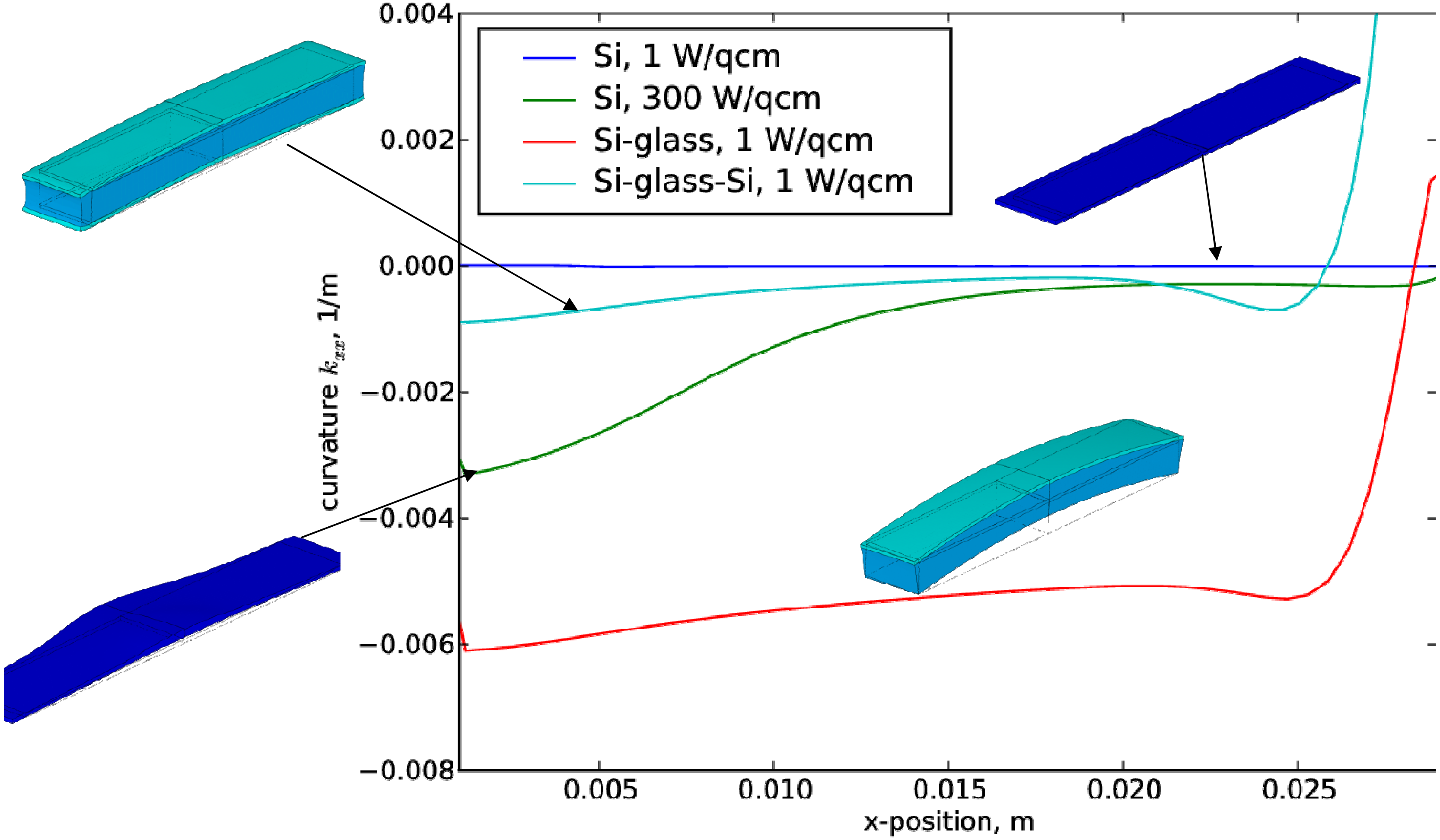
temperature gradient, T_z

displacement, u_z



~no bending, just thermal expansion of Si

Curvature



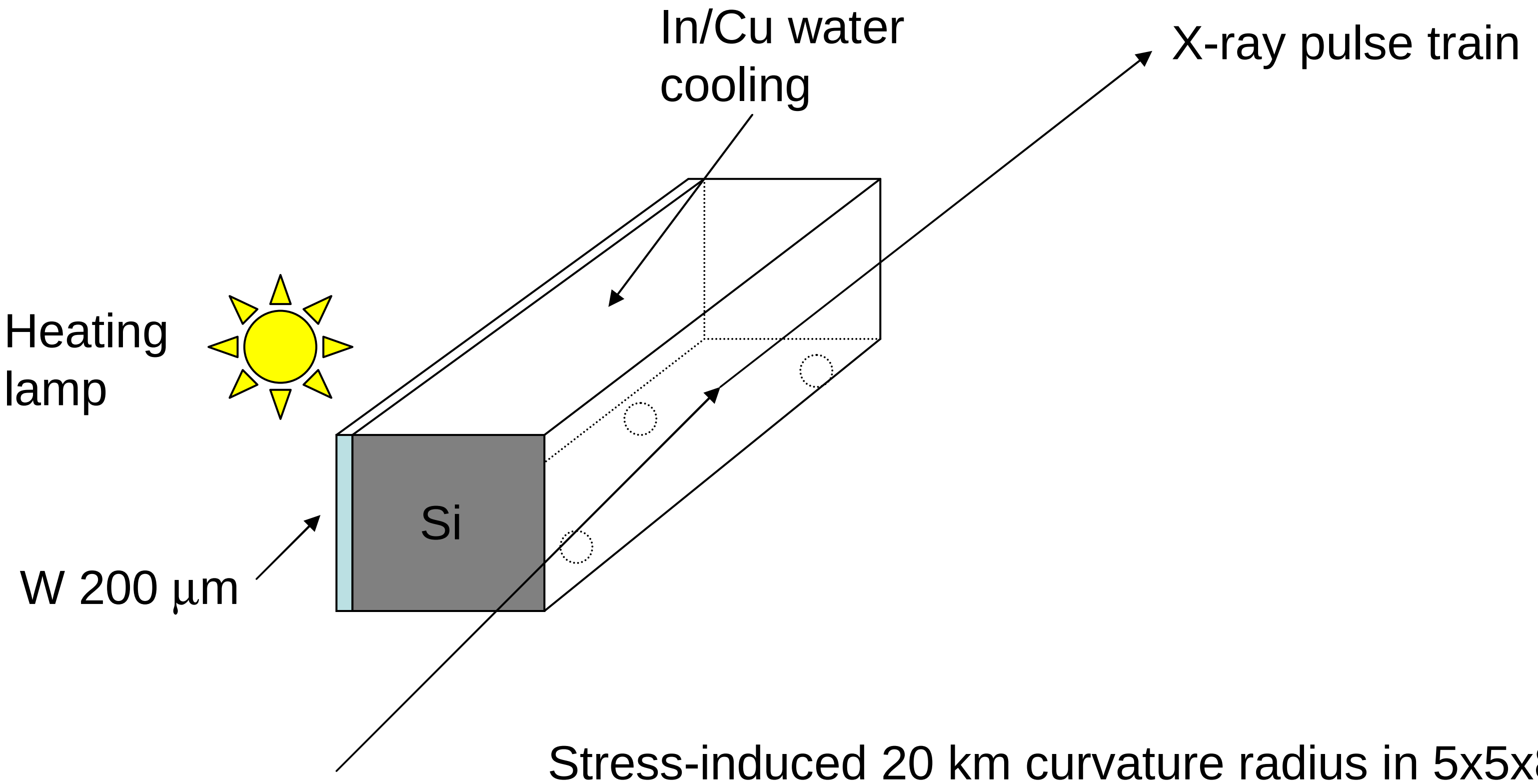
highest curvature with Si on Glass

FEM: X-ray mirror thermal deformations

1. **X-ray mirror (Si wafer):**
   a) Target radius of curvature R = 10 m can be achieved by application of the through-thickness temperature gradient of about 20 °C
   b) the upper limit (RT+20 °C) does not exceed the ma ximum operational temperature of 100 °C

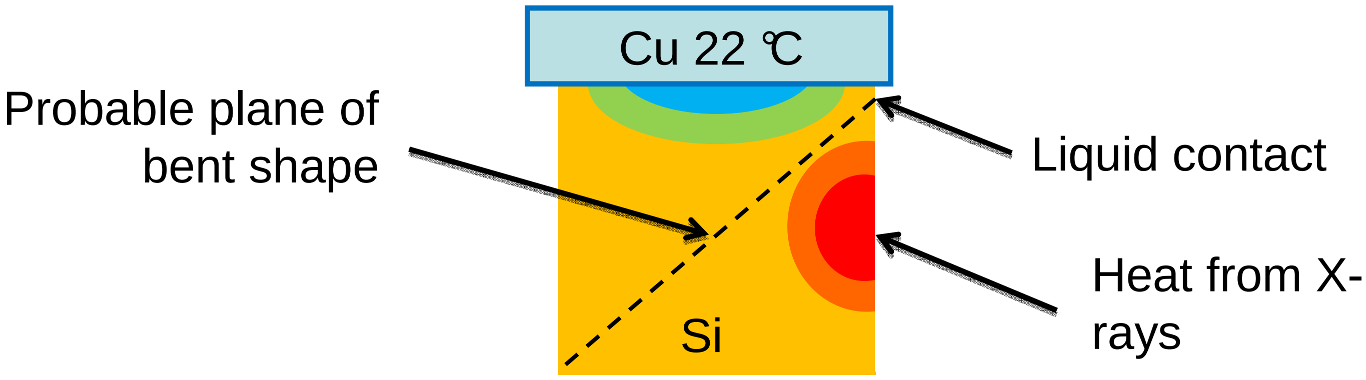
2. **Optics element exposed to the X-ray beam:**
   a) temperature distribution is almost uniform (in steady-state)
   b) X-ray beam with the power of 1 W/cm^2 heats the structure up to 15 °C
   c) minimization or compensation of the thermal expansion mismatch is an effective way to reduce thermal deformations

XFEL Mirror Curvature Control



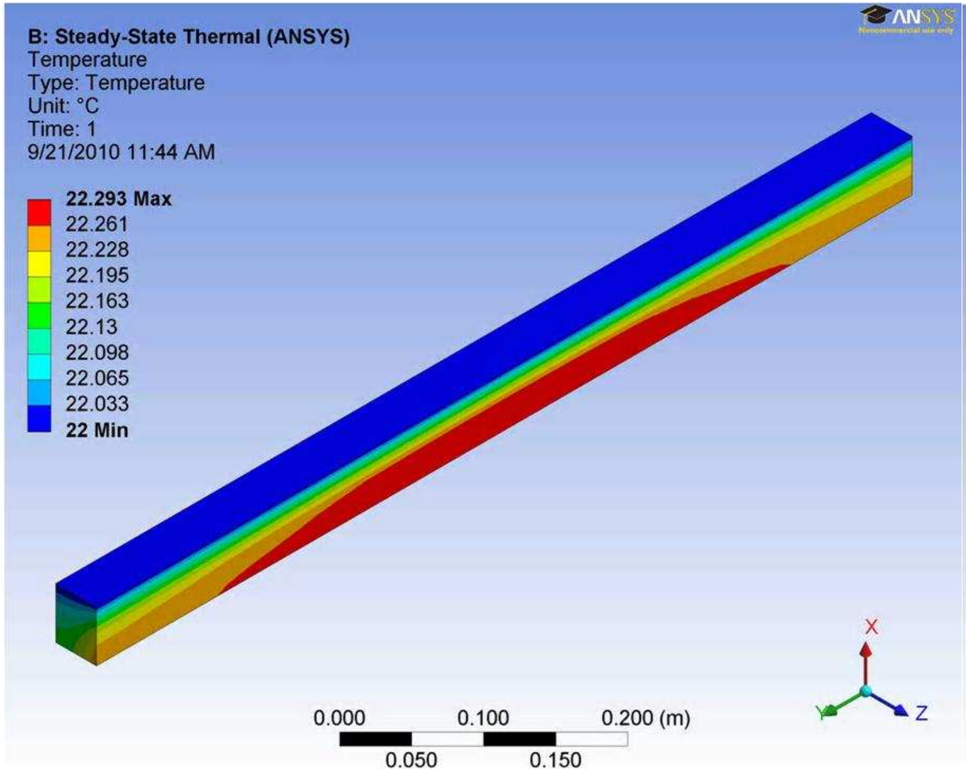
Stress-induced 20 km curvature radius in 5x5x80 cm Si mirror due to 20 ℃ ΔT

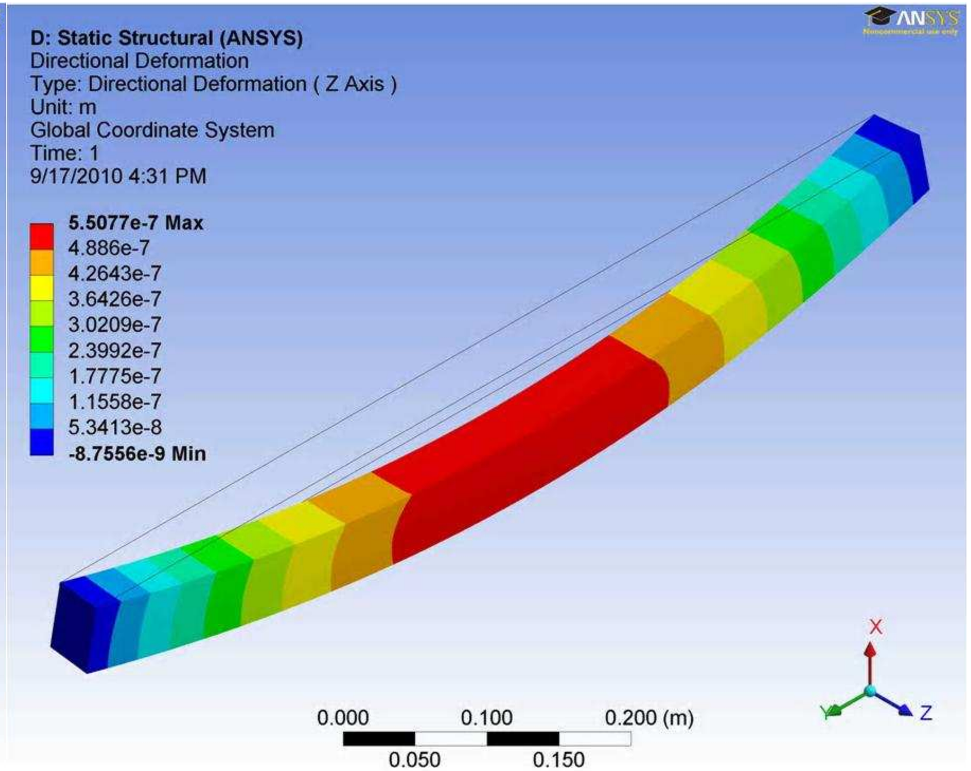
Single cooling surface



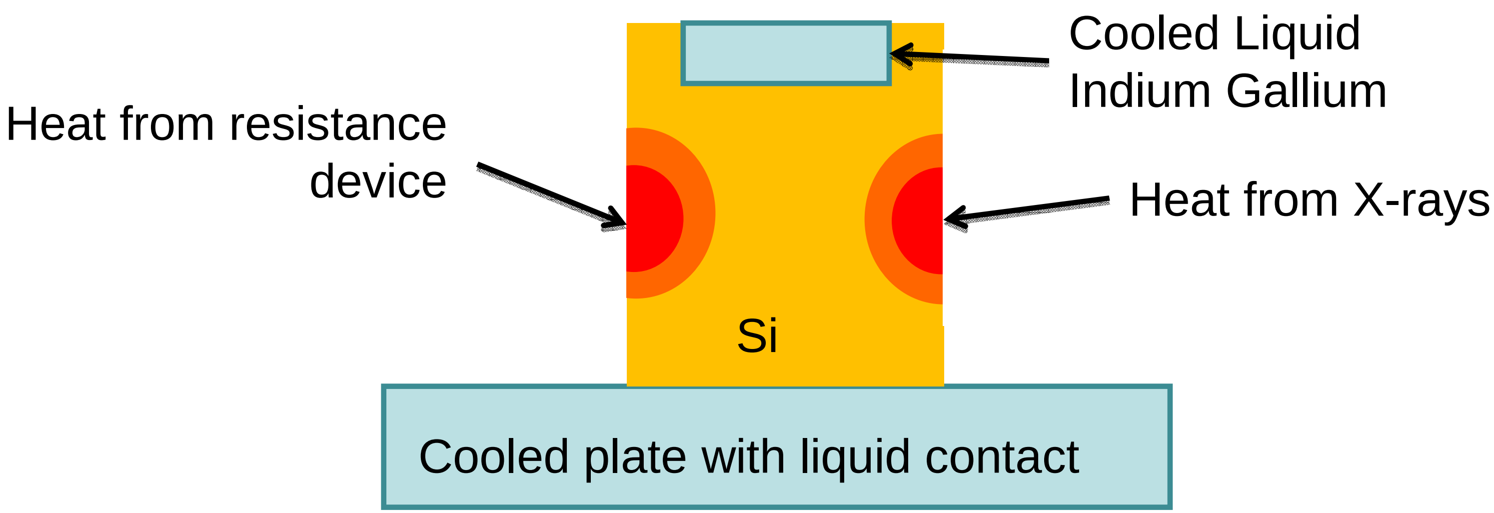
Michael Weinbaum

Single cooling surface only

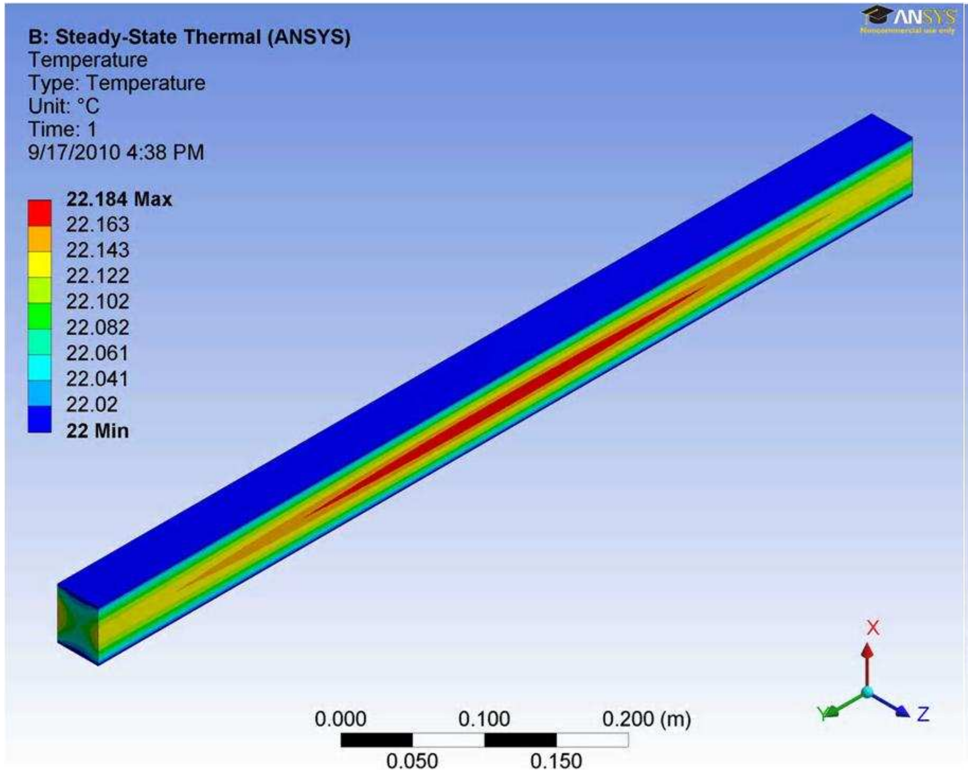


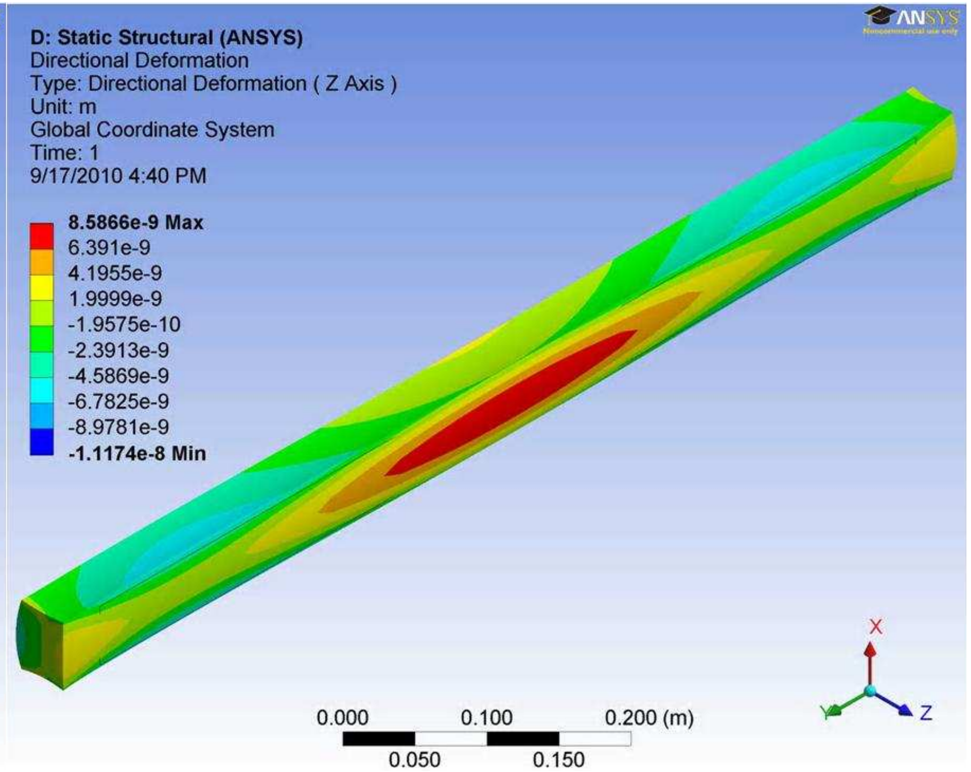


Setup-backlight with two cooling surfaces



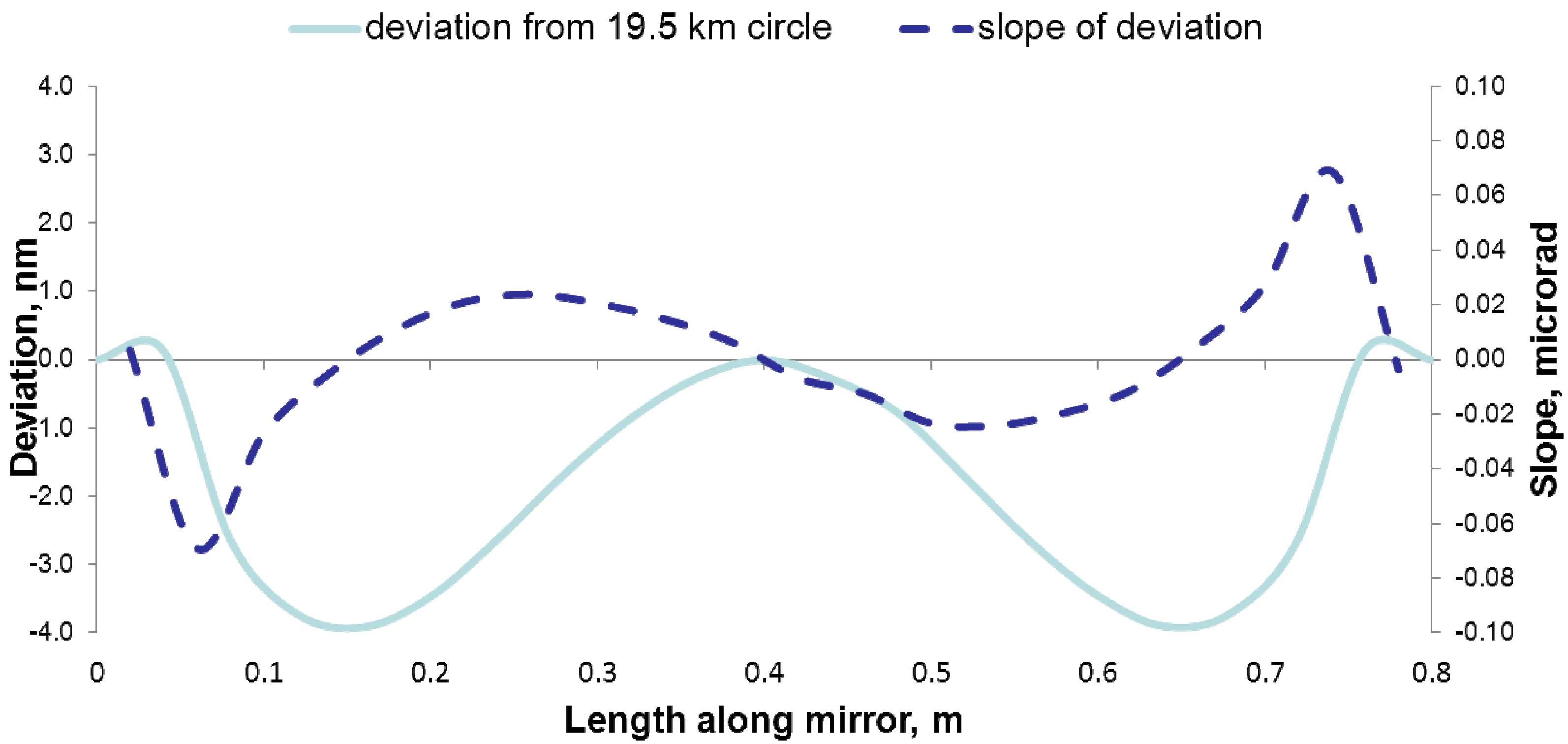
Results- backlighting with two cooling surfaces





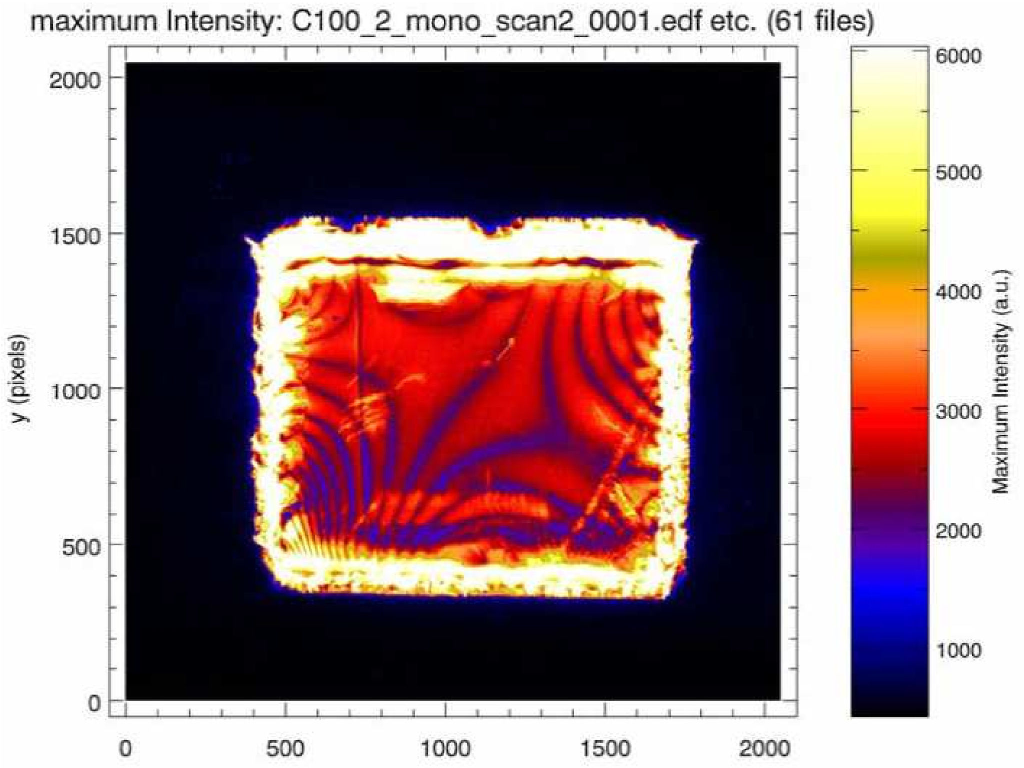
Order of magnitude lower distortion

Results – film and sym. cooling



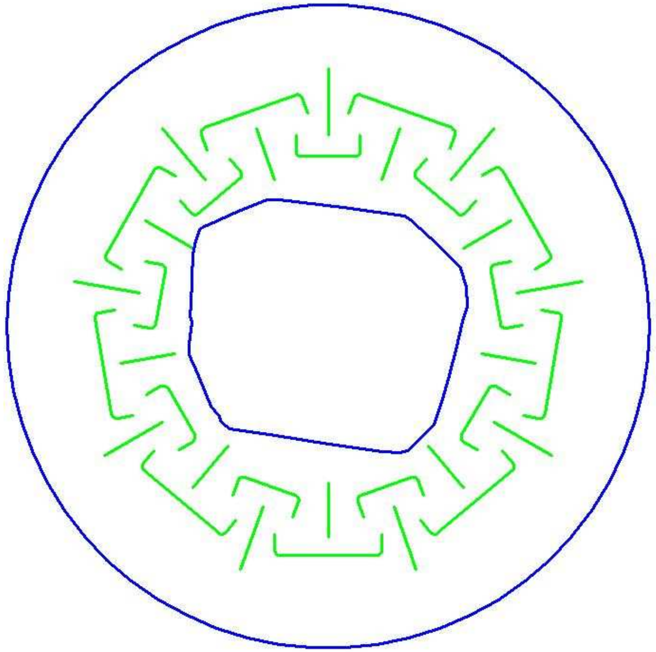
Simulation included 100 micron tungsten film with a 36K temperature change

Stress relief in diamond/CVD ring monochromator



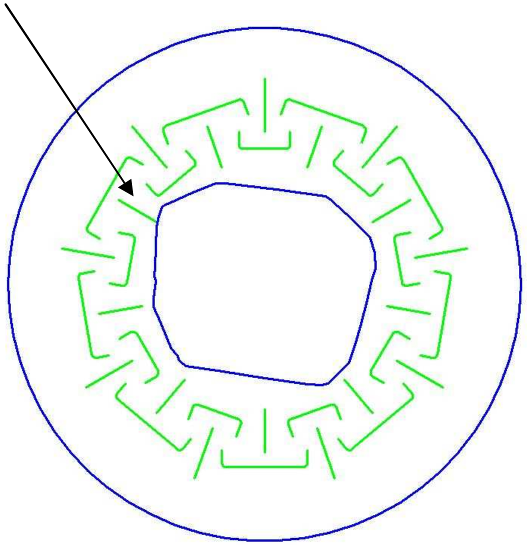
Liubov Samoylova, XFEL

Growers proposed cuts

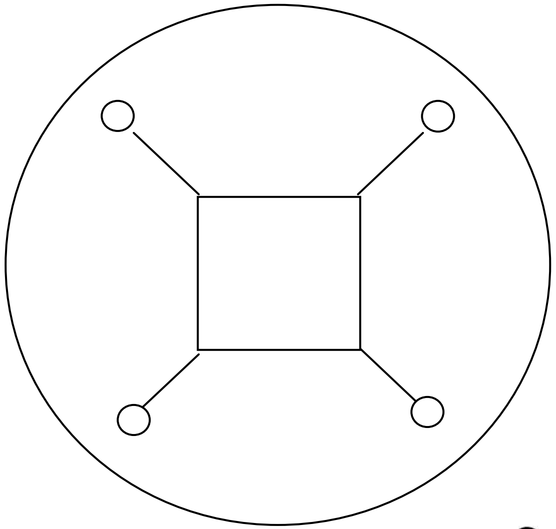


Diamond Monochromator Stress Relief

Useful cut

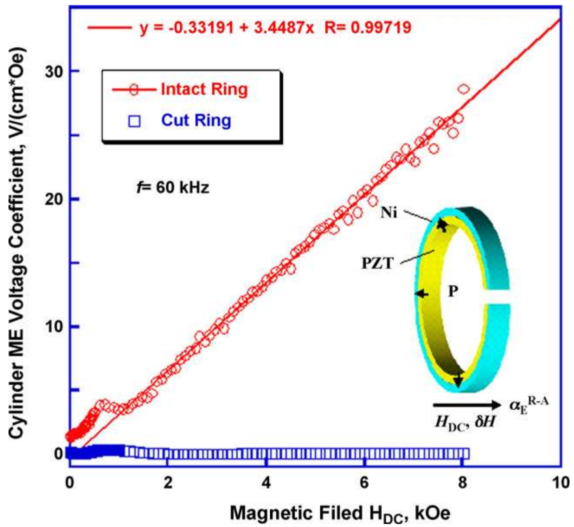


vs.



Fan Yang, XFEL

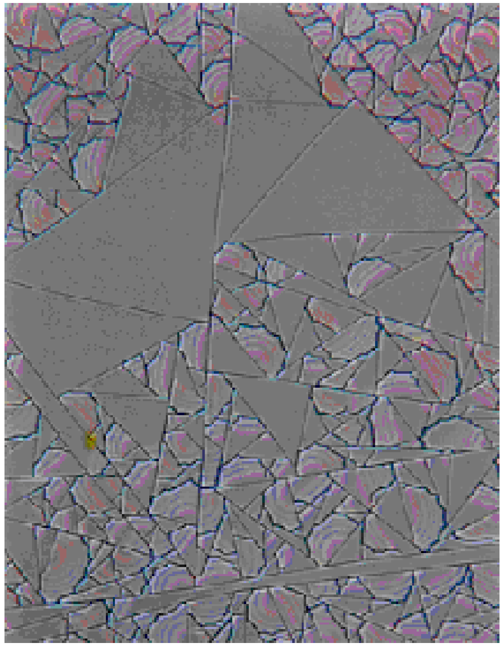
Hoop (circumferential) stress relief σ_θ for a cut ring



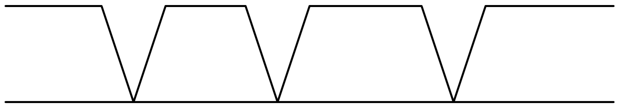
Geometry Effects on Magnetoelectric Performance of Layered Ni/PZT Composites, D.A. Pan, J.J. Tian, S.G. Zhang, J.S. Sun, A.A. Volinsky, L.J. Qiao, Mater. Sci. Eng. B, Vol. 163(2), pp. 114-119, 2009

Stress Relief Through Fracture

Low-K dielectric film fracture in tension

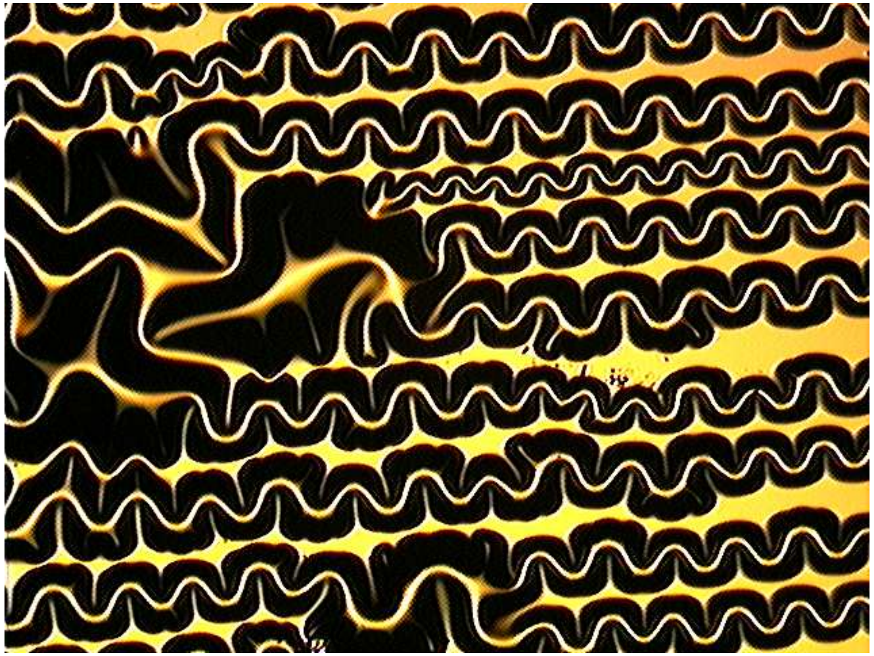


$$h_c = \frac{K^2}{Z\sigma_R^2}$$

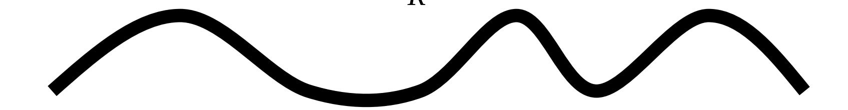


Substrate

TiW film phone cord delamination in compression



$$h_c = \frac{GE}{Z\sigma_R^2}$$



Substrate

Mechanics of Coating Fracture

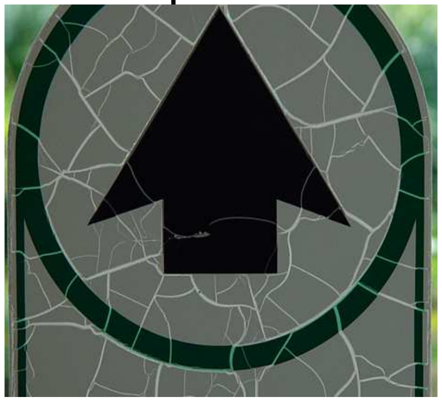
The strain energy release rate of a stressed coating:
(i.e. amount of energy stored in a stressed coating per unit area, J/m^2, *stressed coating is comparable to a loaded spring*)

$$G = Z\frac{\left(1-\nu_c^2\right)\sigma^2 t}{E_c}$$

Coating will delaminate when the strain energy release rate equals the interfacial toughness, $\Gamma_i(\Psi)$, or its adhesion:

$$G = \Gamma_i(\Psi)$$

The coating will crack when the strain energy release rate equals the coating toughness, $\Gamma_{coating}$:



$$G = \Gamma_{coating}$$

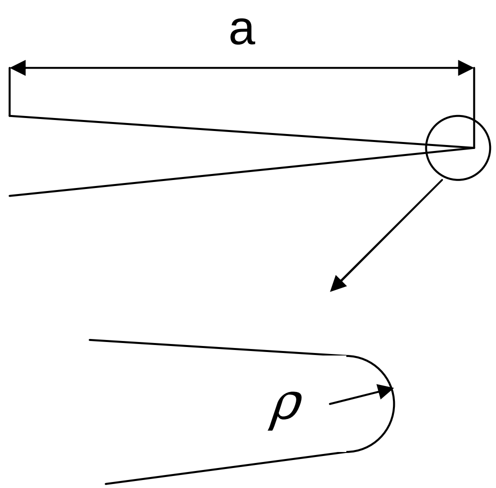
Stress Concentration

Stress at the crack tip is magnified by a factor:

$$\frac{\sigma_{crack\,tip}}{\sigma^{applied}_{thermal}} = C\sqrt{\frac{a}{\rho}}$$

Crack length

Crack tip radius



For a 100 nm crack or defect with 1 nm tip radius, one would find a **10-fold increase** in the stress levels at the crack tip.

Thermal Stress Mechanics

Thermal stress in the coating: $\sigma_{thermal} = \frac{E_c}{1-\nu_c}(\alpha_c - \alpha_s)\cdot\Delta T$

$$G = Z\frac{(1+\nu_c)E_c(\alpha_c-\alpha_s)^2\Delta T^2 t}{(1-\nu_c)}$$

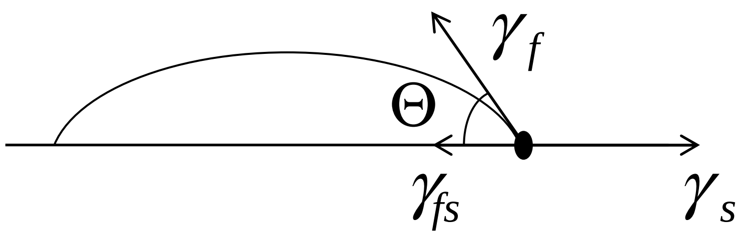
Thermal expansion mismatch causes substantial substrate bending!

Work of Adhesion

Total irreversible energy per unit area of delamination extension required to separate the materials at the interface.

Thermodynamic work of adhesion

$$W_A = \gamma_f + \gamma_s - \gamma_{fs} = \gamma_f (1 + \cos\Theta)$$

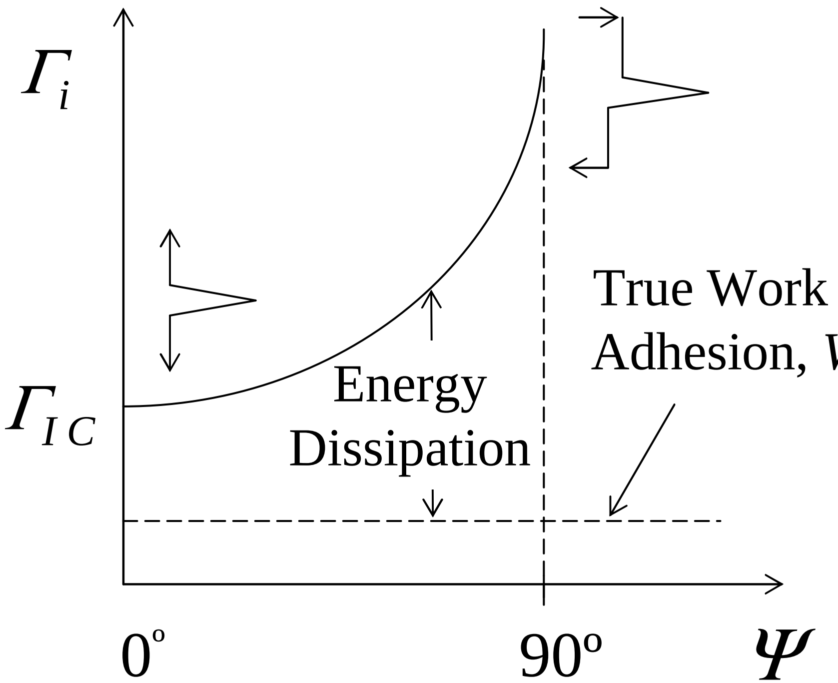


$$G = -\frac{dU_M}{dA} \qquad G \geq \Gamma_i(\Psi)$$

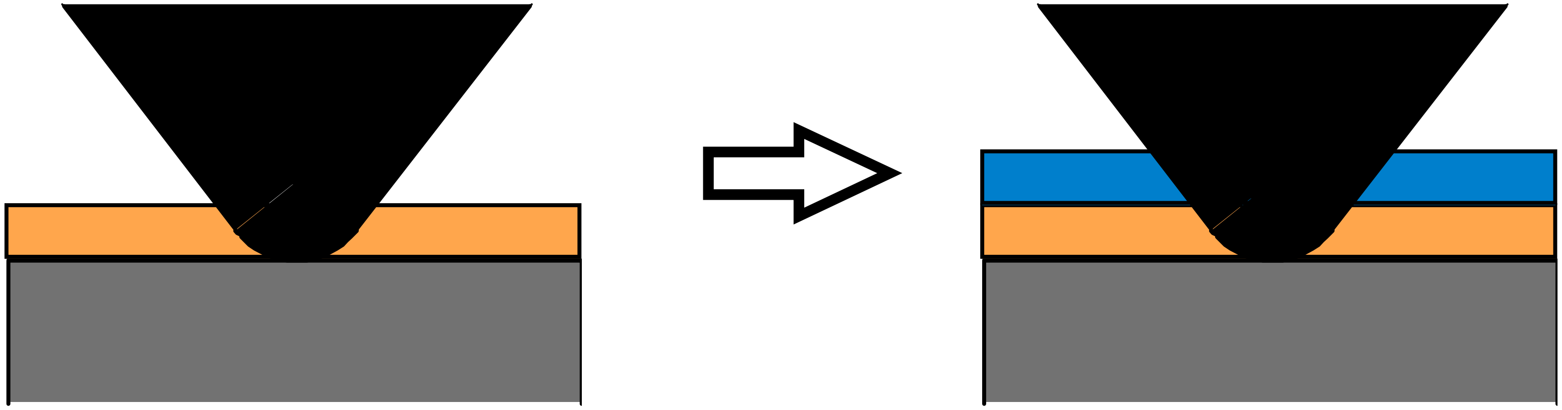
$$\Psi = \tan^{-1}(\tau / \sigma)$$

Practical work of adhesion

$$W_{A,P}(W_A) = W_A + U_f + U_s$$



Single Layer vs. Superlayer Indentation



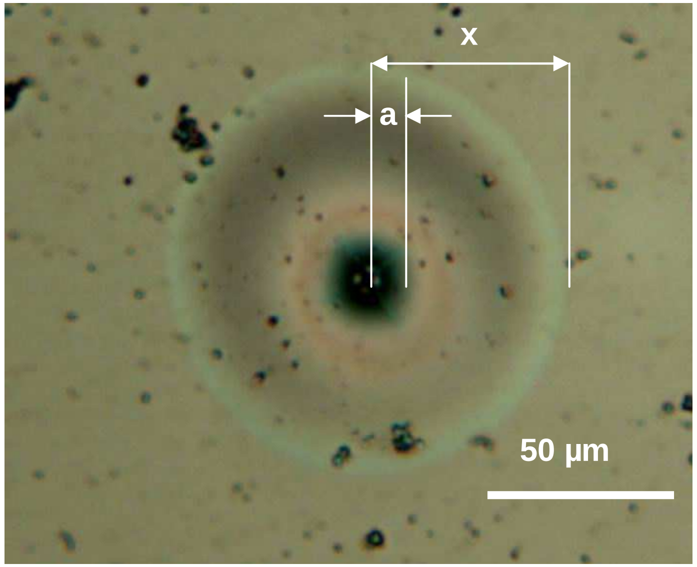
$$\frac{GE_f}{(1-\nu_f)} = \frac{1}{2}h\sigma_I^2(1+\nu_f) + (1-\alpha)(h\sigma_R^2) - (1-\alpha)h(\sigma_I - \sigma_B)^2$$

Nonbuckled: $\alpha = 1$, Buckled (single,double): $0 < \alpha < 1$

Biaxial Film Stress Relation: $\sigma = \varepsilon E/(1-\nu)$

1. D.B. Marshall and A.G. Evans, Measurement of adherence of residually stressed thin films by indentation. I. Mechanics of interface delamination, J. Appl. Phys., **56** (1984) p. 2632-2638.
2. J.W. Hutchinson and Z. Suo, Mixed mode cracking in layered materials, in Advances in Applied Mechanics, 1992, Academic Press, Inc.: New York, p. 63-169.
3. M.D. Kriese and W.W. Gerberich, Quantitative adhesion measures of multilayer films. J. Mater. Res. **14** (7), p. 3007, 1999

Experimental Measurements

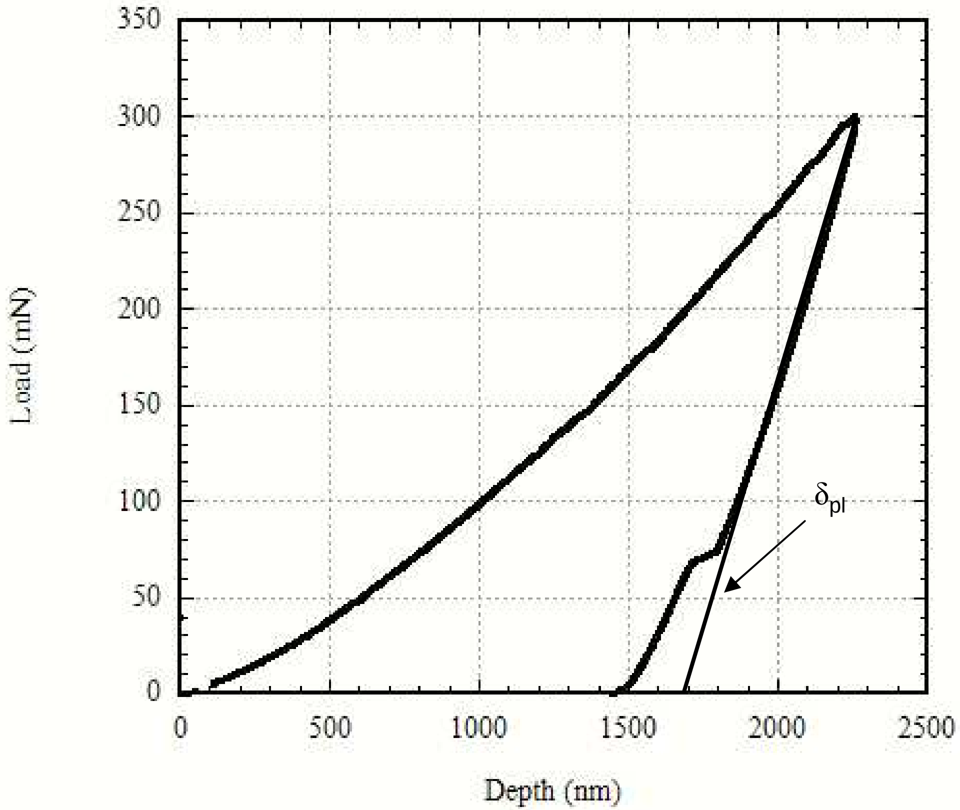


Optical Microscope used for blister measurements

x = blister radius

a = contact radius of indenter tip

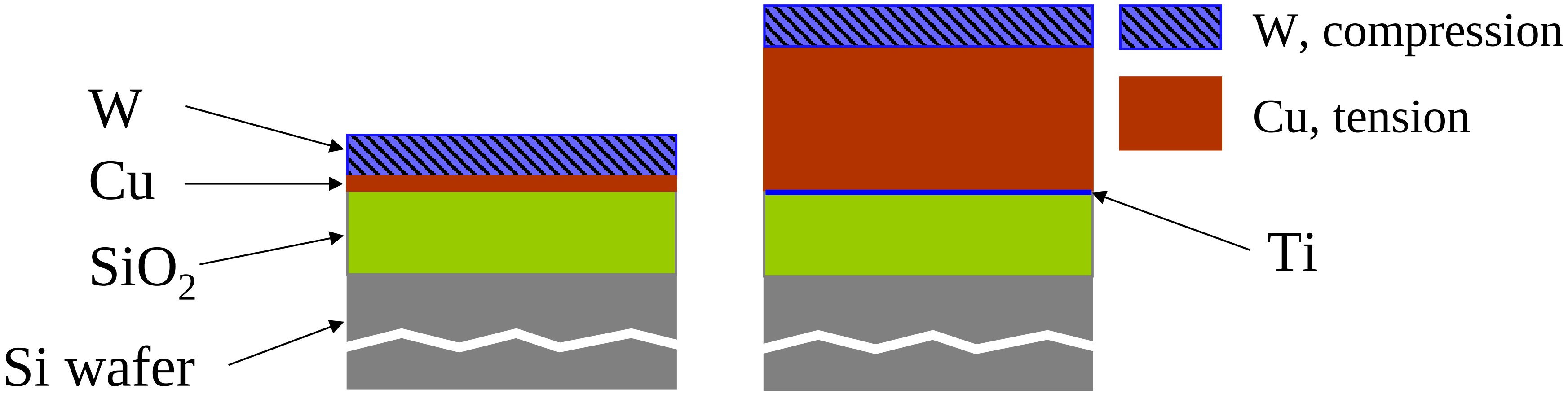
δ_{pl} = plastic indentation depth



Load-Displacement curve from the indenter

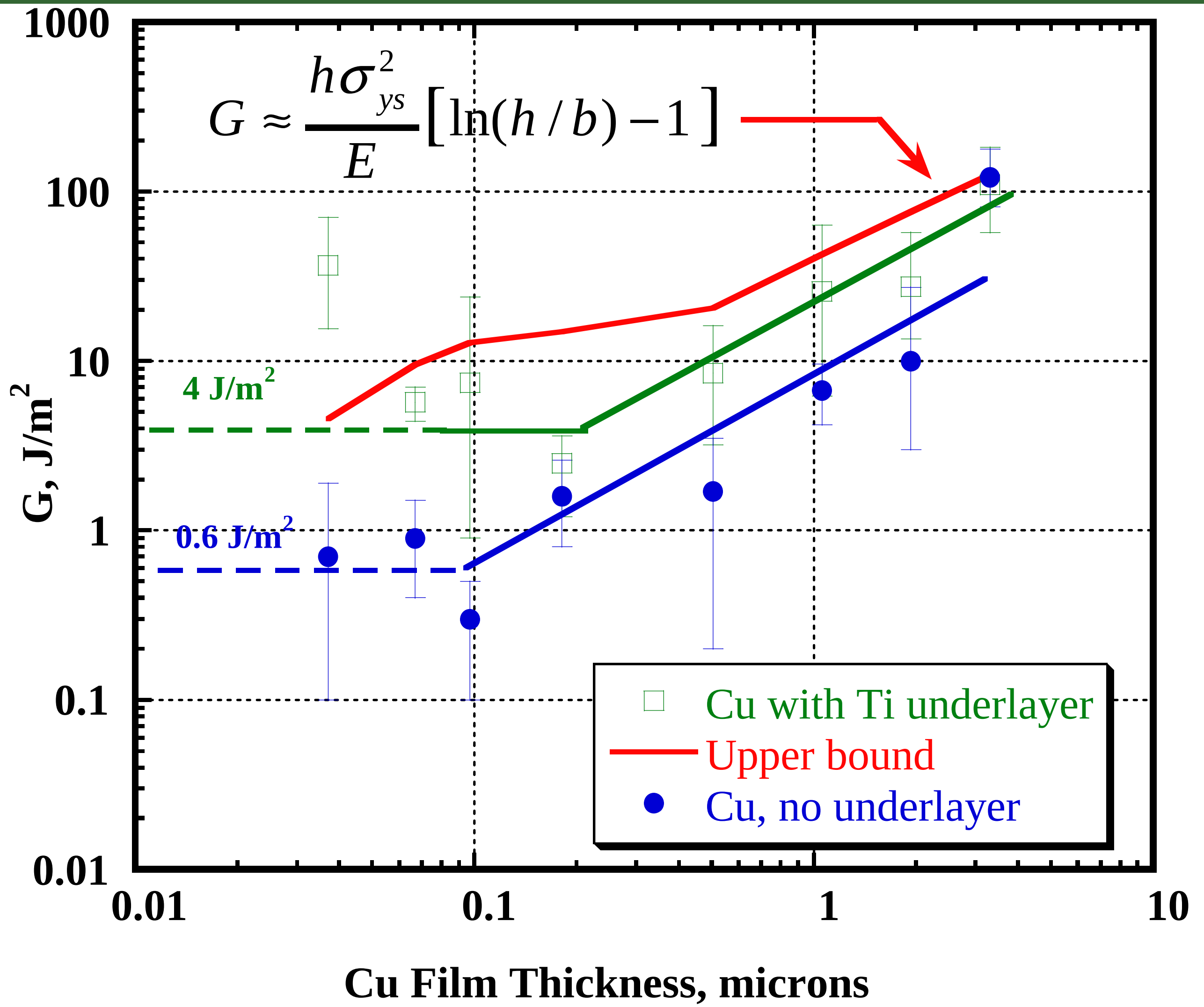
Cu-BASED THIN FILM SYSTEM

Films: 1 µm W overlayer on top of Cu films (40 nm to 3 µm) with and without a 10 nm Ti underlayer



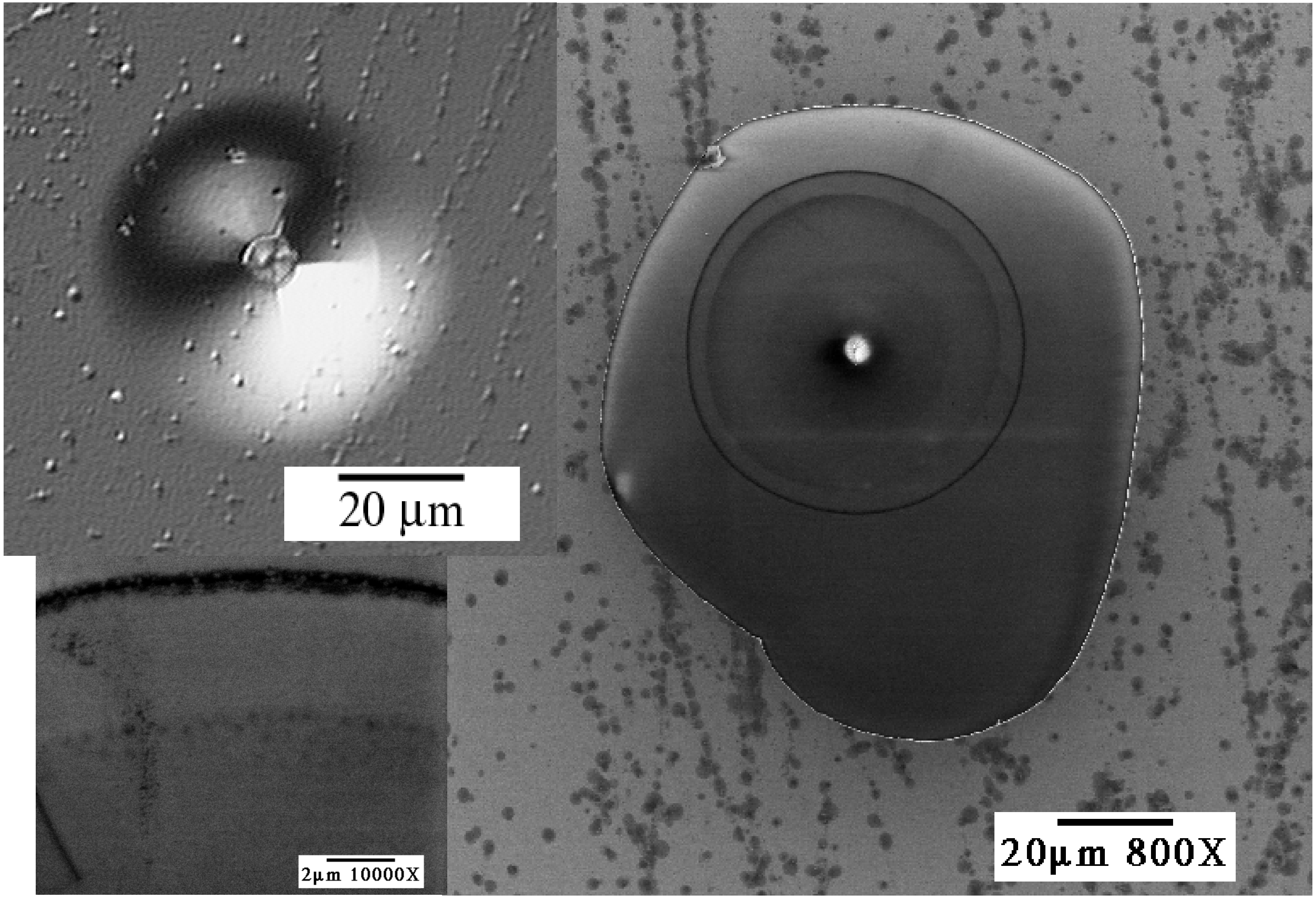
- **Substrates:** Si wafers <100> w/ thermally grown 1.5 µm of SiO_2
- **Processing:** Cleanroom, sputtering in argon (1 µTorr pump down), no etching.

Cu Film Adhesion

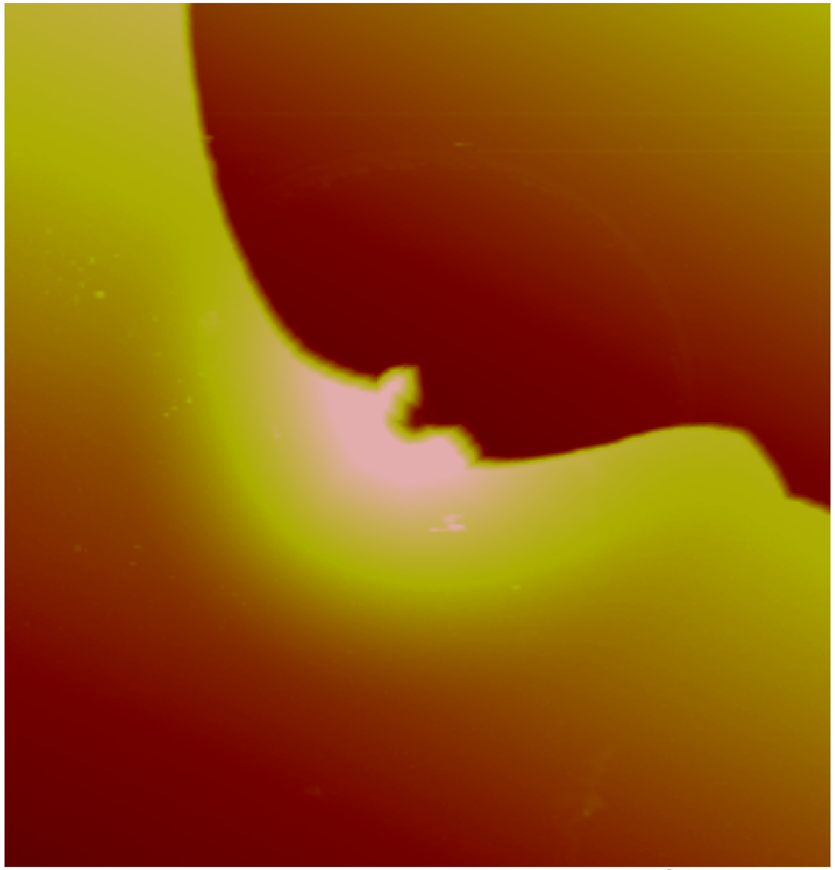


A.A. Volinsky, N.R. Moody, W.W. Gerberich, Acta Mater. Vol. 50/3, pp. 441-466, 2002

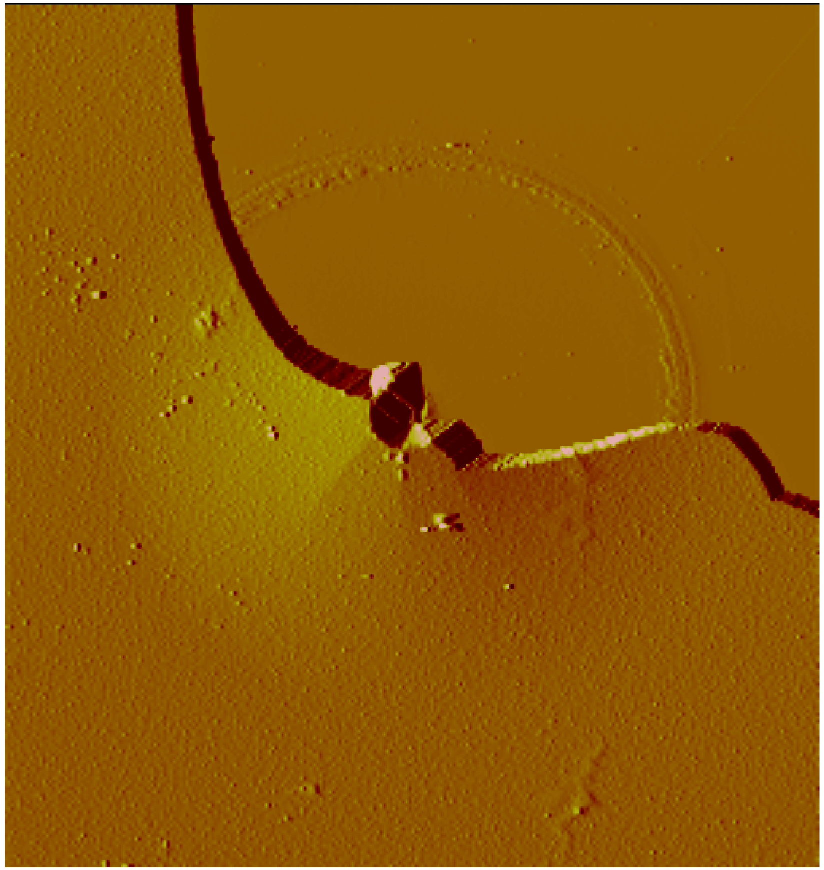
FIDUCIAL MARK. SEM



PARTIAL BLISTER REMOVAL

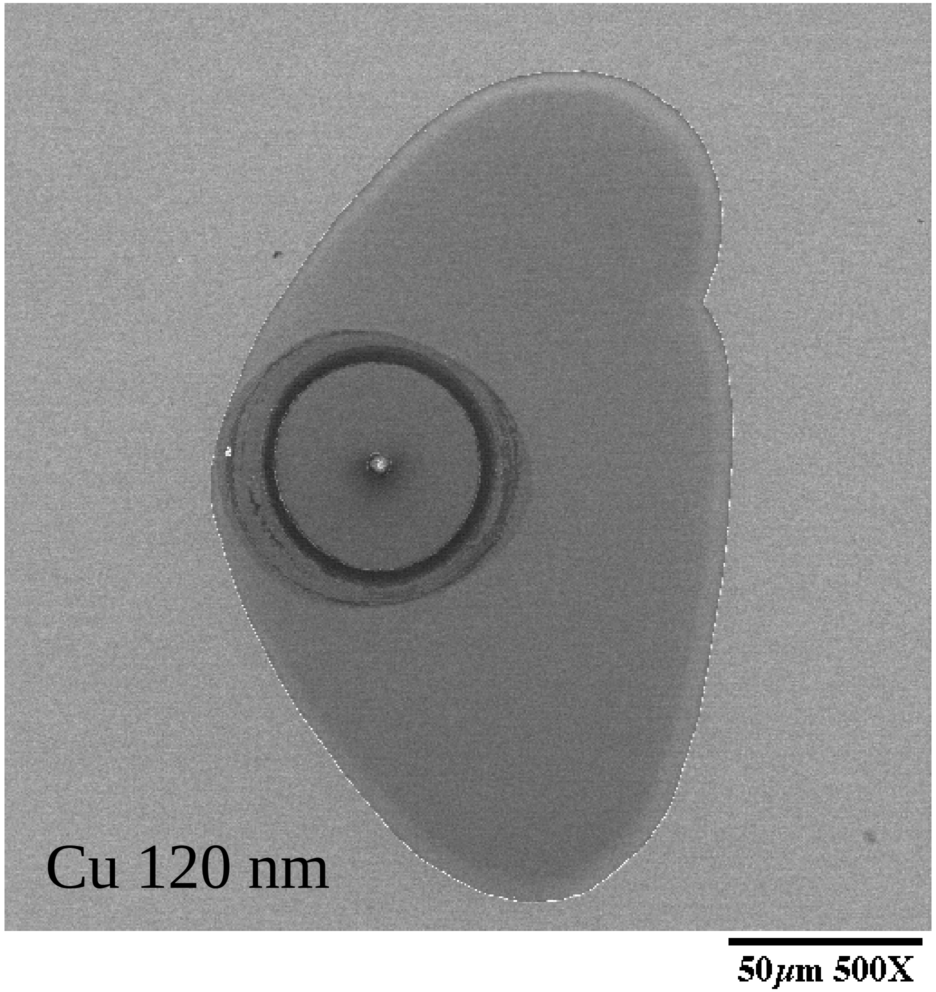


3 microns Z range 25 μm

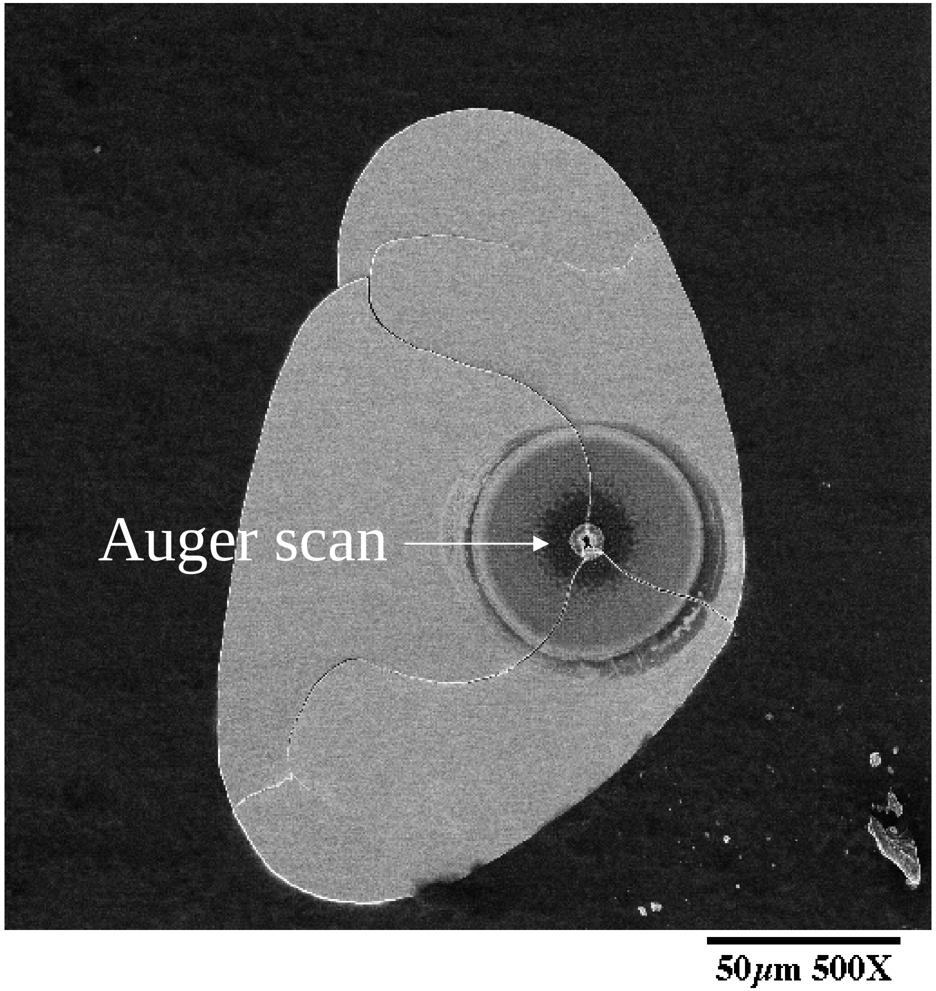


FIDUCIAL MARKS - SEM

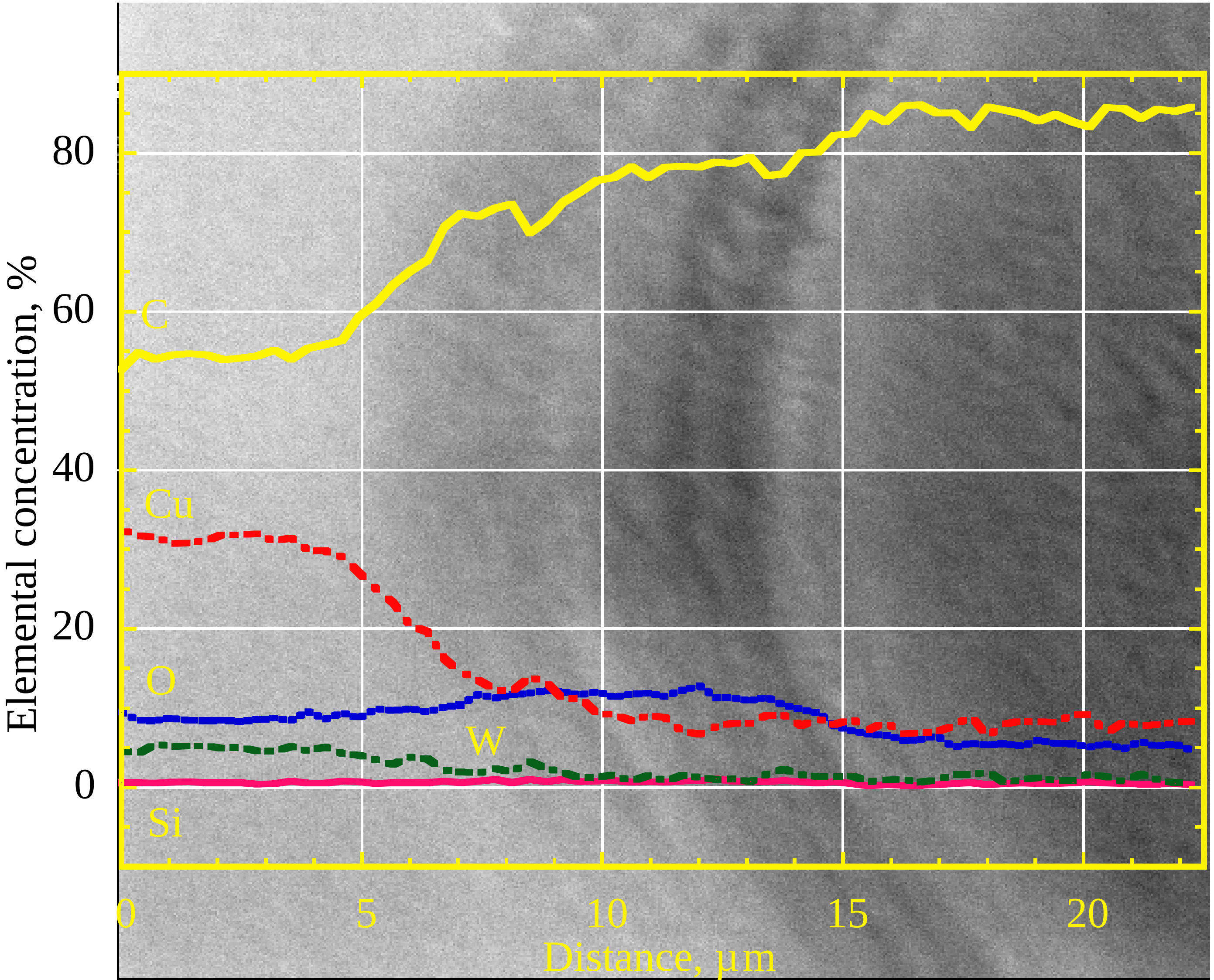
Substrate (SiO_2) side



Sticky tape (Cu) side

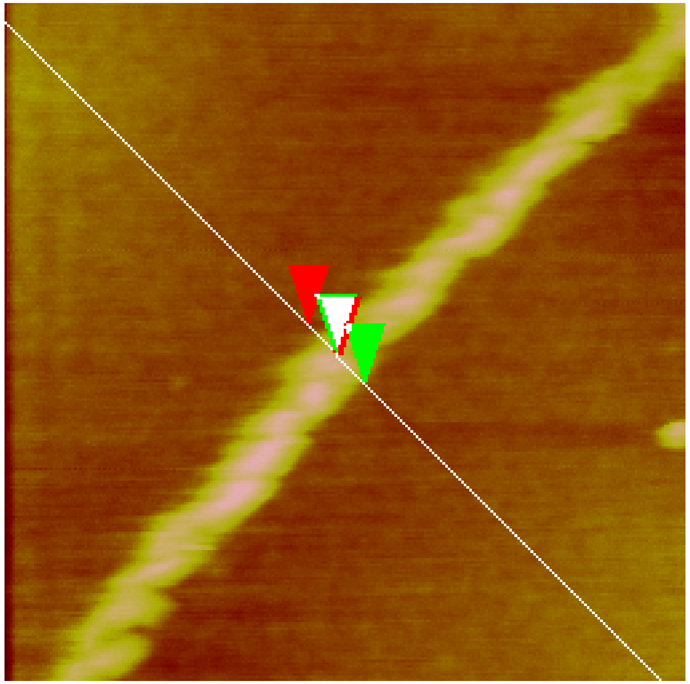


AES SCAN

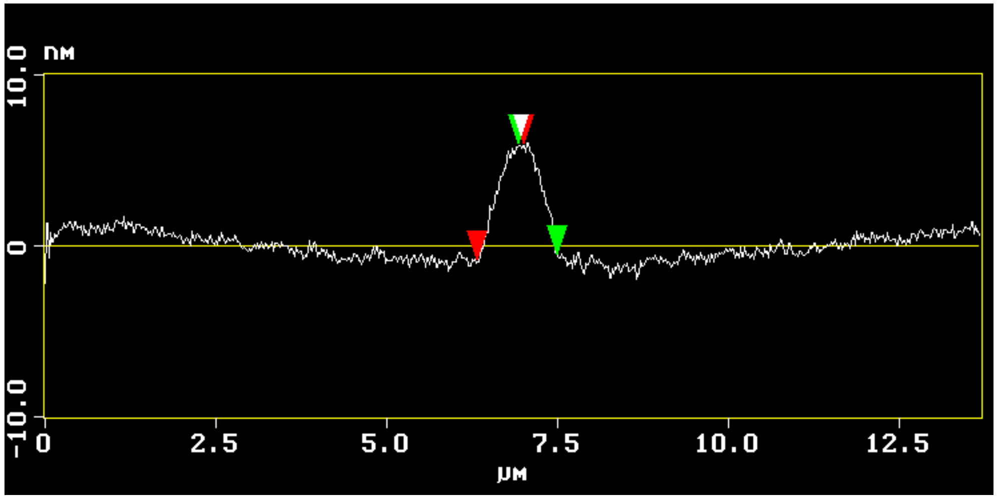


Elemental concentration, %
80
60
40
20
0
C
Cu
O
W
Si
0
5
10
15
20
Distance, µm

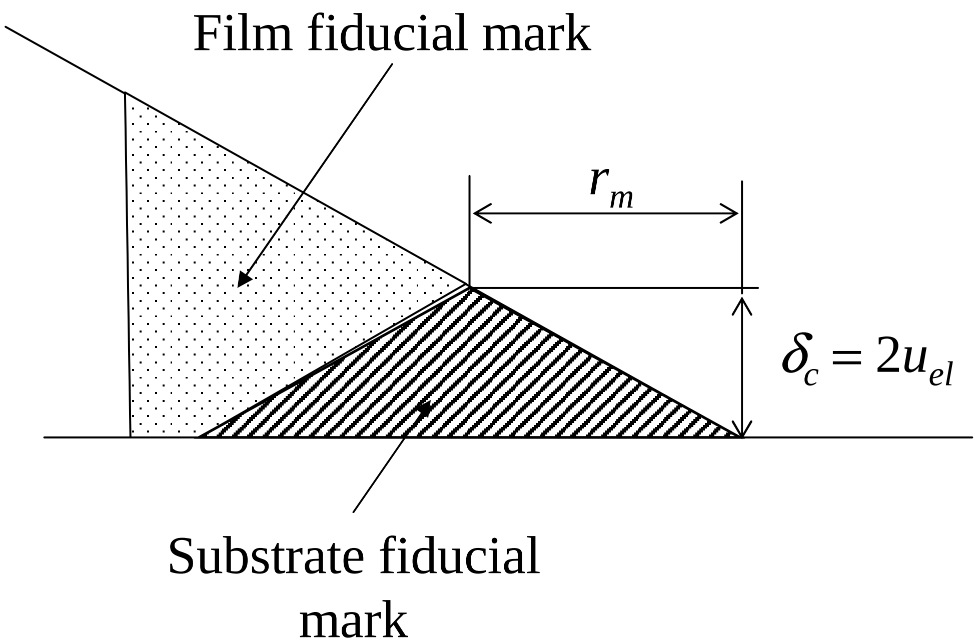
AFM MARK MEASUREMENTS



Surface distance	683.74 nm
Horiz distance(L)	683.59 nm
Vert distance	6.808 nm
Angle	0.571 deg
Surface distance	566.54 nm
Horiz distance	566.41 nm
Vert distance	6.473 nm
Angle	0.655 deg



LINEAR ELASTIC CRACK TIP ANALYSIS



$$u_{el}(r) = \frac{K}{E}\sqrt{\frac{8r}{\pi}}$$

Lawn B., (1993) "Fracture of Brittle Solids", Cambridge University Press, Cambridge

$$K_I = \delta_c E\sqrt{\frac{\pi}{32 r_m}}$$

K_I=0.3 MPa·m$^{1/2}$

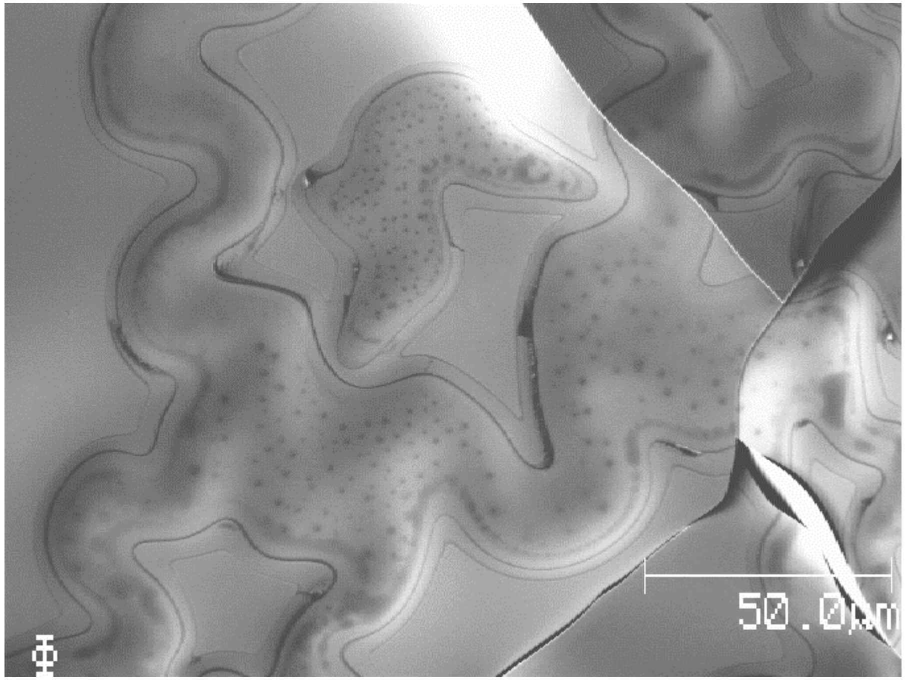
$K_I = (GE)^{1/2}$

G≈0.9 J/m^2 => K_I=0.33 MPa·m$^{1/2}$

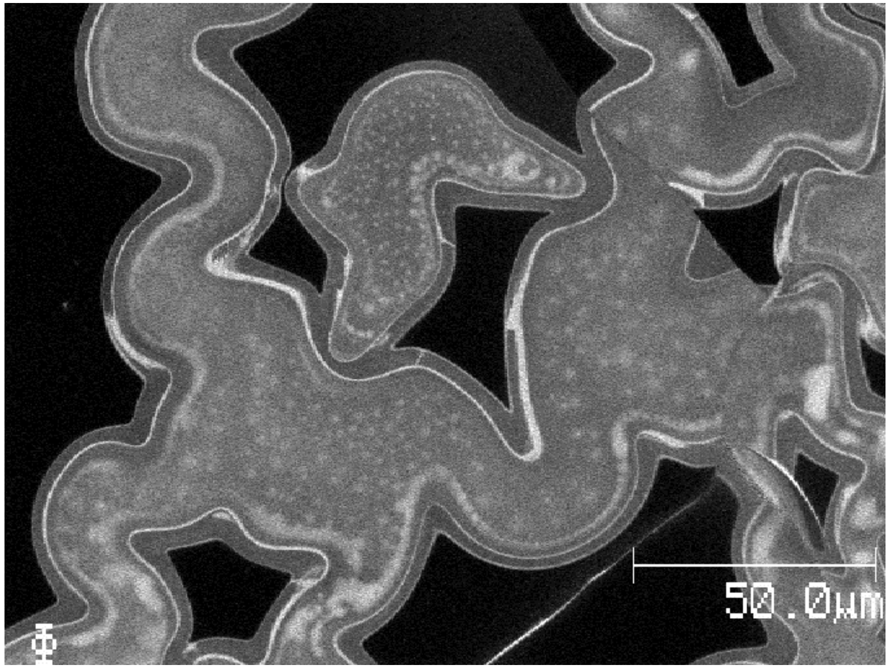
A.A. Volinsky, M.L. Kottke, N.R. Moody, W.W. Gerberich, Engineering Fracture Mechanics 69, pp. 1511-1515, 2002

Auger. Phone Cord Fiducial Marks

SEM



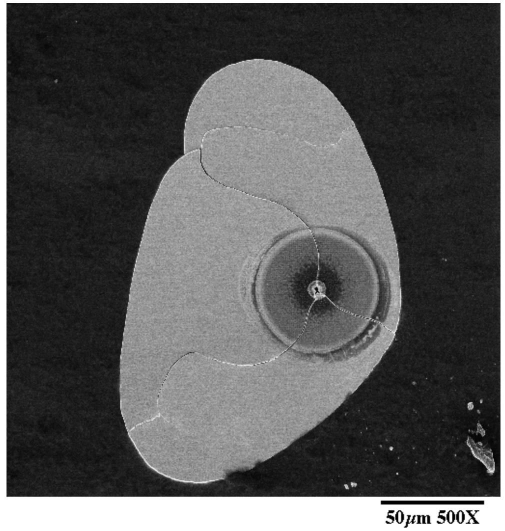
Carbon Map



Mike Kottke

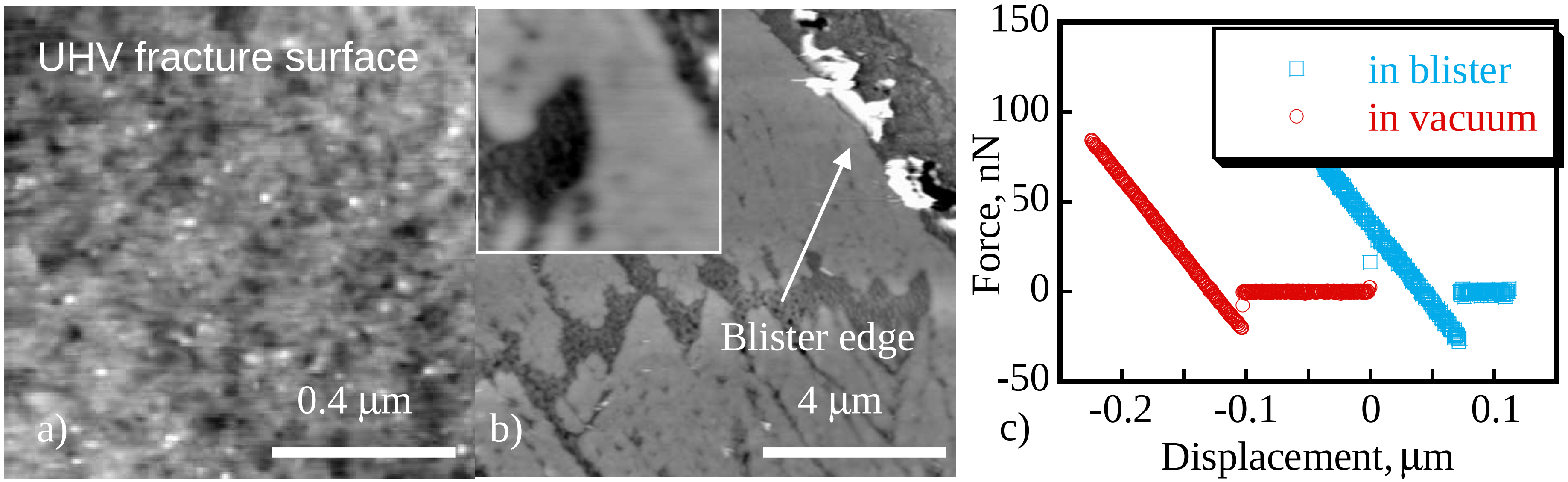
Crack Tip Surface Energy

Conventional methods of surface energy measurement (contact angle technique) only work in air. While film is delaminating, its surface energy is reduced.

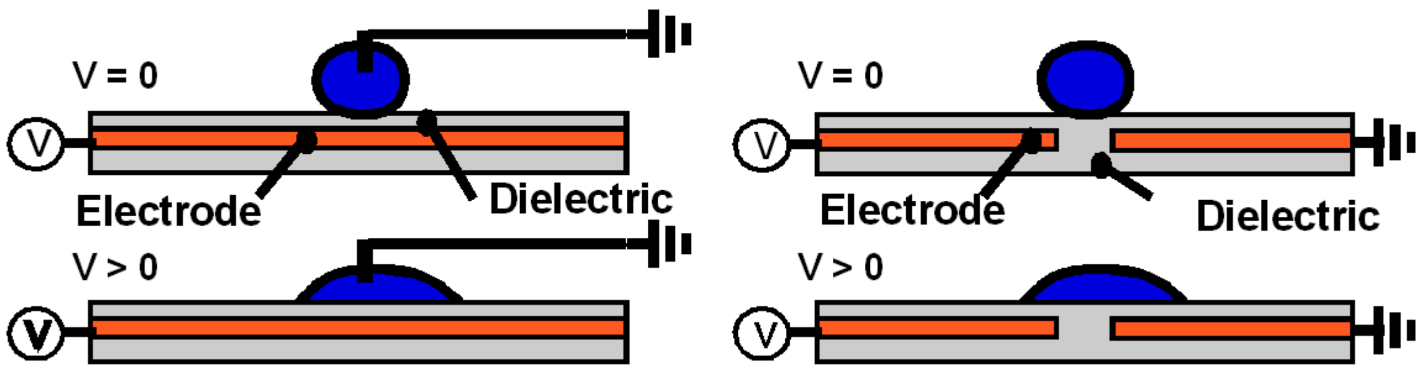


Fiducial Mark and Nanocrack Zone Formation During Thin Film Delamination, A.A. Volinsky, N.R. Moody, M.L. Kottke, W.W. Gerberich, Philosophical Magazine A, Vol. 82, 2002

UHV-AFM (Prof. Szymonski's group). Create fracture surface in UHV, then use AFM tip pull-off data to calculate surface energy.



Electrowetting



Equivalent Electrical Circuit

Drops can be moved by varying electrical field around the drop

Electrowetting Measurement

Electrowetting is typically characterized by the wetting angle

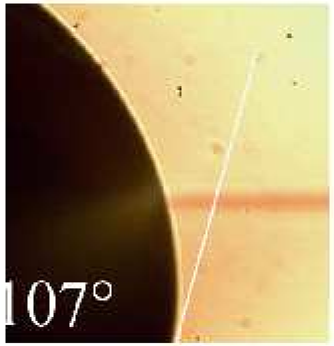
Fitted to Young-Lippman equation assuming parallel plate capacitor

Forces are estimated by modeling surface equilibrium

For many applications electrowetting force is of great interest:

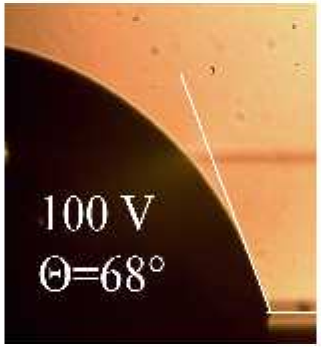
Digital microfluidics, Adaptive cooling, focusing optics, flexible electronics, etc.

0 V



θ = 107 deg

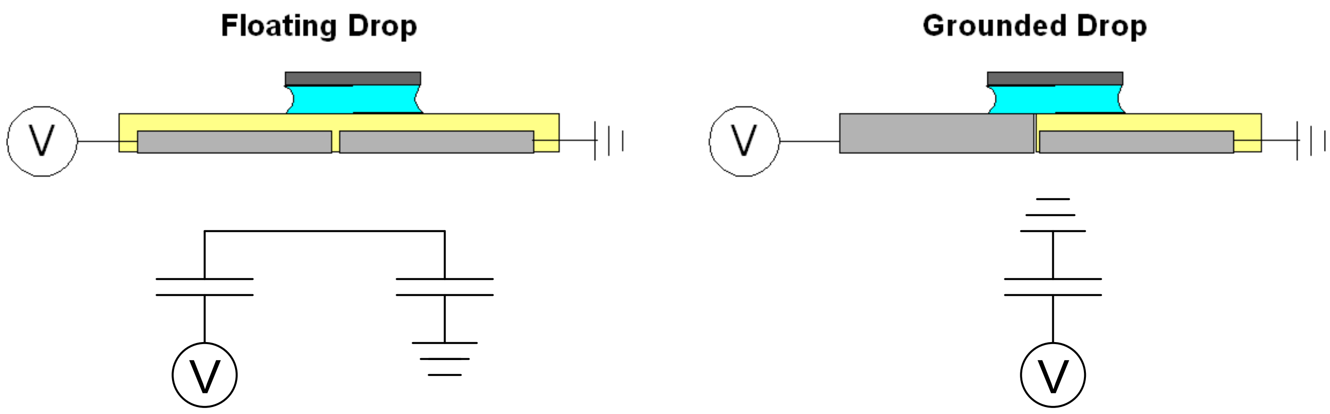
100 V



θ = 68 deg

$$\cos\theta_1 = \cos\theta_o + \frac{\varepsilon_o \varepsilon_r V^2}{2\gamma_{lv}\delta}$$

Electrowetting Configurations



Equivalent Electrical Circuit

Force-Position (Lumped Parameter Model)

$$E = \frac{V_{tot}^2 \varepsilon_0 \varepsilon_R}{8\delta}\left(s^2 - 4x^2\right)$$

$$F_x = \frac{dE}{dx} = -\frac{\varepsilon_0 \varepsilon_R}{\delta} V_{tot}^2 x$$

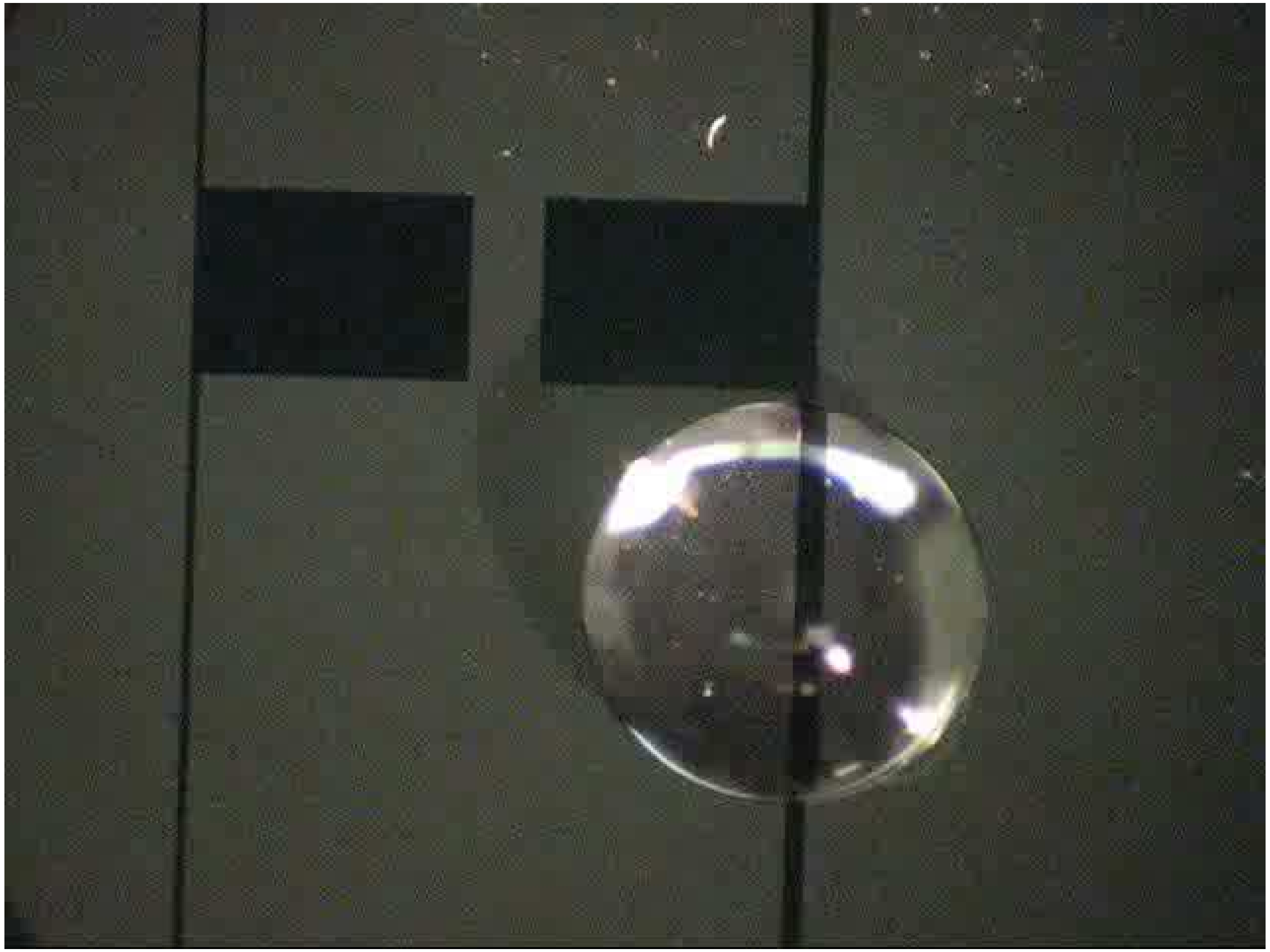
$$F_x = \frac{dE}{dx} = -\frac{\varepsilon_0 \varepsilon_R}{2\delta} s V_{tot}^2$$

s - edge length

x – plate displacement

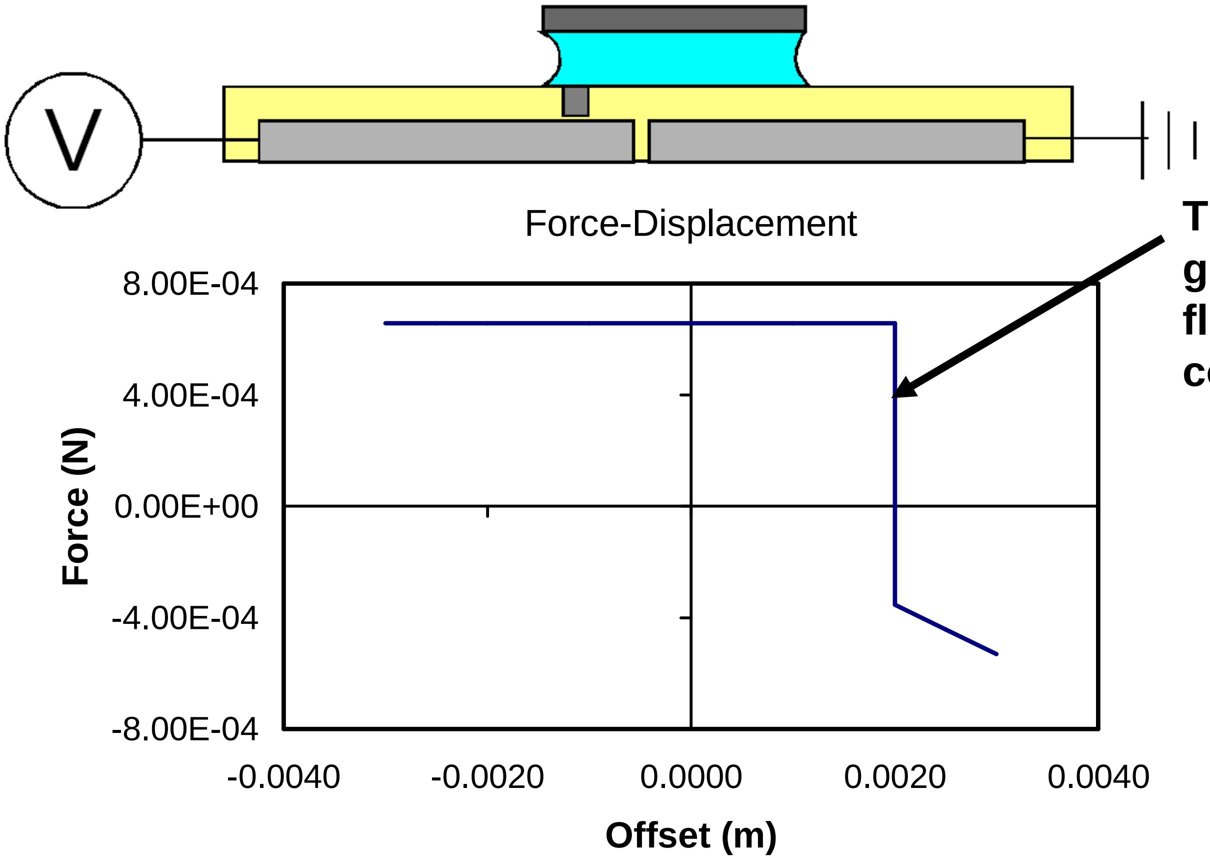
Forces are found by differentiating the system energy with respect to the appropriate displacement variable.

Electrowetting Oscillation. DC Voltage



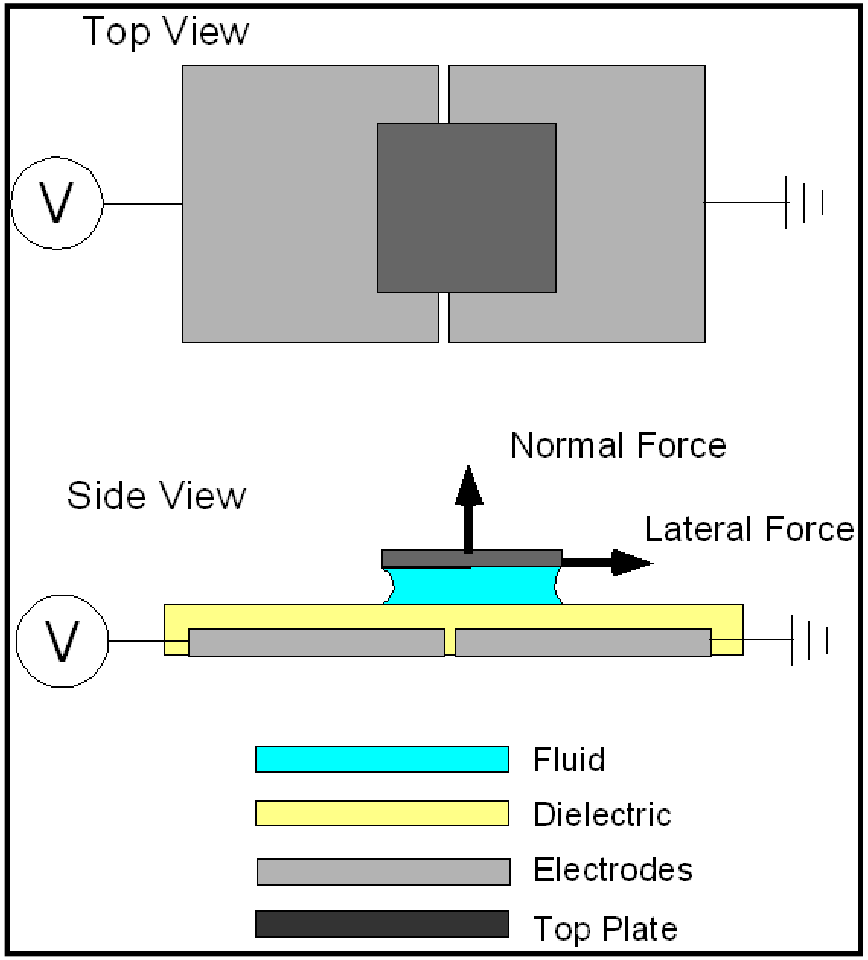
Oscillation Explanation: Local Dielectric Defect

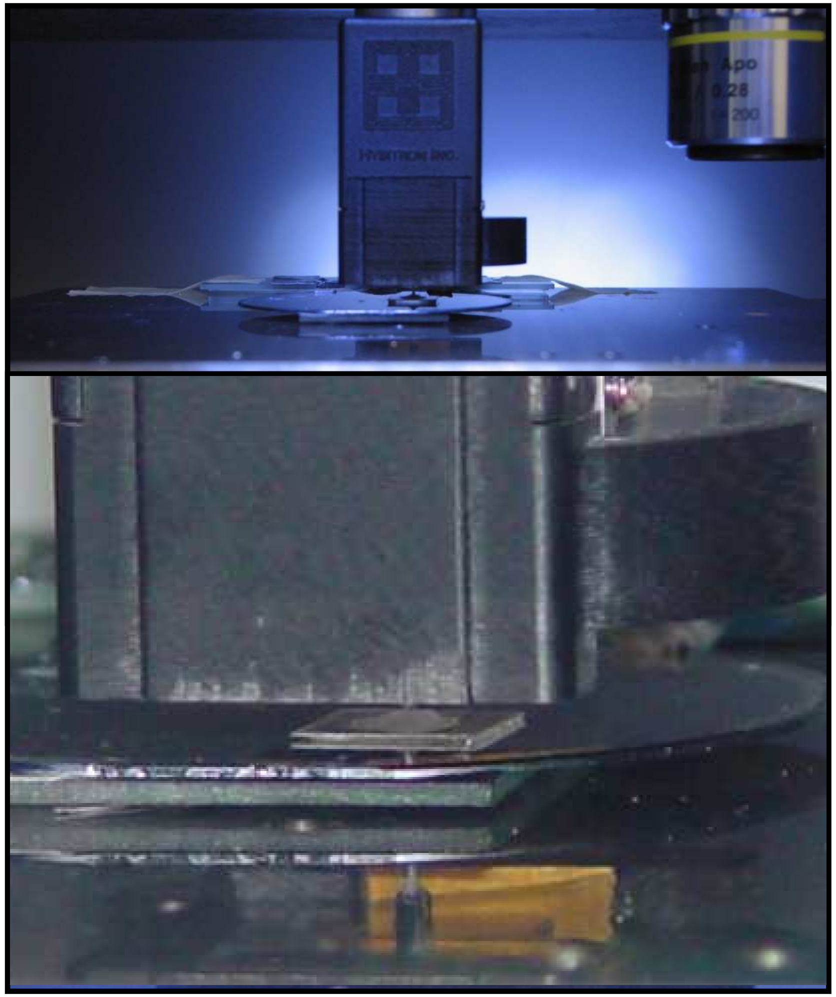
Dielectric Defect Results in Mixed-Mode Behavior



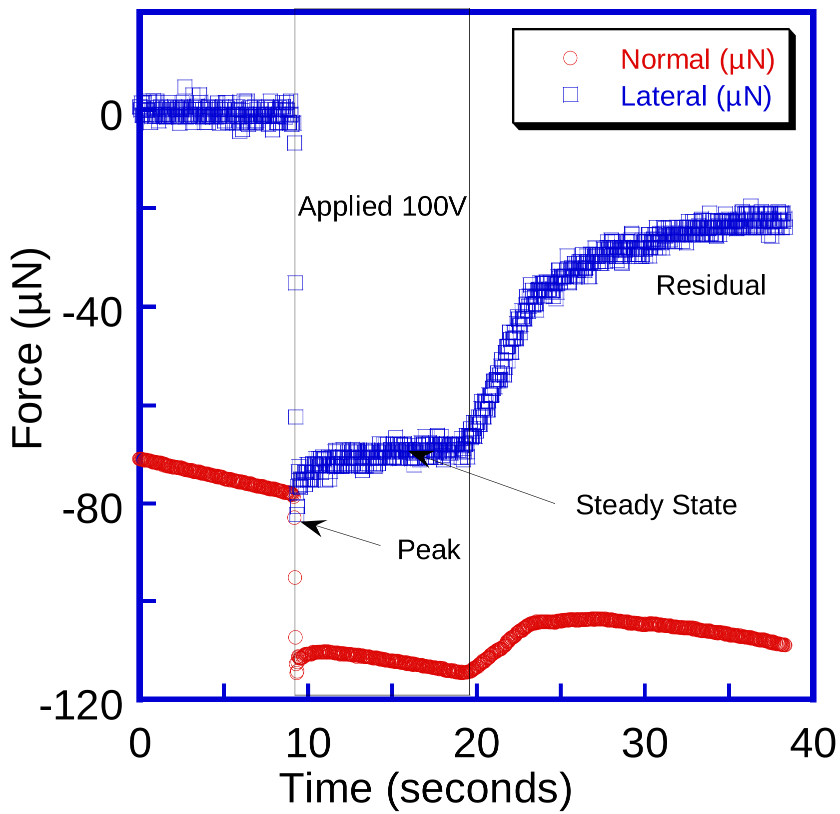
Transition from grounded to floating configuration

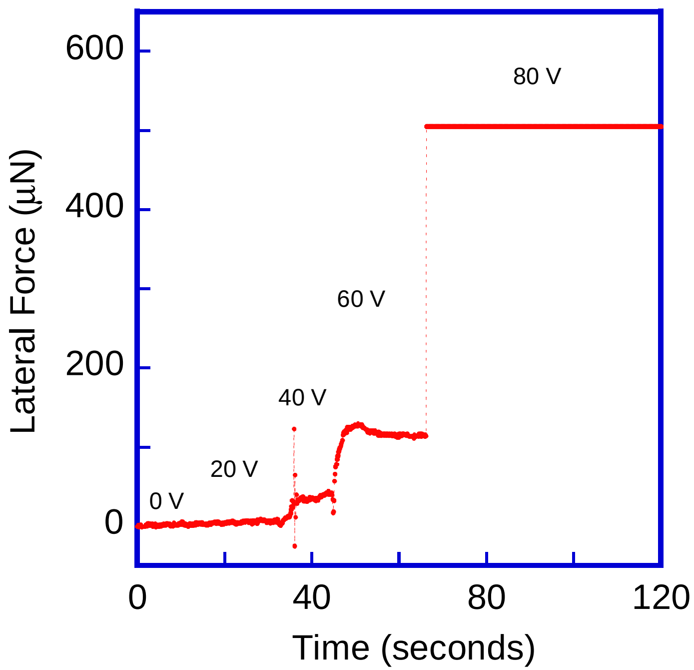
Force Measurement Configuration





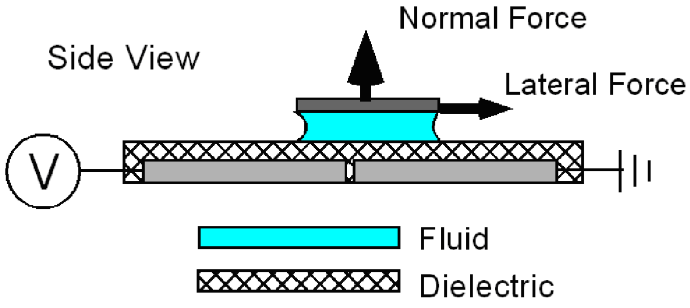
Typical Results

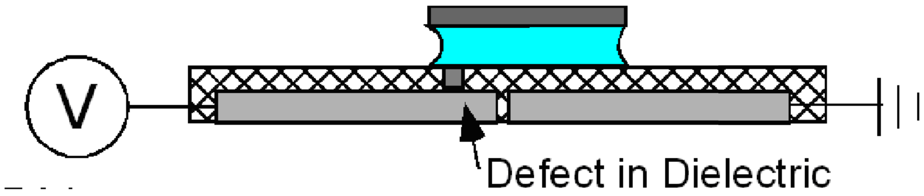




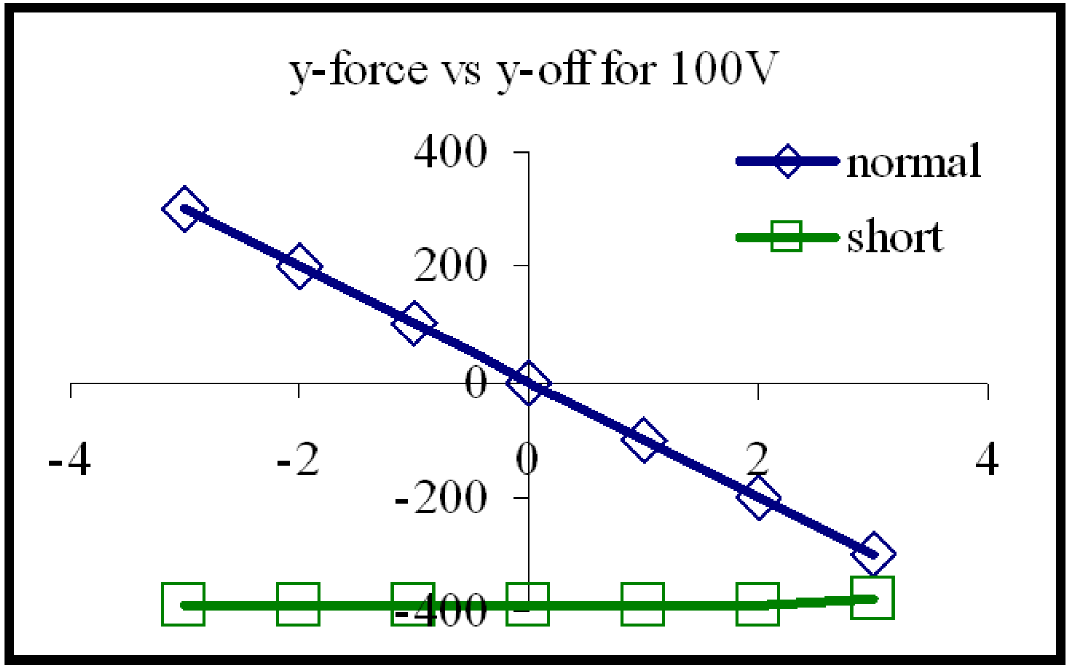
Applied Voltage (V)	Measured Force (µN)	Predicted Force (µN)	Prediction Method
20	6	11	Floating Drop
40	41	44	Floating Drop
60	113	98	Floating Drop
80	505	535	Grounded Drop

Y-force in a normal and defective dielectric layer



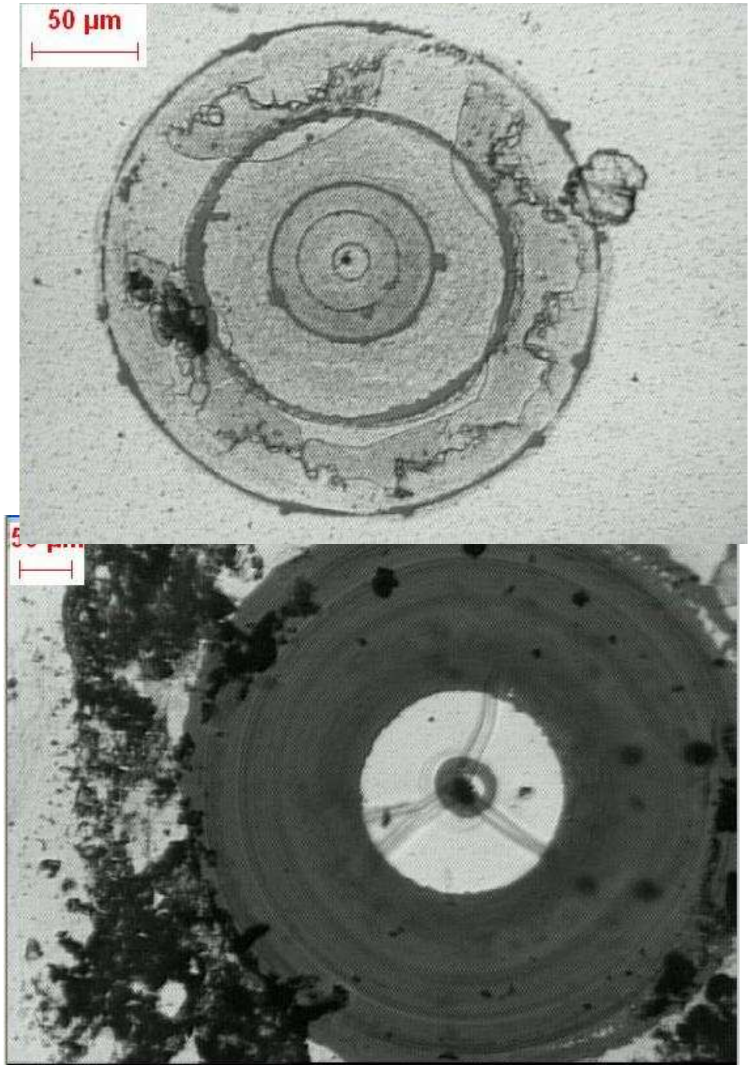


Force measurements can be used to detect defects in the dielectric.

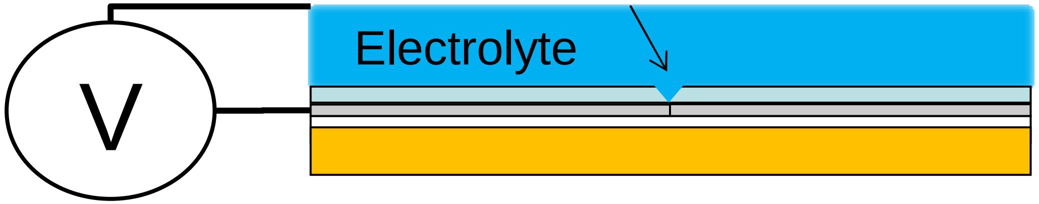


Nathan Crane, Vivek Ramadoss, USF

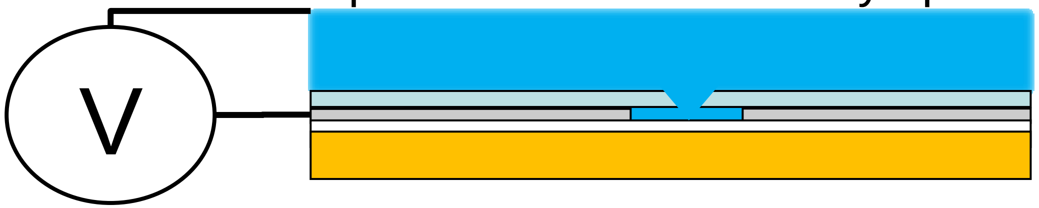
Possible Defect Mechanism



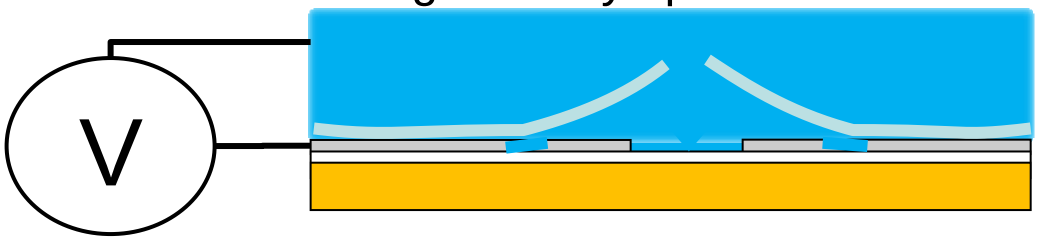
Hole forms in Cytop



Thin aluminum layer corrodes quickly
And pressure builds under Cytop



Cytop delaminates—exposing new Aluminum
Corrosion begins at Cytop/Aluminum Interface



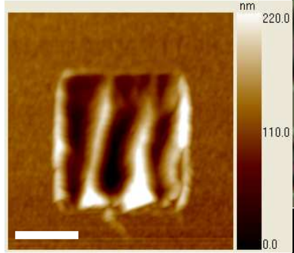
Conclusions for Electrowetting Experiments

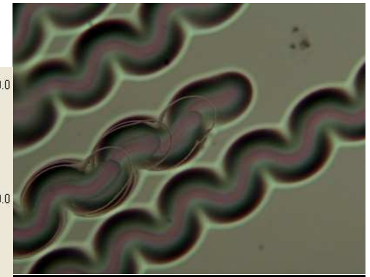
- Electrowetting oscillation under a DC voltage input
- Proposed an explanation for this behavior based on local dielectric defects
- Introduced a method for measuring electrowetting forces in 2-axes simultaneously

1. N.B. Crane, A.A. Volinsky, V. Ramadoss, M. Nellis, P. Mishra, X. Pang, MRS. Proc. Vol. 1052, DD8.1, 2008
2. N. Crane, A.A. Volinsky, P. Mishra, A. Rajgadkar, M. Khodayari, Appl. Phys. Lett., Vol. 96, pp. 104103-3, 2010
3. N. Crane, P. Mishra, A.A. Volinsky, Review of Scientific Instruments, Vol. 81, pp. 043902-7, 2010

Previously Funded Projects

NACE: "Adhesion Measurements of Thin Films in Corrosive Environments" $40K



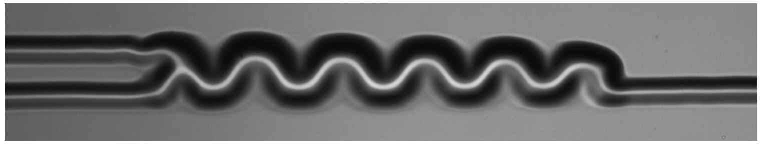


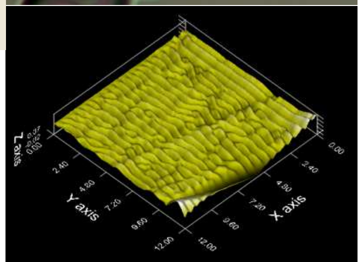
NSF: "Lab-on-a-chip Microchannels

Novel Manufacturing Method" $80K

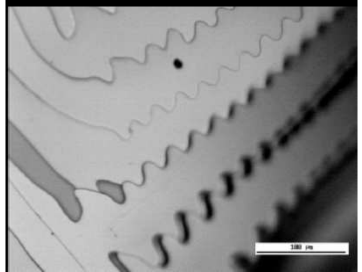
+IREE 2007 $35K

Krakow, Poland

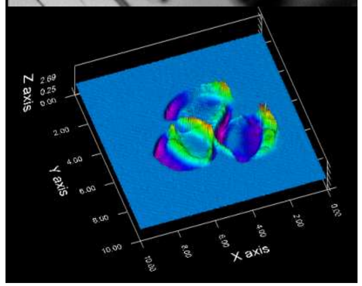




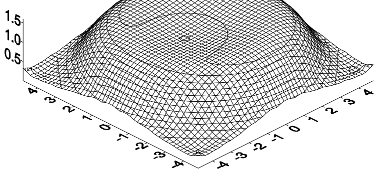
NSF: "Wear-induced Nanoripples in Single Crystals" $60K

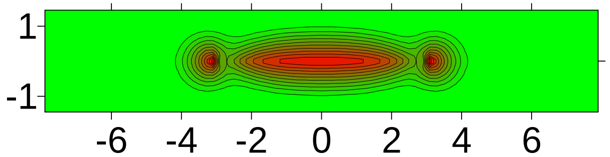


NSF: "Experimental and Computational Investigation of Fracture Patterns in Thin Films and Multilayers" $250K



+IREE 2008 $41K
Dresen, Germany

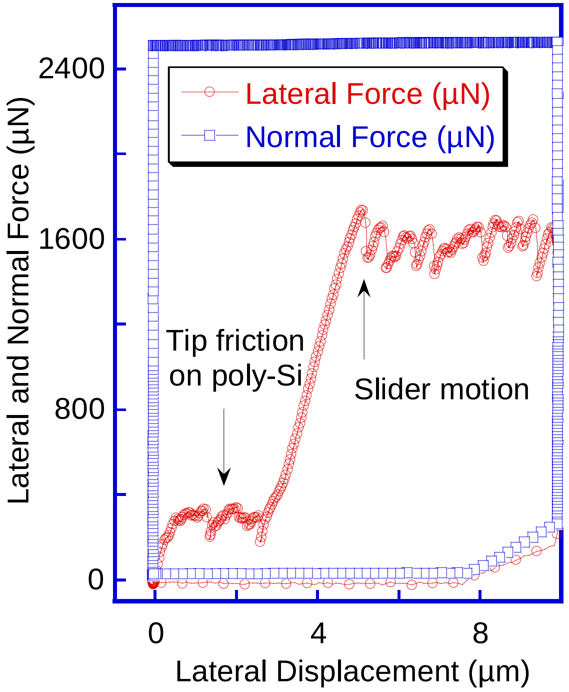


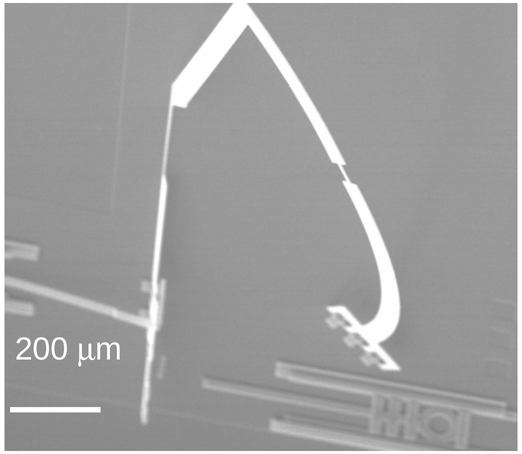


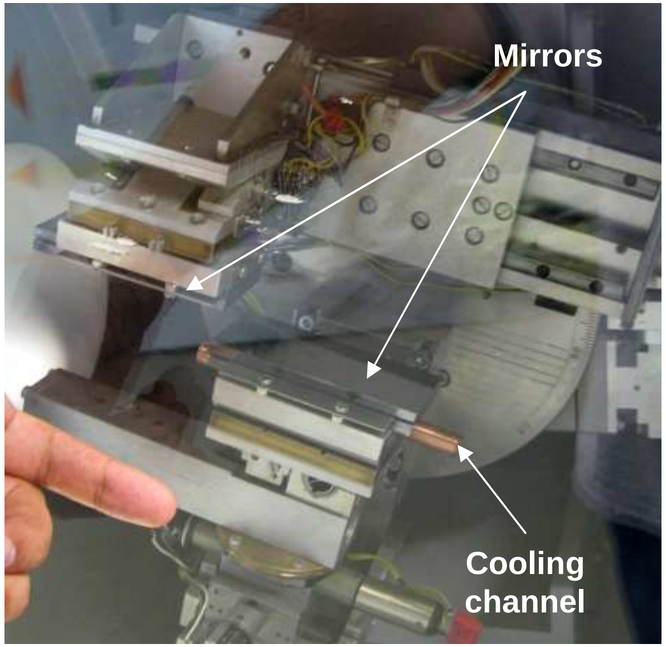
TI: "Nanoindentation and Modeling of Low-K Dielectrics for the TI Advanced Microelectronic Interconnects, their Mechanical Characterization and Reliability" $15K

Currently Funded Projects

NSF: "Uncertainty Quantification for the Kinematic Approach to Compliant Mechanism Design" Co-PI with PI C. Lusk (USF) $370K



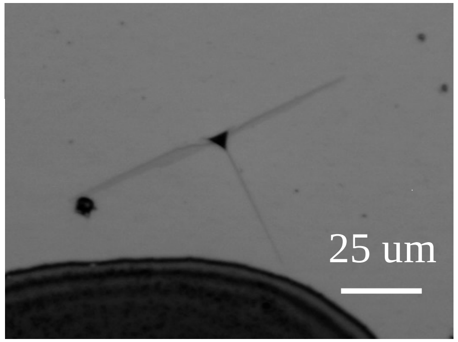




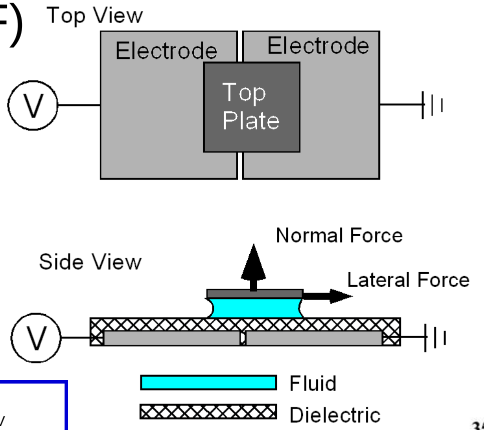
NSF: "IRES: International US-Germany Joint Study of X-Ray Optics Thermomechanical Stability and Control" $150K.

Unfunded Projects

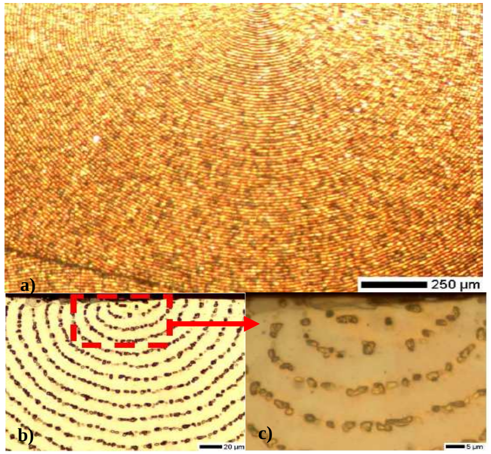
3C-SiC on Si mechanical properties with S. Saddow (USF)

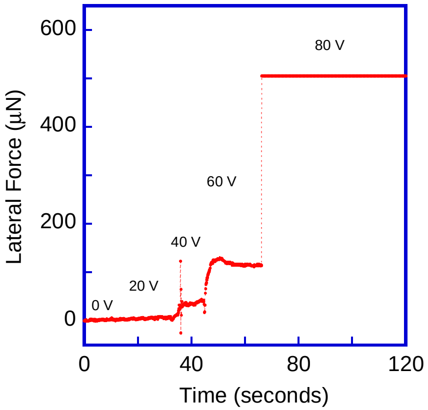


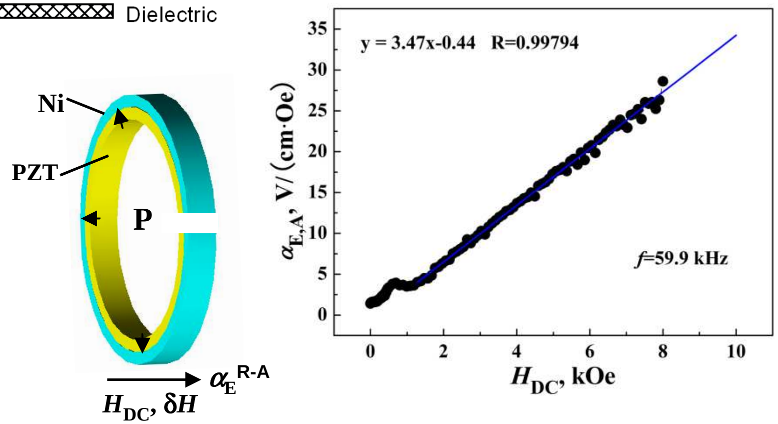
Electrowetting forces with N. Crane (USF)



Pattern formation in Au/Cr/Si films with D. Gracias (JHU)





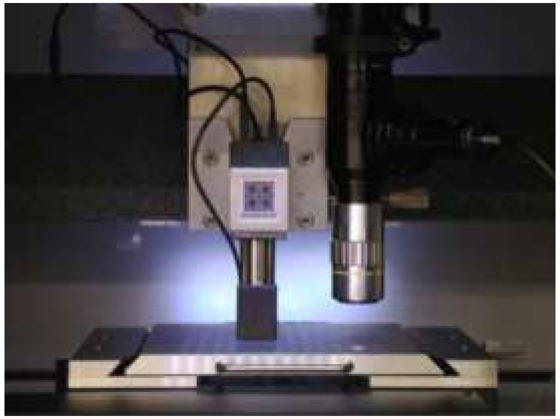


Chromium oxide coatings on steel with Qiao, Gao and Pang,

Magnetoelectric layered composites with D.A. Pan (USTB, China)

Summary

Hysitron Triboindenter™ and other equipment available at USF for collaborative research



We provide value added analysis.