System for the simultaneous Harman based measurement of all thermoelectric parameters from 240 K to 720 K with novel calibration procedure

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ICT2014, Nashville, TN, USA

Topics

1. Introduction
2. ZT-Scanner and Bipolar Transient Harman Measurement
3. Influence of the parasitic thermal phenomena on the *ZT* and λ measurement
4. Novel Two Sample System Calibration (2SSC)
5. ZT-Scanner application on different TE materials
6. Accuracy and precision of measurement
7. Conclusions

Introduction

"The inherent difficulty in thermoelectrics is that direct efficiency measurements require nearly as much complexity as building an entire device"*.

$$ZT = T\frac{\alpha^2}{\rho\lambda}$$

* G. JEFFREY SNYDER AND ERIC S. TOBERER
Materials Science, California Institute of Technology,
nature materials | VOL 7 | FEBRUARY 2008

Separate measurements on different samples

ULVAC ZEM-3

$\alpha, \ \rho$

NETZSCH DSC

λ

NETZSCH FLA

The uncertainty in *ZT* * 50%

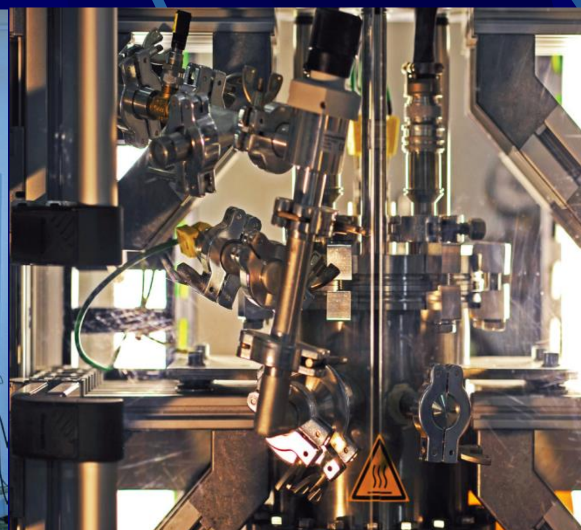
The uncertainty in *ZT* ** ± 20%

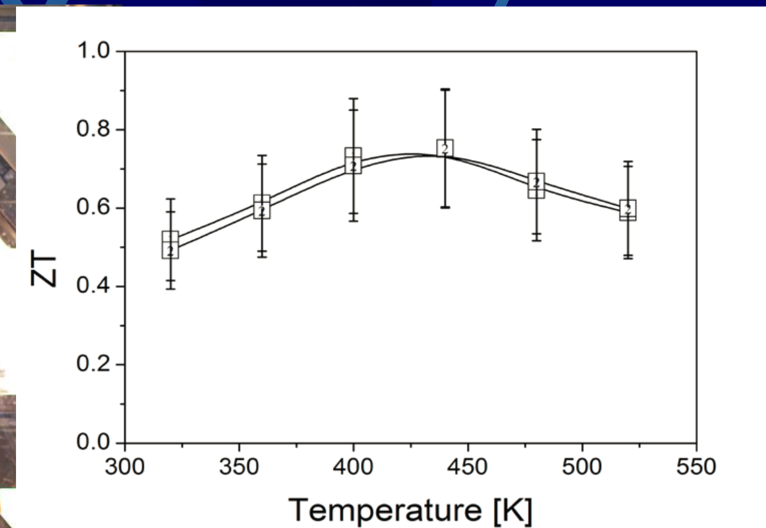
* G. JEFFREY SNYDER AND ERIC S. TOBERER nature materials | VOL 7 | FEBRUARY (2008)

H. WANG, W.D. PORTER, H. BOTNER, J. KÖNIG at al, *J. Electr. Mater.*, **42, 1073 (2013)

ZT-measurement on the same sample

Fraunhofer Institute for Physical Measurement Technique IPM



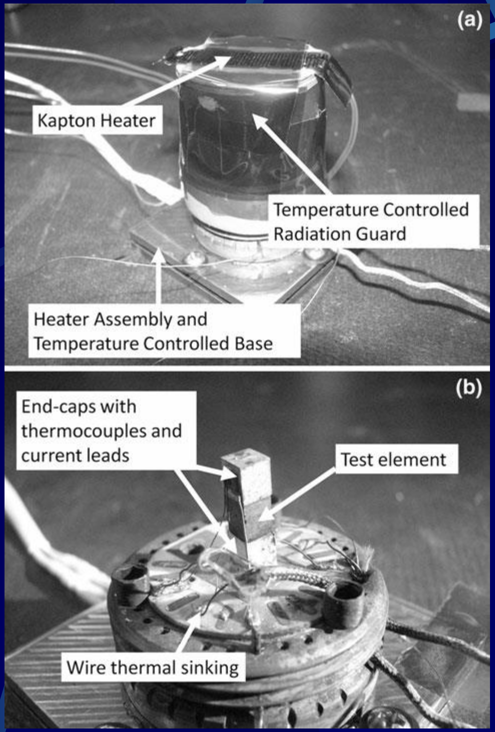


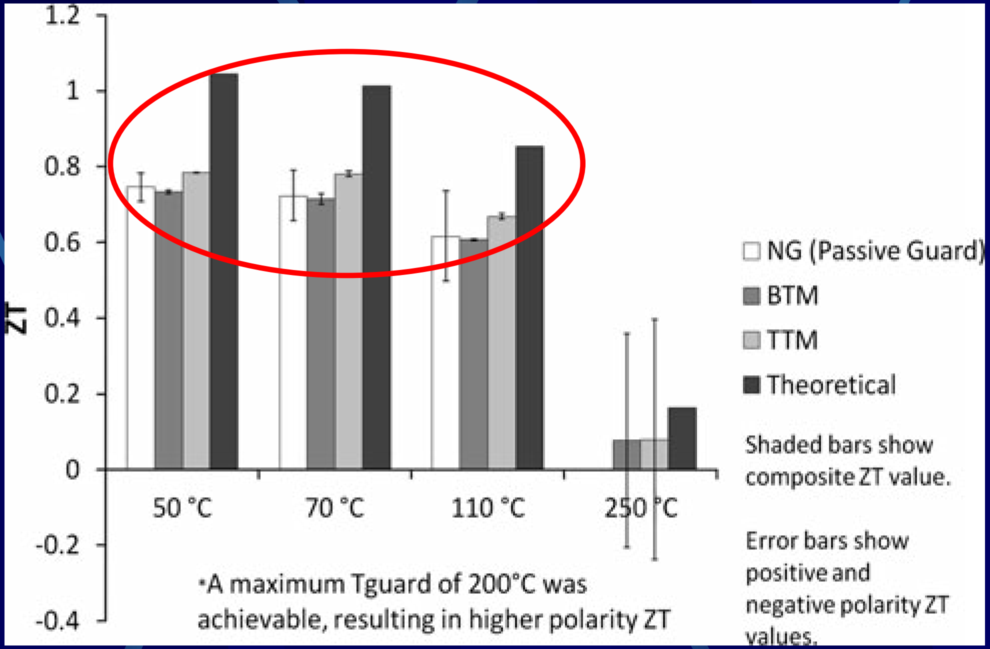
IPM-ZT-Meter-870K

Measurement accuracy

α	$< \pm 5\%$
ρ	$< \pm 10\%$
λ	$< \pm 10\%$
ZT	$\pm 25\%$

Bipolar Transient Harman Measurement



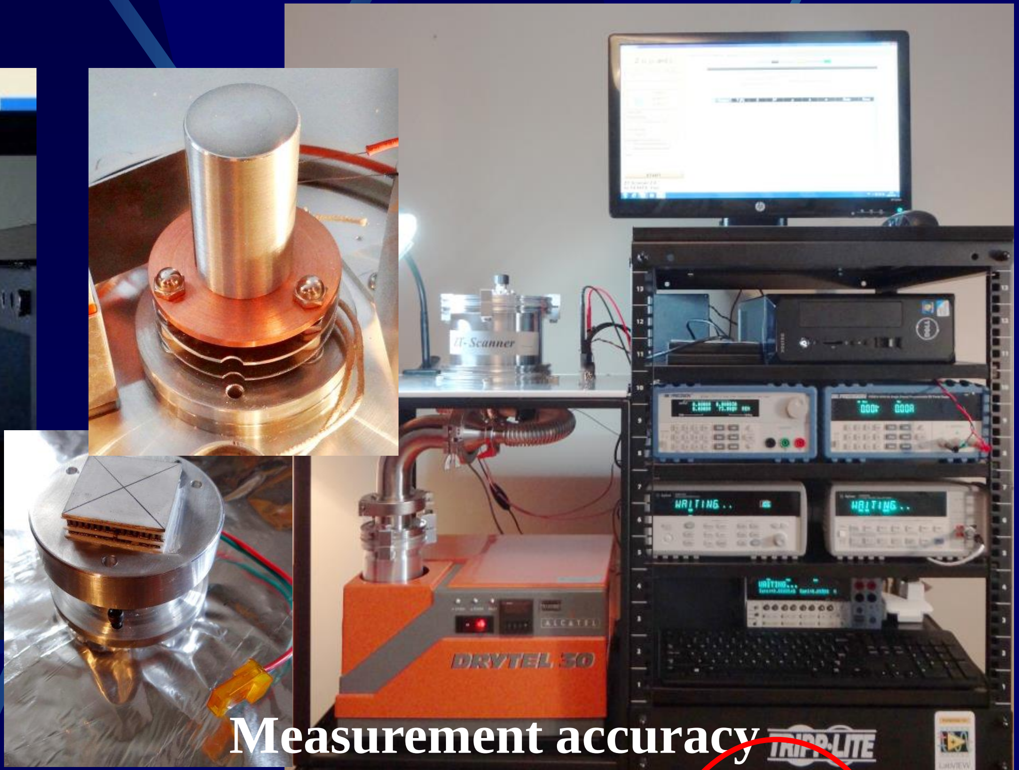


Marlow test setup

From 220 K to 525 K

R.MCCARTY, J.THOMPSON, J.SHARP, A.THOMPSON
Journal of ELECTRONIC MATERIALS,
Vol. 41, No. 6, (2012)

Bipolar Transient Harman Measurement



ZT-Scanner by TEMTE Inc.

From 240 K to 720 K

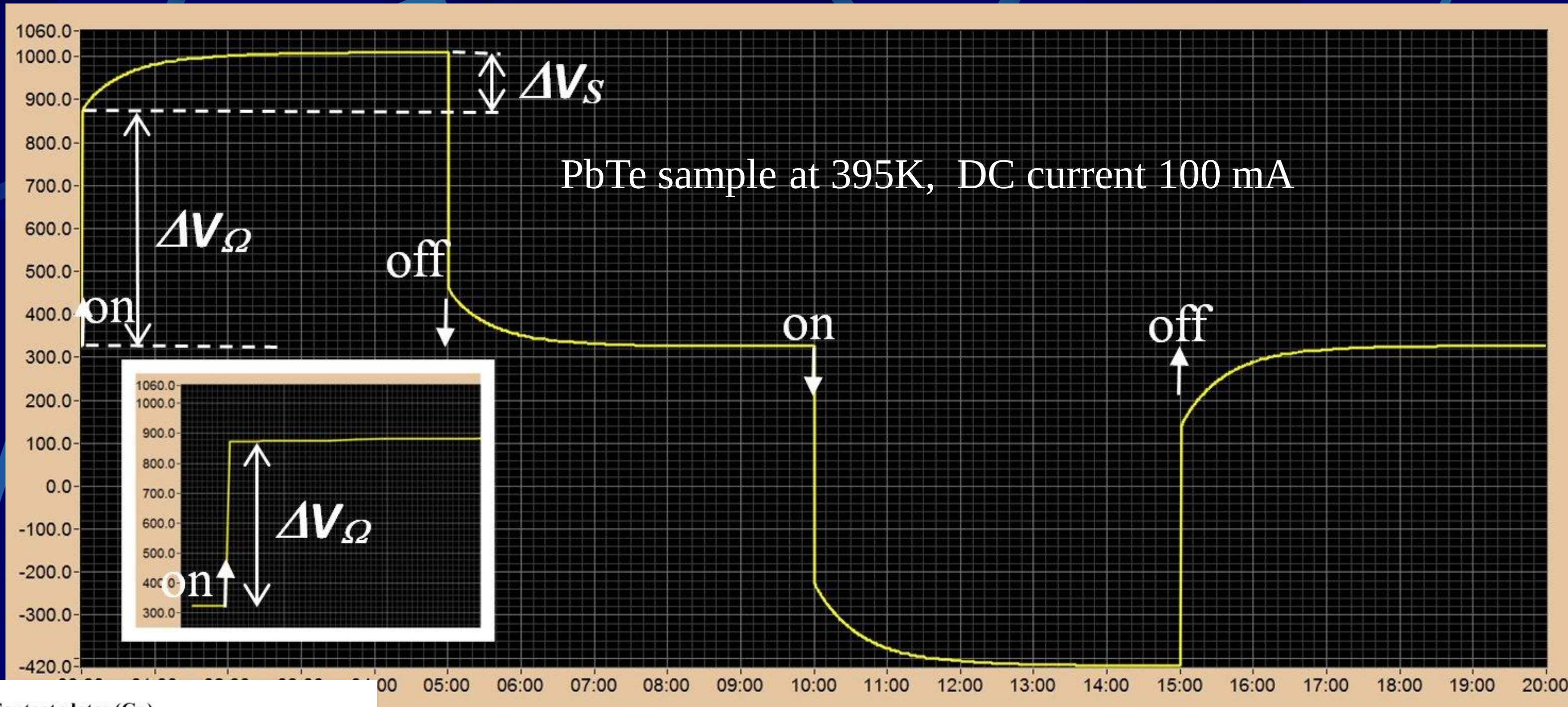
Measurement accuracy

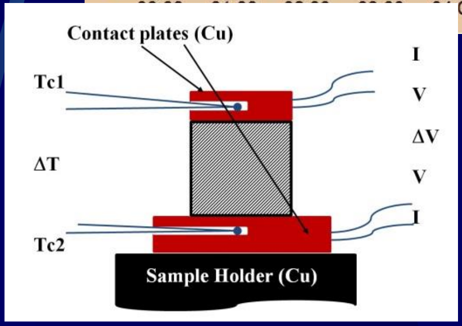
Ambitious !? →

α	$< \pm 0.5\%$
ρ	$< \pm 1.0\%$
λ	$< \pm 1.0\%$
ZT	$< \pm 1.0\%$

Bipolar Transient Harman Measurement*

* R. J. Buist, *Handbook of Thermoelectrics* (1995)





$$ZT=(\Delta V_S/\Delta V_\Omega)$$

$$\alpha = \Delta V_S/\Delta T \qquad \rho = (SF)^{-1} \times \Delta V_\Omega / I_{DC} \qquad \lambda = \alpha^2/Z\rho$$

$SF = A/l$ - sample Shape Factor, A – cross-section

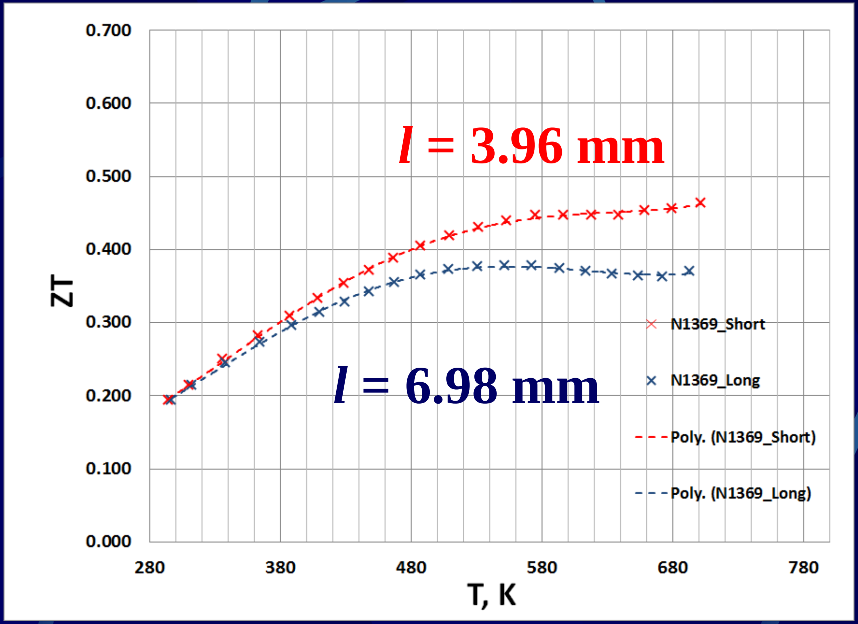
l – sample thickness

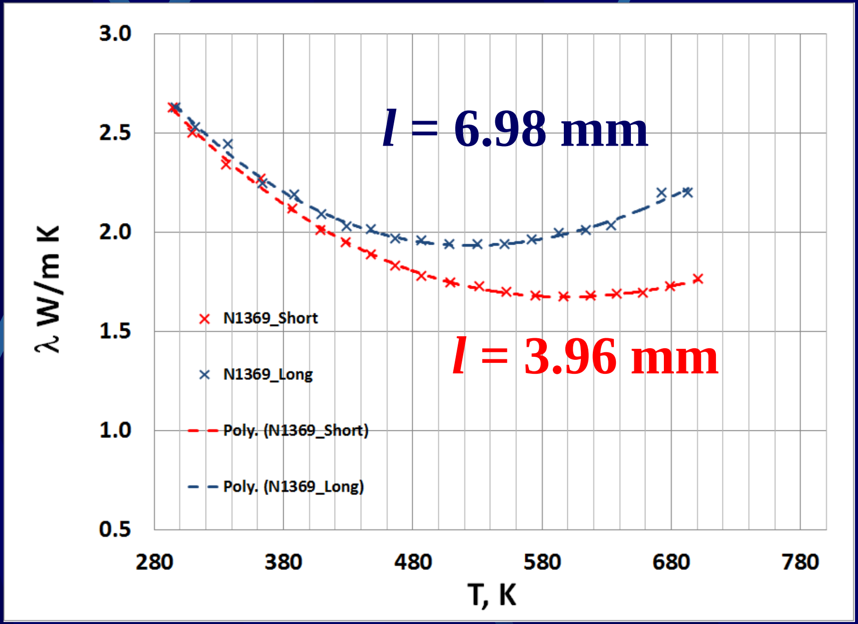
ZT and λ Measurements

$ZT = (\Delta V_S / \Delta V_\Omega)$

$\lambda = \alpha^2 / Z \cdot \rho$

PbTe Hot Extruded





Influence of parasitic thermal phenomena on ***ZT*** and λ Harman measurement.
The problem is well known for almost 60 years!*

*T. C. Harman, *J. Appl. Phys.*, **29**, 1373 (1958)

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Thermal Phenomena & Calibration

Theoretical Study of the Harman- Method for Evaluating the Thermoelectric Performance of Materials and Components at High Temperature

A. Jacquot, M. Jägle, J. König, D.G. Ebling, H. Böttner , **ECT2007**

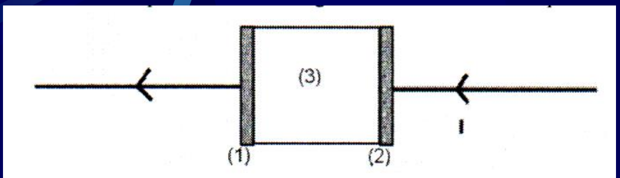


Figure 1: *Arrangement of the sample*

Derivation of the correction factor β

$$Z_{\alpha_M} T = \frac{V_\alpha}{V_\rho}\beta$$

Method of calculation of β

The equation (7) comes also as:

$$Z\overline{T}\,\frac{V_{\rho+c}}{V_\alpha} = a_1\left[a_2 V_{\rho+c}^2 + a_3 + a_4 + a_5\right] \qquad (12)$$

a_1 represents the effect of the contact resistance.

a_2 arises from the effect of the difference of the contact resistances.

a_3 account for the heat losses along the feed lines.

a_4 represents the heat radiated by the feedlines.

a_5 represents the heat radiated by the sample.

Table 1b: *Effect of the sample geometry and emissivity on β. The data used for the calculation are reported in the Table 1a.*

$L_S \setminus \varepsilon$	0	0,5	1
0,2 cm	$V_{\rho+c}$ =9,989e-4 β=1,002 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=2e-3 $a_4(h_M,..)$=0 $a_5(\varepsilon,..)$=1,000	$V_{\rho+c}$ =1,036e-3 β=1,049 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=2e-3 $a_4(h_M,..)$=4,1e-2 $a_5(\varepsilon,..)$=1,005	$V_{\rho+c}$ =1,081e-3 β=1,095 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=2e-3 $a_4(h_M,..)$=8,3e-2 $a_5(\varepsilon,..)$=1,011
1 cm	$V_{\rho+c}$ =9,896e-4 β=1,002 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=2e-3 $a_4(h_M,..)$=0 $a_5(\varepsilon,..)$=1,000	$V_{\rho+c}$ =1,330e-3 β=1,347 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=1e-2 $a_4(h_M,..)$=2.07e-1 $a_5(\varepsilon,..)$=1,130	$V_{\rho+c}$ =1,650e-3 β=1,347 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=1e-2 $a_4(h_M,..)$=4,13e-1 $a_5(\varepsilon,..)$=1,248
2 cm	$V_{\rho+c}$ =1,007e-3 β=1,020 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=2e-2 $a_4(h_M,..)$=0	$V_{\rho+c}$ =1,892e-3 β=1,915 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=2e-2 $a_4(h_M,..)$=4,13e-1	$V_{\rho+c}$ =2,6770-3 β=2,710 $a_1(r_C,..)$=1 $a_2(\Delta r_c,..)$=0 $a_3(\kappa_M,..)$=2e-2 $a_4(h_M,..)$=8,27e-1

Thermal Phenomena & Calibration

We accepted that it is **impossible**:

- Total practical elimination of parasitic thermal phenomena
- Precise theoretical prediction of thermal interaction between the sample and its environment

We believe that it is **possible**:

- Experimental evaluation with high precision of the total impact of all parasitic phenomena for any given temperature
- Compensation of its impact by proper system calibration

Two Samples System Calibration (2SSC)

We introduced novel calibration procedure which we call **2SSC**

(Two Sample System Calibration)

Two Samples System Calibration (2SSC)

Basic hypothesis

Peltier heat αTI during the Harman test generates a temperature difference ΔT across the sample which is inversely proportional to the thermal conductance of the sample K_s and the total equivalent thermal conductance K_p of all parasitic phenomena.

$$\alpha TI = \Delta T\,(K_s + K_p)$$

Parasitic conductance K_p is the distinctive ***system*** parameter which varies with temperature but is independent of the sample size and its nature

1^{st} step of the Two Samples System Calibration (2SSC)

For two samples of the same material and the same DC electrical current

$$\begin{cases} \alpha T I = \Delta T_1 (K_{s1} + K_p) \\ \alpha T I = \Delta T_2 (K_{s2} + K_p) \end{cases}$$

For $A_1 = A_2$ and $l_1 = n l_2$ → $nSF_1 = SF_2$ → $nK_{S1} = K_{S2}$

Shape factors — Thermal conductances

Solving system for K_P

$$K_p = K_{s1} \frac{n\Delta T_2 - \Delta T_1}{\Delta T_1 - \Delta T_2}$$

Parasitic Thermal Conductance

1st step of the Two Samples System Calibration (2SSC)

$$Z = \frac{\alpha^2}{\rho\lambda} = \frac{\alpha^2}{R_{s1}K_{s1}}$$

True value (*Z*)

$$Z_{1meas} = \frac{\alpha^2}{R_{s1}(K_{s1} + K_p)}$$

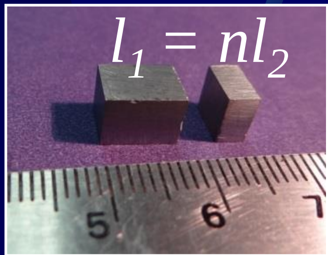
Measured value (Z_{s1})

$$ZT = Z_{1meas}T\left(1 + \frac{n\Delta T_2 - \Delta T_1}{\Delta T_1 - \Delta T_2}\right)$$

$$\lambda = \lambda_{1meas}/\left(1 + \frac{n\Delta T_2 - \Delta T_1}{\Delta T_1 - \Delta T_2}\right)$$

This result needs experimental validation

Experimental Validation of 2SSC

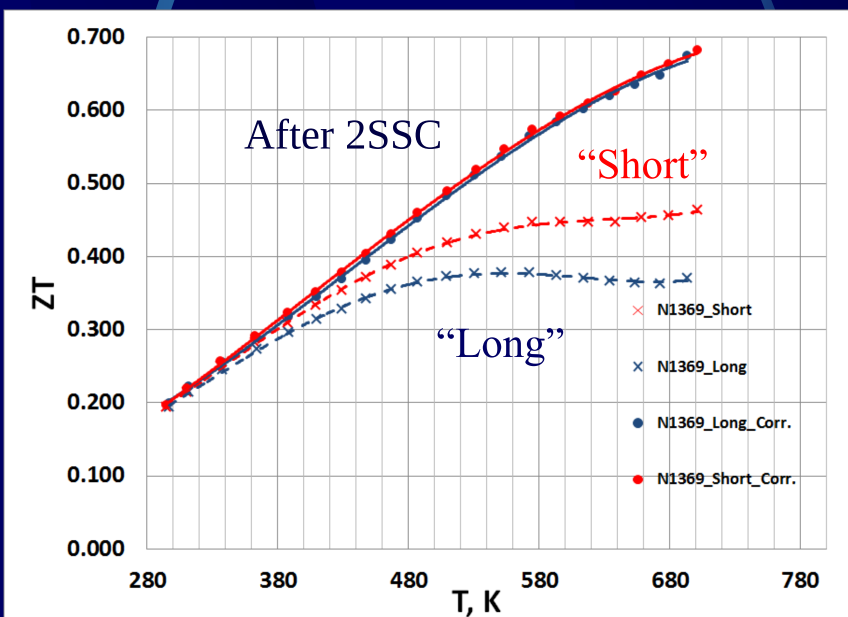


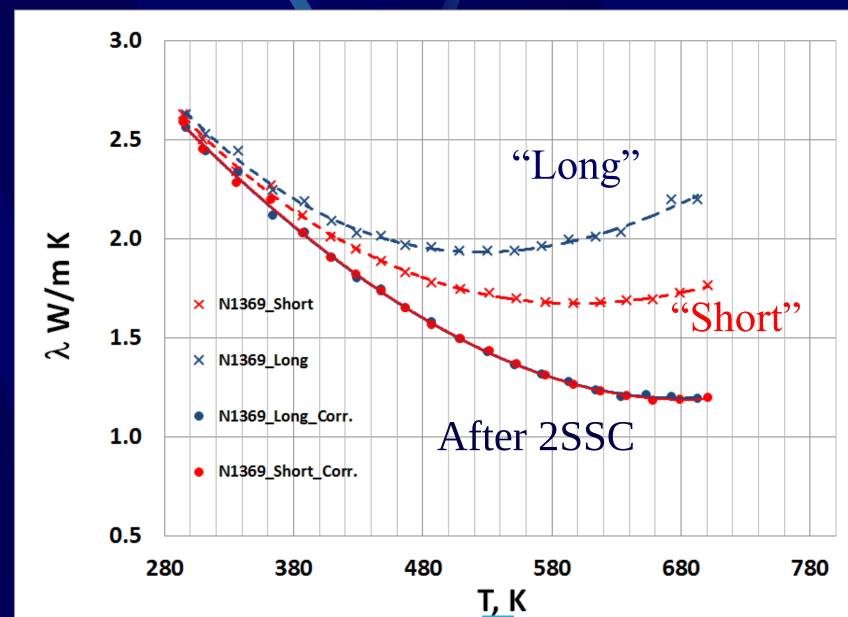
PbTe Hot Extruded n=1.76

"Long" - 6.98 mm
"Short" - 3.96 mm

$$Z_{1meas}T\left(1+\frac{n\Delta T_2-\Delta T_1}{\Delta T_1-\Delta T_2}\right)= \boldsymbol{ZT} = Z_{2meas}T\left(1+\frac{1}{n}\frac{n\Delta T_2-\Delta T_1}{\Delta T_1-\Delta T_2}\right)$$

$$\lambda_{1meas}/\left(1+\frac{n\Delta T_2-\Delta T_1}{\Delta T_1-\Delta T_2}\right)= \boldsymbol{\lambda} = \lambda_{2meas}/\left(1+\frac{1}{n}\frac{n\Delta T_2-\Delta T_1}{\Delta T_1-\Delta T_2}\right)$$





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Pros & Cons

Pros.

- True *ZT* and λ values for an unknown material
- No need in reference sample

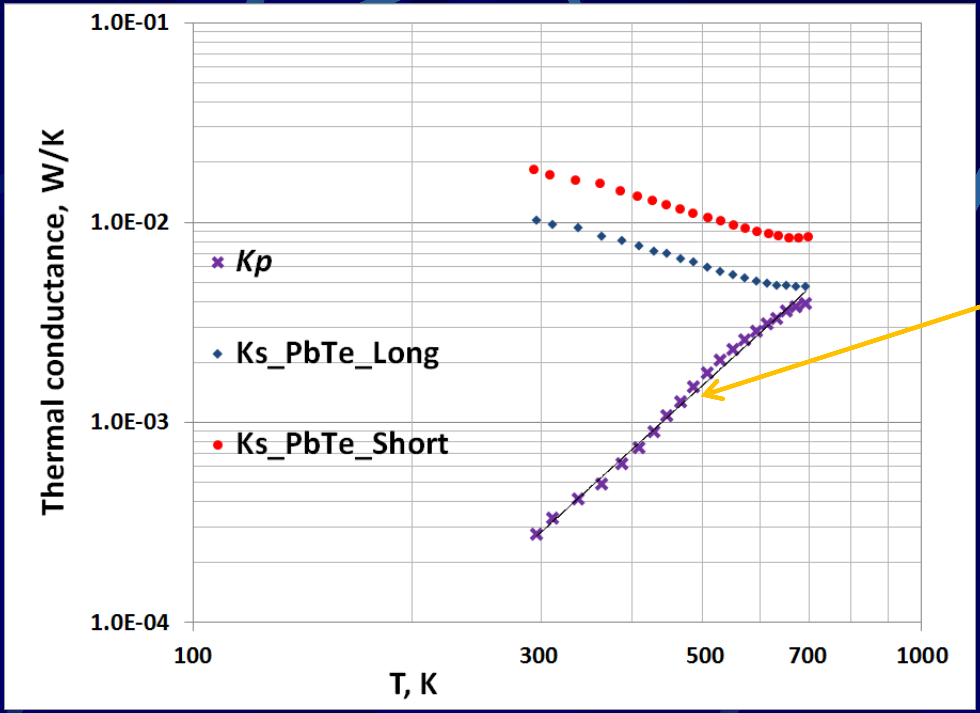
Cons.

- Time consuming procedure
- Potential problem for preparation of two samples with the same properties

There is another option - the 2nd step of 2SSC

2^{nd} step of the Two Samples System Calibration (2SSC)

With $K_p = K_{s1}\dfrac{n\Delta T_2 - \Delta T_1}{\Delta T_1 - \Delta T_2}$ and $K_{s1} = SF_1 \times \lambda$ for different $T\,K$



we define absolute values of $K_P(T)$

$K_P(T)$

is the distinctive signature of the *system* (ZT-Scanner) setup

We consider now the 1st sample as the Reference one with $\lambda_{Ref} \equiv \lambda$

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2^{nd} step of the Two Samples System Calibration (2SSC)

X-sample differs from the reference one not only by the shape factor, but also by its thermal conductivity with $K_X = n\frac{\lambda_X}{\lambda_{Ref}}K_{Ref}$

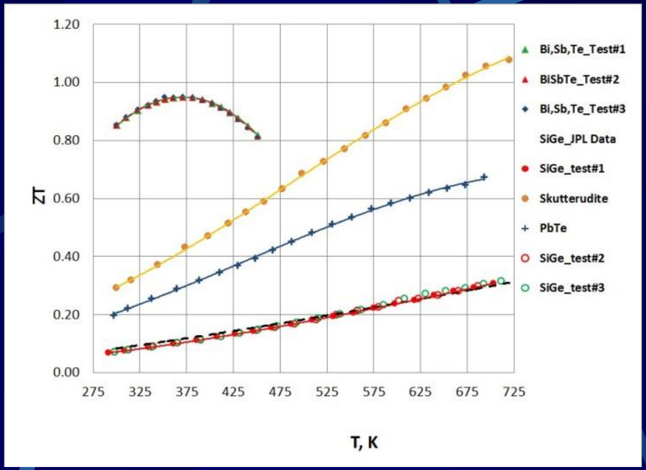
$$Z_XT = Z_{X,meas}T(1 + \frac{1}{n}\frac{\lambda_{Ref}}{\lambda_X}\frac{K_p}{K_{Ref}})$$

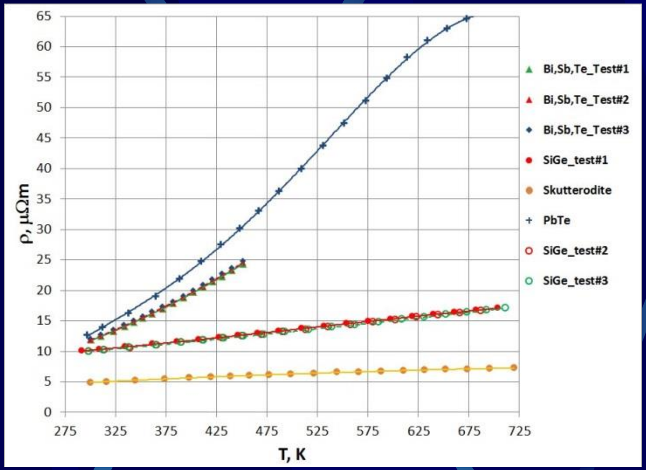
$$\lambda_X = \lambda_{X,meas}/(1 + \frac{1}{n}\frac{\lambda_{Ref}}{\lambda_X}\frac{K_p}{K_{Ref}})$$

Second equation can be solved for λ_X , which then is used in the first one for true Z_XT value calculation

$$\boxed{\lambda_X = \lambda_{X,meas} - \frac{\lambda_{Ref}}{n}\frac{K_p}{K_{Ref}}}$$

Application of the ZT-Scanner with the 2SSC on different TE materials



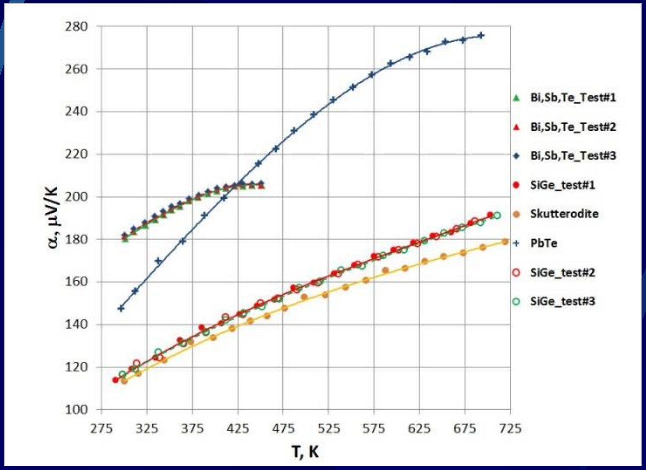


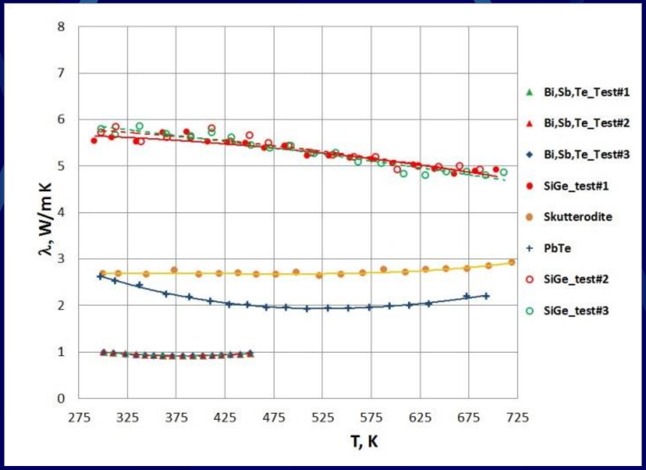
BiSbTe - p

PbTe - n

SiGe - p

Skutterudite-n





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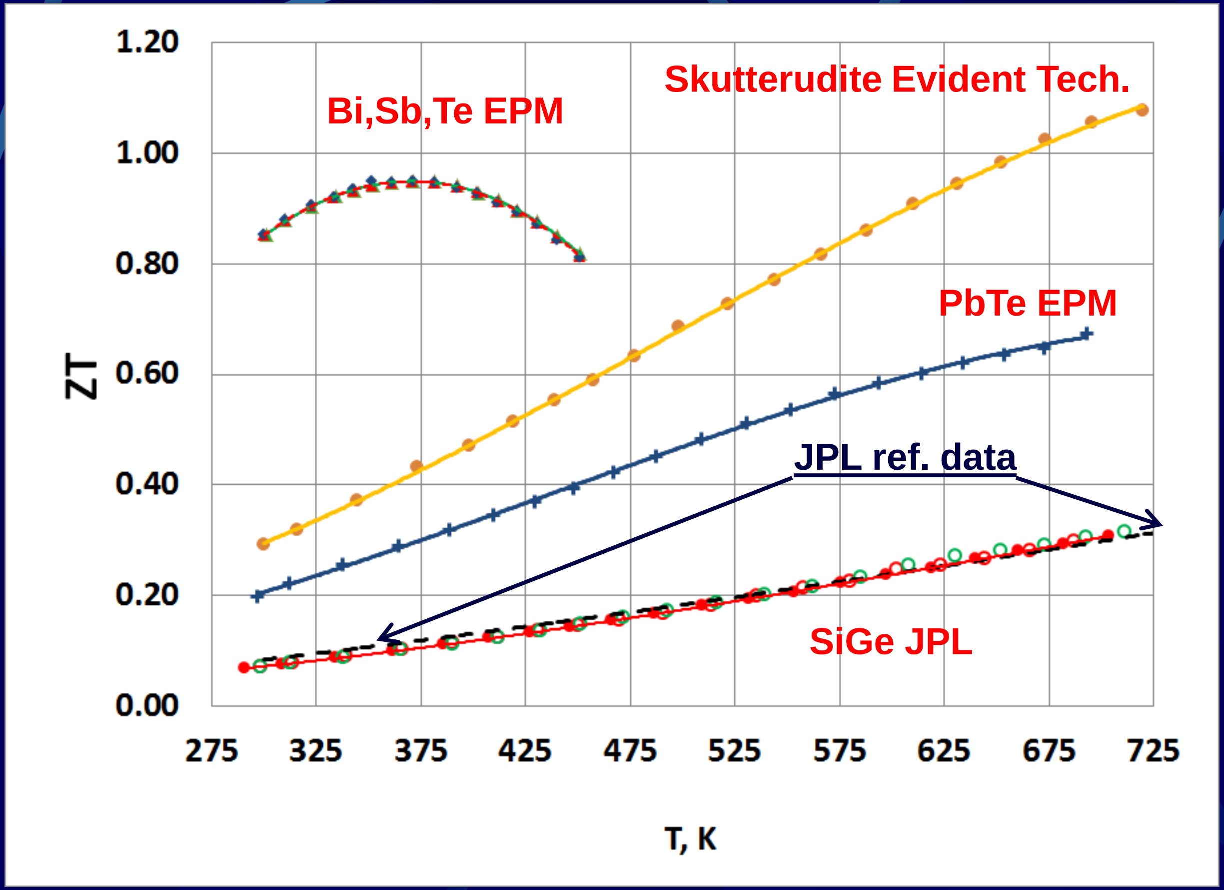
Application of the ZT-Scanner with the 2SSC on different TE materials



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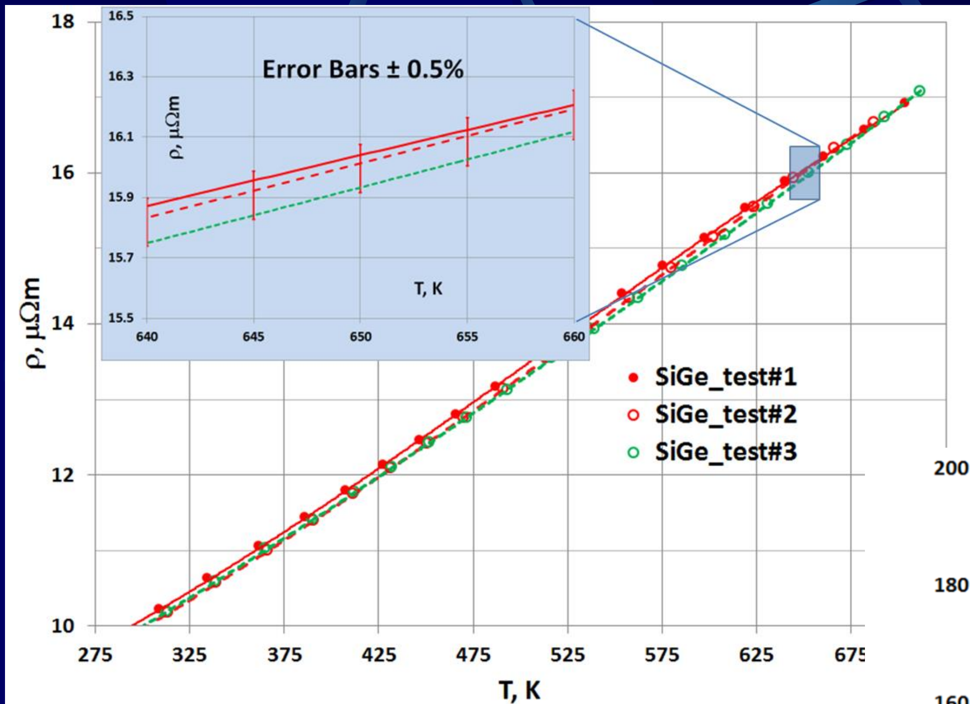
Precision of Measurement



Specific contact resistivity

PbSn solder $R_C = 6\times10^{-7}\ \Omega\ cm^2$

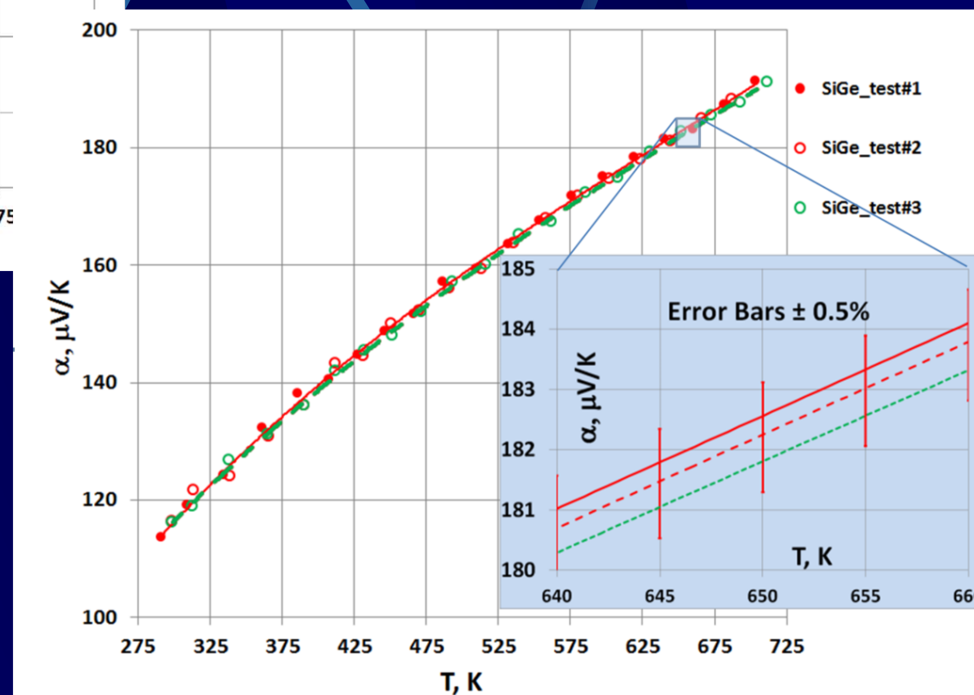
Silver Paste $R_C = 9\times10^{-7}\ \Omega\ cm^2$

Three consecutive $\rho(T)$ and $\alpha(T)$ measurements on **SiGe** sample

Measurement precision

α - < ± 0.5%

ρ - < ± 0.5%



Accuracy of Measurement

Accuracy

Data Acquisition by ZT-Scanner with:
Keithley 2401 power source
Agilent 34420a nano-voltmeter

ΔV < ± 0.1%
ΔT < ± 0.2%

Combined error on sample size (5×5×6 mm³) — SF < ± 0.6%

Relative overestimation of ρ for ($\rho \geq 10$ μΩ m) and $R_C = 9\times10^{-7}$ Ω cm² — < 0.5%

Accuracy of 2SSC is based on $K_p = K_{s1}\dfrac{n\Delta T_2 - \Delta T_1}{\Delta T_1 - \Delta T_2}$ — < ± 1.0%

Measurement accuracy

α	< ± 0.5%
ρ	< ± 1.0%
λ	< ± 1.0%
ZT	< ± 1.0%

Conclusions

- Parasitic thermal interactions is not a critical factor anymore for Harman measurements

- Its impact can be properly compensated by 2SSC procedure

- ***ZT***, α, ρ and λ values can be defined with the accuracy of 1% from 240K to 720K with the ZT-Scanner

- The problem with almost 60 years history of accurate Harman measurement is now solved

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Conclusions

ZT-Scanner is available from TEMTE Inc.

Visit us at www.temte.ca

Contact us at info@temte.ca