Detailed uncertainty analysis of the ZEM-3 measurement system

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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

- **Source:** ULVAC
- **Dimensions:**
  - 0.5mm
  - 1.1mm
- **Source:** Cleveland Electric

![Thermocouple Beads Images]
### Seebeck Uncertainty Sources

<table>
<thead>
<tr>
<th>#</th>
<th>Source</th>
<th>Typical Values</th>
</tr>
</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>
<tr>
<td>5</td>
<td>Wire discrepancy</td>
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</table>

#### Wire Seebeck

- ±2°C
- ±5%

#### Cold-finger Effect

- Upper Probe
- Lower Probe

#### Close-up View

- 22% Difference

![Graph showing temperature difference between T1 and T2](image_url)

- Actual ΔT 1.33 K
- Measured ΔT 1.04 K

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ZEM-3 Uncertainty Analysis
**Resistivity Calculation**

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

- \( y \) - 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**

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

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

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
    - ε=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

- T1/T2 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

- $\text{Si}_{80}\text{Ge}_{20}$ 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

Example Measurement Data

- Graph showing probe to probe voltage vs. sample current for different temperatures.
- Graph showing probe to probe voltage vs. temperature difference for different temperatures.
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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04555-004
Appendix
**Error Propagation**

\[ y = f(x) \]

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

\[
\begin{align*}
\bar{y} \pm U_y &= f(\bar{x} \pm U_x) \\
&\approx f(\bar{x}) \pm \frac{df}{dx}_{x=\bar{x}} U_x
\end{align*}
\]

\[
\begin{align*}
e_{y_x} &= \frac{1}{\bar{y}} \frac{dy}{dx}_{x=\bar{x}} U_x \\
e_{Total} &= \sqrt{e_{y_1}^2 + e_{y_2}^2 + e_{y_3}^2 + \ldots}
\end{align*}
\]

---

**Resistivity and Seebeck**

\[
\rho = \frac{\sum z_i \sum y_i - N \sum z_i y_i wD}{(\sum z_i)^2 - N \sum z_i^2} \frac{1}{L}
\]

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

x- probe temperature difference  
y- probe voltage  
z- electrical current  
N- sample size

---

**Statistical Uncertainty**

\[
U_{Stat} = t_{v, 0.95} \sqrt{\frac{N \sum (y_i - y_c(z_i))^2}{v(N \sum z_i^2 - (\sum z_i)^2)}}
\]
### FEA Verification

#### Temperature Contour

- T1 desired temperature
- T2 measured temperature
- accounts for thermal contact conductance

### Table

<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>
**FEA Uncertainty Results**

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

**Influence of Thermal Contact Conductance**

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

![Graph showing the relationship between furnace temperature and delta temperature](image)

Furnace Temperature 1000°C
Delta Temperature 14°C