Detailed uncertainty analysis of the ZEM-3 measurement system

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NASA/USRA Contract: 04555-004
Objectives

• Develop an uncertainty analysis for a common resistivity and Seebeck coefficient measurement configuration.
• Introduce a software package which includes the uncertainty analysis calculations.
• Establish measurement best practices to minimize measurement uncertainty.
• Demonstrate typical high temperature uncertainty on a Si/Ge sample.

Potentiometric Configuration (4-probe)

Power Factor Uncertainty
+7% / -25%

ULVAC ZEM-3
Linseis LSR-3
LabVIEW VI

• ZEM-3 system has been developed into a LabVIEW VI. (Independent of ULVAC Technologies)
• Software allows for versatile testing profiles.
• Includes full uncertainty analysis on data.
• Open source makes customization possible.

Custom ZEM-3 Software Available

Open Source LabVIEW VI

Contact: Jon Mackey (jam151@zips.uakron.edu or jonathan.a.mackey@nasa.gov)
General Testing Profiles

V-I and Quasi V-ΔT at Temperature

Open Source LabVIEW VI

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## Resistivity Uncertainty

<table>
<thead>
<tr>
<th>#</th>
<th>Source</th>
<th>Typical Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Thermocouple tip radius</td>
<td>0.25 mm</td>
</tr>
<tr>
<td>2</td>
<td>Thermocouple separation length</td>
<td>±0.1 mm</td>
</tr>
<tr>
<td>3</td>
<td>Sample uniformity</td>
<td>±0.1 mm/cm</td>
</tr>
<tr>
<td>4</td>
<td>Caliper uncertainty</td>
<td>±0.01 mm</td>
</tr>
<tr>
<td>5</td>
<td>Statistical variation</td>
<td>Calculated</td>
</tr>
<tr>
<td>6</td>
<td>Wire discrepancy</td>
<td>Calculated</td>
</tr>
<tr>
<td>7</td>
<td>DAQ voltage uncertainty</td>
<td>50 ppm +1.2 μV</td>
</tr>
<tr>
<td>8</td>
<td>DAQ current uncertainty</td>
<td>0.2% +0.3 mA</td>
</tr>
</tbody>
</table>

## Sample Uniformity

![Sample Uniformity Image]

## Typical Thermocouple Beads

- **0.5mm**
  - Source: ULVAC

- **1.1mm**
  - Source: Cleveland Electric

- **0.7mm**
  - Source: ULVAC

- **0.7mm**
  - Source: Cleveland Electric
## Seebeck Uncertainty Sources

<table>
<thead>
<tr>
<th>#</th>
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<th>Typical Values</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Cold-finger effect</td>
<td>10,000 W/(m²K)</td>
</tr>
<tr>
<td>2</td>
<td>Wire Seebeck variation</td>
<td>±5%</td>
</tr>
<tr>
<td>3</td>
<td>Absolute temperature</td>
<td>±2°C</td>
</tr>
<tr>
<td>4</td>
<td>Statistical variation</td>
<td>Calculated</td>
</tr>
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### Cold-finger Effect

**Close-up View**

- Upper Probe
- Lower Probe

**22% Difference**

- Actual $\Delta T$: 1.33 K
- Measured $\Delta T$: 1.04 K

### Wire Seebeck

- ±2°C
- ±5%
Resistivity Calculation

\[ \rho = \frac{\sum z_i \sum y_i - N \sum z_i y_i wD}{(\sum z_i)^2 - N \sum z_i^2} \frac{wD}{L} \]

\( y \)- probe to probe voltage
\( z \)- test electrical current
\( w/D \)- sample width/depth
\( L \)- probe separation length
\( N \)- sample size

Seebeck Calculation

\[ S = -\frac{\sum x_i \sum y_i - N \sum x_i y_i}{(\sum x_i)^2 - N \sum x_i^2} + S_{wire} (T) \]

\( x \)- probe \( \Delta T \)
\( y \)- probe to probe voltage
\( S_{wire} \)- wire Seebeck coefficient
\( T \)- sample temperature
\( N \)- sample size

Error Propagation

\[ U_y = f(\bar{x}) \]

\[ \bar{y} \pm U_y = f(\bar{x} \pm U_x) \approx f(\bar{x}) \pm \frac{df}{dx}\bigg|_{x=\bar{x}} U_x \]

Uncertainty can be calculated from a Taylor Series expansion around the nominal measurement value.
### FEA Model Parameters

- **Autodesk Simulation Multiphysics**
  - Non-linear iterative thermal solver
- **Thermal domain:**
  - Sample **4x4x18mm** rectangular prism
  - Thermal conductivity **4 W/(m²K)**
  - Probes Ø0.5mm x 150mm
    - Thermal conductivity **30 W/(m²K)**
- **Boundary conditions:**
  - Sample ends fixed temperatures
  - Probe ends fixed temperatures
  - Remaining faces radiation coupled
    - $\varepsilon=0.2, 0.5, 0.7, 0.9$ (40% change)
- **Parameters of study:**
  - Furnace temp=200, 600, 1000°C
  - Differential temp=0.1 to 14°C
  - Thermal conductance= **100,000**, **33,000**, **10,000 W/(m²K)** (600% change)

### Grid Independence Study

- Two meshes were generated from primarily brick elements
- **Course mesh** (shown above)
  - 41,000 elements
- **Fine mesh**
  - 55,000 elements
- **Mesh agreement** <0.2% change in results
**Temperature Contour**

- $T_1/T_2$ represent “actual” and “measured” temperatures
- Model fits experiment well at high temperature

**FEA Uncertainty Results**

- Uncertainty is defined as the difference between the desired and measured temperature difference.
- Uncertainty increases with furnace temperature and delta temperature.
Testing Samples & Profile

- Si$_{80}$Ge$_{20}$ samples prepared by milling and spark plasma sintering elemental powders
  - 2at% P doped
  - Ø 1” pucks machined to 4x4x18mm
- Samples measured from 25 to 950°C
- Equilibrium definition:
  - Furnace <5% change in 120 seconds
  - Isothermal <0.1°C in 120 seconds
- Resistivity measurement:
  - -50 to +50 mA increment 5mA
- Seebeck measurement:
  - +1°C/min up to 10°C
Resistivity Uncertainty

- Resistivity uncertainty is fairly temperature independent, due to geometric nature.
- Thermocouple tip radius dominates uncertainty.

Seebeck Uncertainty

- Seebeck uncertainty is highly temperature dependent.
- Cold-finger effect dominates at all temperatures, and is asymmetric.
Overall Results

- Resistivity uncertainty is ±7.0% at all temperatures.
- Absolute Seebeck uncertainty ranges from ±1.0% at room temperature to +1.0%/-13.1% at high temperature.

Power Factor Results

- Power factor uncertainty ranges from ±7.5% at room temperature to +7.3%/-25.0% percent at high temperature.
- These values all assume the conservative parameter values listed.
**Conclusion**

- LabVIEW VI is available to operate the ZEM-3 and calculate the uncertainty.
- Resistivity uncertainty is primarily geometric, and can be reduced with careful preparation.
- Seebeck uncertainty is primarily due to the cold-finger effect, and can be reduced with good thermal contact.
- Power factor uncertainty is ±7.5% at room temperature and +7.3%/-25.0% percent at high temperature.

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JPL

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Appendix
Error Propagation

\[ y = f(x) \]

\[ \bar{y} = f(\bar{x}) \]

\[ \frac{\partial y}{\partial x} \bigg|_{x=\bar{x}} \]

\[ y \pm U_y = f(\bar{x} \pm U_x) \approx f(\bar{x}) \pm \frac{df}{dx} \bigg|_{x=\bar{x}} U_x \]

\[ e_{yx} = \frac{1}{\bar{y}} \frac{\partial y}{\partial x} \bigg|_{x=\bar{x}} U_x \]

Statistical Uncertainty

\[ U_{Stat} = t_{v,95\%} \sqrt{\frac{N \sum (y_i - y_c(z_i))^2}{\nu(N \sum z_i^2 - (\sum z_i)^2)}} \]
### FEA Verification

<table>
<thead>
<tr>
<th>Temperature Celsius</th>
<th>Model $\Delta T_{\text{Probe}}/\Delta T$</th>
<th>Experiment $\Delta T_{\text{Probe}}/\Delta T$</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>0.33</td>
<td>0.18</td>
</tr>
<tr>
<td>600</td>
<td>0.17</td>
<td>0.10</td>
</tr>
<tr>
<td>1000</td>
<td>0.07</td>
<td>0.06</td>
</tr>
</tbody>
</table>

• $T_1$ desired temperature
• $T_2$ measured temperature
• accounts for thermal contact conductance

### Temperature Contour

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Influence of Thermal Contact Conductance

- Thermal contact conductance plays a significant role in the Cold-finger effect and displays a power law dependence.