Scanning Hot Probe Method for Measuring Seebeck Coefficient and Thermal Conductivity of Novel Nano-Structured Materials and Films

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Outline

• Motivation
• Principle of Method/Data Reduction Strategies
• Current Work/Observations/Results
• Challenges/Future Work
Motivation

- Renewed excitement over thermoelectric materials
- Applications?
Motivation (cont’d)

- \[ ZT = \frac{\sigma S^2}{\kappa} T \]
- Improvements by \( \sigma \uparrow, S \uparrow, \kappa \downarrow \)
- How to decrease \( \kappa \)?
Motivation (cont’d)

• Characterization is challenging!
• Need fine resolution of local characterization
• How?

Scanning Hot Probe!
Principle of Method

• Thermal AFM tip heated via Joule heating.
  – Acts as Heat Source, Temperature Gauge and Seebeck Voltage Probe

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FIG. 1. Schematic diagram of the thermal probe.
Principle of Method (cont’d)

• Temperature rise is taken from the TCR of the probe and change in resistance with change in power.

\[ \Delta T = \frac{\Delta R}{R_0 \times TCR} \]

• Thermal resistance is calculated by change in temperature versus heating power.

\[ R_{exp}^{th} = \frac{\Delta T}{P} \]
Principle of Method (cont’d)

• Thermal measurement typically done in non-contact mode.
  – Only air-sample conduction vs. solid-solid, liquid-solid, and air-solid
Principle of Method (cont’d)

- Examples of experimental data from a thermoelectric sample

\[ Y = 1.58015 \times 10^{-5} + 4.06872 \times 10^{-6} X \]

**Graphs:**
- Thermal and deflection signal of non-contact to contact glass measurement.
- DC sample voltage (µV) vs. temperature rise (K).
- Linear fit for Seebeck voltage gold on glass.
Data Reduction Strategies

• Non-Contact Heat Transfer Models
  – Model heat transfer in air, far from sample to obtain convective coefficient
  – Model with well characterized pure (bulk) samples to obtain the thermal contact resistance and exchange radius
  – Model with sample to obtain sample thermal resistance, which gives thermal conductivity by $R_s = \frac{1}{4k_s b'}$, if sample has bulk-like thickness and sample temperature rise which gives true Seebeck coefficient.
Data Reduction Strategies (cont’d)

- Reference Curve Fitting:
  - Measure several samples with known thermal conductivity and obtain the thermal resistance as a function of distance for each.
  - Fit data; use fitting equation to obtain thermal conductivity of sample.
  - Only works for certain range of thermal conductivity values (0.1<k<10 W/mK).
Analysis of reference samples

- Reference samples analyzed:
  - Nb(k=53W/Km), Ti(k=25W/Km), Steel(k=16W/Km), Macor (k=1.4W/Km), k=0.65W/Km, k=0.49W/Km, PEDOT(k=0.37W/Km)
  - For these reference samples, observe the difference in behavior of the thermal resistance vs. distance between the probe and the surface

*Zooming in on the lower thermal conductivity graphs.*
**Region III**: The lower the thermal conductivity, the more similar the values of the thermal resistance are. Measurement of low thermal conductivities (below 2 W/mK) requires much more attentive detail (room temperature and humidity may significantly affect results).

**Region II**: Large variation from the low thermal conductivity region, and significantly higher thermal resistance than the high thermal conductivity region.

**Region I**: The higher the thermal conductivity of the sample, the trend in thermal resistance again becomes similar.
Observations (cont’d)

* Sharp slopes from Non contact to contact are related mainly with a straightforward heat flow from probe to sample.

* Soft slopes from Non contact to contact are related mainly with a spread heat flow from probe to sample.

Other factors that could have an effect: roughness of sample, angle of probe to surface of sample.
Observations (cont’d)

This agrees with work done by Lefevre, et. al;

* Sharp slopes from Non contact to contact are related mainly with a straightforward heat flow from probe to sample.

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Other factors that could have an effect: roughness of sample, angle of probe to surface of sample

\[
\begin{align*}
\frac{\Delta U^2}{U_i^2} &= \frac{3}{4} \frac{\lambda_s/G_{pt}}{G_{pt}/(\pi b + \lambda_s)}', \\
S = \frac{\partial \Delta U^2}{\partial \lambda_s} \propto \lambda_s^{-2}
\end{align*}
\]

\[
S_b = \frac{\partial \Delta U^2}{\partial b}
\]
Current Work

- Finite Elements Model of heat transfer between probe and surface developed to further explore these observations.
Current Work (cont’d)

• Thermal contact resistance and thermal exchange radius constant for low thermal conductivity values (0.1<k < 2 W/mK). Current work to investigate outside this range.

• Thermal contact resistance and thermal exchange radius taken from intersection of Macor (k=1.46 W/mK) and a bulk sample of k=0.49 W/mK.
Results – Bi$_{2-y}$Sb$_y$Te$_3$ thin-film samples

- Thermal Conductivity obtained this way for several thin film samples

<table>
<thead>
<tr>
<th>Type of Sample</th>
<th>Substrate</th>
<th>Name</th>
<th>Thermal Conductivity (W/mK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bi$_2$Te$_3$</td>
<td>SiO$_2$/100nm gold</td>
<td>OC110303A</td>
<td>1.20E+00</td>
</tr>
<tr>
<td>Bi(2-$y$)Sb$_y$Te$_3$</td>
<td>SiO$_2$/100nm gold</td>
<td>OC120814B</td>
<td>9.22E-01</td>
</tr>
<tr>
<td>Bi(2-$y$)Sb$_y$Te$_3$</td>
<td>glass</td>
<td>OC120814B</td>
<td>5.43E-01</td>
</tr>
</tbody>
</table>
Challenges:

• Probes are fragile
  • if probe is damaged during experiment or transportation, experiments must be started over

• Environmental conditions
  • Variations in room temperature; any vibrations or blowing air affect results

• Sample surface roughness
  • Poor contact makes repeatability difficult
Future Work:

• Develop rigorous probe handling and calibration protocol
• Build in remote digital thermometer and humidity monitor for room condition measurement into experimental automation
• Translate data reduction model to be incorporated into experimental automation
• Integrate nano-scale resolution commercial probes for finer local characterization/mapping
• Develop four probe thermal AFM tip for local electrical conductivity mapping
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