Detailed uncertainty analysis of the ZEM-3 measurement system

Jon Mackey
Mechanical Engineering,
University of Akron

Alp Sehirlioglu
Materials Science and Engineering,
Case Western Reserve University

Fred Dynys
NASA Glenn Research Center

NASA Cooperative Agreement: NNX08AB43A
NASA/USRA Contract: 04555-004

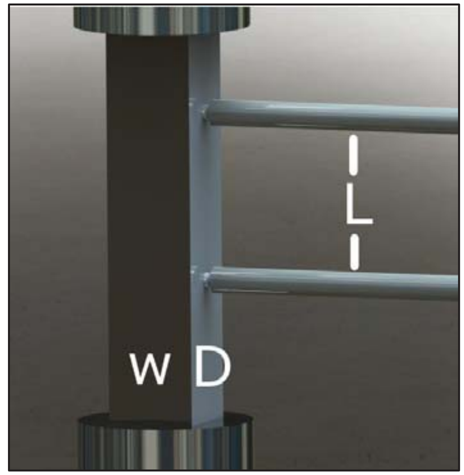
The University of Akron

Case Western Reserve University EST. 1826
think beyond the possible

NASA

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%

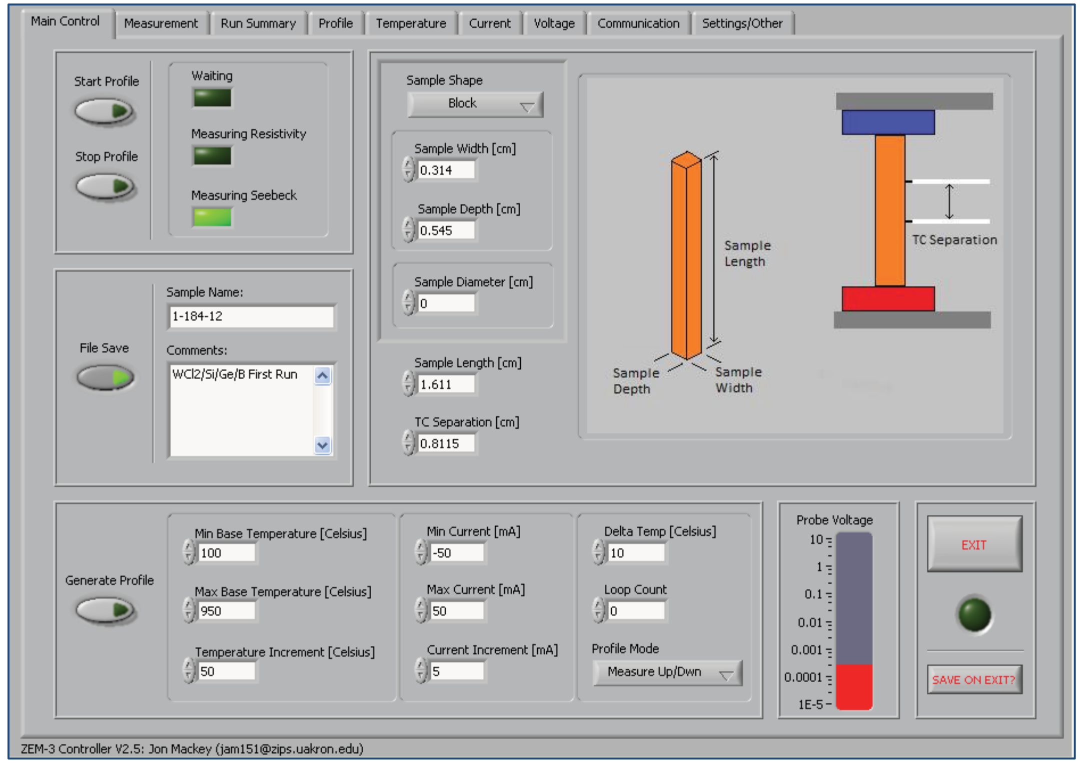
ULVAC ZEM-3

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.

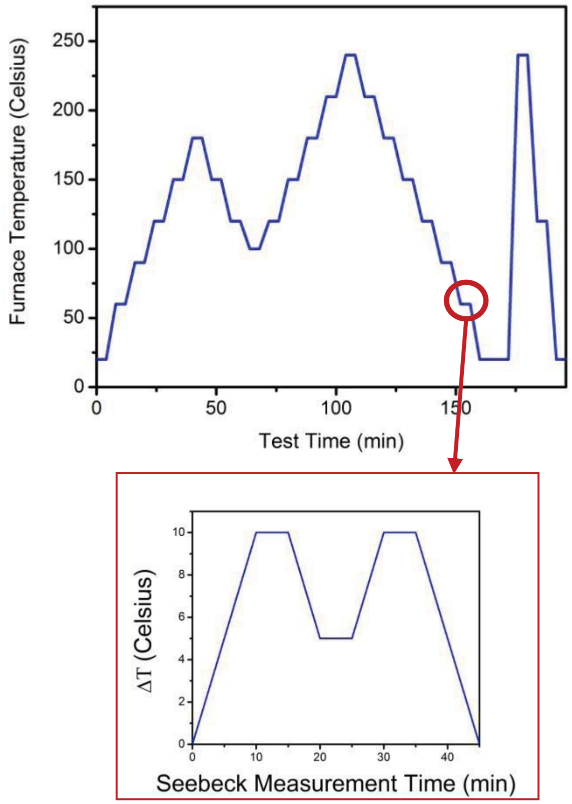
Custom ZEM-3 Software Available



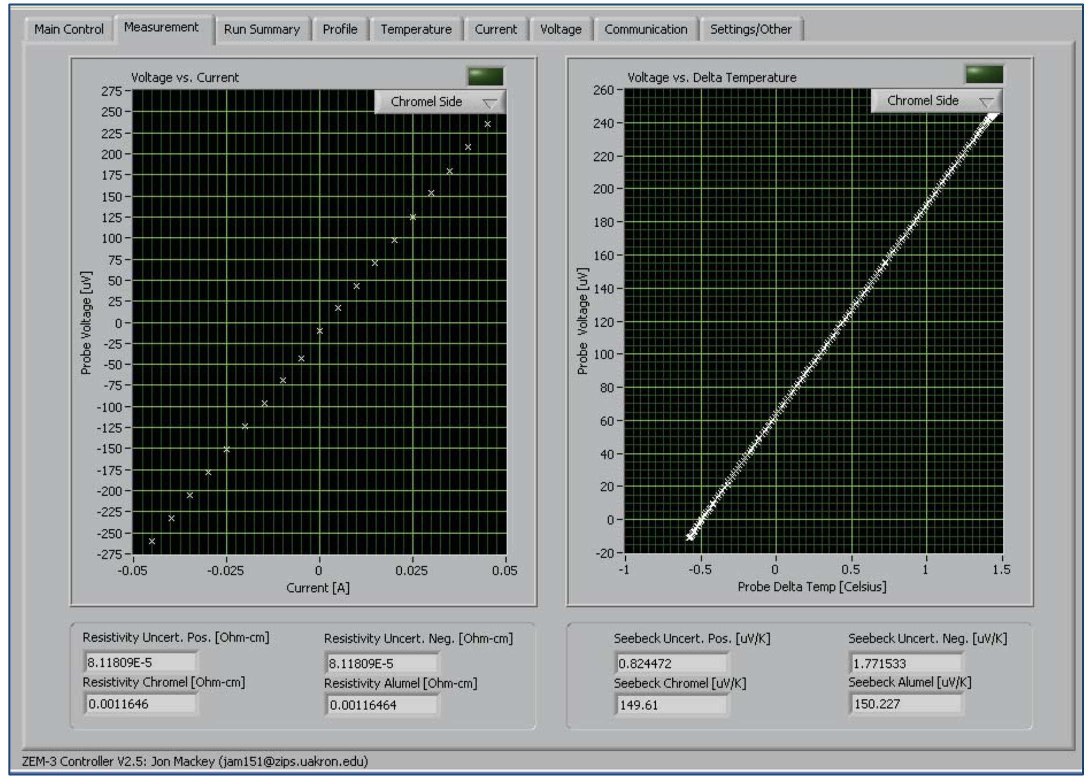
Open Source LabVIEW VI

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

General Testing Profiles



V-I and Quasi V-ΔT at Temperature



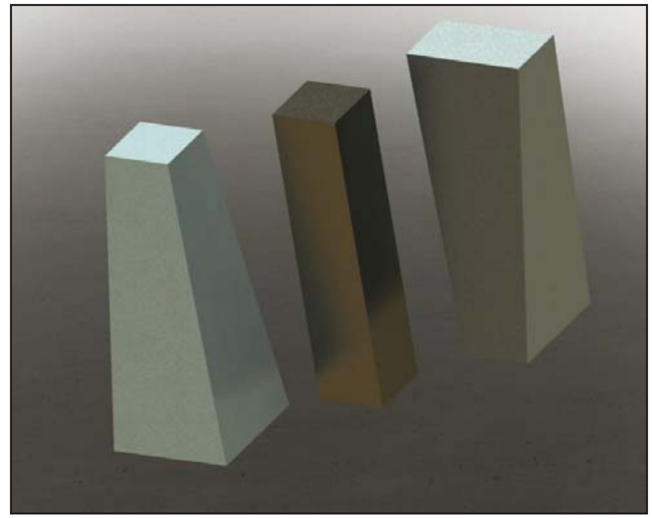
Open Source LabVIEW VI

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

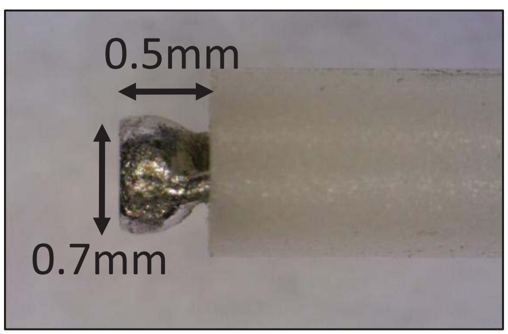
Resistivity Uncertainty

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

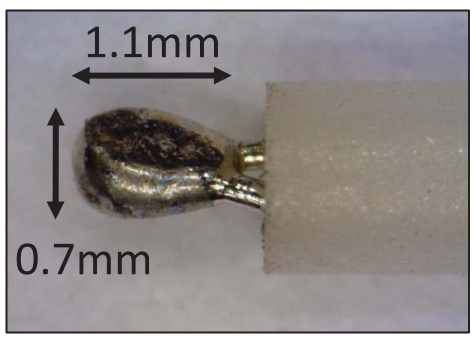
Sample Uniformity



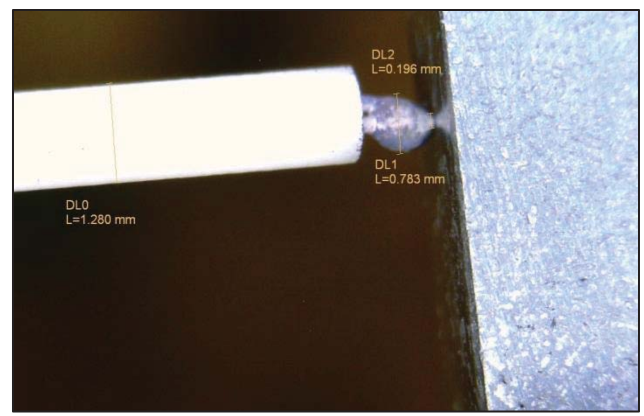
Typical Thermocouple Beads



Source: ULVAC



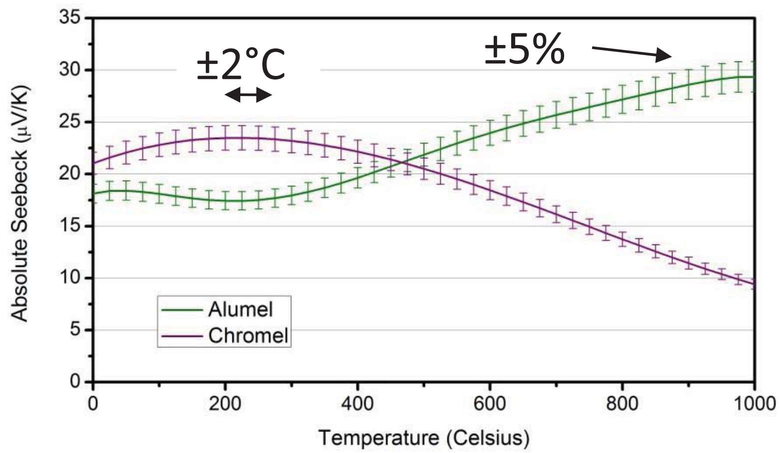
Source: Cleveland Electric



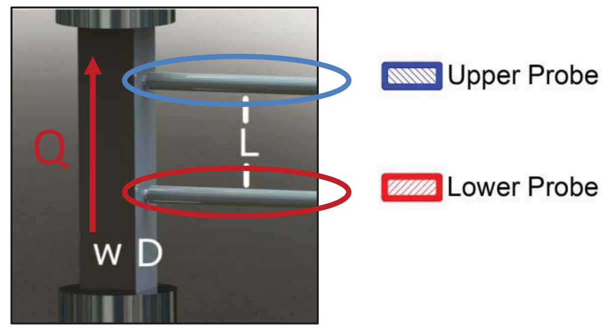
Seebeck Uncertainty Sources

#	Source	Typical Values
1	Cold-finger effect	10,000 W/(m^{2}K)
2	Wire Seebeck variation	±5%
3	Absolute temperature	± 2°C
4	Statistical variation	Calculated
5	Wire discrepancy	Calculated
6	DAQ voltage uncert.	50 ppm + 1.2 μV
7	DAQ temperature uncert.	50 ppm + 1.2 μV

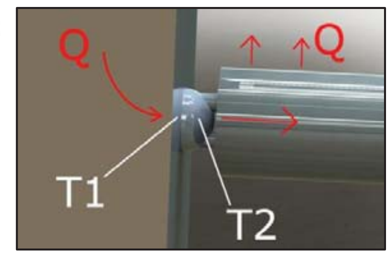
Wire Seebeck

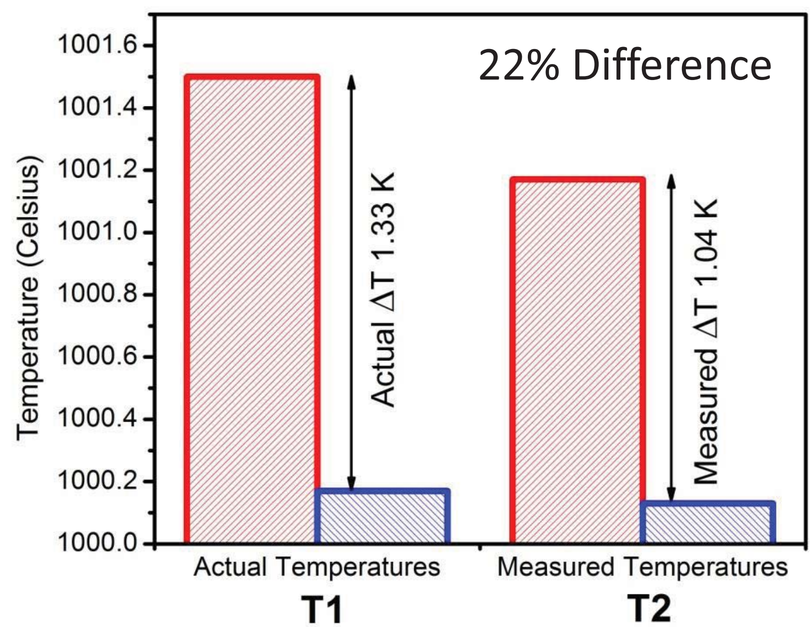


Cold-finger Effect



Close-up View





Resistivity Calculation

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

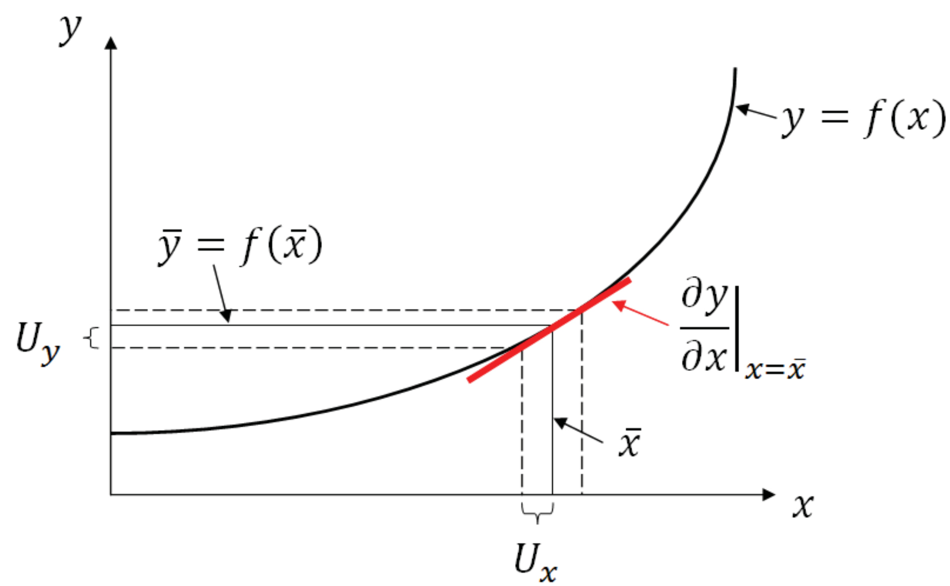
y- probe to probe voltage
z- test electrical current
w/D- sample width/depth
L- probe separation length
N- sample size

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 ΔT
y- probe to probe voltage
S_{wire}- wire Seebeck coefficient
T- sample temperature
N- sample size

Error Propagation



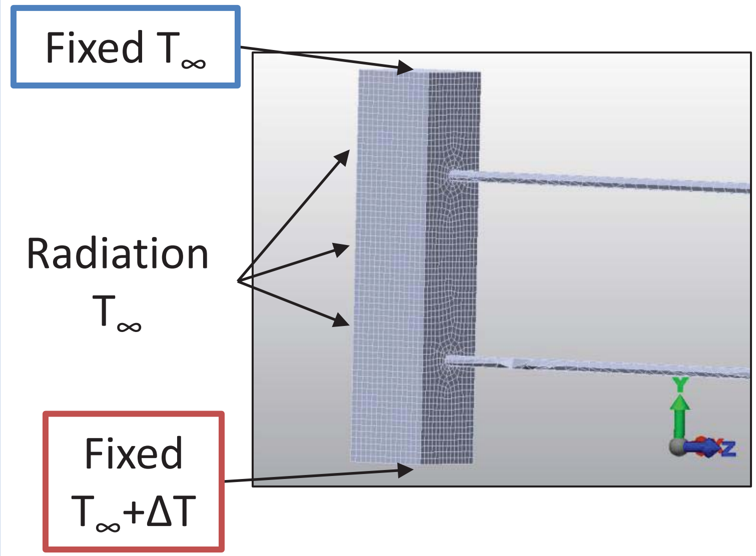
$$\bar{y} \pm U_y = f(\bar{x} \pm U_x) \approx f(\bar{x}) \pm \left.\frac{df}{dx}\right|_{x=\bar{x}} U_x$$

Uncertainty can be calculated from a Taylor Series expansion around the nominal measurement value

FEA Model Parameters

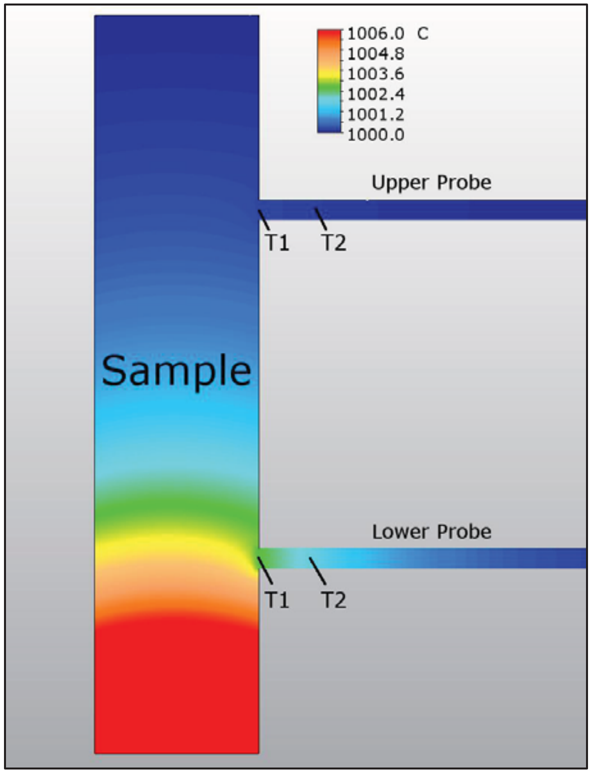
- Autodesk Simulation Multiphysics
  - Non-linear iterative thermal solver
- Thermal domain:
  - Sample 4x4x18mm rectangular prism
    - Thermal conductivity 4 W/(m^{2}K)
  - Probes Ø0.5mm x 150mm
    - Thermal conductivity 30 W/(m^{2}K)
- Boundary conditions:
  - Sample ends fixed temperatures
  - Probe 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/(m^{2}K) (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

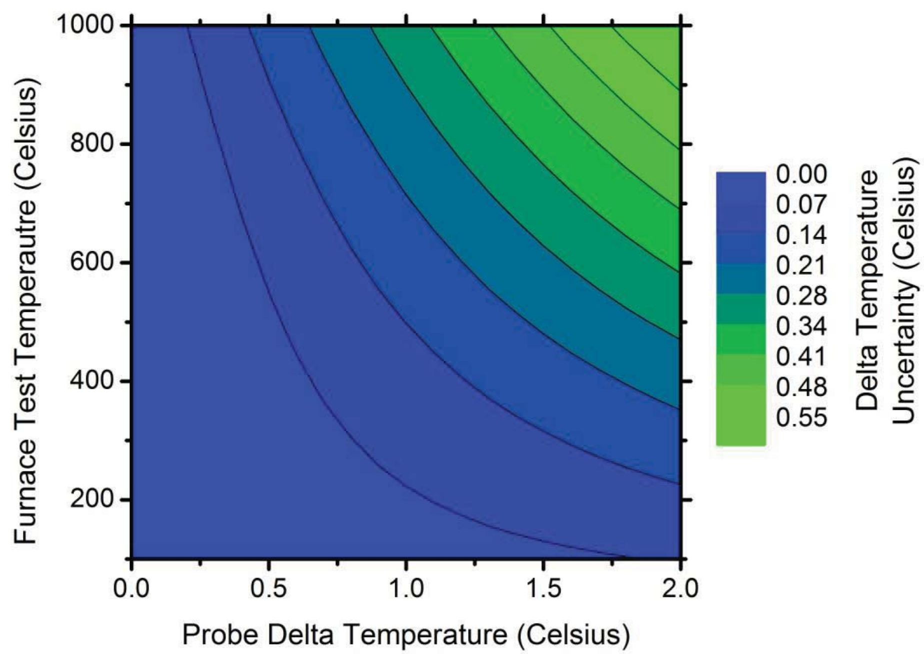
Temperature Contour



- T1/T2 represent "actual" and "measured" temperatures
- Model fits experiment well at high temperature

FEA Uncertainty Results

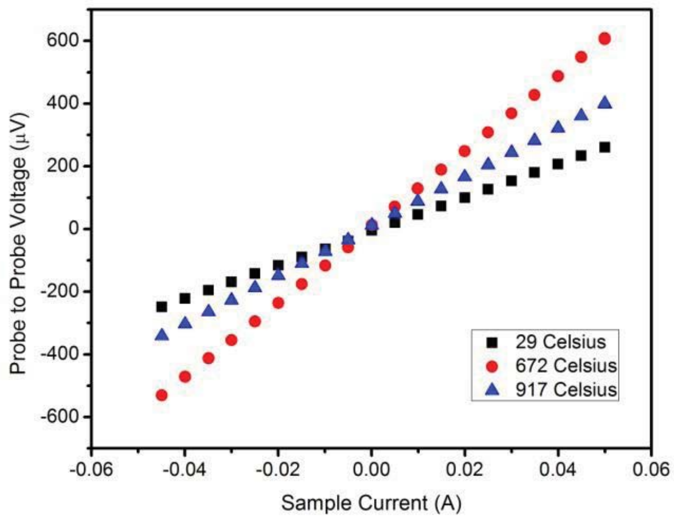
- Uncertainty is defined as the difference between the desired and measured temperature difference.
- Uncertainty increases with furnace temperature and delta temperature.

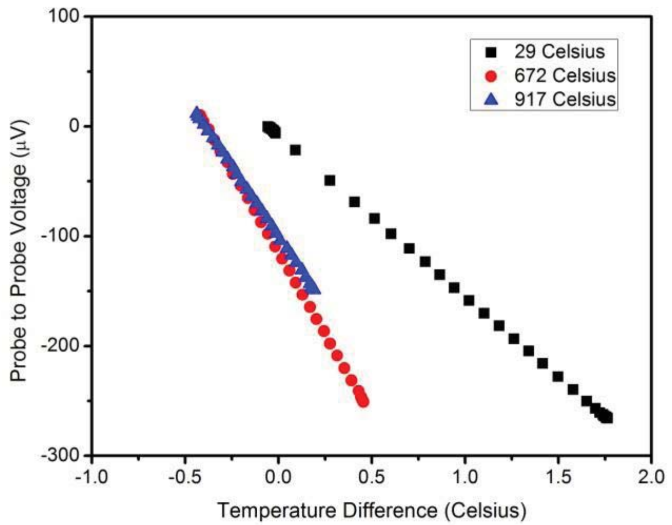


Testing Samples & Profile

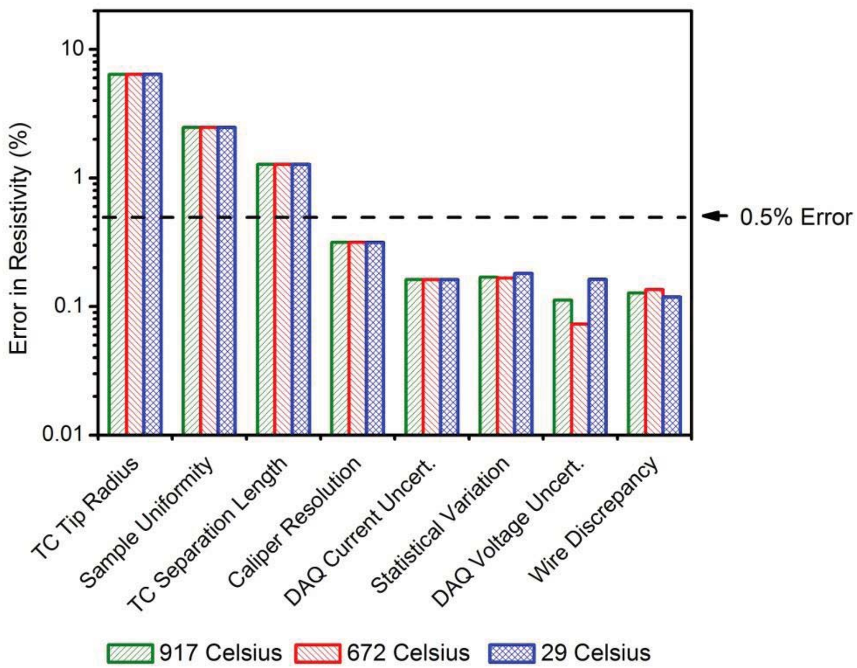
- $Si_{80}Ge_{20}$ samples prepared by milling and spark plasma sintering elemental powders
  - 2at% P doped
  - Ø 1" pucks machined to 4x4x18mm
- Samples measured from 25 to 950°C
- Equilibrium definition:
  - Furnace <5% change in 120 seconds
  - Isothermal <0.1°C in 120 seconds
- Resistivity measurement: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

Example Measurement Data



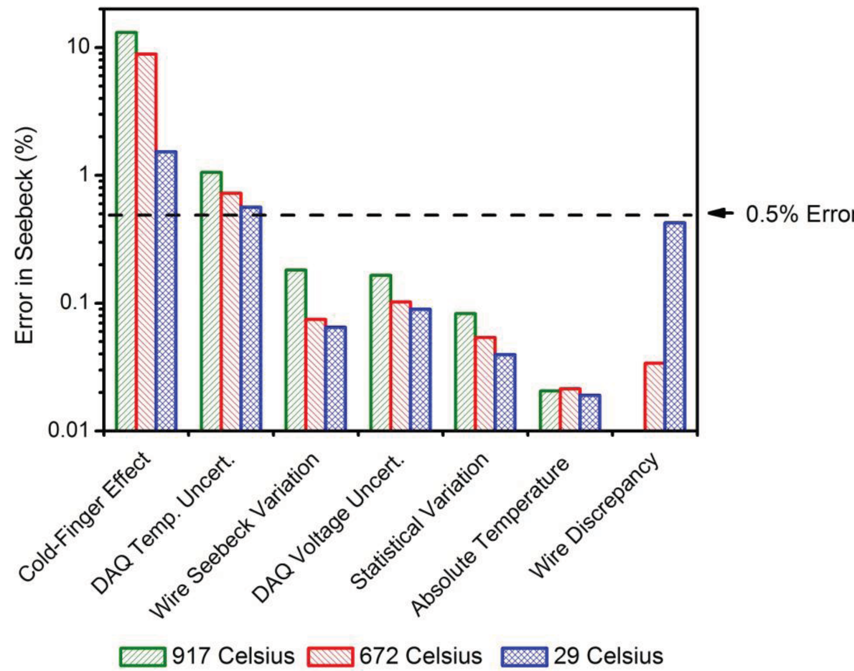


Resistivity Uncertainty



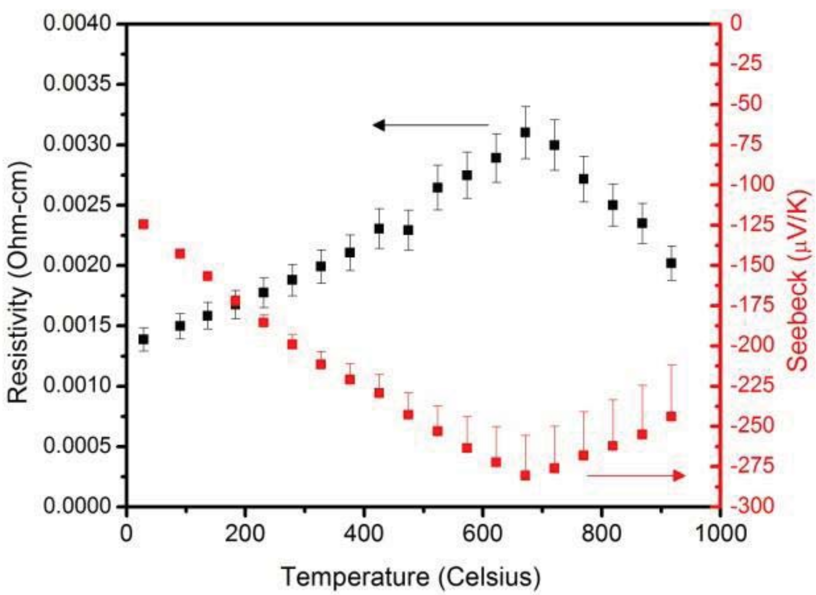
- Resistivity uncertainty is fairly temperature independent, due to geometric nature.
- Thermocouple tip radius dominates uncertainty.

Seebeck Uncertainty



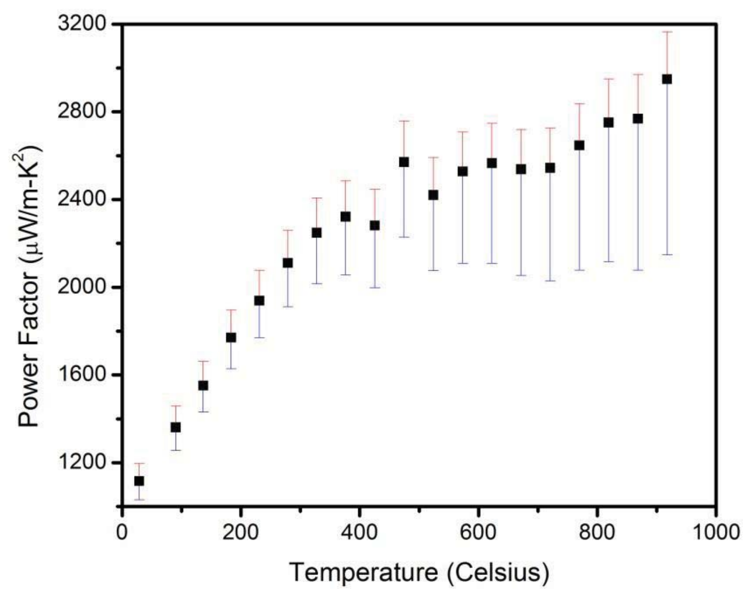
- Seebeck uncertainty is highly temperature dependent.
- Cold-finger effect dominates at all temperatures, and is asymmetric.

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.

Acknowledgements

Tom Sabo, Ray Babuder, Ben Kowalski, Clayton Cross

NASA Glenn Research Center

Dr. Sabah Bux, Dr. Jean-Pierre Fleurial

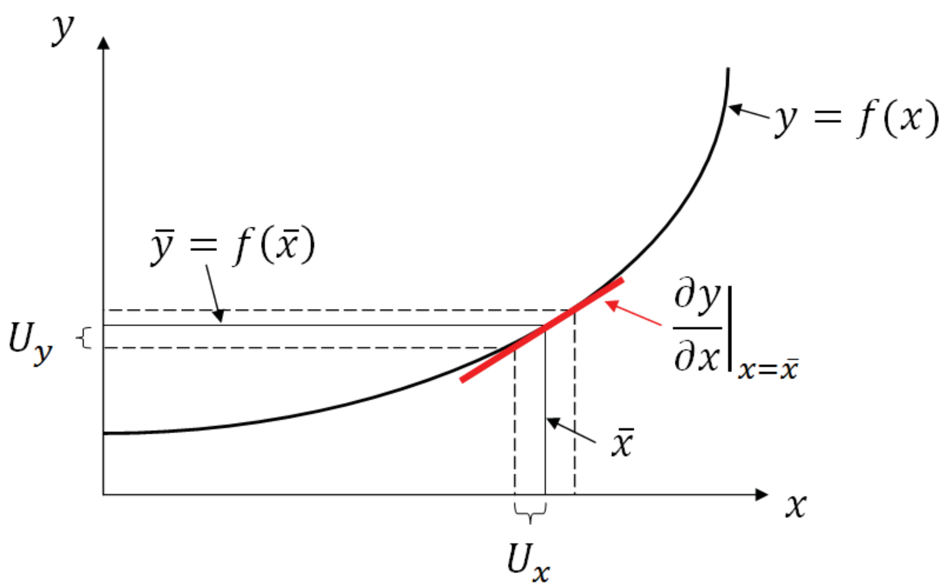
JPL

NASA Cooperative Agreement: NNX08AB43A

NASA/USRA Contract: 04555-004

Appendix

Error Propagation



$$\bar{y} \pm U_y = f(\bar{x} \pm U_x) \approx f(\bar{x}) \pm \left.\frac{df}{dx}\right|_{x=\bar{x}} U_x$$

$$e_{y_x} = \frac{1}{\bar{y}} \left.\frac{\partial y}{\partial x}\right|_{x=\bar{x}} U_x$$

$$e_{Total} = \sqrt{{e_{y_1}}^2 + {e_{y_2}}^2 + {e_{y_3}}^2 + \cdots}$$

Resistivity and Seebeck

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

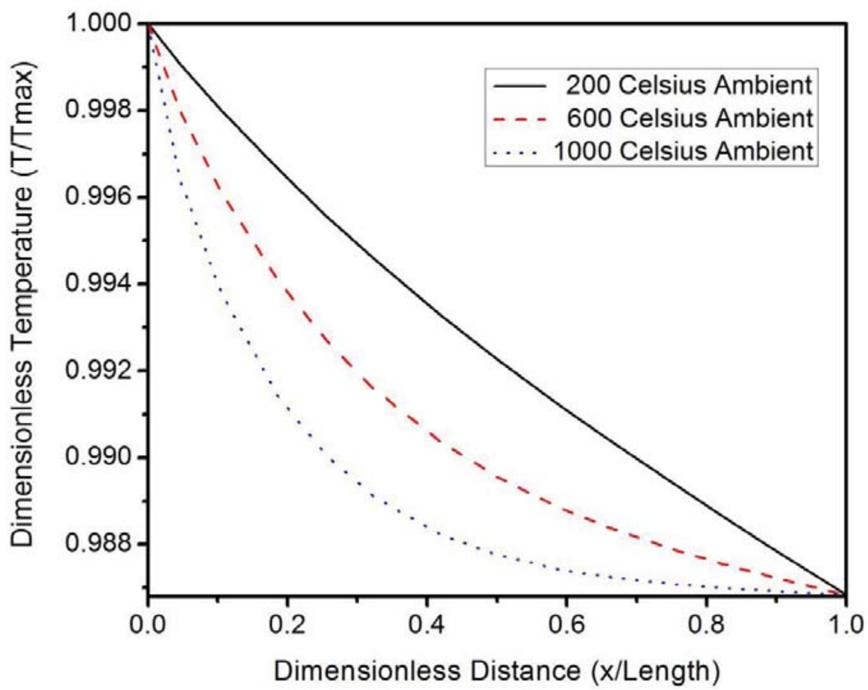
$$S = -\frac{\sum x_i \sum y_i - N \sum x_i y_i}{(\sum x_i)^2 - N \sum {x_i}^2} + S_{Wire}(T)$$

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

Statistical Uncertainty

$$U_{Stat} = t_{\nu,95\%} \sqrt{\frac{N \sum \left(y_i - y_c(z_i)\right)^2}{\nu\left(N \sum {z_i}^2 - (\sum z_i)^2\right)}}$$

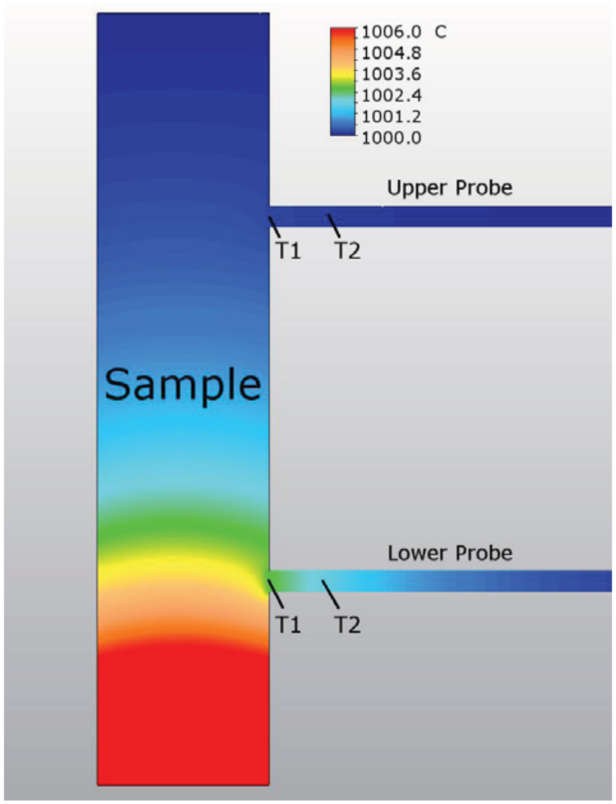
FEA Verification



Temperature Celsius	Model $\Delta T_{Probe}/\Delta T$	Experiment $\Delta T_{Probe}/\Delta T$
200	0.33	0.18
600	0.17	0.10
1000	0.07	0.06

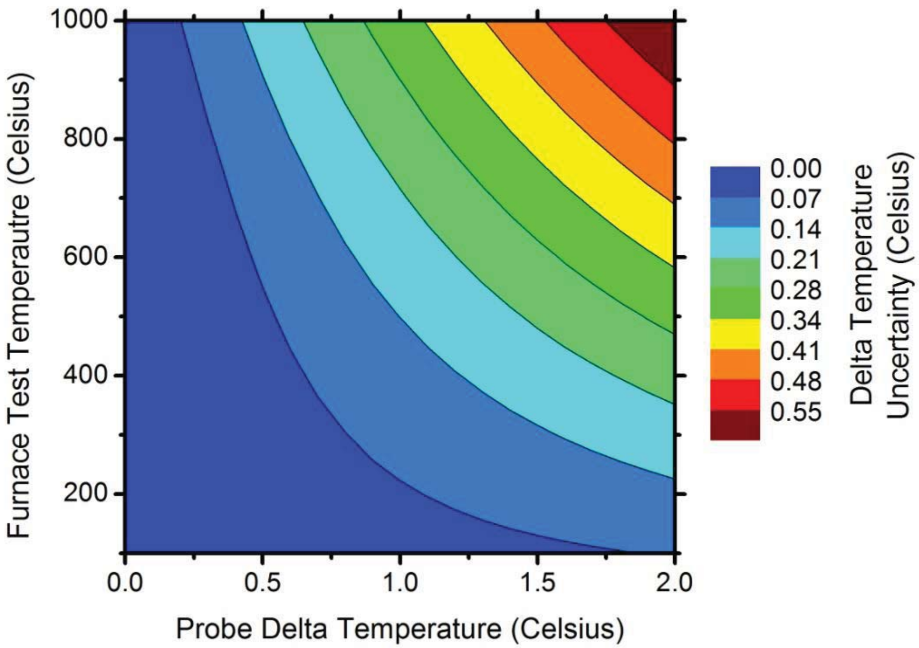
Temperature Contour

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



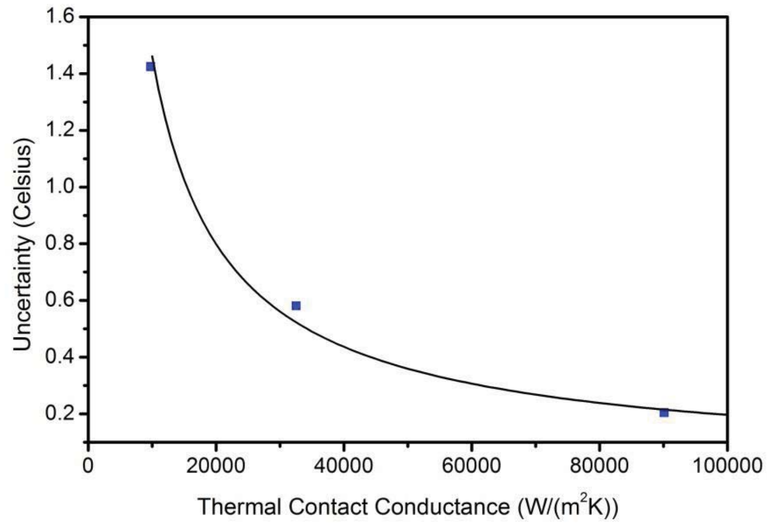
FEA Uncertainty Results

- 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.



Furnace Temperature 1000°C
Delta Temperature 14°C