SOLAR ENERGY
TECHNOLOGIES OFFICE
U.S. Department Of Energy

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THERMOPHYSICAL PROPERTY MEASUREMENTS OF HEAT TRANSFER MEDIA AND CONTAINMENT MATERIALS

Topic Area 2B: Gen3 Research & Analysis

Gen3 CSP Summit

Principal Investigator: Shannon K. Yee, Associate Professor

Co-PI: Andrey Gunawan, Research Engineer II

Thursday, August 26th 2021

Georgia Institute of Technology

People: STEEL and Georgia Tech


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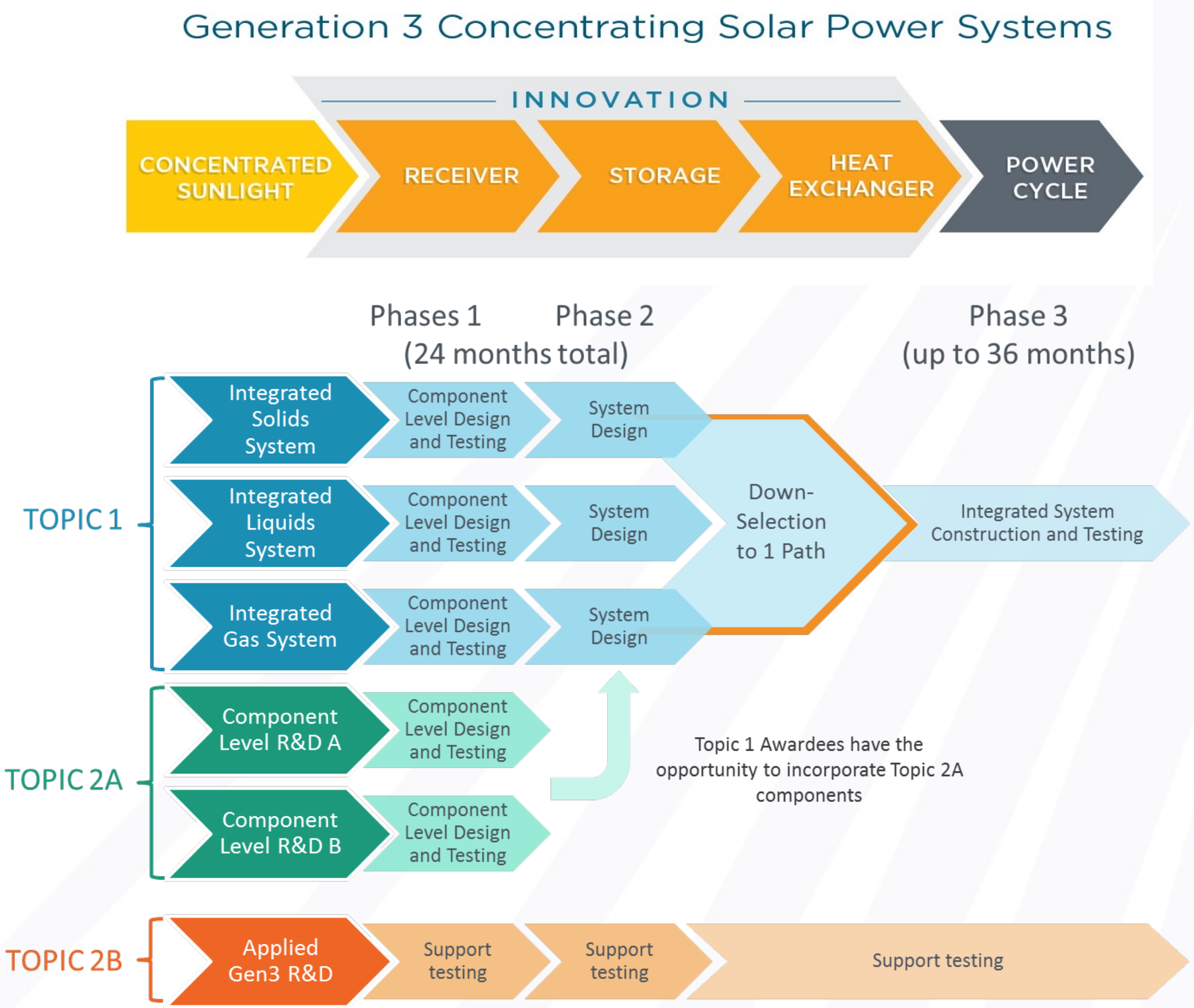
Associate Professor

Objectives of Gen3 CSP Program



Thermophysical Property Measurements of

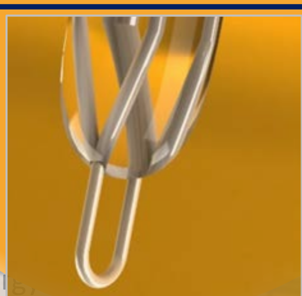
Heat Transfer Media (e.g., molten salts) & **Containment Materials** (e.g., high temperature alloys)

LIBRARY NEXT

Thermophysical Property Database

HEAT LAB

Immersion Electrothermal Probe



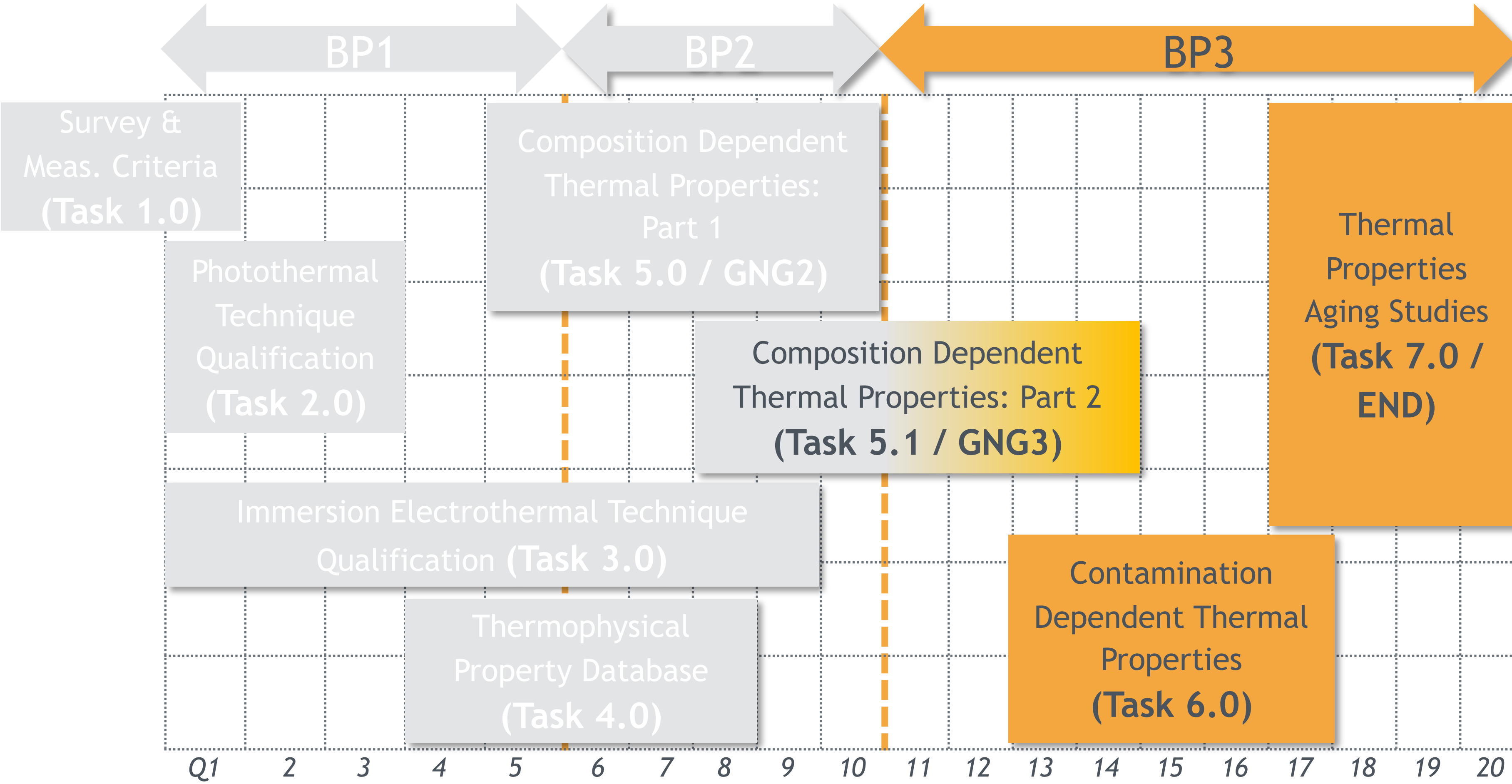
HEAT LAB

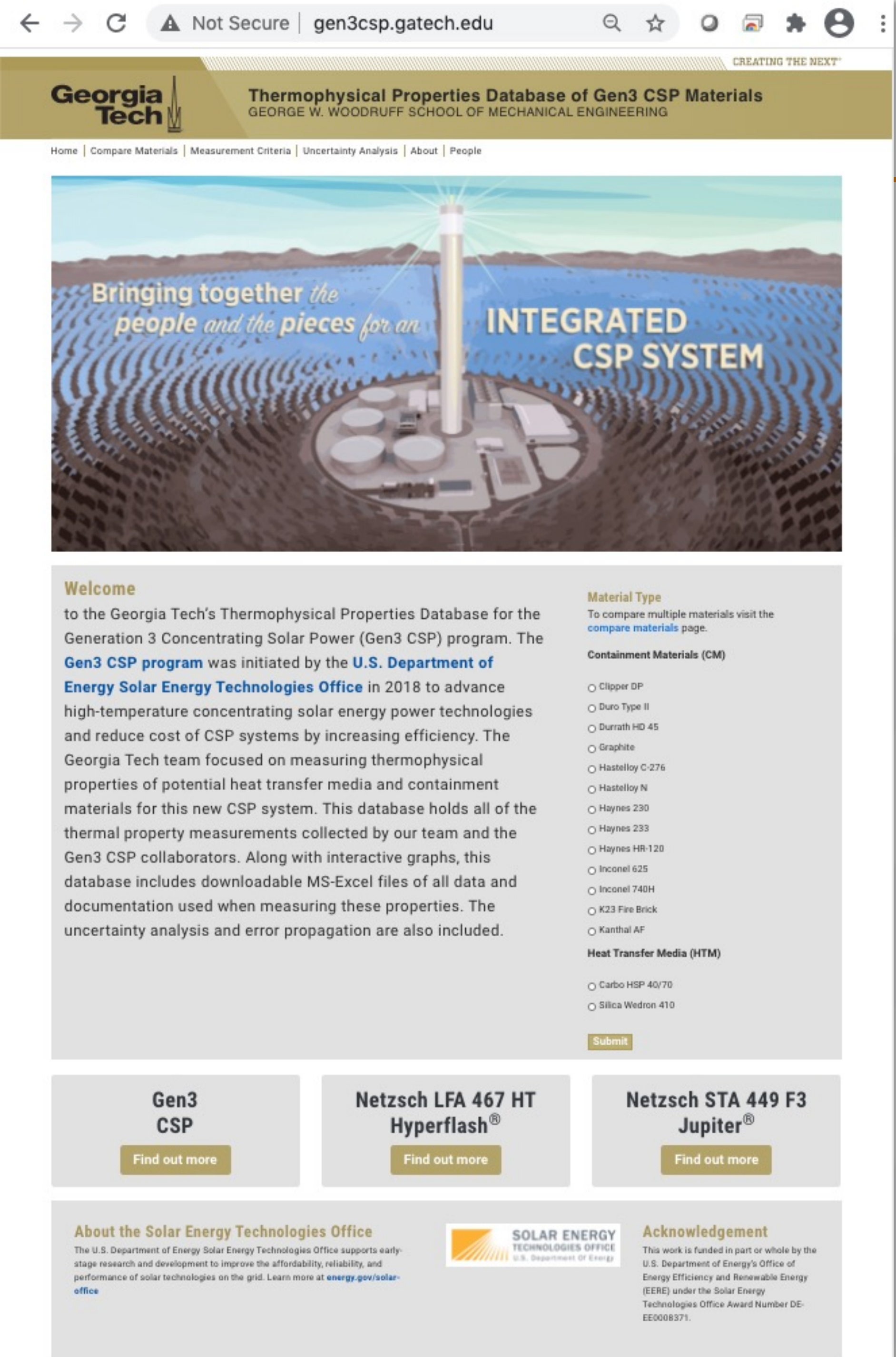
Advanced Photo-thermal Technique

Measurement Accuracy @ High Temperatures in Corrosive Environments

Project Objectives - The Technology's Critical Path

The overall objective of this project is to gather thermophysical property data—specifically, thermal conductivity, thermal diffusivity, and specific heat—of heat transfer medias (HTMs) and containment materials (CMs) used in Topic Area 1 and 2A at high temperatures (>700 °C)





gen3csp.gatech.edu Homepage

- Main access to thermophysical properties of 25+ HTM and CMs
- Access to pages:
  1. Compare Materials
  2. Measurement Criteria
  3. Uncertainty Analysis
  4. About
  5. People
- Information about:
  - Gen3 CSP program
  - Measurement tools
- SETO information
- Acknowledgement

Georgia Tech

Thermophysical Properties Database of Gen3 CSP Materials

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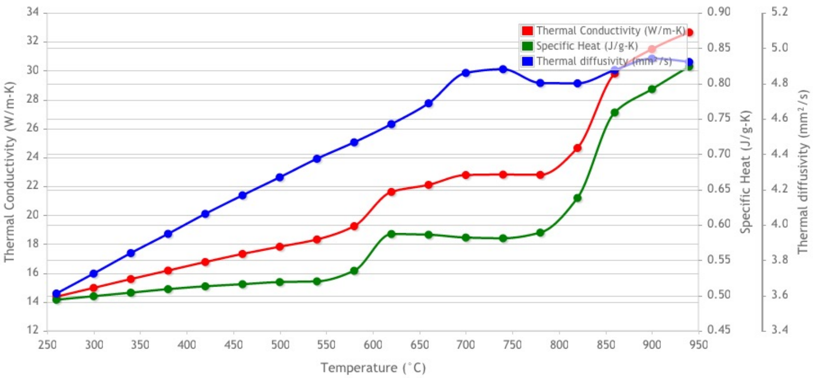
Home | Compare Materials | Measurement Criteria | Uncertainty Analysis | About | People

Inconel 740H



Toggle Error Bars:

Download data here: Inconel740H.xlsx

Notes:

- Measurement uncertainties at 95% confidence level are included in the downloadable MS. Excel file above
- Detailed uncertainty analysis and error propagation methodology used to develop those measurement uncertainties is described in the Uncertainty Analysis page

More information

Information about each thermal property can be found below.

Thermal diffusivity (mm^2/s)

- Each data point shown in the plot is an average of (minimum of) three (3) measurements
- Instrument: NETZSCH LFA 467 HT HyperFlash®
- Purge gas: Argon
- Protective gas: Argon
- Sample thickness: 2.03 – 2.15 mm
- Sample diameter: 10 mm

Specific Heat Capacity (J/g-K)

- Each data point shown in the plot is an average of (minimum of) three (3) measurements
- Instrument: NETZSCH STA 449 F3 Jupiter®
- Purge gas: Argon
- Protective gas: Argon
- Sample mass: 91.5 – 93.8 mg

Thermal Conductivity (W/m-K)

- Thermal conductivity, thermal diffusivity, specific heat and density are related through, $k = \alpha c_p \rho$. Therefore, best estimation of the thermal conductivity is calculated based on that relation.
- Density of Inconel 740H is assumed to be constant at 8.05 g/cm^3 [1]

References:

1. Inconel® alloy 740H®. [Online]. Available: https://www.specialmetals.com/assets/smc/documents/alloys/inconel/inconel-alloy-740-h.pdf.

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Individual material page

(Showing Inconel 740H as an example)

- Temperature dependence plots:
  i. Thermal conductivity
  ii. Specific Heat
  iii. Thermal diffusivity
- Toggle switch for turn on/off error bands
- Data (.xlsx) download link
- Experimental parameters
- Reference(s)

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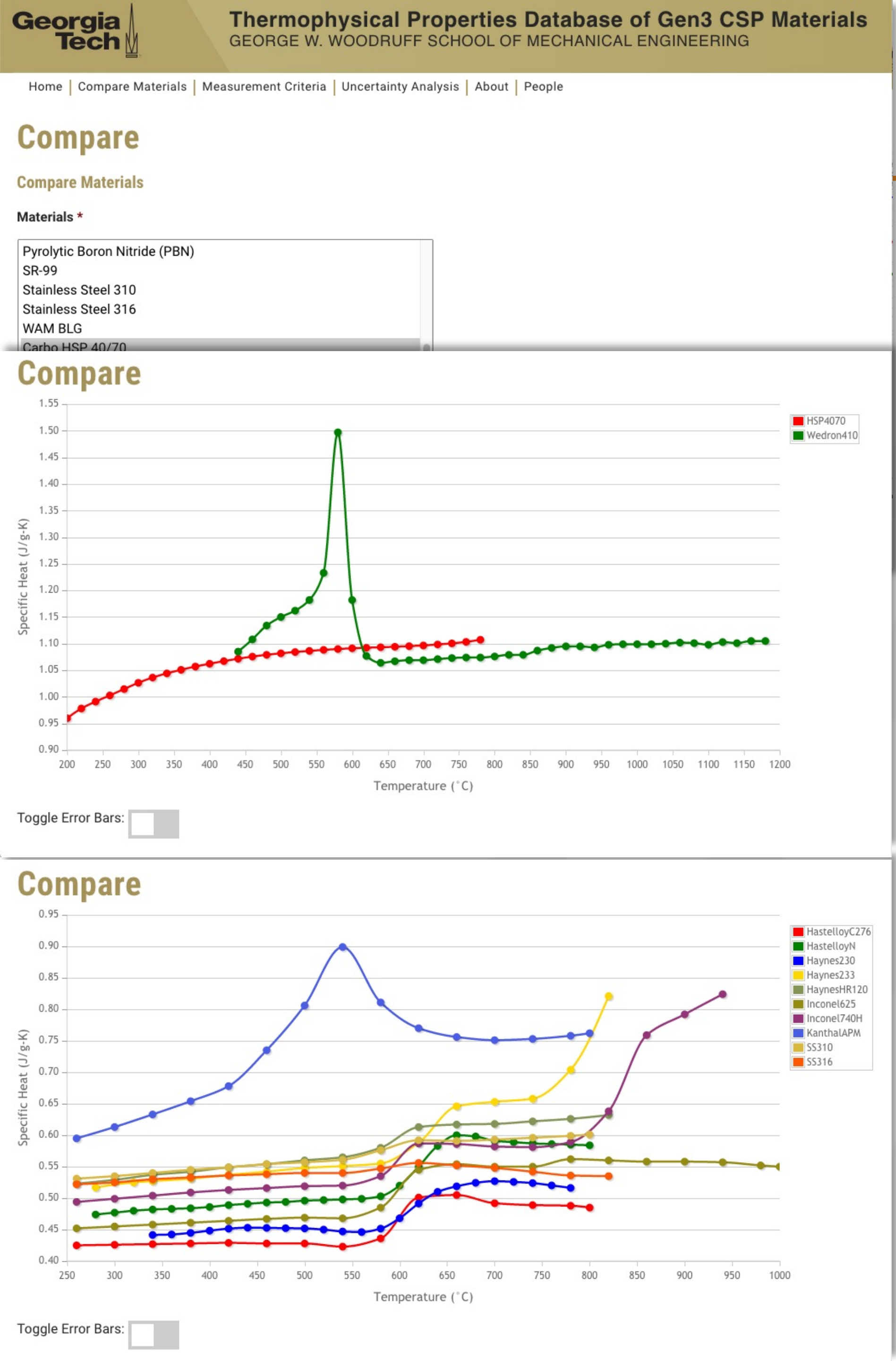

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Compare Materials

- (i) Choose the materials, and (ii) choose the interested thermophysical property
- Toggle switch for turn on/off error bands
- Up to 10 materials for comparison

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Measurement Criteria

Measurement Acceptance Criteria*

Standards, Principles, and Required Documentation

*A PDF version of this document can be downloaded here.

A goal of this Gen3CSP project is to establish a thermophysical property database of high temperature (700-1250 °C) (i) heat transfer media (HTMs) and (ii) containment materials (CMs). This database will be made public containing thermophysical properties collected using (a) our electrothermal immersion technique, (b) our modified photothermal technique, and (c) from third parties (*i.e.*, literature and other Gen3CSP collaborators). This document outlines criteria for an "acceptable" measurement or dataset to be included in this database and the accompanying supporting information.

Acceptable Measurement Criteria: Thermophysical property (thermal diffusivity, thermal conductivity, and specific heat) measurements performed for the Gen3CSP program will meet the following criteria:

1. Sufficient details to identify the material (*e.g.*, composition, vendor's product/catalog ID number, *etc.*),
2. Nominal measurement value,
3. Measurement conditions (*e.g.*, temperature, N_2/Ar environment, *etc.*),
4. Uncertainty quantification individually reported with each measurement,
   - Measurement uncertainty of less than 15% of the nominal value is preferred, but exceptions can be made for difficult/exotic materials.
   - Monte-Carlo uncertainty is the preferred approach when using a multi-parameter thermal model; simplified quadrature uncertainty can be used when an analytical relation exists.
5. Reference to appropriate measurement standard or supporting peer-reviewed publication detailing the measurement technique.

This data will be made available to the public with each measurement set that is curated in this thermophysical property database via a web interface hosted at Georgia Tech. Raw data of each measurement will be curated separately but will be made available for subsequent analysis upon request. Each dataset included in the database will be reviewed by senior personnel (*i.e.*, the PI, Co-PI, Research Engineer/Scientist, Postdoc, *etc.*) to ensure that these criteria are met.

Notes:

- Our electrothermal immersion technique is a modification of the 3-omega technique that can be immersed in a high temperature fluid. This technique is currently under development (as of January 2019). This technique will be qualified by using known standards allowing for at most 10% variation from the accepted value. The technique methodology will be documented in a peer-reviewed publication.
- Our modified photothermal technique is a modification on the flash diffusivity technique. Specifically, this technique has been modified to use infrared optics to correct for collimator/aperture effects that become pronounced at high temperatures. This instrument was purchased from Netzsch (LFA 467 HT Hyperflash®) and modifications are underway. To measure the volumetric specific heat of bulk samples, we will use a modified DSC/TGA purchased from Netzsch (STA 449 F3 Jupiter®) with modification to the platinum furnace and sample holders specifically designed for more accurate specific heat measurements (and not phase transitions). The manufacturer's specific heat uncertainty is claimed at 3.5%; this will be verified through testing known standards.
- Third party measurements from others within the Gen3CSP program and beyond will also be welcome, but we will insist upon the aforementioned criteria for inclusion in the database.

We would also like to thank Prof. David G. Cahill, Prof. Chris Dames, and Prof. Patrick Hopkins, who were kind enough to review the above criteria as our standard for acceptable measurements.

David G. Cahill

David Cahill is the Willett Professor of Engineering and Professor of Materials Science and Engineering at the University of Illinois at Urbana-Champaign. He joined the faculty of the Department of Materials Science and Engineering at the U. Illinois after earning his Ph.D. in condensed matter physics from Cornell University, and working as a postdoctoral research associate at the IBM Watson Research Center. His current research program focuses on developing a microscopic understanding of thermal transport at the nanoscale; extremes of low and high thermal conductivity in materials; the interactions between phonons, electrons, photons, and spin; and the kinetics and thermodynamics of aqueous and electrochemical interfaces with materials. He received the 2018 Innovation in Materials Characterization Award of the Materials Research Society (MRS); the 2015 Touloukian Award of the American Society of Mechanical Engineers; the Peter Mark Memorial Award of the American Vacuum Society (AVS); and is a fellow of the MRS, AVS, and APS (American Physical Society).

Chris Dames

Chris Dames received his Ph.D. in Mechanical Engineering from the MIT in 2006. His B.S. and M.S. are from UC Berkeley (1998, 2001). He was a faculty member at UC Riverside from 2006-2011 before joining UC Berkeley in 2011, and he has also worked as a research engineer for Solo Energy Corp. (1998-1999). His research interests emphasize fundamental studies of heat transfer and energy conversion at the nanoscale, using both theoretical and experimental methods. Some topics of current interest include graphene, nanocrystalline materials, mean free path distributions, thermoelectrics, biological systems, and highly anisotropic and nonlinear transport including thermal rectification. His research has been recognized with a DARPA Young Faculty Award (2009) and NSF CAREER award (2011).

Patrick E. Hopkins

Patrick Hopkins is a Professor in the Department of Mechanical and Aerospace Engineering at University of Virginia (U.Va.). He received his Ph.D. in Mechanical and Aerospace Engineering from the U.Va. in 2008, following a B.S. in Mechanical Engineering and a B.A. in Physics at U.Va. in 2004. He spent 3 years as a Harry S. Truman Postdoctoral Fellow at Sandia National Laboratories in Albuquerque, NM from 2008 – 2011. Hopkins began his faculty appointment at U.Va. in 12/2011 as an Assistant Professor, and was promoted to Associate Professor with tenure in 8/2015. Hopkins is a recipient of the AFOSR and ONR Young Investigator Awards, the ASME Bergles-Rohsenow Young Investigator Award in Heat Transfer, and the Presidential Early Career Award for Scientists and Engineers (PECASE).

About the Solar Energy Technologies Office

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Acknowledgement

This work is funded in part or whole by the U.S. Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the Solar Energy Technologies Office Award Number DE-EE0008371.

- Our guideline in doing the measurements & reporting datasets in the database
- This document was peer-reviewed by experts from the appropriate Heat Transfer community (with DOE endorsement)

★ **It was submitted to osti.gov:**

OSTI ID: 1719141

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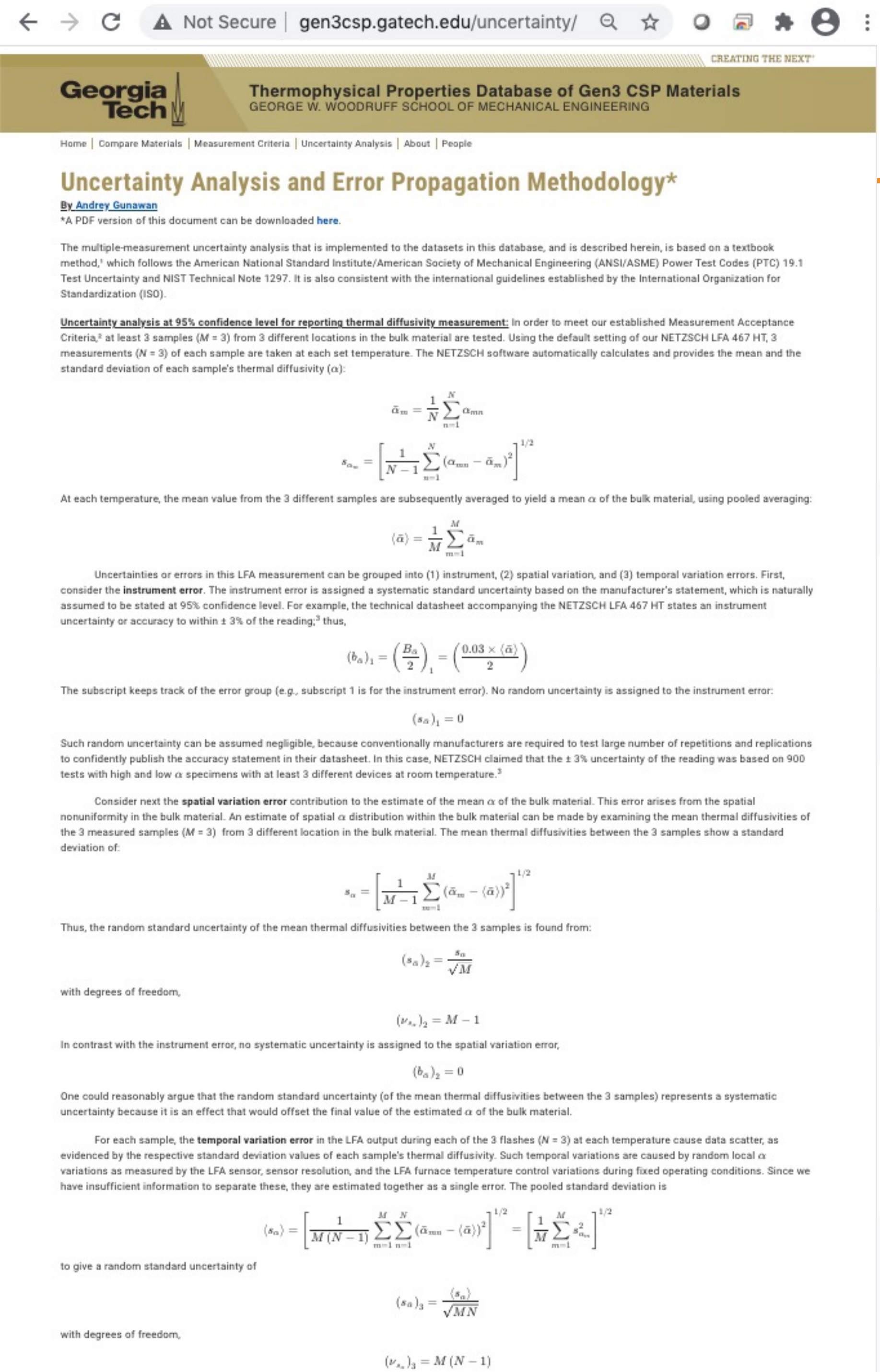
Uncertainty Analysis



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Uncertainty Analysis and Error Propagation Methodology*

By Andrey Gunawan

*A PDF version of this document can be downloaded here.

The multiple-measurement uncertainty analysis that is implemented to the datasets in this database, and is described herein, is based on a textbook method,[1] which follows the American National Standard Institute/American Society of Mechanical Engineering (ANSI/ASME) Power Test Codes (PTC) 19.1 Test Uncertainty and NIST Technical Note 1297. It is also consistent with the international guidelines established by the International Organization for Standardization (ISO).

Uncertainty analysis at 95% confidence level for reporting thermal diffusivity measurement: In order to meet our established Measurement Acceptance Criteria,[2] at least 3 samples (M = 3) from 3 different locations in the bulk material are tested. Using the default setting of our NETZSCH LFA 467 HT, 3 measurements (N = 3) of each sample are taken at each set temperature. The NETZSCH software automatically calculates and provides the mean and the standard deviation of each sample's thermal diffusivity (α):

$$\bar{\alpha}_m = \frac{1}{N}\sum_{n=1}^{N} \alpha_{mn}$$

$$s_{\alpha_m} = \left[\frac{1}{N-1}\sum_{n=1}^{N} (\alpha_{mn} - \bar{\alpha}_m)^2\right]^{1/2}$$

At each temperature, the mean value from the 3 different samples are subsequently averaged to yield a mean α of the bulk material, using pooled averaging:

$$\langle\bar{\alpha}\rangle = \frac{1}{M}\sum_{m=1}^{M} \bar{\alpha}_m$$

Uncertainties or errors in this LFA measurement can be grouped into (1) instrument, (2) spatial variation, and (3) temporal variation errors. First, consider the **instrument error**. The instrument error is assigned a systematic standard uncertainty based on the manufacturer's statement, which is naturally assumed to be stated at 95% confidence level. For example, the technical datasheet accompanying the NETZSCH LFA 467 HT states an instrument uncertainty or accuracy to within ± 3% of the reading;[3] thus,

$$(b_{\bar{\alpha}})_1 = \left(\frac{B_{\bar{\alpha}}}{2}\right)_1 = \left(\frac{0.03 \times \langle\bar{\alpha}\rangle}{2}\right)$$

The subscript keeps track of the error group (e.g., subscript 1 is for the instrument error). No random uncertainty is assigned to the instrument error:

$$(s_{\bar{\alpha}})_1 = 0$$

Such random uncertainty can be assumed negligible, because conventionally manufacturers are required to test large number of repetitions and replications to confidently publish the accuracy statement in their datasheet. In this case, NETZSCH claimed that the ± 3% uncertainty of the reading was based on 900 tests with high and low α specimens with at least 3 different devices at room temperature.[3]

Consider next the **spatial variation error** contribution to the estimate of the mean α of the bulk material. This error arises from the spatial nonuniformity in the bulk material. An estimate of spatial α distribution within the bulk material can be made by examining the mean thermal diffusivities of the 3 measured samples (M = 3) from 3 different location in the bulk material. The mean thermal diffusivities between the 3 samples show a standard deviation of:

$$s_{\alpha} = \left[\frac{1}{M-1}\sum_{m=1}^{M} (\bar{\alpha}_m - \langle\bar{\alpha}\rangle)^2\right]^{1/2}$$

Thus, the random standard uncertainty of the mean thermal diffusivities between the 3 samples is found from:

$$(s_{\bar{\alpha}})_2 = \frac{s_{\alpha}}{\sqrt{M}}$$

with degrees of freedom,

$$(\nu_{s_\alpha})_2 = M - 1$$

In contrast with the instrument error, no systematic uncertainty is assigned to the spatial variation error,

$$(b_{\bar{\alpha}})_2 = 0$$

One could reasonably argue that the random standard uncertainty (of the mean thermal diffusivities between the 3 samples) represents a systematic uncertainty because it is an effect that would offset the final value of the estimated α of the bulk material.

For each sample, the **temporal variation error** in the LFA output during each of the 3 flashes (N = 3) at each temperature cause data scatter, as evidenced by the respective standard deviation values of each sample's thermal diffusivity. Such temporal variations are caused by random local α variations as measured by the LFA sensor, sensor resolution, and the LFA furnace temperature control variations during fixed operating conditions. Since we have insufficient information to separate these, they are estimated together as a single error. The pooled standard deviation is

$$\langle s_{\alpha}\rangle = \left[\frac{1}{M(N-1)}\sum_{m=1}^{M}\sum_{n=1}^{N} (\bar{\alpha}_{mn} - \langle\bar{\alpha}\rangle)^2\right]^{1/2} = \left[\frac{1}{M}\sum_{m=1}^{M} s_{\alpha_m}^2\right]^{1/2}$$

to give a random standard uncertainty of

$$(s_{\bar{\alpha}})_3 = \frac{\langle s_{\alpha}\rangle}{\sqrt{MN}}$$

with degrees of freedom,

$$(\nu_{s_\alpha})_3 = M(N-1)$$

- This online document describe the detailed uncertainty analysis and error propagation methodology used by Georgia Tech.

★ **It was submitted to osti.gov:**

OSTI ID: 1719142

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About

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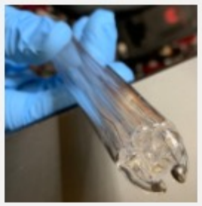
Tool Overview

Description of each of the tools that we used can be found below:



Electrothermal Immersion Technique

Our electrothermal immersion technique is a modification of the 3-omega technique that can be immersed in a high temperature fluid. This technique is currently under development (as of July 2020). This technique will be qualified by using known standards allowing for at most 10% variation from the accepted value. The technique methodology will be documented in a peer-reviewed publication.

Modified Photothermal Technique

Our modified photothermal technique is a modification on the flash diffusivity technique. This instrument was purchased from Netzsch (i.e. LFA 467 HT Hyperflash). Specifically, this technique has been modified to use infrared optics to correct for collimator/aperture effects that become pronounced at high temperatures.

Digital Scanning Calorimetry

To measure the volumetric specific heat of bulk samples, we will use a modified DSC/TGA purchased from Netzsch (STA 449 F3 Jupiter) with modification to the platinum furnace and sample holders specifically designed for more accurate specific heat measurements (and not phase transitions). The manufacturer's specific heat uncertainty is claimed at 3.5%; this will be verified through testing known standards.

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Acknowledgement

This work is funded in part or whole by the U.S. Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the Solar Energy Technologies Office Award Number DE-EE0008371.

Information about measurement tools:

- Thermal conductivity and specific heat of molten salts HTM (Electrothermal Immersion Technique)
- Thermal diffusivity of CMs (LFA 467 HT Hyperflash)
- Specific heat capacity of CMs (STA 449 F3 Jupiter)

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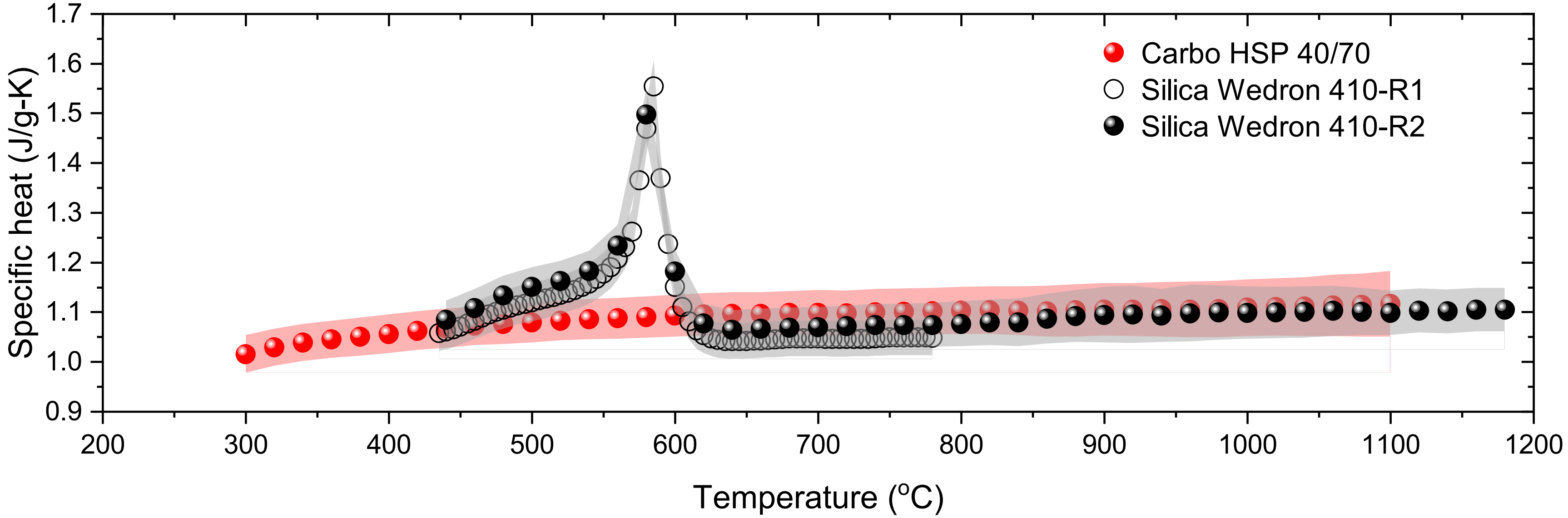
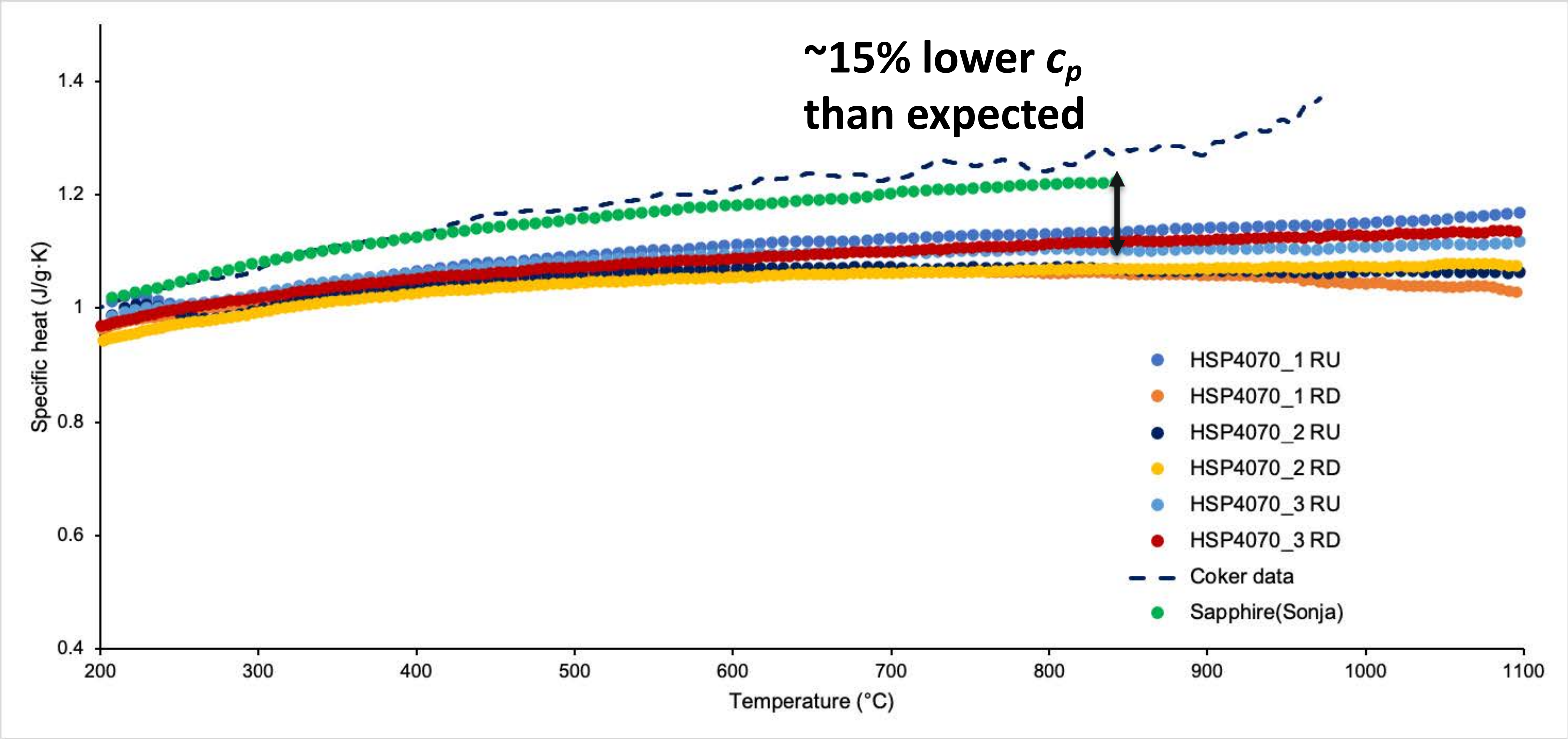
There are differences between materials within the same class of particulate HTMs



- Carbo HSP 40/70 is different than Silica Wedron 410 particles, which experience a phase change in crystalline structure (from alpha-quartz to beta-quartz) at ~573 °C.

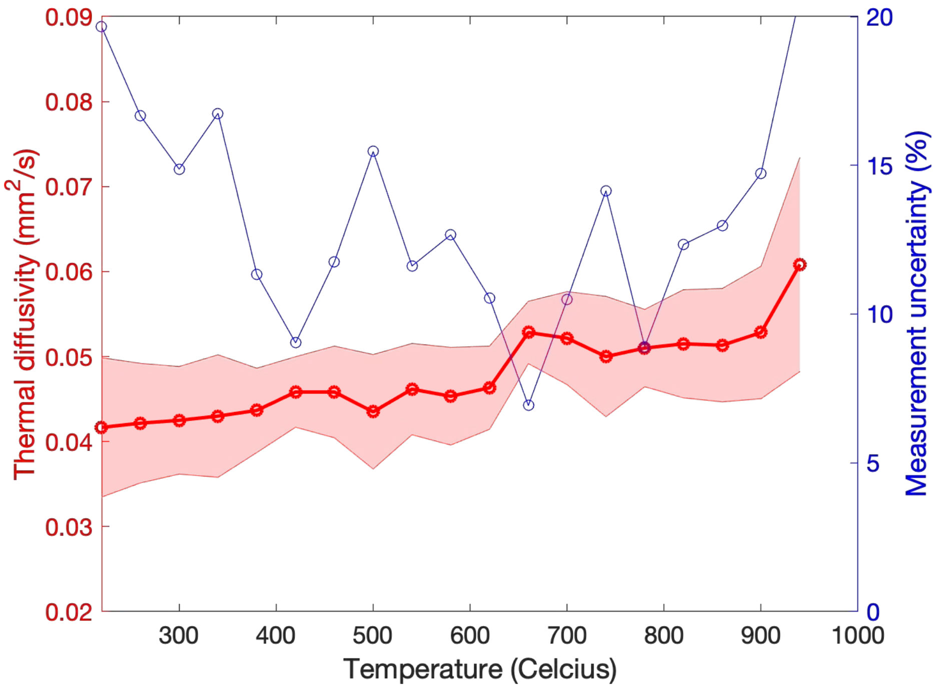
Maskalunas, J., "High-temperature thermal properties of particles for concentrated solar power and thermal-Energy storage system", *Master's Thesis*, Univ. Wisconsin-Madison 2020 (advisor: Nellis & Anderson)
https://sel.me.wisc.edu/publications/theses/maskalunas20.zip

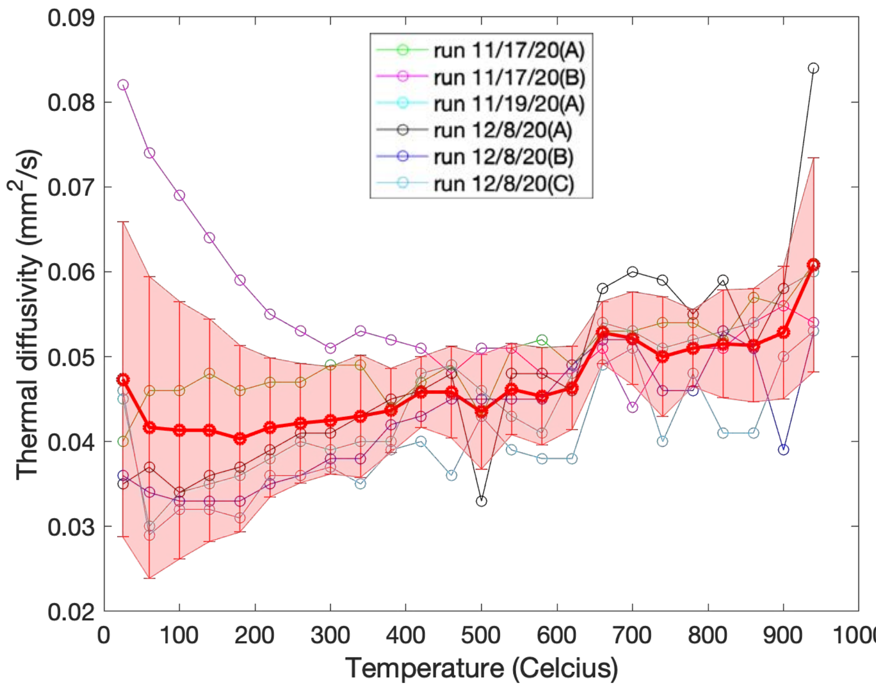
Different (DSC) setup/protocols may result in different c_p measurements



- Difference due to a sapphire disc placed underneath a DSC crucible, which is a common practice to prevent the crucible from sticking onto the sample holder, during high-temperature measurement cycle.
- To test this hypothesis, we measured the c_p of Netzsch's standard (pure) sapphire and compared it with the measurements.

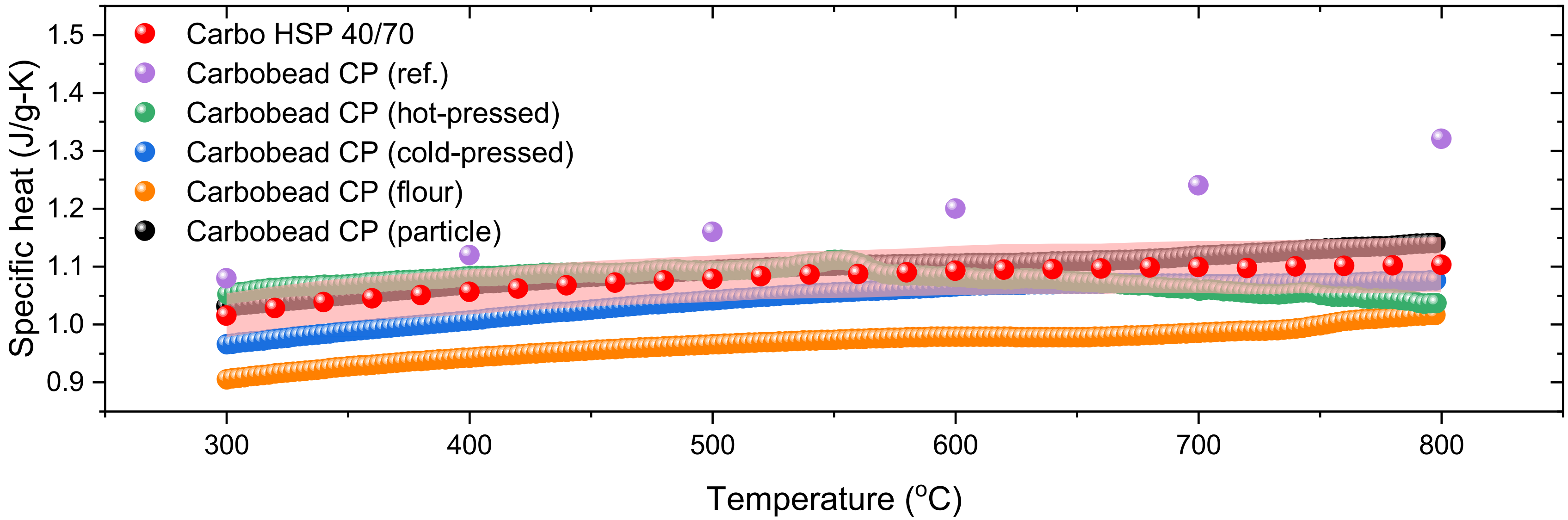
Not all LFAs can measure particles thermal diffusivity accurately





- Measurement uncertainty is still >15%
- Netzsch's LFA is not designed to measure thermal diffusivity of particles accurately

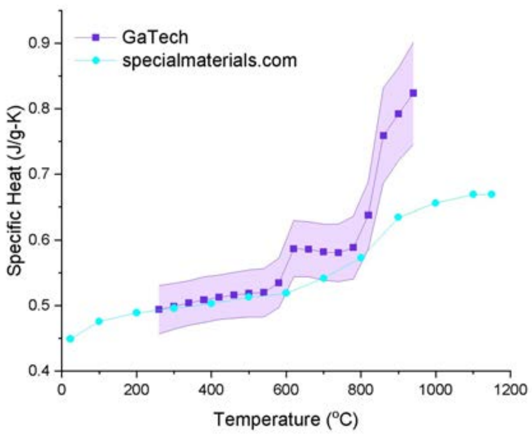
c_p does not vary with form-factor of the particles (up to 800 °C)

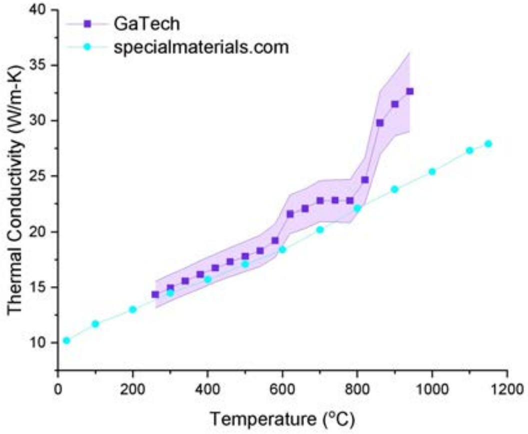


- c_p does not vary with form-factor of the particles
- Carbo HSP 40/70 is in line with Carbobead CP

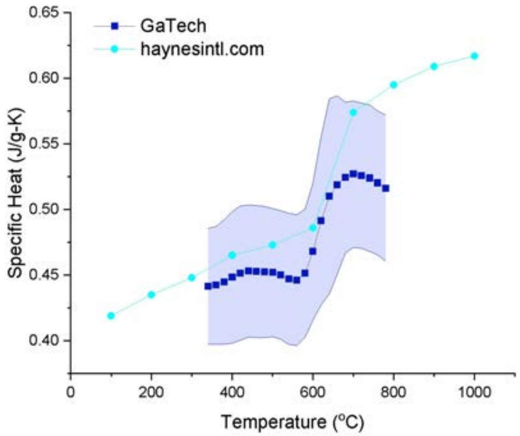
Ni-based alloy often exhibit high-temperature phase transition that are not previously captured in supplier's spec sheet

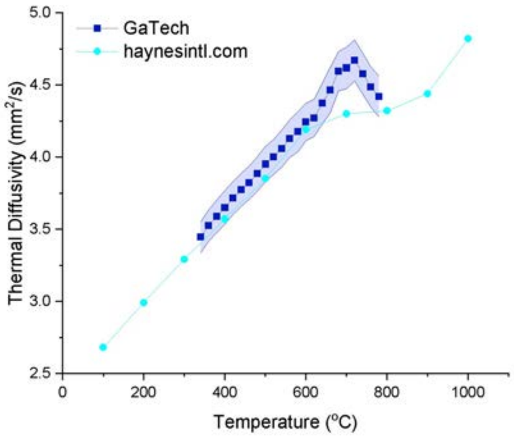
Inconel 740H

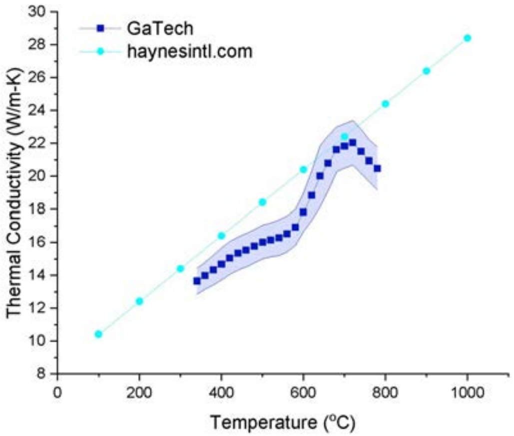




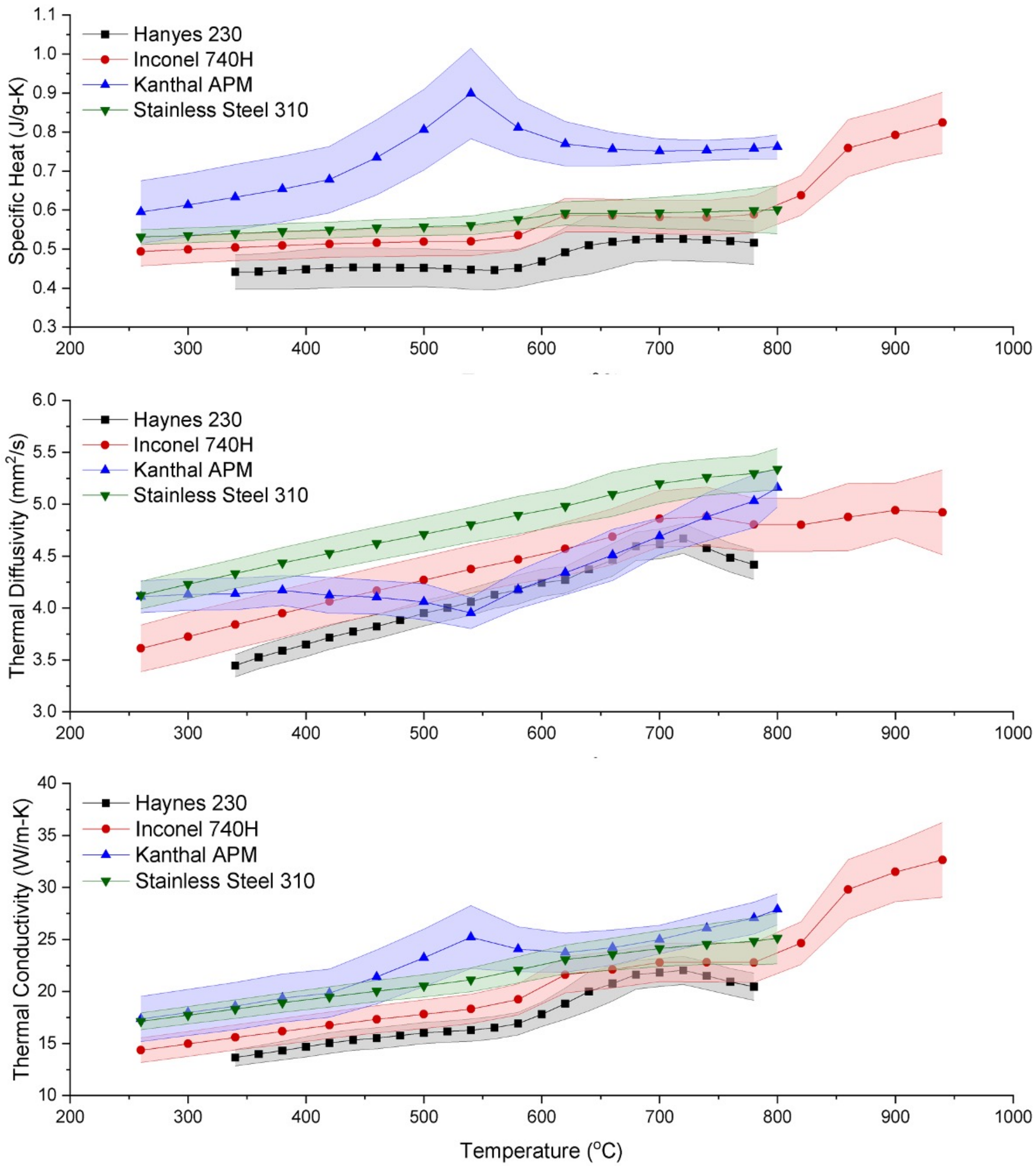
Haynes 230



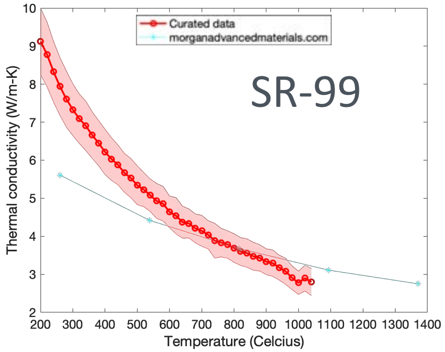




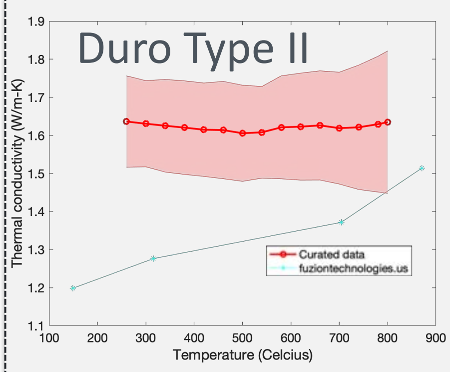
There are differences between materials within the same class

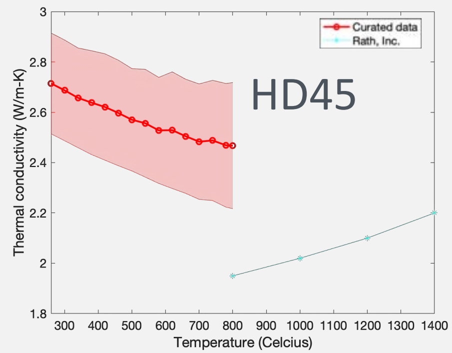


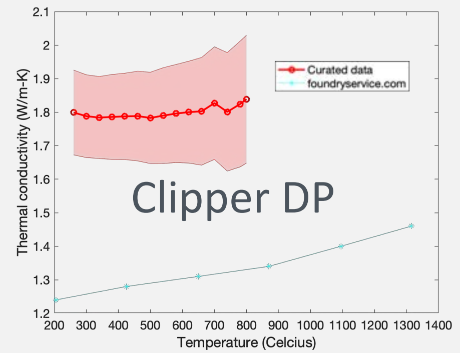
We have filled the knowledge gap for high temperature containment materials



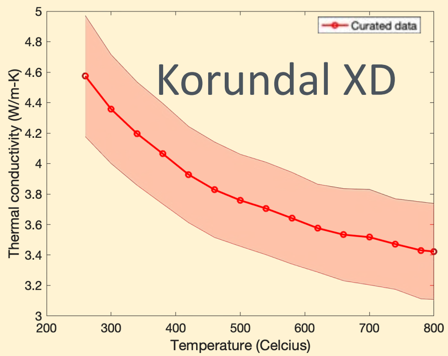
Measured data substantially different from supplier's spec sheet

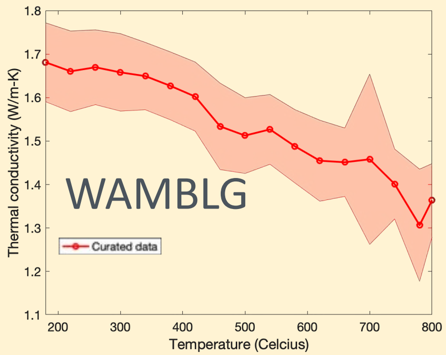


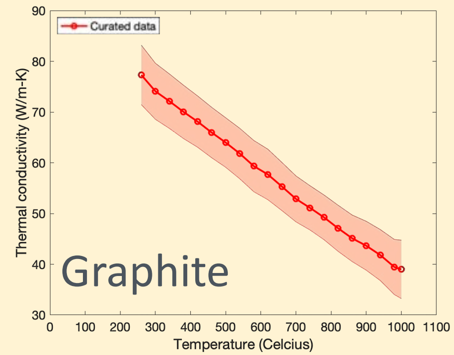


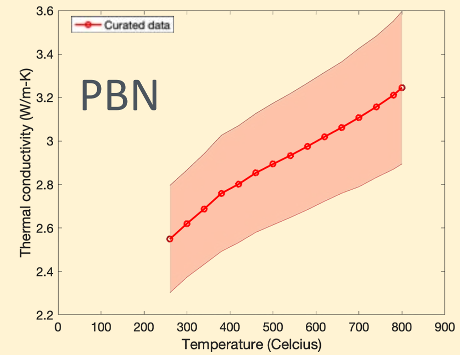


No Previous Data Available

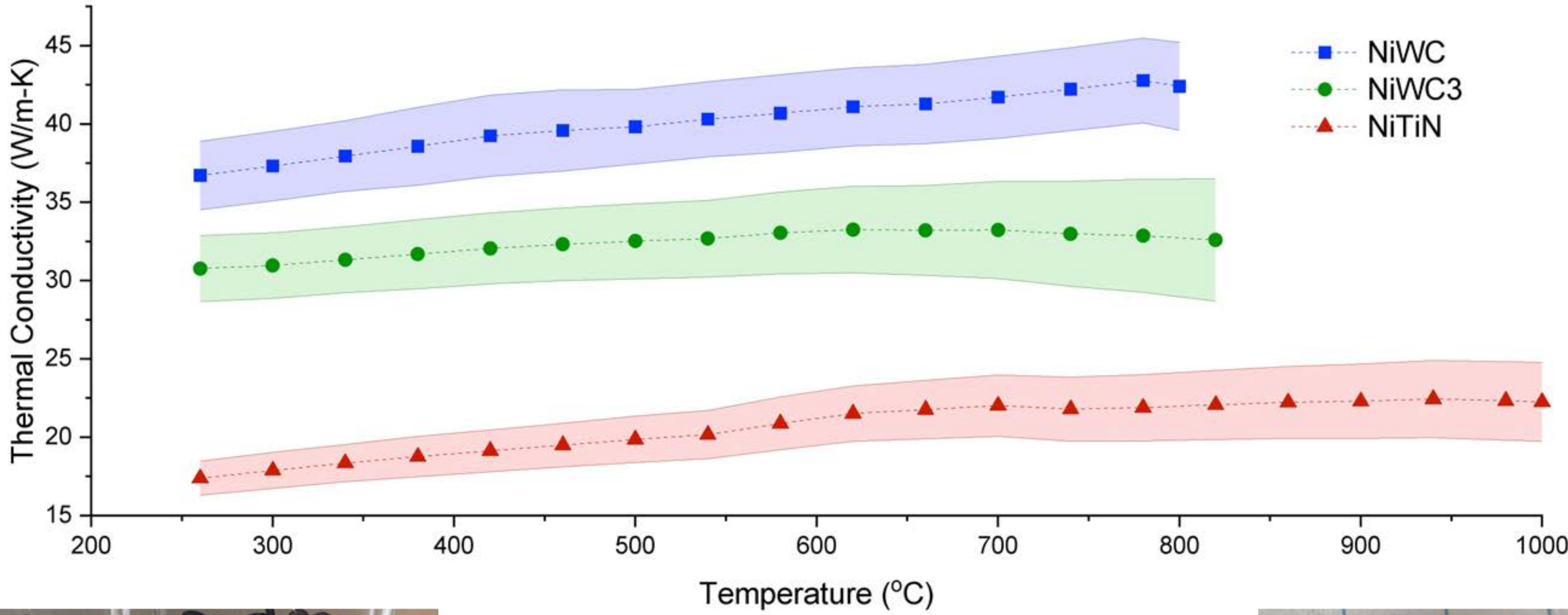


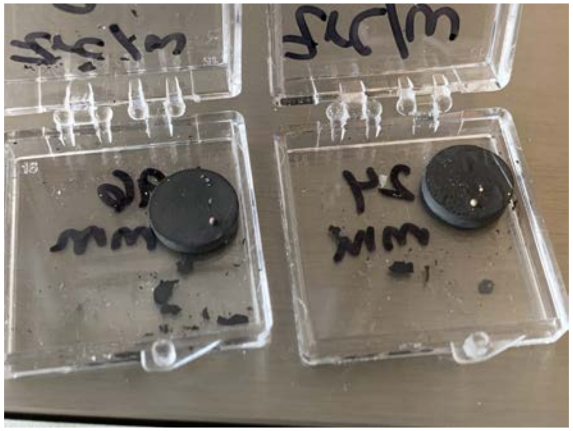






Cermets remain challenging to work with and measure

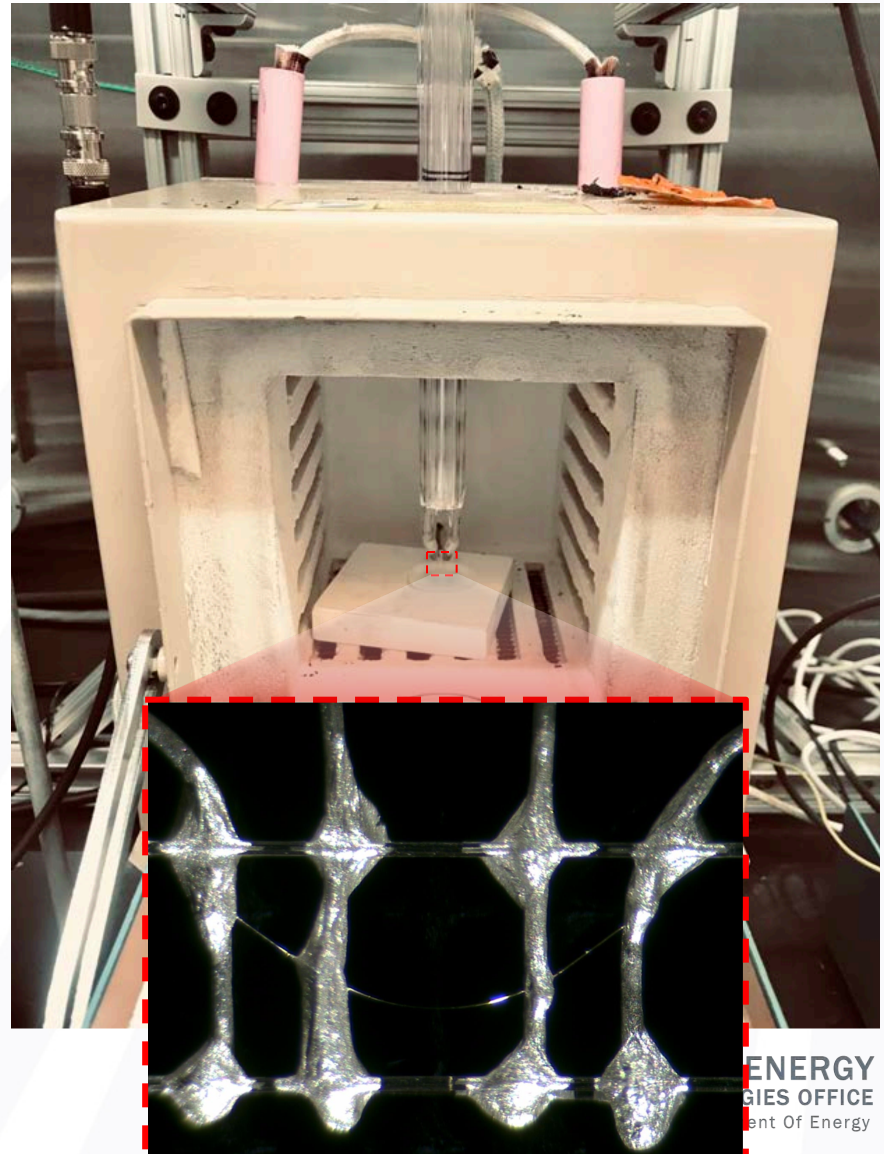




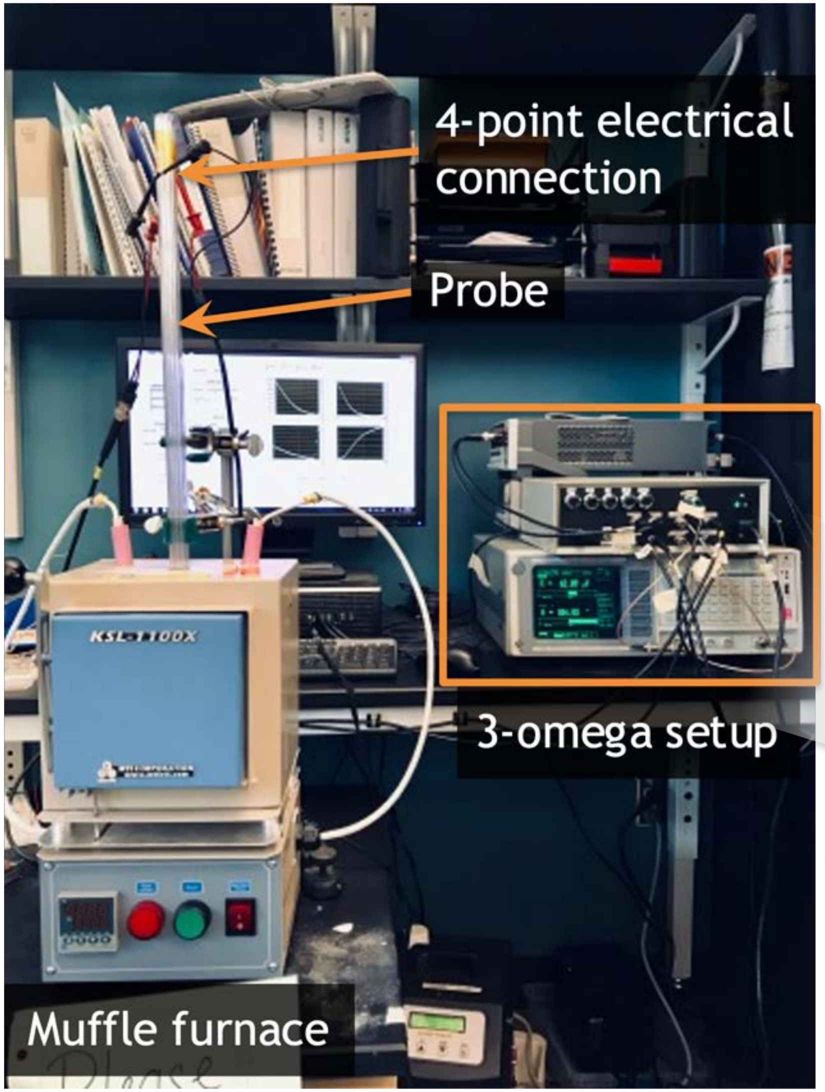
(Left) Bubbles formed on surface of ZrC/W cermet post testing

(Right) ZrC/W samples sintered to Pt crucible

Final Design of Immersion Probes



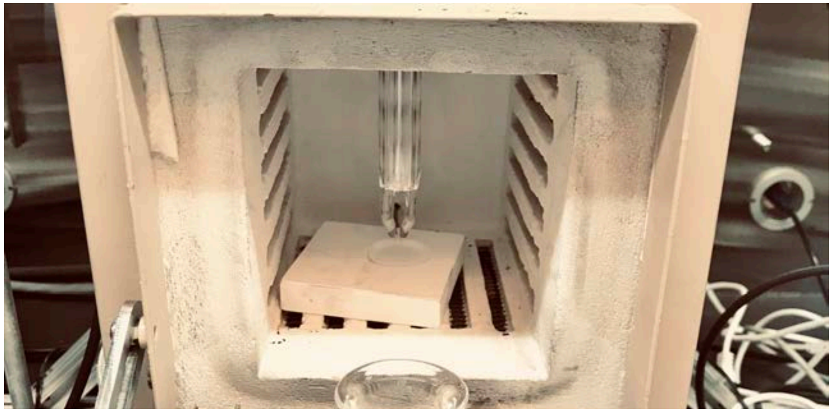
Final Immersion Probes Setup

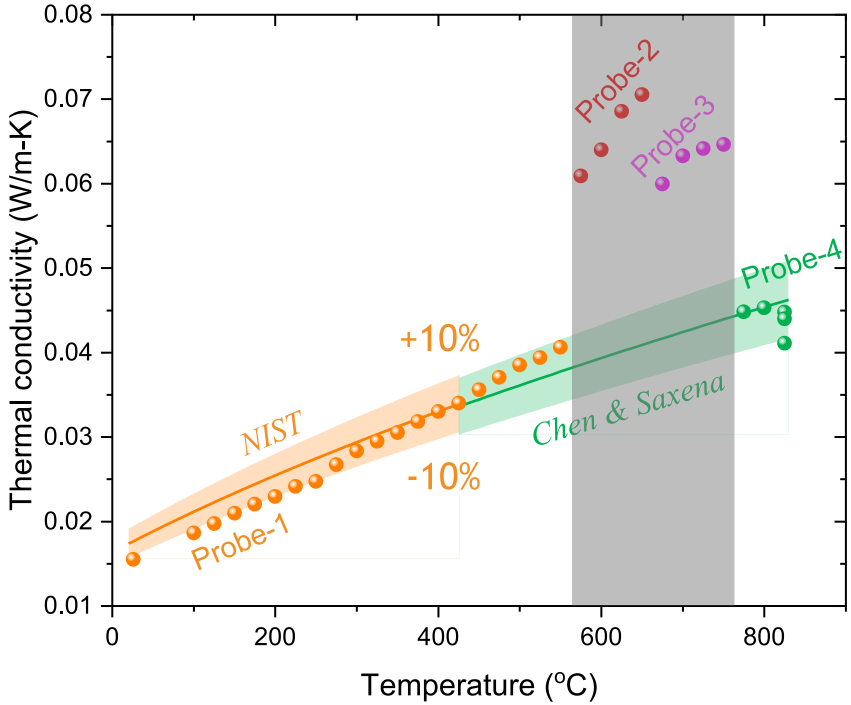


Accuracy of immersion sensors at elevated temperature

Sensor capable of measuring k of standard >800°C with <10% difference from literature†

Most k measurements are 10% accurate. If our measurement of standard (Ar gas) at elevated temperature is <10% accurate, it qualifies our immersion probe technique (*i.e.* it is on-parr or better than the SoA)





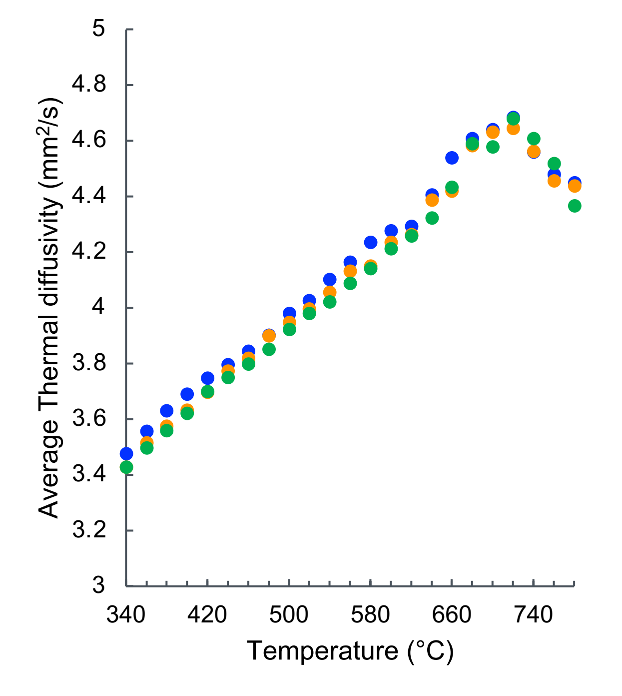
- k measurement of Ar between 26-550°C and 775-825°C is <10% accurate
- The 3-ω measurement at 825°C were repeated three (3) times
- Accuracy varied between probes, which may be caused by variation during fabrication
- Accuracy of C_P of the ceramic core is very important for phase-fitting (based on sensitivity analysis)

†Chen & Saxena, *Molecular Physics*, 29 (2), pp.455-466, 1975

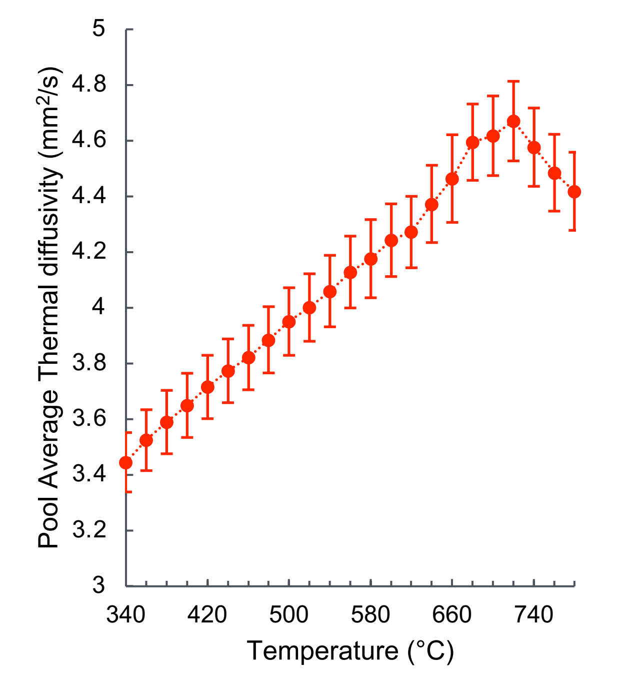
Lessons learned

- High temperature molten salts are very challenging
  - Corrosive nature, unexpected volatility, material mismatch (in everyway imaginable), special skill sets and equipment requirements, and data analysis non-trivial
- Cermets are non-stable composites
- Ni alloys exhibit high temperature phase transitions not previously captured in supplier spec sheets
- Oxidation, corrosion, or sublimation of high temperature materials (e.g., graphite foams) limit use
- Data discrepancies with material supplier spec sheets
- Filled the knowledge gap of high temperature data
- Confident in measuring CM properties accurately and precisely

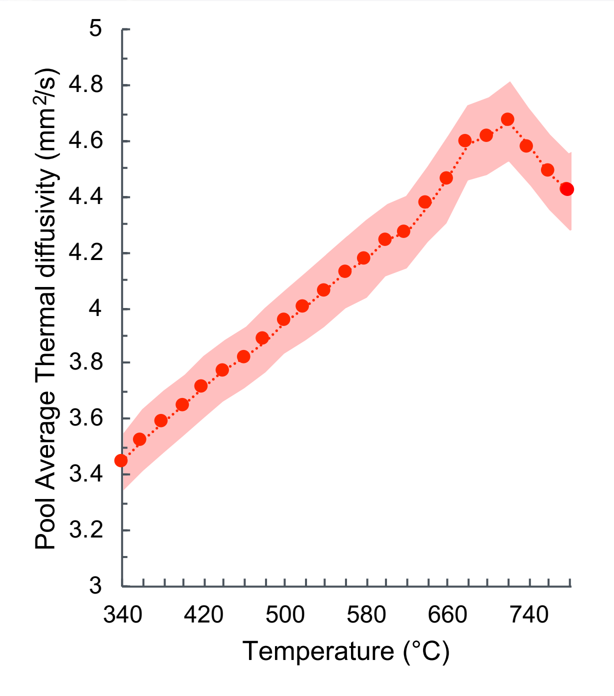
Uncertainty Analysis at 95% Confidence Level:† Thermal Diffusivity Measurement with LFA



Raw data from 3 measurements with 3 different samples (from 3 different location on the same block of CM material)



Pool average (marker) with 95% uncertainty (error bars) estimated from Uncertainty Analysis*



We are using shaded error bars/band on the website

†Gunawan, "Uncertainty Analysis and Error Propagation Methodology for Reporting Thermophysical Properties Measurement of Gen3 CSP Materials", *OSTI ID: 1719142*

*Figliola & Beasley, "Theory and Design for Mechanical Measurements", *5th Ed.*, Wiley 2010 (Chapter 5)

Uncertainty Analysis at 95% Confidence Level:† Thermal Diffusivity Measurement with LFA (cont'd)

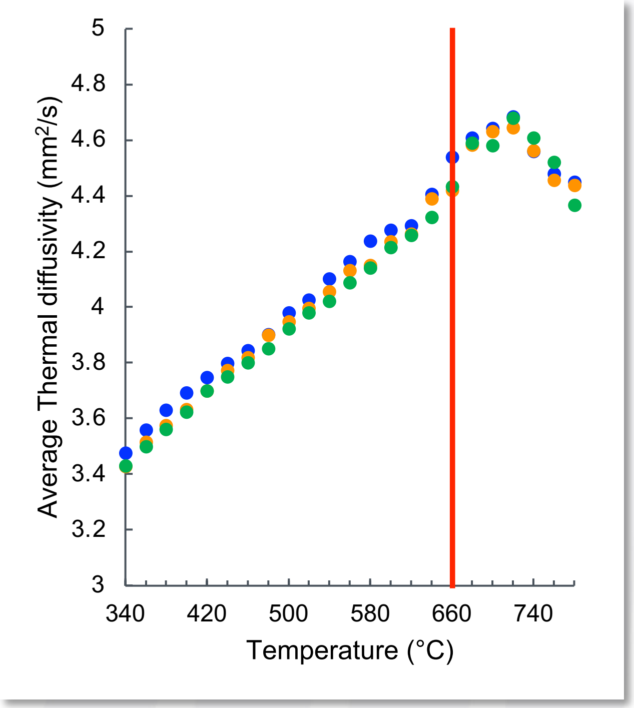
This approach adhere to the American National Standard Institute/American Society of Mechanical Engineers (ANSI/ASME) Power Test Codes (PTC) 19.1 Test Uncertainty (2), which is the U.S. engineering test standard*

- The mean value (at each temperature) from 3 different samples are averaged to yield a mean thermal diffusivity (α) of the bulk material using **pooled averaging** (Ch. 4*)

$$\langle \bar{\alpha} \rangle = \frac{1}{3} \sum_{m=1}^{3} \bar{\alpha}_m = 4.464\ \mathrm{mm^2/s}$$

- Errors for the LFA measurement are due to (1) instrument error, (2) spatial variation errors, and (3) temporal variation errors.
- First, consider the **instrument error**. The instrument error is assigned a systematic uncertainty (only) based on the manufacturer's (Netzsch's) statement of ± 3% of the reading, which is assumed to be stated at 95% confidence. The standard uncertainties are assigned as

$$(b_{\bar{\alpha}})_1 = \frac{(0.03 \times 4.464)}{2} = 0.067\ \mathrm{mm^2/s} \qquad (s_{\bar{\alpha}})_1 = 0$$



†Gunawan, "Uncertainty Analysis and Error Propagation Methodology for Reporting Thermophysical Properties Measurement of Gen3 CSP Materials", *OSTI ID: 1719142*

*Figliola & Beasley, "Theory and Design for Mechanical Measurements", *5th Ed.*, Wiley 2010 (Chapter 5)

Uncertainty Analysis at 95% Confidence Level:† Thermal Diffusivity Measurement with LFA (cont'd)

-2- Key Technical Data STA 449 *F3 Jupiter®*	
Drift dynamic	10…20 µg (after TG baseline correction)
Sensor exchange	Yes, *Quick*-Connect connection
Sensor (DSC/DTA) data	
Sensor types	Quickly exchangeable sensors (see table below) ▪ Standard DSC ▪ Optional: TG, DTA, DSC- c_p
Sensor data	See table below for detection limit, time constant, enthalpy accuracy and precision, DSC measuring range
c_p	0 ... 5 J/(g*K)
c_p accuracy/precision	▪ -150°C to 700°C: ± 1.0% ▪ RT to 1000°C: ± 2.5% ▪ RT to 1500°C: ± 3.5%
TAWN	▪ Sensitivity: > 3 (heating rate 0.1 K/min) ▪ Resolution: < 0.8 (heating rate 20 K/min) ➔ Data is valid for steel furnace (TAWN tests require c linear cooling) and TGA-DSC sensors type P and K.
Atmosphere	
Gas atmospheres	Inert, oxidizing, reducing, corrosive gases (non toxic, n mable), vacuum, static, dynamic

	LFA 467 *HT HyperFlash®*
Temperature range	RT ... 1250°C (furnace temperature 1500°C)
Heating rate (max.)	50 K/min
Furnace cooling device	External chiller
Thermal diffusivity	0.01 mm²/s ... 2000 mm²/s
Thermal conductivity	0.1 W/(m·K) ... 4000 W/(m·K)
Accuracy	▪ Thermal diffusivity[1]: ± 3% ▪ Specific heat[2]: ± 5%

Uncertainty Analysis at 95% Confidence Level:† Thermal Diffusivity Measurement with LFA (cont'd)

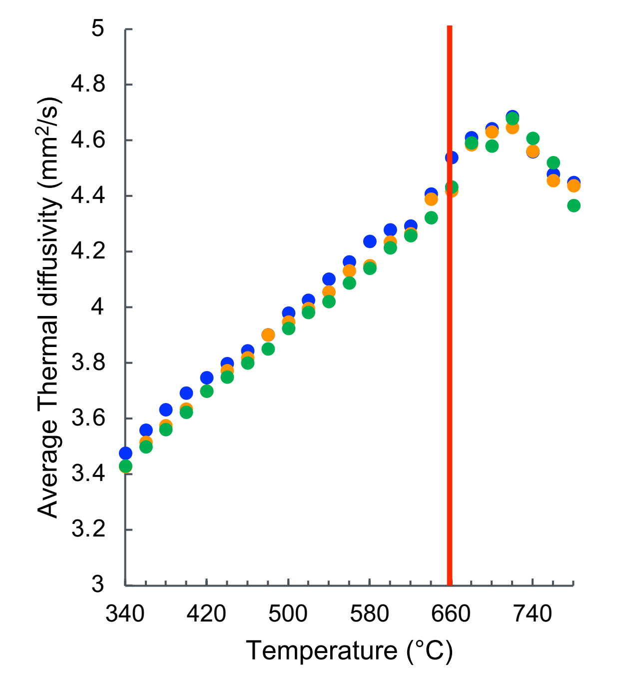
- Consider next the spatial error contribution to the estimate of the mean thermal diffusivity $\bar{\alpha}$. This error arises from the spatial uniformity in the bulk material. An estimate of spatial α distribution within the bulk material can be made by examining the mean α of the 3 measured samples from 3 different location in the bulk material. The mean α within the bulk material show a standard deviation of

$$s_\alpha = \sqrt{\frac{\sum_{m=1}^{3}(\bar{\alpha}_m - \langle\bar{\alpha}\rangle)^2}{2}} = 0.065\ \mathrm{mm^2/s}$$

Thus, the random standard uncertainty of the α is found from:

$$(s_{\bar{\alpha}})_2 = \frac{s_\alpha}{\sqrt{3}} = 0.038\ \mathrm{mm^2/s}$$

with degrees of freedom, $\nu = 2$. We do not assign a systematic uncertainty to this error, so $(b_{\bar{\alpha}})_2 = 0$.



†Gunawan, "Uncertainty Analysis and Error Propagation Methodology for Reporting Thermophysical Properties Measurement of Gen3 CSP Materials", *OSTI ID: 1719142*

*Figliola & Beasley, "Theory and Design for Mechanical Measurements", *5th Ed.*, Wiley 2010 (Chapter 5)

Uncertainty Analysis at 95% Confidence Level:† Thermal Diffusivity Measurement with LFA (cont'd)

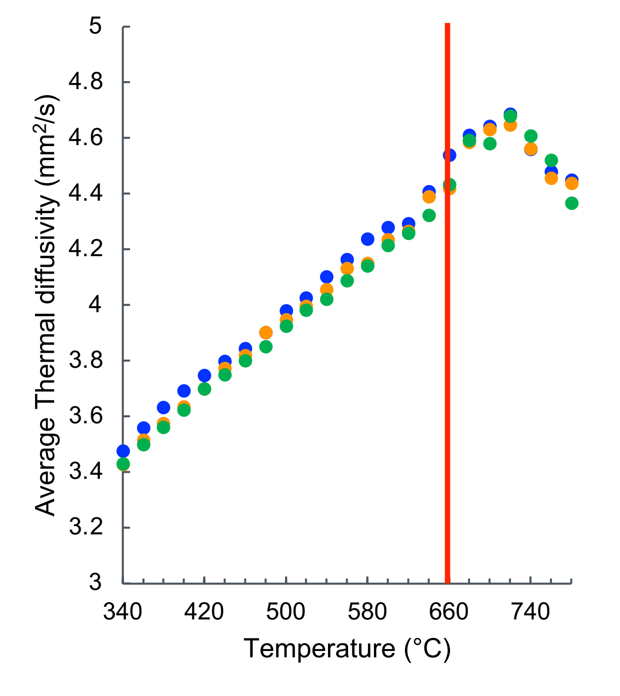
- **Time variation error** in α output during each of the 3 flashes at each temperature (for each sample) cause data scatter. Such time variations are caused by random local α variations as measured by the LFA sensor, sensor resolution, and furnace temperature control variation during fixed operating conditions. Since we have insufficient information to separate these, they are estimated together as a single error. The pooled standard deviation is

$$\langle s_\alpha \rangle = \sqrt{\frac{\sum_{m=1}^{3}\sum_{n=1}^{3}(\bar{\alpha}_{mn} - \langle\bar{\alpha}\rangle)^2}{M(N-1)}} = \sqrt{\frac{1}{M}\sum_{m=1}^{3} s_{\bar{\alpha}_m}^2} = 0.038\ \mathrm{mm^2/s}$$

to give a random standard uncertainty of

$$(s_{\bar{\alpha}})_3 = \frac{\langle s_\alpha \rangle}{\sqrt{9}} = 0.013\ \mathrm{mm^2/s}$$

with degrees of freedom, $\nu = 6$. We assign $(b_{\bar{\alpha}})_3 = 0$.



†Gunawan, "Uncertainty Analysis and Error Propagation Methodology for Reporting Thermophysical Properties Measurement of Gen3 CSP Materials", *OSTI ID: 1719142*

*Figliola & Beasley, "Theory and Design for Mechanical Measurements", *5th Ed.*, Wiley 2010 (Chapter 5)

Uncertainty Analysis at 95% Confidence Level:† Thermal Diffusivity Measurement with LFA (cont'd)

- The **measurement systematic standard uncertainty** is

$$b_{\bar{\alpha}} = [(b_{\bar{\alpha}})_1^2 + (b_{\bar{\alpha}})_2^2 + (b_{\bar{\alpha}})_3^2]^{1/2} = 0.067\ \mathrm{mm^2/s}$$

and the **measurement random standard uncertainty** of

$$s_{\bar{\alpha}} = [(s_{\bar{\alpha}})_1^2 + (s_{\bar{\alpha}})_2^2 + (s_{\bar{\alpha}})_3^2]^{1/2} = 0.040\ \mathrm{mm^2/s}$$

with degrees of freedom are found using:

$$\nu = \frac{\left(\sum_{k=1}^{K}(s_{\bar{\alpha}}^2)_k + (b_{\bar{\alpha}}^2)_k\right)^2}{\sum_{k=1}^{K}((s_{\bar{\alpha}}^4)_k/\nu_k) + \sum_{k=1}^{K}((b_{\bar{\alpha}}^4)_k/\nu_k)} = 36$$

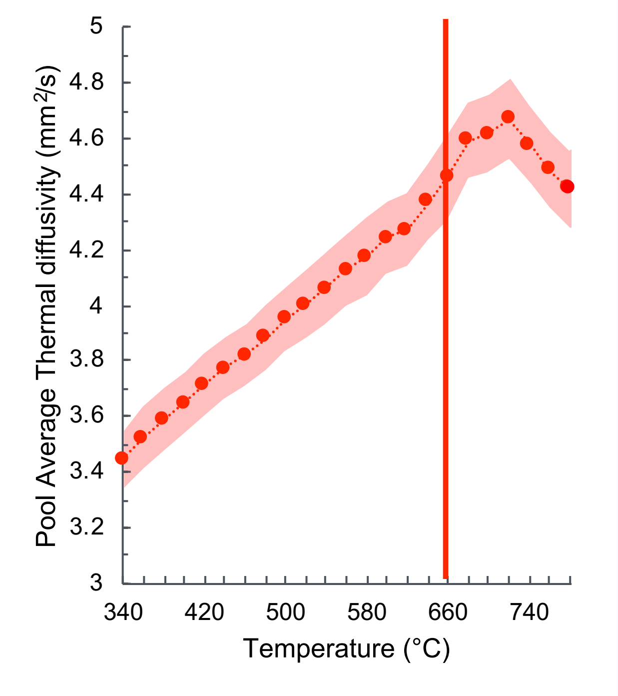
Note that when the ν in the systematic uncertainties are large, the second term in the denominator is small.

- The combined standard uncertainty in the mean α is

$$u_\alpha = \left[b_{\bar{\alpha}}^2 + s_{\bar{\alpha}}^2\right]^{1/2} = 0.078\ \mathrm{mm^2/s}$$

- Assigning $t_{36,95} = 2.021$ (from Table 4.4*), **the best estimate of the mean α with 95% confidence** is

$$\alpha' = \bar{\alpha} \pm t_{29,95}\left[b_{\bar{\alpha}}^2 + s_{\bar{\alpha}}^2\right]^{1/2} = 4.464 \pm 0.158\ \mathrm{mm^2/s}\ (< 15\%\ )$$



†Gunawan, "Uncertainty Analysis and Error Propagation Methodology for Reporting Thermophysical Properties Measurement of Gen3 CSP Materials", *OSTI ID: 1719142*

*Figliola & Beasley, "Theory and Design for Mechanical Measurements", *5th Ed.*, Wiley 2010 (Chapter 5)

Propagation of Uncertainty (at 95% Confidence Level) to a Result:†

Thermal Conductivity Best Estimation

- To estimate thermal conductivity, $k = \alpha \times c_p \times \rho$, once we have
  - $\alpha' = \bar{\alpha} \pm t_{\nu,95} u_\alpha$
  - $c_p' = \overline{c_p} \pm t_{\nu,95} u_{c_p}$
  - $\rho = \text{constant}$

  here we assume a negligible systematic and random errors in ρ
- The random and systematic standard uncertainties propagate through to the result (k), calculating about the operating point as stablished by the mean values for α and c_p. That is,

$$s_{\bar{k}} = \left[\left(\frac{\partial k}{\partial \alpha} s_{\bar{\alpha}} \right)^2 + \left(\frac{\partial k}{\partial c_p} s_{\overline{c_p}} \right)^2 \right]^{1/2}$$

and

$$b_{\bar{k}} = \left[\left(\frac{\partial k}{\partial \alpha} b_{\bar{\alpha}} \right)^2 + \left(\frac{\partial k}{\partial c_p} s_{\overline{c_p}} \right)^2 \right]^{1/2}$$

†Gunawan, "Uncertainty Analysis and Error Propagation Methodology for Reporting Thermophysical Properties Measurement of Gen3 CSP Materials", *OSTI ID: 1719142*

*Figliola & Beasley, "Theory and Design for Mechanical Measurements", *5th Ed.*, Wiley 2010 (Chapter 5)

Propagation of Uncertainty (at 95% Confidence Level) to a Result:[†]
Thermal Conductivity Best Estimation

- The degrees of freedom in the k is determined by

$$\nu = \frac{\left[\left(\frac{\partial k}{\partial \alpha} s_{\bar{\alpha}}\right)^2 + \left(\frac{\partial k}{\partial c_p} s_{\overline{c_p}}\right)^2 + \left(\frac{\partial k}{\partial \alpha} b_{\bar{\alpha}}\right)^2 + \left(\frac{\partial k}{\partial c_p} b_{\overline{c_p}}\right)^2\right]^2}{\left[\left(\frac{\partial k}{\partial \alpha} s_{\bar{\alpha}}\right)^4 / \upsilon_{s_\alpha} + \left(\frac{\partial k}{\partial c_p} s_{\overline{c_p}}\right)^4 \Big/ \upsilon_{s_{c_p}}\right] + \left[\left(\frac{\partial k}{\partial \alpha} b_{\bar{\alpha}}\right)^4 / \upsilon_{b_\alpha} + \left(\frac{\partial k}{\partial c_p} b_{\overline{c_p}}\right)^4 \Big/ \upsilon_{b_{c_p}}\right]}$$

- **The best estimate of the thermal conductivity**, using $t_{\upsilon,95}$, can be reported as

$$k' = \bar{k} \pm t_{\upsilon,95}\left[b_{\bar{k}}^2 + s_{\bar{k}}^2\right]^{1/2} \quad (95\%)$$

- We can calculate the % measurement uncertainty:

$$\% = \frac{u_k}{\bar{k}} = \frac{t_{\upsilon,95}\left[b_{\bar{k}}^2 + s_{\bar{k}}^2\right]^{1/2}}{\bar{k}} \quad < 15\%$$

4. Uncertainty quantification individually reported with each measurement,
   a. Measurement uncertainty of less than 15% of the nominal value is preferred, but exceptions can be made for difficult/exotic materials.
   b. Monte-Carlo uncertainty is the preferred approach when using a multi-parameter thermal model; simplified quadrature uncertainty can be used when an analytical relation exists.
5. Reference to appropriate measurement standard or supporting[+] peer-reviewed publication detailing the measurement technique.

[†]Gunawan, "Uncertainty Analysis and Error Propagation Methodology for Reporting Thermophysical Properties Measurement of Gen3 CSP Materials", *OSTI ID: 1719142*

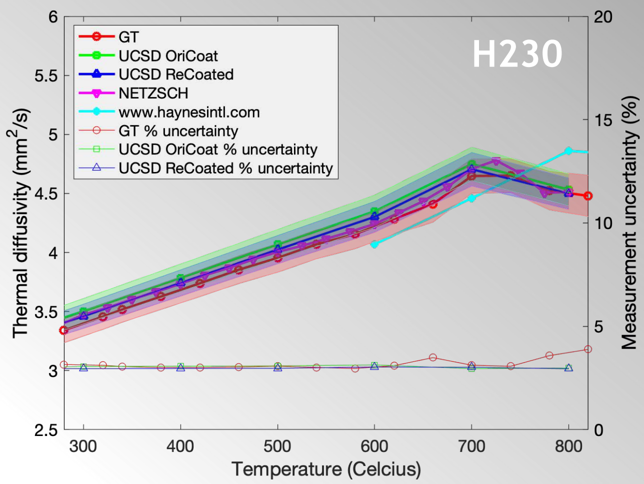
[+]Yee & Gunawan, "Measurement Acceptance Criteria for Reporting Thermophysical Properties Measurement of Gen3 CSP Materials: Standards, Principles, and Required Documentation", *OSTI ID: 1719141*

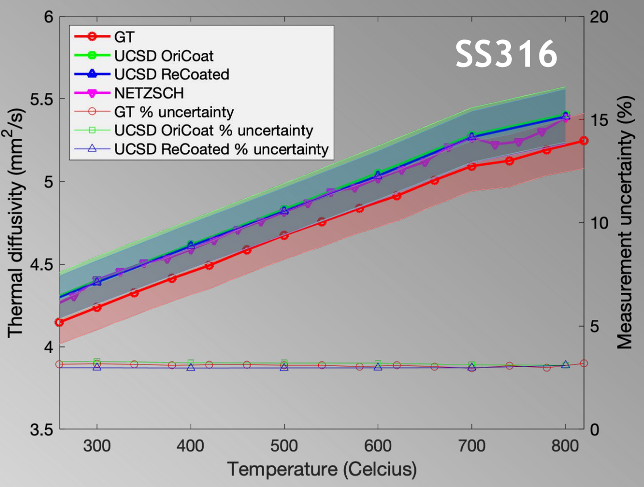
*Figliola & Beasley, "Theory and Design for Mechanical Measurements", 5th *Ed.*, Wiley 2010 (Chapter 5)

Round Robin Data from UCSD & Georgia Tech

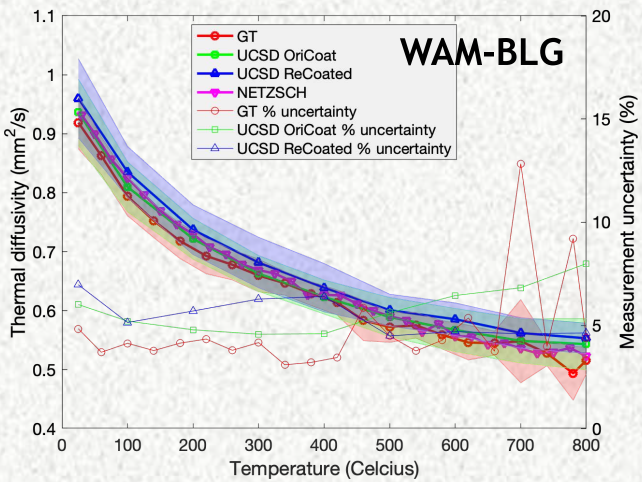
using Netzsch LFA 467 HT HyperFlash®

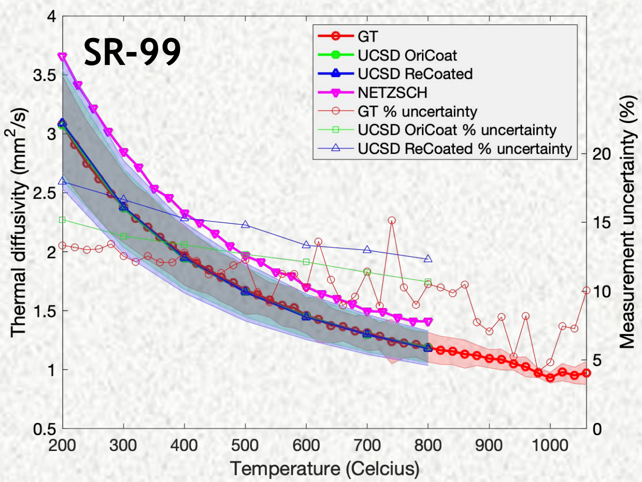
Metal



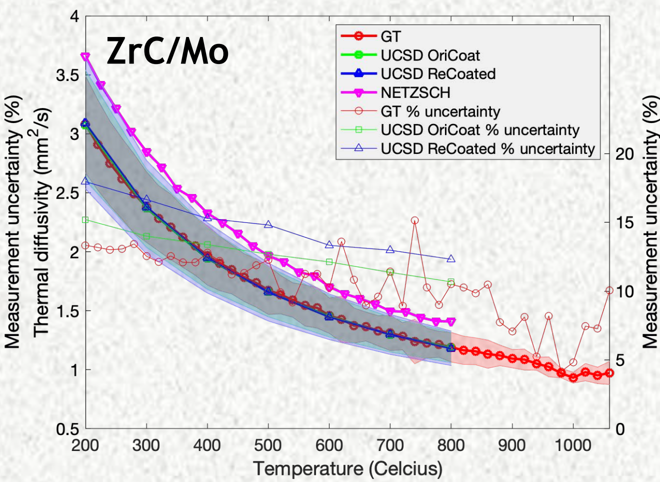


Ceramic





CerMet



SOLAR ENERGY
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THERMOPHYSICAL PROPERTY MEASUREMENTS OF HEAT TRANSFER MEDIA AND CONTAINMENT MATERIALS

Topic Area 2B: Gen3 Research & Analysis

Gen3 CSP Summit

Principal Investigator: Shannon K. Yee, Associate Professor
Co-PI: Andrey Gunawan, Research Engineer II

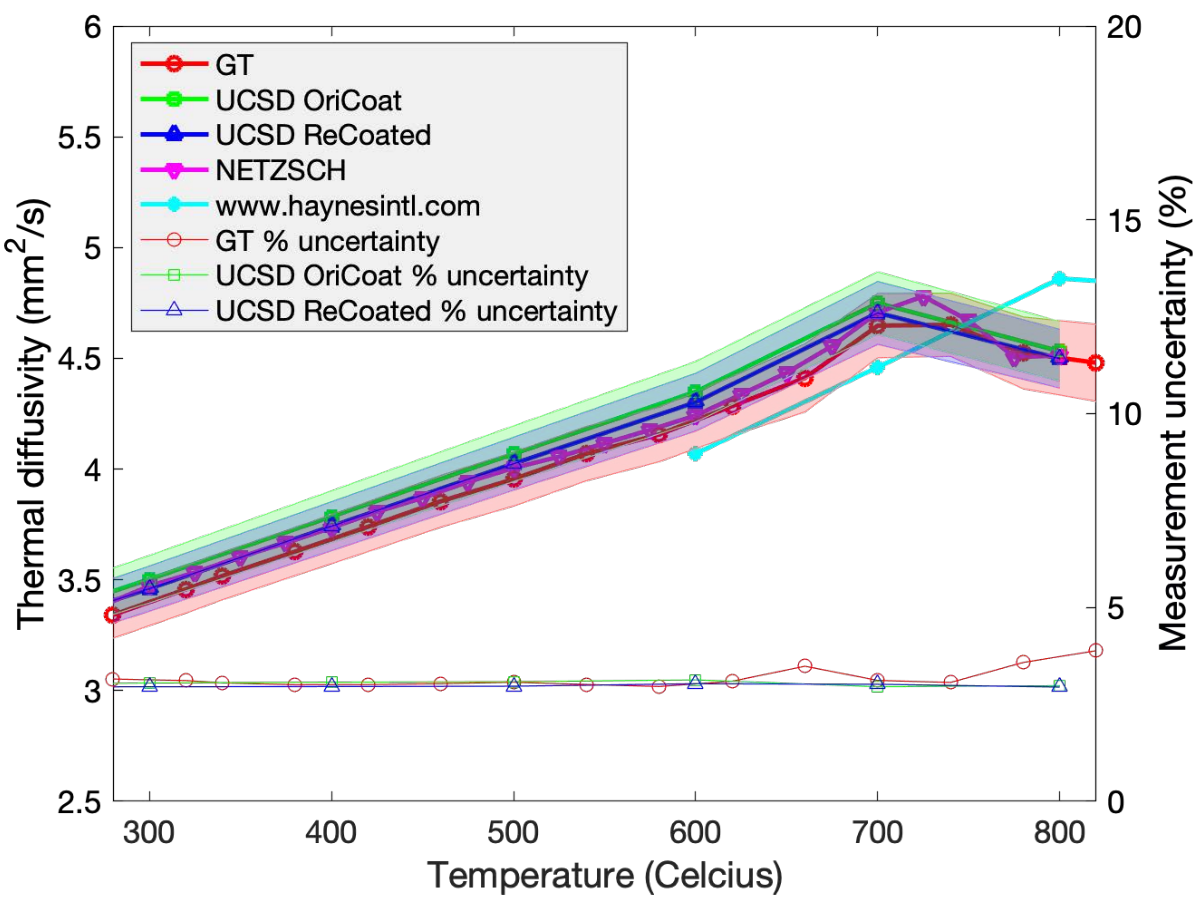
Thursday, August 26th 2021

Georgia Institute of Technology

SOLAR ENERGY
TECHNOLOGIES OFFICE
U.S. Department Of Energy

Additional slides

CM#2 Haynes 233 - comparison



SOLAR ENERGY TECHNOLOGIES OFFICE
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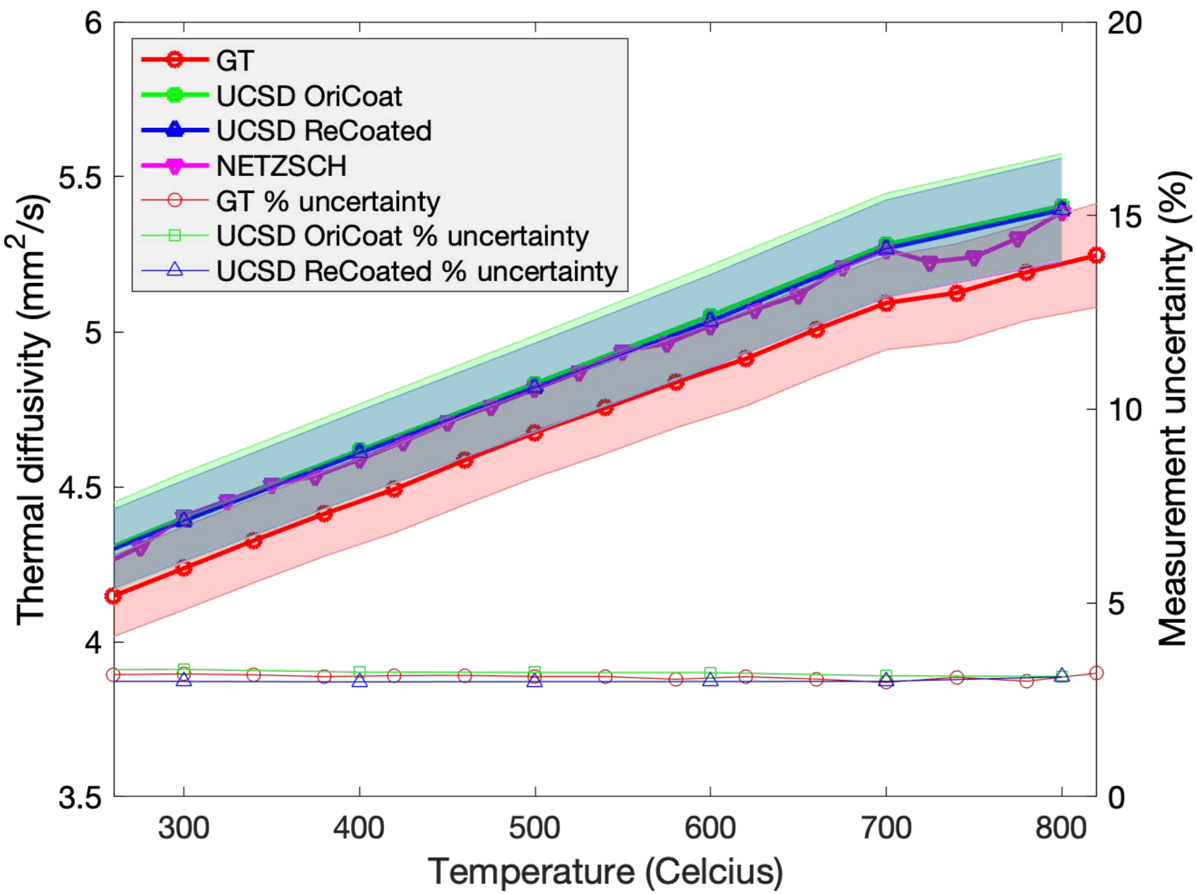
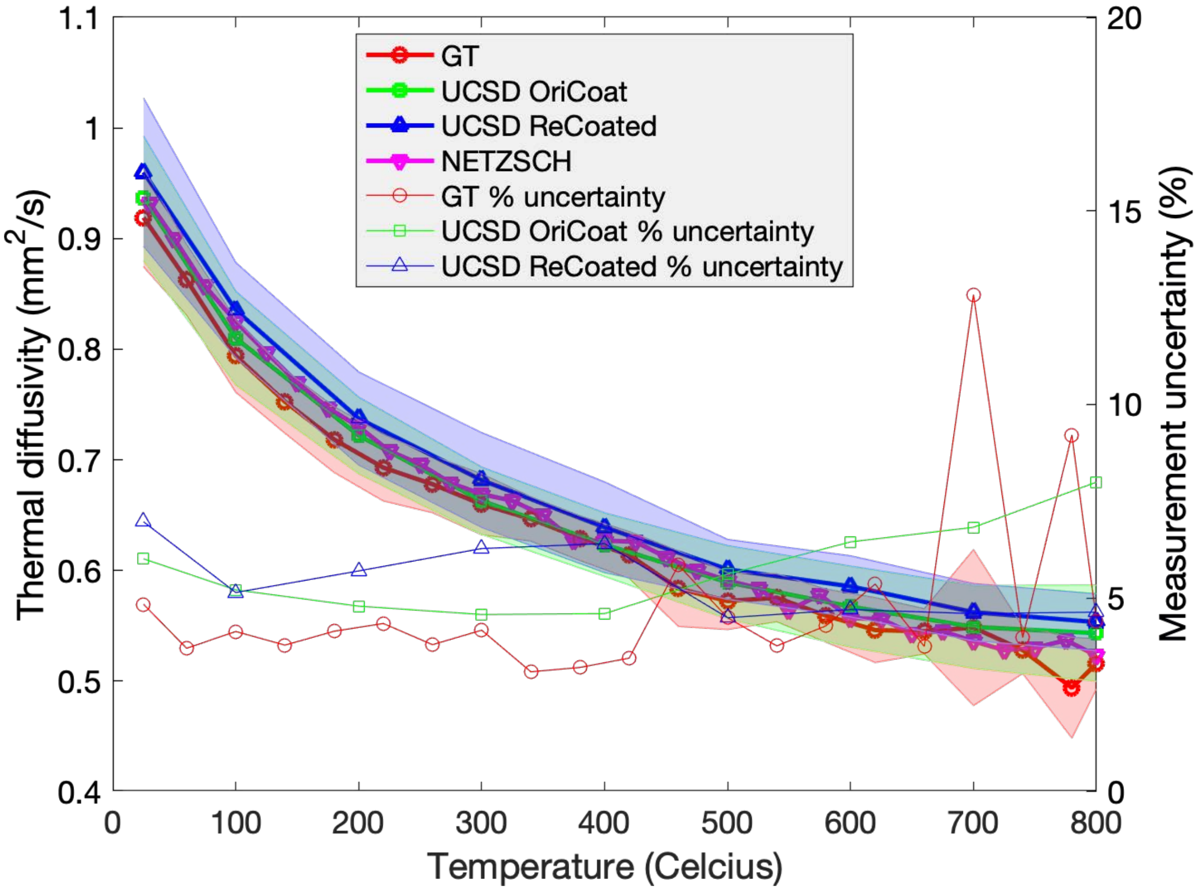
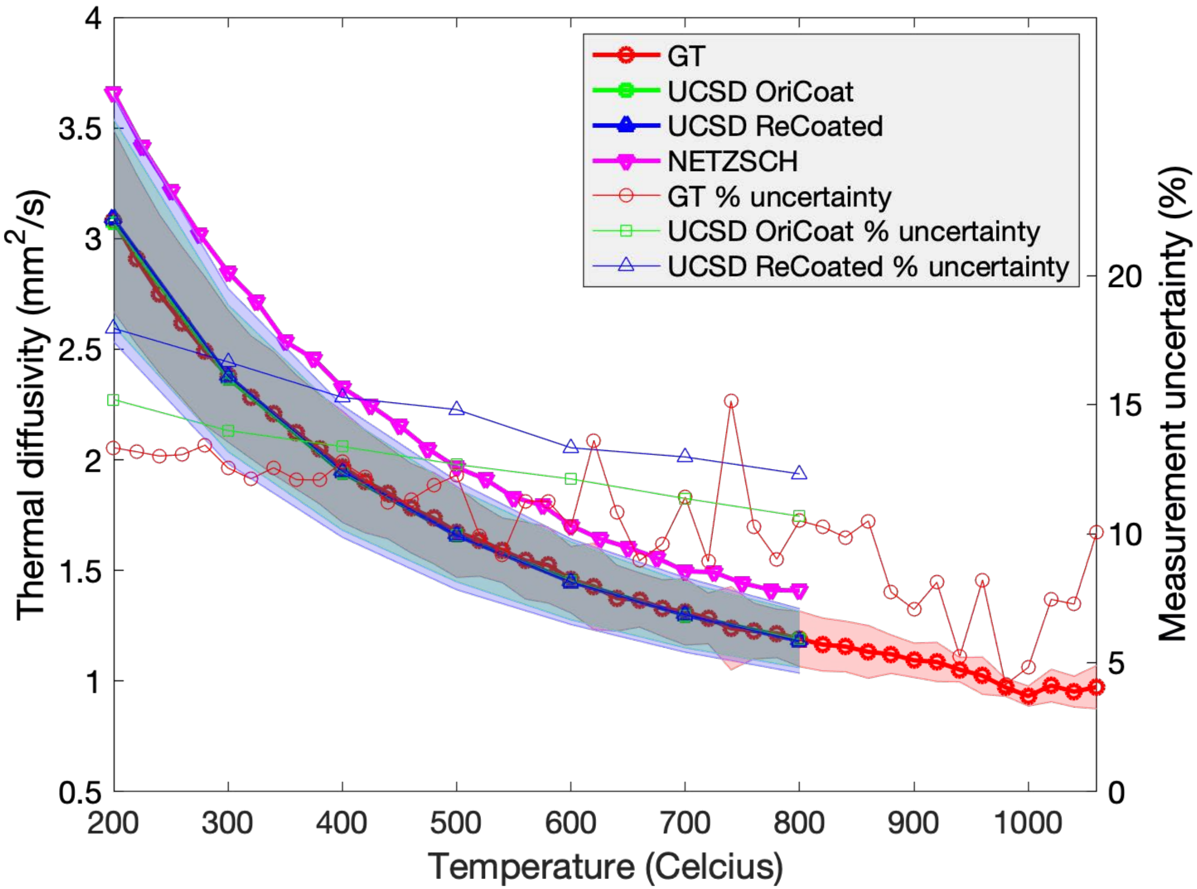
CM#12 Stainless Steel 316 – comparison



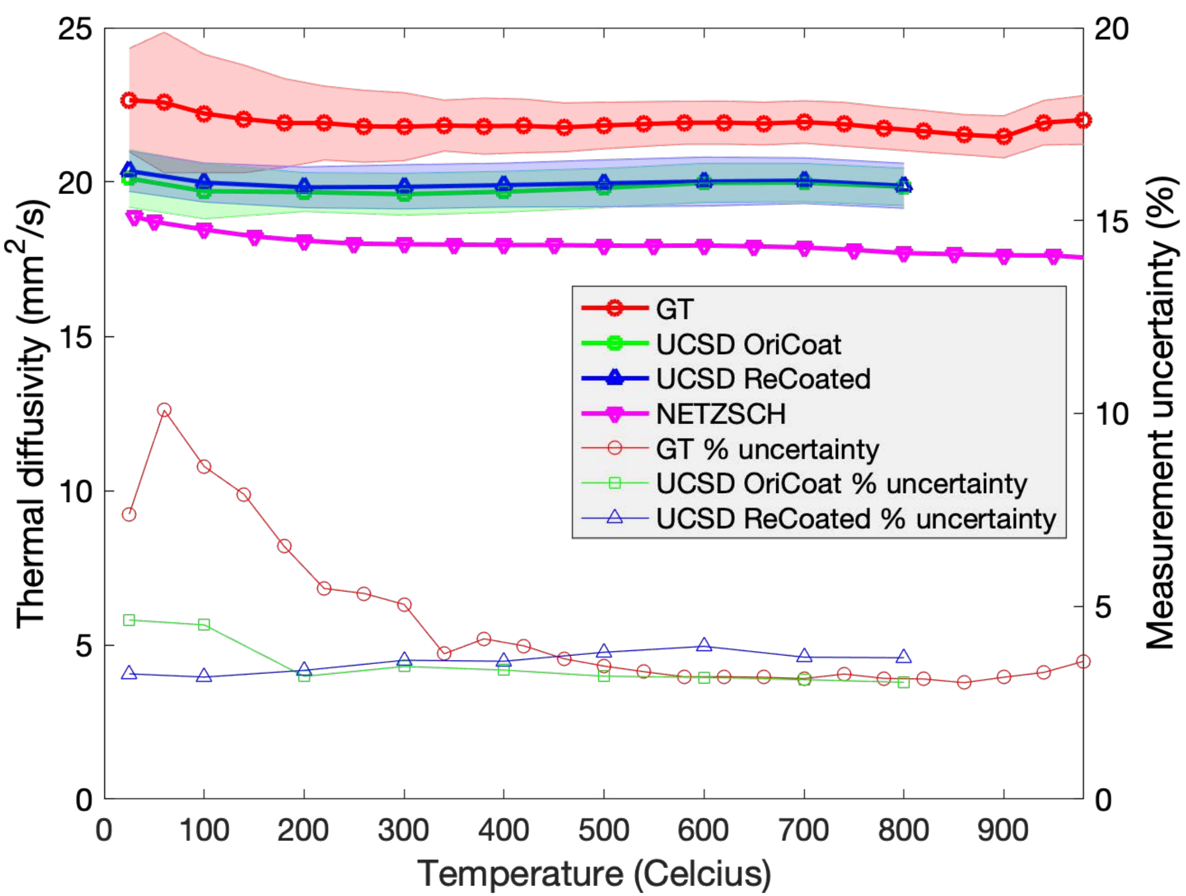
CM#4 WAM®-BLG - comparison (standard model)



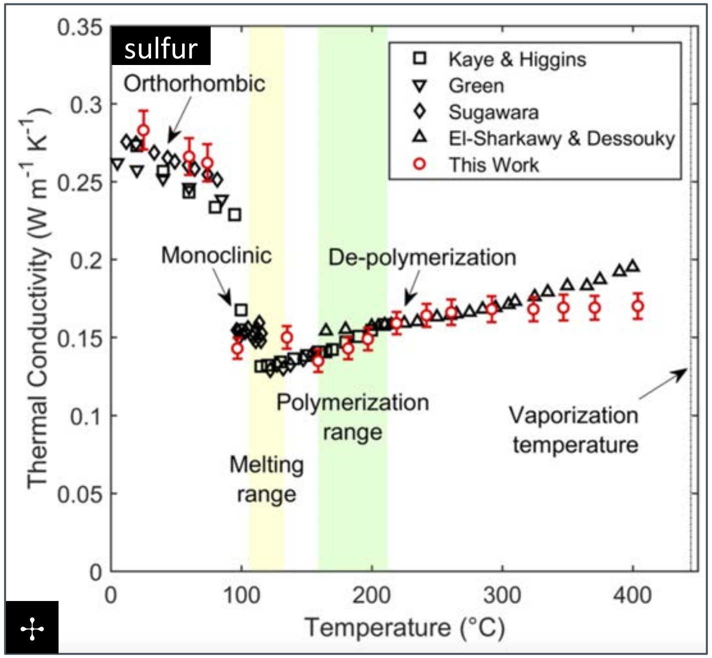
CM#11 SR-99 – comparison

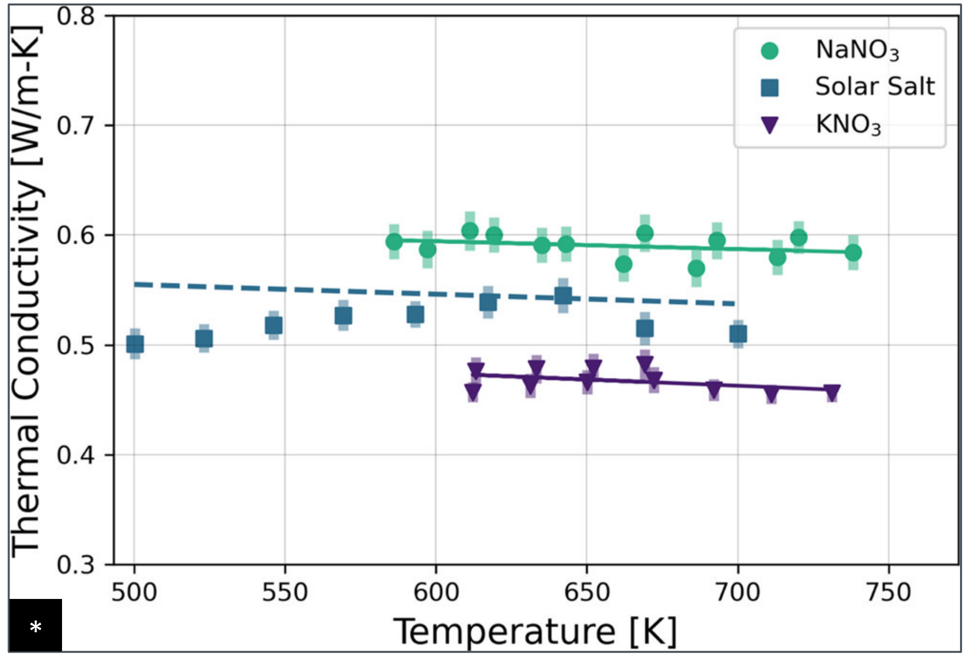


CM#*X* ZrC/Mo - comparison



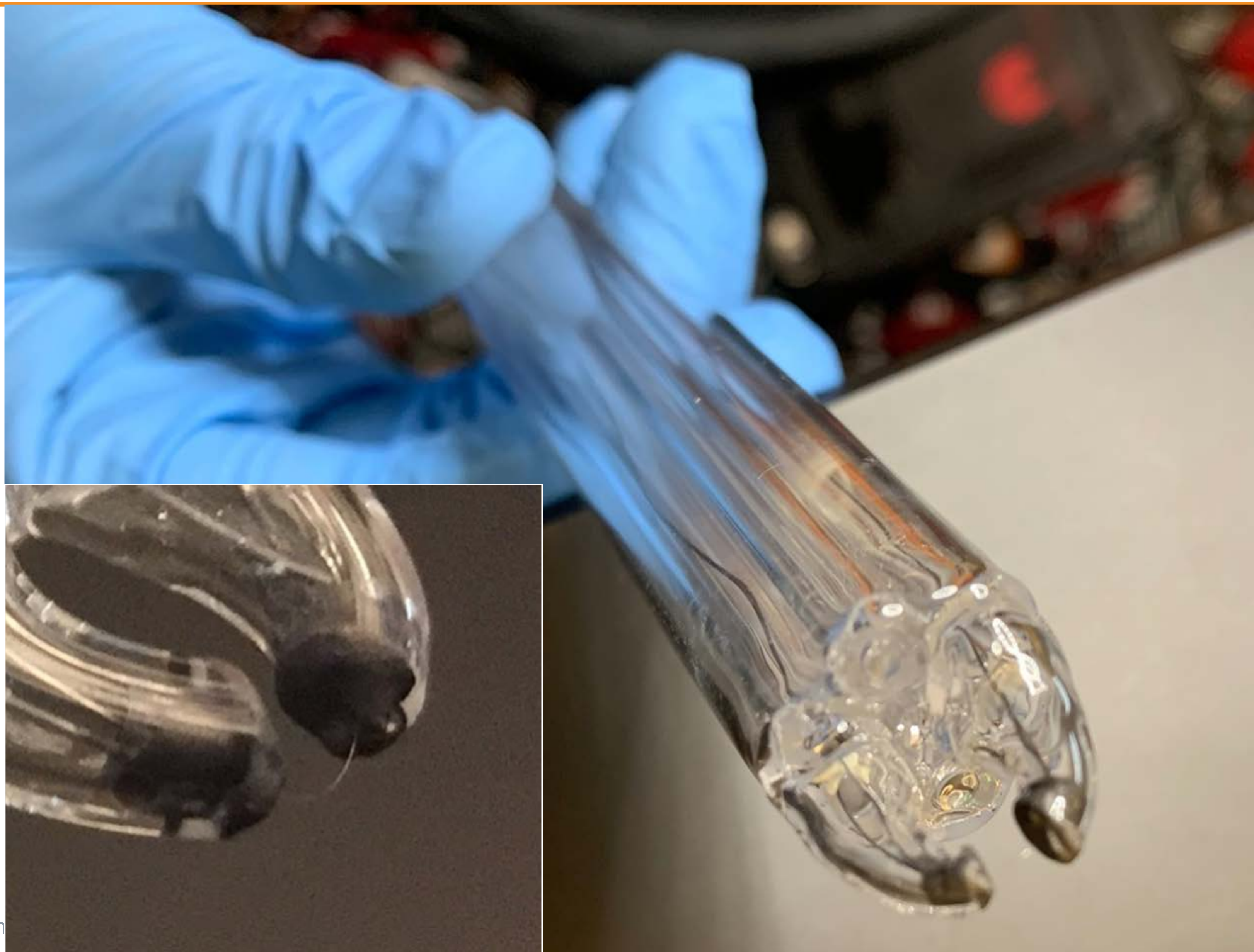
High-temperature molten salts are challenging to measure



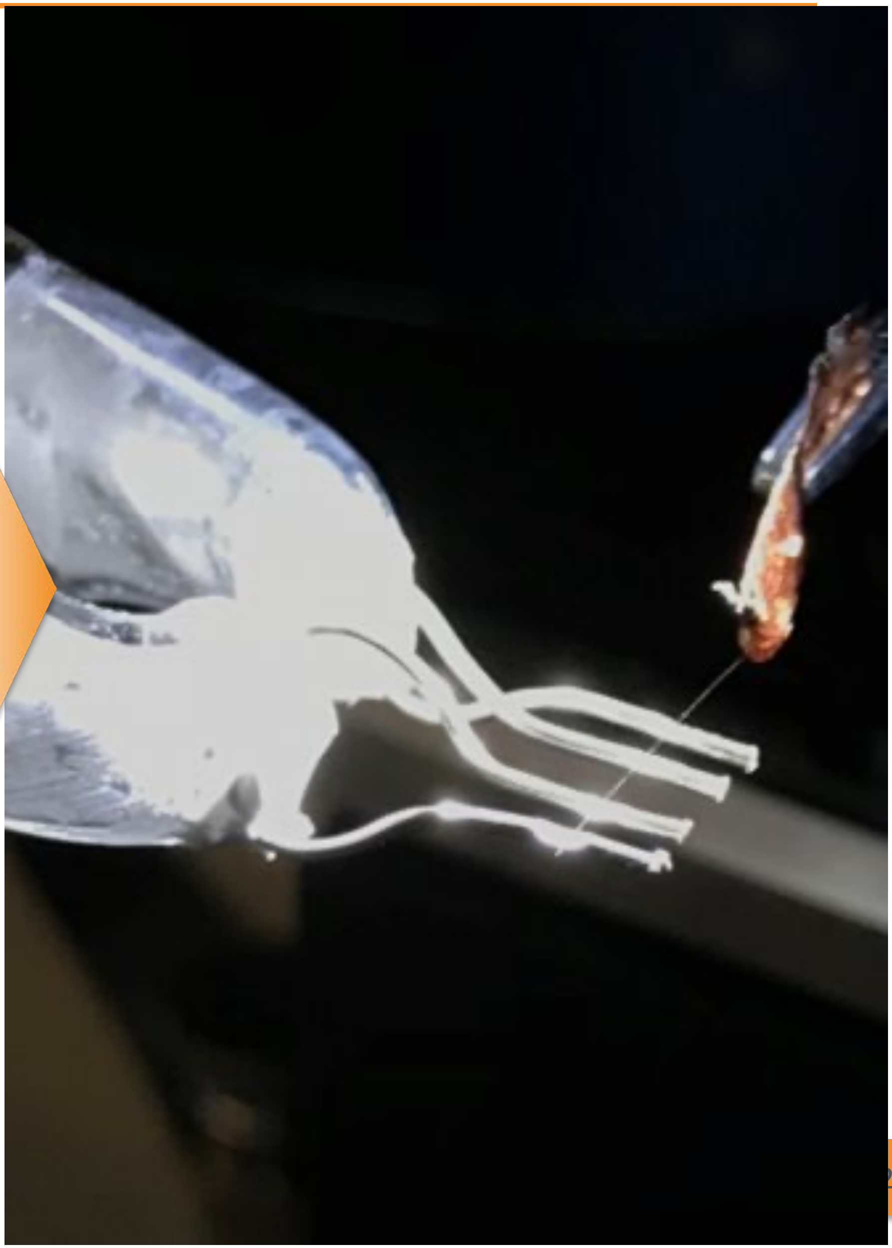


+Wingert, Zhao, Kodera, Obrey, Garay, *Rev. Sci. Instrum.*, 91(5), 2020 (https://doi.org/10.1063/1.5138915)
*Zhao, Wingert, Garay, *ACS J. Chem. Eng. Data.*, 2021 (https://dx.doi.org/10.1021/acs.jced.0c00621)
Wang, Rincon, Li, Zhao, Vidal, *ASME J Sol. Energy Eng.*, 143(4), 2020 (https://doi.org/10.1115/1.4049253)

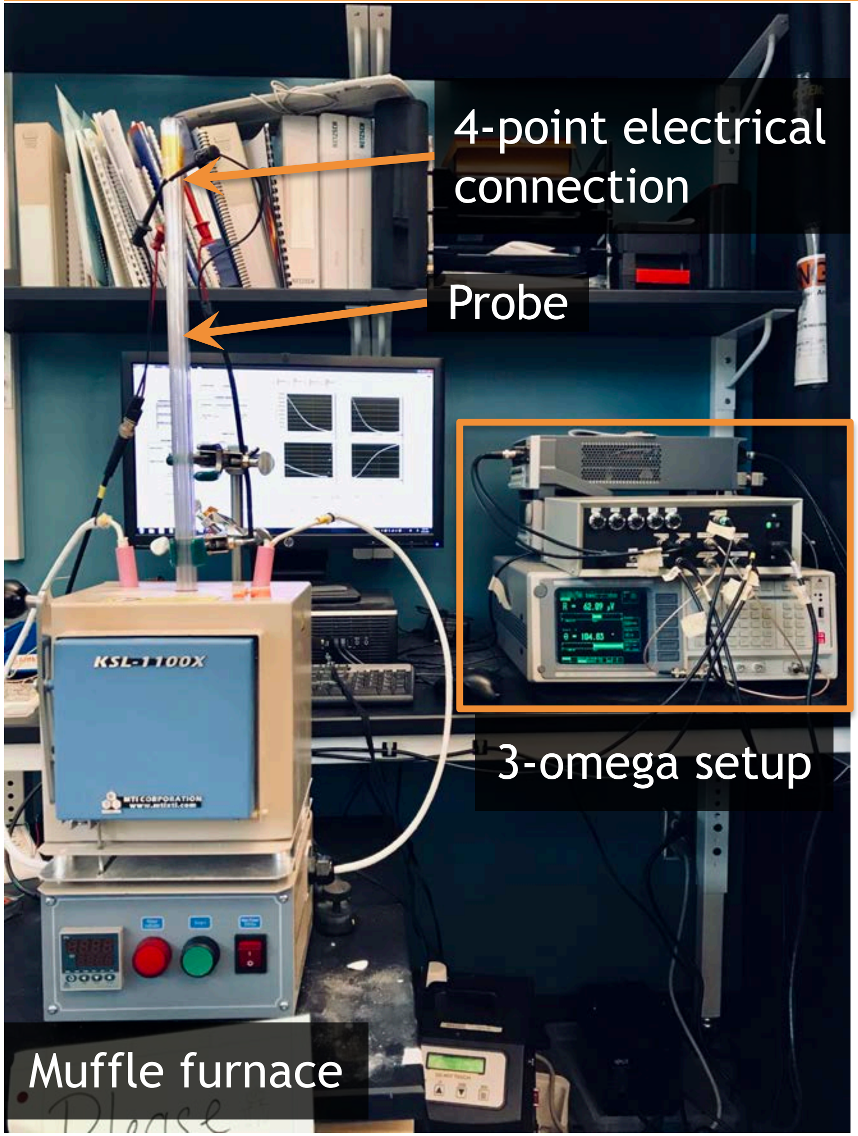
Design of Immersion Probes

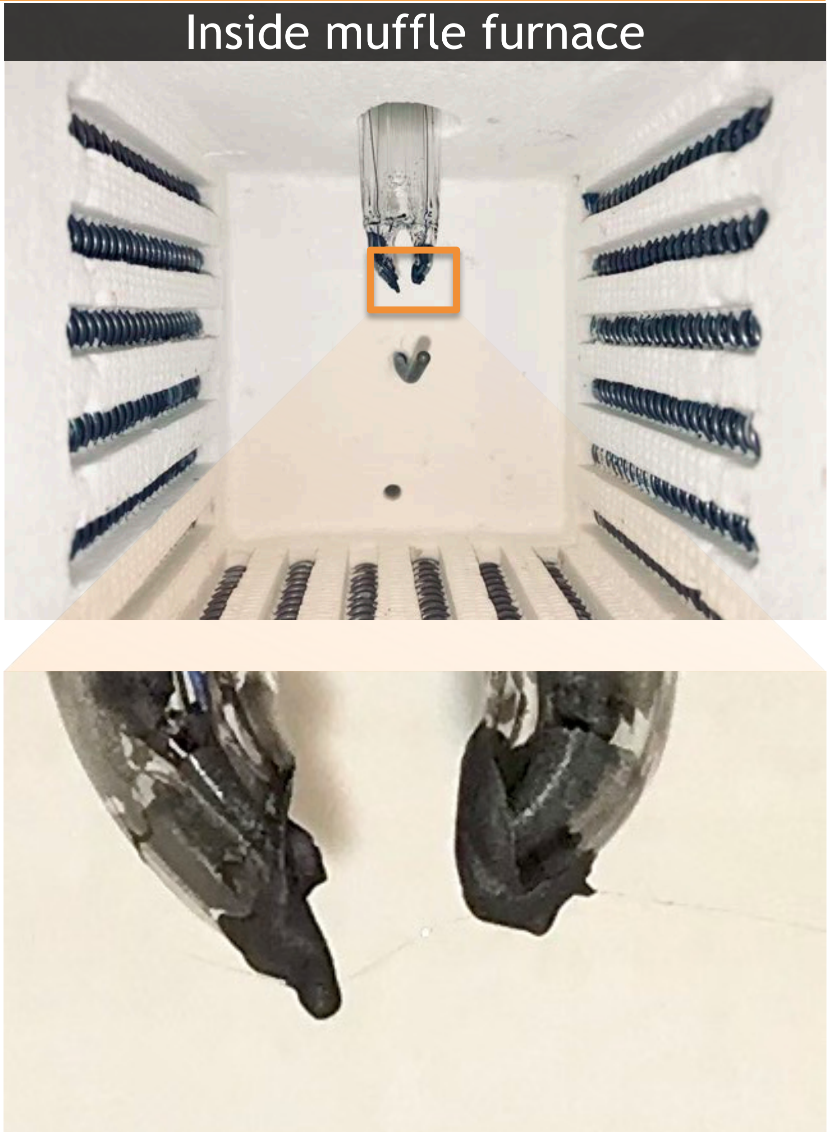


Immersion Probe Modification

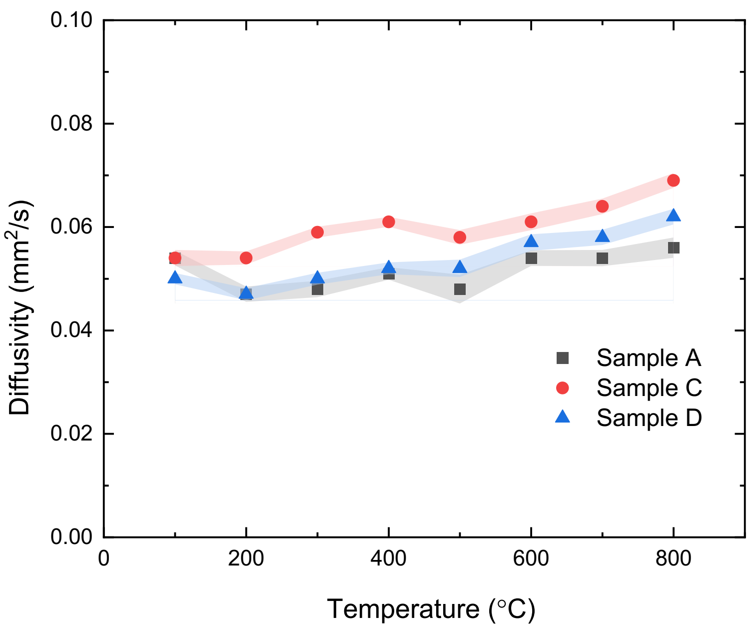


Immersion Probes Setup

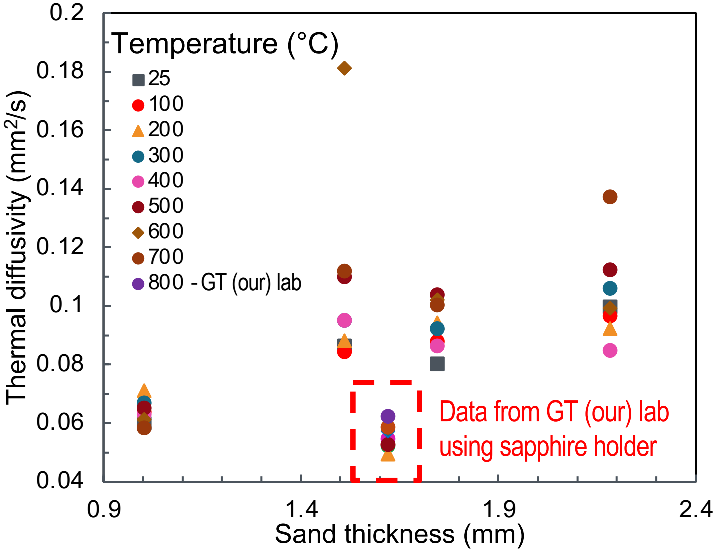


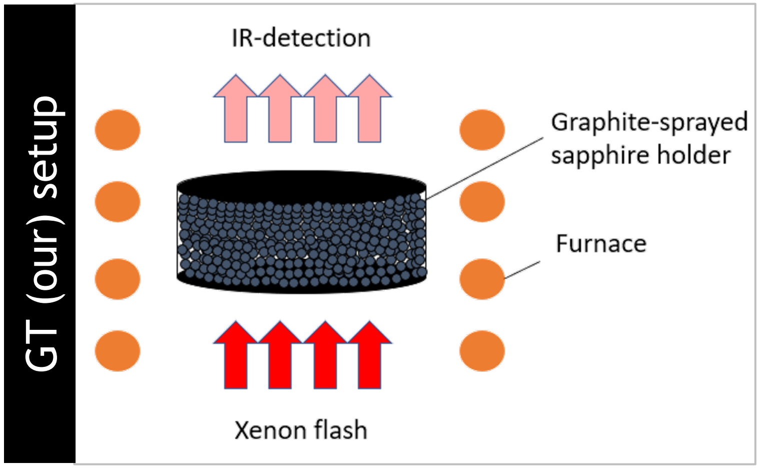


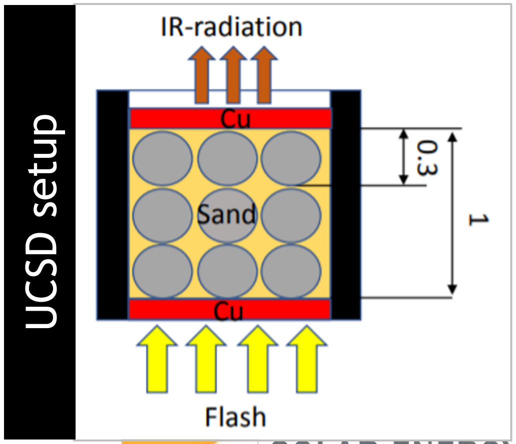
Not all LFA are designed to measure particles' thermal diffusivity accurately



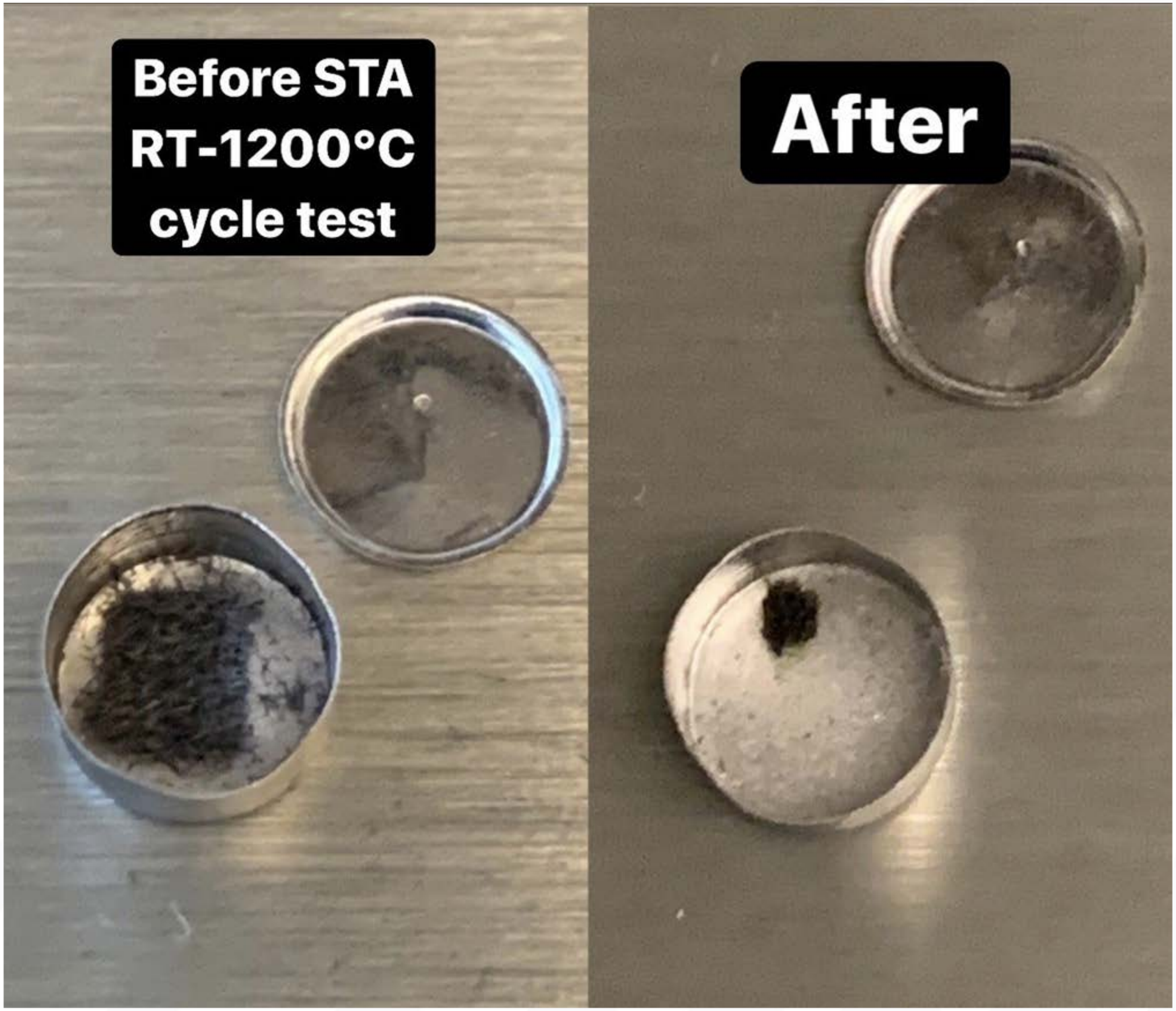
Comparison with UCSD Laser Flash data for HSP 40/70







MIT’s graphite foam samples



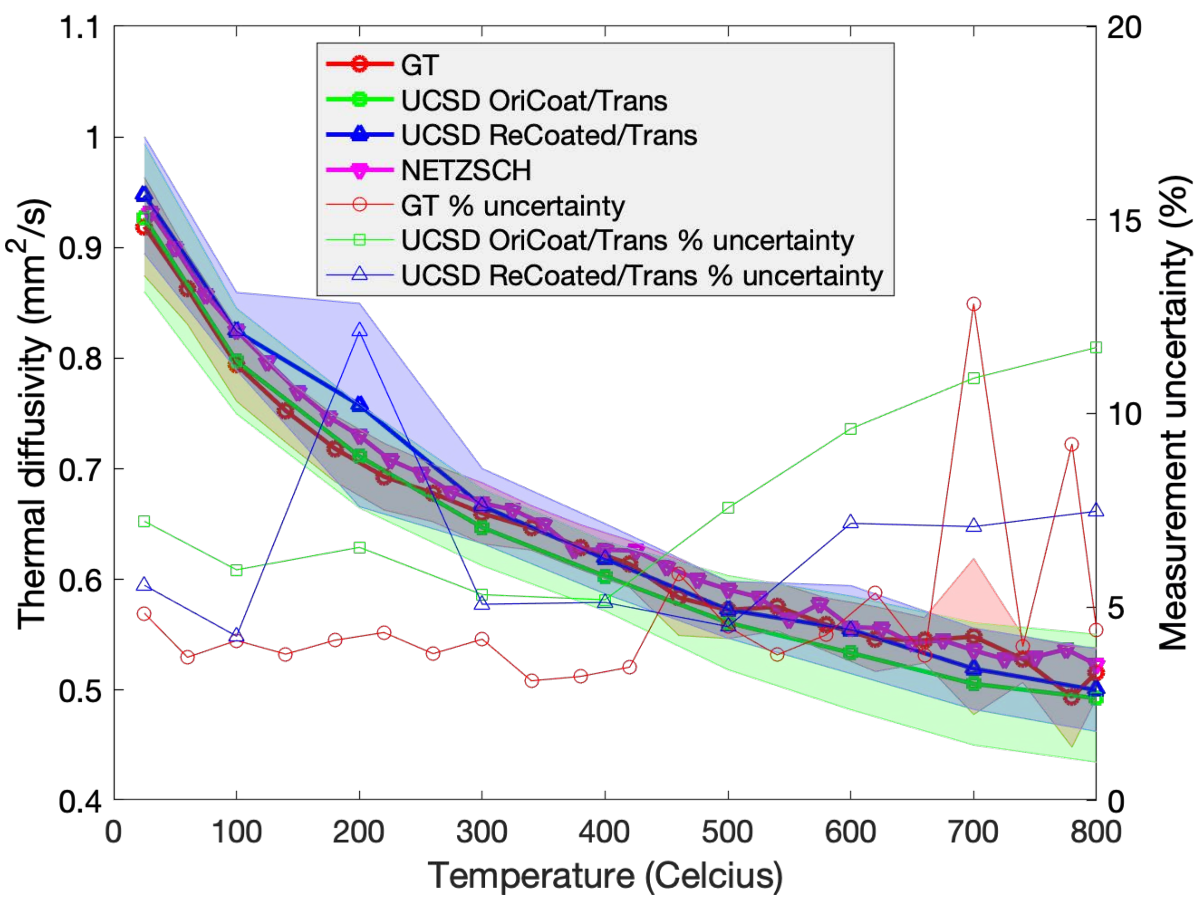
Molten Salt Measurements, December 2020

First test with molten salt

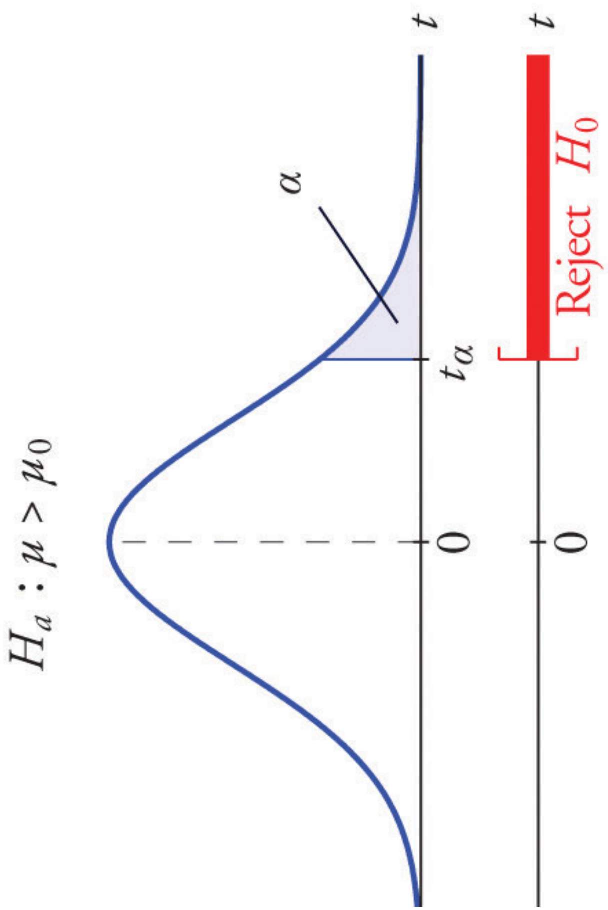
- Cured and annealed probe (at 825°C) with salt in quartz crucible beneath it
- Held for measurement at 700°C
- Saw salt depositions on all parts of probe in oven, all over crucible, on top of oven




CM#4 WAM®-BLG - comparison (transparent model)

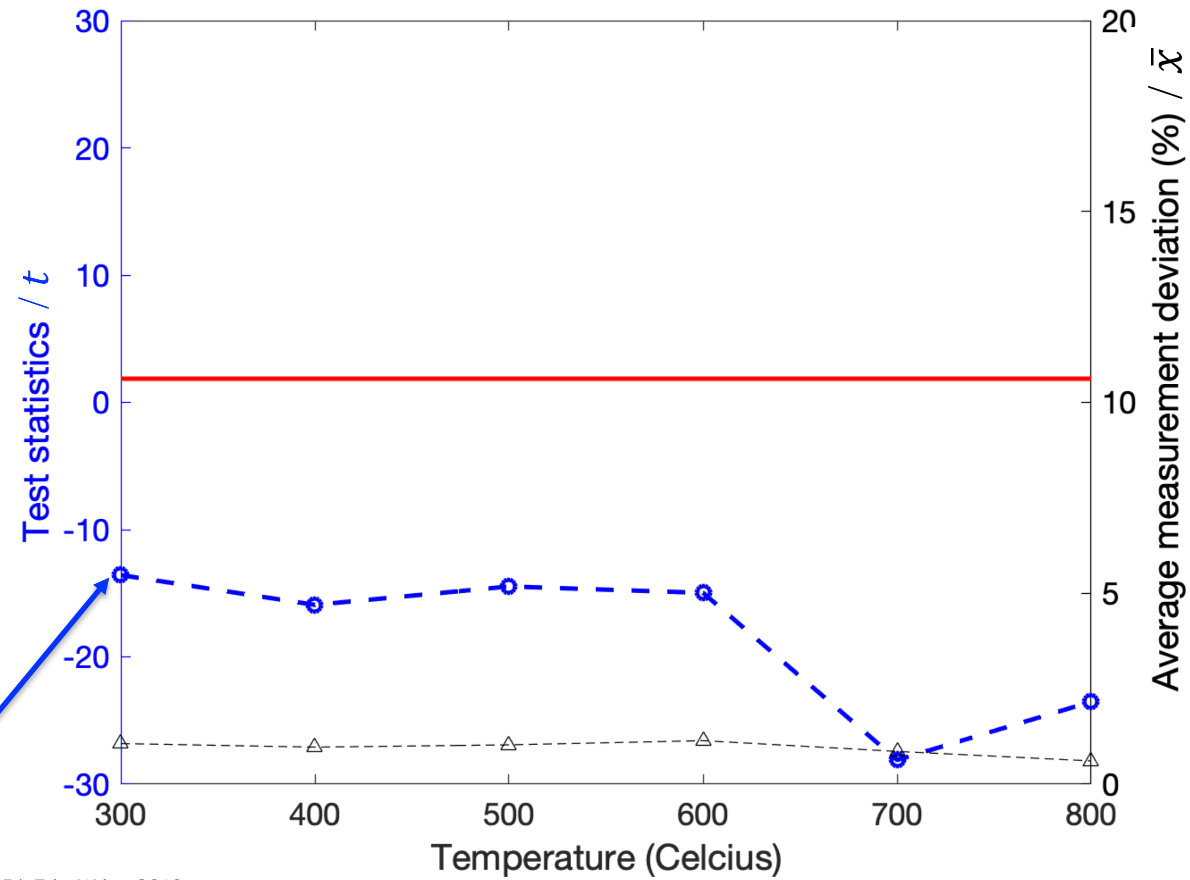


CM#2 Haynes 233 - One-sided student's *t*-test



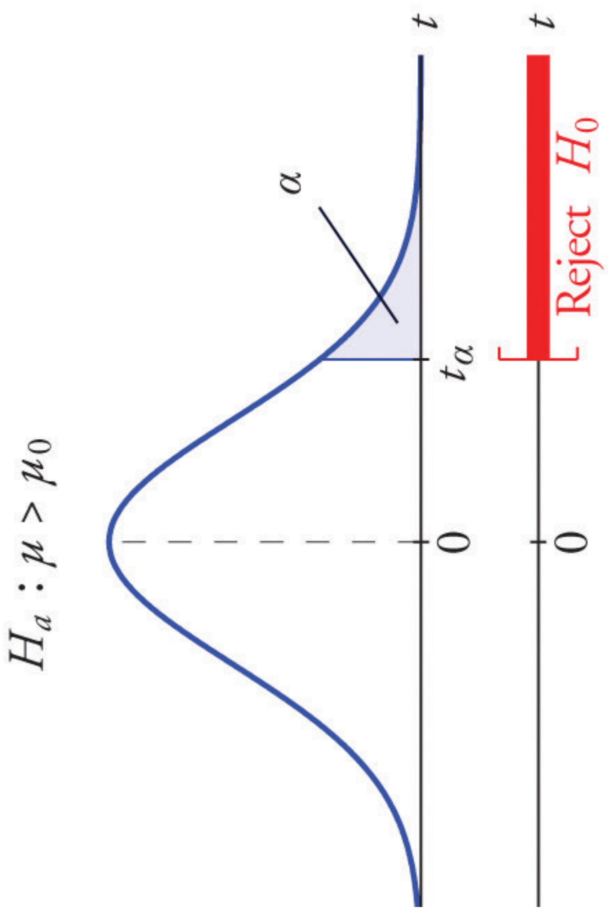
on:

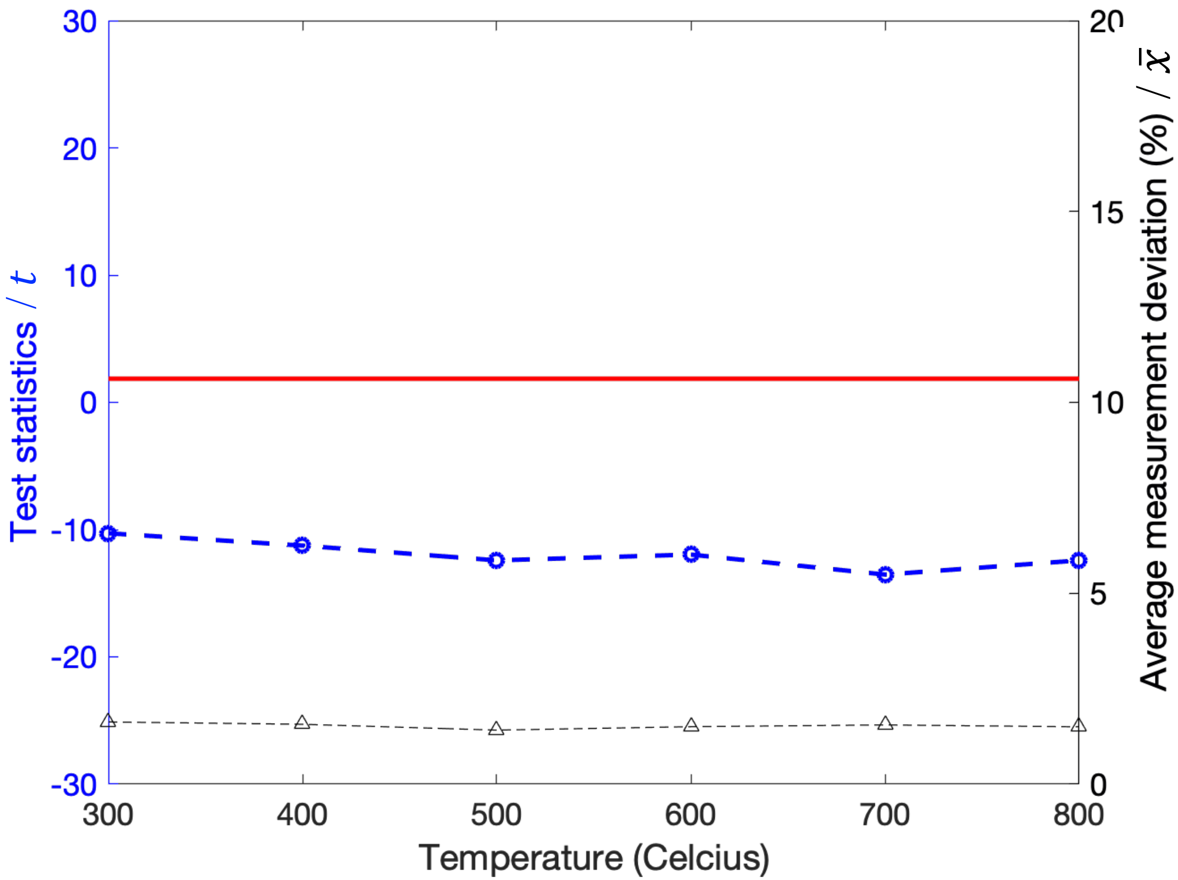
2



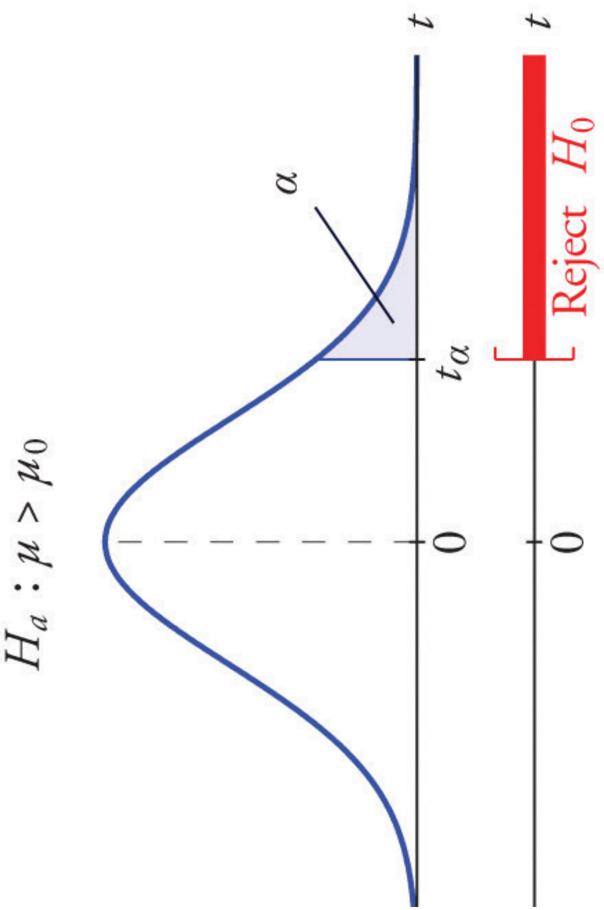
nts", *5th Ed.*, Wiley 2010

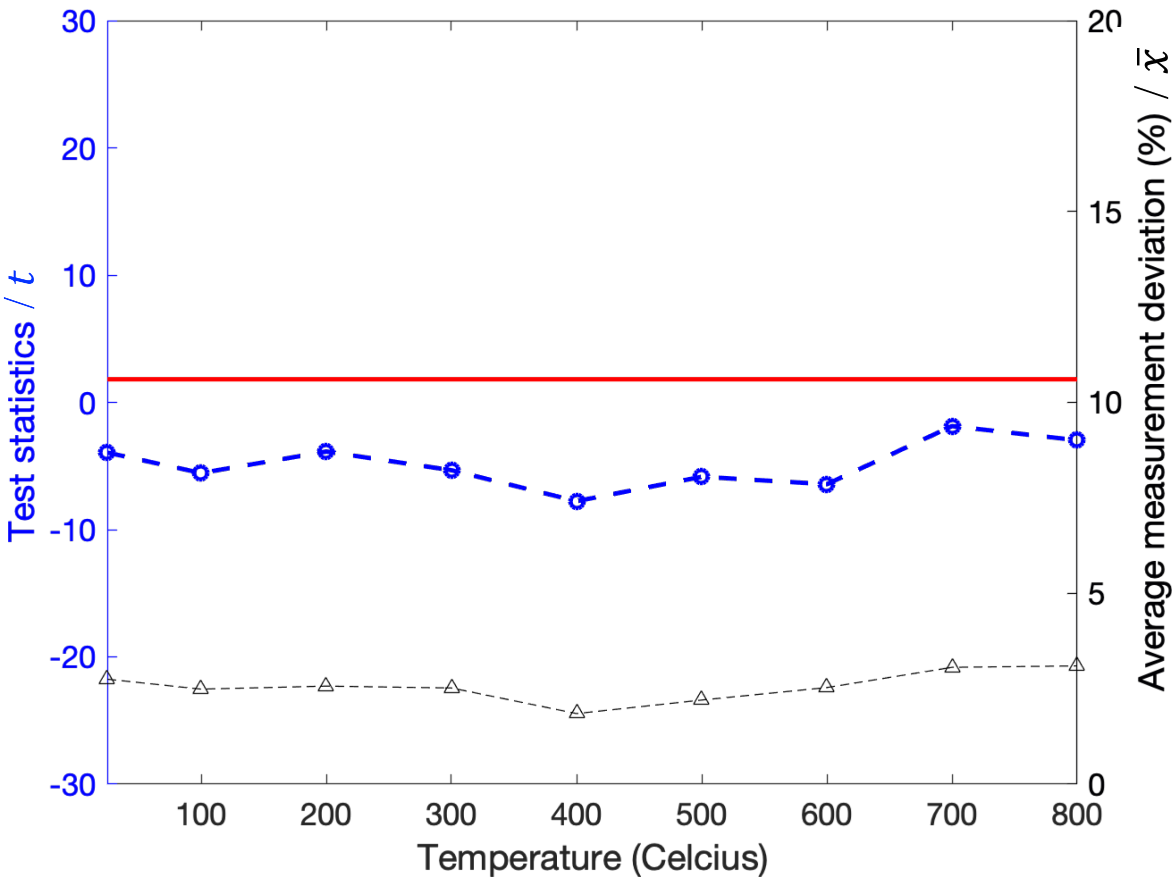
CM#12 Stainless Steel 316 - One-sided student's *t*-test





CM#4 WAM®-BLG - One-sided student's *t*-test





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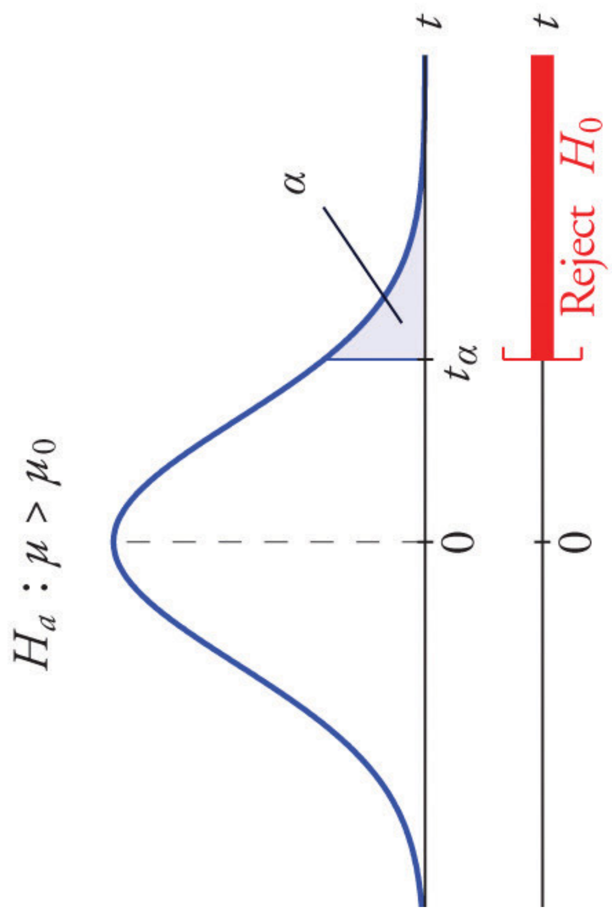
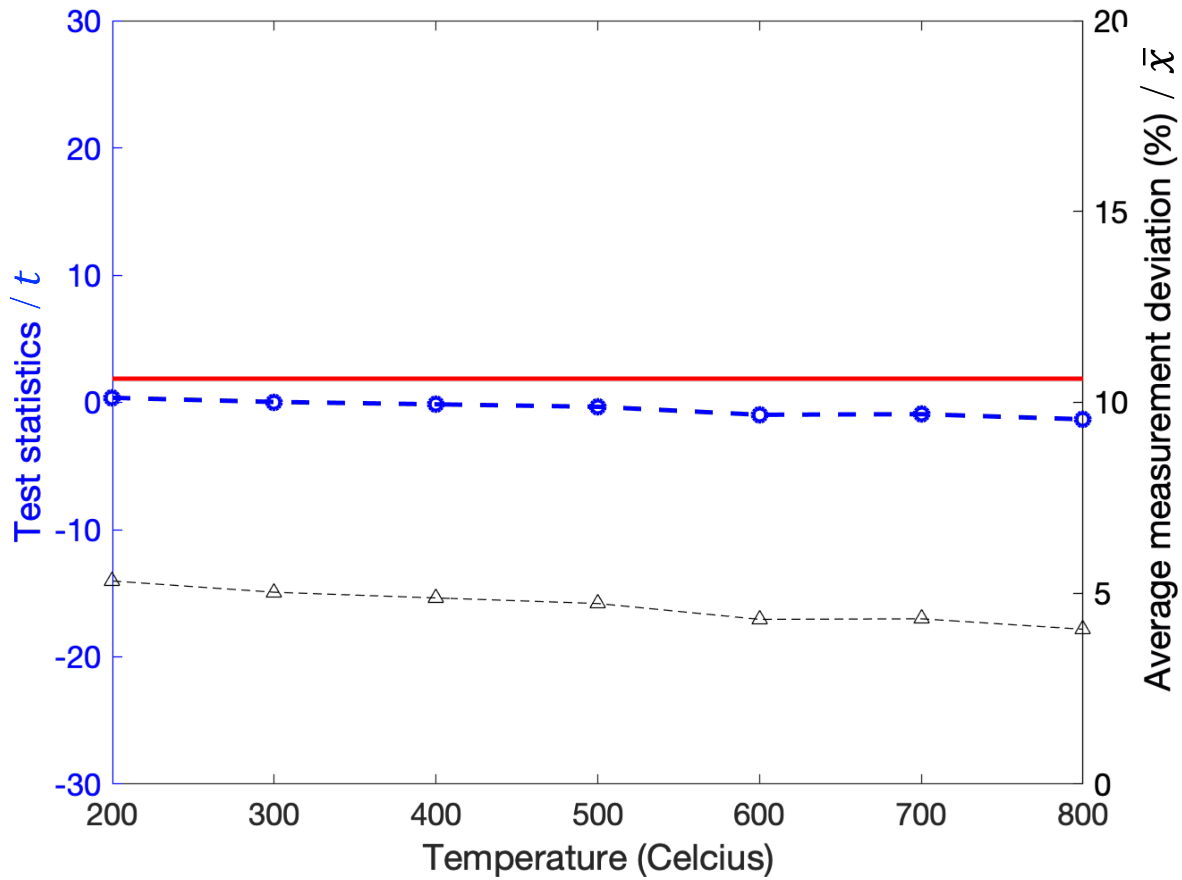
CM#11 SR-99 - One-sided student's *t*-test





CM#*X* ZrC/Mo - One-sided student's *t*-test