Measuring thermal properties of nanosystems

Prof. Patrick E. Hopkins

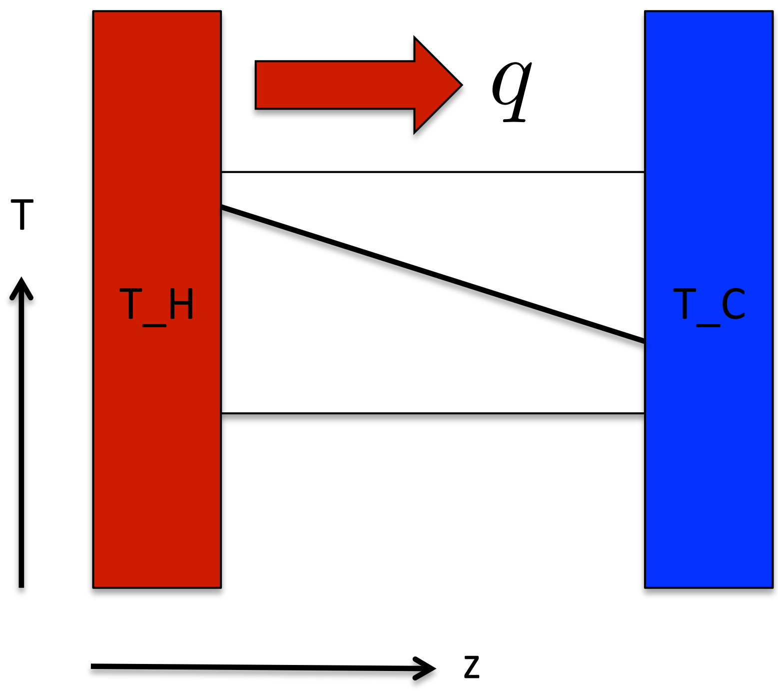
MEC 331

4/4/12

Heat Transfer

Steady state = The Fourier Law

$$q = -\kappa \frac{\partial T}{\partial z}$$



Transient = The Heat Equation

$$\rho C \frac{\partial T}{\partial t} = \kappa \frac{\partial^2 T}{\partial z^2} + q(t)$$

$$q = \text{Flux}$$
$$\rho = \text{mass density}$$
$$\kappa = \text{Thermal conductivity}$$
$$T = \text{temperature}$$
$$C = \text{Heat Capacity}$$

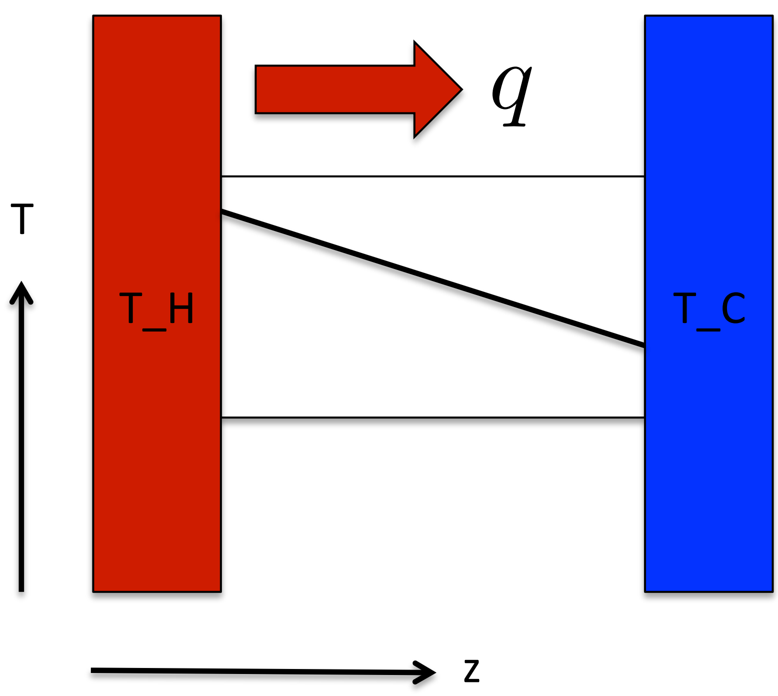
Outline

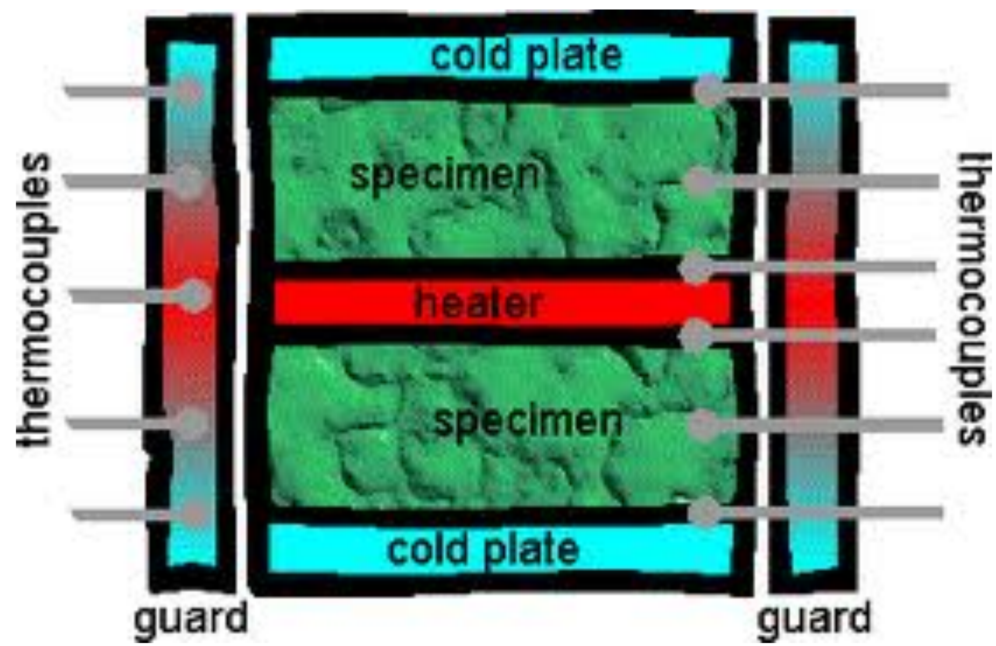
- Steady state electrical resistivity
- Transient techniques
  - "RC"
  - Heat capacity
  - Laser flash
  - Transient Electro-thermal
  - ns pulse
  - fs pulse
- Periodic techniques
  - Angstrom method
  - 3ω
  - FDTR
  - TDTR

Steady state measurements - bulk

"Guarded hot plate"

$$q = -\kappa \frac{\partial T}{\partial z}$$





Steady state measurements - nano

Electrical resistivity

Fourier Law

$$q = -\kappa \frac{\partial T}{\partial z}$$

Joule heating

$$q = I^2 R$$

$$\kappa V \frac{\partial^2 T}{\partial x^2} + I^2 R(T) = 0$$

$$\overline{R} = R_0 \left[\frac{2}{L\sqrt{\frac{I^2 R_0 \alpha}{w d L \kappa}}} \tan\left(\frac{L\sqrt{\frac{I^2 R_0 \alpha}{w d L \kappa}}}{2} \right) \right]$$

Measured resistance is related to thermal conductivity (and a lot of other known quantities)

Y. C. Tai, C. H. Mastrangelo, and R. S. Muller. Thermal conductivity of heavily doped low-pressure chemical vapor deposited polycrystalline silicon films. Journal of Applied Physics, 63:1442–1447, 1988.

Steady state measurements - nano

$$\overline{R} = R_0 \left[\frac{2}{L\sqrt{\frac{I^2 R_0 \alpha}{w d L \kappa}}} \tan\left(\frac{L\sqrt{\frac{I^2 R_0 \alpha}{w d L \kappa}}}{2} \right) \right]$$

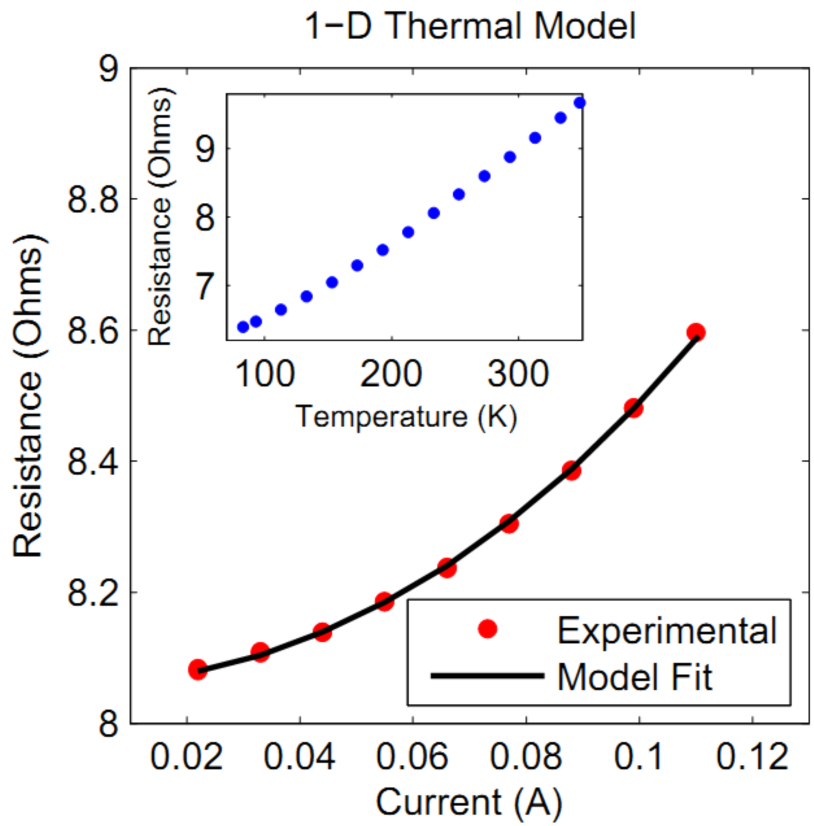


Fig. from: English, Phinney, Hopkins, and Serrano, “Thermal conductivity of single crystal silicon microbridges measured by electrical resistance thermometry and time domain thermoreflectance,” under review

Steady state measurements - nano

Electrical resistivity - assumptions

Equilibrium resistance

Current

$$\overline{R} = R_0 \left[\frac{2}{L\sqrt{\frac{I^2 R_0 \alpha}{w\, d\, L\, \kappa}}} \tan\left(\frac{L\sqrt{\frac{I^2 R_0 \alpha}{w\, d\, L\, \kappa}}}{2} \right) \right]$$

TCR

What you measure

Sample geometry

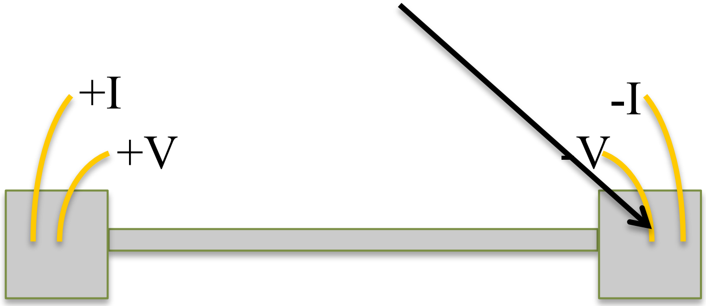
NEED TO KNOW SAMPLE GEOMETRY!!!

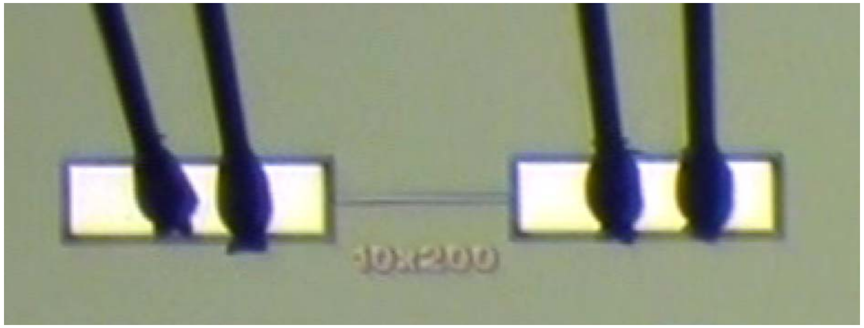
Y. C. Tai, C. H. Mastrangelo, and R. S. Muller. Thermal conductivity of heavily doped low-pressure chemical vapor deposited polycrystalline silicon films. Journal of Applied Physics, 63:1442–1447, 1988.

Fig. from: English, Phinney, Hopkins, and Serrano, "Thermal conductivity of single crystal silicon microbridges measured by electrical resistance thermometry and time domain thermoreflectance," under review.

Steady state measurements - nano

Electrical/thermal contact resistances are inherently present in measurements





How do you make these contacts in a nanosystem??

When would these contact resistances matter in terms of sample geometry???

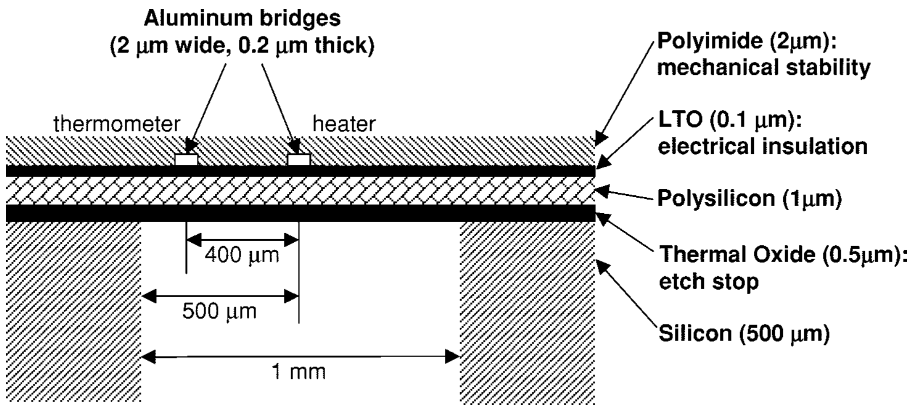
P. E. Hopkins and L. M. Phinney. Thermal conductivity measurements on polycrystalline silicon micro- bridges using the 3w technique. Journal of Heat Transfer, 131:043201, 2009.

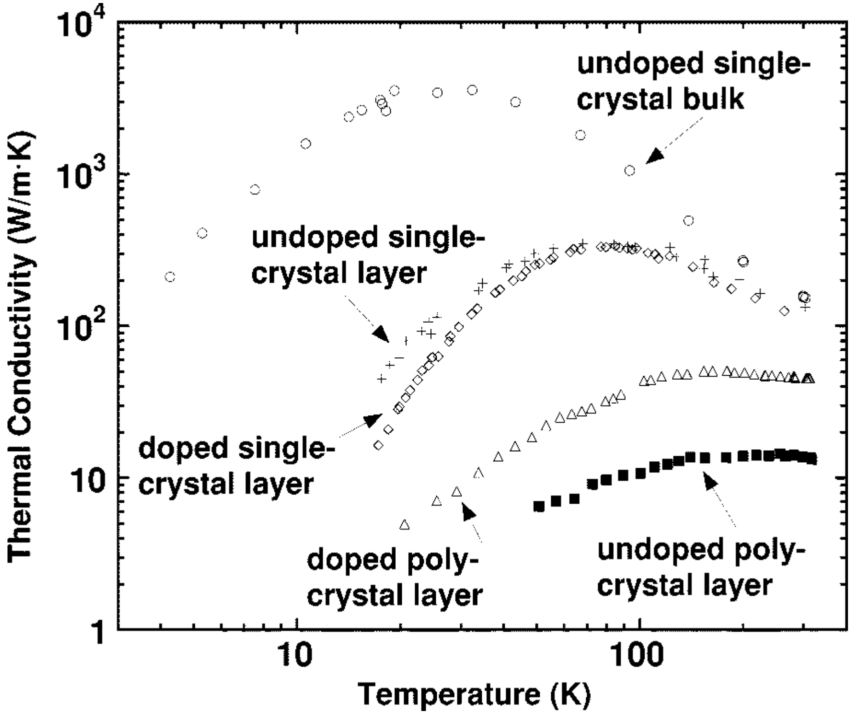
Steady state measurements - nano

Lithography

Thermal Conductivity of Doped Polysilicon Layers

Angela D. McConnell, Srinivasan Uma, *Member, IEEE*, and Kenneth E. Goodson, *Associate Member, IEEE*





A. D. McConnell, S. Uma, and K. E. Goodson. Thermal conductivity of doped polysilicon layers. Journal of Microelectromechanical Systems, 10:360–369, 2001.

Steady state measurements - nano

Lithography

Thermal conductivity of individual silicon nanowires

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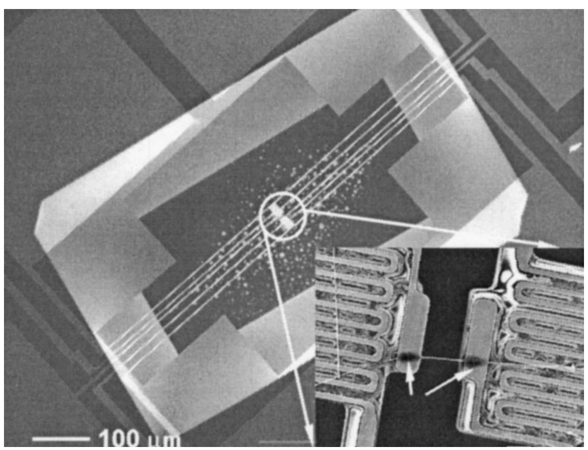
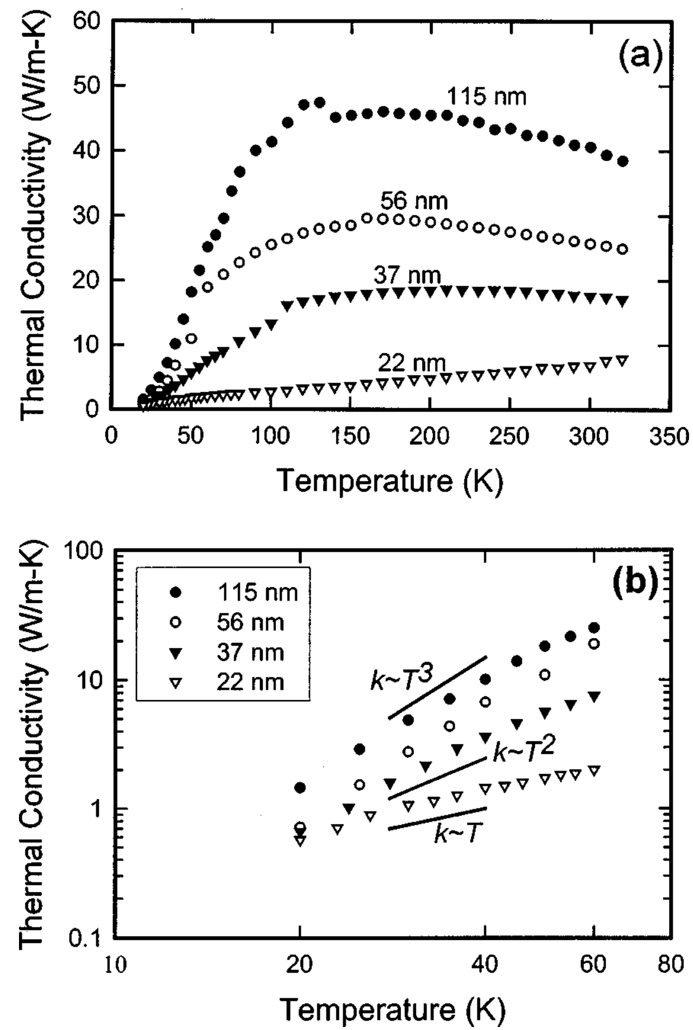
(Received 27 May 2003; accepted 13 August 2003)





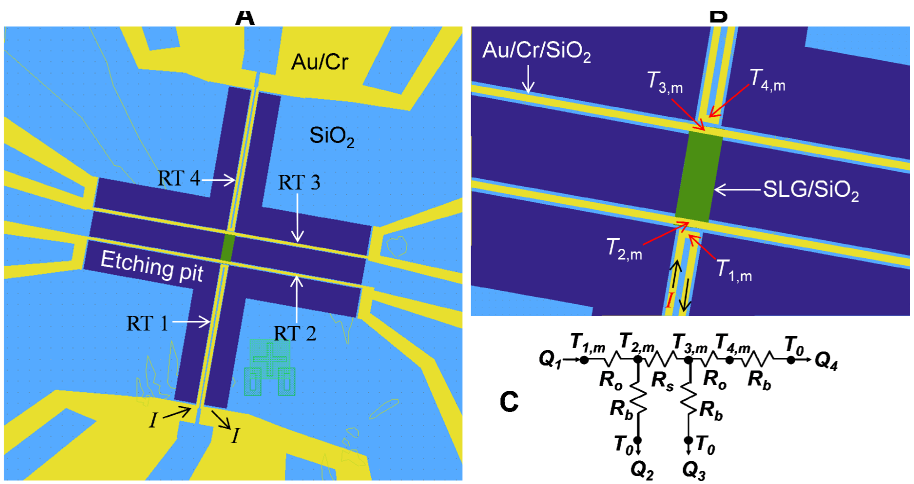
D. Li, Y. Wu, P. Kim, L. Shi, P. Yang, and A. Majumdar. Thermal conductivity of individual silicon nanowires. Applied Physics Letters, 83:2934–2936, 2003.

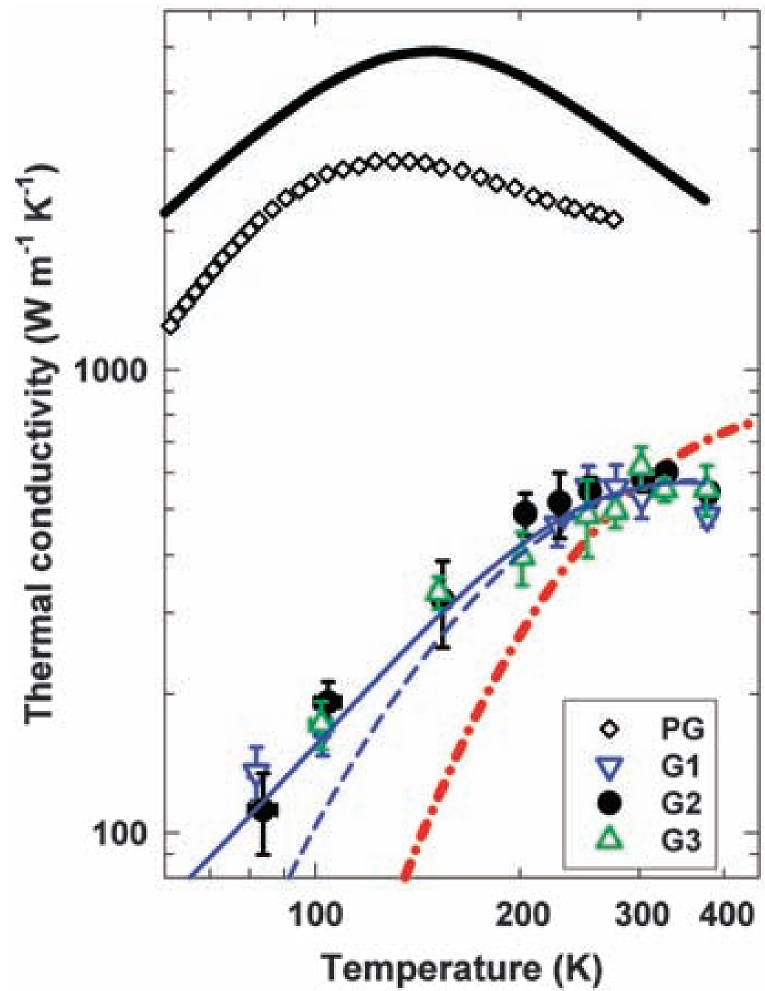
Steady state measurements - nano

Lithography

Two-Dimensional Phonon Transport in Supported Graphene

Jae Hun Seol,[1] Insun Jo,[2] Arden L. Moore,[1] Lucas Lindsay,[3,4] Zachary H. Aitken,[5] Michael T. Pettes,[1] Xuesong Li,[1,6] Zhen Yao,[2] Rui Huang,[5] David Broido,[3] Natalio Mingo,[7] Rodney S. Ruoff,[1,6] Li Shi[1,6]*





J. H. Seol, I. Jo, A. L. Moore, L. Lindsay, Z. H. Aitken, M. T. Pettes, X. Li, Z. Yao, R. Huang, D. A. Broido, N. Mingo, R. S. Ruoff, and L. Shi. Two-dimensional phonon transport in supported graphene. Science, 328:213–216, 2010.

Steady state vs. transient

Steady state = The Fourier Law

$$q = -\kappa \frac{\partial T}{\partial z}$$

Transient = The Heat Equation

$$\rho C \frac{\partial T}{\partial t} = \kappa \frac{\partial^2 T}{\partial z^2} + q(t)$$

Heat capacity enters the picture

The source term can make a difference

Steady state, or long time, experiments, are subjected to MAJOR convection and conduction losses. High *T* issues (i.e., RT and above)

Source can be "single shot/impulse" or "periodic"

Transient measurements

"RC" techniques

$$\rho C \frac{\partial T}{\partial t} = \kappa \frac{\partial^2 T}{\partial z^2} + q(t)$$

Solution results in "thermal" time constant in exponential decay

$$\Delta T(t) = \Delta T_0 \exp\left[\frac{-t}{\tau}\right]$$

$$\tau = \frac{CV}{Ah}$$

Thermal conductance

How does $\Delta T(t)$ change with *C, V, A,* and *h*?

Transient measurements

$$\tau = \frac{CV}{Ah}$$

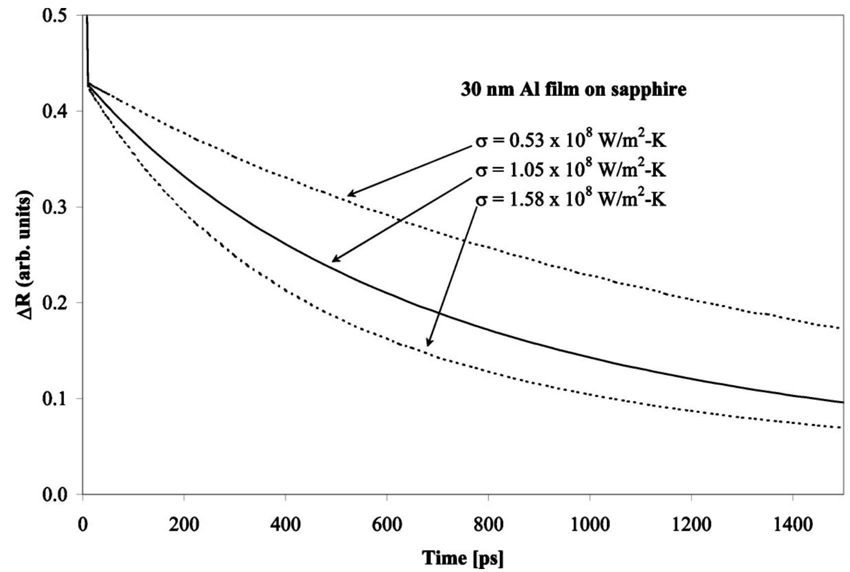


Fig. 2 Modeled thermal response of 30 nm Al film on a sapphire substrate with $\sigma = 1.05 \times 10^8$ W/m^2 K. The dotted lines are the thermal response for the same film with ±50% change in σ.

SO WHAT DO YOU MEASURE????

R. J. Stevens, A. N. Smith, and P. M. Norris. Measurement of thermal boundary conductance of a series of metal-dielectric interfaces by the transient thermoreflectance technique. Journal of Heat Transfer, 127(3): 315–322, 2005.

Steady state vs. transient

Steady state = The Fourier Law

$$q = -\kappa \frac{\partial T}{\partial z}$$

Steady state techniques are the only measurements that are directly related to thermal conductivity

Transient = The Heat Equation

$$\rho C \frac{\partial T}{\partial t} = \kappa \frac{\partial^2 T}{\partial z^2} + q(t)$$

Transient techniques measure quantities that are related to the thermal diffusivity or thermal effusivity of the sample

$$D = \frac{\kappa}{C} \qquad E = \sqrt{\kappa C}$$

Thermal effusivity

Thermal effusivity
From Wikipedia, the free encyclopedia

A material's thermal effusivity is a measure of its ability to exchange thermal energy with its surroundings.

If two semi-infinite bodies initially at temperatures T1 and T2 are brought in perfect thermal contact, the temperature at the contact surface Tm will be given by their relative effusivities.

$$T_m = T_1 + (T_2 - T_1)\,\frac{E_2}{E_1+E_2}$$

This expression is valid for all times for semi-infinite bodies in perfect thermal contact. It is also a good first guess for the initial contact temperature for finite bodies.

Notes on heat capacity

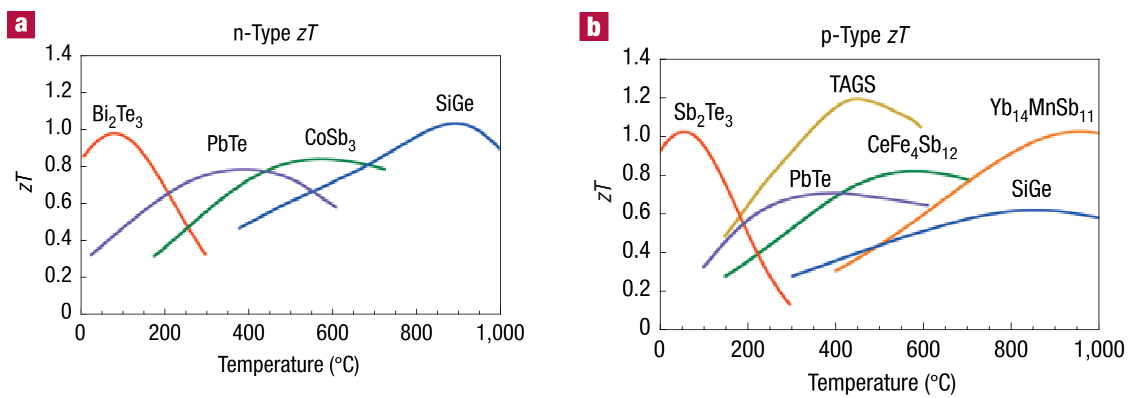
So if *C* is well known, then *k* can be "inferred" with transient measurements

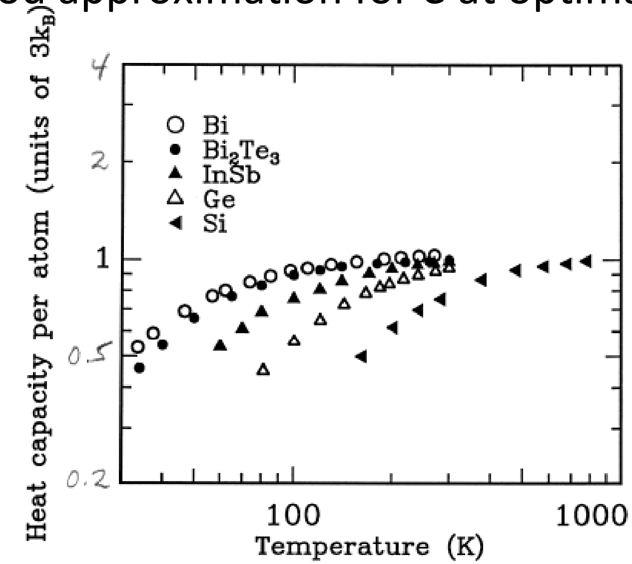
- In many cases, *C* is well known from careful measurements on bulk materials
- When scaled for porosity, *C* is ~ independent of microstructure (i.e., only atomic density is important)
- Materials with similar bonding and atomic weights have similar heat capacities
- Electronic heat capacity is too small to matter in most considerations

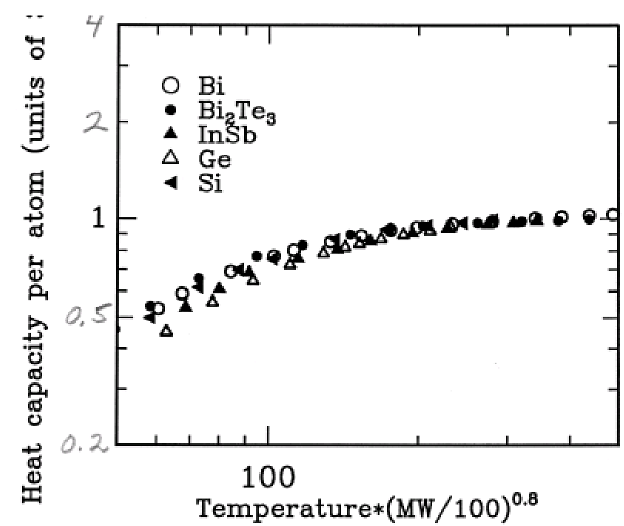
Notes on heat capacity

- For many semiconductors, classical equipartition is a good approximation for C at optimal thermoelectric operating temperatures

$$C = C_V = 3Nk_{\mathrm{B}}$$



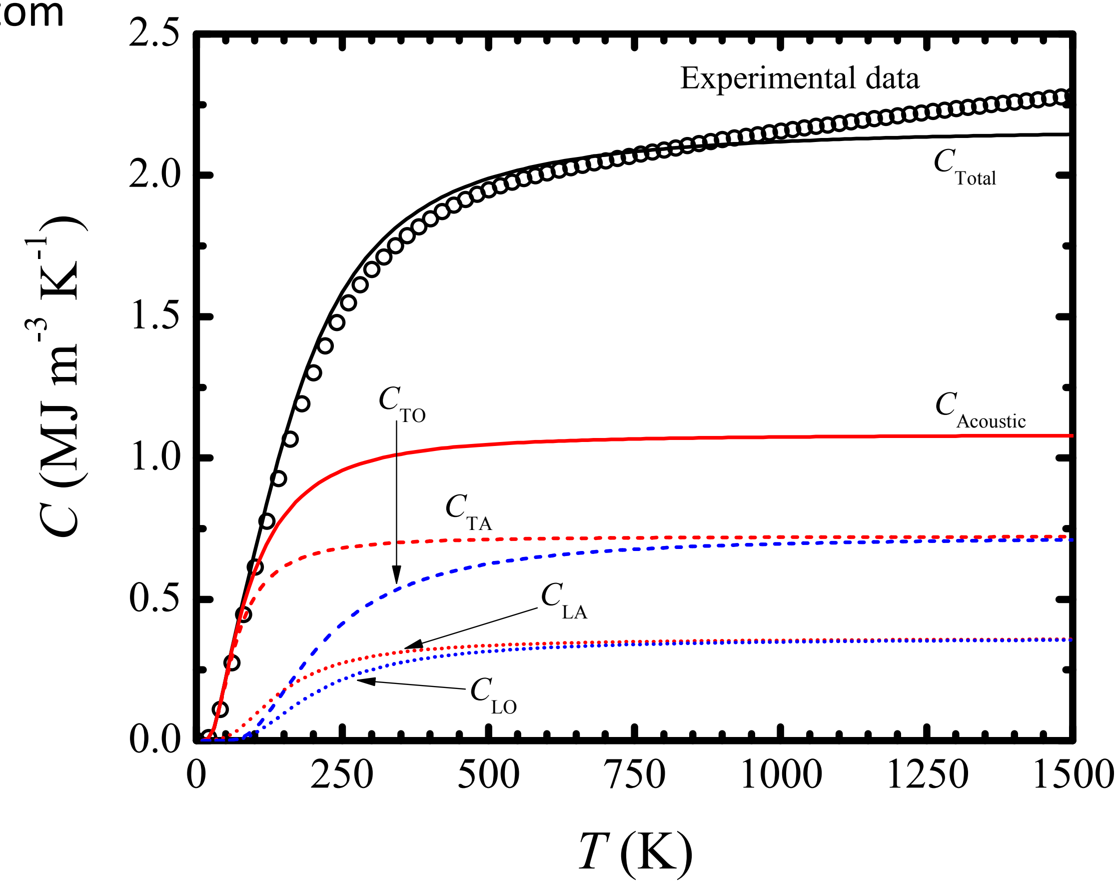




G. J. Snyder and E. S. Toberer. Complex thermoelectric materials. Nature Materials, 7:105–114, 2008.
David Cahill, “Measurement of Thermal conductivity,” “Thermal_School09.pdf”, on http://users.mrl.illinois.edu/cahill/presentations.html

Notes on heat capacity

- At high temperatures, anharmonicity also increases the heat capacity
- Thermal expansion causes the vibrational modes to soften increasing the vibrational entropy per atom



Heat capacity of silicon

Grüneisen Parameters

TABLE I. The Grüneisen parameters $\gamma_{TA(X)}$ and $\gamma_{TA(L)}$ for Si, diamond, and Ge are calculated from Eq. (10). The estimated $\gamma_{TA(X)}$ and $\gamma_{TA(L)}$ are compared with experimental data (Ref. 1) and tight-binding calculation results.

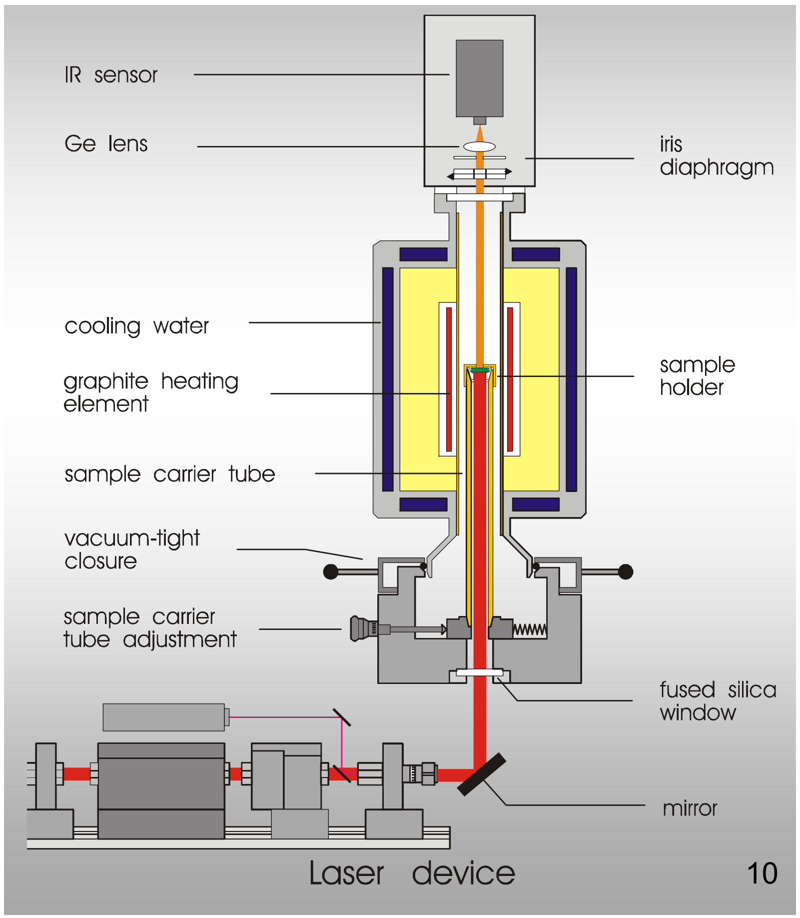
	Si	Diamond	Ge
$\gamma_{TA(X)}$ (*TB* calc.)	−1.08	0.042	
$\gamma_{TA(X)}$ (expt.)	−1.4		−1.53
$\gamma_{TA(X)}$ [Eq. (10)]	−1.67	0.017	−1.08
$\gamma_{TA(L)}$ (*TB* calc.)	−1.15	−0.047	
$\gamma_{TA(L)}$ (expt.)	−1.3		−0.4
$\gamma_{TA(L)}$ [Eq. (10)]	−1.54	−0.060	−0.77

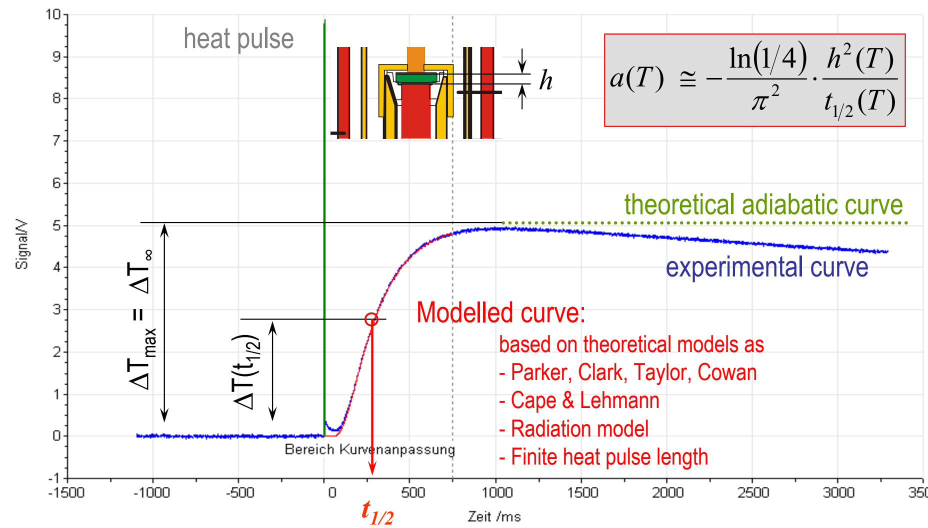
PRB 43, 5024 (1991)

What does this say about Debye temperatures?

Transient measurements

Laser Flash





- Very dependent on surface emissivity
- Terrible sensitivity in nanosystems

Transient measurements

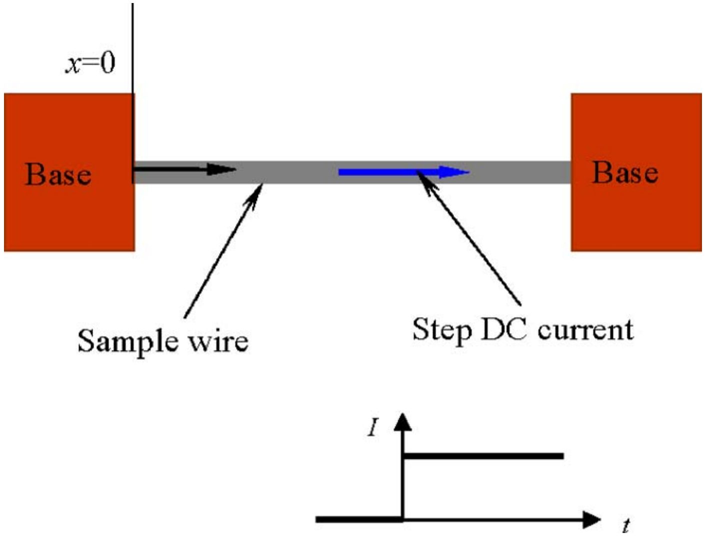
Transient electro-thermal technique (TET technique)

JOURNAL OF APPLIED PHYSICS **101**, 063537 (2007)

Thermal characterization of microscale conductive and nonconductive wires using transient electrothermal technique

Jiaqi Guo and Xinwei Wang[a)]
Department of Mechanical Engineering, N104 Walter Scott Engineering Center, University of Nebraska-Lincoln, Lincoln, Nebraska 68588-0656

Tao Wang
State Key Laboratory of Clean Energy Utilization, Zhejiang University, Hangzhou, People's Republic of China 310027



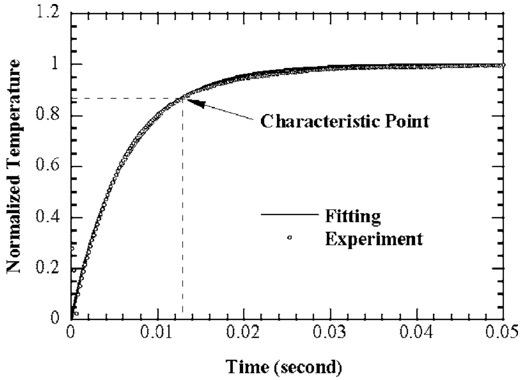


FIG. 5. The normalized temperature vs the theoretical fitting for the SWCNT bundle.

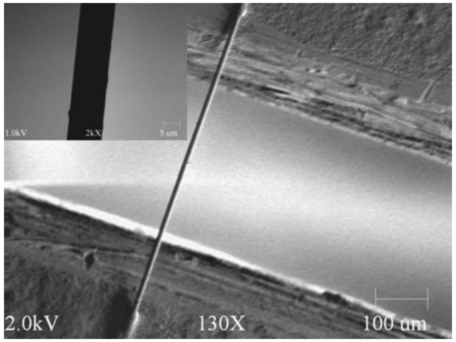
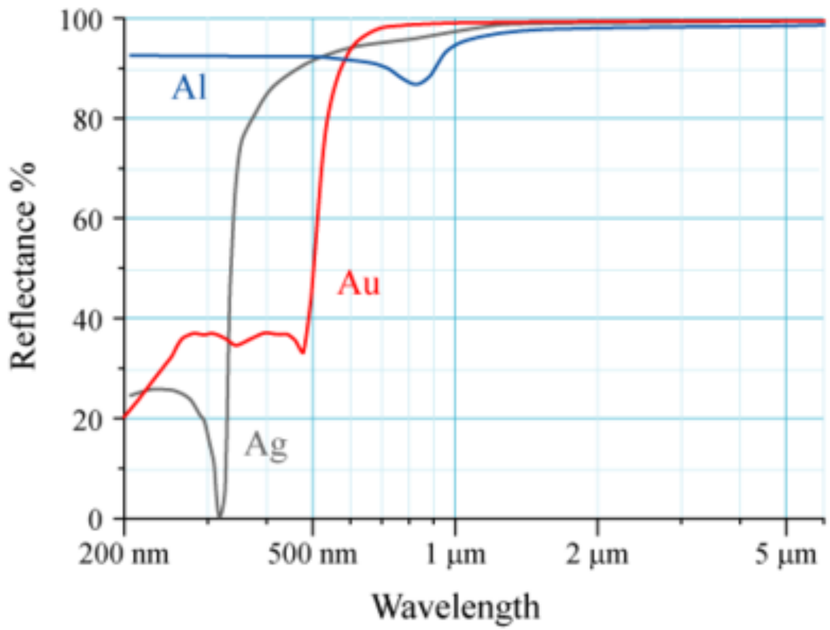
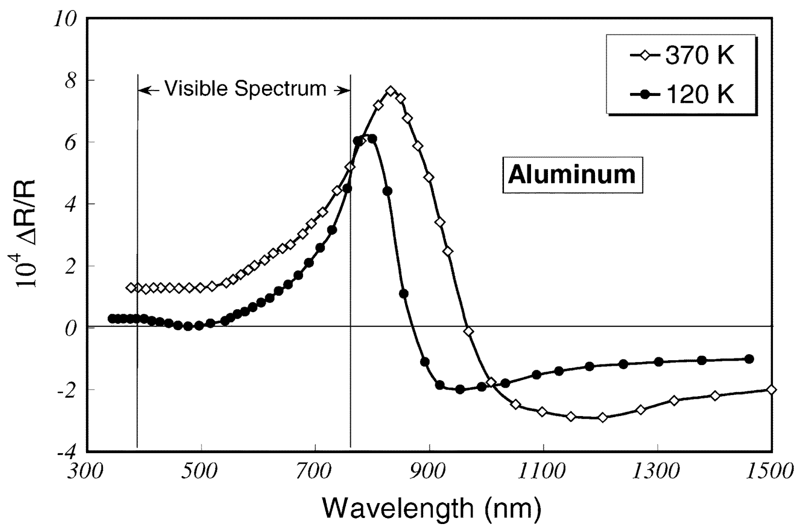


FIG. 6. SEM picture of coated polyester fiber (sample 2).

Transient measurements (optical)

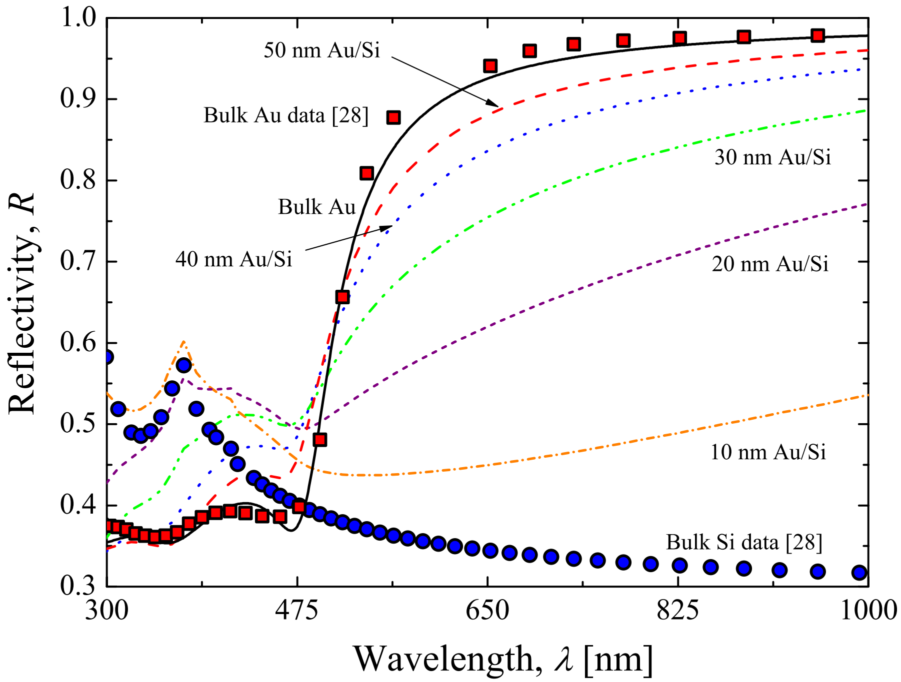
Reflectivity vs. Thermoreflectivity $\frac{\partial R}{\partial T}$

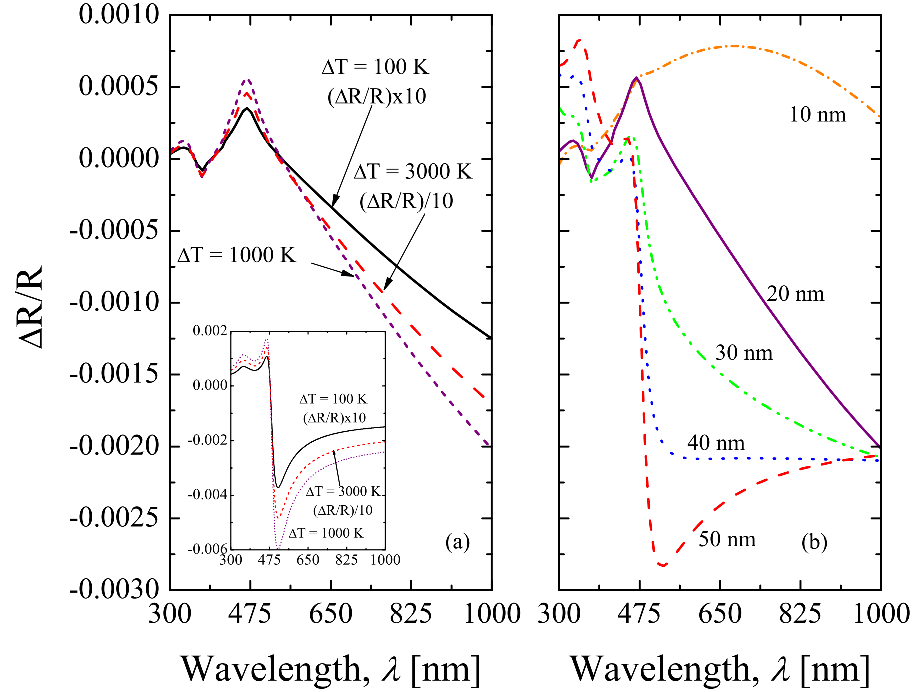




Transient measurements (optical)

Thermoreflectivity $\dfrac{\partial R}{\partial T}$





P. E. Hopkins. Influence of electron-boundary scattering on thermoreflectance calculations after intra- and interband transitions induced by short-pulsed laser absorption. Physical Review B, 81:035413, 2010.

Transient ThermoReflectance (TTR) measurements (optical)

Pump-probe: nanosecond pump

Full relaxation of thermal energy before next pulse arrives

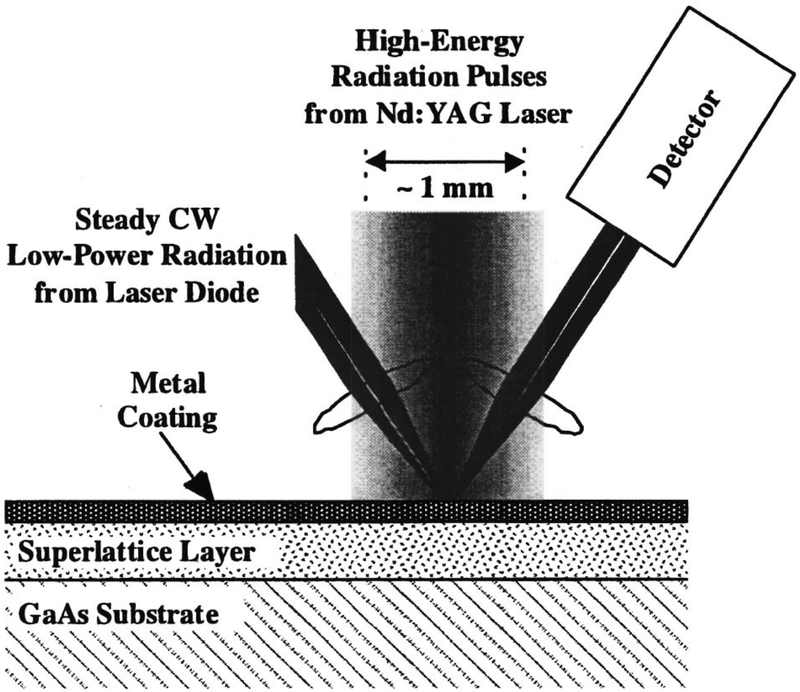


FIG. 1. The thermoreflectance method for measuring the vertical thermal resistance of Bi_2Te_3/Sb_2Te_3 superlattice layers.

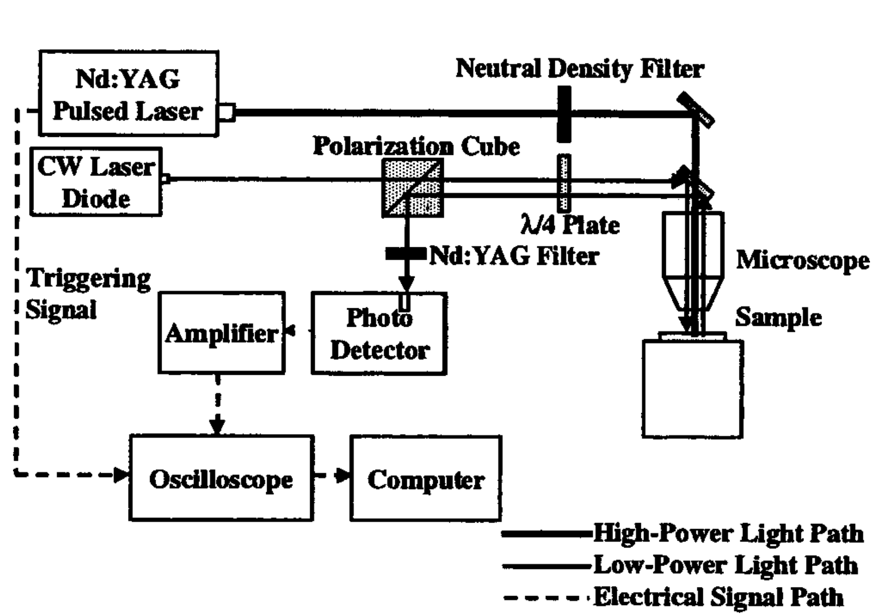
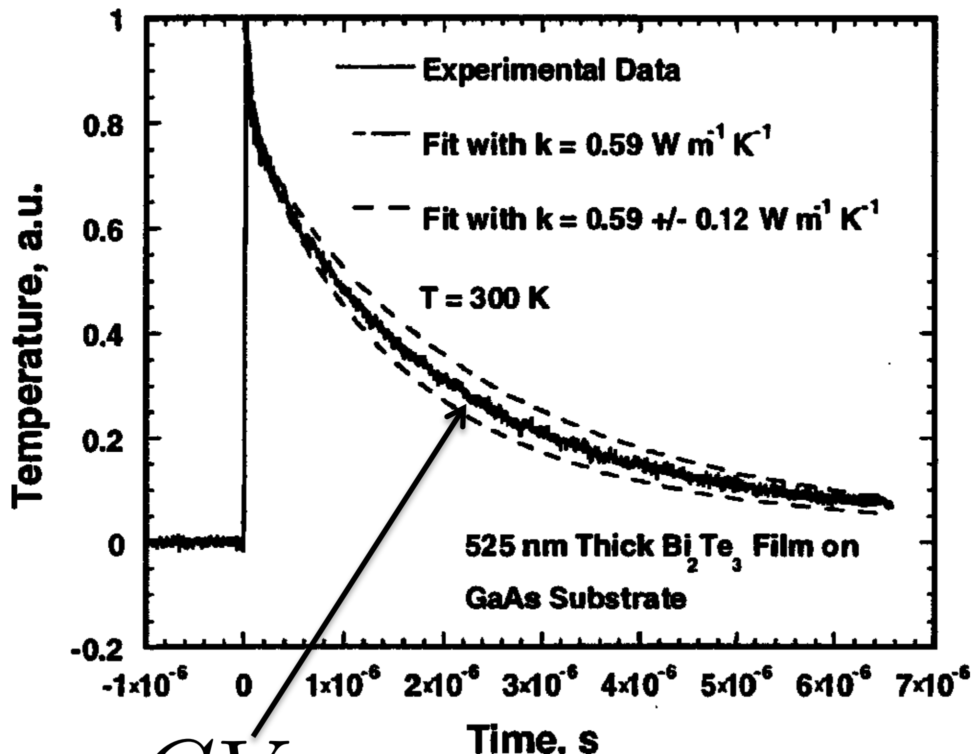


FIG. 2. Diagram describing the paths for radiation and electrical signals in the experimental setup.

M. N. Touzelbaev, P. Zhou, R. Venkatasubramanian, and K. E. Goodson. Thermal characterization of Bi_2Te_3/Sb_2Te_3 superlattices. Journal of Applied Physics, 90:763–767, 2001.

Transient ThermoReflectance (TTR) measurements (optical)

Pump-probe: nanosecond pump



$$\tau = \frac{CV}{Ah}$$

Pump pulse gives heating event, and decay is monitored after heating event. Time of pulse dictates spatial resolution

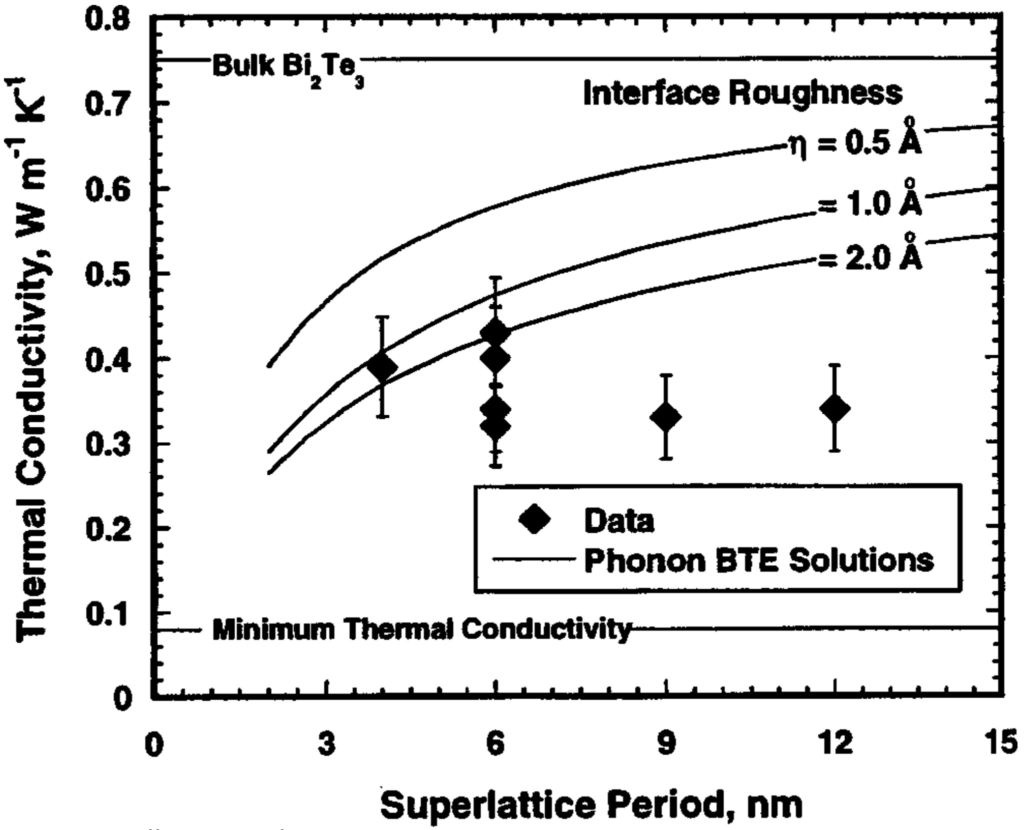
$$\tau = \frac{\delta^2 C}{\kappa}$$

$$\delta = \sqrt{\frac{\tau \kappa}{C}}$$

M. N. Touzelbaev, P. Zhou, R. Venkatasubramanian, and K. E. Goodson. Thermal characterization of Bi_2Te_3/Sb_2Te_3 superlattices. Journal of Applied Physics, 90:763–767, 2001.

Transient ThermoReflectance (TTR) measurements (optical)

Pump-probe: nanosecond pump



M. N. Touzelbaev, P. Zhou, R. Venkatasubramanian, and K. E. Goodson. Thermal characterization of Bi_2Te_3/Sb_2Te_3 superlattices. Journal of Applied Physics, 90:763–767, 2001.

$$\delta = \sqrt{\frac{\tau\kappa}{C}}$$

For low diffusivity materials, assuming τ = 1 ns

$$\delta = 30\,\text{nm}$$

For high diffusivity materials

$$\delta = 300\,\text{nm}$$

BUT – limited by temporal processes > τ = 1 ns

Transient ThermoReflectance (TTR) measurements (optical)

Pump-probe: femtosecond pump

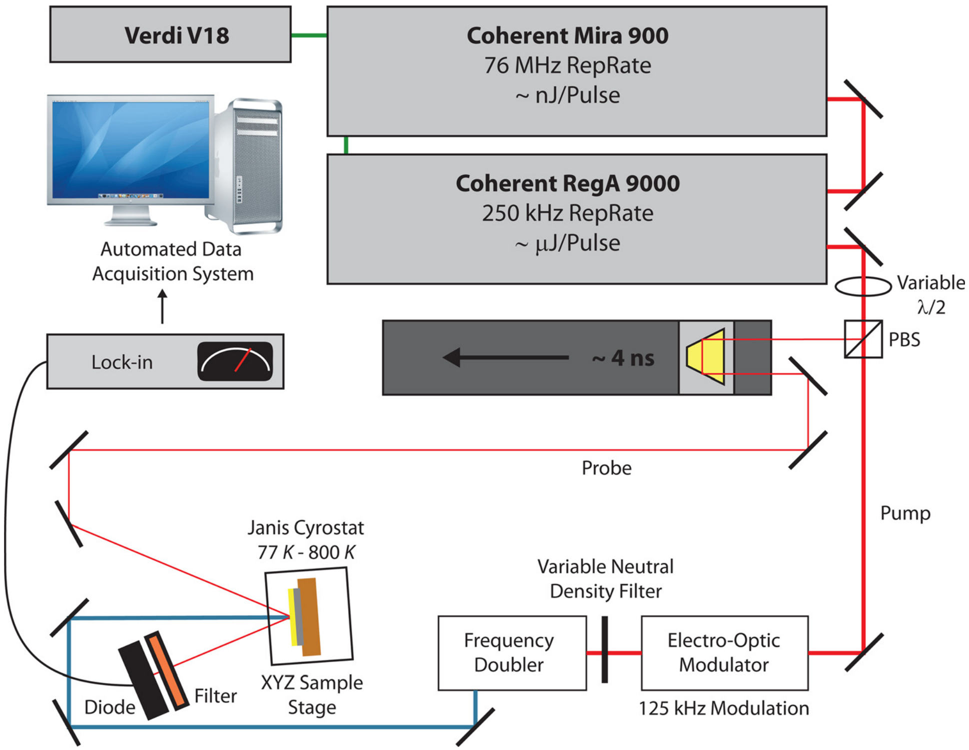


Fig. 2 Schematic of transient thermoreflectance setup at University of Virginia.

P. M. Norris, J. L. Smoyer, J. C. Duda, and P. E. Hopkins. Prediction and measurement of thermal transport across interfaces between isotropic solids and graphitic materials. Journal of Heat Transfer, 134:020910, 2012.

$$\delta = \sqrt{\frac{\tau \kappa}{C}}$$

For low diffusivity materials, assuming τ = 100 fs

$$\delta = 0.3\,\text{nm}$$

For high diffusivity materials

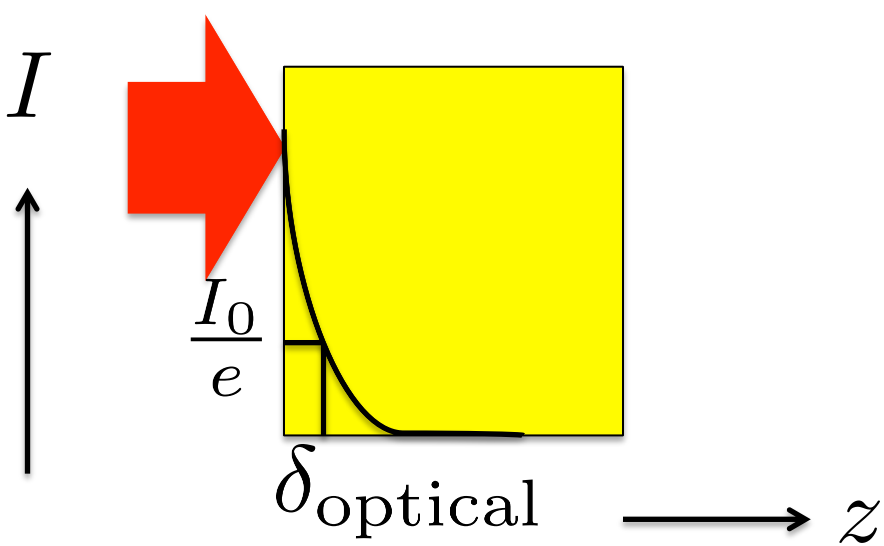
$$\delta = 3.0\,\text{nm}$$

Transient ThermoReflectance (TTR) measurements (optical)

Pump-probe: femtosecond pump

In this case, spatial resolution limited by optical penetration depth of metal

$$\delta_{\text{optical}} = \frac{\lambda}{4\pi k}$$



FANTASTIC temporal resolution (limited by pulse width)

Pulse absorption (~100 fs)

↓

Fermi relaxation and ballistic transport (few hundred fs)

↓

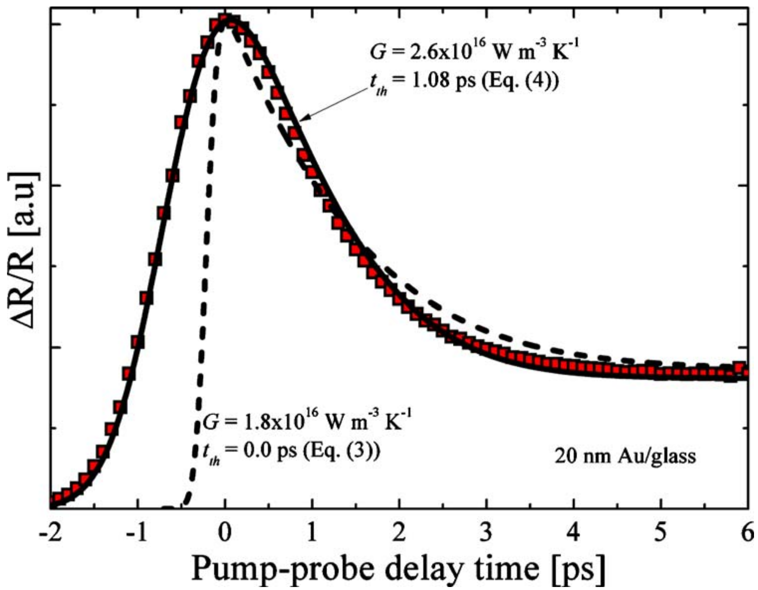
Electron-phonon coupling (a few ps)

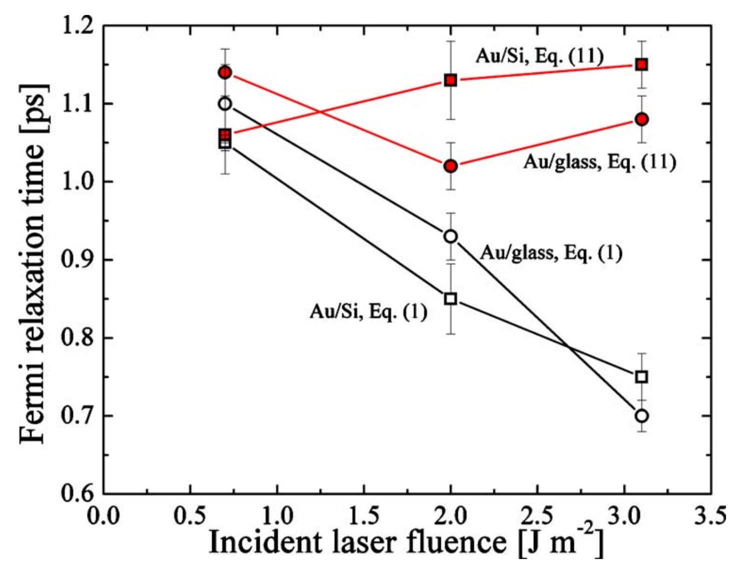
↓

Thermal diffusion (hundreds of ps to ns)

Transient ThermoReflectance (TTR) measurements (optical)

Pump-probe: femtosecond pump

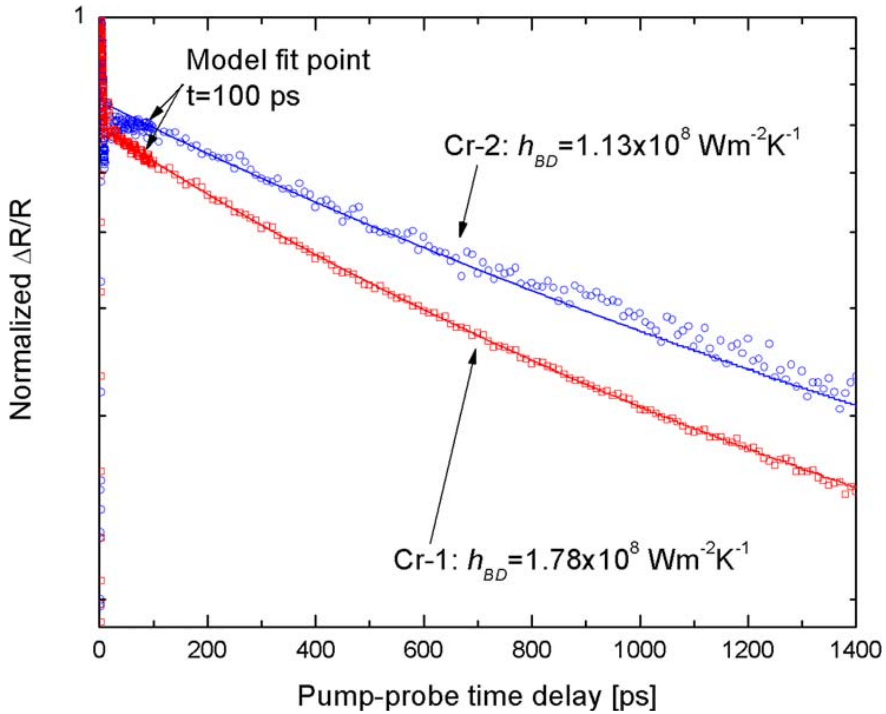


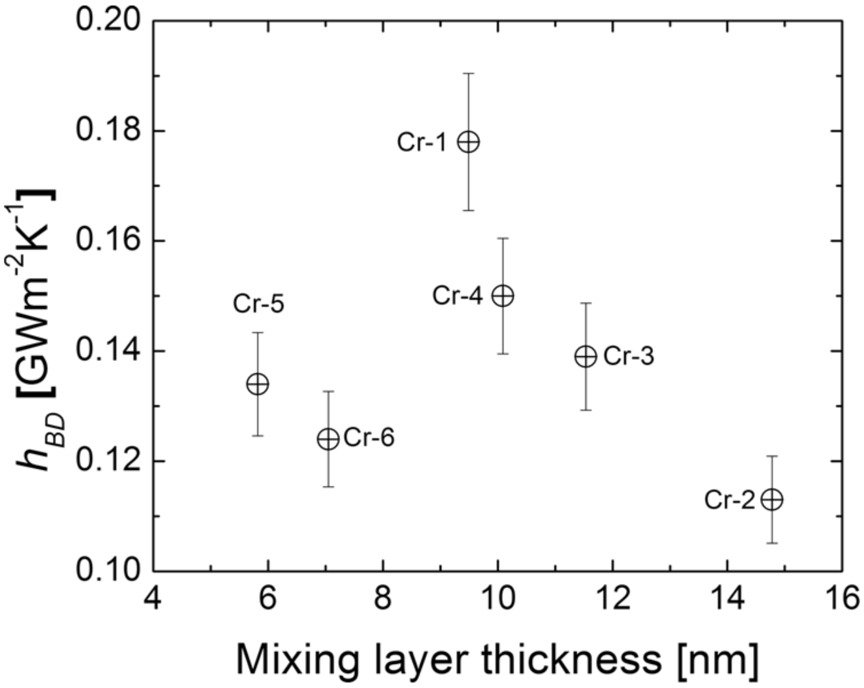


P. E. Hopkins, L. M. Phinney, and J. R. Serrano. Reexamining electron-fermi relaxation in gold films with a nonlinear thermoreflectance model. Journal of Heat Transfer, 133:044505, 2011

Transient ThermoReflectance (TTR) measurements (optical)

Pump-probe: femtosecond pump





P. E. Hopkins, P. M. Norris, R. J. Stevens, T. Beechem, and S. Graham. Influence of interfacial mix- ing on thermal boundary conductance across a chromium/silicon interface. Journal of Heat Transfer, 130:062402, 2008.

Steady state vs. transient vs. periodic

Steady state = The Fourier Law

$$q = -\kappa \frac{\partial T}{\partial z}$$

Transient = The Heat Equation

$$\rho C \frac{\partial T}{\partial t} = \kappa \frac{\partial^2 T}{\partial z^2} + q(t)$$

Heat capacity enters the picture

The source term can make a difference $q\left(t\right)$ vs. $q\left(t, \omega\right)$

If source term is periodic (and not “single shot, or instantaneous), then you get a modulated temperature on your samples surface

1) This yields both steady state and transient components
2) Makes data analysis easier since you can work in frequency domain

Periodic measurements

What separates periodic measurements from everything else????

$$\delta_{\mathrm{thermal}} = \sqrt{\frac{\kappa}{\pi f C}} = \sqrt{\frac{2\kappa}{\omega C}}$$

Thermal penetration depth

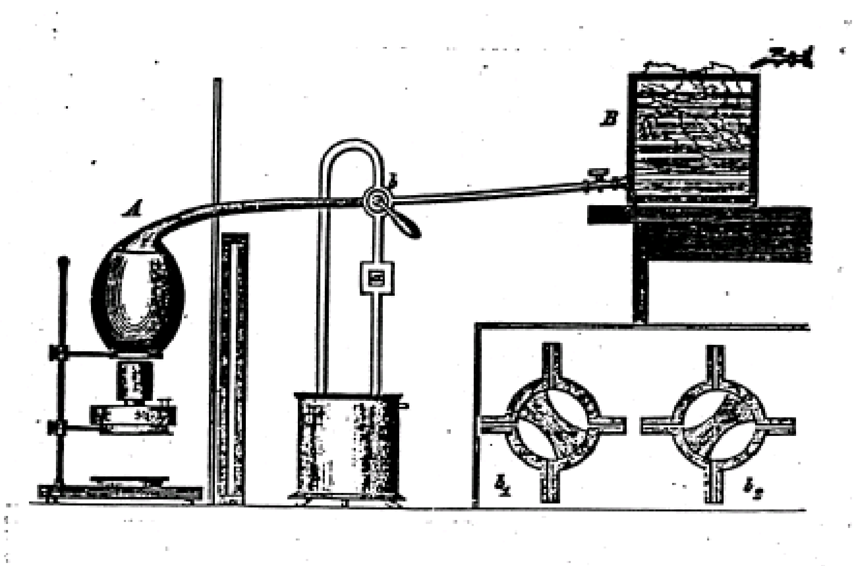
Ångström method

Used fixed temperature boundary conditions

$$T\left(x=0\right)=0^{\circ}\mathrm{C} \qquad 0<t<\Gamma/2$$

$$T\left(x=0\right)=100^{\circ}\mathrm{C} \qquad \Gamma/2<t<\Gamma$$

where Γ is the period of temperature oscillations produced by alternating flow of ice water and steam



Frequency dependent temperature rise leads to temperature fluctuation at end of sample with some phase lag based on RC

Modified Ångström method

JOURNAL OF APPLIED PHYSICS VOLUME 95, NUMBER 4 15 FEBRUARY 2004

Thermal contact conductance of adhered microcantilevers

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Leslie M. Phinney[b)]
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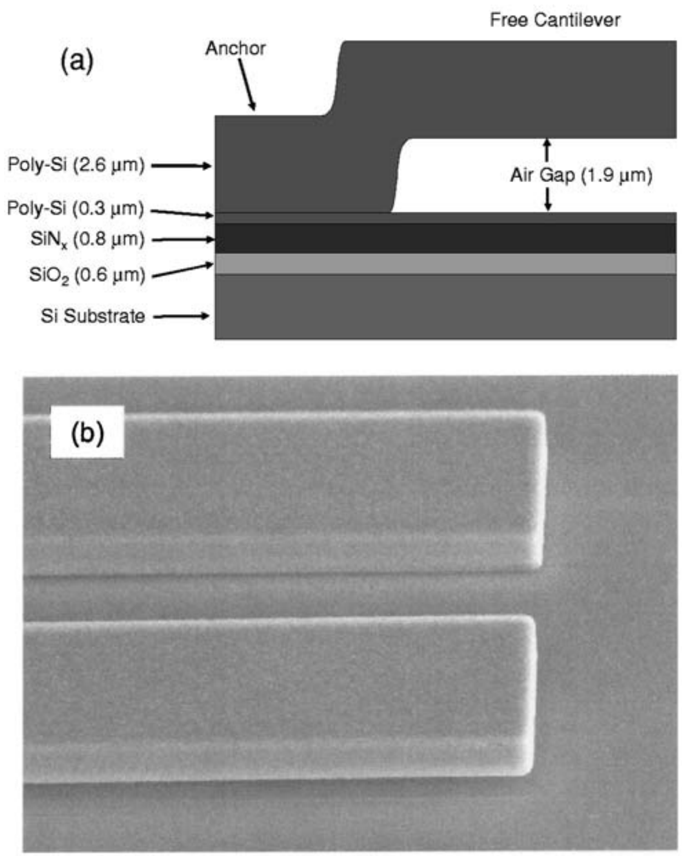
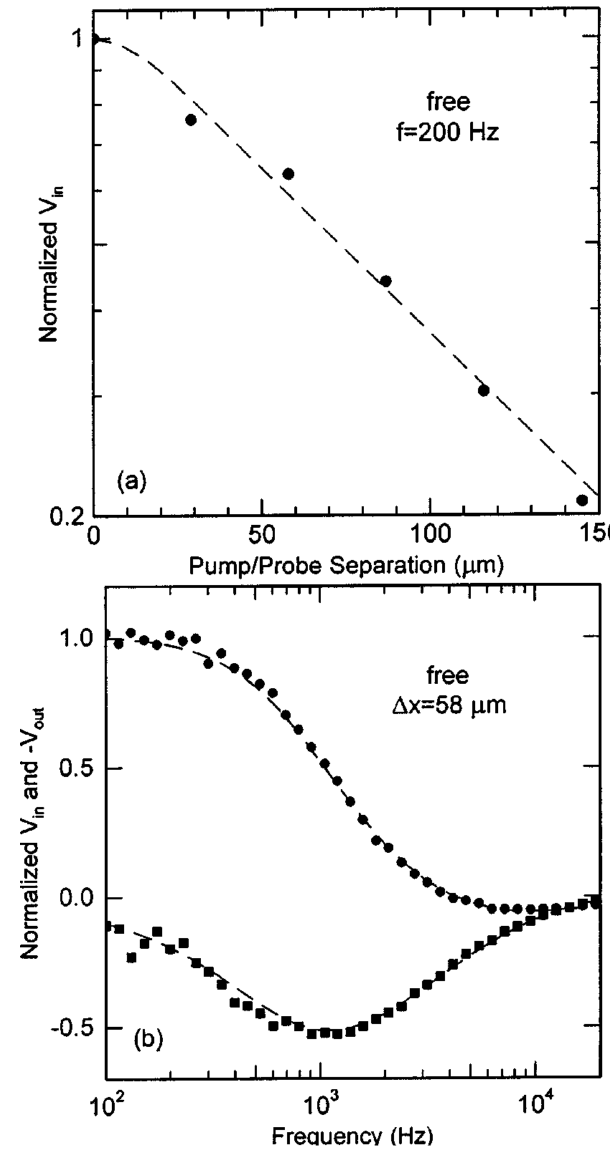
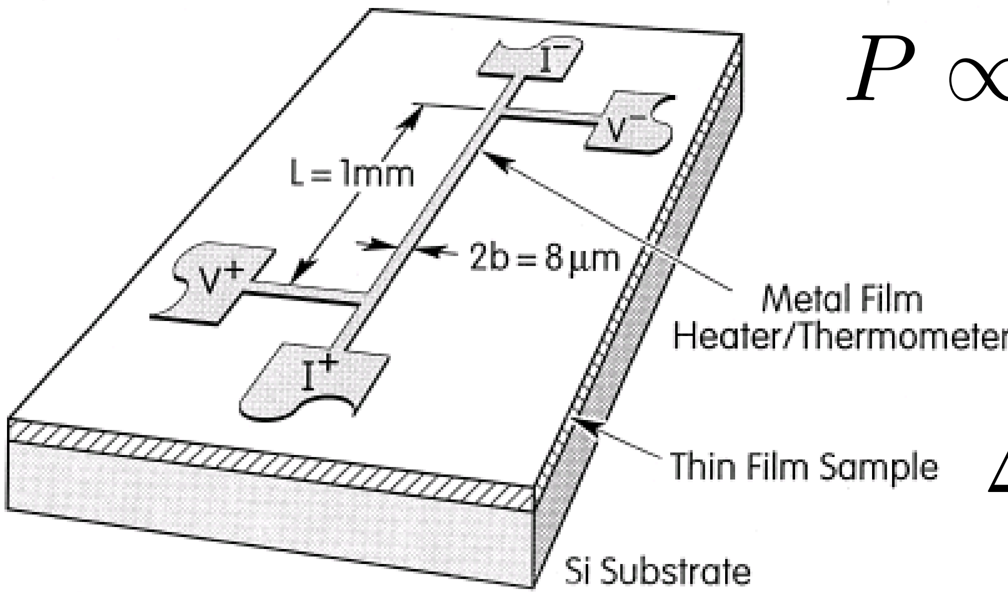




3ω technique

Uses single metal film for heater/thermometer (Birge, 1987); (Cahill, 1990).

$$I \propto \exp\left[i\omega t\right]$$

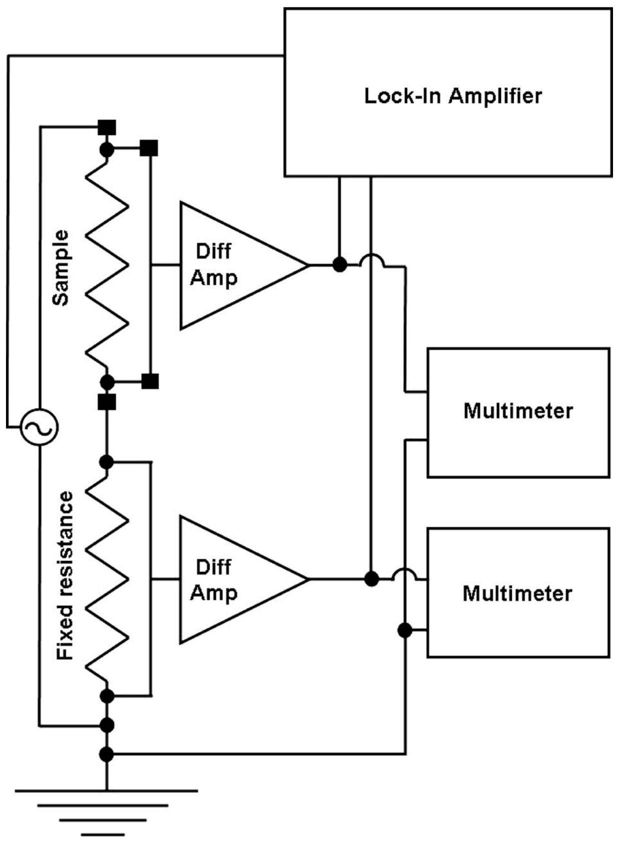


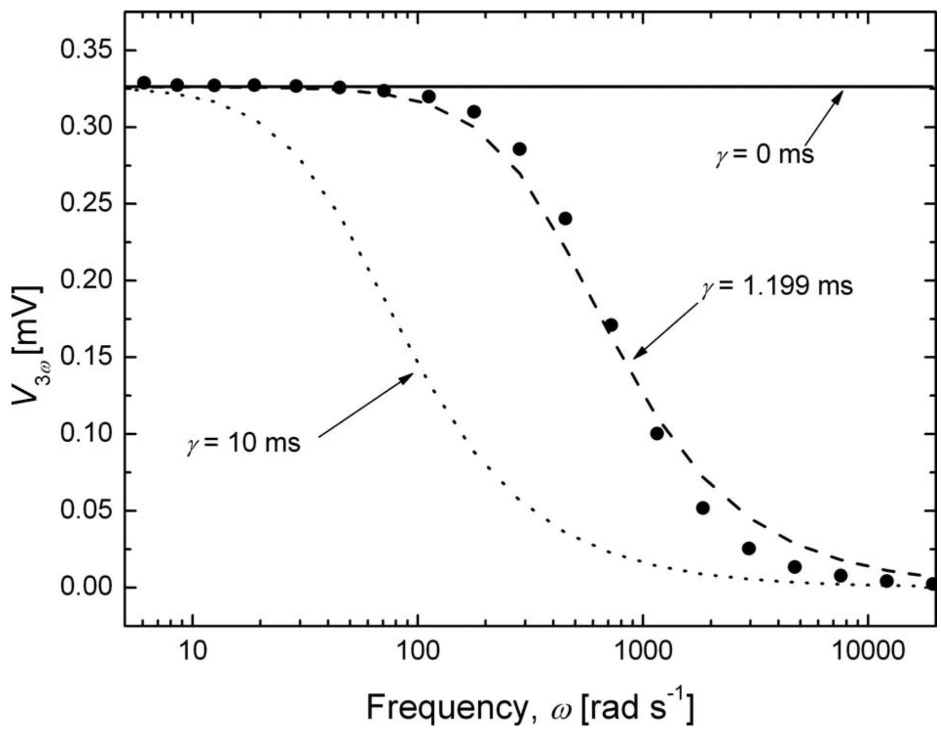
$$P \propto \Delta T \propto \exp\left[i2\omega t\right]$$

$$\Delta R \propto \exp\left[i2\omega t\right]$$

$$\Delta V = I\Delta R \propto \exp\left[i3\omega t\right]$$

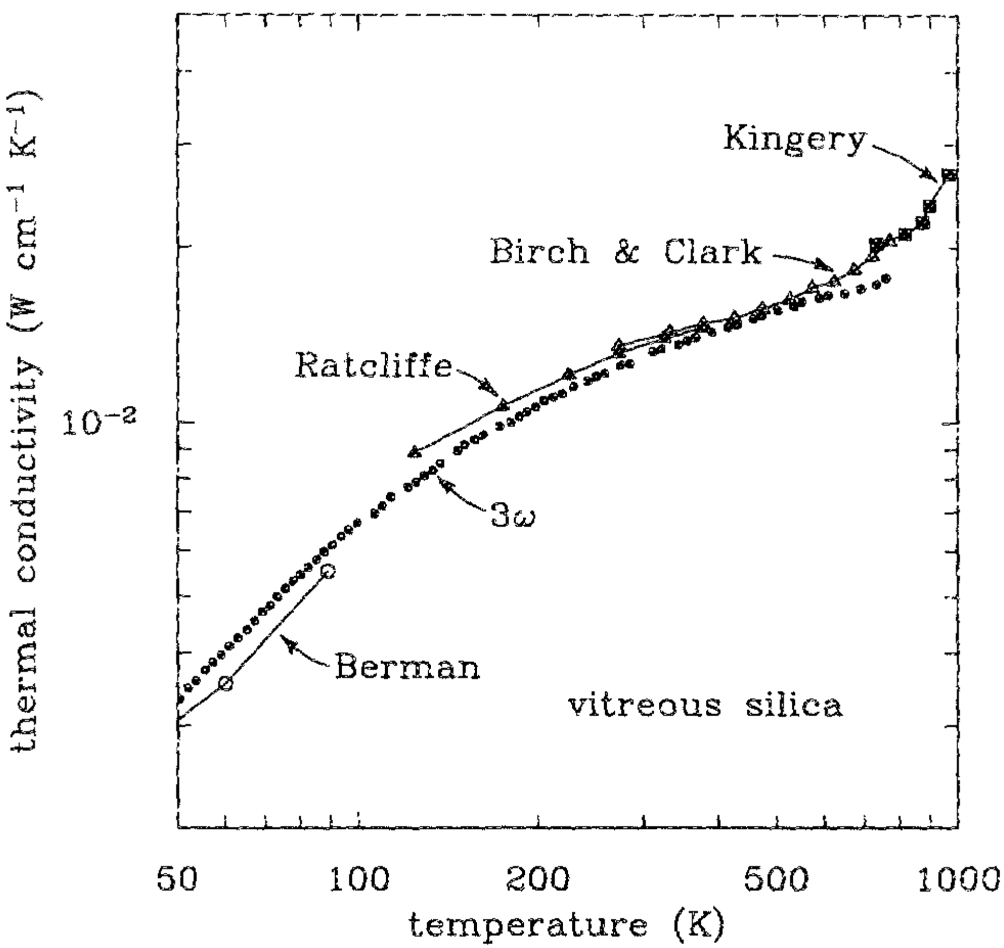
3ω technique





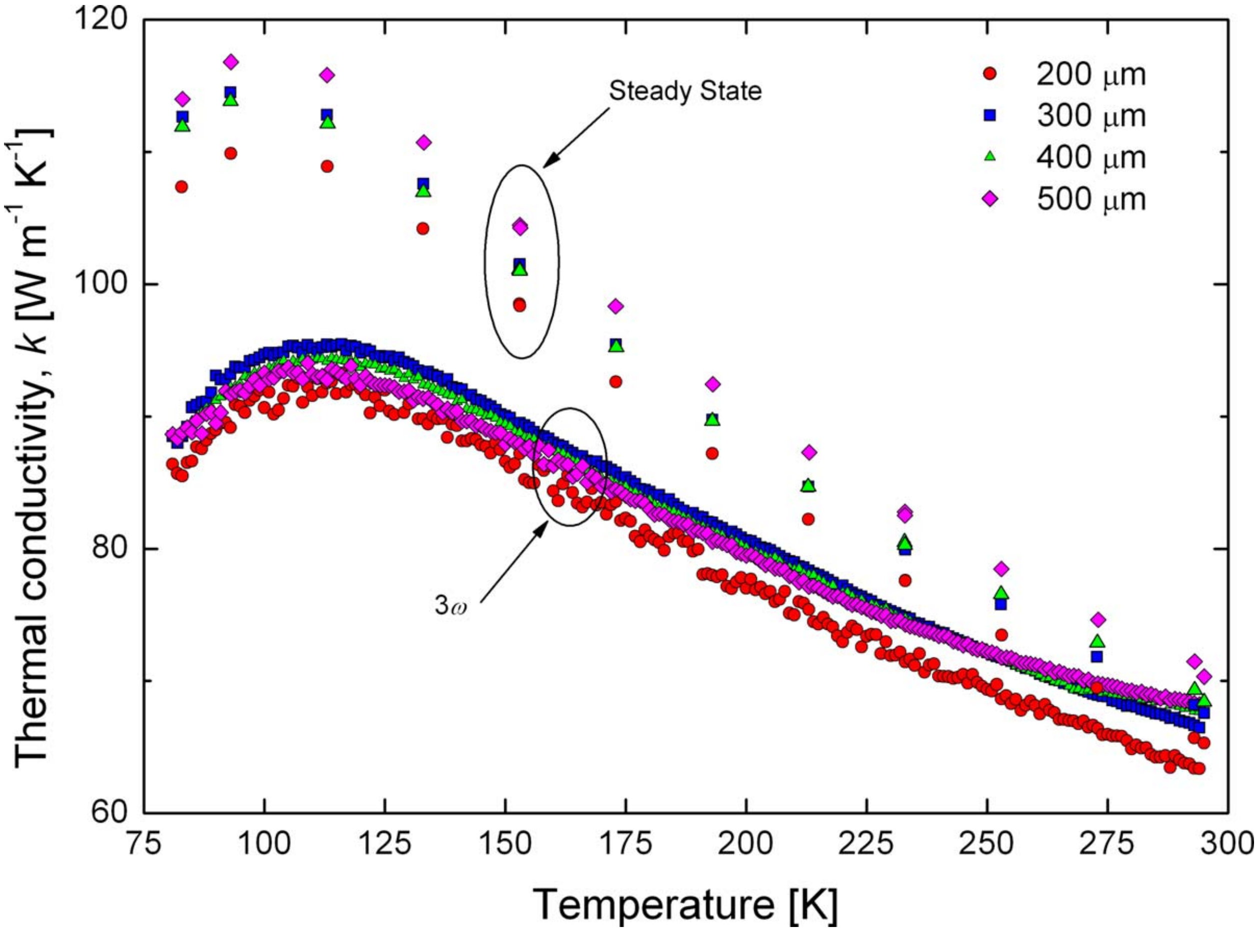
P. E. Hopkins and L. M. Phinney. Thermal conductivity measurements on polycrystalline silicon micro- bridges using the 3w technique. Journal of Heat Transfer, 131:043201, 2009.

3ω technique



D. G. Cahill. Thermal conductivity measurement from 30 to 750 K: The 3ω method. Review of Scientific Instruments, 61:802–808, 1990.

3ω vs. steady state



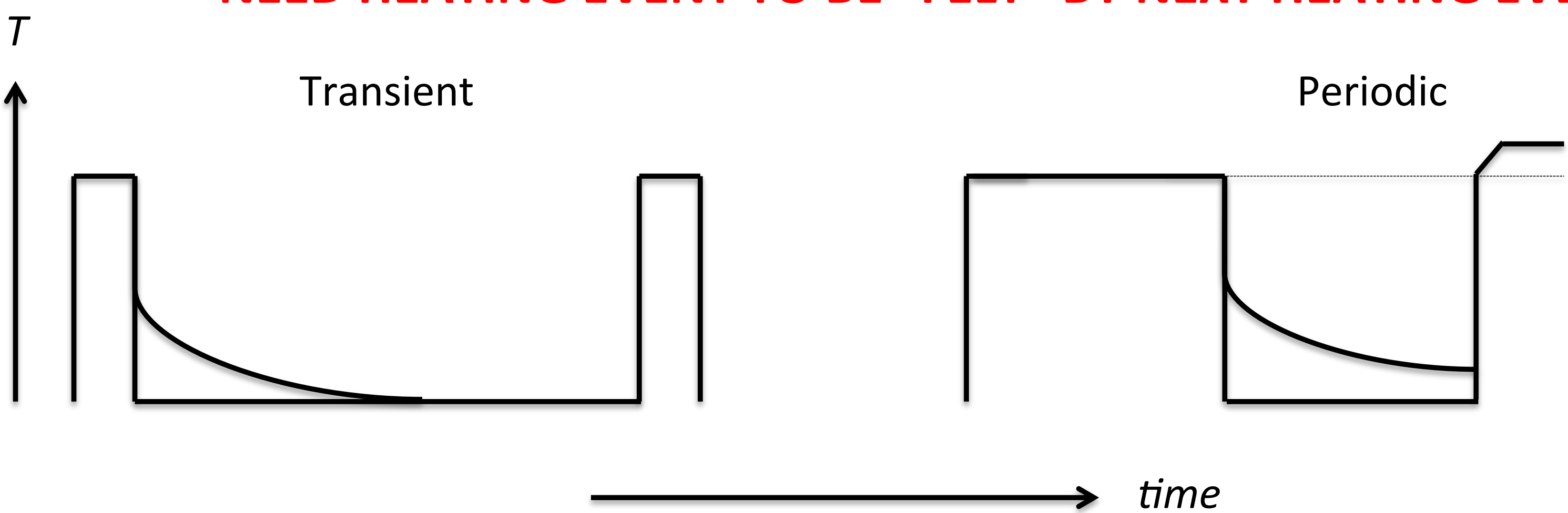
What could differences be caused by?

What about optical periodic heating techniques?

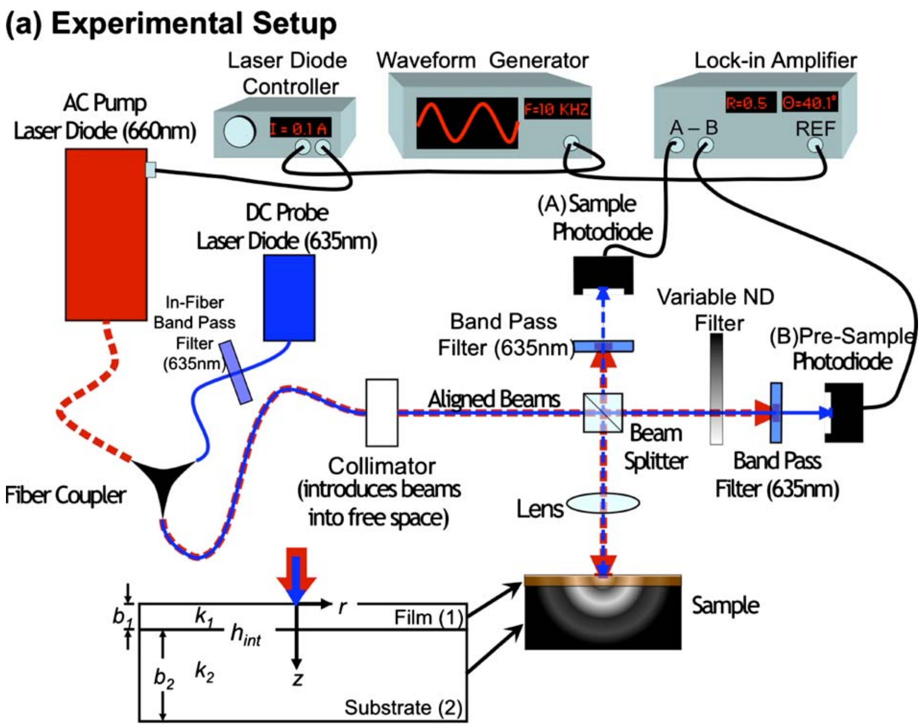
$$q\left(t\right) \ \text{vs.} \ q\left(t,\omega\right)$$

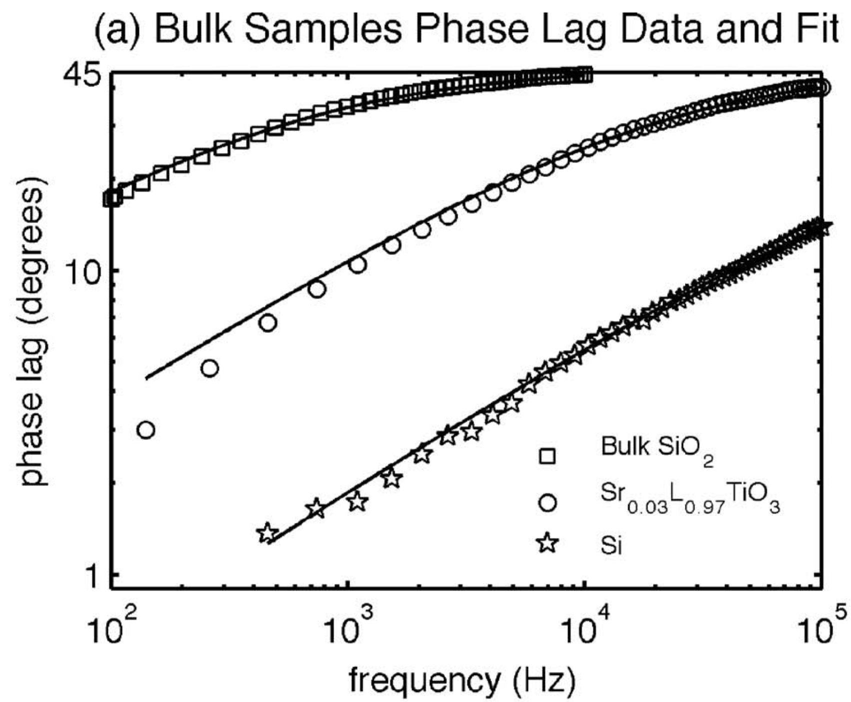
If source term is periodic (and not "single shot, or instantaneous), then you get a modulated temperature on your samples surface

THE KEY IS THE DUTY CYCLE!!!!
NEED HEATING EVENT TO BE "FELT" BY NEXT HEATING EVENT



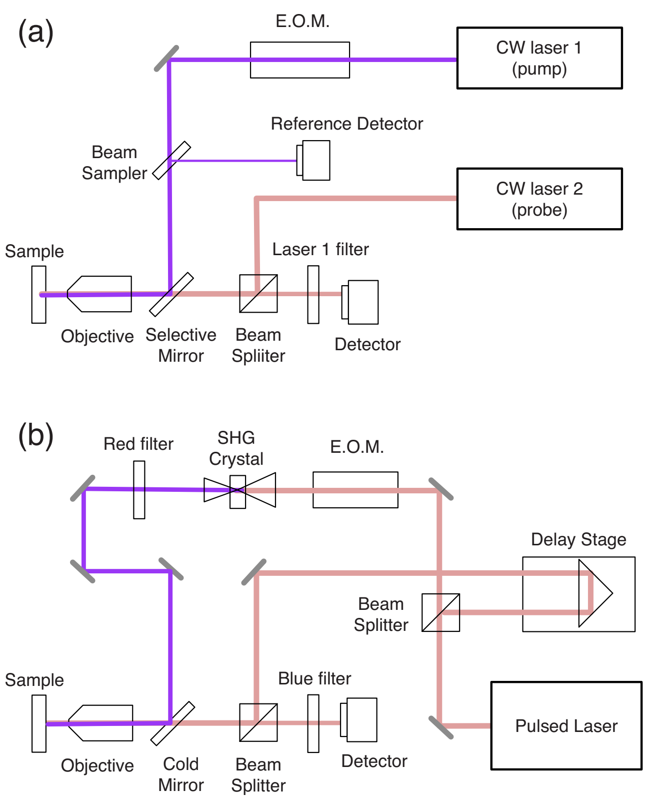
CW-Frequency domain thermoreflectance

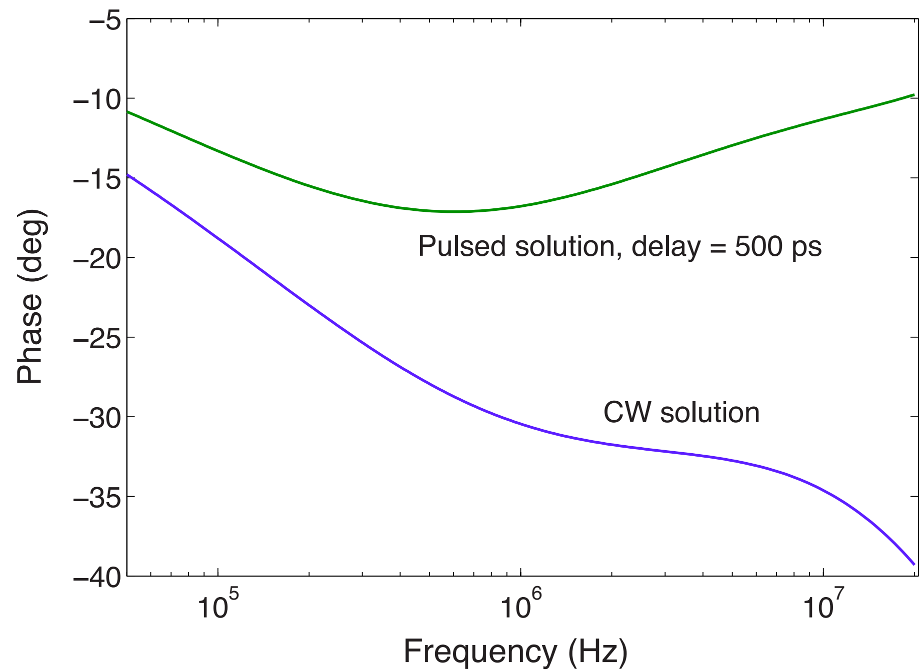




J. A. Malen, K. Baheti, T. Tong, Y. Zhao, J. A. Hudgings, and A. Majumdar. Optical measurement of thermal conductivity using fiber aligned frequency domain thermoreflectance. Journal of Heat Transfer, 133(8):081601, 2011.

FDTR – cw vs. pulsed



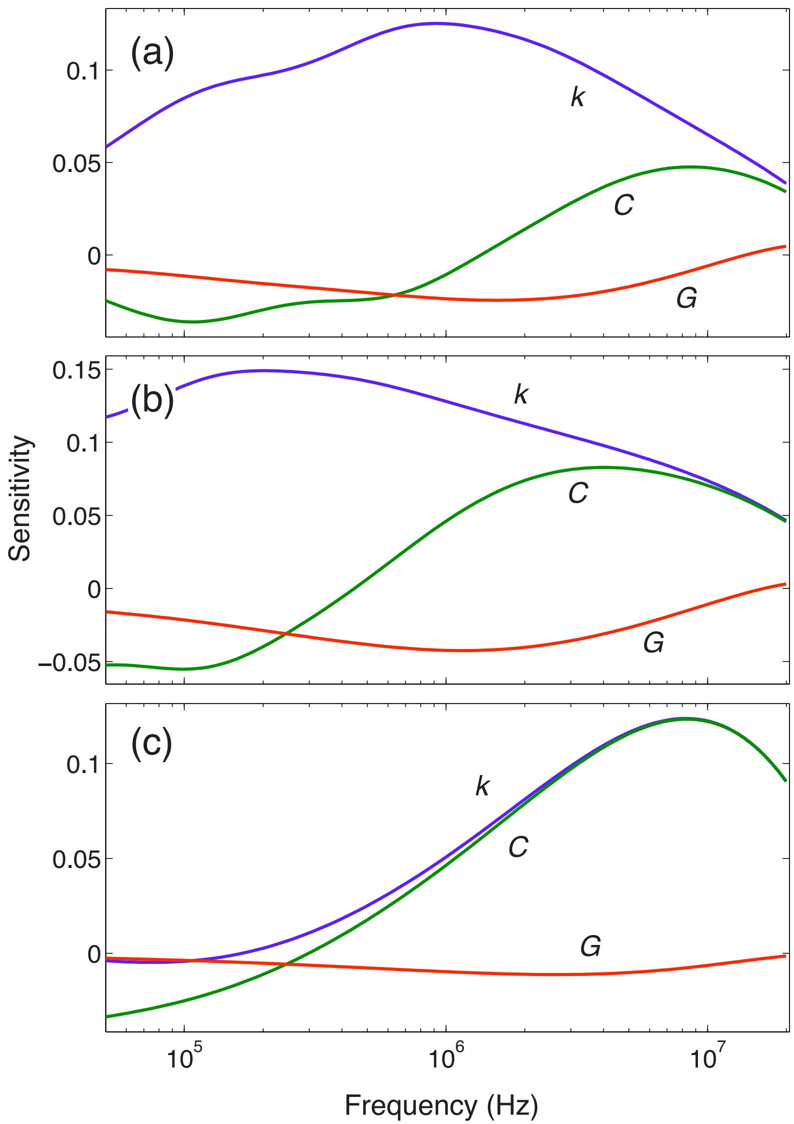


$$\delta_{\text{cw}} = \sqrt{\frac{\kappa}{\pi f C}}$$

$$\delta_{\text{pulsed}} \propto \sqrt{\tau \frac{\kappa}{C}}$$

A. J. Schmidt, R. Cheaito, and M. Chiesa. A frequency-domain thermoreflectance method for the characterization of thermal properties. Review of Scientific Instruments, 80:094901, 2009.

FDTR – pulsed



$$D = \frac{\kappa}{C} \qquad E = \sqrt{\kappa C}$$

Silicon

Where are the diffusivity and effusivity regimes?

Sapphire

In what materials can you accruately determine both C and κ?

Pyrex (SiO_2)

A. J. Schmidt, R. Cheaito, and M. Chiesa. A frequency-domain thermoreflectance method for the characterization of thermal properties. Review of Scientific Instruments, 80:094901, 2009.

FDTR – pulsed

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Characterization of thin metal films via frequency-domain thermoreflectance

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(Received 30 October 2009; accepted 12 December 2009; published online 27 January 2010)

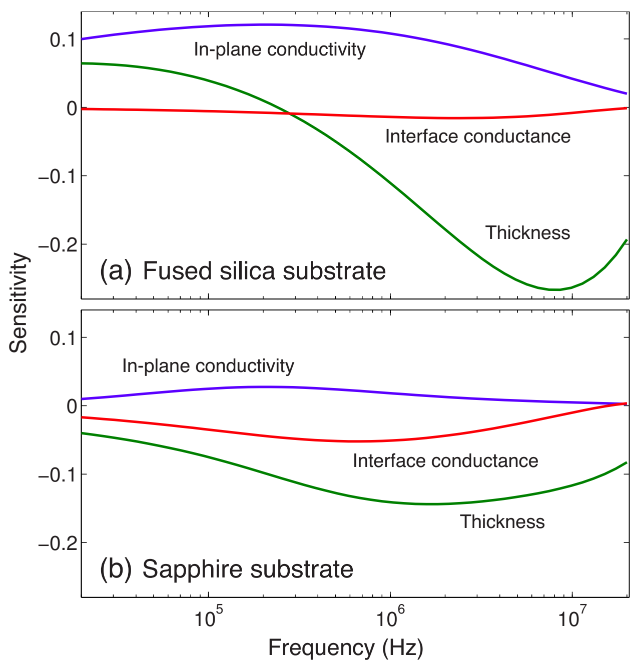


FIG. 5. (Color online) (a) The sensitivity parameter, Eq. (5), for in-plane thermal conductivity, metal-substrate boundary conductance, and metal thickness, for a sample consisting of an 80 nm film of Au deposited on a fused silica substrate. (b) The same sensitivities, calculated for a sapphire substrate. The phase angle in Eq. (5) is taken in radians.

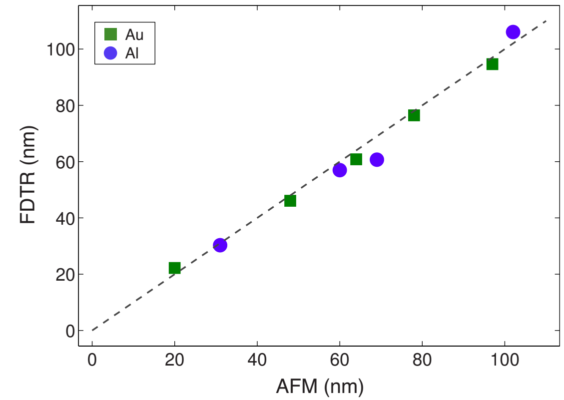


FIG. 3. (Color online) Film thickness data obtained for Au and Al films on fused silica substrates. The ordinate is the thickness determined from AFM cross sections while the abscissa is the FDTR value. Error bars based on two standard deviations are approximately the size of the symbols used.

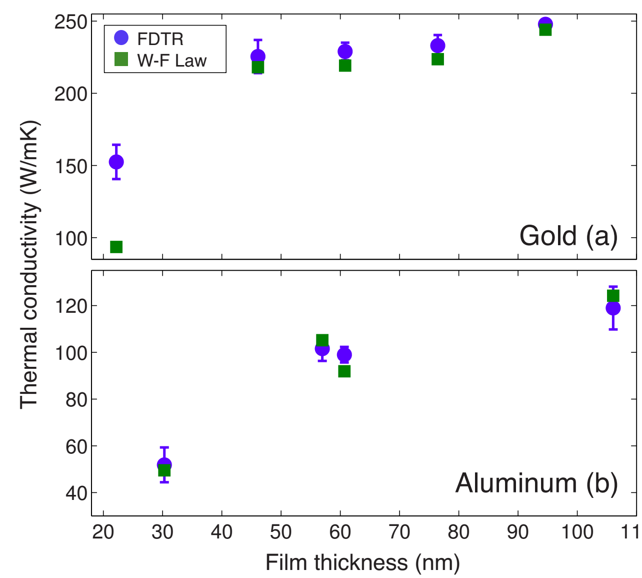
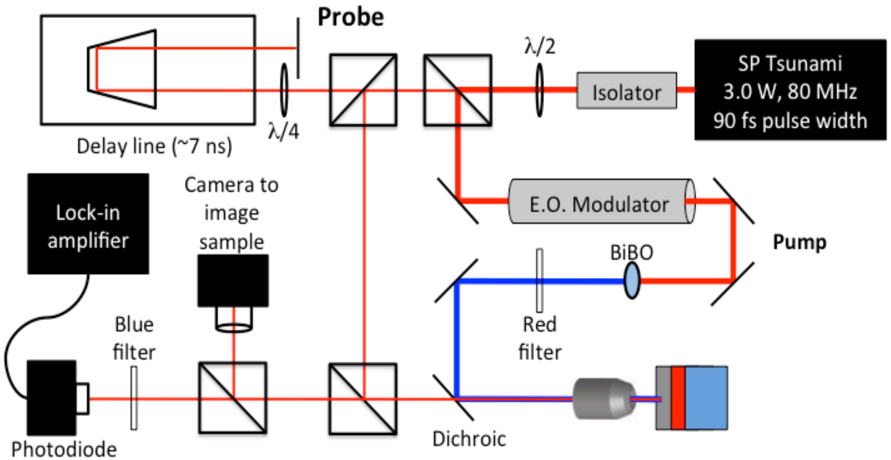
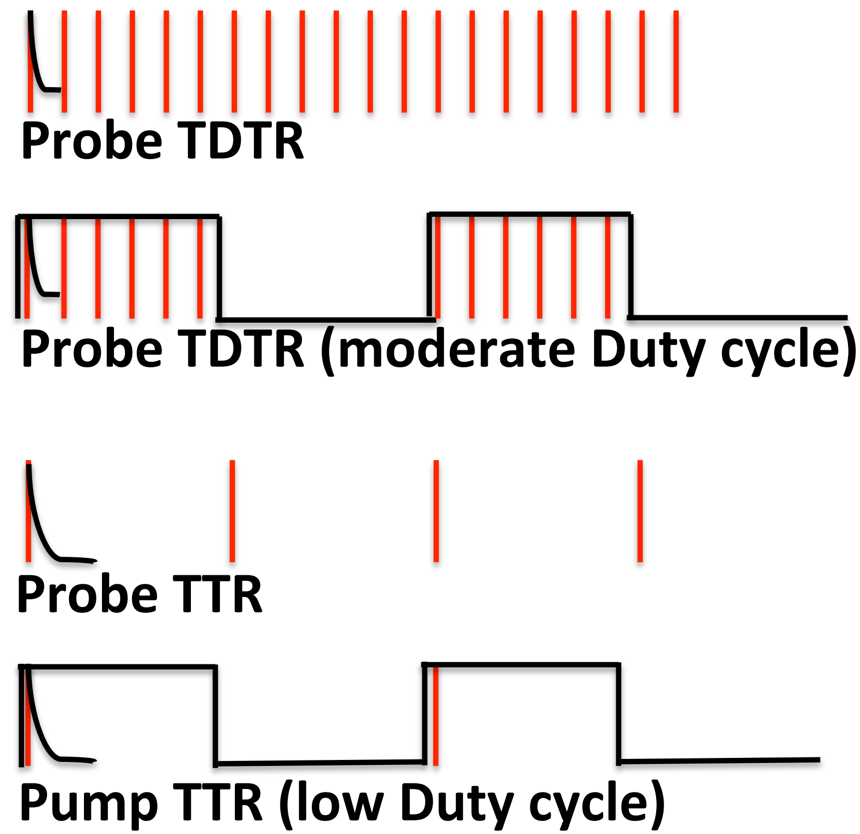


FIG. 4. (Color online) Thermal conductivity data obtained for (a) Au and (b) Al films on fused silica substrates. Circles are values obtained with the FDTR method, while the squares are values computed from electrical conductivity measurements using the WF law.

Can we achieve transient AND periodic thermometry?

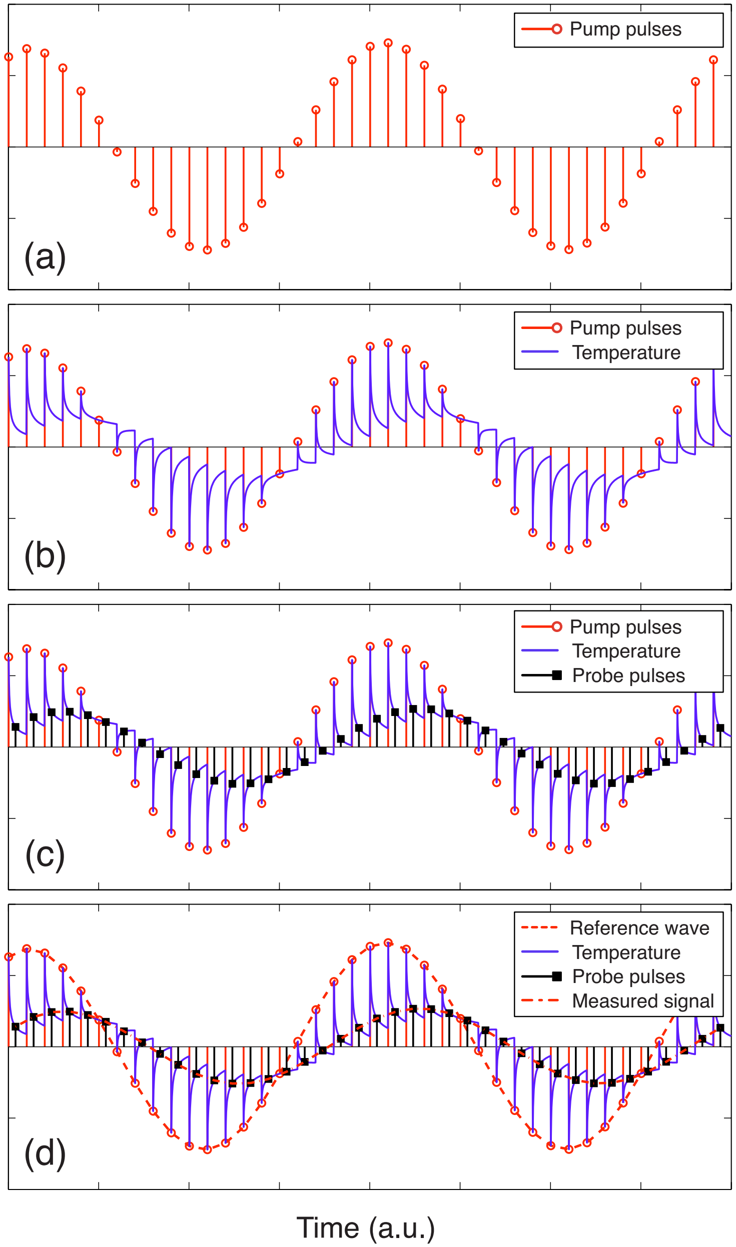
Time domain thermoreflectance

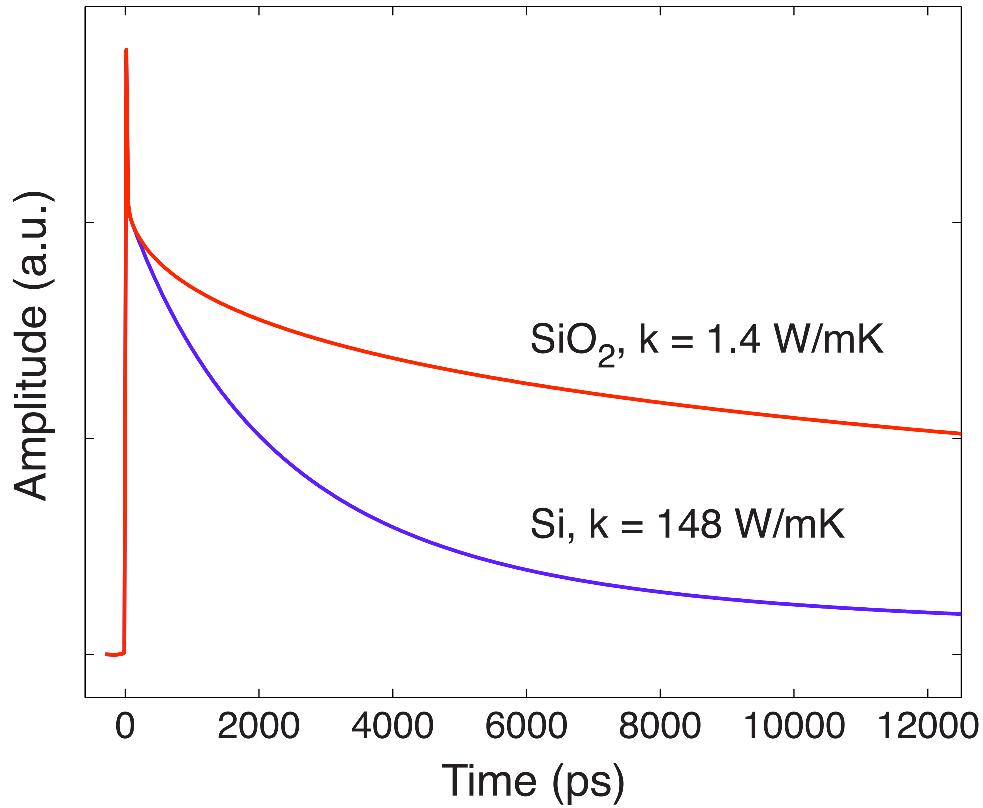




Use both the transient AND periodic response from the short pulsed heating event. Use high rep. rate laser and modulate at some frequency with moderate to high Duty cycle.

TDTR





A. J. Schmidt, X. Chen, and G. Chen. Pulse accumulation, radial heat conduction, and anisotropic ther- mal conductivity in pump-probe transient thermoreflectance. Review of Scientific Instruments, 79:114902, 2008.

TDTR – depth profiling by enhancing sensitivities to different parameters

Effect of dislocation density on thermal boundary conductance across GaSb/GaAs interfaces

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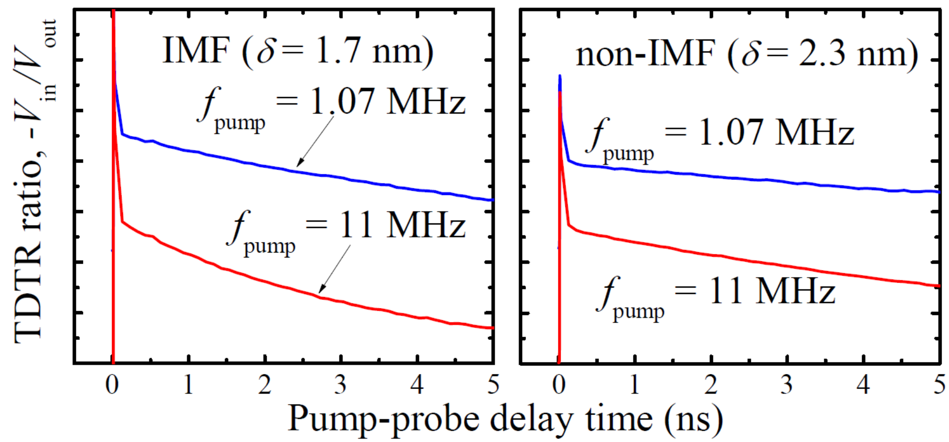
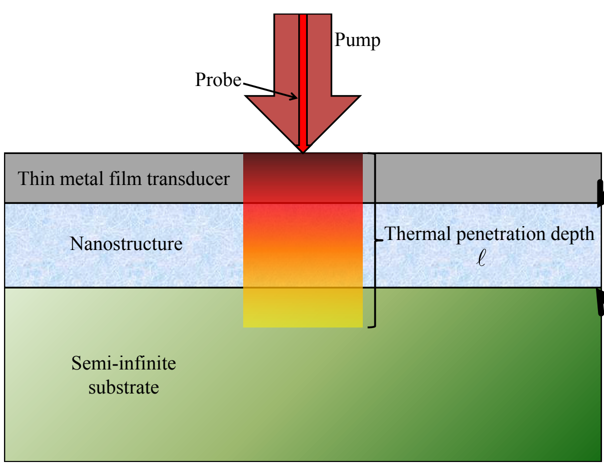


Increase how "deep" you probe in TDTR by decreasing the frequency



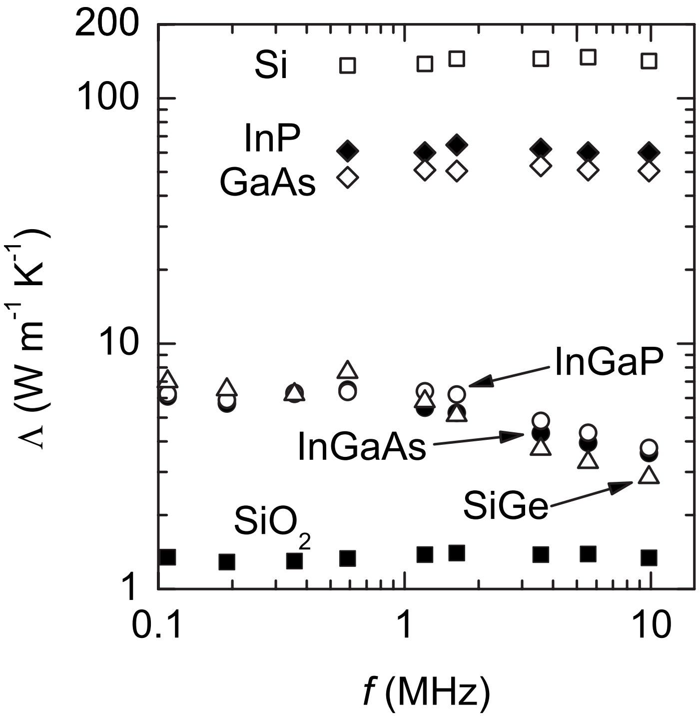
h_K – Al/GaSb (f = 11 MHz)

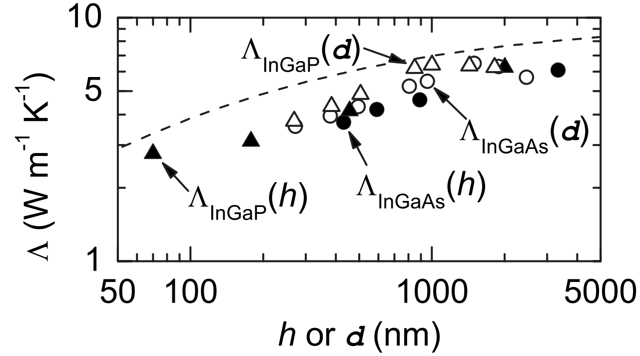
$$\ell \approx \sqrt{\frac{\kappa}{\pi C f_{\mathrm{pump}}}}$$

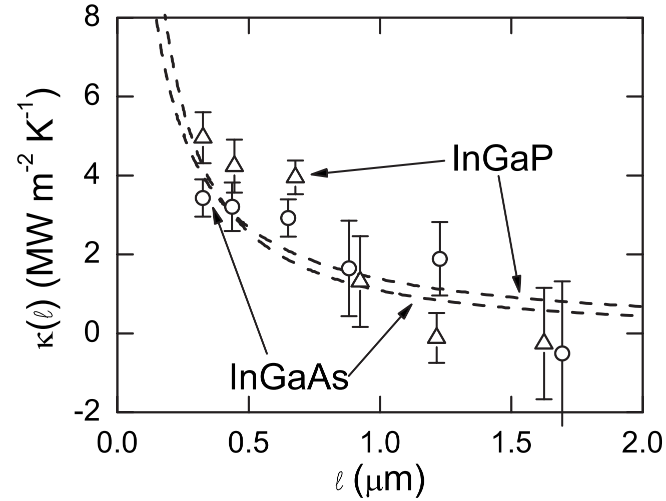
h_K – GaSb/GaAs (f = 1.07 MHz)

TDTR – modulation frequency dependence

$$\delta_{\text{thermal}} = \sqrt{\frac{\kappa}{\pi f C}} = \sqrt{\frac{2\kappa}{\omega C}}$$







Y. K. Koh and D. G. Cahill. Frequency dependence of the thermal conductivity of semiconductor alloys. Physical Review B, 76:075207, 2007.

Phonon "escape" from thermal penetration depth