Steady-State Thermoreflectance: A Practical Guide

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Steady-State Thermoreflectance (SSTR): Comparison to TDTR & FDTR

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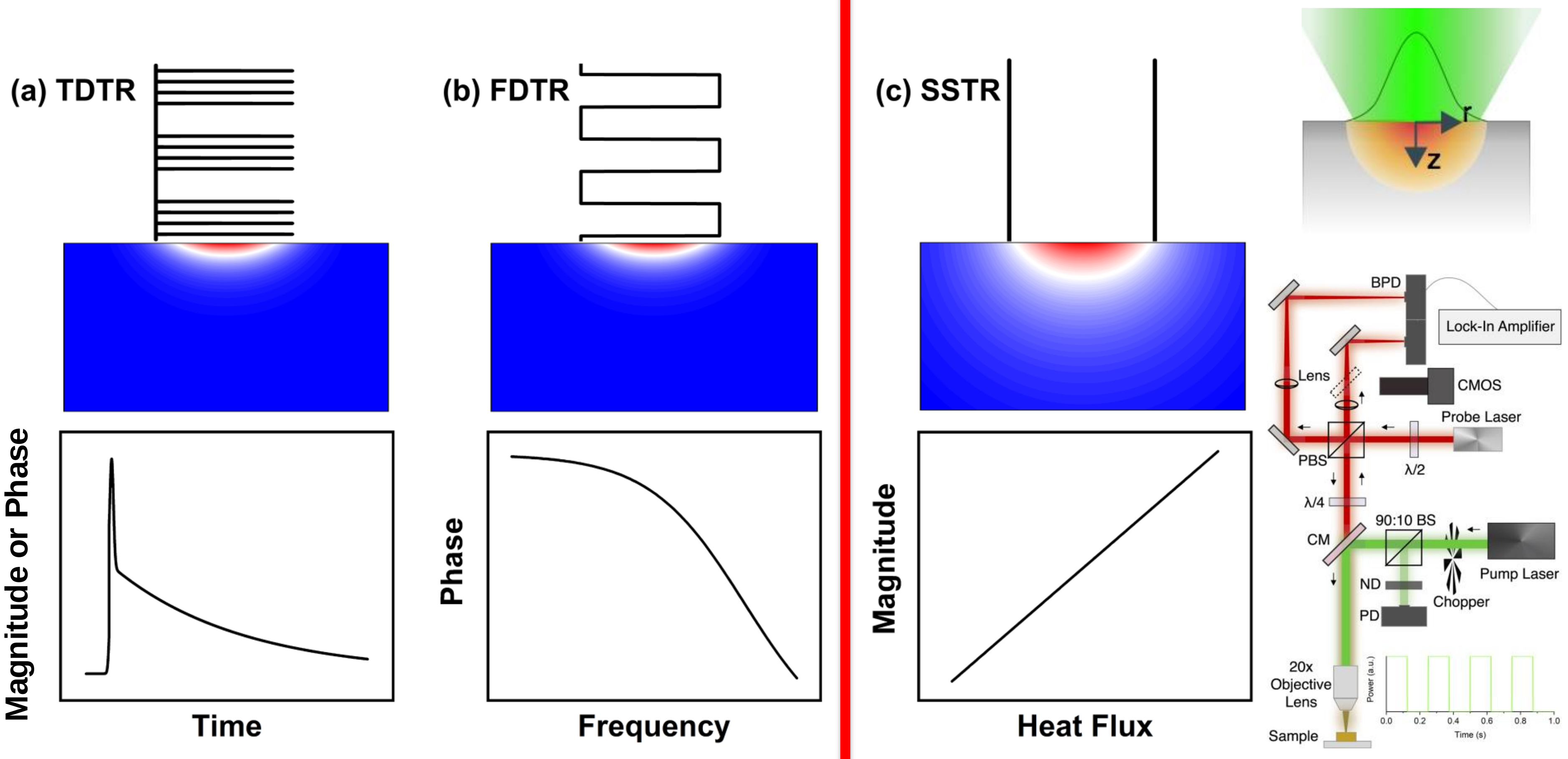

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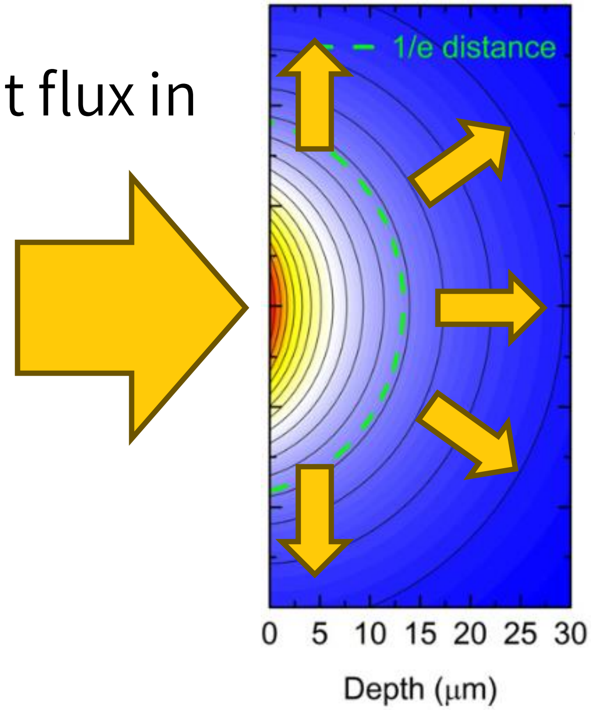
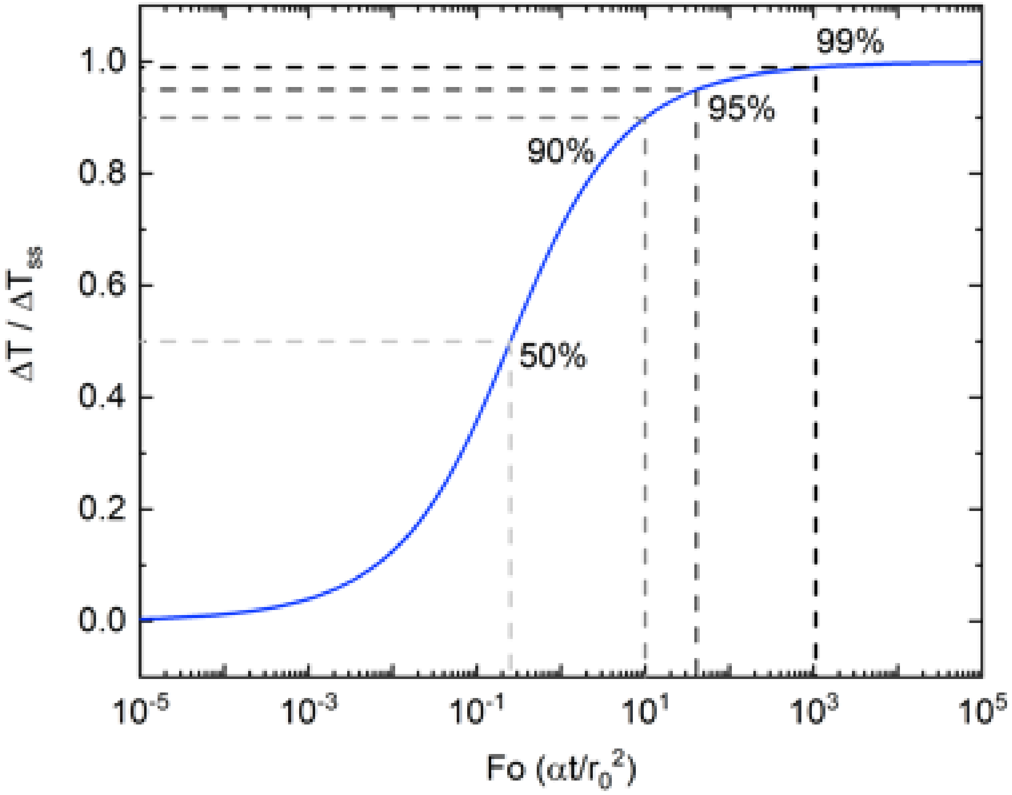
What does “steady-state” mean?

$$q'' = -\kappa \nabla T$$

Heat flux in



= Heat flux out

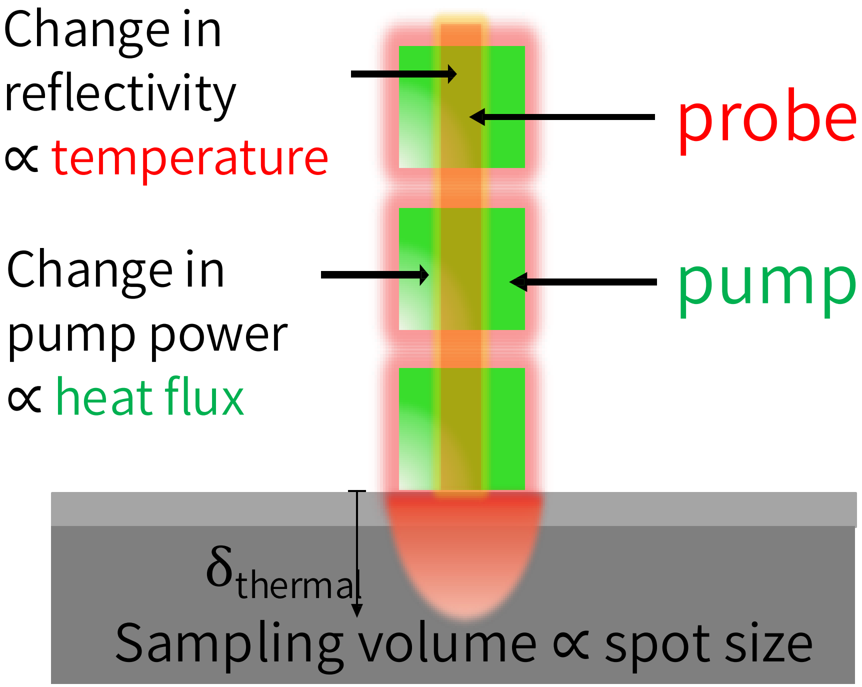


α = Thermal diffusivity

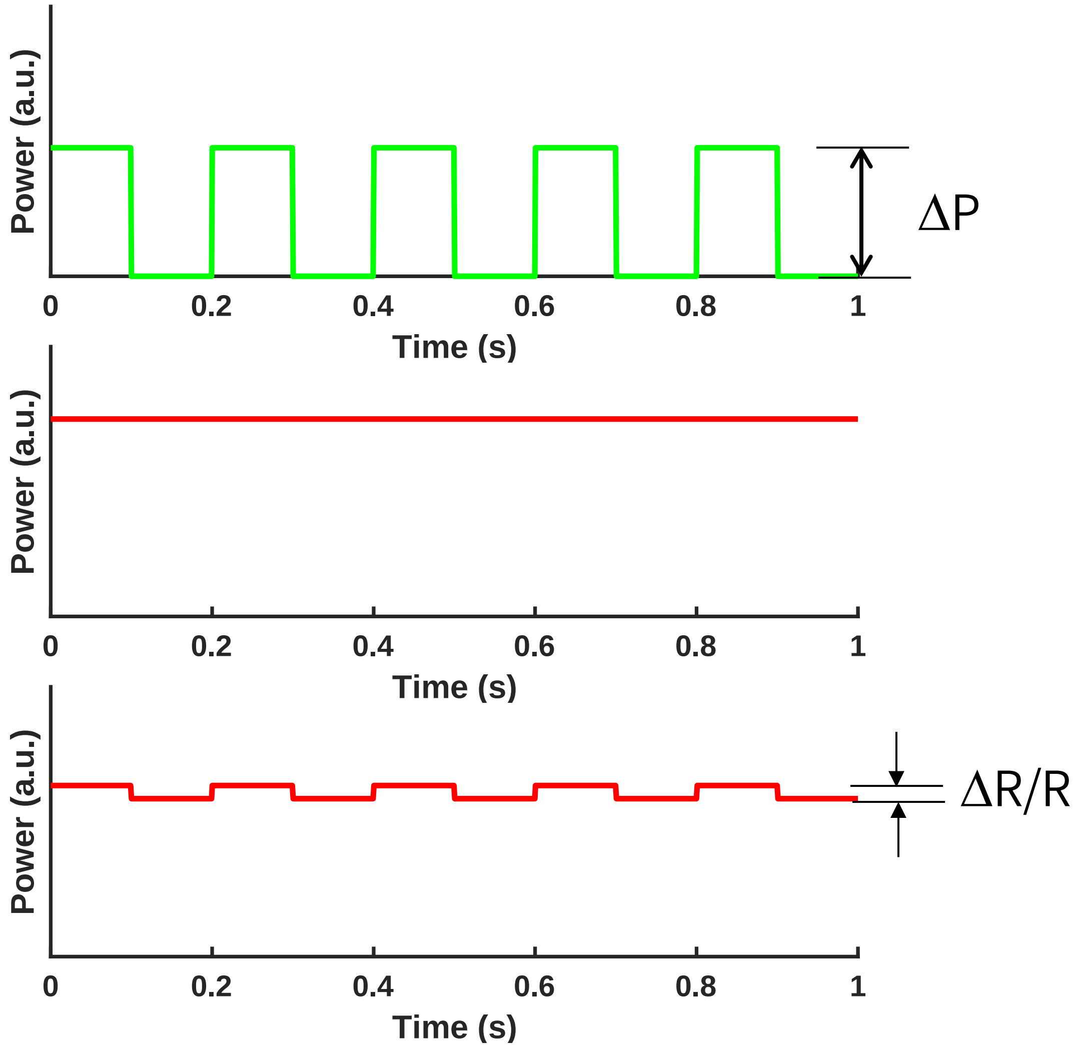
r_0 = 1/e^2 radius of laser

Steady-State Thermoreflectance (SSTR): How it works

Measurement Concept: Heat a small volume of the sample to a steady-state temperature, measure ΔT between “on” and “off” state



Depth measurement proportional to spot size
Spot size can be as small as <1 micron



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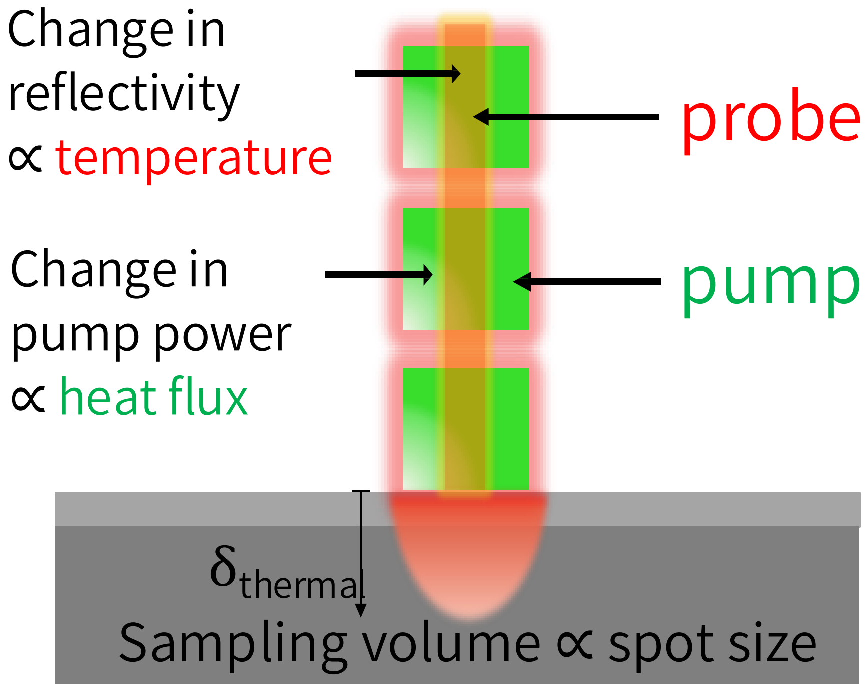

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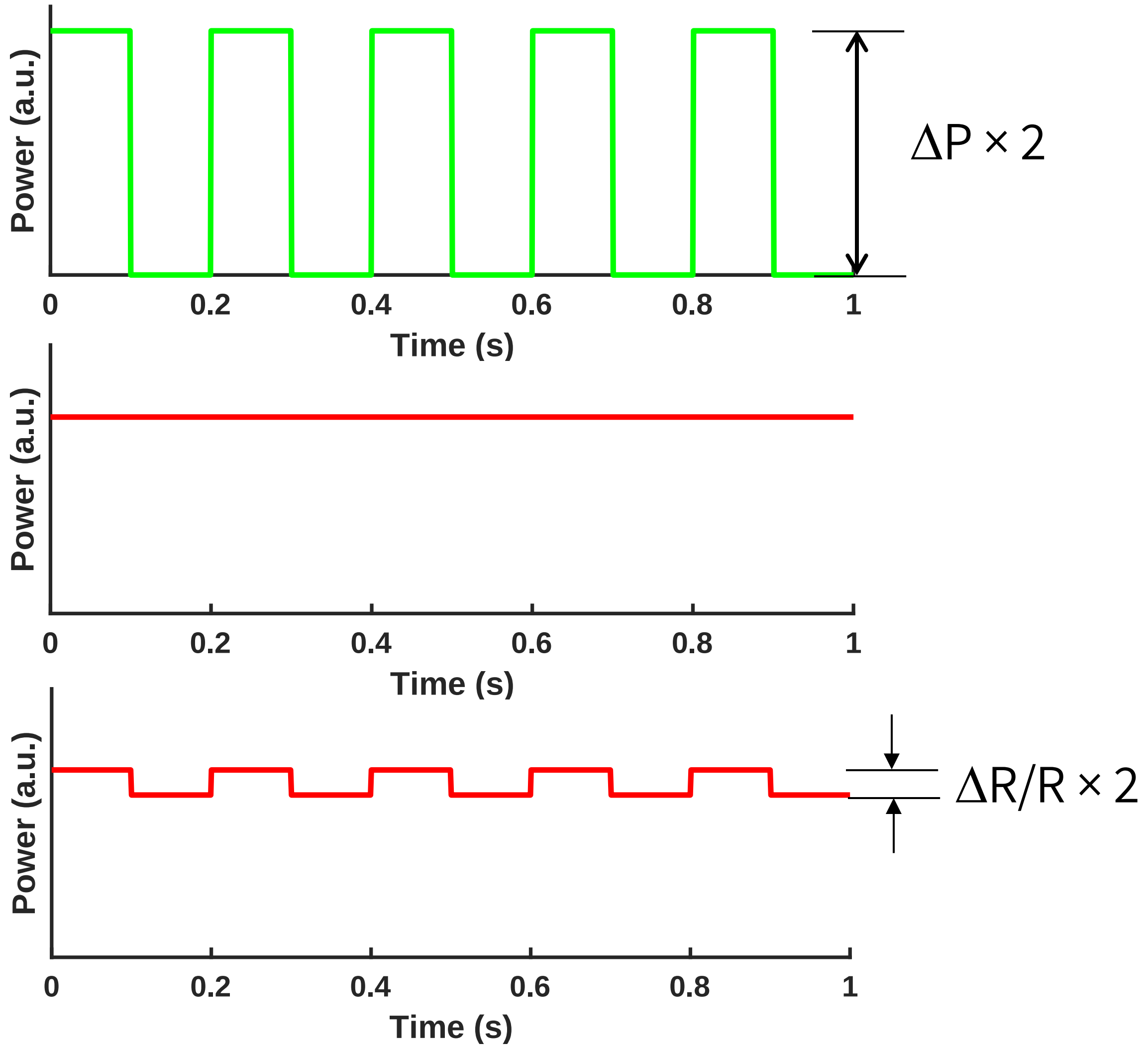
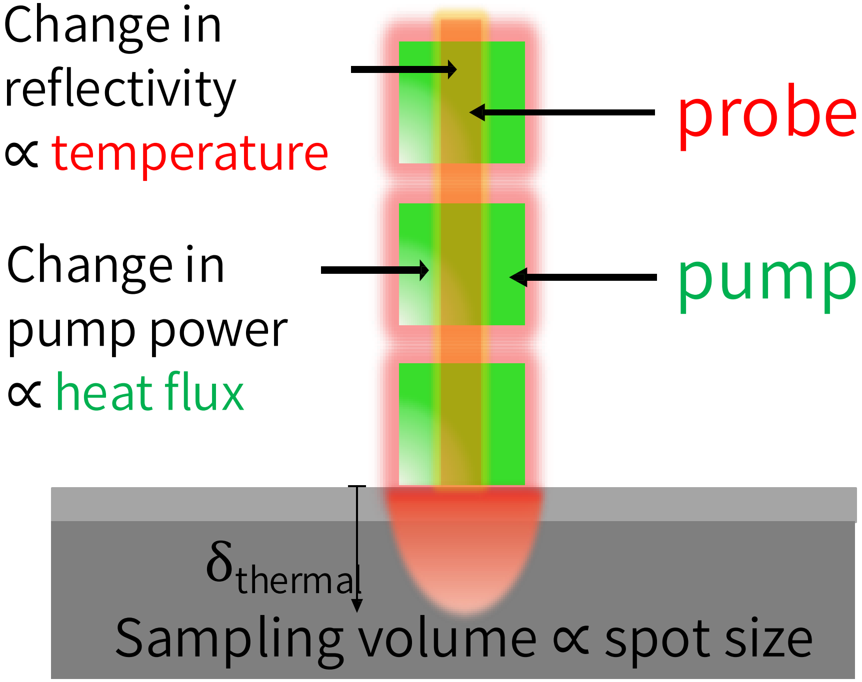


Steady-State Thermoreflectance (SSTR): How it works

Measurement Concept: Repeat process with multiple powers to improve accuracy



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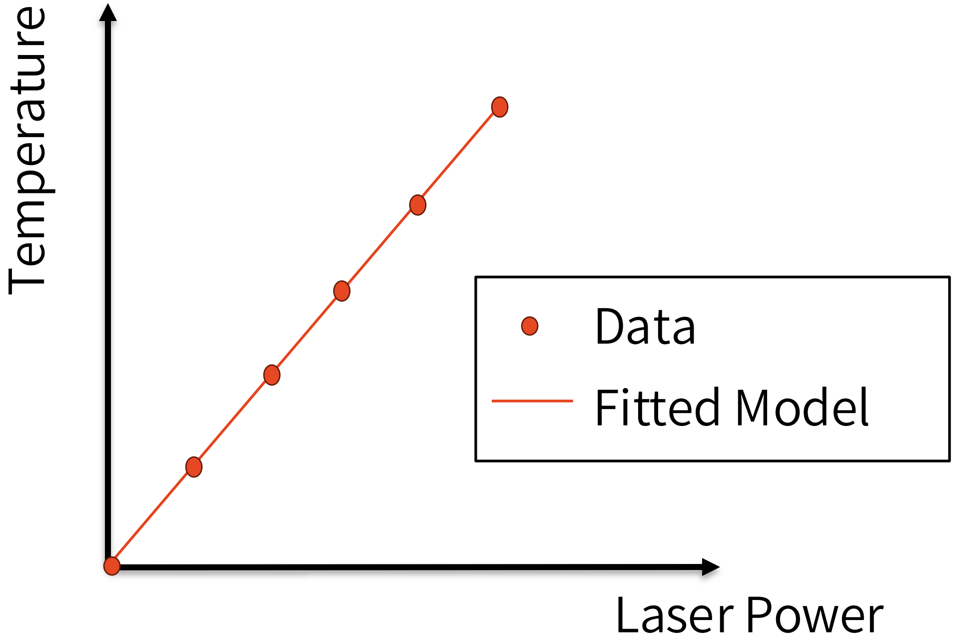
Fourier's Law

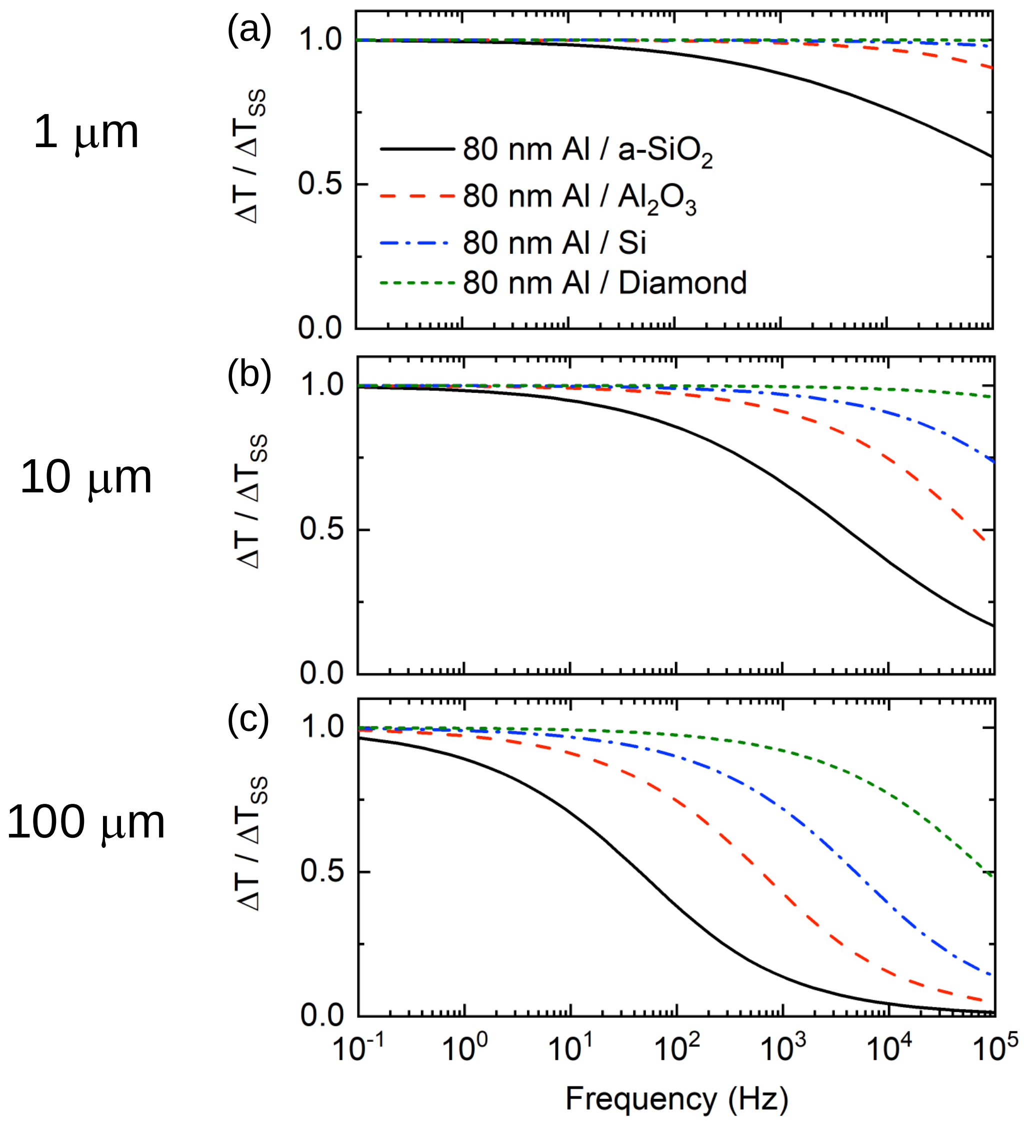
$$q'' = -\kappa \nabla T$$



Increasing laser power leads to increasing temperature rise
Slope of this relation gives us thermal conductivity

Steady-State Thermoreflectance (SSTR): Lock-in Amplifier

<u>Laser Diameter</u>



Goal: **Heat at a low enough frequency to induce a steady-state temperature rise**

$$Q \propto e^{i\omega t} \Rightarrow T \propto e^{i\omega t}$$

For some conditions, T is not in a steady-state

→ Must include for ω in the thermal model

Even if sample is in steady-state, it is best practice to include ω in thermal model

Understanding the Data Analysis

- Temperature rise is proportional to photodetector voltage

$$\boldsymbol{\Delta T \propto \frac{\Delta R}{R} \propto \frac{\Delta V}{V}}$$

  - Linear relation with small temperature changes
- Heat flux proportional to pump power

$$\boldsymbol{|\Delta q''| \propto \Delta P}$$

- Therefore, we can claim

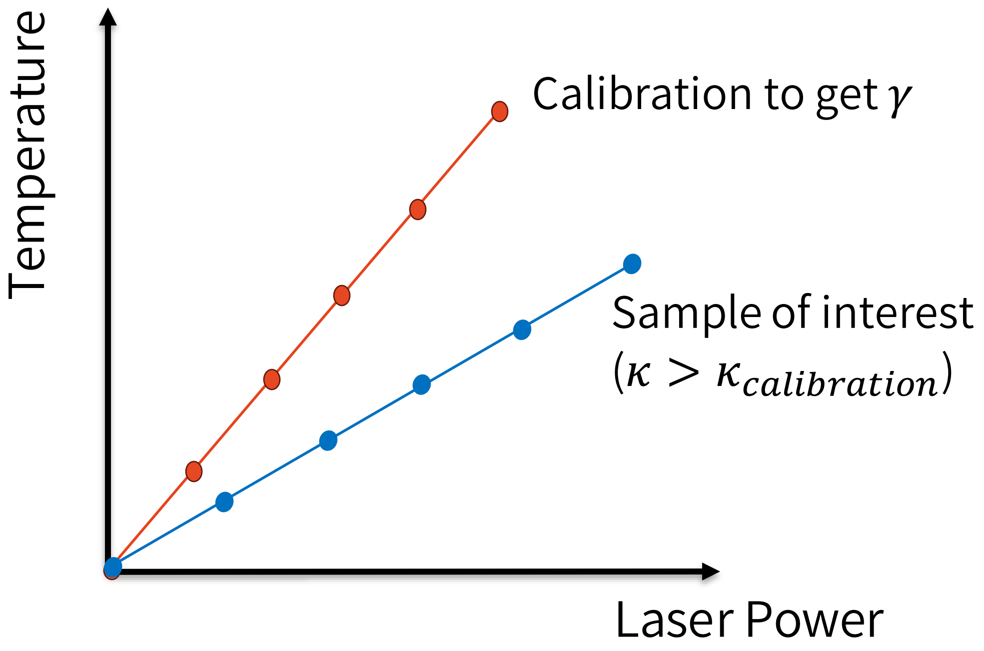
$$\boldsymbol{\left(\frac{\Delta V}{V \Delta P}\right) = \gamma \left(\frac{\Delta T(\kappa)}{\Delta |q''|}\right)}$$

- Use a Calibration to get γ

$$\boldsymbol{\gamma = \left(\frac{\Delta V}{V \Delta P}\right)_{\mathrm{cal}} \left(\frac{\Delta T(\kappa_{\mathrm{cal}})}{\Delta |q''|}\right)^{-1}}$$

Fourier's Law

$$q'' = -\kappa \nabla T$$



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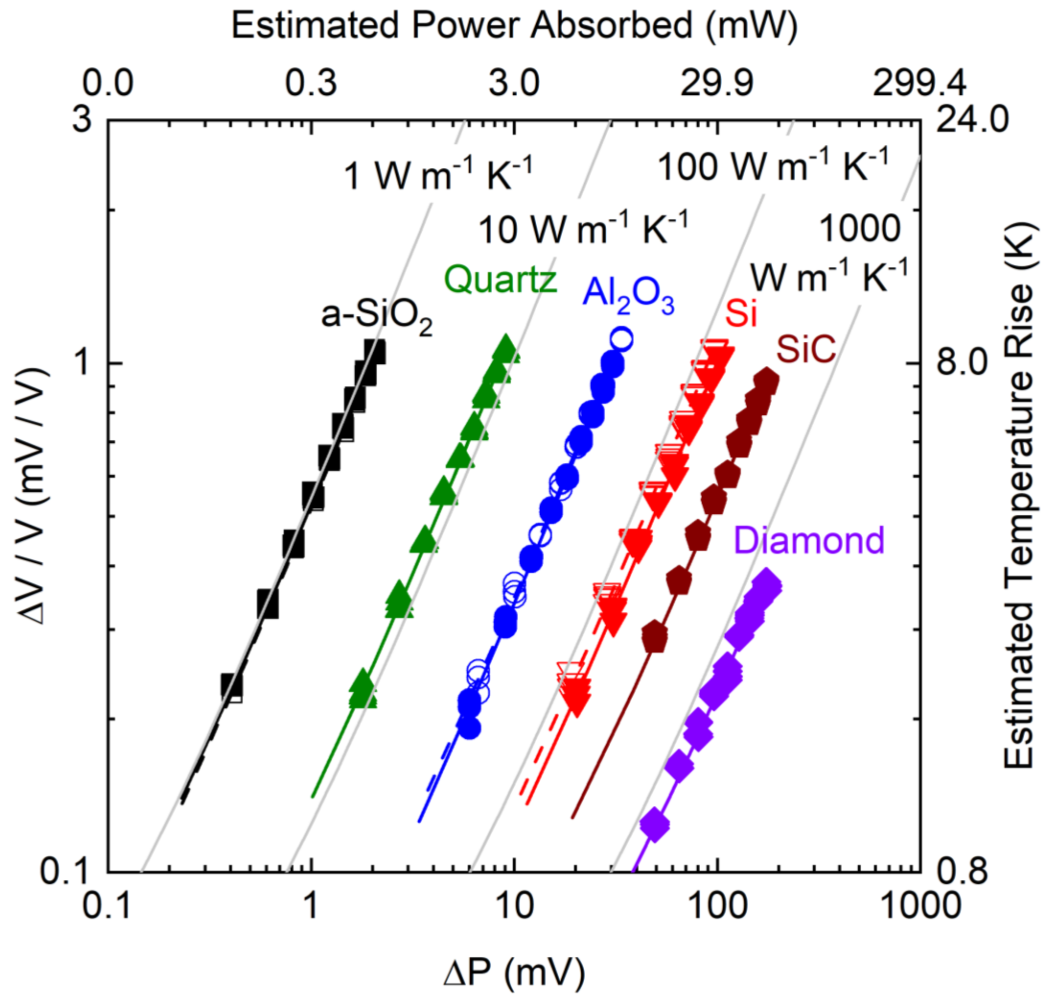
Slope of this relation gives us thermal conductivity

Steady-State Thermoreflectance (SSTR): Analysis Options

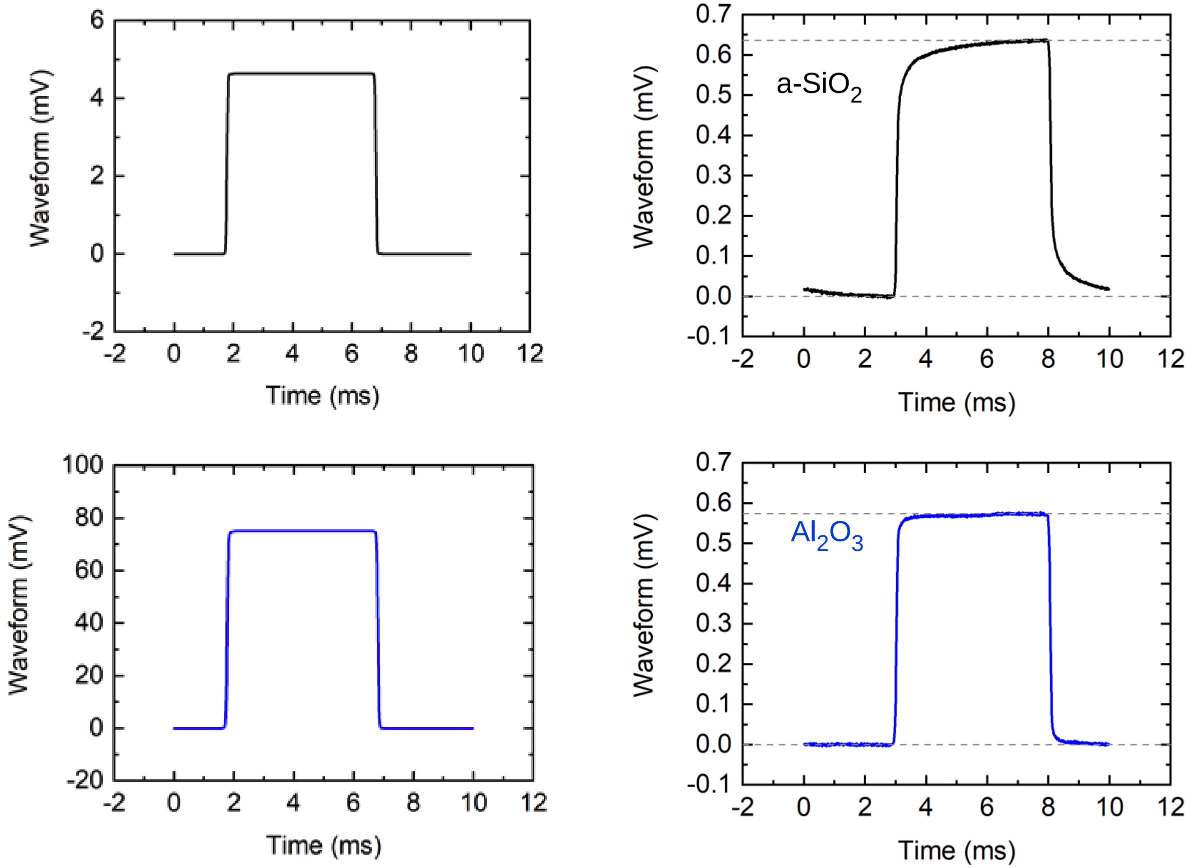
Lock-in Amplifier



Periodic Waveform Analyzer with Boxcar Averager

Pump Waveform

Temperature Response



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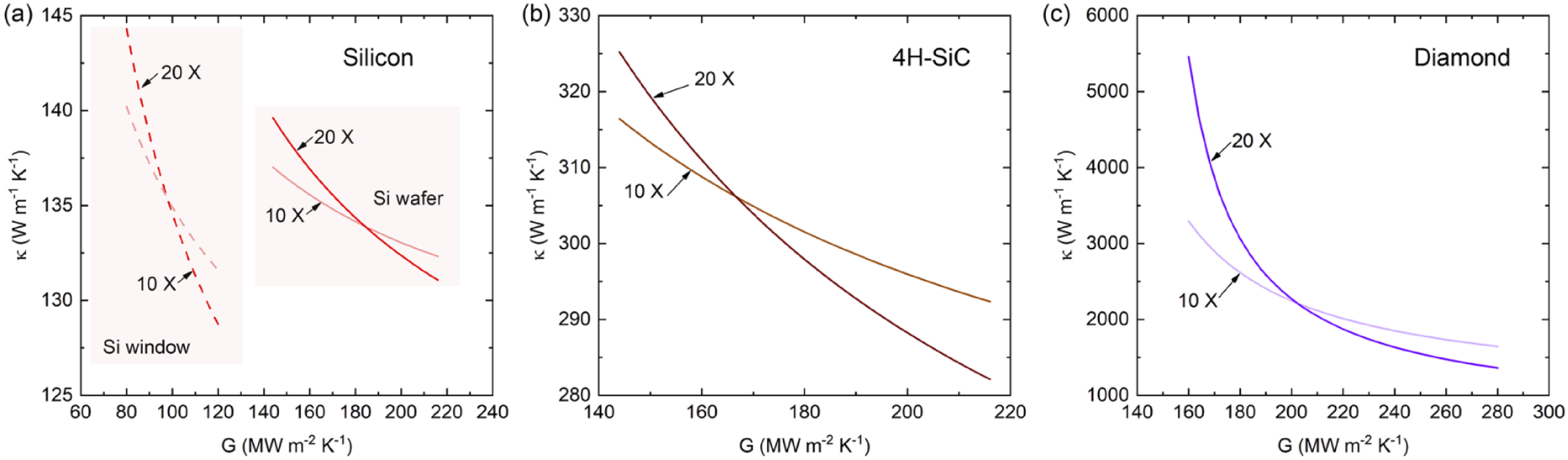
Using multiple spot sizes to determine multiple parameters

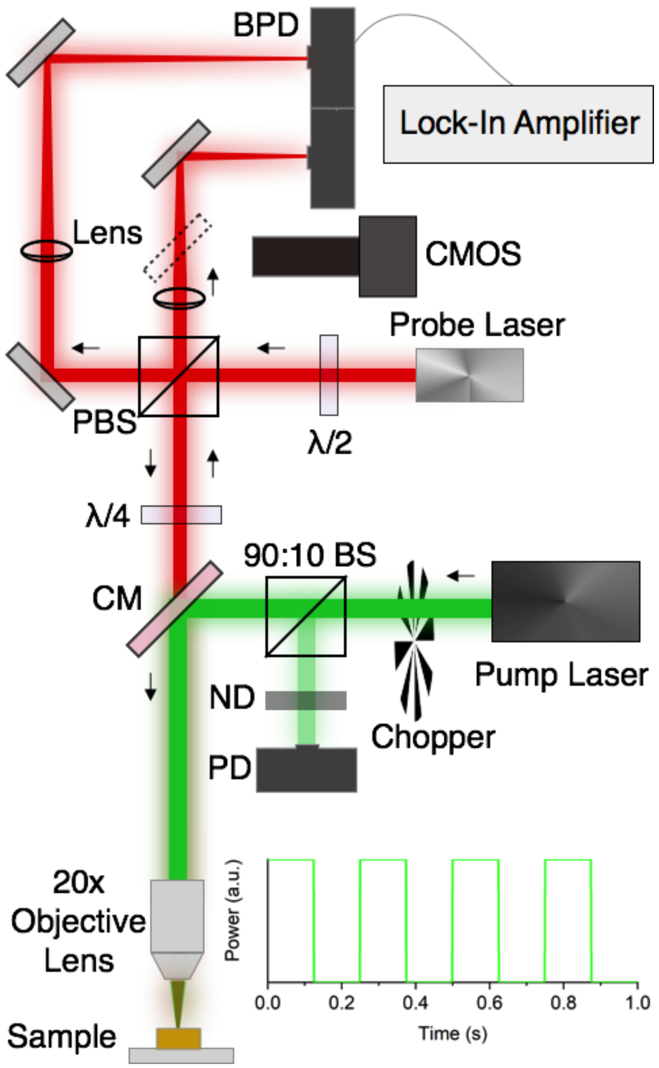
What about thermal boundary conductance (G)?

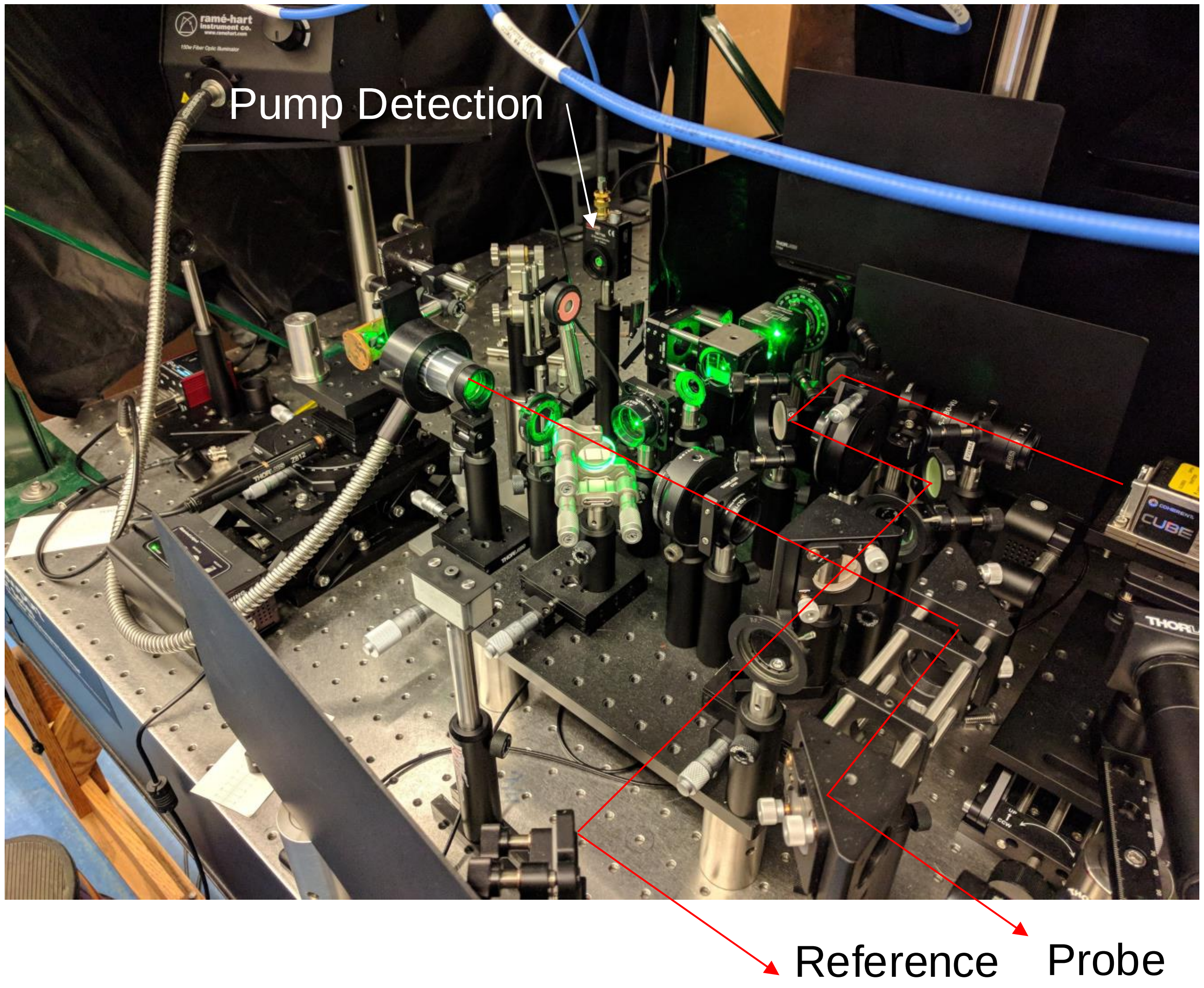
- Can be significant for high-κ materials (κ>100 W/m-K)
- Using different laser diameters allows measurement of both κ and G



How to build an SSTR free-space system

Steady-State Thermoreflectance (SSTR)





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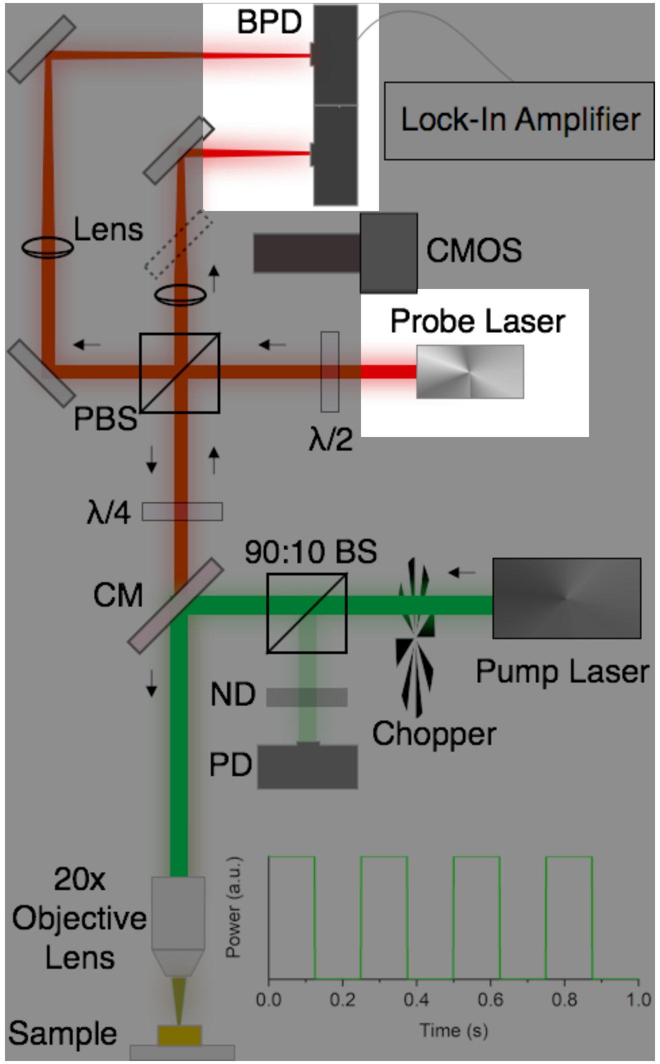

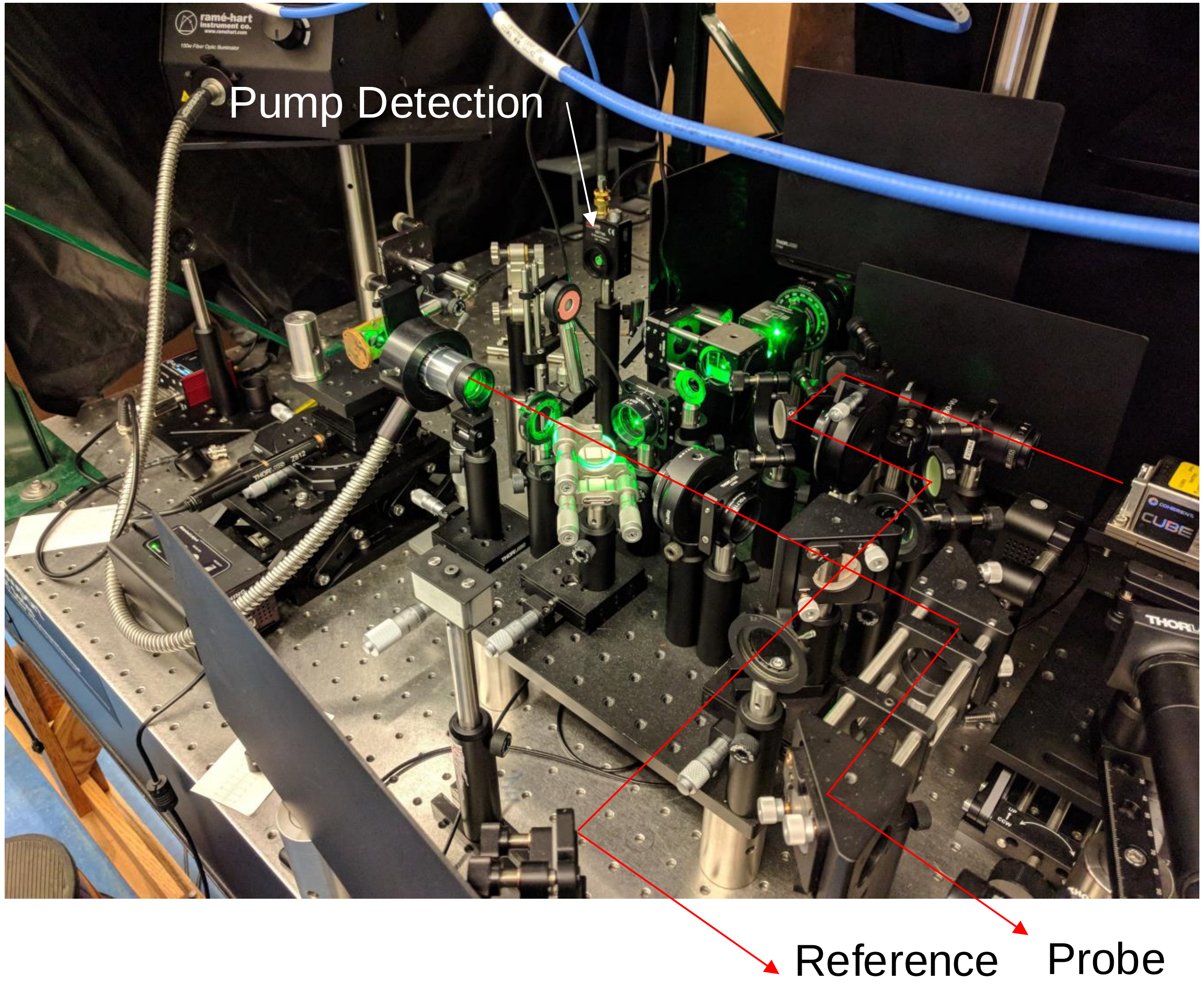

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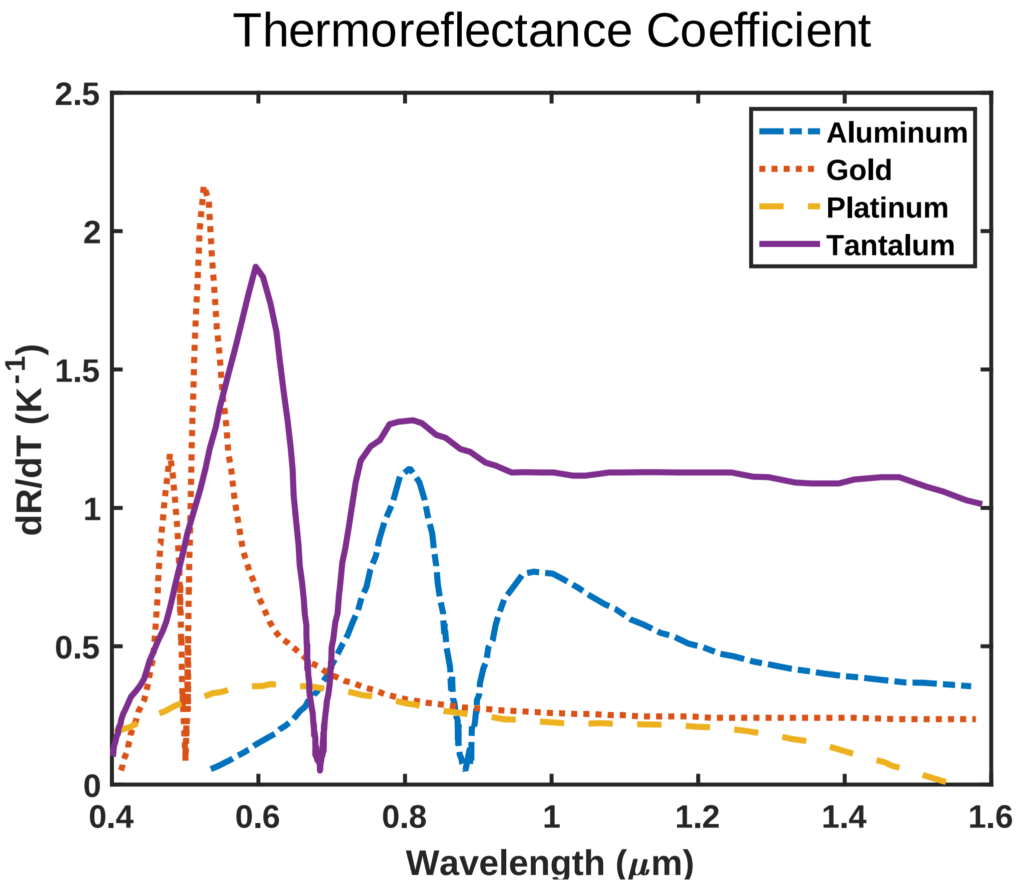
System Design: Optimizing Pump and Probe Wavelengths

Selecting Probe Wavelength: let's choose Aluminum as our transducer



Fourier's Law

$$q'' = -\kappa \nabla T$$

$$\Delta V_{Probe} \propto \beta(\lambda) D(\lambda) \Delta T$$

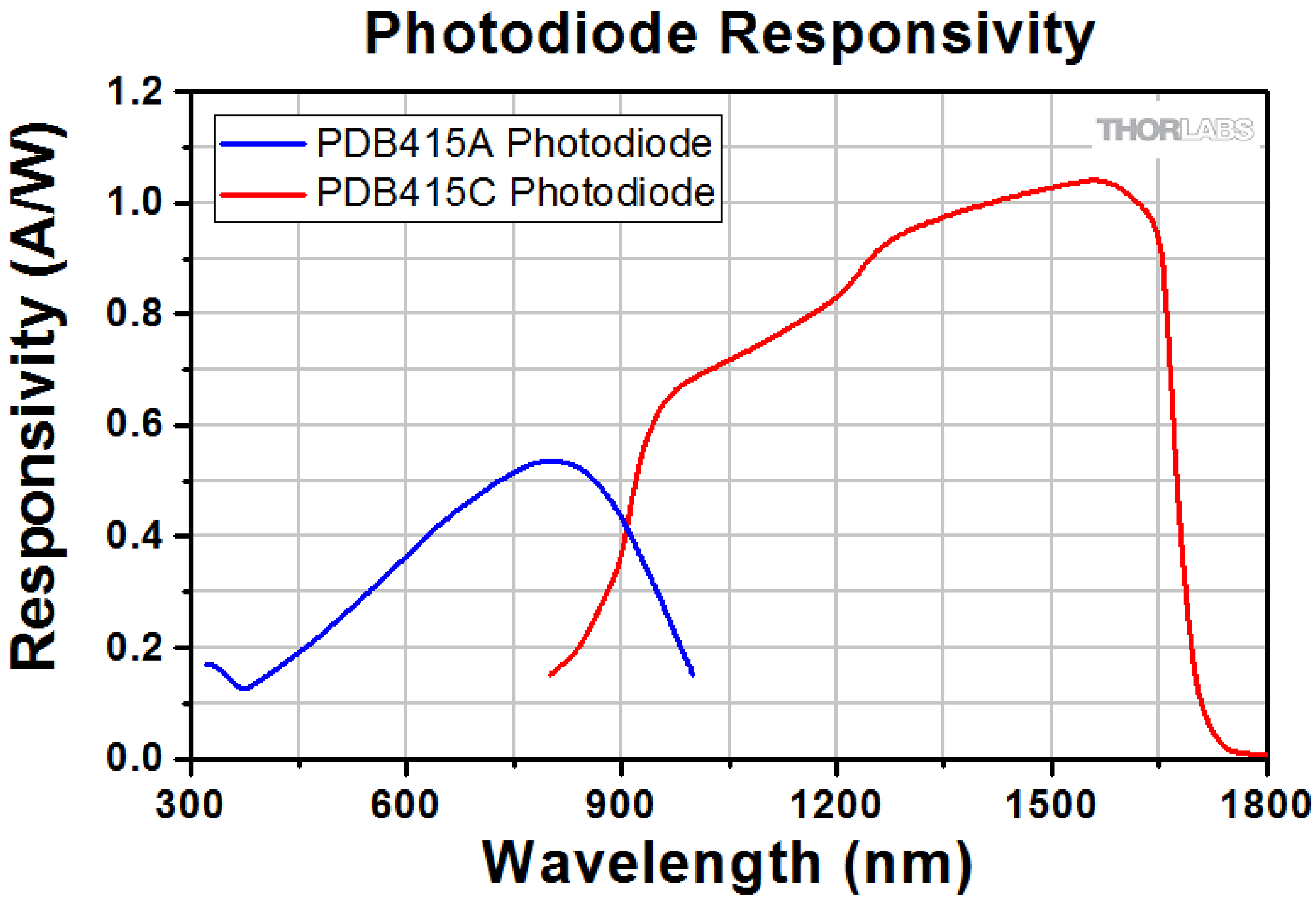
$\beta(\lambda)$ = Thermoreflectance Coefficient

$D(\lambda)$ = Detector Responsivity

Wilson, R. B., et al. *Optics express* 20.27 (2012): 28829-28838.

System Design: Probe Wavelength Selection

Photodetector Responsivity

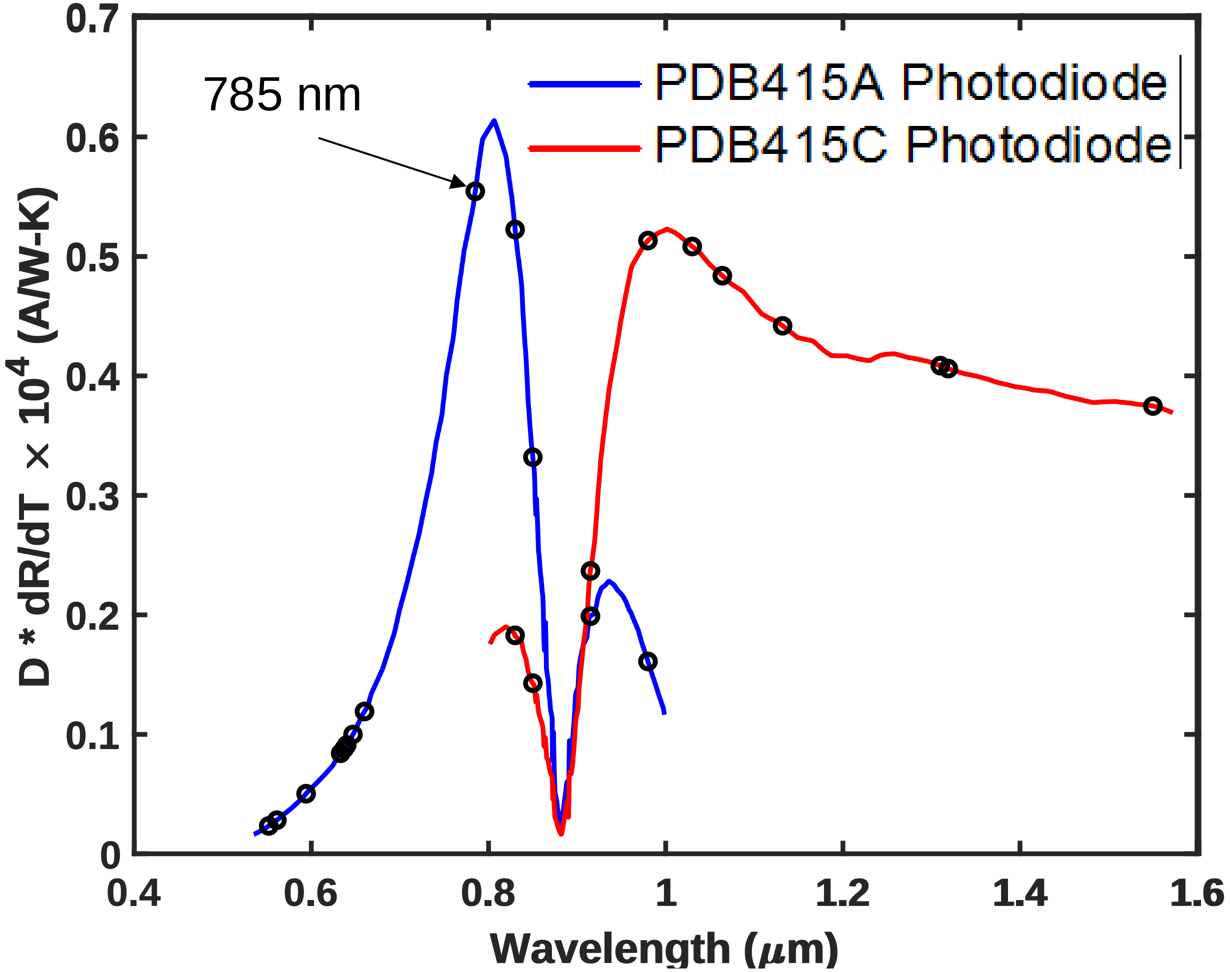


https://www.thorlabs.com

Item #	PDB415A	PDB415C
Detector Type	Si/PIN	InGaAs/PIN
Wavelength Range	320 - 1000 nm	800 - 1700 nm
Typical Max Responsivity	0.53 A/W	1.0 A/W
Active Detector Diameter	0.8 mm	0.3 mm
Bandwidth (3 dB)	DC - 100 MHz	
Common Mode Rejection Ratio	>25 dB	
Transimpedance Gain[a]	50 x 10^3 V/A	
Minimum NEP[b]	12.03 pW/$Hz^{1/2}$ (DC - 100 MHz)	6.99 pW/$Hz^{1/2}$ (DC - 100 MHz)
RF Output Conversion Gain[a,c]	26.5 x 10^3 V/W	50 x 10^3 V/W
CW Saturation Power	135 μW @ 820 nm	72 μW @ 1550 nm

System Design: Probe Wavelength Selection

Probe Figure of Merit as a function of probe laser wavelength



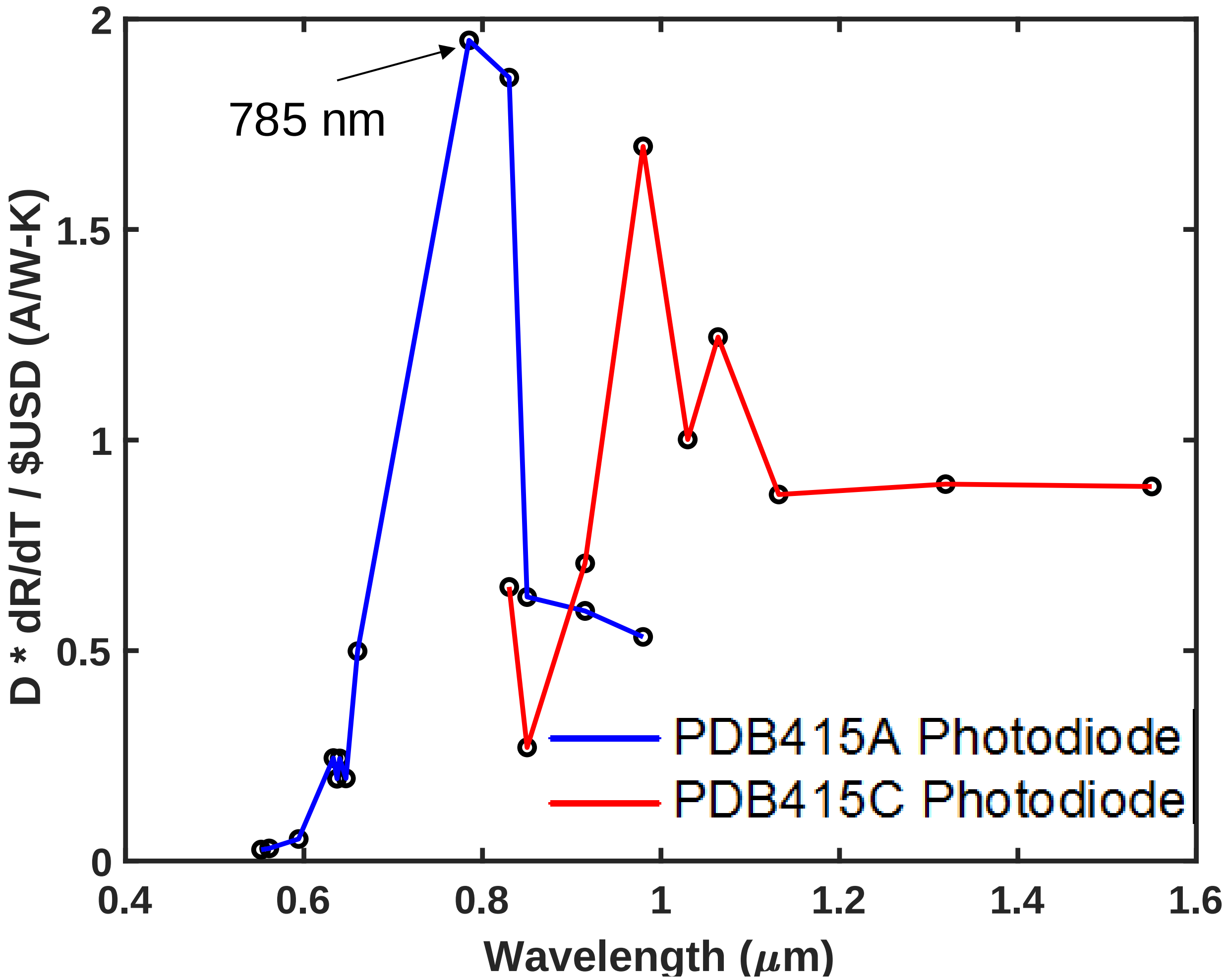
Commonly Available Laser Wavelengths

OBIS	Matchbox
375	830
405	850
445	915
458	980
473	1030
488	1064
505	1123
514	1319
520	1550
532	
552	
561	
594	
633	
637	
640	
647	
660	
785	

System Design: Probe Wavelength Selection

What about cost?

Conclusion: 785 nm is best option for probe



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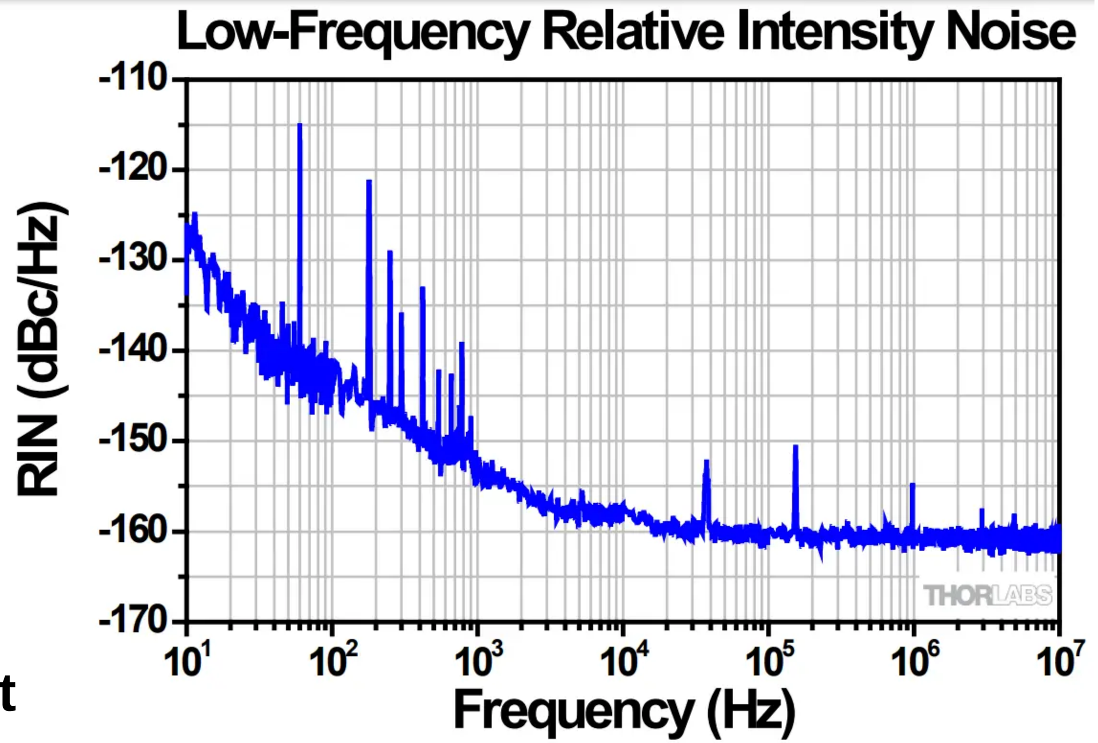
Some final notes on probe noise and detection

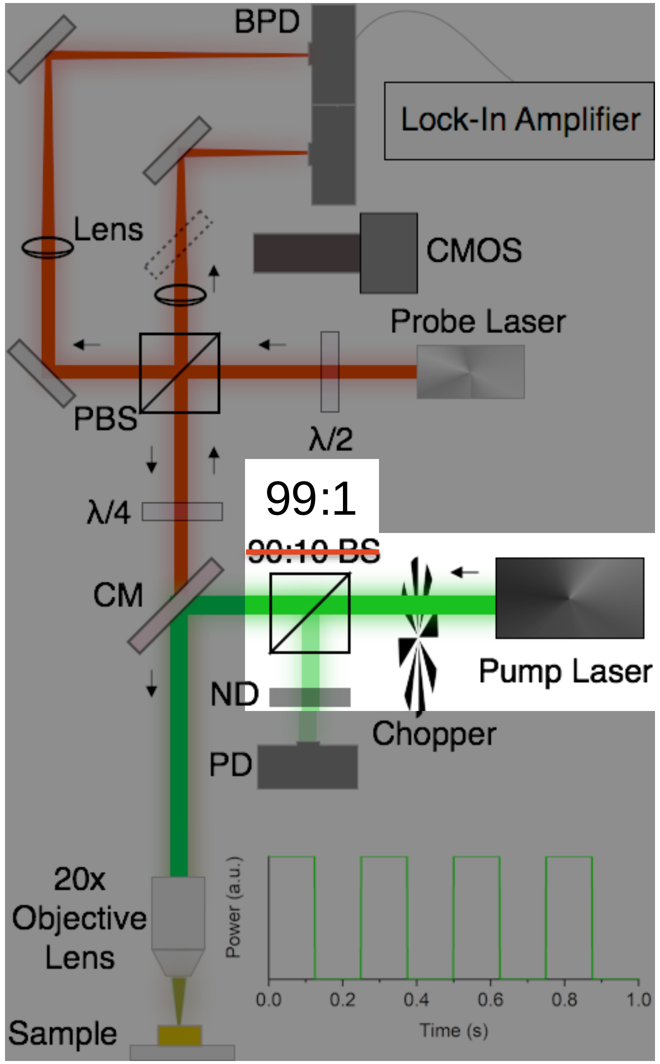
- Balanced photodetector is best performing for low frequency detection
- Path matching probe signal and reference arm does not significantly affect performance

- Probe laser has a noise spectrum
- Detector + lock-in amplifier has a noise spectrum

Adjust probe power to optimize signal-to-noise at your operating frequency



https://device.report/manual/6145843

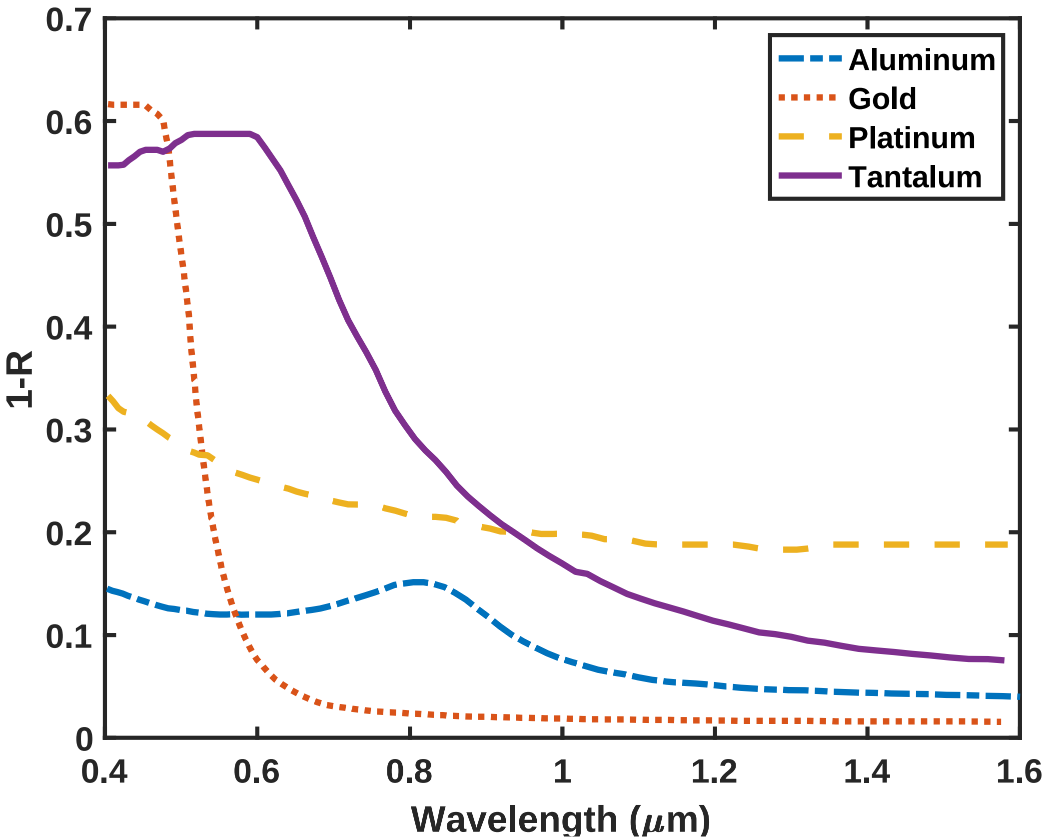


Pump selection comes down to how much *absorbed power* is needed in your sample.

This can be determined based on the the **maximum κ** to be measured

System Design: Optimizing Pump and Probe Wavelengths

Selecting Pump Wavelength



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Fourier's Law

$$q'' = -\kappa \nabla T$$

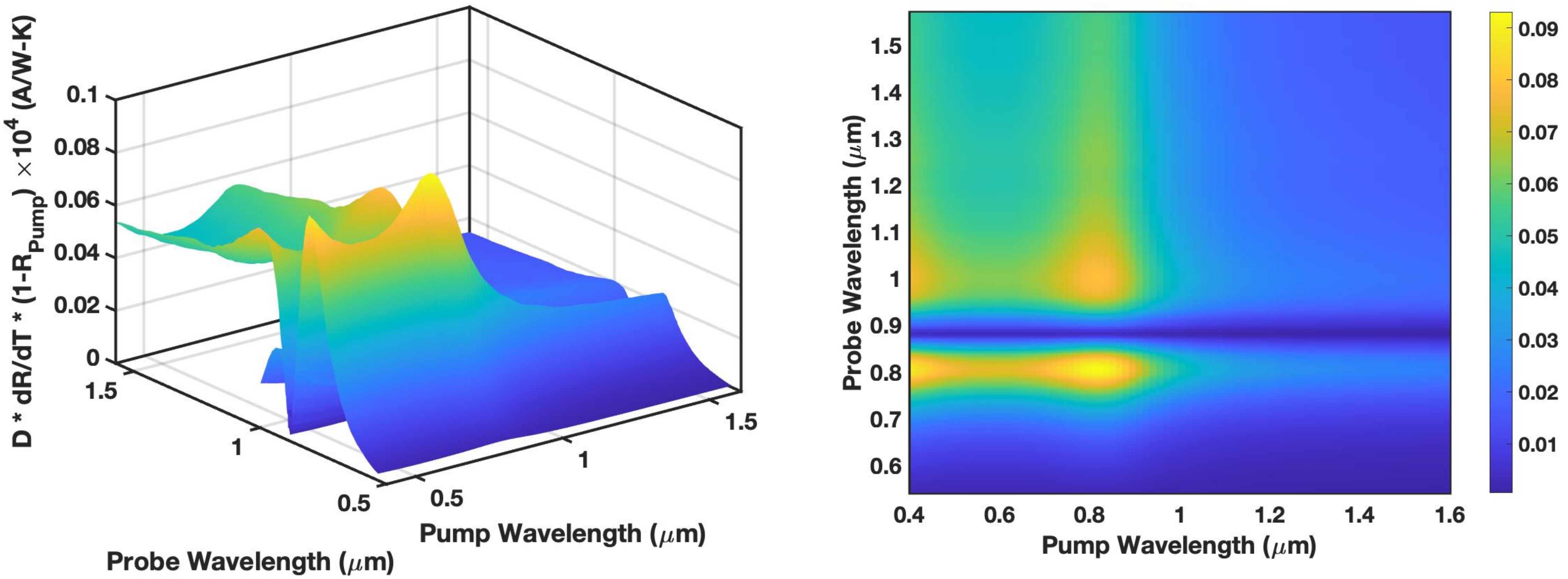
$$\Delta V_{Pump} \propto (1 - R(\lambda))$$

$R(\lambda)$ = Reflectance

System Design: Pump and Probe Wavelength Selection

Combined Figure of Merit for selecting pump and probe wavelength

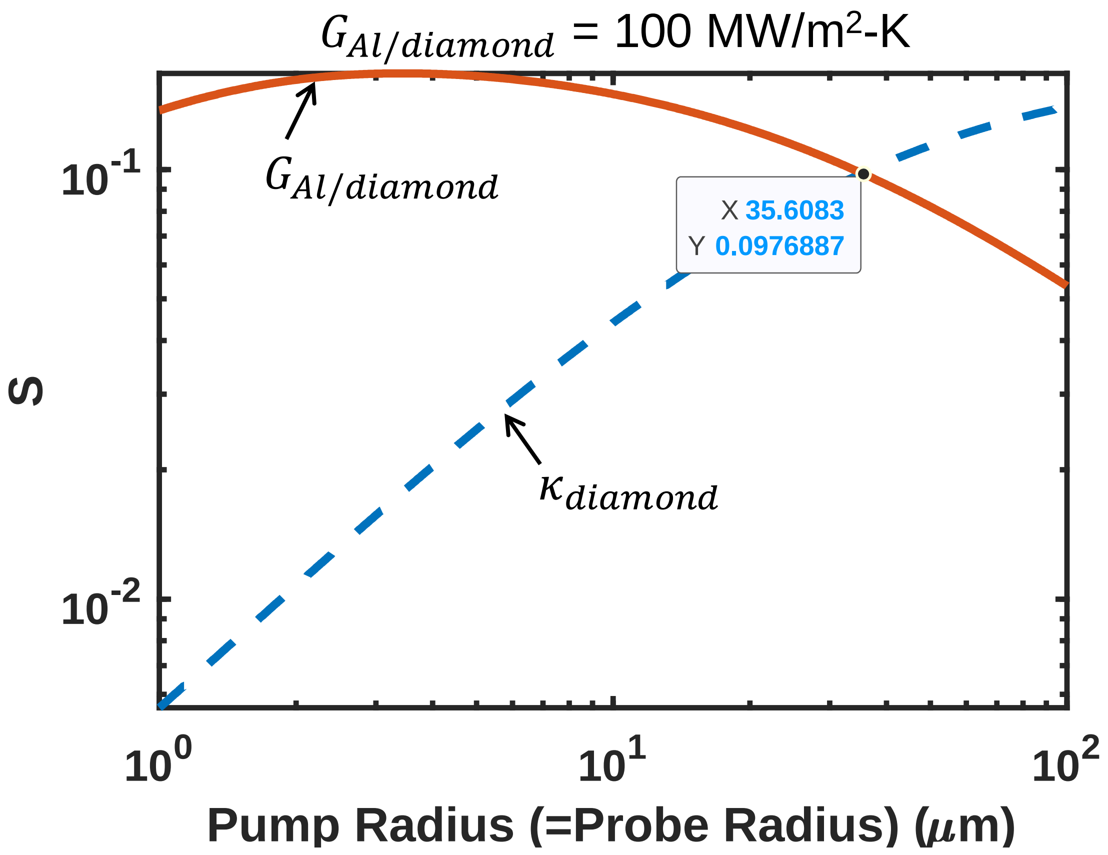
$$\Delta V_{Probe} \propto \beta(\lambda_{probe}) D(\lambda_{probe}) \Delta T \propto \beta(\lambda_{probe}) D(\lambda_{probe}) (1 - R(\lambda_{pump}))$$



Conclusion: ~800 nm wavelength is ideal for both pump and probe

System Design: Determining Pump Power Needed

Consider the hardest case to measure: low G, κ = 3000 W/m-K



Measurement Sensitivity

$$S_x = \frac{|T_{1.1x} - T_{0.9x}|}{T_x}$$

Layer 1: Aluminum

- κ = 180 W/m-K
- C_v = 2.42 J/cm³-K
- Thickness = 80 nm

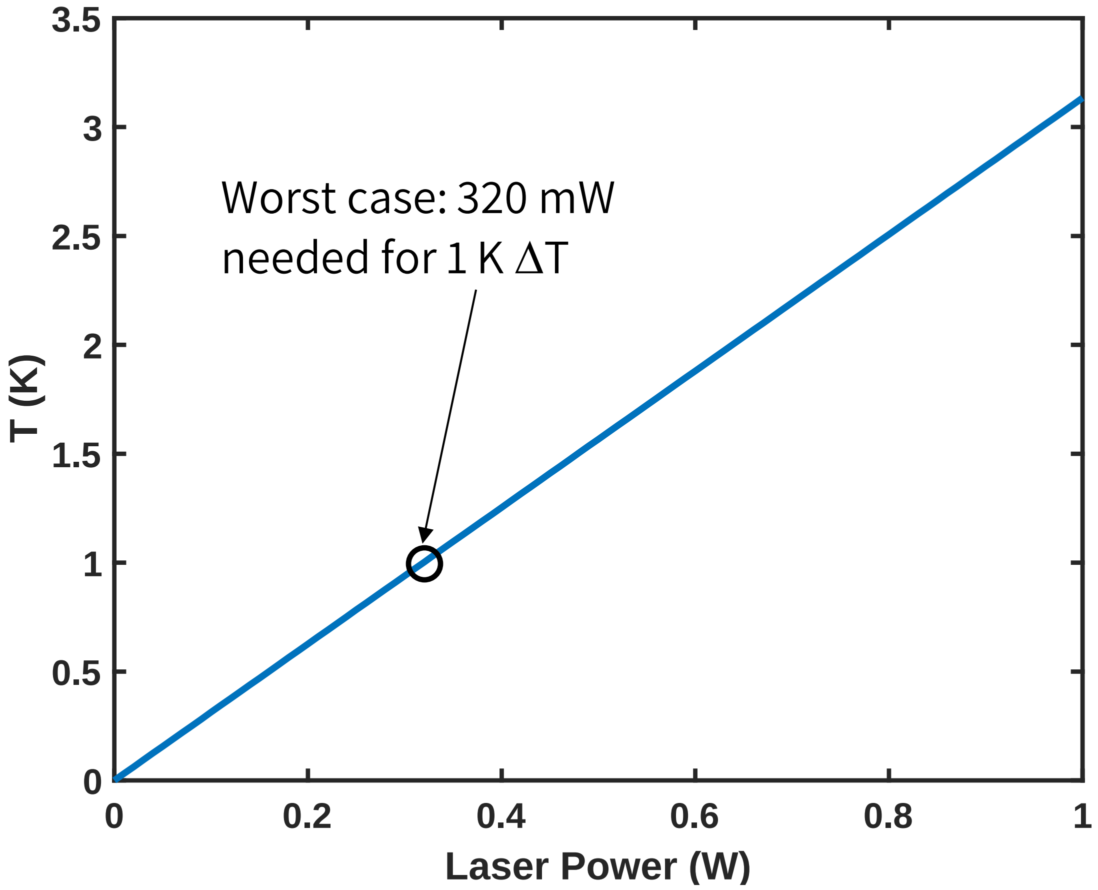
Layer 2: Diamond

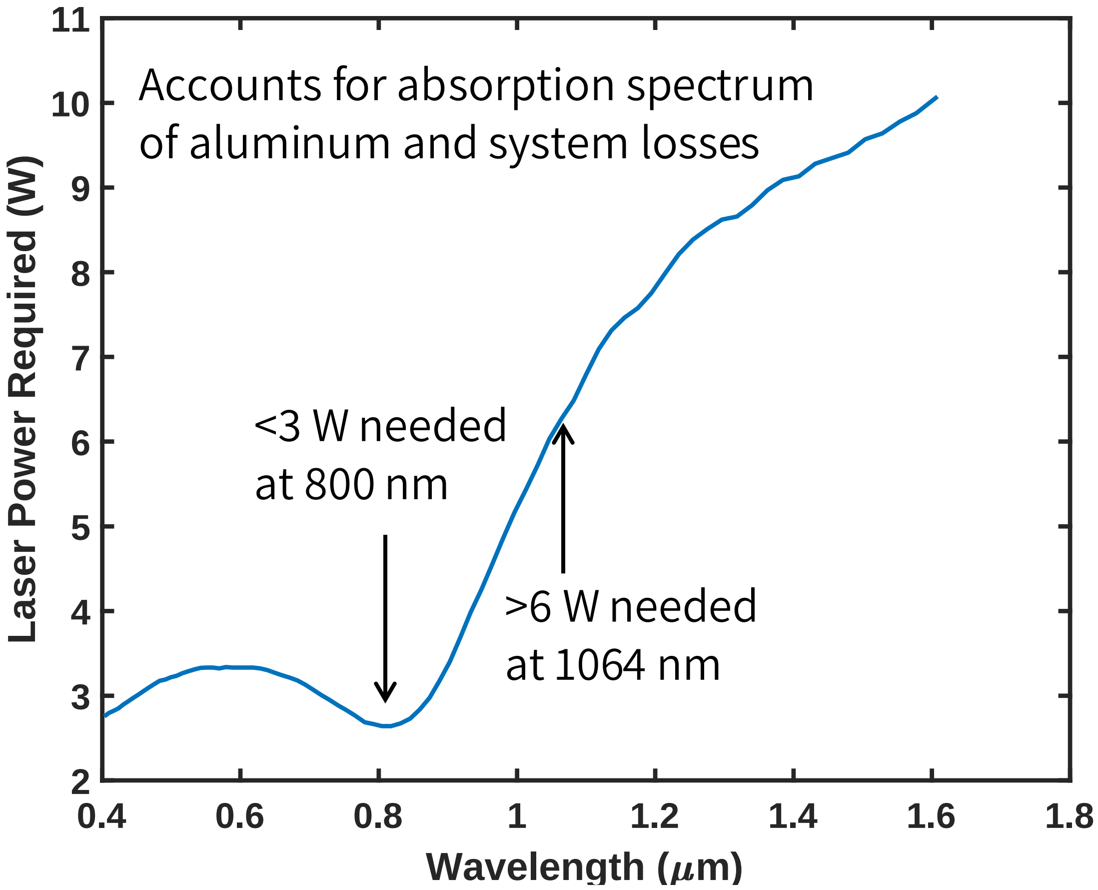
- κ = 3000 W/m-K
- C_v = 1.78 J/cm³-K

Worst case: ~35 μm radius → assume 50 μm to be conservative

System Design: Determining Pump Power Needed

Temperature rise as a function of absorbed pump laser power…



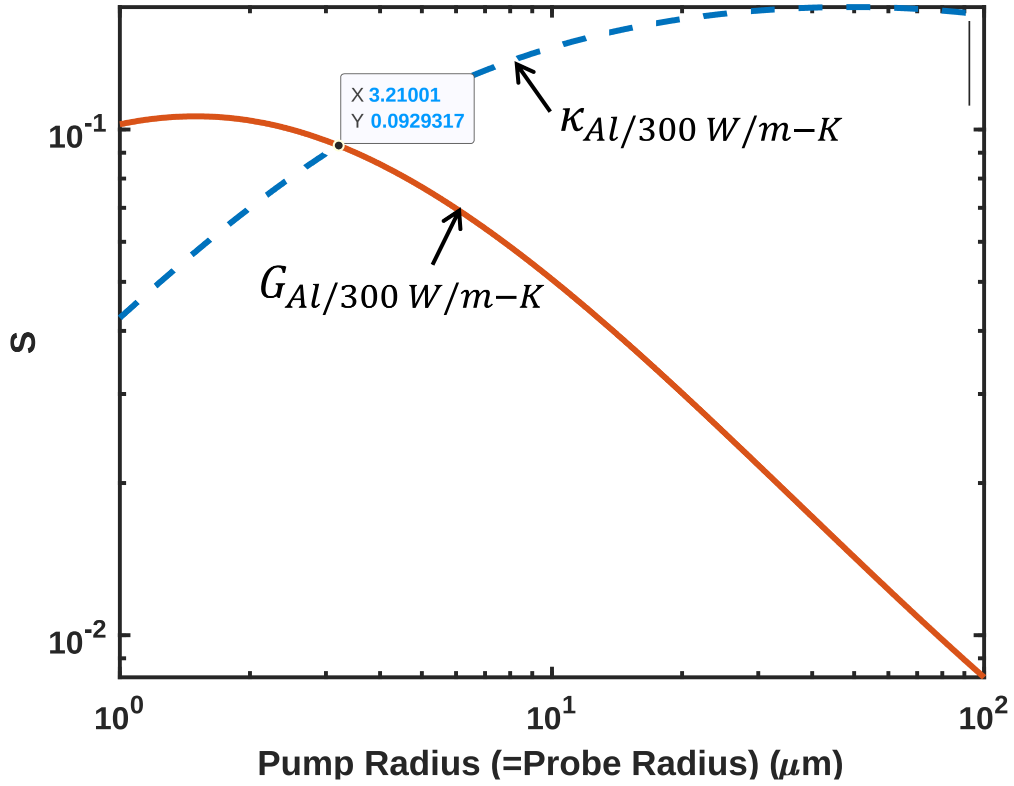


…translates to laser power needed for pump specification

System Design: Determining Pump Power Needed

What if we only need a system capable of measuring up to κ = 300 W/m-K?

$G_{Al/300\ W/m-K}$ = 100 MW/m²-K



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Layer 2: Diamond
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Sensitivity crossover ~3.2 μm radius → assume maximum of 5 μm needed

System Design: Determining Pump Power Needed

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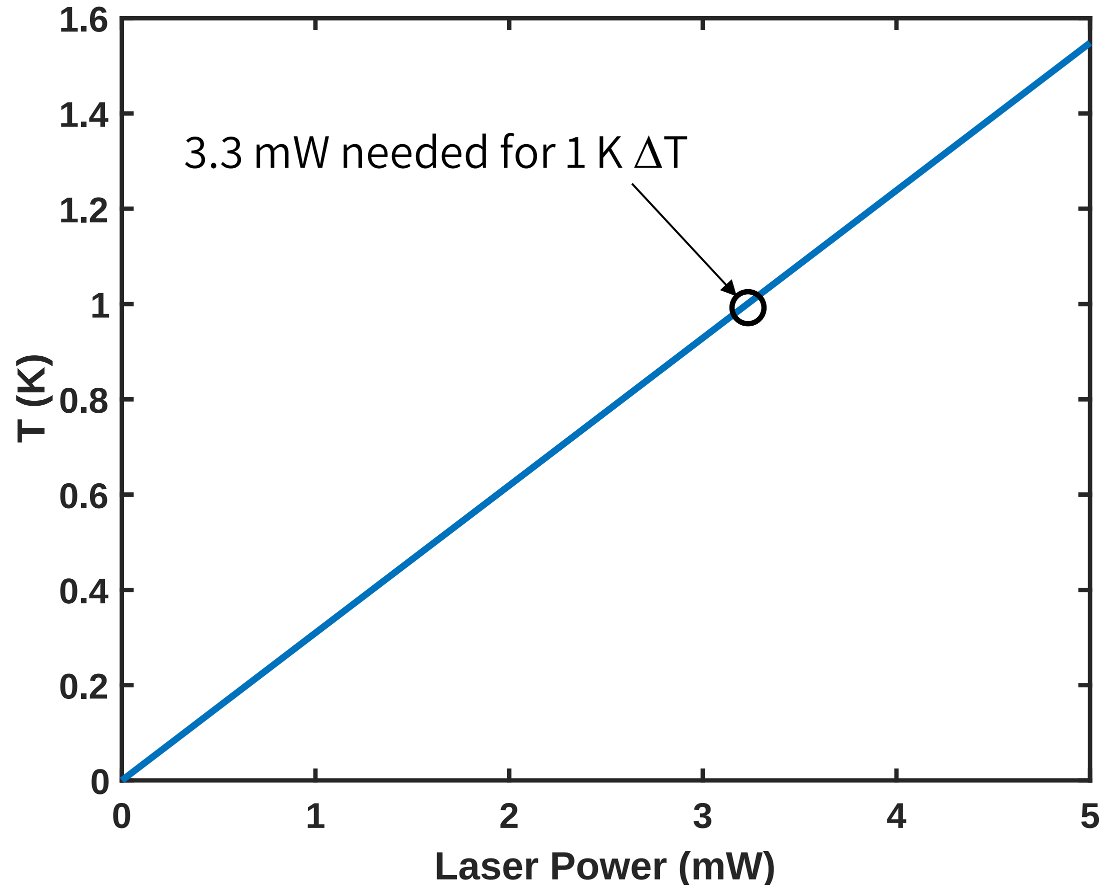
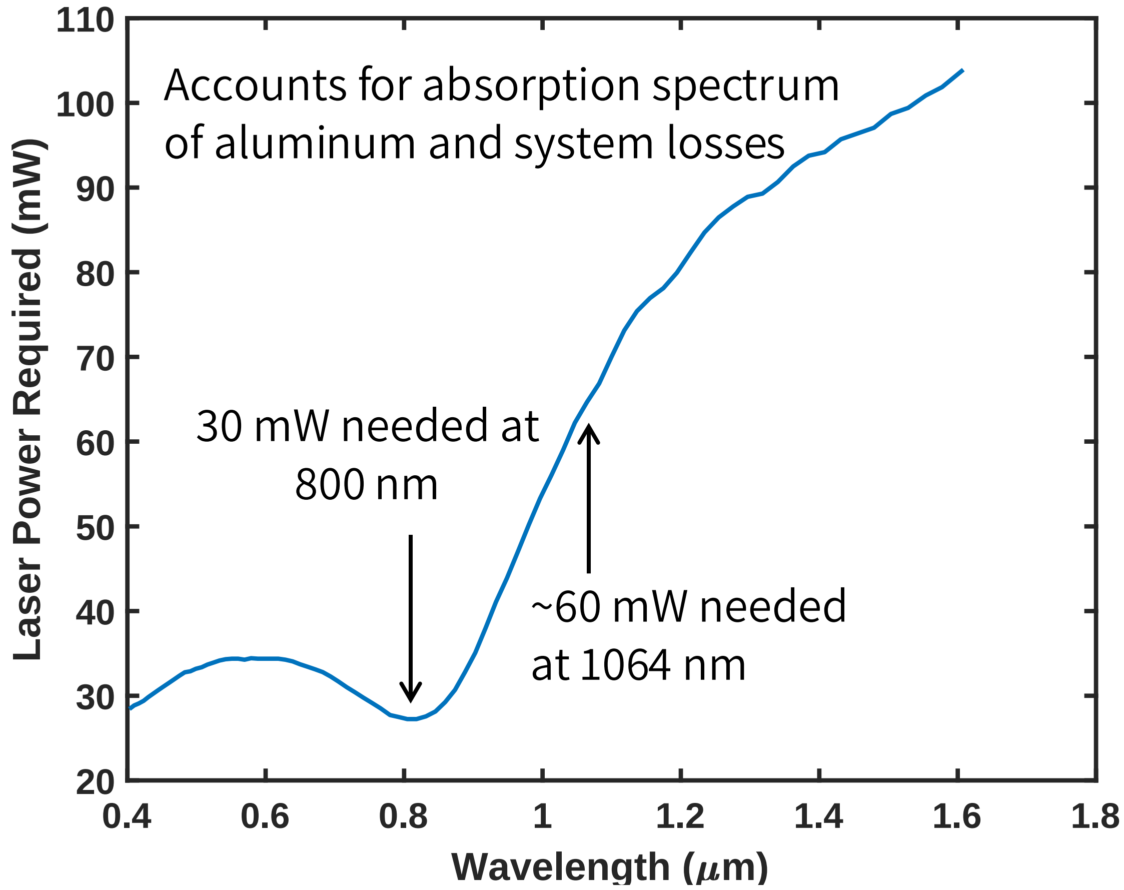
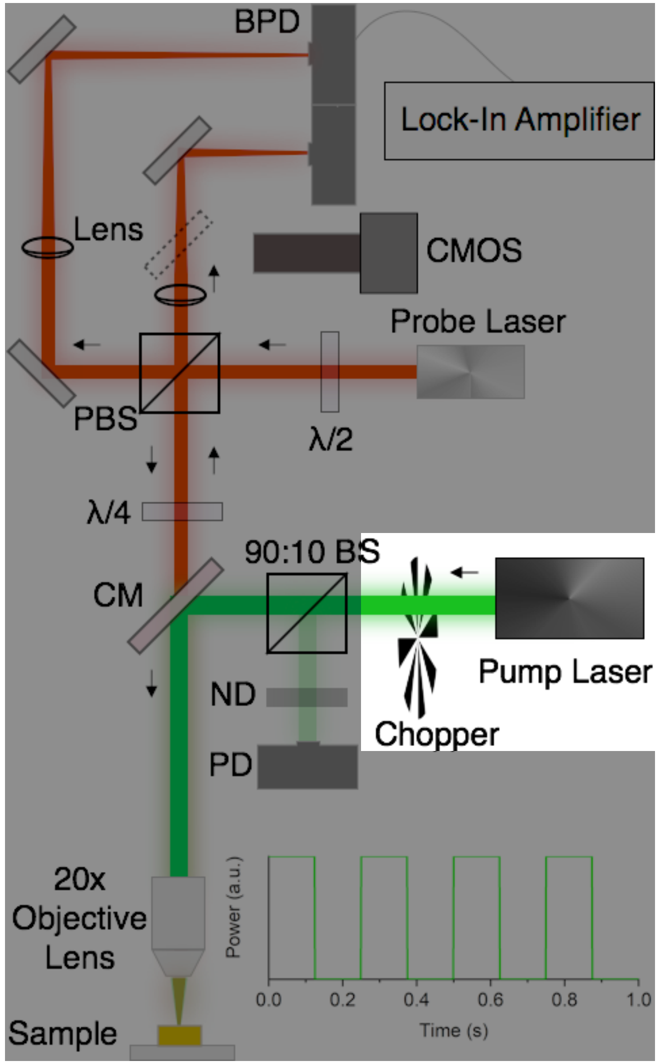




Very manageable to obtain – **most FDTR systems can be converted to SSTR mode!**

Pump Laser Modulation in SSTR

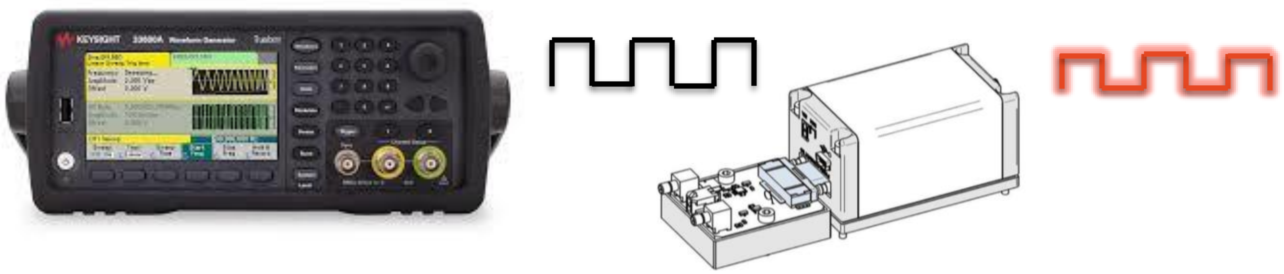


Pump Modulation Options

Mechanical Chopper

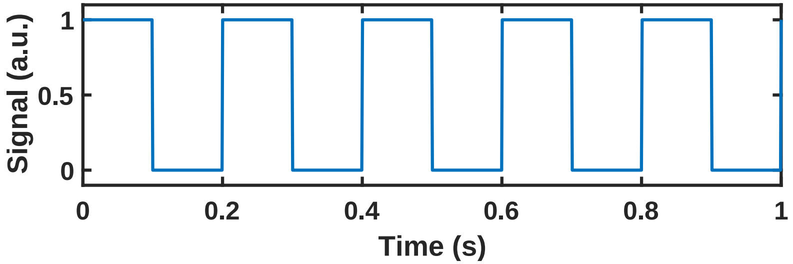
Electro-Optic or Acousto-Optic Modulator

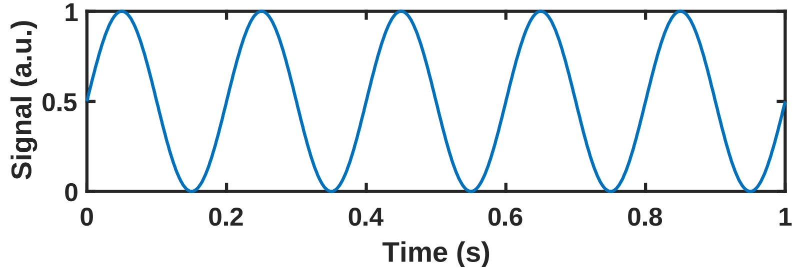
Drive Current Modulation



Pump Laser Modulation in SSTR

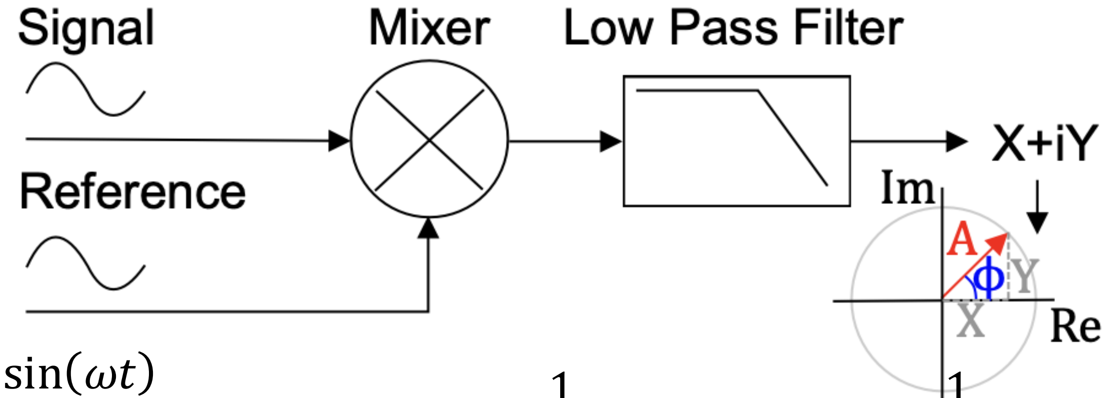
Does modulation waveform matter? **Depends on demodulator! For sine wave demodulation, no.**





$$V_{sig} = V_0 \, [\sin(\omega t + \phi) + f(t)]$$

$$V_{sig} \times V_{ref} = \frac{1}{2} \, V_0 \, V_1 [\cos(2\omega t + \phi) + \sin(\omega t) \, f(t) + \cos(\phi)]$$



$$V_{ref} = V_1 \sin(\omega t)$$

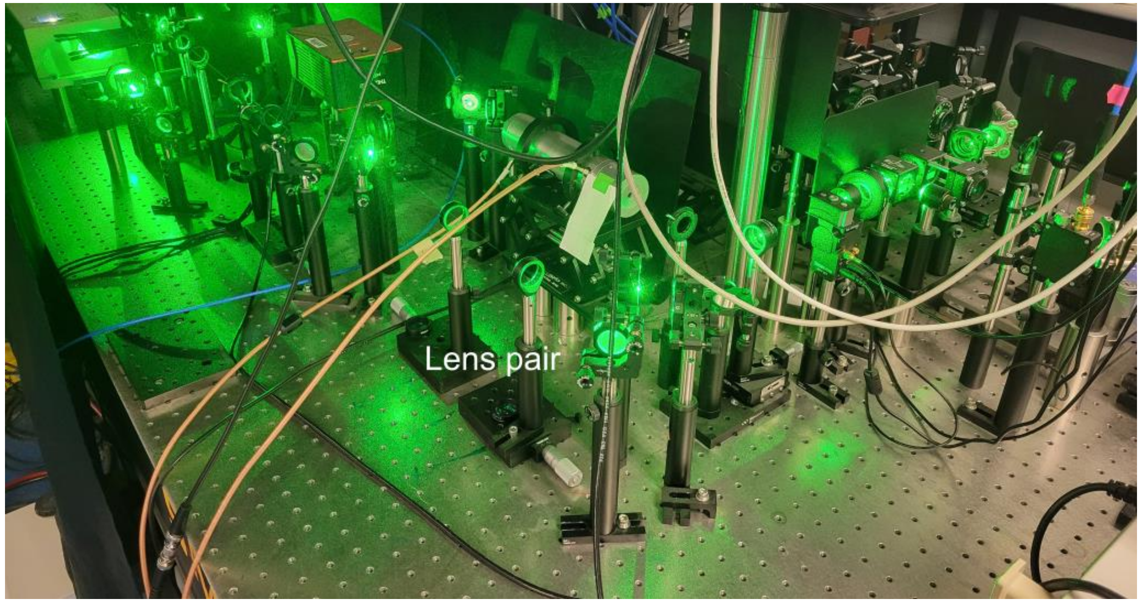
$$X = \frac{1}{2} V_0 V_1 \cos(\phi) \quad Y = \frac{1}{2} V_0 V_1 \sin(\phi)$$

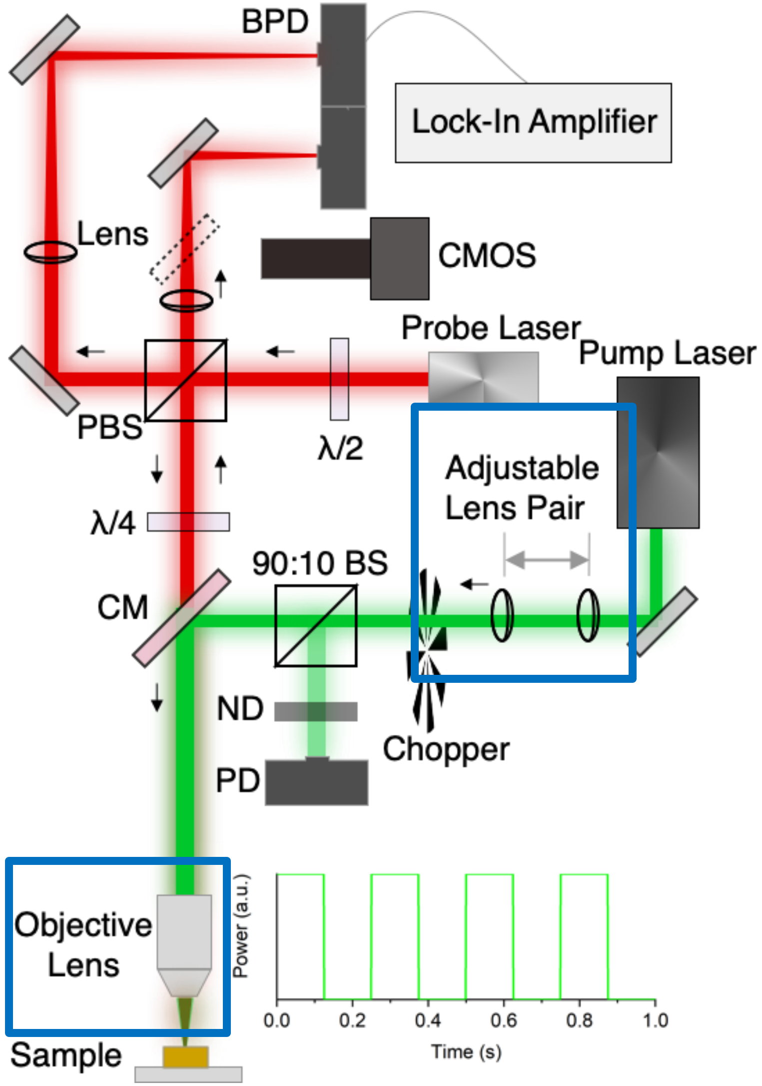
Recommendation:

- For lock-in amplification, drive and demodulate with sine wave
    - For chopper, only square wave is possible → still demodulate with sine wave

How to adjust pump laser spot size

- Adjustable lens pair allows changing spot size of pump
- Common objective lenses available for different magnification at sample
  - 2x
  - 5x
  - 10x
  - 20x
  - 50x





Some Common Questions

Does γ change with modulation frequency?

- Generally no, as long as you remain below detector bandwidth

Does γ change with temperature?

- Yes, near linear relationship for Al transducer
- No significant change for Au transducer

Does γ change with each transducer deposition?

- Generally no, but best practice is to include a calibration sample

Does γ change day-to-day?

- Generally no, but it is best practice to run calibration

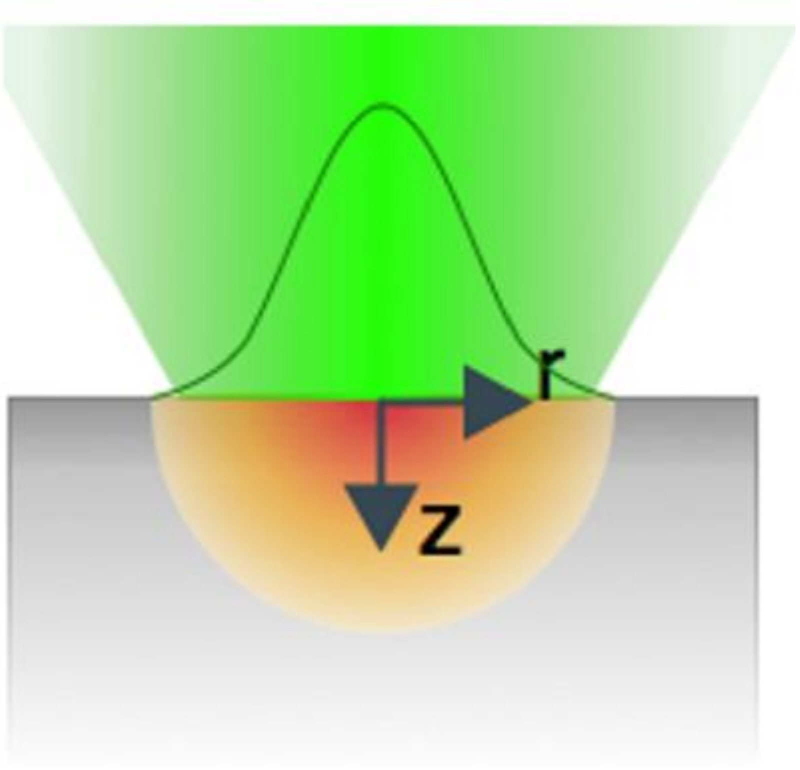
What is the recommended temperature rise for SSTR?

- ~5-10 K, but should be based on SNR
- With longer averaging times, smaller ΔT(~1 K) can work

What can you do with SSTR?

Additional capabilities of SSTR-F

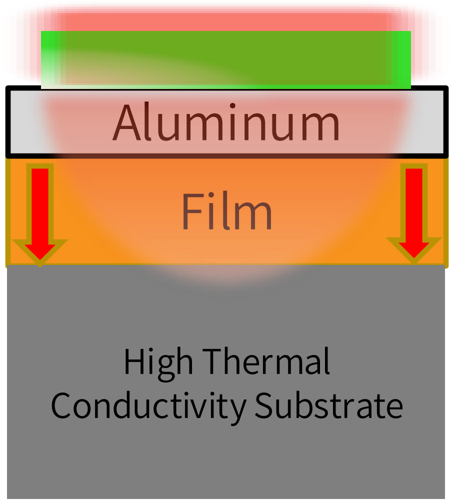
Cross-plane, In-plane and sub-surface capabilities



For bulk materials, SSTR is sensitive to

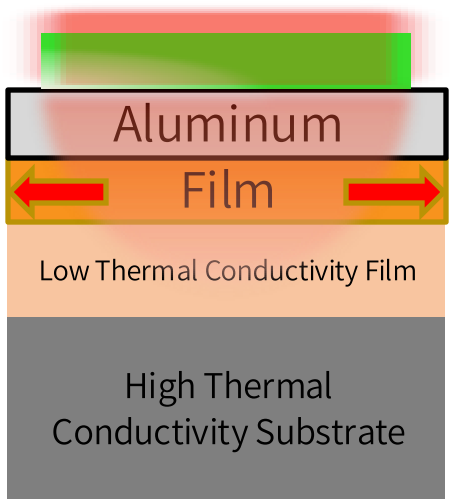
$$\kappa = \sqrt{\kappa_r \kappa_z}$$

Cross-plane Measurement



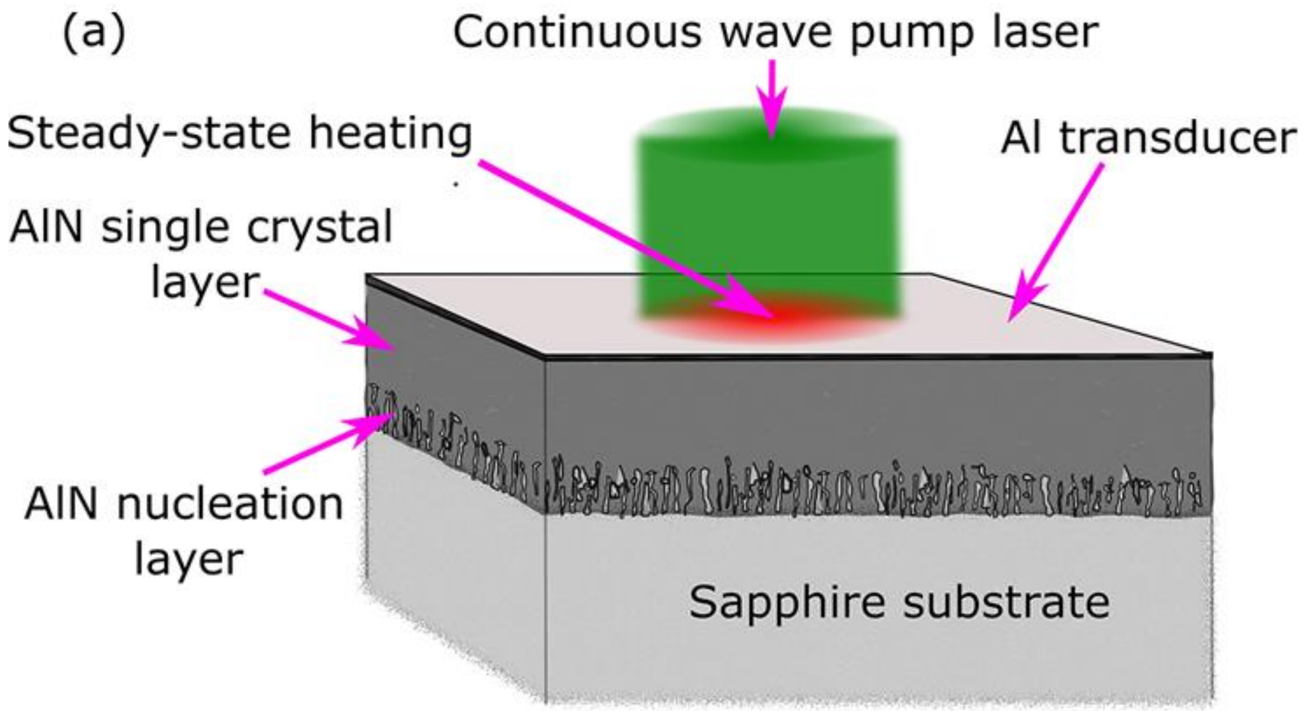
- Primary heat flow into substrate (cross-plane)
- Substrate is a heat sink
- Cross-plane case most valid for insulating films

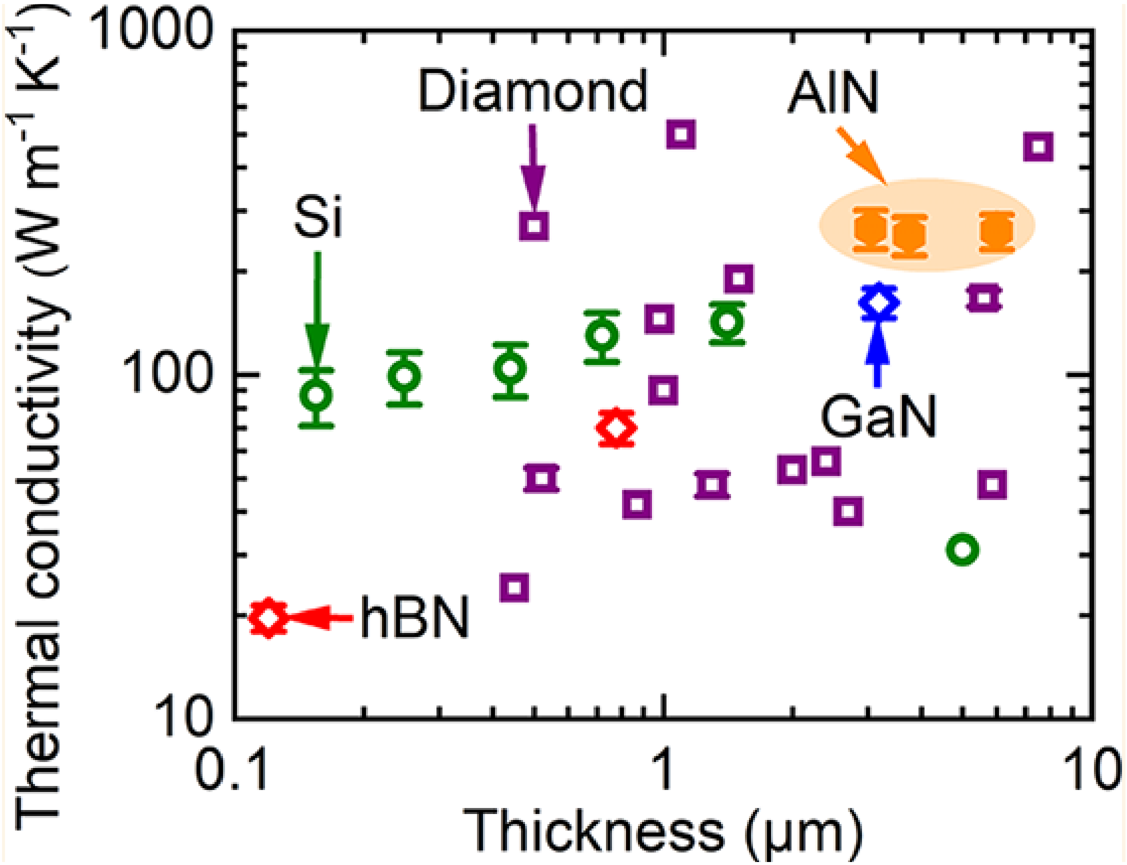
In-plane Measurement



- Primary heat flow is in-plane
- In-plane dominant for conductive films on insulating substrates/films
- Path of least resistance is in-plane

In-plane thermal conductivity of thin films
e.g., anisotropy effects in AlN thin films





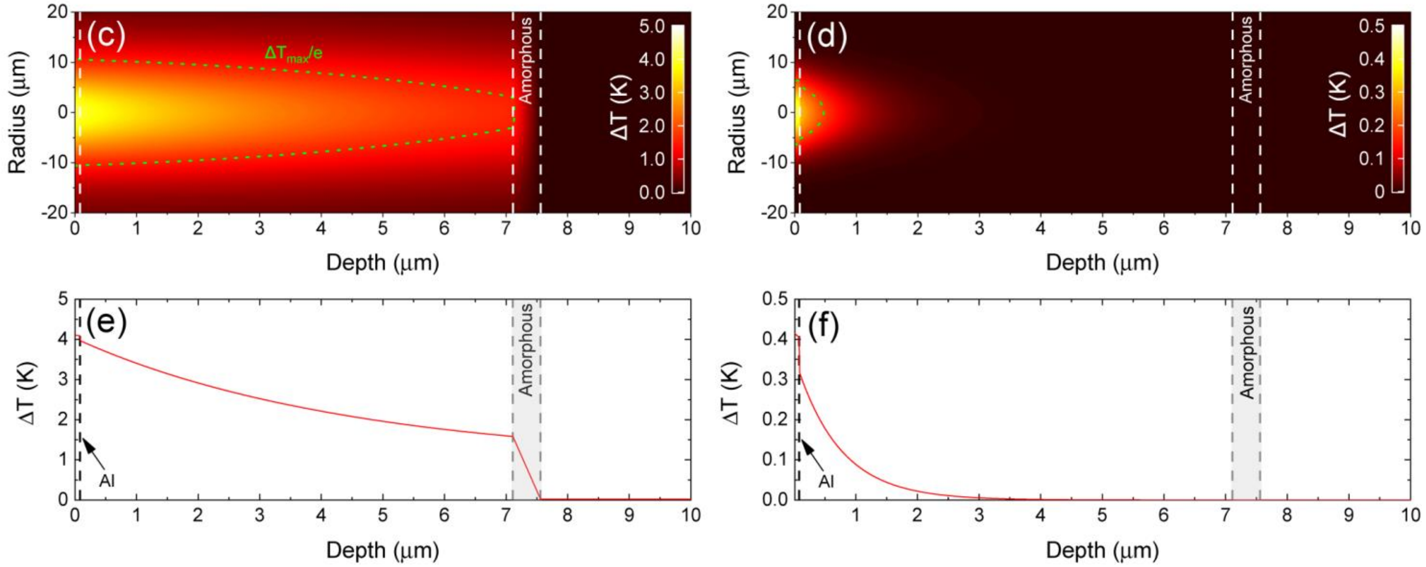
ACS Nano **15**, 9588 (2021)

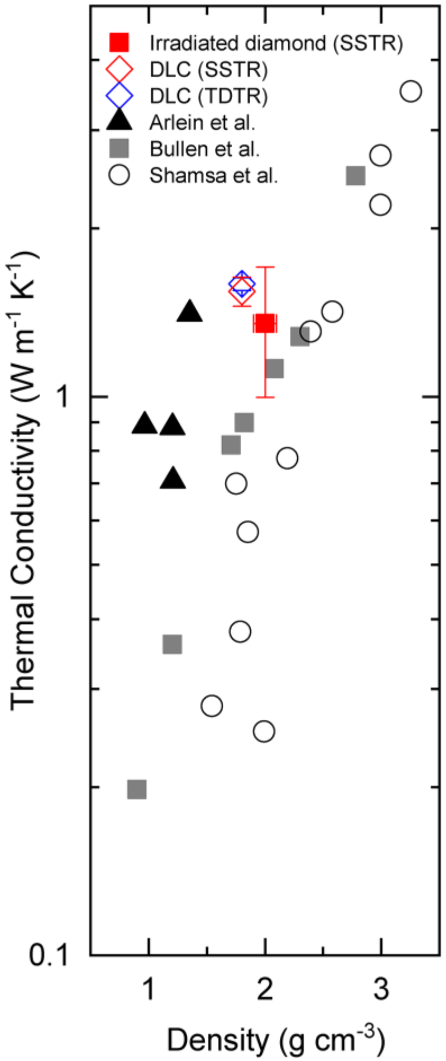
Sub-surface defect detection

e.g., measure thermal conductivity of thin region with point defects 7 μm under diamond surface

SSTR can measure at variable depths under the surface controlled by the laser spot size

TDTR/FDTR restricted to ~1 μm beneath surface in diamond





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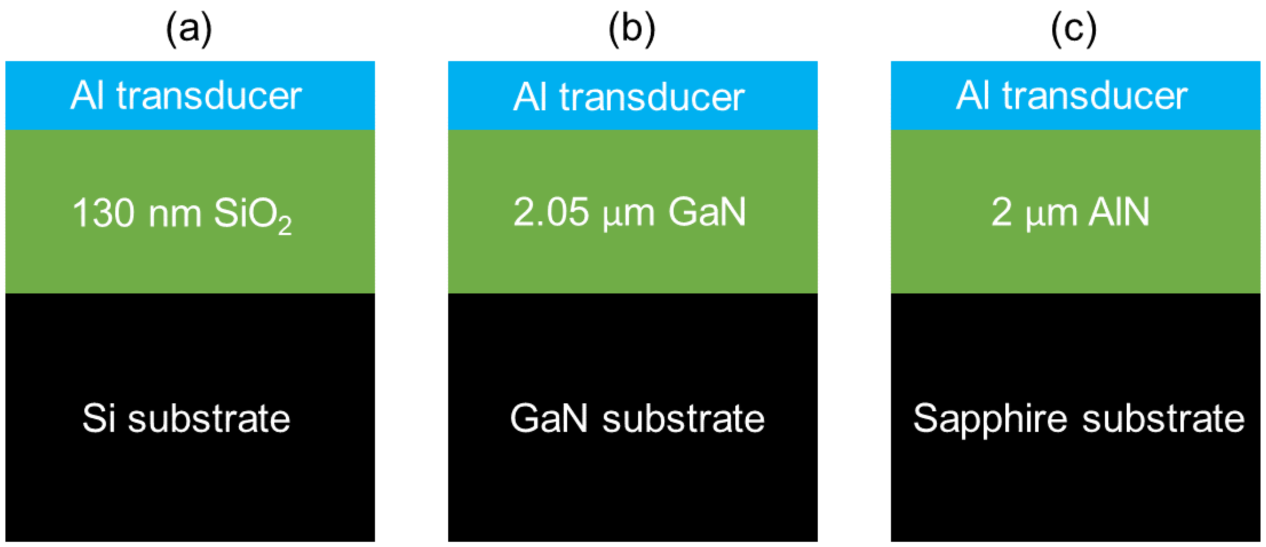
Sub-surface interfaces and heat sinks

e.g., measure thermal conductivity of buried interfaces, sub-mounts & substrates under GaN and AlN thin films



- Variable spot size allows measurement of multiple properties
- Measurement of layer-by-layer thermal conductivity in material stack/composite

Substrates	Thermal conductivity ($W\ m^{-1}\ K^{-1}$)		
	spot size 10 μm	spot size 20 μm	literature
Si	141 ± 27	140 ± 18	140[30]
GaN	194 ± 27	185 ± 16	195[41]
Sapphire	35.1 ± 5.9	34.5 ± 4.2	35[42]

Rev. Sci. Instrum. **92**, 064906

Thank You!

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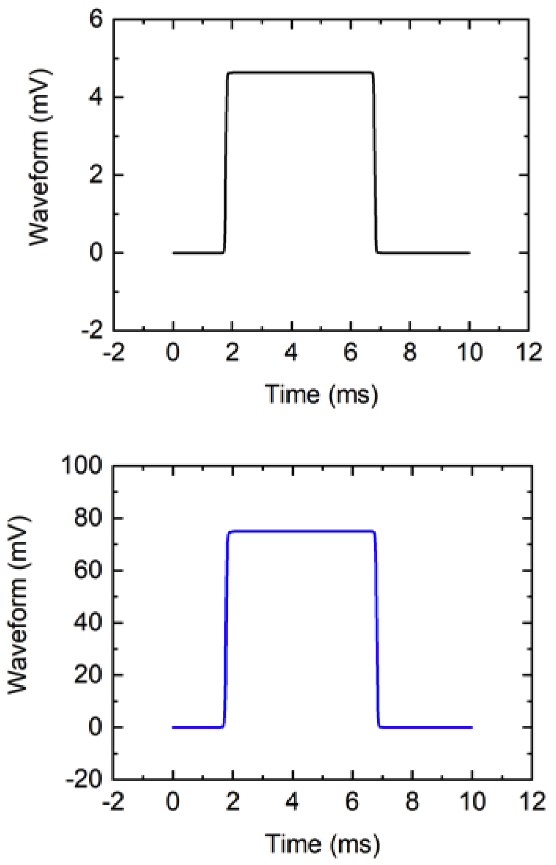
LASER THERMAL

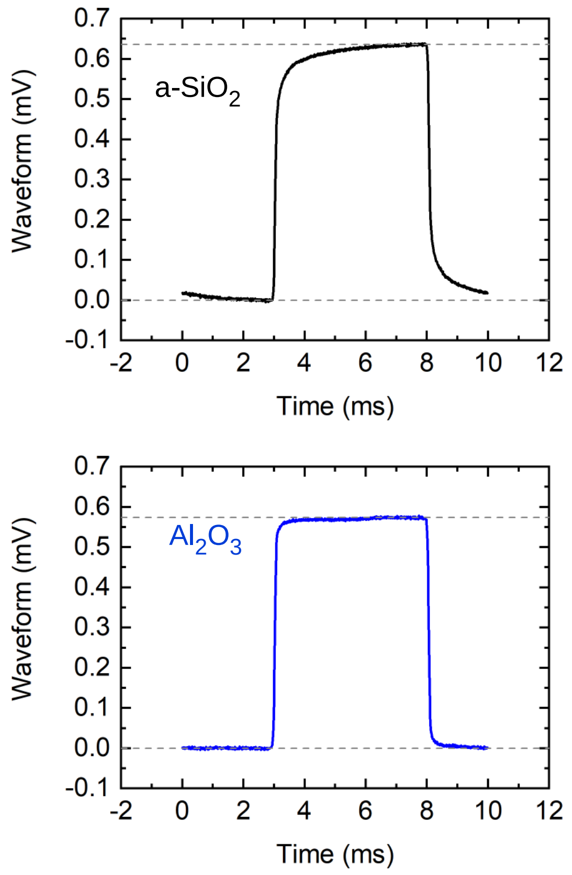
Steady-State Thermoreflectance (SSTR): Analysis Options

Periodic Waveform Analyzer with Boxcar Averager

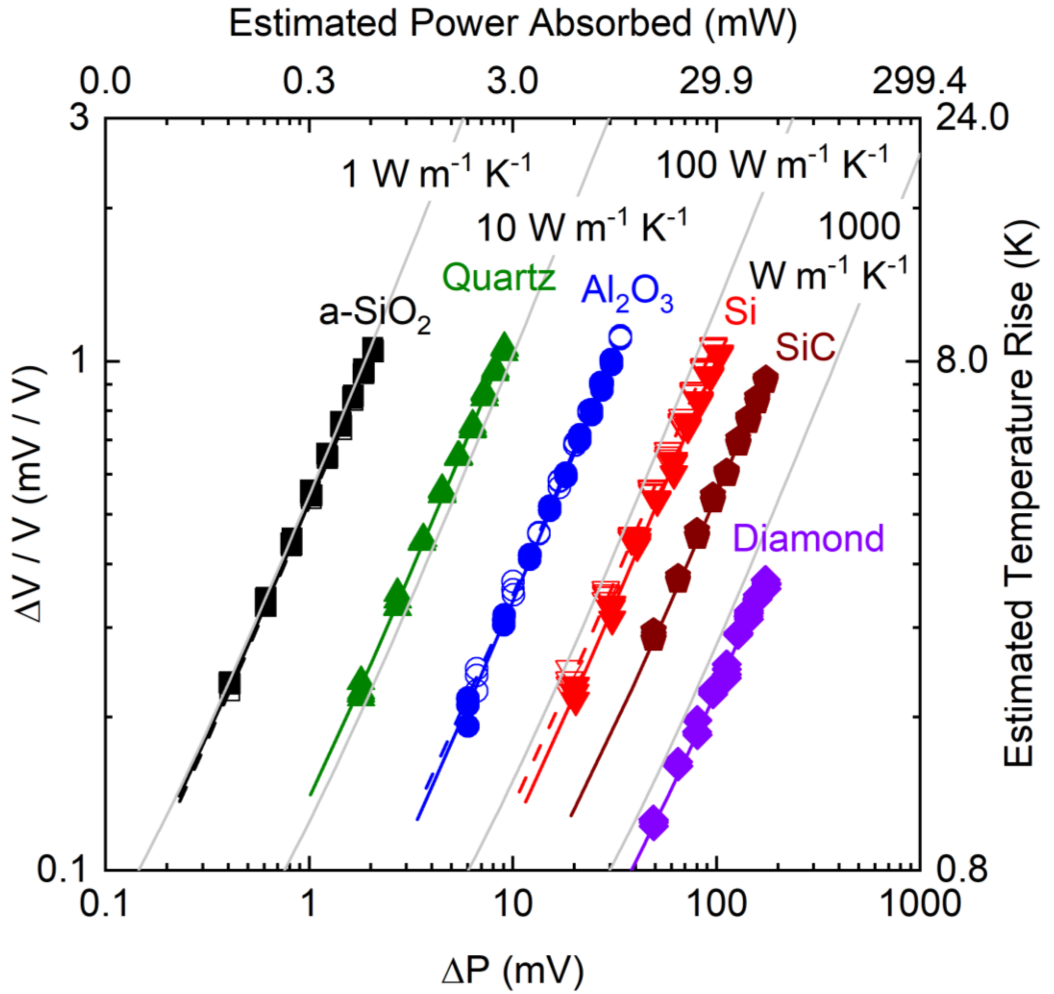
Pump Waveform



Temperature Response



Lock-in Amplifier



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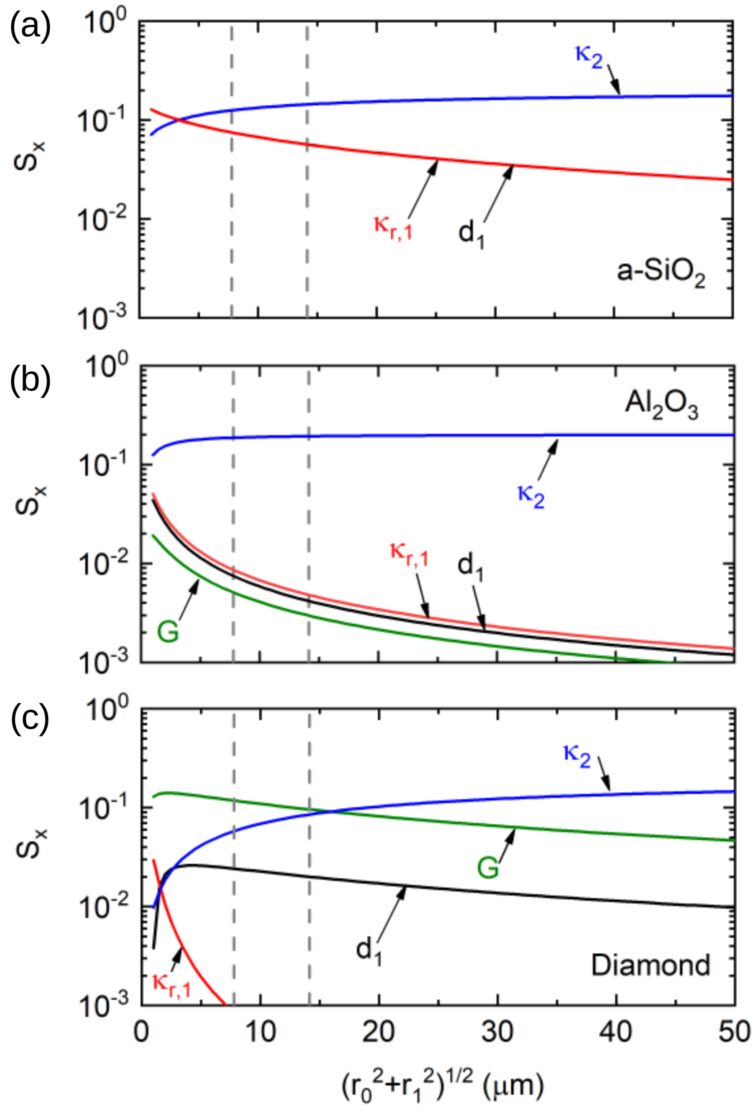
Sensitivity Analysis

Sensitivity to parameter is defined by change in temperature response to ±10% change in parameter magnitude

$$S_x = \frac{|\Delta T_{1.1x}(r_{01}) - \Delta T_{0.9x}(r_{01})|}{\Delta T_x(r_{01})}$$

S_x is defined as a function of effective pump/probe radius

Changing r_{01} allows us to change sensitivity to various parameters of interest



Sensitivity to thin films