

High-throughput Thermal Resistance Mapping for 2D MXene via Probe Thermal Resistance Calibration in Scanning Thermal Microscopy (SThM)

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Co-authors: Brian C Wyatt, Krutarth Kiran Kamath, Babak Anasori, Raisul Islam



School of Materials Engineering



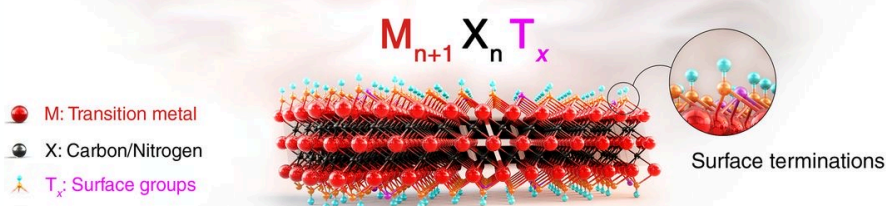
Outline

- **Thermal Properties of Ti_3C_2 MXene**
- **Thermal Characterization Techniques**
- **Scanning Thermal Microscopy (SThM)**
- **Qualitative $\rightarrow$ Quantitative : *Most challenging***
- **Thermal Resistance Mapping of Single Flake Ti_3C_2 MXene**

Outline

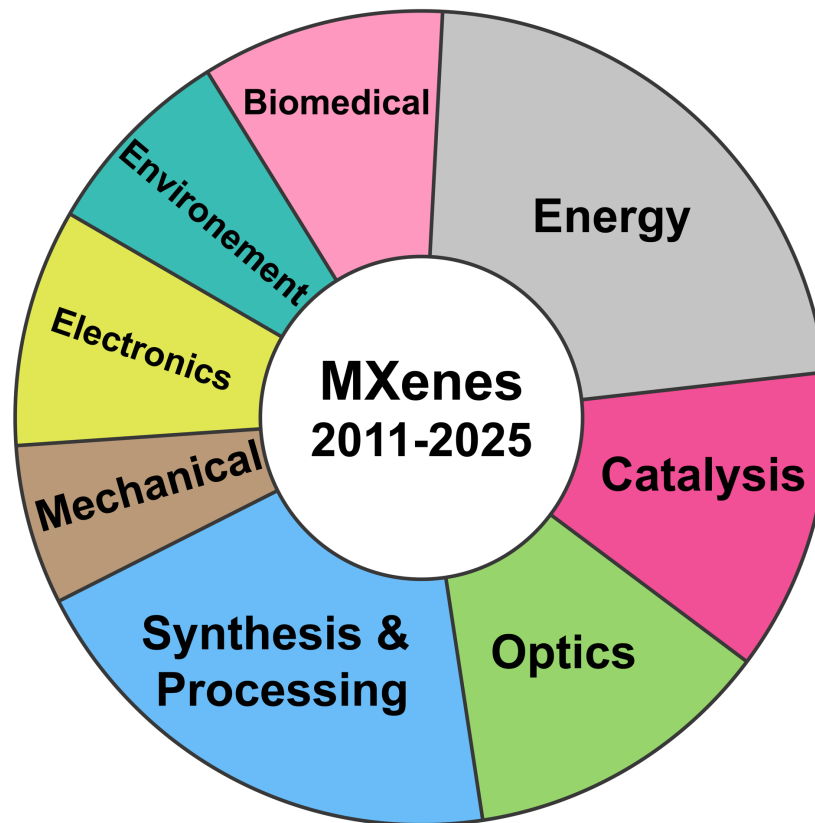
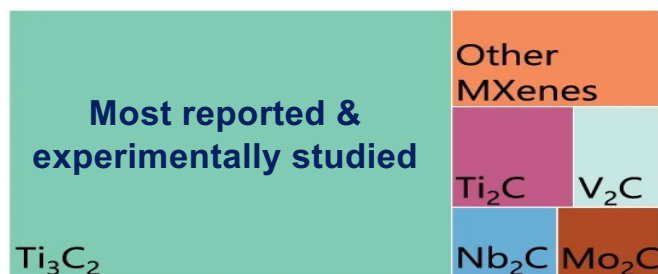
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- Qualitative $\rightarrow$ Quantitative

MXene: Properties and Applications



Anasori, B. & Gogotsi, Y. Graphene and 2D materials, 2022

- **Surface Chemistry** → *Multiple functionalization*
- **High capacitance** up to 1500 F/cm³
- **High Thermal Stability**
- **Layered structure** → *easy to scale*

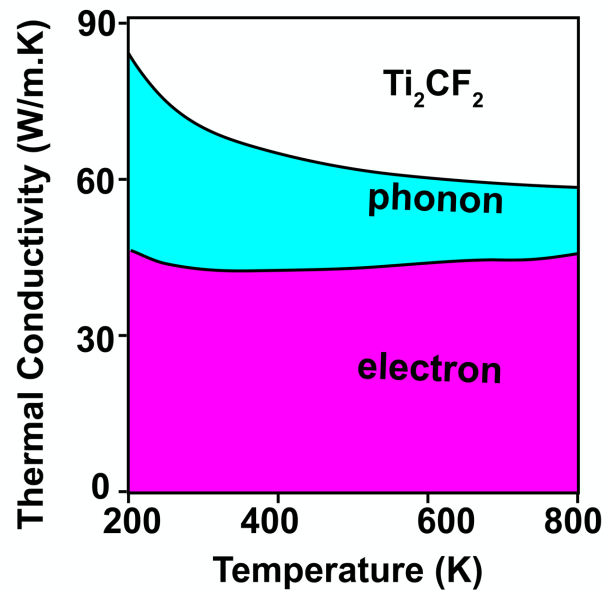


VahidMohammadi et al. Science, 2021

Thermal Transport in MXene

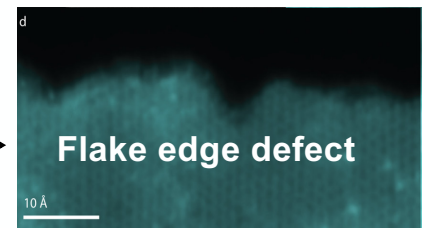
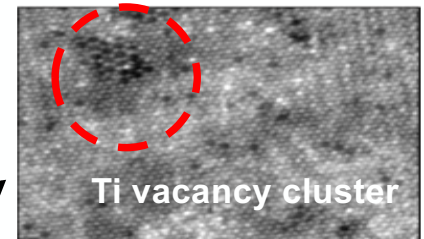
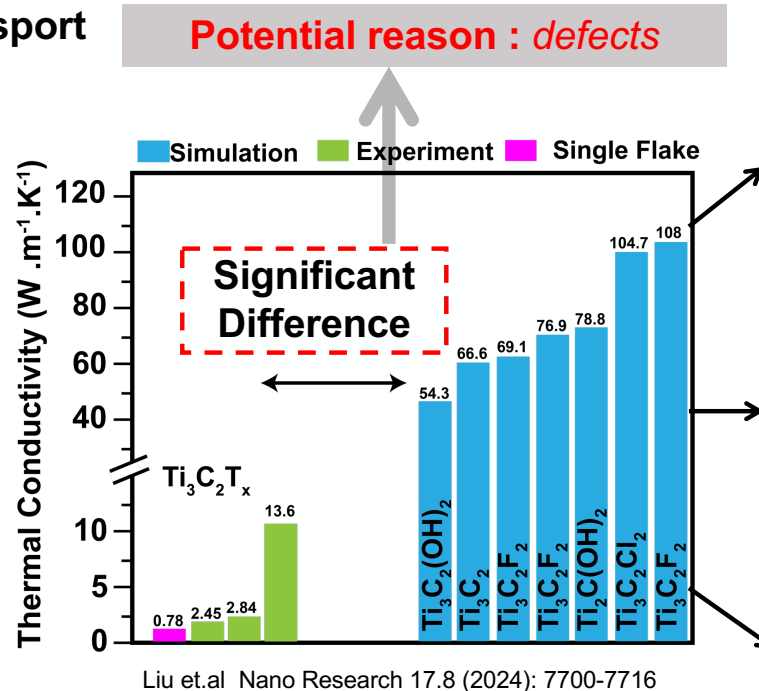
Michałowski, P. P. et al., *Nat Nanotechnology*, 2022

- Phonon dominated thermal transport

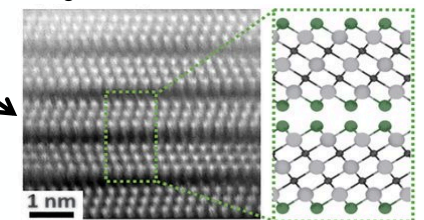


Wang, Ao, et al. *Physical Review Materials* 6.1 (2022): 014009

Karlsson, L. H., Birch, J., Halim, J., Barsoum, M. W. & Persson, P. O. *Nano Lett*, 2015



Sang, X. et al., *ACS Nano*, 2016



Non-uniform functionalization

Outline

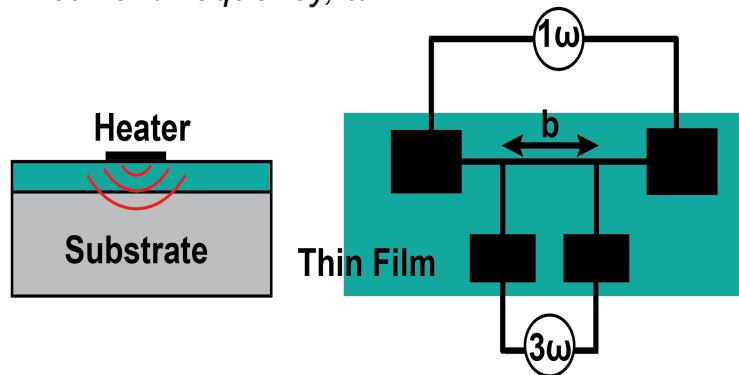
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Thin Film Thermal Characterization

Christofferson et al. ASME (2008): 041101

3-Omega Technique

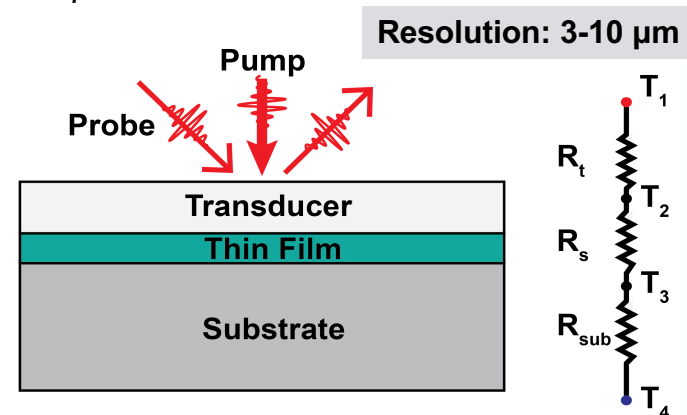
- Temperature sensor built on film
- Measures thermal diffusivity - function of current frequency, ω



→ Complex sample preparation – potential membrane damage for MXene

Time-domain Thermo-reflectance (TDTR)

- Use a laser source for “pump” & “probe”
- Probe measures reflectivity - function of temperature



→ Sample must be optically reflective, transducer required – not compatible for all MXene

Both methods are not ideal for MXenes

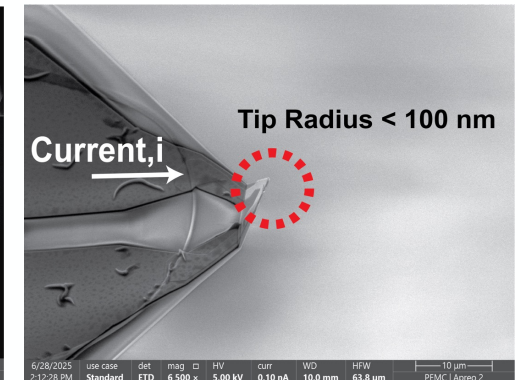
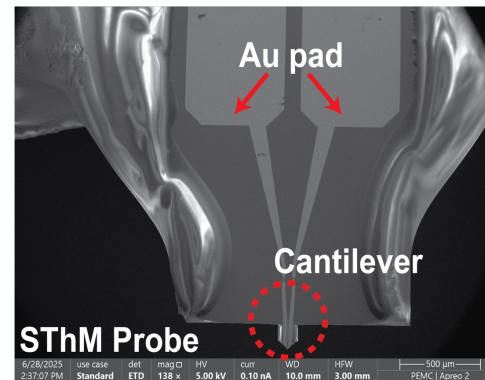
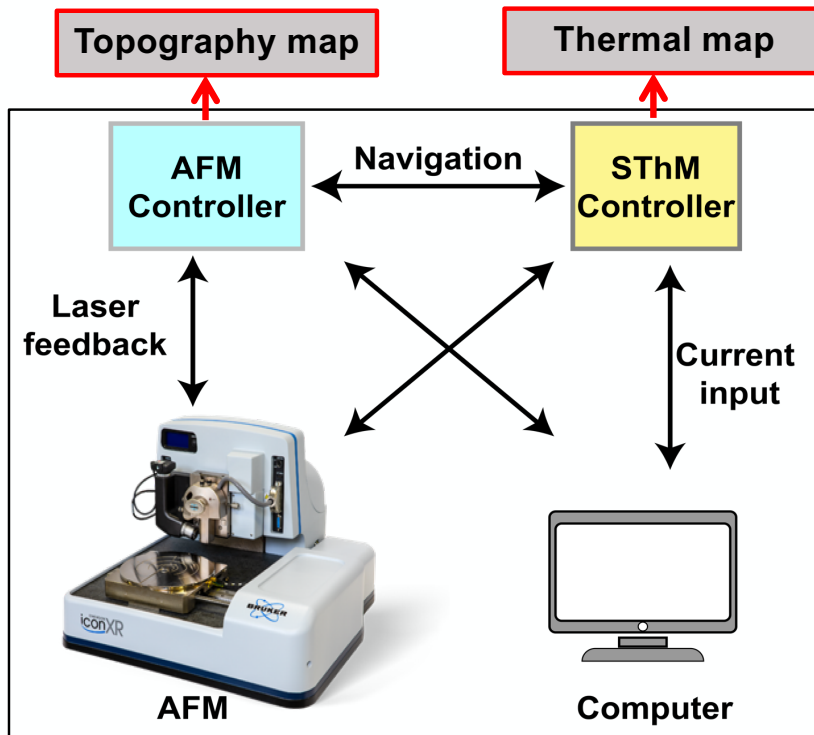
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Scanning Thermal Microscopy (SThM)

- Atomic force microscopy (AFM) based system
- Thermal sensor probe $\rightarrow \Delta R/R_0 = \alpha \Delta T$, $\alpha: TCR$

R. Munde, [..], R. Islam et al. JAP arXiv:2511.09960 (2025) (Under review)



- High spatial resolution (*sub-100 nm*)
- High throughput measurements (*scan imaging*)
- No additional sample preparation (*just like AFM*)
- No limitation on sample type & thickness (*semiconductor, metal or dielectric*)

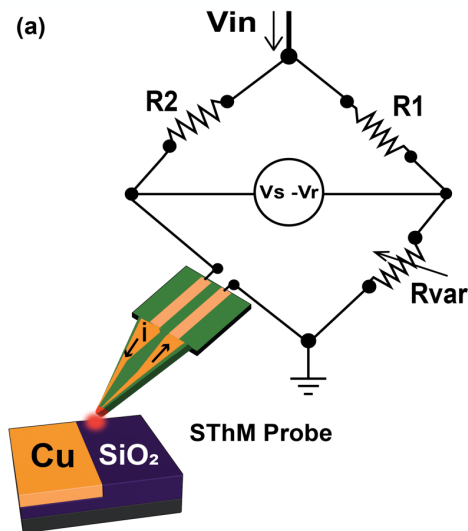
SThM: Mechanism & Output

R. Munde, [...], R. Islam et al. JAP arXiv:2511.09960 (2025) (Under review)

- SThM probe mounted on Wheatstone bridge
- Capable of resolving $\Delta T \sim 0.1^\circ\text{C}$

- Local thermal conductivity contrast
(Grain boundaries, surface defects, dislocations, etc.)

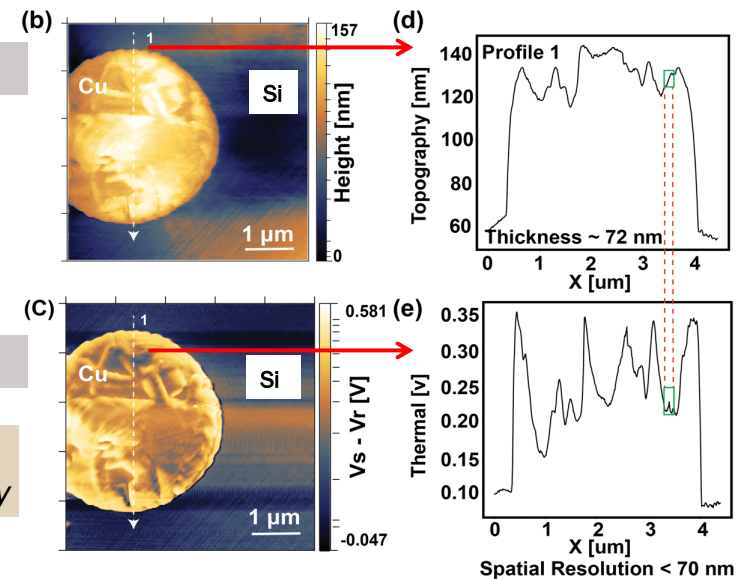
sample courtesy: Tiwei Wei, UCLA



Topography

Thermal

High thermal voltage
→ low thermal conductivity



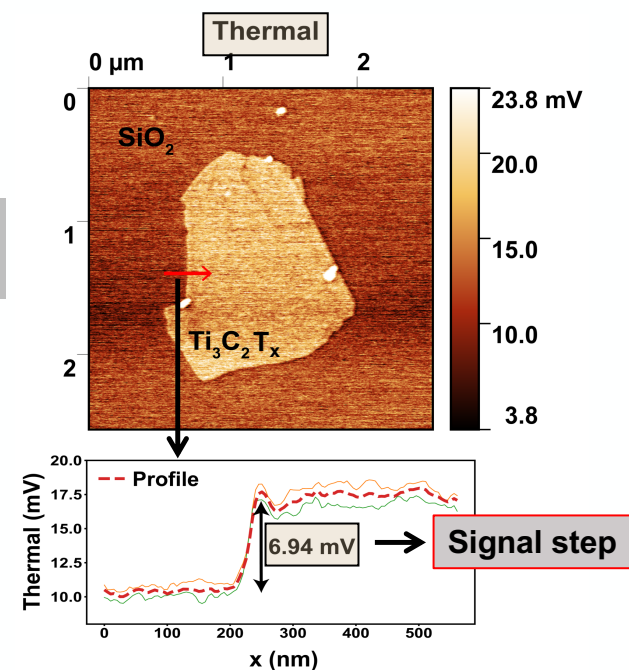
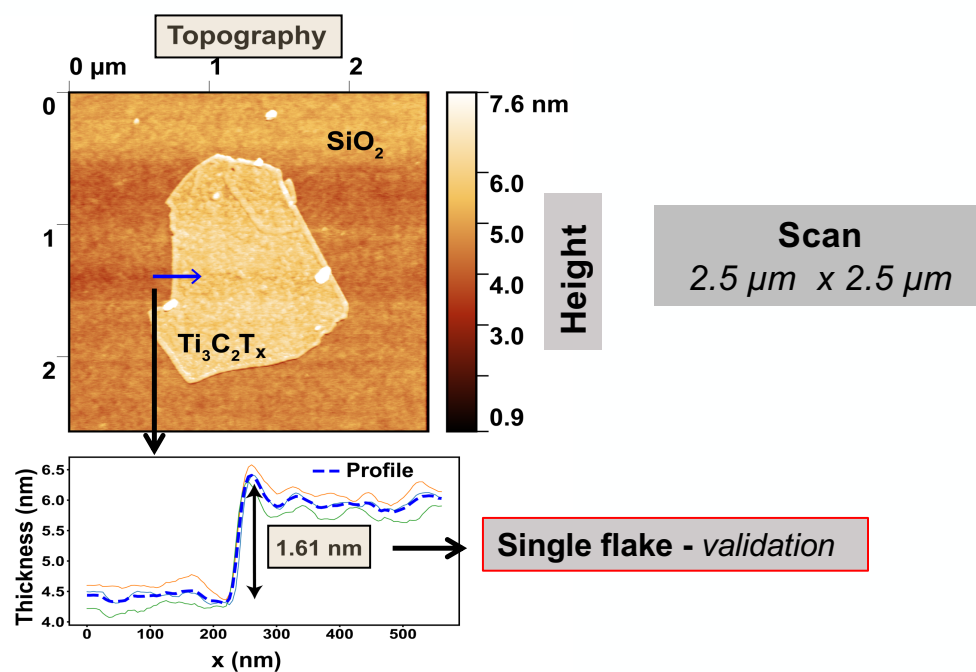
Thermal signal output ($V_s - V_r$) → To interpret local thermal conductivity

Single Flake Ti_3C_2 MXene

Thakur Small Methods 7.8 (2023): 2300030

- Spin coated on SiO_2 substrate

- Clear contrast between SiO_2 and flake



High thermal voltage for flake $\rightarrow$ low thermal conductivity (relative to SiO_2)

Outline

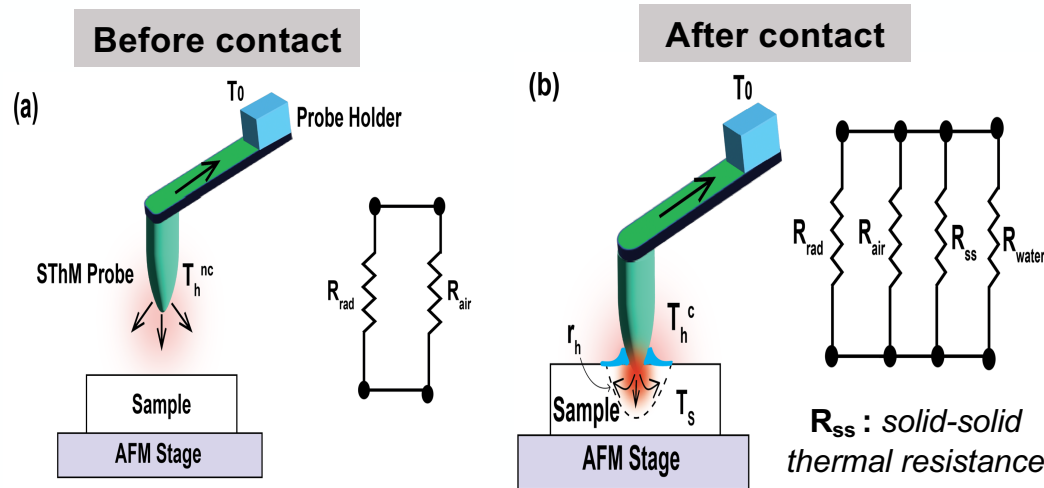
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Qualitative → Quantitative: SThM Calibration

R. Munde, [...], R. Islam et al. JAP arXiv:2511.09960 (2025) (Under review)

$$R_c = R_{\text{tip}} + R_{\text{interface}} + R_{\text{spreading}}$$

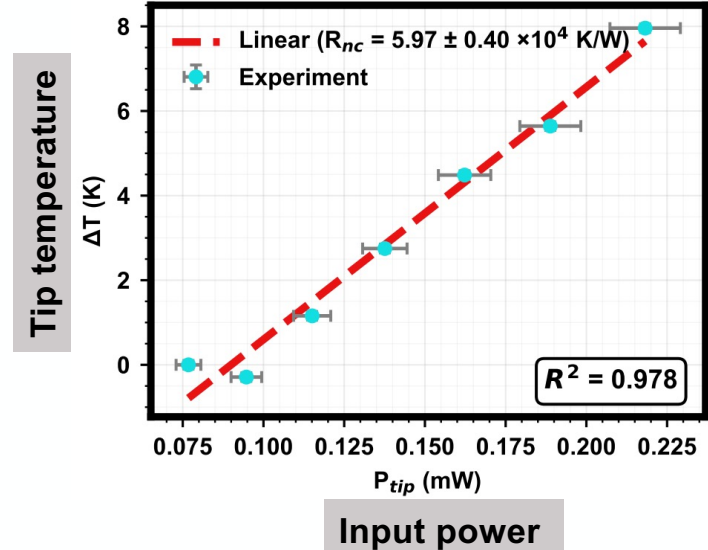
- Developed a circuit-based calibration method
- Conventional methods use a heating stage → sensitive to parasitic behavior



After some reasonable assumptions:

$$\frac{V^{nc} - V^c}{V^{nc}} = \frac{R_{nc}}{R_{nc} + R_c}$$

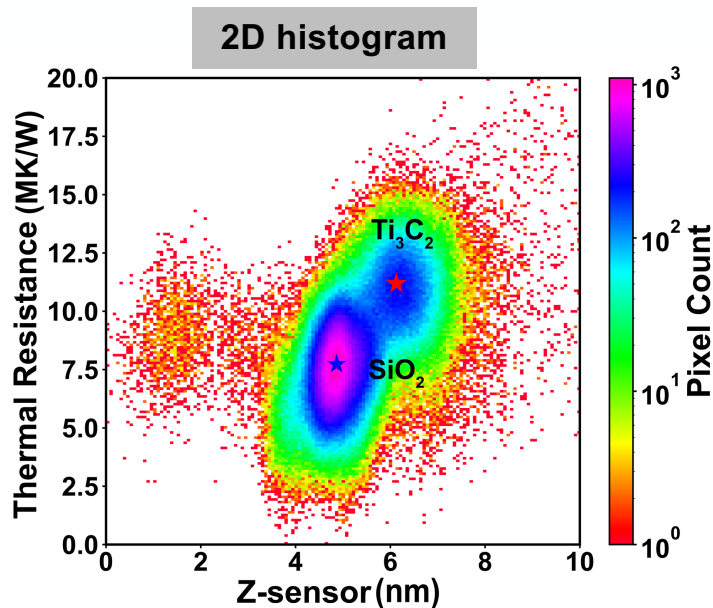
V^{nc} : before contact thermal signal
 V^c : after contact thermal signal
 R_{nc} : before contact thermal resistance



$R_{nc} = 5.97 \times 10^4 \text{ K/W}$, matches with the literature

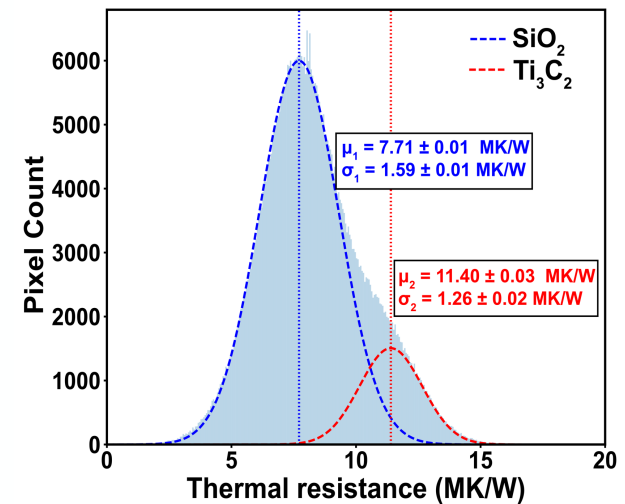
Qualitative → Quantitative: Thermal conductivity

- Pixel-wise analysis → *considers all signals*
- Material thermal resistance → *shows denser distribution*



Uncertainty:
water meniscus
(upto 6%), σ

- Double-Gaussian fitting → *Extraction of thermal resistances from Ti_3C_2 & SiO_2*
- Mean thermal resistance (μ) & std deviation (σ) → *Thermal conductivity*



SiO_2
1.4 W/m.K



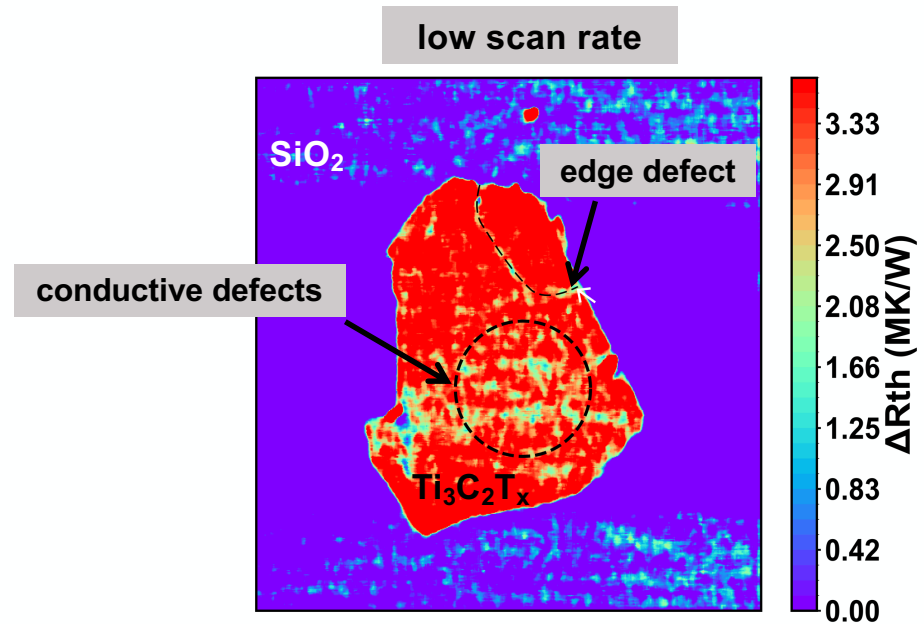
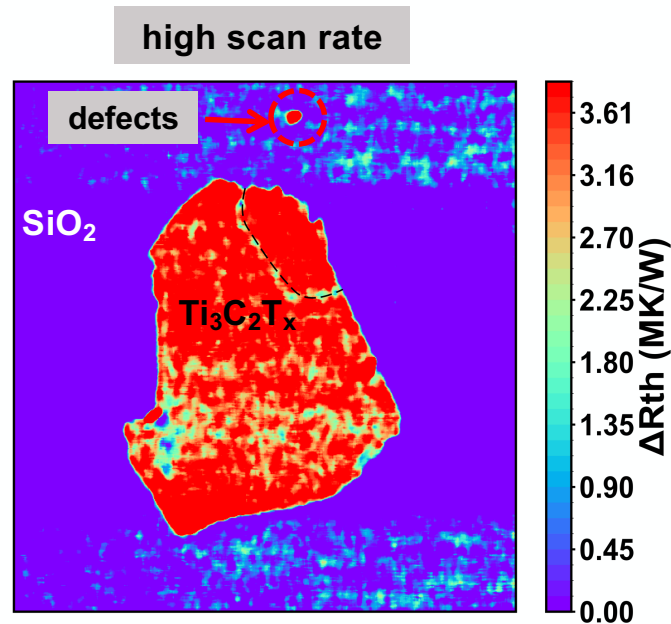
Ti_3C_2
Thermal Conductivity, k
 0.93 ± 0.22 W/(m.K)

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Thermal resistance map

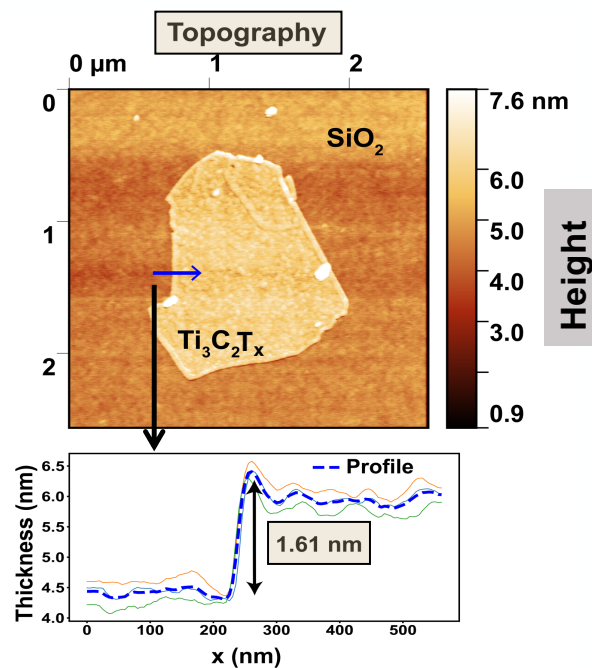
- Thermal resistance map $\rightarrow$ *local heat transport*
- Non-uniform thermal conductivity $\rightarrow$ *surface terminations, defects*
- Spatial resolution < 100 nm



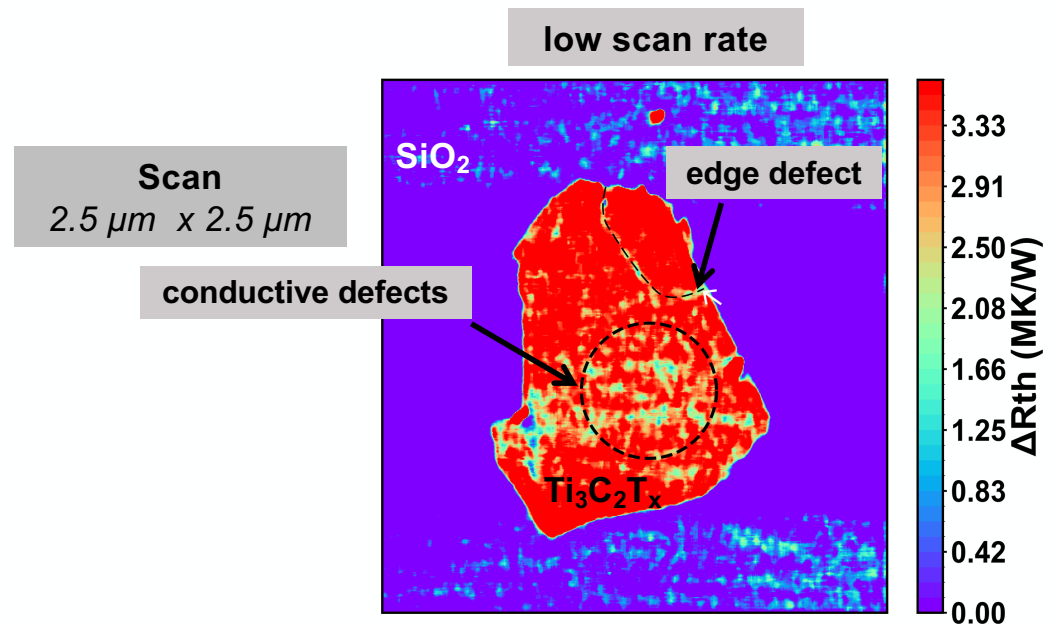
Low scan rate: Provides better resolution (*With cost*)

Revealing Thermally Induced Defects in Materials

- Spin coated on SiO_2 substrate



- Spatial resolution < 100 nm



Study of thermally induced defects: *A New Frontier*

Summary

- **Thermal Properties of Ti_3C_2 MXene** → *Phonon dominated*
- **Thermal Characterization Techniques** → *Traditional methods are not Ideal*
- **Scanning Thermal Microscopy** → *Provides nanoscale thermal characterization*
- **Qualitative** → **Quantitative** : *Most challenging*
- **Thermal Resistance Mapping of Single Flake Ti_3C_2 MXene** → *Useful for defect study*

Acknowledgements

Dr. Raisul Islam

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Special thanks to:

Brian C. Wyatt

Argonne National Laboratory

Krutarth Kiran Kamath

Ph.D. Candidate, MSE, Purdue University

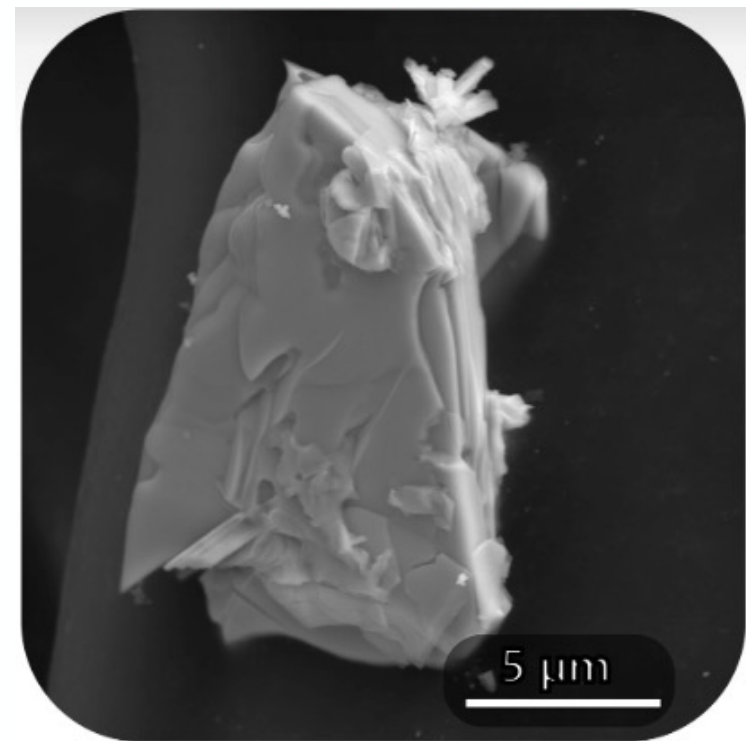
Prof. Babak Anasori

Associate Professor, MSE, Purdue University

Questions ?

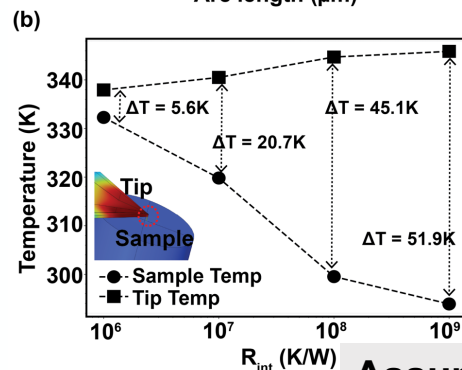
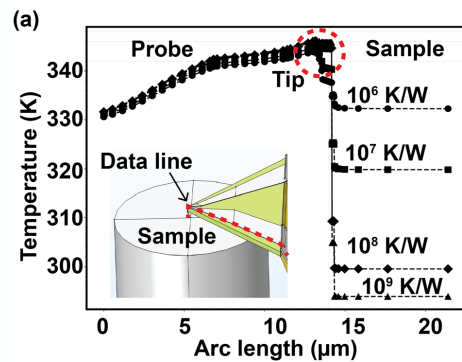
Wyatt Science 389.6764 (2025): 1054-1058.

- **Scanning Thermal Microscopy**
→ *provides nanoscale thermal characterization*
- **Thermal Resistance Mapping of Single Flake Ti_3C_2 MXene**
→ *useful for defect study*
- **Potential to study high entropy MXene**
→ *Thermal conductivity contrast for each metal*

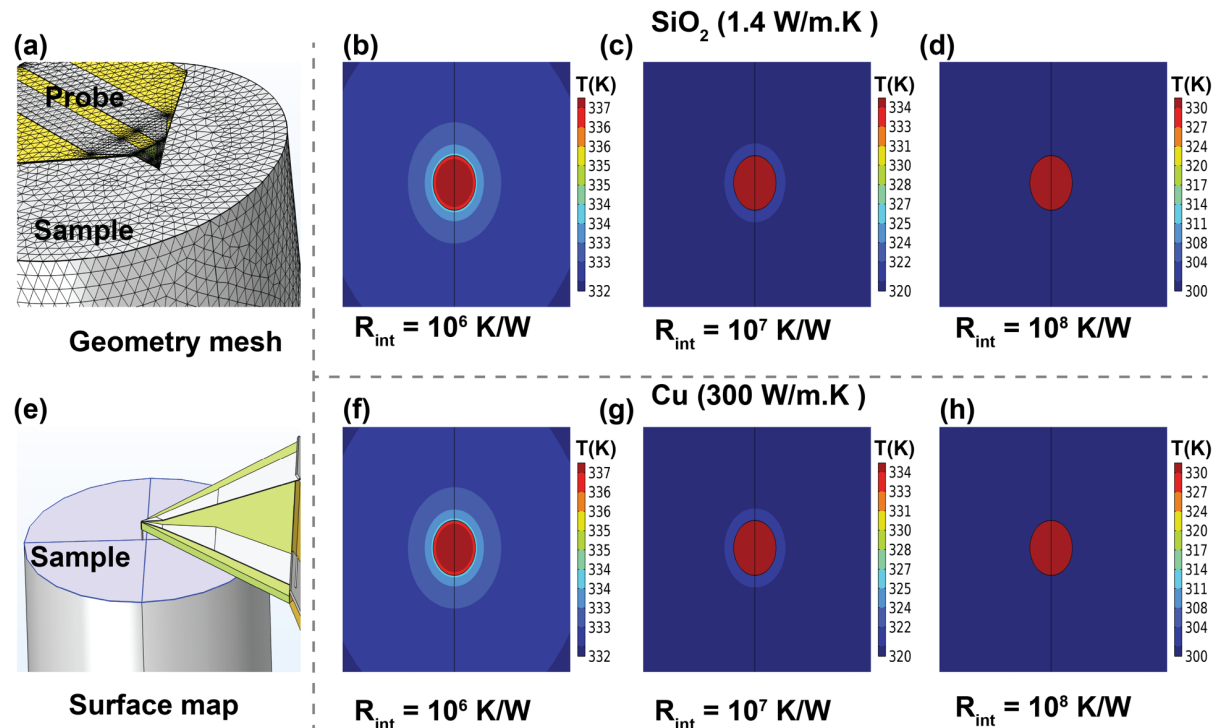


Heat Transport at Tip-Sample Interface

- Probe-sample interface thermal resistance modeling



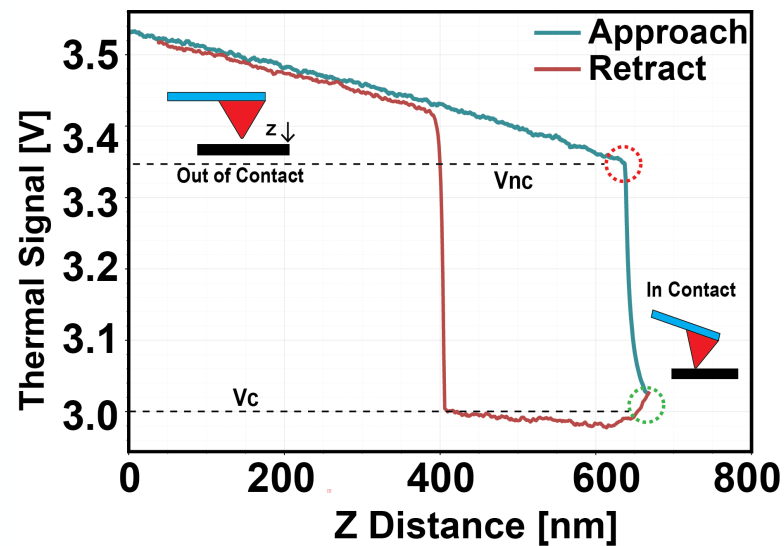
- Probe-sample thermal spreading resistance FEM study



Assumption: for similar roughness sample – same interface thermal resistance

Approach-Retract Curve

- Approach-retract curve obtained with “point & shoot” method



Helps to determine non-contact thermal signal → important for quantitative measurements