FINAL SCRIPT: APPROVED FOR FILMING

Submission ID #: 68908
Scriptwriter Name: Poornima G
Project Page Link: https://review.jove.com/account/file-uploader?src=21016988

Title: The Frequency Domain Thermoreflectance Technique for Thermal Property Measurements

Authors and Affiliations:
Alesanmi R. Odufisan[1], Benjamin Stern[2], Ryohei Nagahiro[3], Rosemary Wynnychenko[2,4], Sevan Chanakian[5], Junichiro Shiomi[3,6], Eleonora Isotta[7], Oluwaseyi Balogun[2]

[1]Department of Theoretical and Applied Mechanics, Northwestern University
[2]Department of Mechanical Engineering, Northwestern University
[3]Institute of Engineering Innovation, The University of Tokyo
[4]Department of Physics, Wellsley College
[5]Department of Chemical Engineering and Materials Science, Michigan State University
[6]Department of Mechanical Engineering, The University of Tokyo
[7]Department of Materials Science and Engineering, Northwestern University

Corresponding Authors:

Alesanmi R. Odufisan	alesanmiodufisan2028@u.northwestern.edu
Oluwaseyi Balogun	o-balogun@northwestern.edu

Email Addresses for All Authors:

Benjamin Stern	benjaminstern2027@u.northwestern.edu
Ryohei Nagahiro	nagahiro-r@photon.t.u-tokyo.ac.jp
Rosemary Wynnychenko	rw103@wellesley.edu
Sevan Chanakian	sevchanakian@gmail.com
Junichiro Shiomi	shiomi@photon.t.u-tokyo.ac.jp
Eleonora Isotta	e.isotta@mpie.de
Oluwaseyi Balogun	o-balogun@northwestern.edu

Author Questionnaire

1. Microscopy: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**

2. Software: Does the part of your protocol being filmed include step-by-step descriptions of software usage? **Yes, all done**

3. Filming location: Will the filming need to take place in multiple locations? **No**

4. Testimonials (optional): Would you be open to filming two short testimonial statements **live during your JoVE shoot**? These will **not appear in your JoVE video** but may be used in JoVE's promotional materials. **Yes**

Current Protocol Length
Number of Steps: 23
Number of Shots: 53 (17 SC)

Introduction

Videographer: Obtain headshots for all authors available at the filming location.

INTRODUCTION:

1.1. **Oluwaseyi Balogun:** We study how micro-scale defects in materials, such as grain boundaries, affect the material's thermal conductivity with micron resolution.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.2. **Benjamin Stern:** The continuum-scale assumption that thermal properties are size-independent breaks down at small length scales.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

CONCLUSION:

1.3. **Oluwaseyi Balogun:** We have measured thermal conductivity suppression across grain boundaries in silicon and thermoelectric chalcogenides, and demonstrated that the amount of suppression correlates with the structural features of the boundary, such as misorientation angle.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.4. **Benjamin Stern:** We can measure thermal conductivity on a micron scale, rather than relying on bulk measurements provided by, for example, Laser Flash Analysis, which cannot distinguish local heterogeneity.

1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.5. **Benjamin Stern:** Grain boundaries are typically assumed to be structureless defects. However, we use FDTR to show that grain boundaries have unique thermal properties depending on their structure.

1.5.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Videographer: Obtain headshots for all authors available at the filming location.

Testimonial Questions (OPTIONAL):

Videographer: Please capture all testimonial shots in a wide-angle format with sufficient headspace, as the final videos will be rendered in a 1:1 aspect ratio. Testimonial statements will be presented live by the authors, sharing their spontaneous perspectives.

- Testimonial statements will **not appear in the video** but may be featured in the journal's promotional materials.
- **Provide the full name and position** (e.g., Director of [Institute Name], Senior Researcher [University Name], etc.) of the author delivering the testimonial.
- Please **answer the testimonial question live during the shoot**, speaking naturally and in your own words in **complete sentences**.

How do you think publishing with JoVE will enhance the visibility and impact of your research?

1.6. **Oluwaseyi Balogun, Professor of Mechanical Engineering at Northwestern University**: (authors will present their testimonial statements live)

1.6.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 4.2.1*

Can you share a specific success story or benefit you've experienced—or expect to experience—after using or publishing with JoVE? (This could include increased collaborations, citations, funding opportunities, streamlined lab procedures, reduced training time, cost savings in the lab, or improved lab productivity.)

1.7. **Oluwaseyi Balogun, Professor of Mechanical Engineering at Northwestern University**: (authors will present their testimonial statements live)

1.7.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 3.2.1*

Protocol

2. Equipment Setup for Measurement

Demonstrator: Benjamin Stern

2.1. To begin, turn the power switch of the 532-nanometer diode laser to the **ON** position to activate the power supply **[1]**. Wait until the enable light turns on, indicating that the laser crystal's temperature is stable **[2]**. Then, turn the laser switch to initiate the emission of the 532-nanometer laser **[3]** and use a power meter to confirm that the input probe laser power is between 20 and 30 milliwatts before proceeding **[4]**.

2.1.1. WIDE: Talent turning the power switch of the 532 nanometer diode laser to the **ON** position.

2.1.2. Close-up shot of the enable light turning on as the laser temperature stabilizes.

2.1.3. Talent switching on the laser to begin the 532 nanometer emission.

2.1.4. Talent using a power meter to check that the laser power is within 20–30 milliwatts.

2.2. Next, turn on the pump laser diode controller **[1]**. Enable thermoelectric cooling by setting the correct resistance value according to the diode laser specifications. After the temperature stabilizes, switch on the laser current **[2]** and press the emission button **[3]**.

2.2.1. Talent turning on the pump laser diode controller.

2.2.2. Show the device panel with resistance settings, where the value is adjusted to 10 kiloohms for thermoelectric cooling. Talent switching on the laser current once the temperature is stable.

Added shot 2.3.3 Press the emission button

2.3. Then, turn on the erbium-doped fiber amplifier or EDFA **[1]** and set the output power to the desired value in watts **[2]**.

2.3.1. Talent powering on the EDFA unit.

2.3.2. Display the power adjustment interface where the output is set to 1 watt.

2.4. Set the radio frequency or RF, RF amplitude, and bias voltage on the signal generator to modulate the pump laser intensity **[1]**.

2.4.1. Show the signal generator interface where the RF frequency, amplitude, and bias voltage are adjusted.

2.5. Now, turn on the lock-in amplifier and photodetector **[1]**. Ensure the reference port is connected to the RF signal generator output **[2]**, and the signal input port is connected to the photodetector **[3]**.

2.5.1. Talent powering on the lock-in amplifier and photodetector.

2.5.2. Close-up shot of pointing to the cable connection between the RF signal generator output and the reference port. **Videographer's NOTE**: 2.5.2 Can't be a single close-up shot. I changed the angle to show what was needed throughout the clip.

2.5.3. Close-up shot of pointing to the photodetector connected to the signal input port. **Videographer's NOTE:** 2.5.3 is at the end of 2.5.2

2.6. Turn on the temperature controller for the periodically poled lithium niobate crystal **[1]** and enable the proportional-integral-derivative or PID control **[2]**.

2.6.1. Talent turning on the temperature controller for the PPLN crystal.

2.6.2. Show the PID control interface being activated.

2.7. Mount the sample on the multi-axis precision stage equipped with a micrometer knob for Z-position adjustment **[1]**. Use thin double-sided tape to secure the sample and prevent it from sliding during measurement **[2]**.

2.7.1. Talent mounting the sample on the multi-axis precision stage. **Videographer's NOTE:** 2.7.1 - 2.7.2 combined

2.7.2. Close-up shot of the sample being fixed onto the stage using thin double-sided tape.

2.8. Next, mount the cold mirror above the objective to begin focusing the lasers on the sample surface **[1]**. Observe the sample surface through the CCD camera and adjust the Z-knob on the stage until the surface comes into focus on the live camera feed **[2]**. Continue to fine-tune until the laser spot reaches its minimum size **[3]**. Once in focus, remove the cold mirror **[4]**.

2.8.1. Talent placing the cold mirror above the objective.

2.8.2. Talent adjusting the Z-knob.

2.8.3. Close-up of the laser spot minimizing in size during fine adjustment.

2.8.4. Talent carefully removing the cold mirror after achieving focus.

2.9. Now, using the LabVIEW *(lab view)* virtual instrument, adjust the tilt angles of the Gimbal steering mirror to align the pump laser along the optical axis of the probe laser **[1]**. Monitor the thermoreflectance amplitude and look for the maximum signal to confirm the lasers are coincident on the sample surface **[2]**.

2.9.1. SCREEN: 2.9.1.mp4 00:10-00:27.

2.9.2. Show the overlapping pump and probe laser spots coinciding on the sample surface, indicating maximum thermoreflectance amplitude.

3. Point Measurements

3.1. Mount the sample on the multi-axis translation stage **[1]**.

3.1.1. Talent mounting the sample carefully onto the multi-axis translation stage. **Videographer's NOTE:** 3.1.1 can re-use shot 2.7.1

3.2. View the sample through the CCD camera **[1]** and adjust its position to ensure that the probe laser is reflected at a clean, impurity-free spot on the surface **[2]**.

3.2.1. Talent looking at the sample through the CCD camera.

3.2.2. Talent adjusting the stage position.

3.3. Then, select the desired pump power level **[1]** and perform the focus and alignment procedures as demonstrated earlier **[2]**.

3.3.1. Show the power control panel where the pump laser power is adjusted to the selected level. **Videographer's NOTE:** 3.3.1 can re—use shot 2.3.2

3.3.2. Shot of the fully aligned pump and probe lasers on the sample surface. **Videographer's NOTE:** 3.3.2 can re-use 2.9.2. Showing it on the surface is not possible

3.4. Decide on the frequency range for the point measurement **[1]**. Verify that the chosen thermal diffusion model is sensitive and appropriate for the material properties within that range **[2]**.

3.4.1. SCREEN: 3.4.1---3.5.3.mp4 00:10-00:18.

3.4.2. SCREEN: 3.4.1---3.5.3.mp4 00:18-00:21. *Video editor: Highlight the Sensitivity-1 box on the left side of the screen where the cursor points*

3.5. Select the proper sensitivity level on the lock-in amplifier for the chosen frequency range **[1]**. Program the point measurement LabVIEW virtual instrument with the lock-in sensitivity level, frequency range, and total number of frequencies to be measured **[2]**. If required, configure multiple stages with varying sensitivity levels to optimize the signal-to-noise ratio **[3]**.

3.5.1. SCREEN: 3.4.1---3.5.3.mp4 00:24-00:35.

3.5.2. SCREEN: 3.4.1---3.5.3.mp4 00:36-00:48.

3.5.3. SCREEN: 3.4.1---3.5.3.mp4 00:49-00:52.

3.6. Run the point measurement and save the recorded data as a text file **[1]**. Record the phase data from the lock-in amplifier three times: once for the probe laser signal, once for the reference laser signal, and once for the noise **[2]**. Block the unused beams during each recording, and block both beams when collecting noise data **[3-TXT]**.

3.6.1. SCREEN: 3.6.1.mp4 00:05-00:12.

3.6.2. SCREEN: 3.6.1.mp4 00:13-00:24.

3.6.3. Talent adjusting the settings for blocking beams. **TXT: Repeat the measurements to obtain a final averaged dataset**

4. Image Measurements

4.1. To begin, mount the sample on the multi-axis translation stage and position the laser spot directly over the intended measurement area **[1]**. Ensure that the selected region is free of dirt and irregularities in surface roughness **[2]**. Avoid moving the translation axes of the stepper motor stage to their extremes to prevent coupling with the Z-axis **[3]**.

4.1.1. Talent mounting the sample on the multi-axis translation stage. **Videographer's NOTE:** 4.1.1 can re-use 2.7.1

4.1.2. Show the CCD camera feed as the laser spot is positioned on a clean and even area of the sample surface. **Videographer's NOTE:** 4.1.2 can re-use 3.2.1

4.1.3. Close-up shot of the stage controls.

4.2. Now, program the measurement area into the image measurement LabVIEW virtual instrument **[1]**. Run a quick scan over the designated area, for example, three steps in both the X and Y directions, while monitoring the CCD camera live feed **[2]**. Identify and confirm the precise boundaries of the measurement area **[3]**.

4.2.1. SCREEN: 4.2.1.mp4. 00:00-00:08

4.2.2. Show the live CCD camera feed capturing a quick scan in both X and Y directions.

4.2.3. SCREEN: 4.2.1.mp4 00:08-00:10

4.3. Now, select the desired pump power level and complete the focus and alignment procedure **[1]**.

4.3.1. Show the pump power level being set to the required value on the control interface. **Videographer's NOTE:** 4.3.1 same as 2.3.2

4.4. Select at least five frequencies at which the sample will be scanned **[1]**. Verify that the chosen thermal model is appropriately sensitive to the sample's measured properties within this frequency range **[2]**.

4.4.1. Show the panel where frequencies are being selected.

4.4.2. Display the selected frequency range.

4.5. Block the probe laser and record the in-phase X and quadrature Y readings for the reference laser from the lock-in amplifier display **[1]**. Then, block both the probe and reference lasers to capture the X and Y noise readings **[2]**. When data collection is complete, unblock the probe laser **[3]**.

4.5.1. Show the panel where data appears.

4.5.2. Show the panel displaying data for the noise after both lasers are blocked.

4.5.3. Shot of unblocking the probe laser after completing data acquisition.

4.6. Input the number of steps, representing the number of point measurements, for each scan direction into the LabVIEW VI **[1]** and start the map measurement **[2]**.

4.6.1. SCREEN: 4.6.1.mp4.

4.6.2. SCREEN: 4.6.2.mp4. 00:00-0:08

4.7. Once the map measurement is complete, repeat the procedure for recording X and Y readings for both the reference and noise signals **[1]**. Finally, calculate the average X and Y values from before and after the measurement for both signals to obtain the final corrected results **[2]**.

4.7.1. SCREEN: 4.7.1.mp4.

4.7.2. LAB MEDIA: 4.7.2.png.

Results

5. Results

5.1. The gold transducer layer thickness measured using the picosecond ultrasonic method showed coherent oscillations in the time-domain waveform **[1]**, with a corresponding Fourier amplitude peak at 29 gigahertz **[2]**, giving a thickness of around 55.5 nanometers **[3]**.

5.1.1. LAB MEDIA: Figure 3A.

5.1.2. LAB MEDIA: Figure 3A. *Video editor: Emphasize the inset orange Fourier transform graph's peak near 29 gigahertz*.

5.1.3. LAB MEDIA: Figure 3A. *Video editor: Zoom in on the text "Gold Thickness = (55.5 ± 0.3) nm"*.

5.2. Knife-edge measurements determined the pump and probe laser spot sizes with average 1 by e radii of approximately 10 micrometers along both X and Y directions **[1]**, confirming circular symmetry of the laser spots **[2]**.

5.2.1. LAB MEDIA: Figure 3B.

5.2.2. LAB MEDIA: Figure 3C.

5.3. The thermoreflectance phase sensitivity curves demonstrated distinct frequency-dependent behavior for the substrate thermal conductivity and interfacial conductance, confirming uncorrelated properties between 100 kilohertz and 10 megahertz **[1]**.

5.3.1. LAB MEDIA: Figure 4A. *Video editor: Focus on the blue and orange solid lines labeled κ (Si) and G (Au–Si).*

5.4. The measured thermoreflectance phase decreased with increasing modulation frequency **[1]** and showed a turning point near 1 megahertz, where heat flow transitioned from three-dimensional to one-dimensional **[2]**.

5.4.1. LAB MEDIA: Figure 4B. *Video editor: Highlight the blue phase data points along the curve*.

5.4.2. LAB MEDIA: Figure 4B. *Video editor: Emphasize the data points near the 100 mark on the X-axis, basically the base of the curve showing a depression*.

5.5. Monte Carlo simulations of probe spot size uncertainty produced Gaussian distributions for fitted substrate thermal conductivity and interfacial conductance values, with corresponding uncertainties of around 5 and 3.3% **[1]**.

5.5.1. LAB MEDIA: Figure 5A. *Video editor: Highlight the black dashed lines in both the purple and green histograms*.

5.6. Increasing uncertainties in model inputs led to linear increases in uncertainty of fitted thermal properties, with pump and probe spot sizes contributing the largest propagation errors **[1]**.

5.6.1. LAB MEDIA: Figure 5B. *Video editor: Focus on the lines for "pump radius" and "probe radius" trending upward.*

5.7. Local thermal conductivity mapping across a vertical silicon–silicon interface showed a 3% reduction in conductivity at the interface **[1]**, while the interfacial conductance map remained nearly uniform **[2]**.

5.7.1. LAB MEDIA: Figure 6A. *Video editor: Highlight the blue vertical stripe between 25 and 50 marks on the X-axis*.

5.7.2. LAB MEDIA: Figure 6B.

- thermoelectric

Pronunciation link: https://www.merriam-webster.com/dictionary/thermoelectric
IPA: /ˌθɜr·moʊ·ɪˈlɛk·trɪk/
Phonetic Spelling: ther-moh-ih-LEK-trik

- diode

Pronunciation link: https://www.merriam-webster.com/dictionary/diode
IPA: /ˈdaɪ·oʊd/
Phonetic Spelling: DIE-ohd

- amplifier

Pronunciation link: https://www.merriam-webster.com/dictionary/amplifier
IPA: /ˈæm·plɪˌfaɪ·ər/
Phonetic Spelling: AM-pli-fai-er

- gimbal

Pronunciation link: https://www.merriam-webster.com/dictionary/gimbal

IPA: /ˈdʒɪm·bəl/
Phonetic Spelling: JIM-buhl

- photodetector

Pronunciation link: https://www.merriam-webster.com/dictionary/photodetector
IPA: /ˌfoʊ·toʊ·dɪˈtɛk·tər/
Phonetic Spelling: FOH-toh-di-TEK-ter

- proportional-integral-derivative

Pronunciation link: https://www.merriam-webster.com/dictionary/proportional-integral-derivative
IPA: /prəˌpɔr·ʃəˈnæl·ɪn·tə·ˌgræl·dɪˈrɪv·ə·tɪv/
Phonetic Spelling: pruh-POR-shuh-nal-IN-tuh-GRAL-di-RIV-uh-tiv

- multi-axis

Pronunciation link: https://www.merriam-webster.com/dictionary/multi-axis
IPA: /ˌmʌl·tiˈæk·sɪs/
Phonetic Spelling: MUL-tee-AK-sis

- micrometer

Pronunciation link: https://www.merriam-webster.com/dictionary/micrometer
IPA: /maɪˈkrɒm·ɪ·tər/
Phonetic Spelling: my-KROM-ih-ter

- thermoreflectance

Pronunciation link: No confirmed link found
IPA: /ˌθɜr·moʊ·rɪˈflɛk·təns/
Phonetic Spelling: THER-moh-ri-FLEK-tans

- interfacial

Pronunciation link: https://www.merriam-webster.com/dictionary/interfacial
IPA: /ˌɪn·tərˈfeɪ·ʃəl/
Phonetic Spelling: in-ter-FAY-shuhl