SAND2008-3084P

1513 Microsystems Thermal/Fluids Experimental Capabilities and Results

Microfluids and Microrheology

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Microscale Heat Transfer

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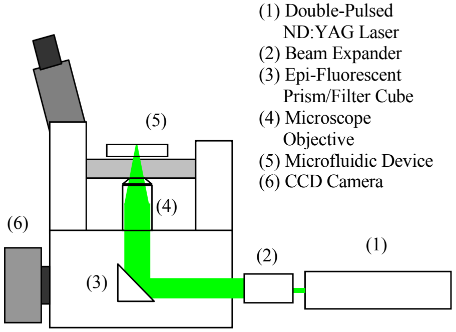
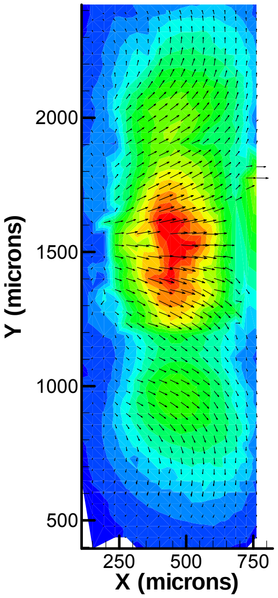
Outline

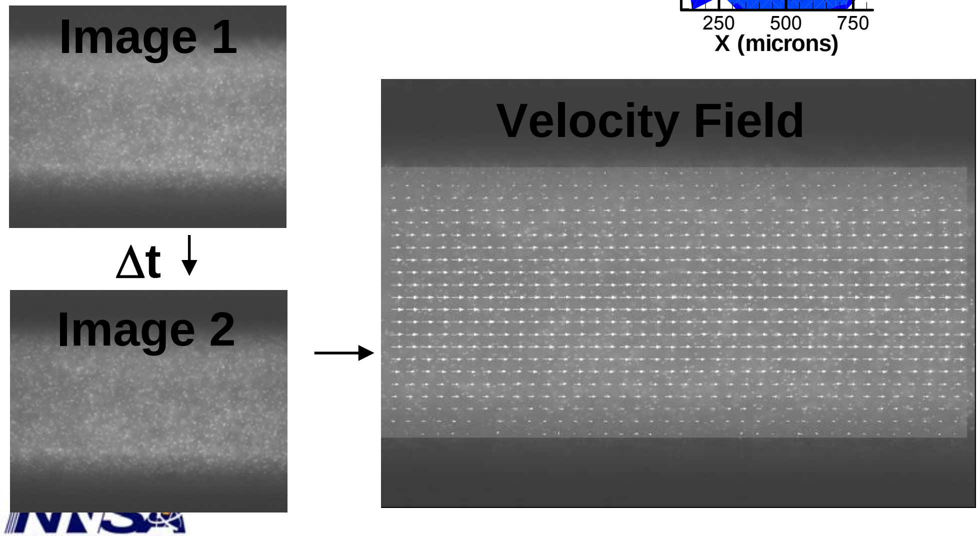
- 1513 Experimental Capabilities
- Thermal Actuator Experiments
  - Model Validation
  - Discovery
- Thermal Conductivity Measurement
- LDRD Proposal

Micro Particle-Imaging Velocimetry



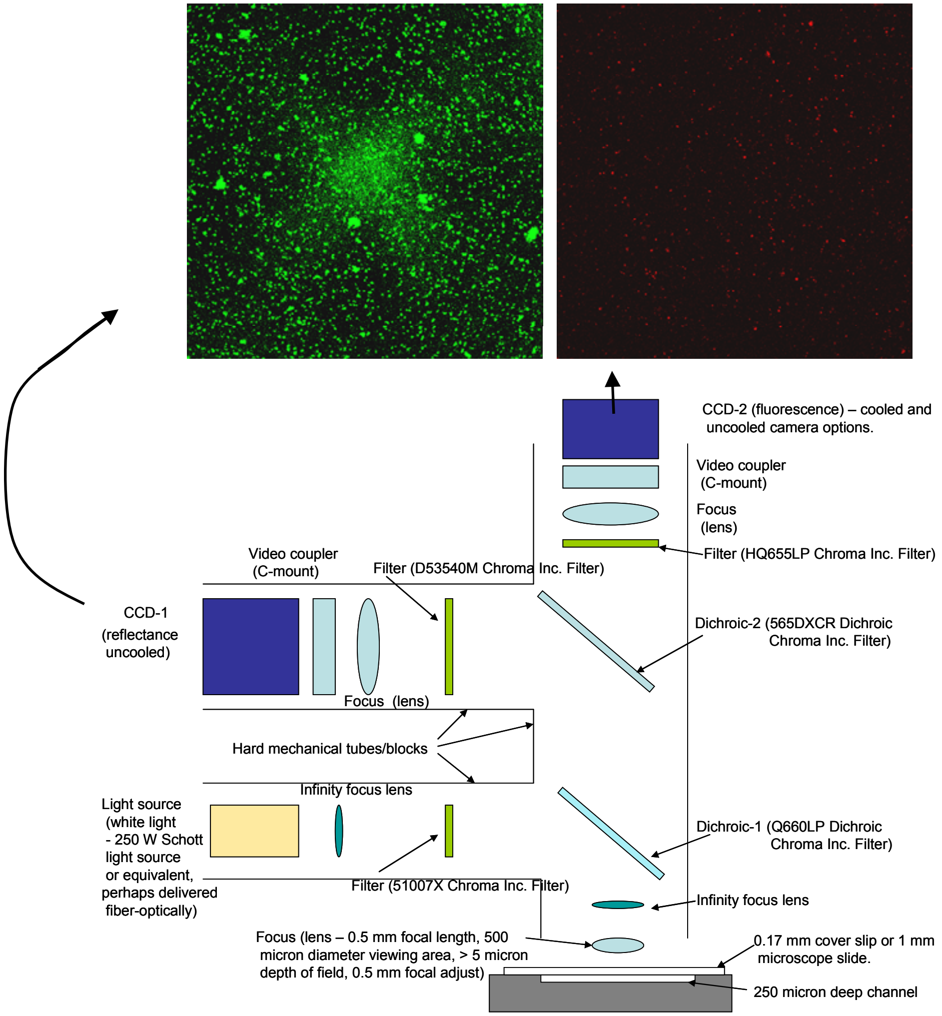
Micro-PTV system.

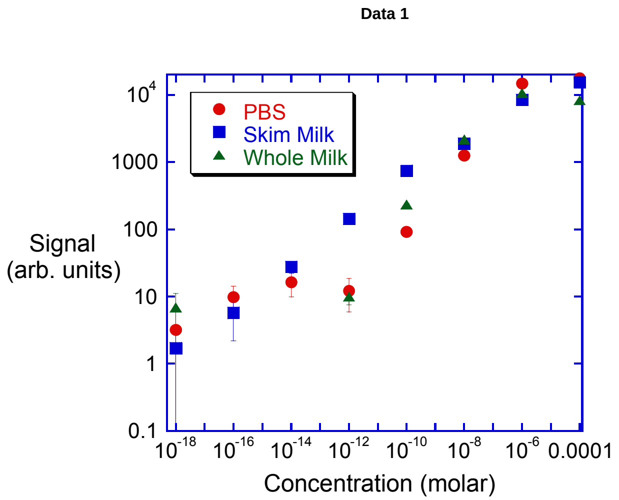


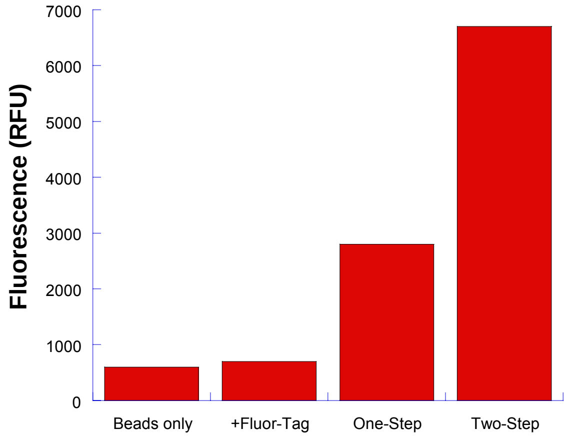


- Entire flowfield illuminated by pulsed light source
- Focus plane and measurement depth determined by optics
- Images of fluorescent microparticles captured at known time delay
- Small regions in image compared with cross correlation technique to determine displacement of particles between frames
- Spatial resolution of 1x1x1 micron attainable
- Velocities of microns/minute to kilometers/sec measurable
- Advanced PIV capabilities include:
- Dynamic interface tracking
- 2-Color PIV
- Microscale Particle-Tracking Velocimetry in Air

Optical Characterization of Bead-Based Detection Systems







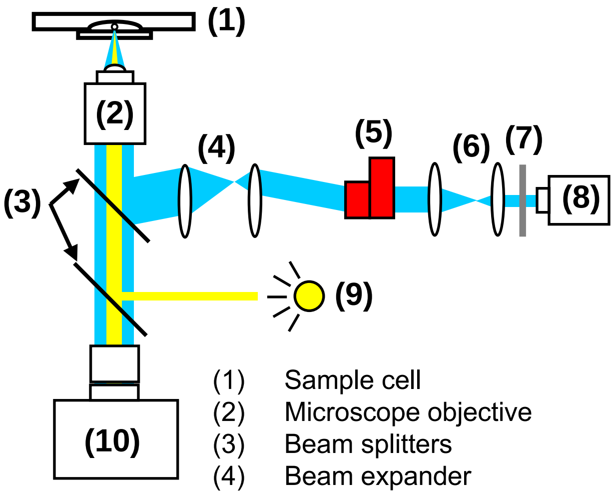
BoNT/A Simulant "Sandwich" Reactions

2-Dye Micro-PLIF for temperature measurement

- This technique measures temperature by gathering the fluorescence signal of two fluorescent dyes, one temperature *sensitive (Rhodamine B)*, the other temperature *insensitive (Rhodamine 110)*
  - Using two dyes simultaneously results in an *in situ* calibration
  - Measured intensity ratio is dependent only on temperature
  - Results in instantaneous temperature field data
  - Measurement error less than ±0.25 K
- Using Scanning Confocal Microscope results in high depth resolution, decreased background light intensity, and highly selective wavelength selection.
  - Confocal pinhole design virtually eliminates background signal and allows for 1-2 micron imaging depth
  - Dual-grating system allows for bandwidth selection with nanometer accuracy

Optical Tweezers

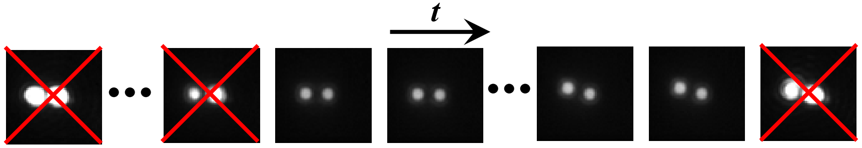
- Focused laser beam captures colloidal microparticles in aqueous and non-polar solvents
- Time-shared traps manipulate multiple particles



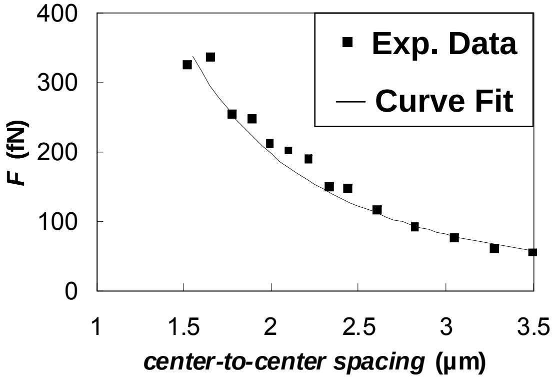
(1)	Sample cell
(2)	Microscope objective
(3)	Beam splitters
(4)	Beam expander
(5)	Acousto-Optic Deflector (AOD)
(6)	Beam expander
(7)	Neutral density filter
(8)	Nd:YAG laser
(9)	Mercury arc lamp
(10)	High speed camera

Applications:

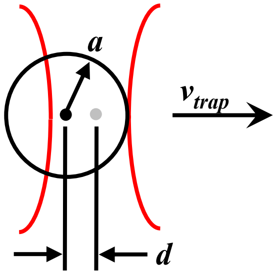
- fN-scale interparticle force measurements from relative diffusion of multiple particles



- Curve fitting to *F* determines zeta potential and screening length of particles



- Rheological measurements from displacement of particle from trap center



η = fluid viscosity

k_{trap} = trap stiffness

$$\boxed{\eta = \frac{k_{trap} d}{6\pi v a}}$$

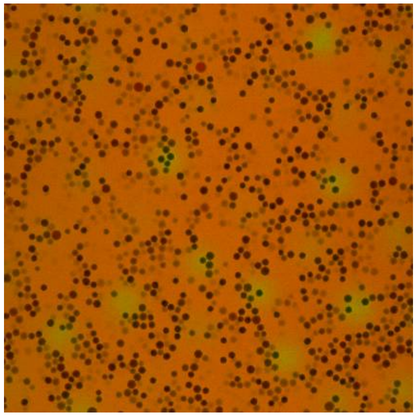
Basic Material Characterization

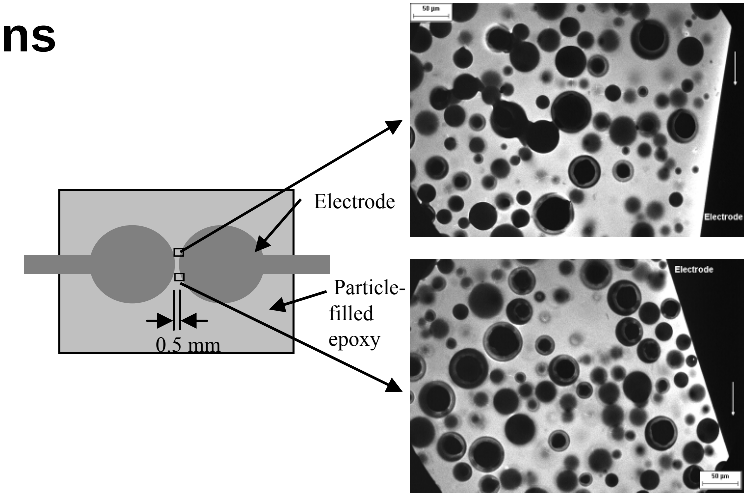
- Rheology
  - Measurement of viscosity and non-Newtonian properties
  - Experience with complex liquids such as suspensions & polymers, wide temperature ranges, various flow configurations
- Surface tension, Wetting properties
  - Pendant drop, Wilhelmy plate, Du Nouy ring techniques
  - Feed Through Goniometer to study dynamic wetting of complex materials
  - Experience with testing complex liquids including suspensions and high viscosity polymers
  - Measurement with adsorption/desorption of surfactants

Confocal Microscopy

- Capability to image three dimensional structures with high depth resolution. Three dimensional structures are constructed from stacks of 2D cross sections. Dynamic 3D imaging for slow processes (50 - 256x512 images in <30sec) Also provides advanced 2-D fluorescence microscopy with improved signal to noise resolution.

➡ **Microstructure of suspensions**

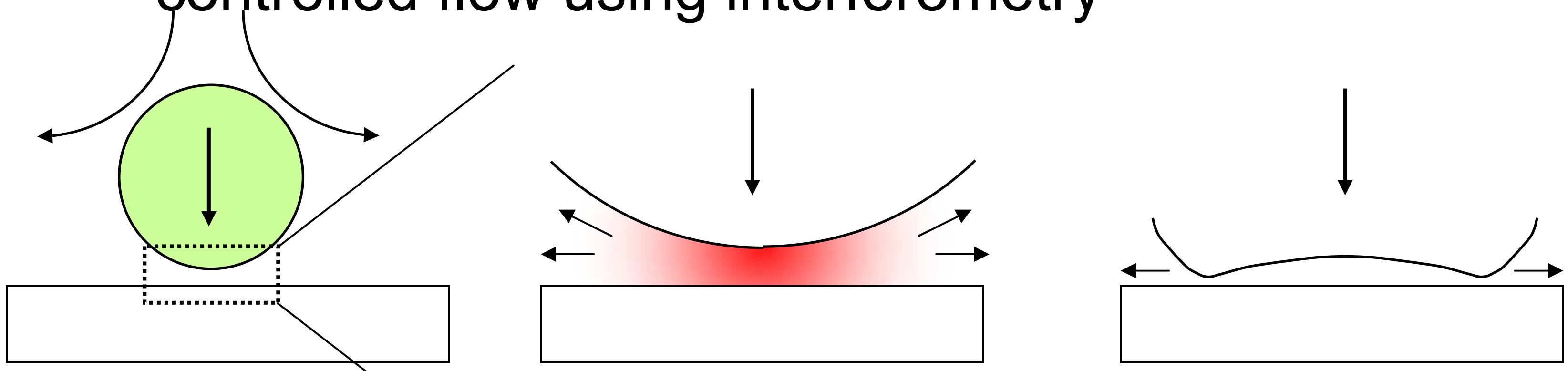


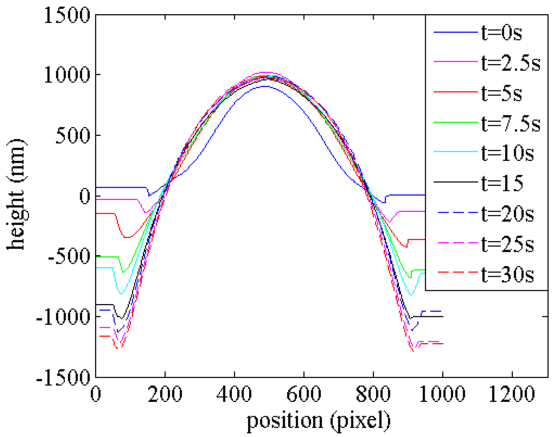


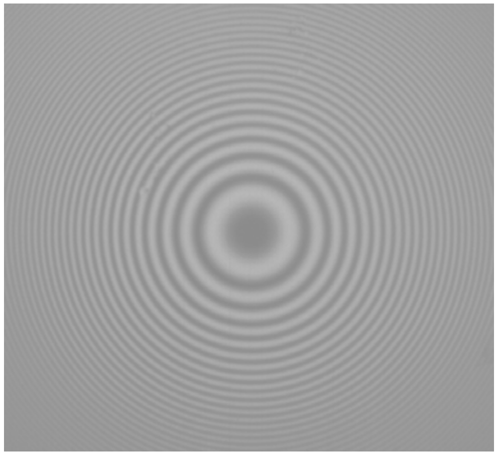
➡ **Chemical segregation, general fluorescence microscopy, particle tracking, temperature measurement**

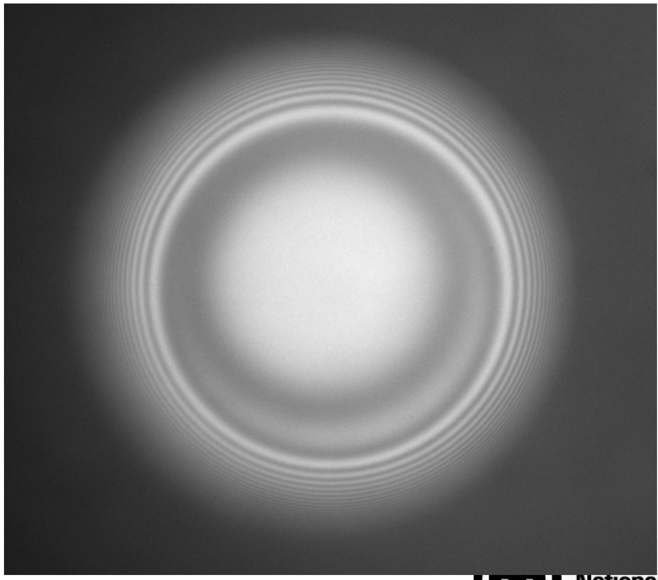
Emulsion Experience: Coalescence

- Study of coalescence in liquid-liquid emulsions under controlled flow using interferometry







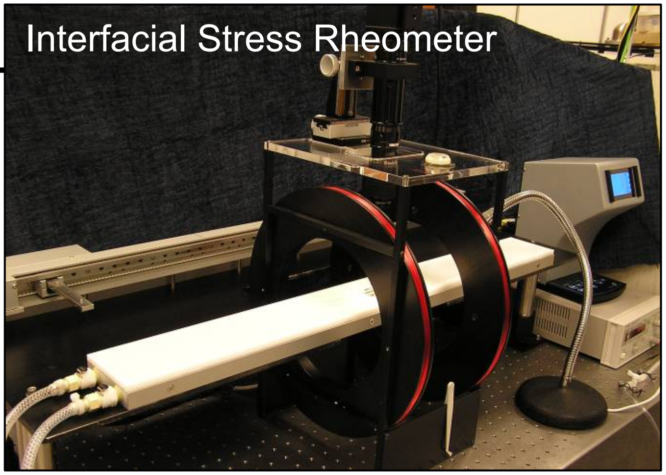


Interfacial Rheology

- Surface active agents, such as surfactants and micro/nanoparticles, can impart rheological properties to interfaces
- Interfacial rheology plays an important role in the stability of foams and emulsions and in the processing of Langmuir-Blodgett films
- We have the ability to measure the rheology for *shear* (Interfacial Stress Rheometer) and *dilatational* deformations (Surface Dilational Rheometer – not shown)
- Molecular structure in monolayer films can impart visco-elastic rheology

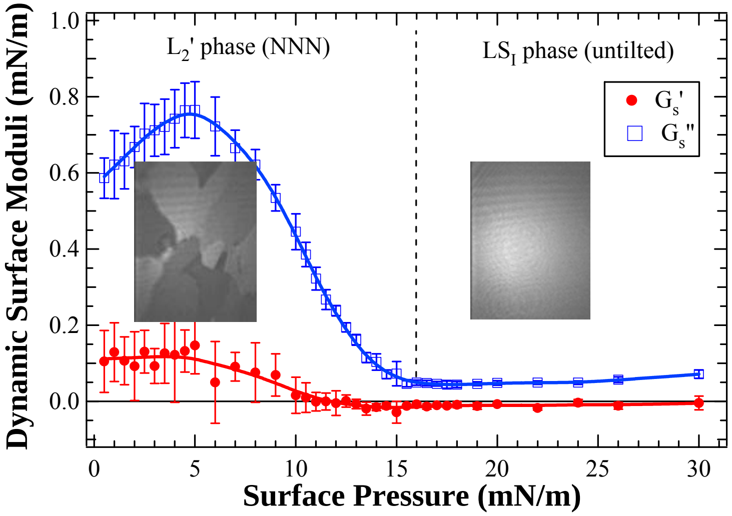
References

C.F. Brooks et al., *Langmuir* **1999**, v15, p2450.
C.F. Brooks et al., *Langmuir* **2002**, v18, p2166.


Interfacial Stress Rheometer


Magnetic Needle at Air-Water Interface

Surface Rheology of a Monolayer of CH_3-$(CH_2)_{19}$-OH



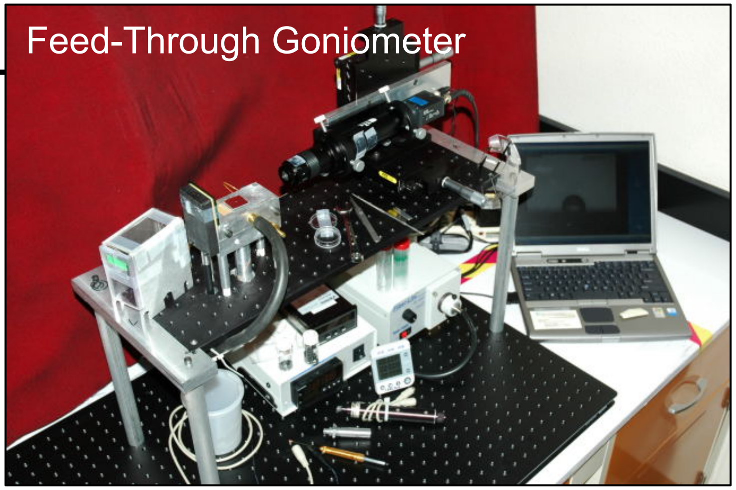
Dynamic Contact Angle

- Modeling of multiphase flow processes in solid geometries requires knowledge of the velocity dependece of the three-phase contact angle (in addition to interfacial tension and static contact angle).

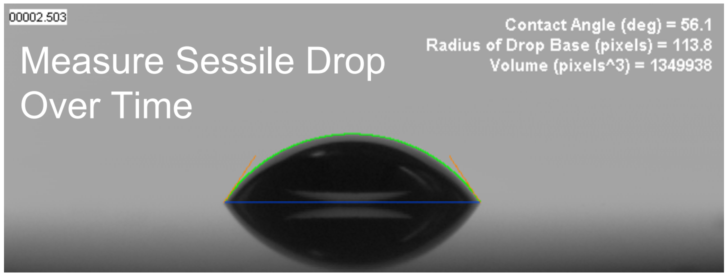
- Velocity dependence of the contact angle is important in dynamic processes with small length scales, where capillary/interfacial forces dominate
  - capillary wicking in microfluidic channels
  - Injection molding and encapsulation/potting
  - coating and printing
  - water removal in PEM fuel cells

- The Feed-Through Goniometer measures the contact angle as a function of velocity as liquid is injected through a hole in the test substrate

- Can be used to measure
  - smooth ideal surfaces (e.g. silicon) and directionally dependent wetting on rough surfaces (e.g. machined Al)
  - Simple liquids, polymers, suspensions, and curing epoxies



Feed-Through Goniometer



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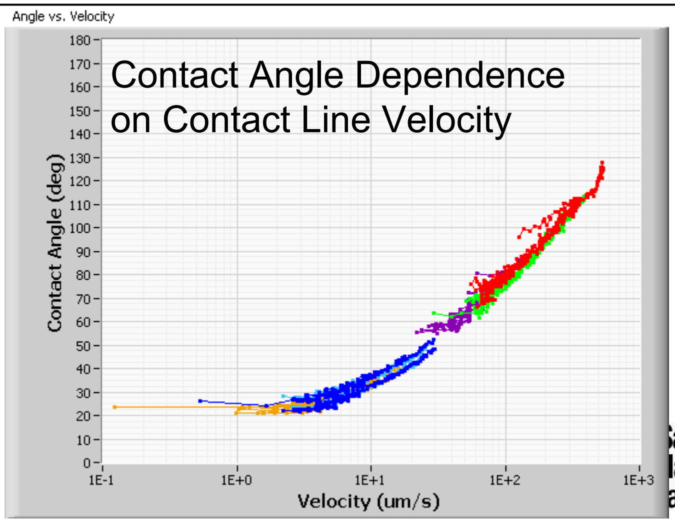
Contact Angle (deg) = 56.1
Radius of Drop Base (pixels) = 113.8
Volume (pixels^3) = 1349938

Measure Sessile Drop Over Time

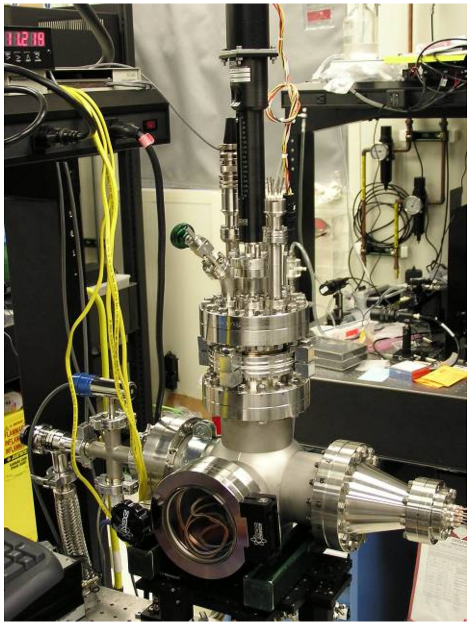


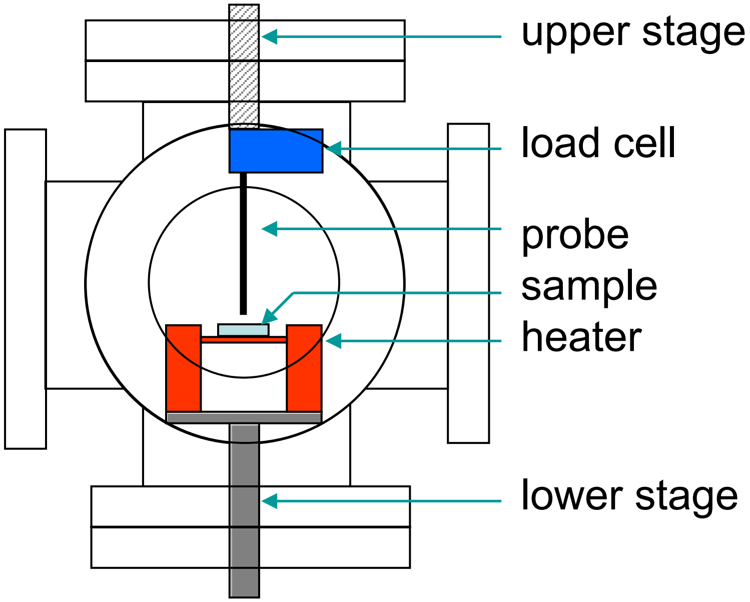
Contact Angle Dependence on Contact Line Velocity

References
C.F. Brooks et al., *Langmuir* **2006**, v22, p9928.

High Temperature Interfacial Characterization

High Temperature, controlled vacuum / atmosphere capability to probe the interfacial properties of liquids





- Micro-indentation (anodized coating over molten aluminum)
- Surface tension measurement
- Porous imbibition

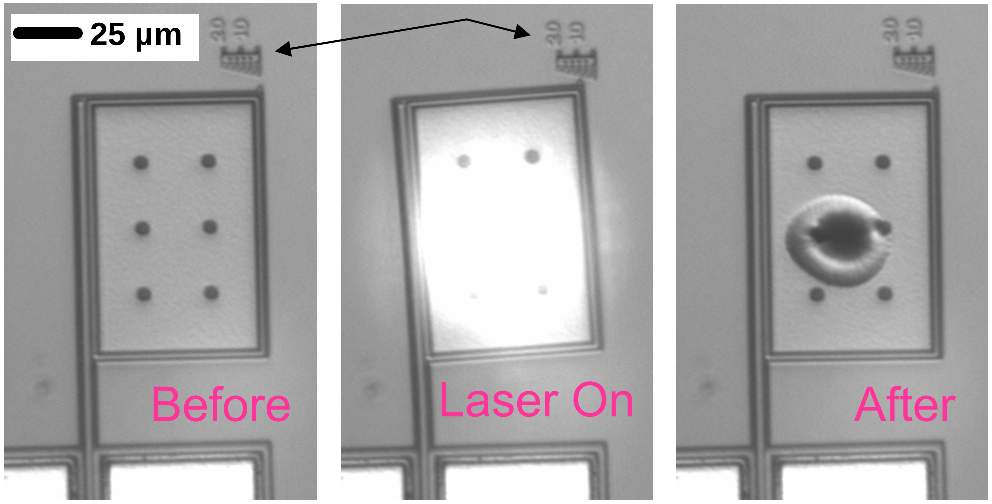
Optical Testing of Microsystems

Optical Testing Capabilities

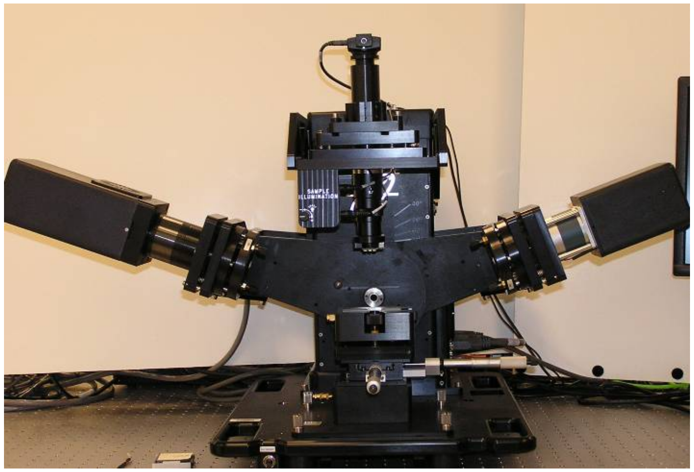
- Able to image microsystems while heated with a laser
- Current heating lasers include 808 nm and 532 nm CW lasers up to 2.5 Watts
- Determined the performance of optically powered thermal microactuators
- Investigated the damage characteristics of polysilicon MEMS actuators and test structures
- M2000 Multiwavelength Ellipsometer, J.A. Woollam
- 245 nm to 1690 nm
- 45 deg. to 90 deg. angles
- Temperature stage – room temperature to 300 deg. C

Laser Powered Flexure Thermal Actuator

Scale Indicates Motion



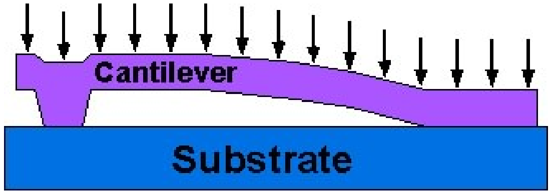
M2000 Multiwavelength Ellipsometer



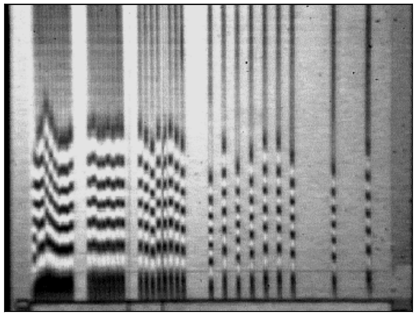
Thermomechanical Effects in Microsystems

Thermomechanical Testing Capabilities

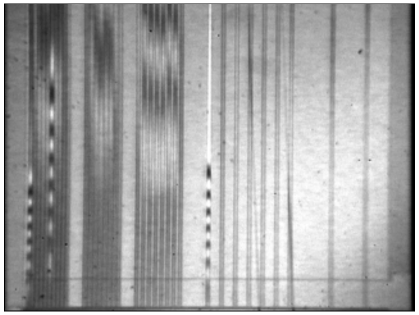
- Temperature stages with optical access up to 1000˚C
- One stage has vacuum and gas capabilities.
- Nd:YAG laser repair of adhered microcantilevers
- Interferometric imaging



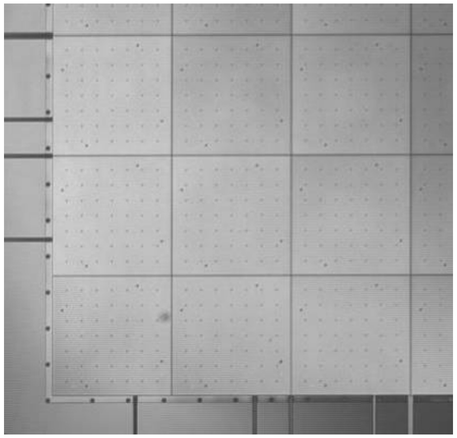
Schematic of an adhered cantilever irradiated by a laser for repair



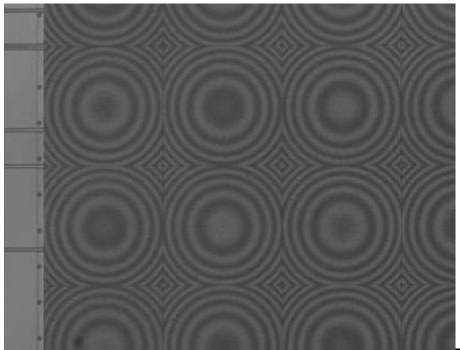
Prior to laser irradiation

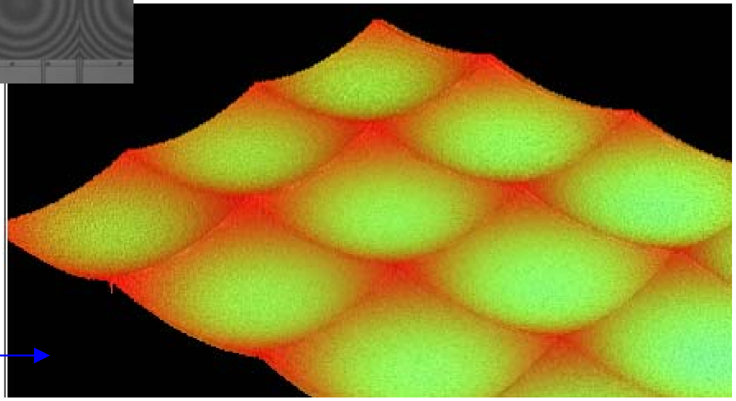


After laser irradiation



T = 26 deg. C before temperature cycle





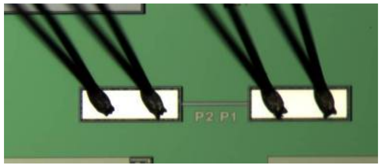
T = 27 deg. C after temp. cycle peak to valley ~1 μm

Thermal Conductivity Measurements

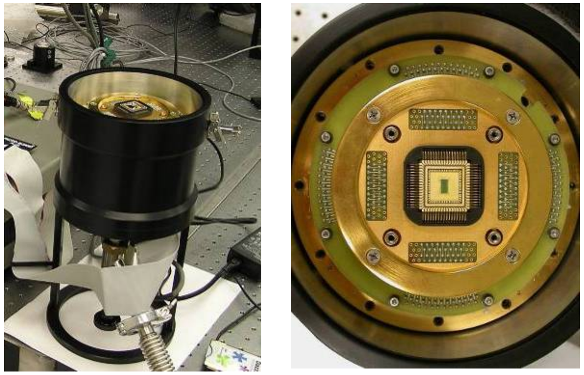
Thermal Conductivity Measurement Capabilities

- Steady state electrical resistance technique, have electronics to make 3ω measurements
- Used bridge test structures fabricated with standard micromachining process
- Two cryostats, one with temperature range from 77K to 350K and the other from 77K to 700K
- Other sample configurations possible including thin films if a metal sensor can be deposited on the film

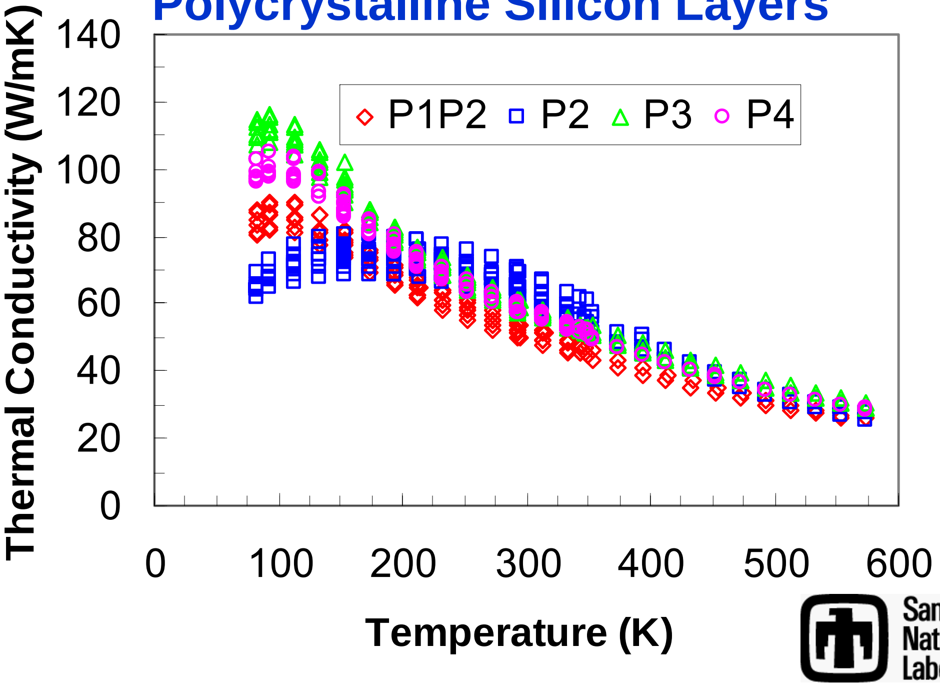
Polysilicon Bridge Test Structures



Cryostats



Thermal Conductivity of SNL SUMMIT Polycrystalline Silicon Layers

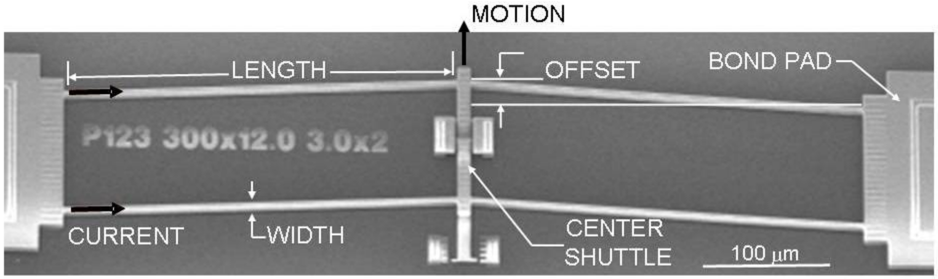


Thermometry for Microsystems

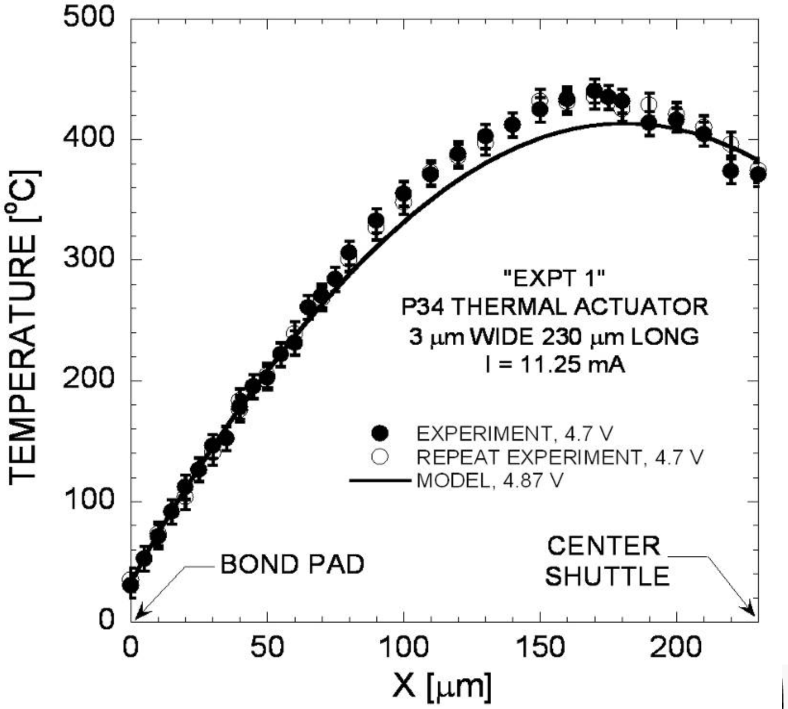
Microthermometry Capabilities

- Non-invasive and non-contact
- In-plane spatial resolution ~1μm
- Raman microscopy for semiconducting and insulating materials including single crystal and polycrystalline silicon, SiC, and GaN
- Thermal reflectance technique for metals
- Primarily, steady state measurements due to long signal collection times.
- Diagnostics for transient measurements for periodic operation.
- Reported first spatially resolved temperature measurements for an electrothermal microactuator.

MEMS Microthermal Actuator



Actuator Beam Temperature Profile

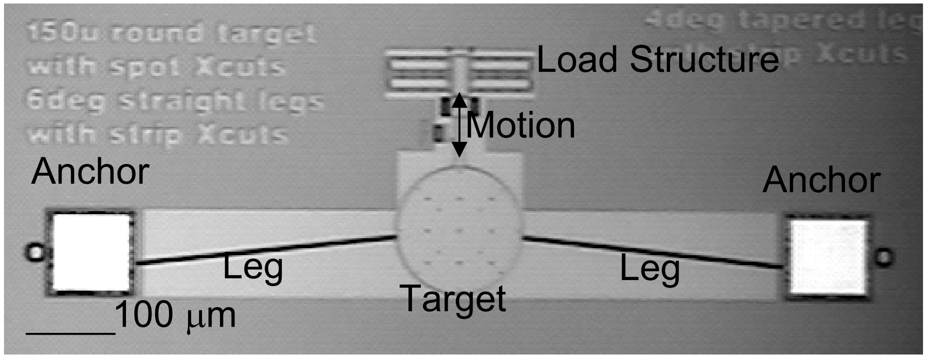


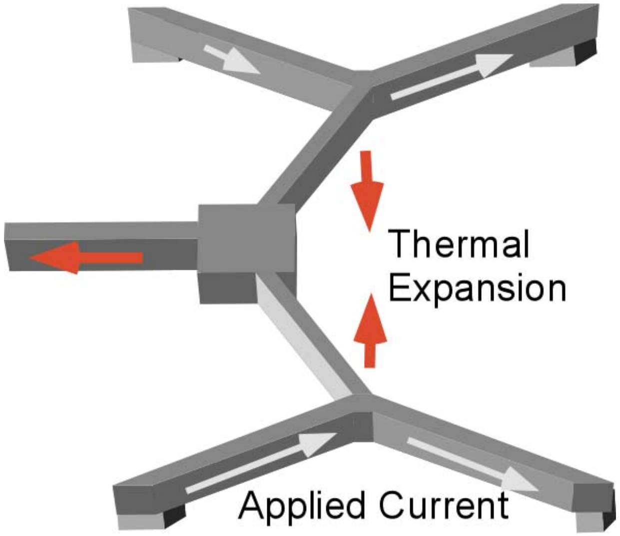
Outline

- 1513 Experimental Capabilities
- Thermal Actuator Experiments
  - Model Validation
  - Discovery
- Thermal Conductivity Measurement
- LDRD Proposal

Microthermal Actuators for Microsystems

- Thermal Actuators (TAs) have much higher forces (200 μN) than electrostatic comb drives (20 μN).
- 100% compatible with SUMMiT-V™ process
- More resistant to stiction than lower force actuators.



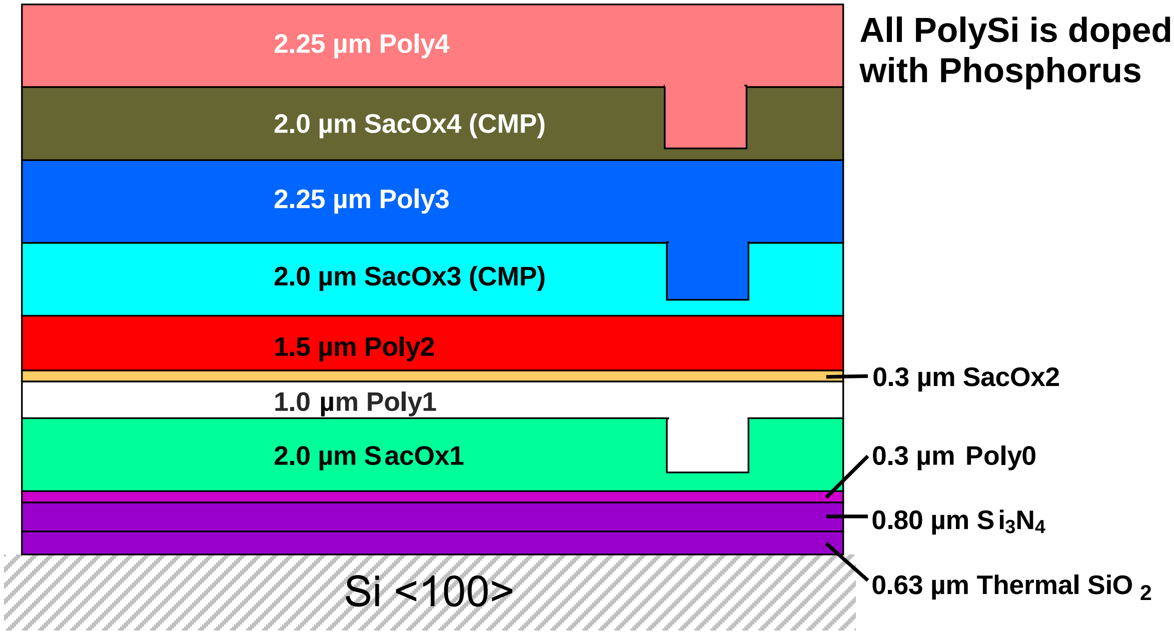


- Opto-Thermal Actuators (OTAs) use optical energy instead of electrical energy.
- Have no electrical connections to the outside world
- Compatible with radiation and high temperature environments
- Will not electrostatically attract particles.

SUMMiT™ V

Sandia's Ultra-planar Multi-level MEMS Technology

SUMMiT™V Layer Descriptions



2.25 µm Poly4

2.0 µm SacOx4 (CMP)

2.25 µm Poly3

2.0 µm SacOx3 (CMP)

1.5 µm Poly2

0.3 µm SacOx2

1.0 µm Poly1

2.0 µm SacOx1

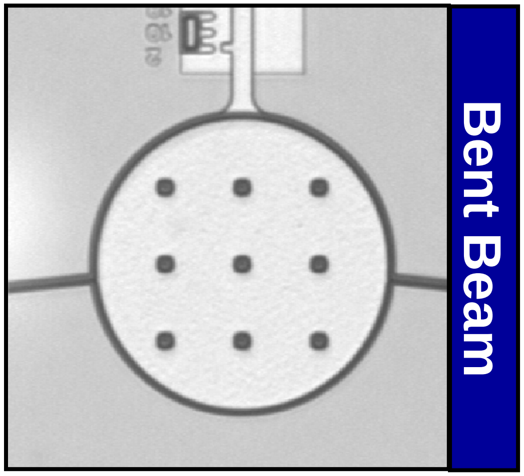
0.3 µm Poly0

0.80 µm Si_3N_4

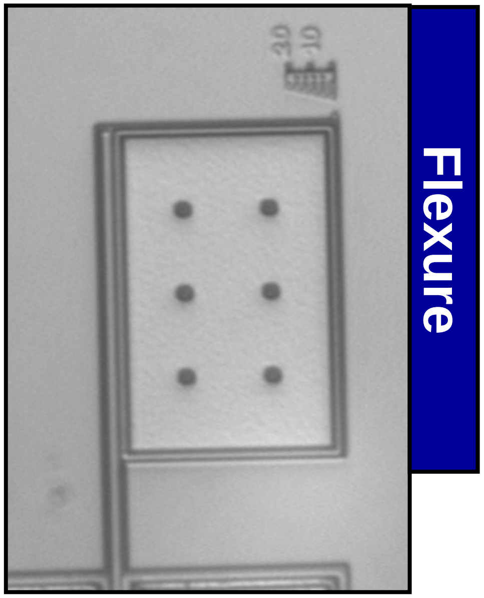
0.63 µm Thermal SiO_2

Si <100>

All PolySi is doped with Phosphorus



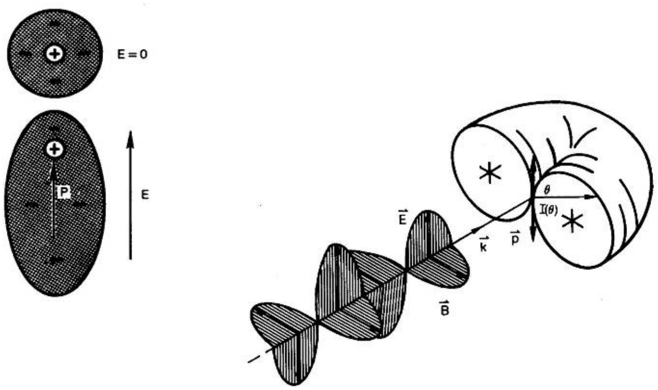
Bent Beam



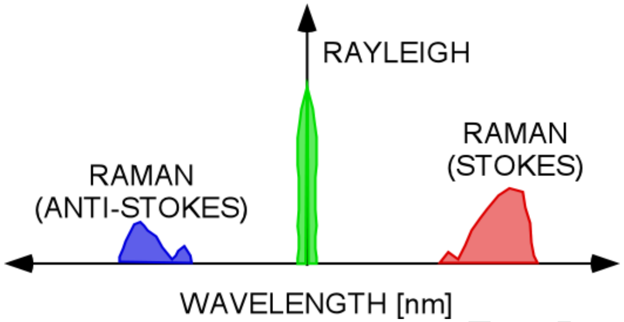
Flexure

- 5-level sacrificial micromachining
- Actuators use top two layers (Poly3 and Poly4)

Temperature Measurements Using Raman Microscopy – Description



Light Scattering by a Laser-Induced Dipole



Light Scattering Spectrum $E_s \sim P$

- Light is scattered by <u>inducing</u> a <u>radiating dipole</u> in the scattering medium.
- The scattered light field, E_s is proportional to the strength of the induced dipole moment, *P*.

$$E_s \sim P \sim \alpha\, E_L \sim \alpha \cos \omega t$$

- The polarizability, α, is a measure of how readily a medium is “polarized” (how easily light induced dipoles) and α can be expanded in terms of the equilibrium and ‘vibrating’ positions of the molecules in the scattering medium, in this case a solid-state lattice.

$$\alpha = \alpha_o + (\partial\alpha/\partial Q)Q$$

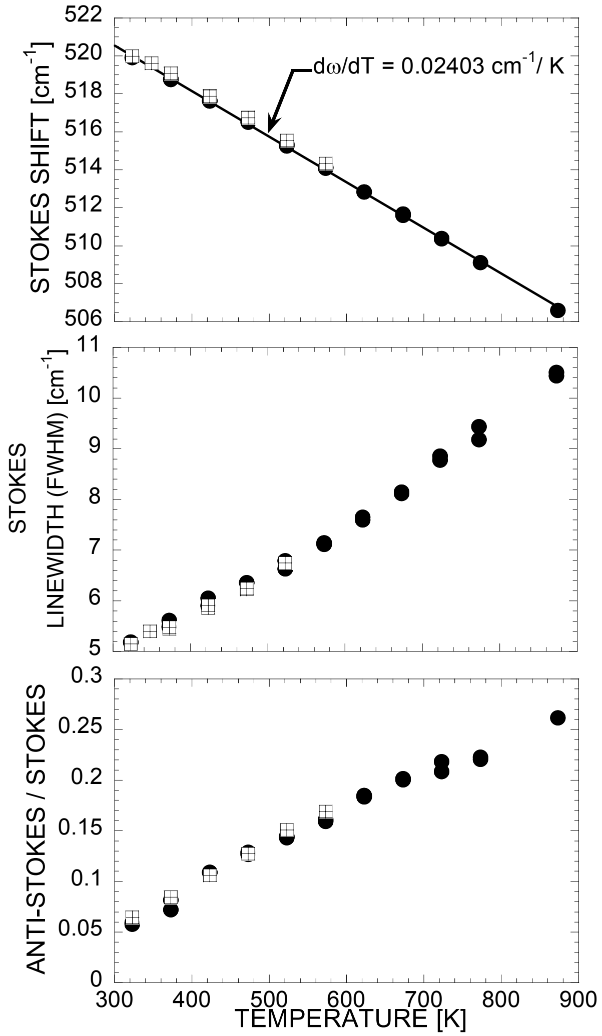
- If the lattice vibrates at a certain allowed phonon frequency, Ω, then $Q \sim \cos \Omega t$ and

$$E_s \sim \alpha_o \cos \omega t + (\partial\alpha/\partial Q)\,[\cos(\omega + \Omega)t + \cos(\omega - \Omega)t]$$

Rayleigh Scattering

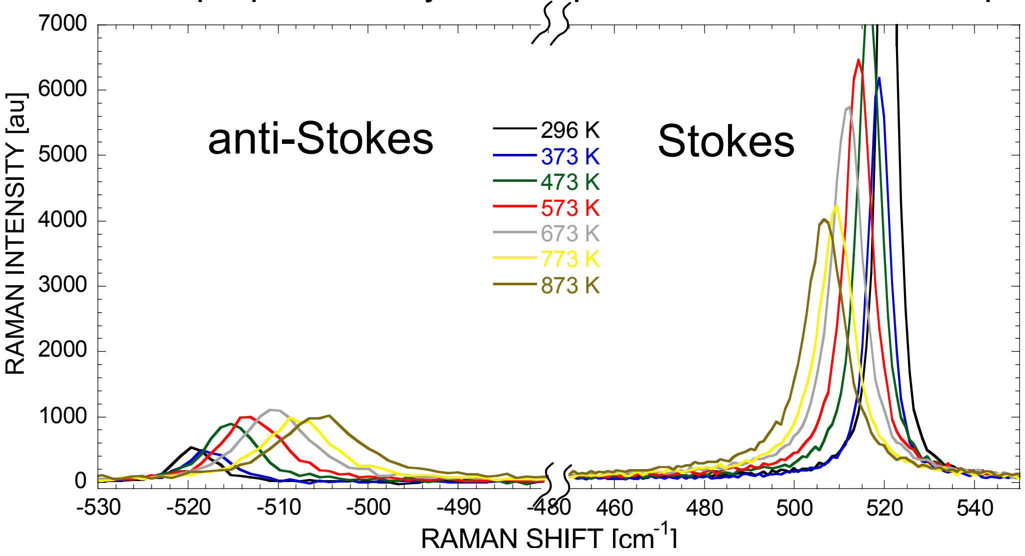
Raman Scattering

Polycrystalline Silicon Raman Spectra: Temperature Sensitivity



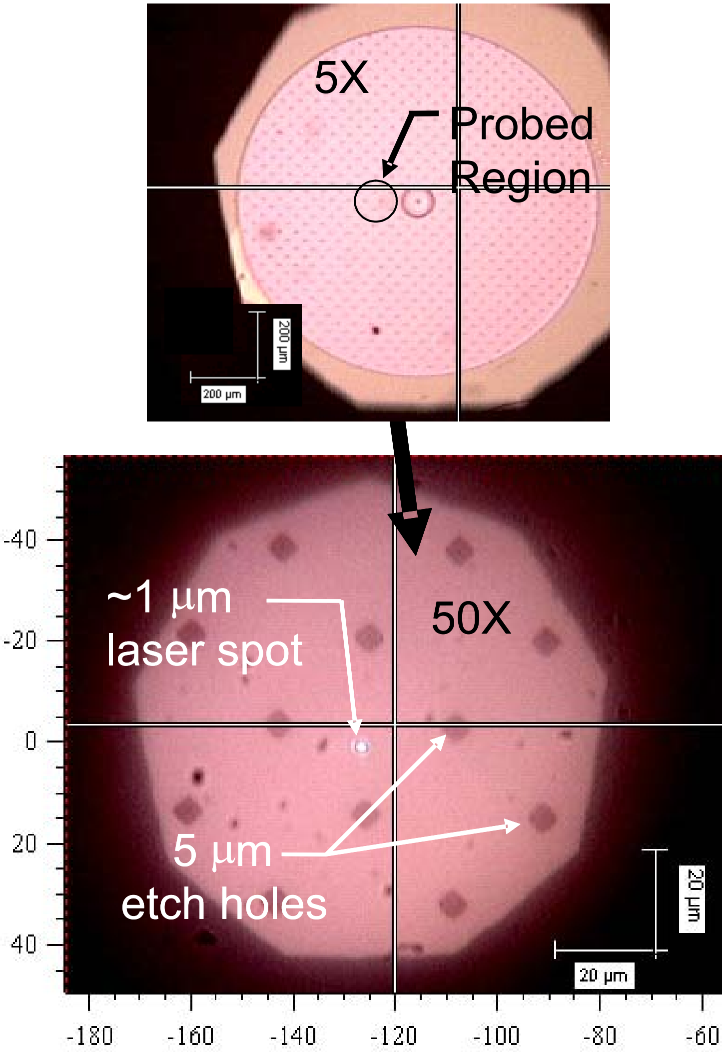
Temperature Sensitive Properties

- Temperature-dependent information extracted from PolySi Raman Spectra.
- Stokes (red-shifted) peak position – Shifts with increasing temperature due to lattice expansion.
- Stokes linewidth (full-width at half maximum) – increases with temperature due to increased optical phonon relaxation time.
- Ratio of Stokes to anti-Stokes (blue-shifted) areas -- Increased with temperature as a result of increasing population of thermally excited optical phonons.
- Some of these properties may also depend on stress and/or doping.



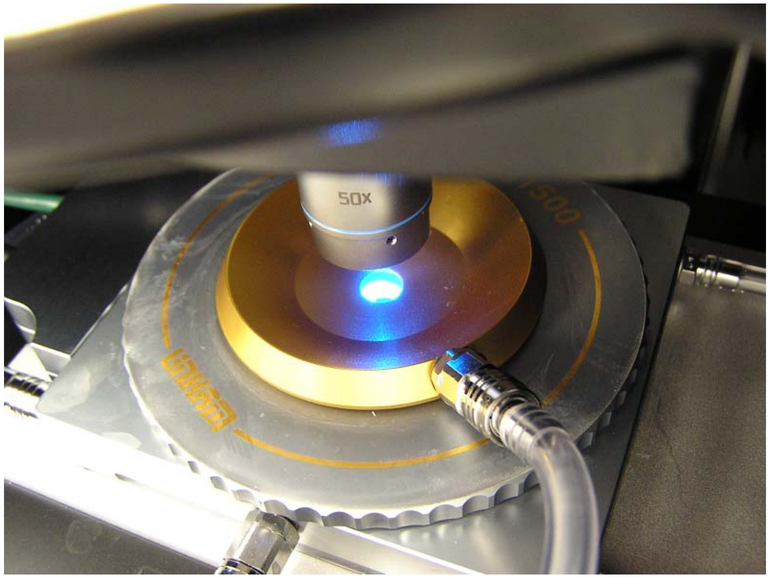
Raman Spectra from PolySi Part

Micro-Raman Probing of Polysilicon

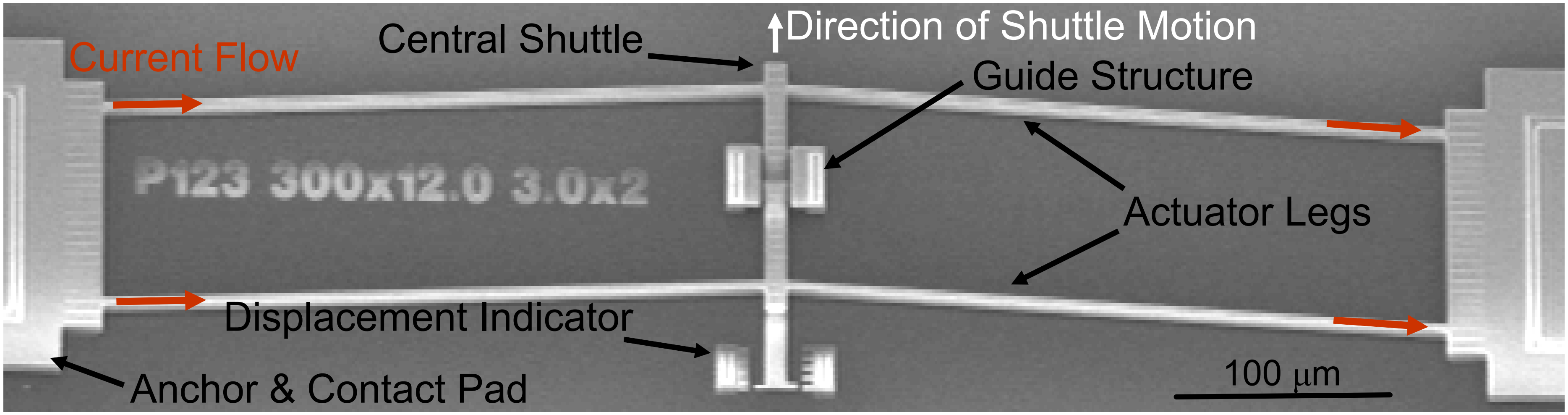


Polysilicon Surface under Raman Microscope

Laser-Illuminated Sample in Hot Stage



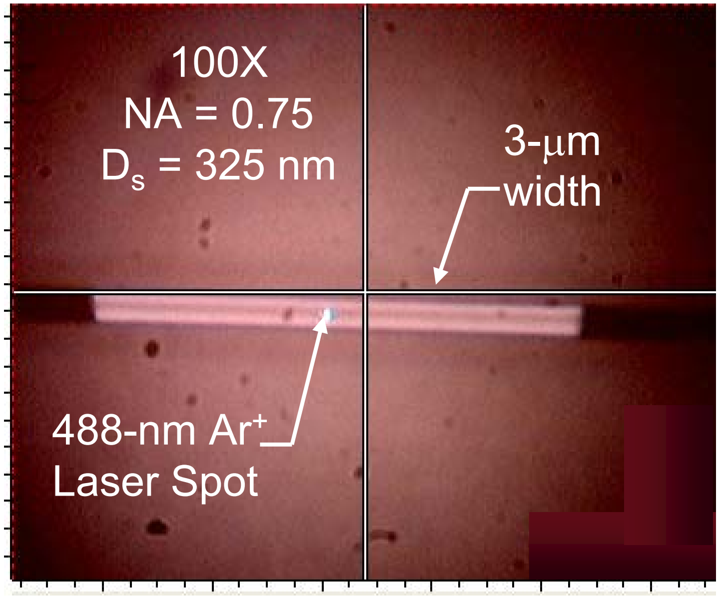
MEMS Thermal Actuators



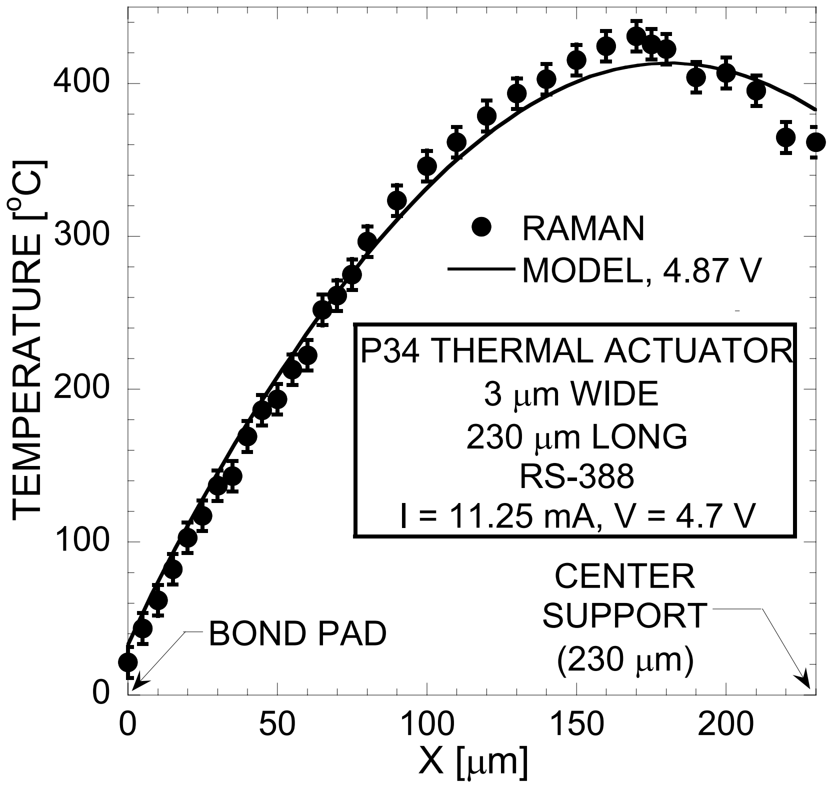
- Utilizes constrained thermal expansion due to Joule heating to achieve motion.
- Operates at low voltages and higher currents (12 V 20 mA typical) compared to other MEMS actuators.
- Capable of relatively high output forces (2.5 mN) and displacements (over 50 μm).

(M. Baker, Sandia National Laboratories, NM)

Raman Thermometry of a Thermal Actuator



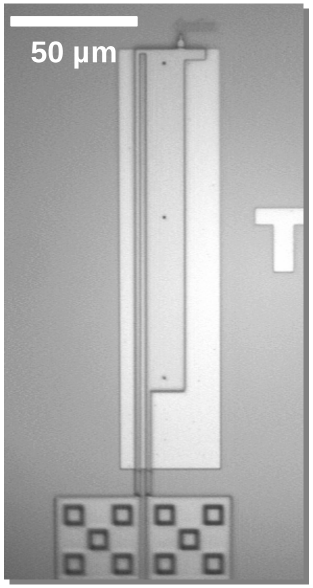
Actuator Leg Under Raman Microscope



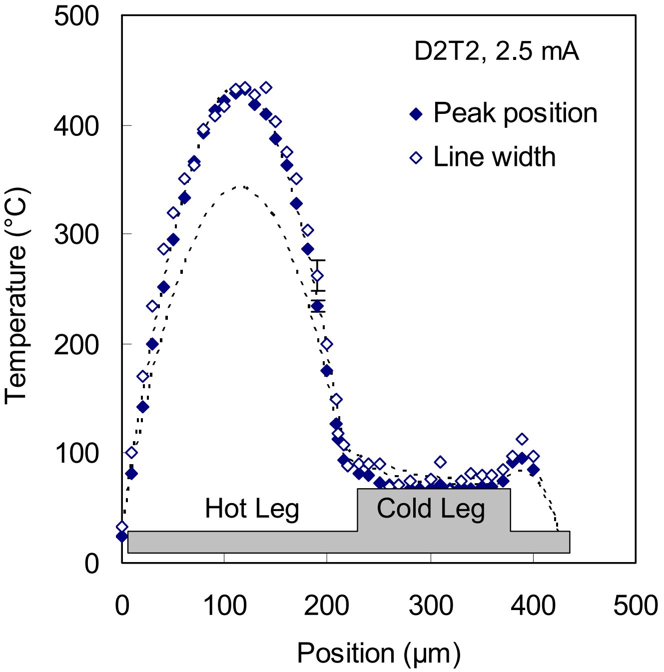
- To our knowledge, these were the first known data for such actuators, which are being studied in parallel by research groups at Brigham Young and Wisconsin
- The outstanding spatial resolution and unambiguous nature of the Si Raman signature are a significant improvement over IR methods

Kearney, Phinney, and Baker, *JMEMS*, **15**, pp. 314-321, 2006.

Raman Thermometry of Flexure Thermal Actuators



- Hot leg length = 200 µm
- Hot leg width = 2 µm
- Cold leg length = 160 µm
- Flexure length = 50 µm
- Leg separation distance = 3 µm
- Thickness = 2.5 µm
- Gap height = 2.0 µm



- Micron-scale resolution of instrument allows for temperature profiles to be obtained with 10 µm resolution. Displacement was 4.75 µm.
- Results are then used to validate numerical models of actuator temperature and displacement

Serrano, Phinney, and Kearney, *JMM*, **16**, pp. 1128-1134 (2006).

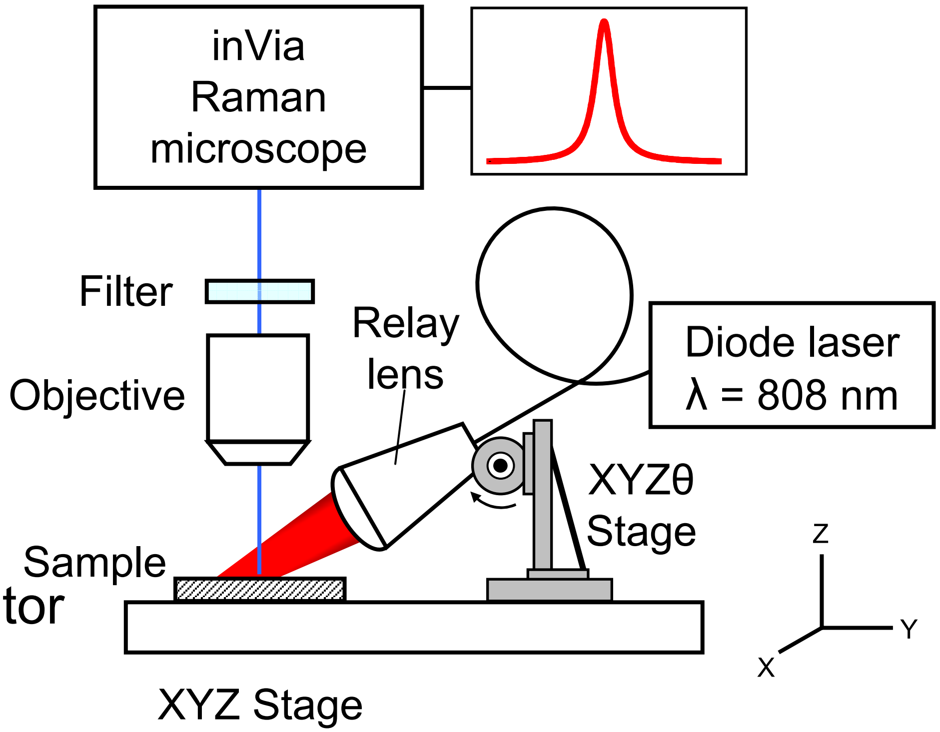
Thermometry of Laser Heated MEMS

No direct temperature measurements of laser-heated MEMS existed, so...

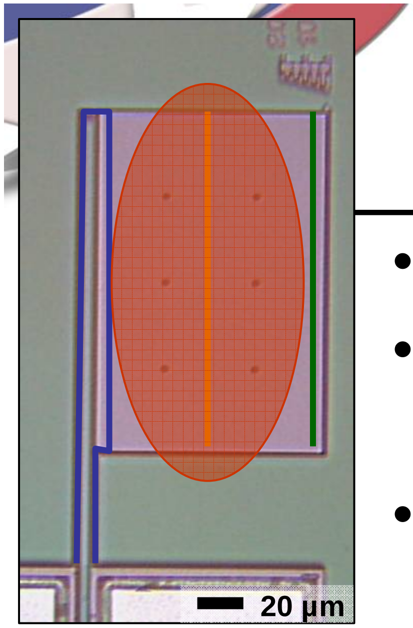
...use μRaman to map temperature of laser-heated devices

- **Goal:** Obtain further insight into thermal and optical behavior of laser-heated devices through direct temperature measurements

- Test two MEMS structures:
  - Cantilever plates, and
  - Optically-powered flexure actuator

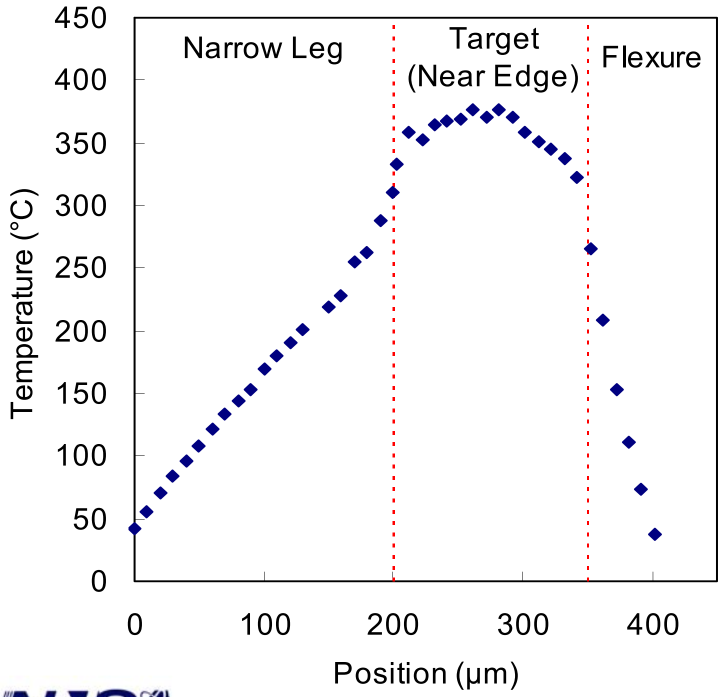


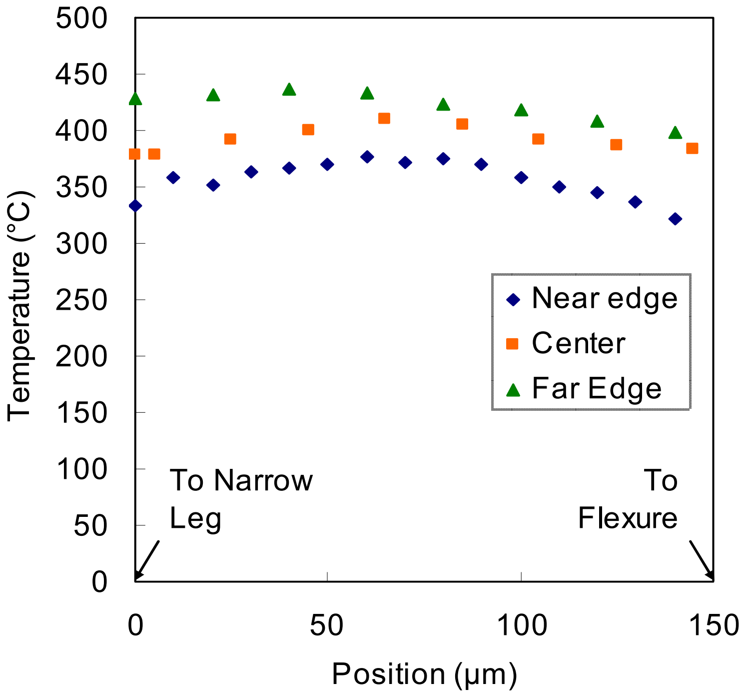
Serrano and Phinney, *Proc. InterPACK2007*, IPACK2007-33571 (2007)
Serrano and Phinney, *JMEMS*, **17,** 166-174 (2008)



Optically-Powered Flexure Actuator

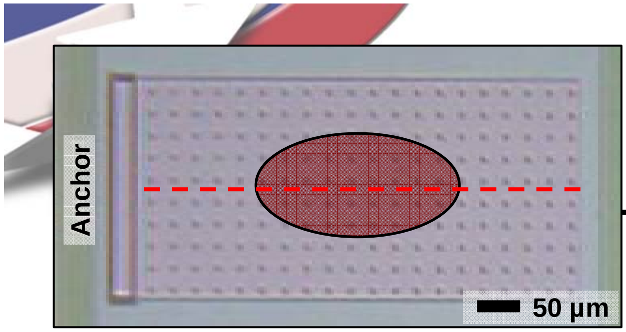
- Steady temperature increase along narrow leg
- Target temperature uniform at ~375°C up to middle, then decreases as flexure is approached
- Upper-right corner is hottest point on actuator due to device motion and longest conduction pathway



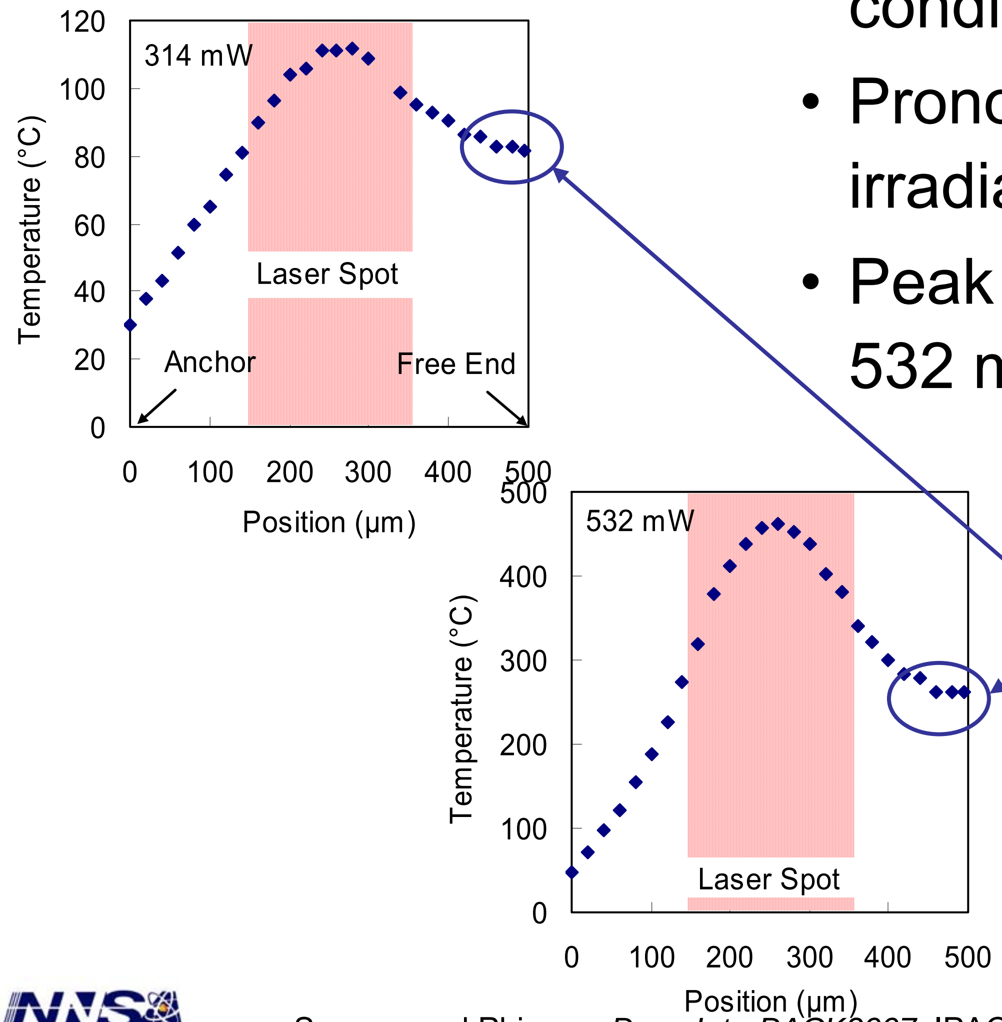


Serrano and Phinney, *Proc. InterPACK2007*, IPACK2007-33571 (2007).
Serrano and Phinney, *JMEMS*, **17,** 166-174 (2008).

Cantilever Plate



- Effect of different boundary conditions clearly evident
- Pronounced increase within irradiated region
- Peak temperature of 460°C at 532 mW

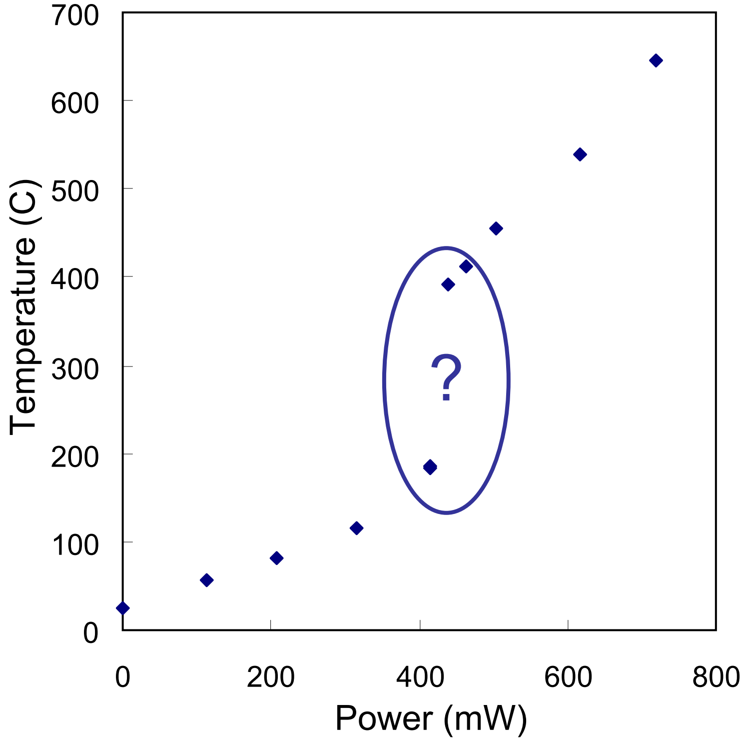


Adiabatic edge observed at free end, validating extended surface model assumptions

Serrano and Phinney, *Proc. InterPACK2007*, IPACK2007-33571 (2007).
Serrano, Phinney and Rogers, *IJHMT*, in review (2008).

Optical-Thermal Phenomena

- Center temperature is ~linear with power < 400 mW and temperatures < 200°C
- In the vicinity of 440 mW, temperature “jumps” by 200°C to over 400°C
- Temperature remains linear with power for higher powers

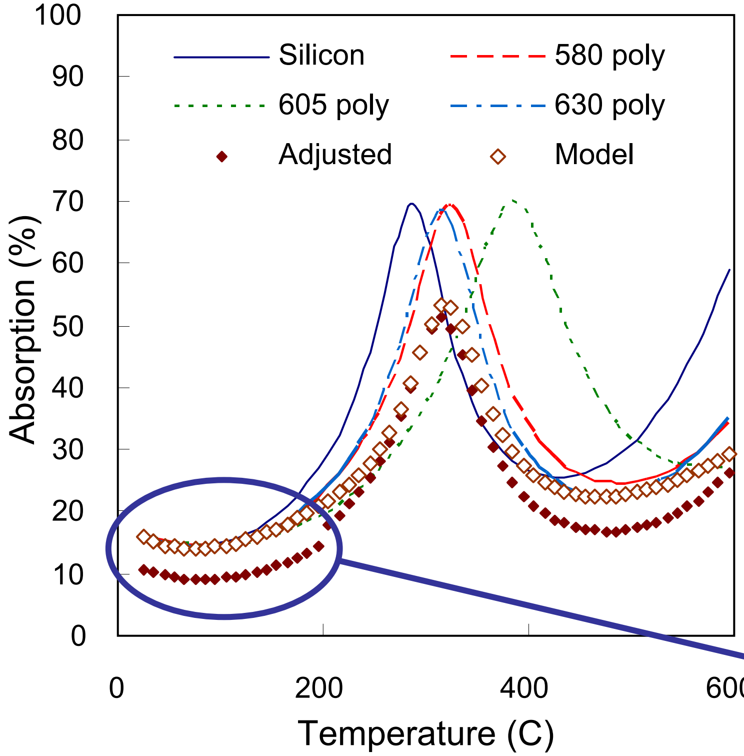


Why the abrupt jump?

Serrano and Phinney, *Proc. InterPACK2007*, IPACK2007-33571 (2007).
Serrano and Phinney, *JMEMS,* **17,** 166-174 (2008).

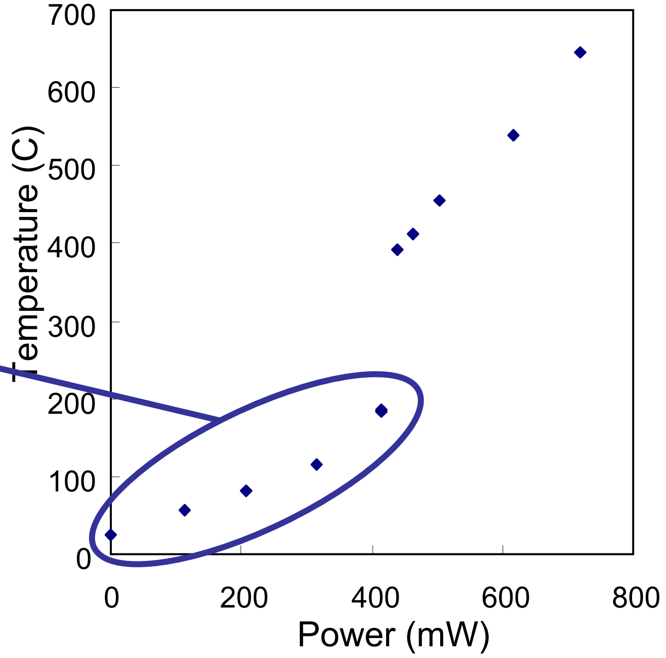
Optical-Thermal Phenomena

Small Thickness + T-dependant refractive index = T-dependant absorption



Cantilever plate: 60° incidence; 808 nm; literature temperature dependence for optical constants

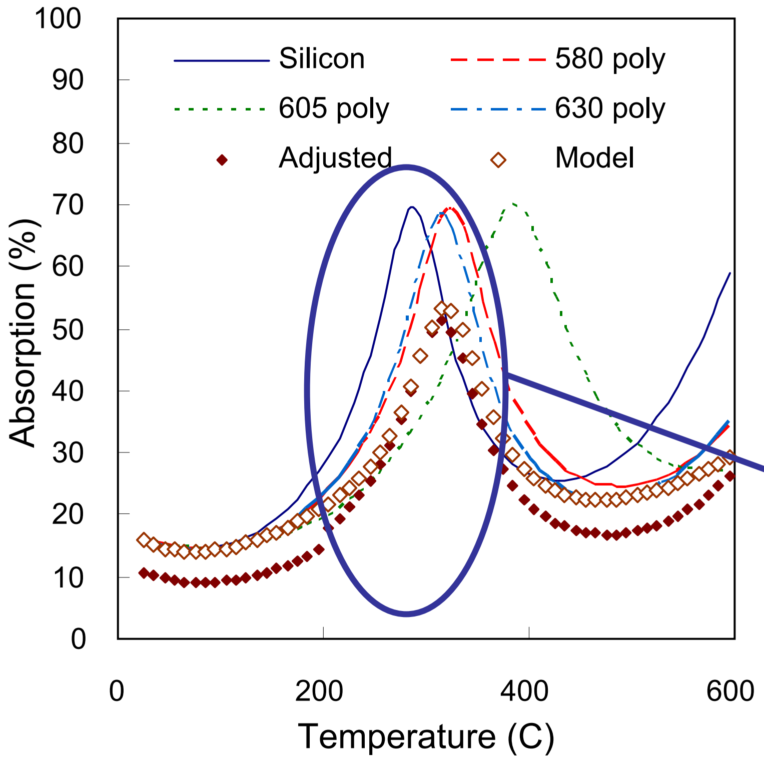
Flat absorption at lower T leads to initial linear temperature increase



Serrano, Phinney and Rogers, *IJHMT*, in review (2008).

Optical-Thermal Phenomena

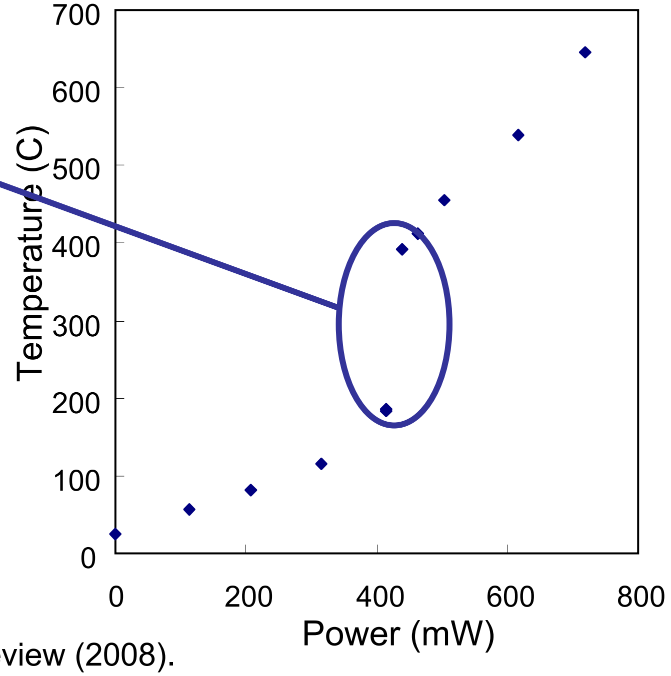
Small Thickness + T-dependant refractive index = T-dependant absorption



Flat absorption at lower T leads to initial linear temperature increase

Absorption peak leads to “positive feedback” effect:

forbidden T’s

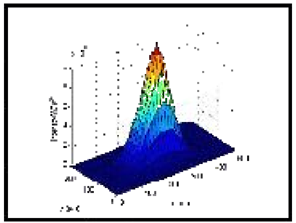


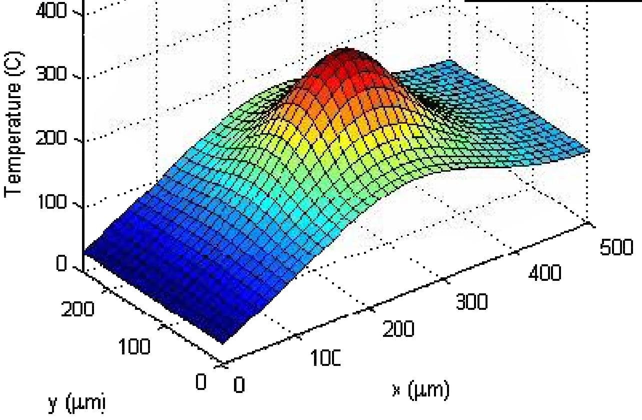
Serrano, Phinney and Rogers, *IJHMT*, in review (2008).

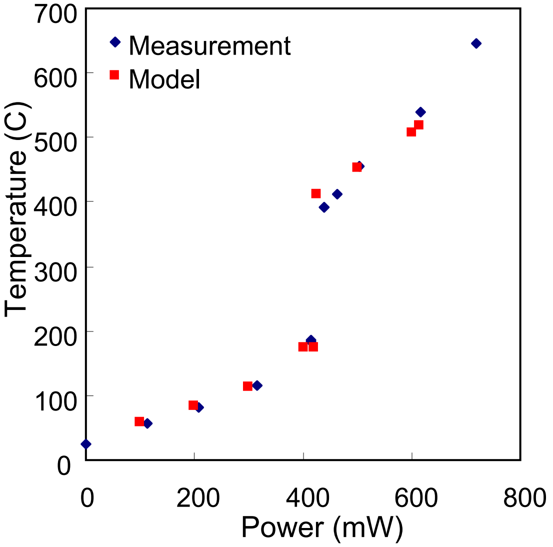
Simulating Optical-Thermal Phenomena

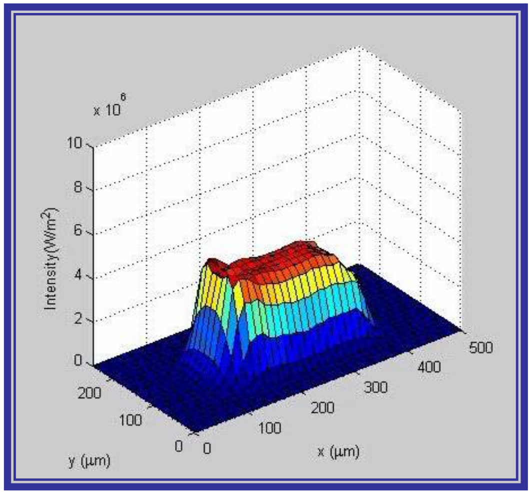
- Coupled optical-thermal phenomena must be considered for accurate device modeling
- With temperature-dependant absorption in model, temperature jump is also predicted

- Non-uniform absorption across surface drives system towards equilibrium much faster than without



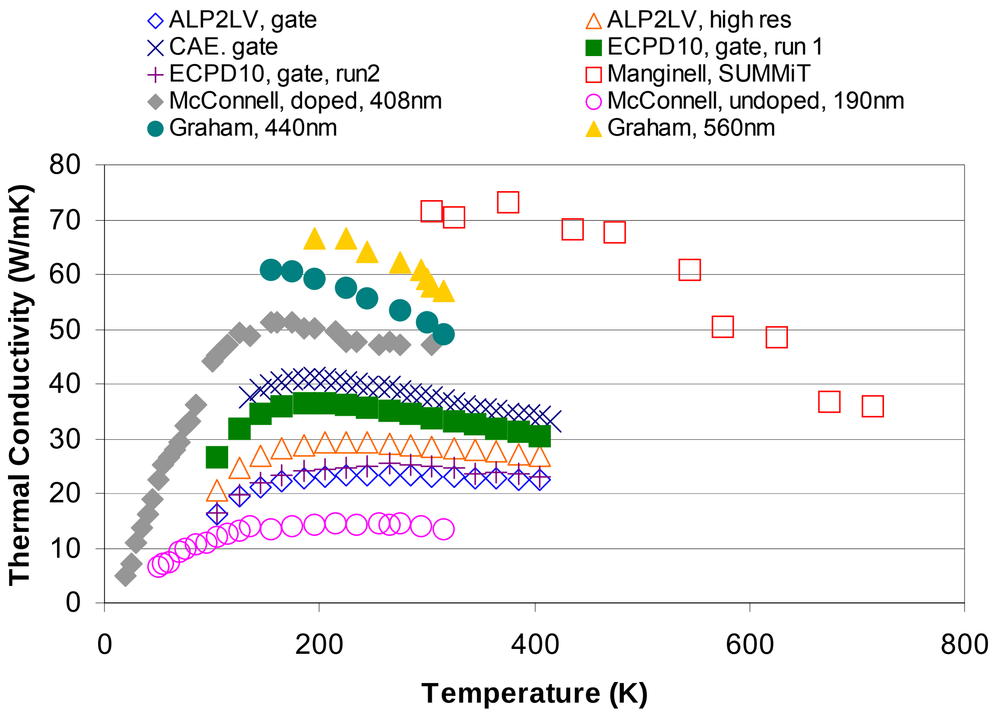


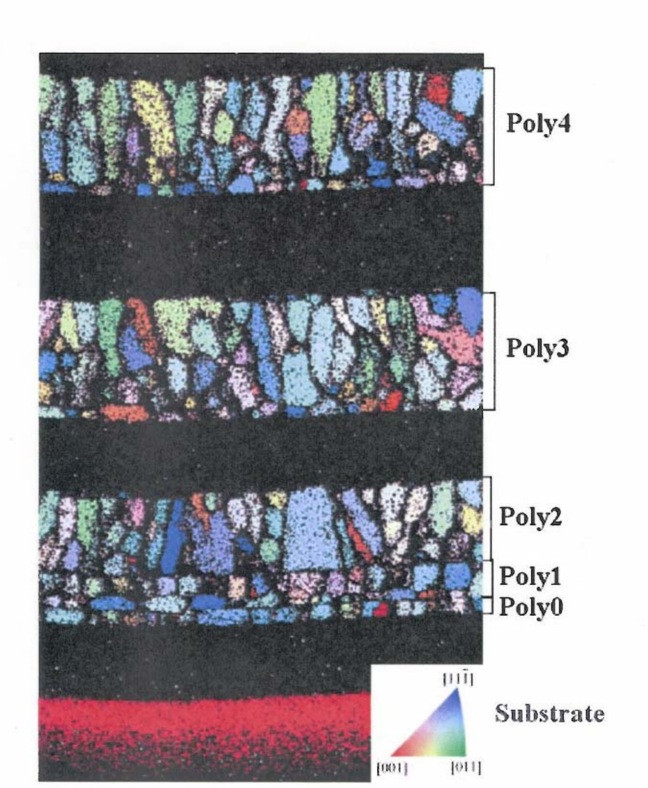




Serrano, Phinney and Rogers, (IJHMT, in review, 2008)

Thermal Conductivity Motivation

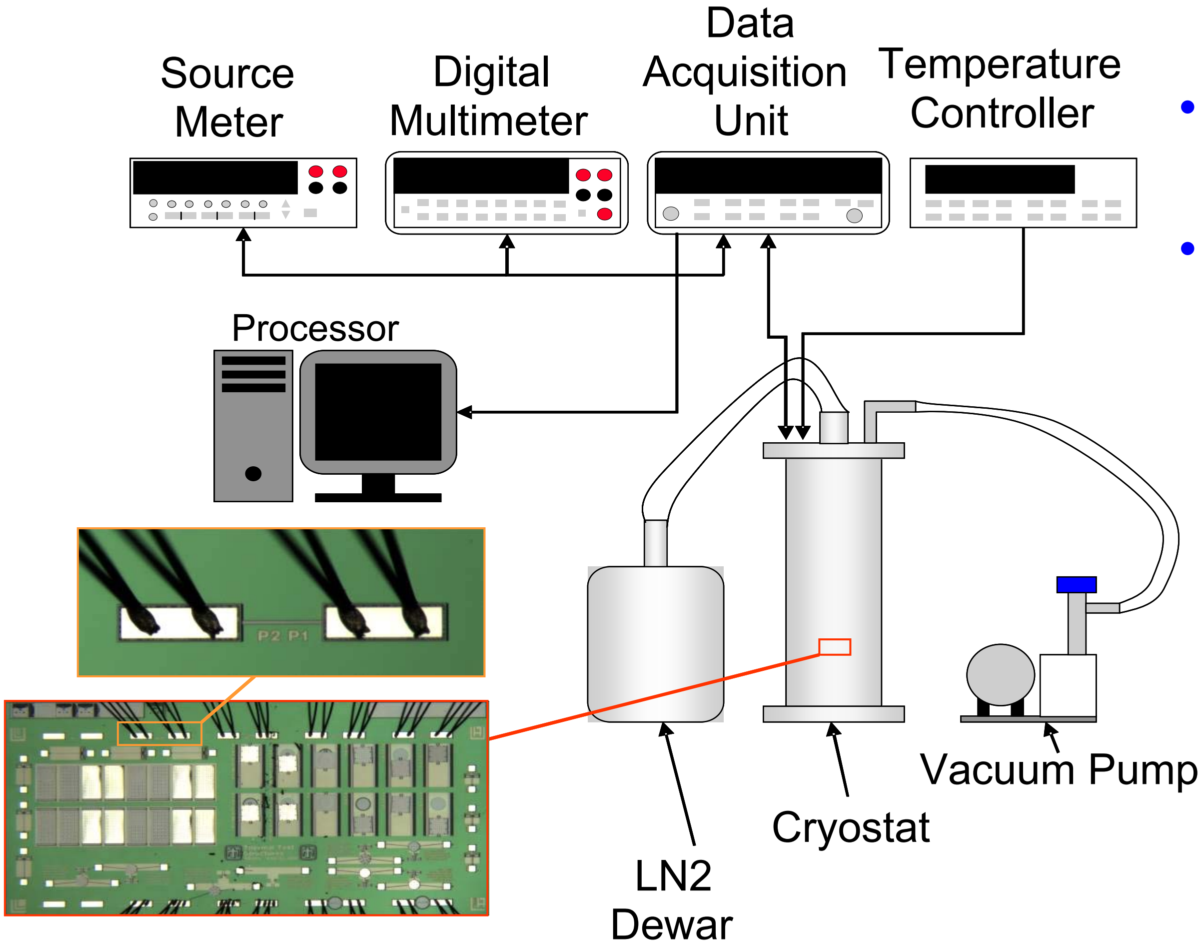




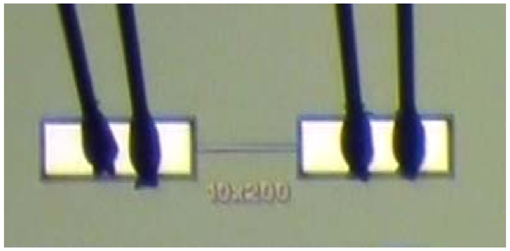
- Polycrystalline silicon (polysilicon) structure
- Range in measured thermal conductivities
- Understanding measurement technique and quantifying errors

T. Buchheit, SNL, 2004

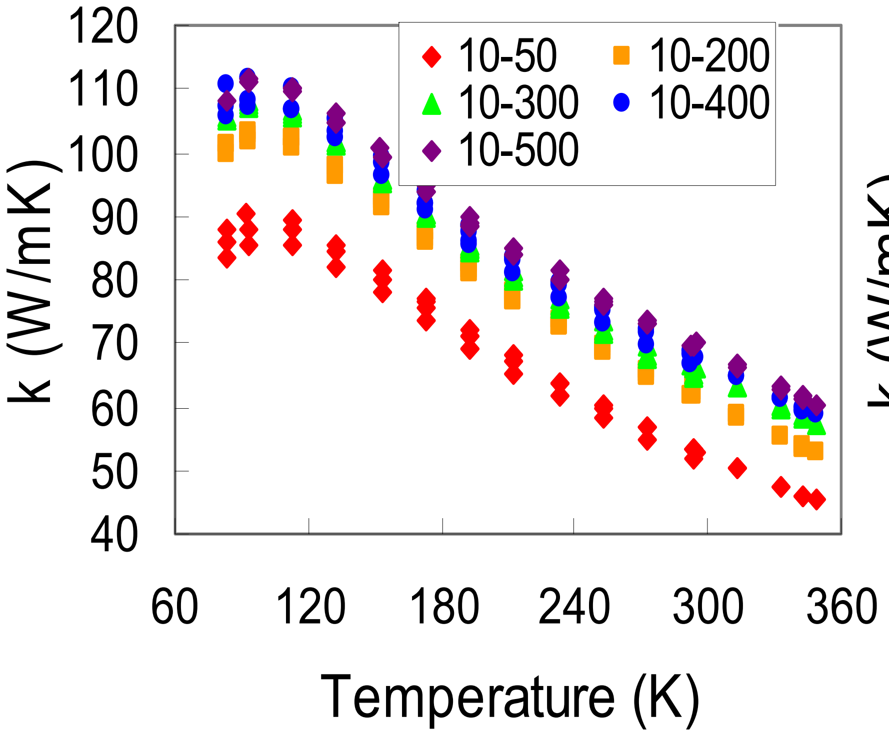
Experimental Methods and Test Structures



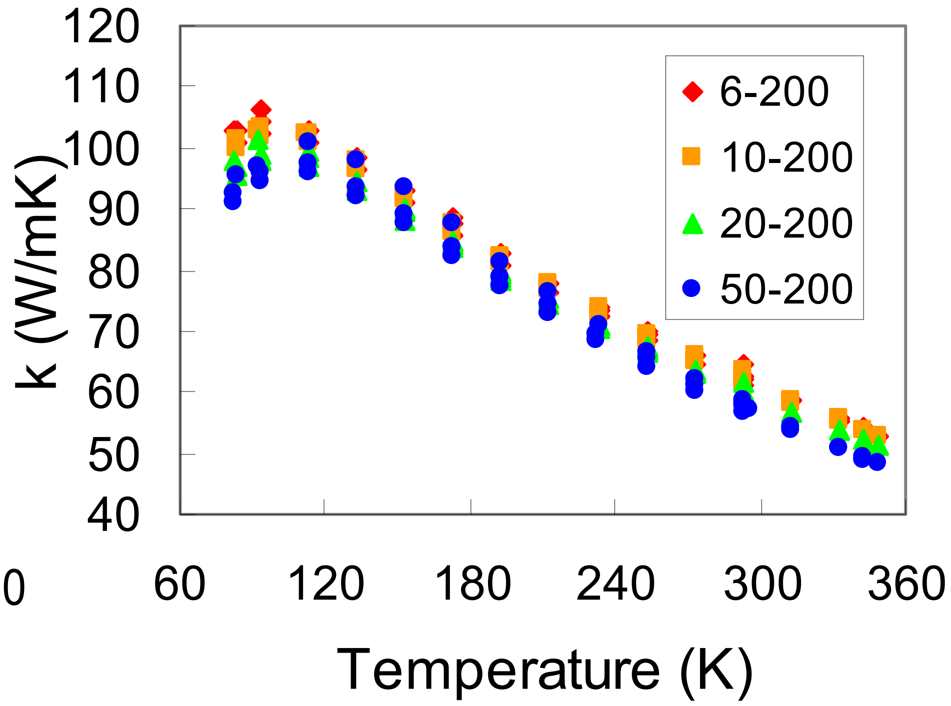
- Test structures are made of Poly4
- Lengths 50 – 500 μm, Widths 6 – 50 μm



Experimental Data



Thermal Conductivity of SNL SUMMiT Poly4 for
10 μm wide test structures with 50-500 μm lengths



Thermal Conductivity of SNL SUMMiT Poly4 for
200 μm long test structures with 6-50 μm widths

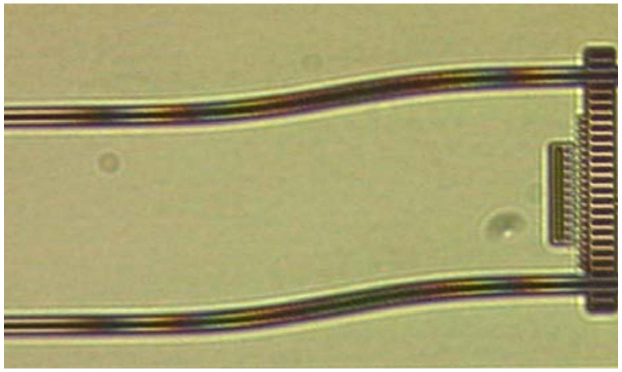
LDRD Proposal

Thermal Actuator Based Devices
- SUMMiT^{TM}V
- SOI

Time-dependent Failure Mechanisms
- Plastic deformation/creep
- Debris generation

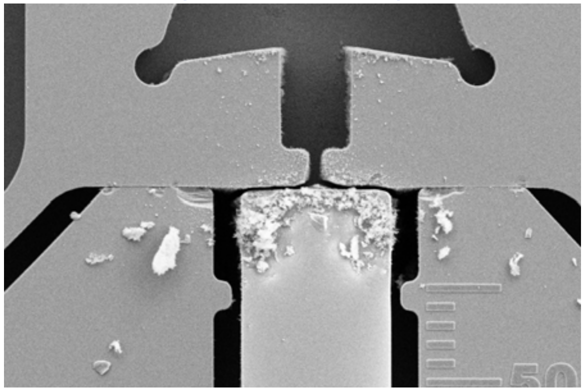
Collaboration Opportunities
- Reliability Model
- Suggestions/Discussion

Plastic Deformation of Thermal Actuator Legs after Operation



M. Baker, SNL, 1749-2

Debris Generation due to Contacting/Impacting Surfaces

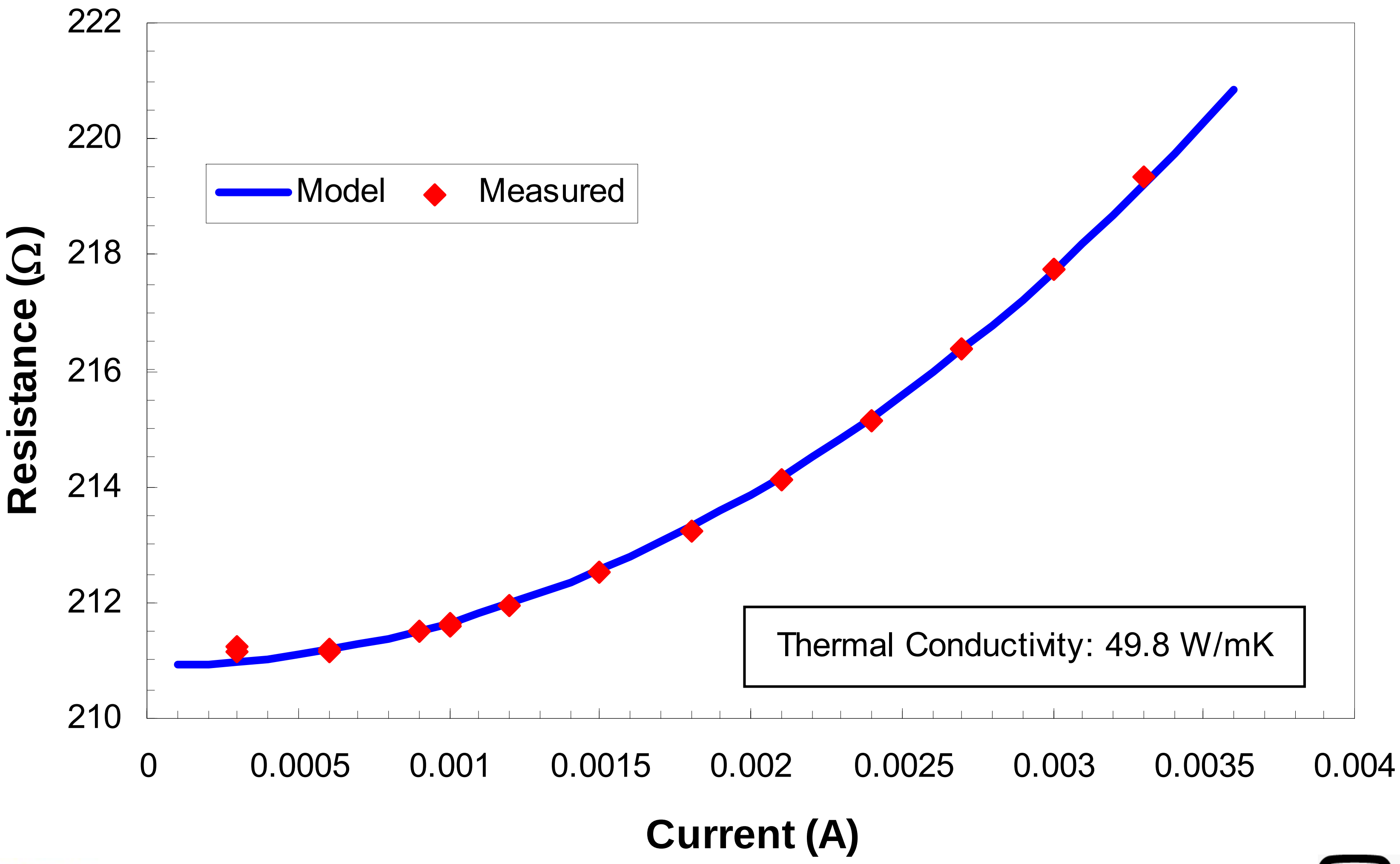


D. Luck, SNL, 1749-1

Summary

- Sandia's Microscale Science and Technology Department has a wide variety of experimental capabilities and expertise to probe fluidic, thermal, and material property phenomena at small scales.
- Both model validation and discovery experiments are performed.
- Thermal conductivity measurements benefitted from simulations and experiments.
- MEMS Reliability LDRD provides collaboration opportunities.

Typical Data



Advanced MicroPIV capabilities

- Dynamic interface tracking
- 2-Color PIV
- Microscale Particle-Tracking Velocimetry in Air