Best Practices for Characterizing Engineering Properties of Hydrogen Storage Materials

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contracted with

National Renewable Energy Laboratory

Contract and Technical Manager: Phil Parilla

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ST052

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- Overview -

Timeline

- Start – Feb 2007
- End – Continuing
- 92% complete

Budget

- Total project funding
  - DOE $1,108K
  - Contractor $222K
- Funding FY11 $141K
- Funding FY12 $130K

FY11 budget reduced 20%, FY12 budget scaled back to $130K.

Barriers

- Technical Targets: On-Board Hydrogen Storage Systems
- Barriers addressed
  - A. System Weight and Volume.
  - C. Efficiency.
  - D. Durability/Operability.
  - E. Charging/Discharging Rates.
  - J. Thermal Management.
  - Q. Reproducibility of Performance.

Partners

- NREL: Dr. Parilla, Contract Management
- Authors: University of California Berkeley- California Institute of Technology - Pacific Northwest Laboratories - Texas A&M University - Los Alamos National Laboratory - Université Paris-Sud - VU University Amsterdam and the Delft University of Technology - University of Nevada Reno
- International Energy Agency Hydrogen Implementing Agreement (IEA) Task 22, Dr. Kuriyama AIST, Japan
- Review by experts: IEA Task 22 & others

- Relevance -

- Hydrogen storage materials R&D is a challenging subset of energy storage and environmental materials R&D that also includes analogous activities in areas such as natural gas storage, CO2 separation and sequestration. The creation of Best Practice standards in this field will certainly be of great value to the materials research community at large.
- There are many challenges in the accurate characterization of the hydrogen storage properties of new materials.
- There is a need for consistent measurement practices and improved communication of technical results.
- This project addresses this need through the creation of a reference document detailing best practices and limitations in measuring hydrogen storage properties of materials.
- Accurate measurement methods and metrics are required to determine how new materials compare to all of these targets.
- Progressive sections of this document have been made available for public use by pdf download from the DOE website.

- Objectives -

What ?

- ***To prepare a reference document detailing best practices and limitations in measuring hydrogen storage properties of materials***
- Document reviewed by experts in the field (IEA, IPHE, Industry)
- Document to be made available to researchers at all levels in the DOE hydrogen storage program

Why ?

- To reduce errors in measurements
- Improve reporting and publication of results
- To improve efficiency in measurements
- Reduce the expenditure of efforts based on incorrect results
- Reduce the need for extensive validation
- To increase the number of US experts in this field (students, etc.)

- Benefit to DOE and Researchers -

- Accurate measurement metrics are required to determine how new materials compare to all to DOE targets.
- Accurate measurements practices are also required whenever data is generated that will be used for modeling and engineering of scaled-up systems.
- This project's goal is the establishment of uniform practices in the measurement and presentation of hydrogen storage materials performance.
- The project delivers a public resource as an aid:
  - to the hydrogen storage research community,
  - to students, academic and industry researchers world wide (for hydrogen storage and materials development in general), and
  - to improve international communications on these issues between government, university, and industry.

- Milestones 2011/2012 -

Milestone	Results	%Comp
Finalize Thermodynamic section	Final review and integration of reviewer's edits and comments on Thermodynamics section completed.	100%
Finalize Cycle-Life Section	Final review and integration of reviewer's edits and comments on Cycle-Life section completed.	100%
Complete H2 Storage Properties Sections	Final review and integration of sections 1-5 completed and delivered to DOE for upload to web.	100%
Draft Thermal Properties Section	Draft version in progress.	85%
Mechanical Properties Section	Outline of Draft version in progress.	15%

- **Budget:** Scaled back from prior years, Deliverables unchanged, milestones spread out over longer time period.
- **Go/No-Go FY12:** If the deliverables has not been completed or is determined to provide no value to the program the project will be terminated (9/12).

- Approach: Project Overview -

- **Task 1: General Introduction * (Added at request of DOE)**
  - General introduction to hydrogen storage materials R&D.
  - Overview of measurement techniques and best choice related to purpose of study.
- **Task 2: Kinetics**
  - Emphasis on measurement conditions and material properties that strongly influence the results of kinetic measurements
  - Benefits and limitations of applying mechanistic analysis to kinetics data.
- **Task 3: Capacity**
  - Hydrogen capacity has been the key metric for the success and failure of materials to be considered for practical hydrogen storage.
  - The objective of this task is to clarify issues that can impact these measurements.
- **Task 4: Thermodynamic Stability**
  - Review methods and present new techniques for precisely determining equilibrium thermodynamics.
  - Define protocols to separate true equilibrium conditions from kinetic effects.
- **Task 5: Cycle-life Properties**
  - Cycle-life measurements are critical for evaluating the performance of hydrogen storage materials for applications where hundreds of cycles will be required.
  - Define how such tests should be performed, what parameters may impact the results, and what properties are e.g., capacity fade, or degradation in kinetics, are most critical in performance evaluation.
- **Task 6: Engineering Thermal Properties**
  - Review measurement techniques currently being used for measuring thermal conductivity and heat capacity properties of hydrogen storage materials.
  - This task will include an evaluation of common thermal property measurement methods used in other applied materials fields that may be appropriate for hydrogen storage materials.
- **Task 7: Engineering Mechanical Properties**
  - Examine benefits and limitations of methods for measuring porosity, skeletal, apparent, and packing densities.
  - The validity of translating measurements on small samples to full systems scale performance will be examined.
  - Currently used and alternative methods for measuring material expansion forces will be presented.

Completed / In Progress

- Collaborations -

- **Contributions to this project from world experts including written materials, examples, presentation or editorial review of draft documents from:**
  - Dr. Phil Parilla (contract manager) and Dr. Thomas Gennett, National Renewable Energy Laboratory, Golden CO, USA. (Introduction, Kinetics, Capacity, Spillover)
  - Dr. Gary Sandrock Consultant to U.S. Department of Energy. (Introduction, Kinetics)
  - Dr. George Thomas Consultant to U.S. Department of Energy. (Introduction, Kinetics)
  - Professor Sam Mao University of California Berkeley. (Introduction, Kinetics, Capacity, Thermodynamics sections)
  - Dr. Michael Miller of Southwest Research Institute, San Antonio TX. (Kinetics)
  - Dr. Anne Dailly, and Dr. Frederick Pinkerton of General Motors GM R&D Center. (Capacity)
  - Professor Channing Ahn, California Institute of Technology, USA, IEA Task 22. (Capacity, Spillover)
  - Professor Evan Gray, Griffith University, Brisbane, Australia , IEA Task 22. (Capacity)
  - Dr. Ole Martin Løvvik of the Institute for Energy Technology in Kjeller Norway. (Kinetics)
  - Dr. Nobuhiro Kuriyama and Dr. Tetsu Kiyobayashi , AIST, Japan, IEA Task 22. (Introduction, Kinetics sections)
  - Dr. Eric Poirier of NRC Canadian Neutron Beam Centre Chalk River Laboratories, Canada (Capacity section)
  - Dr. Kevin Ott, Dr. Anthony Burrell, and Dr. Troy Semelsberger of Los Alamos National Laboratory (Capacity, Thermodynamics sections)
  - Professor Klaus Yvon, University of Geneva, Switzerland IEA Task 22. (Capacity section)
  - Professor Gavin Walker, University of Nottingham, United Kingdom IEA Task 22. (Thermodynamics Section)
  - Professor Richard Chahine, Université du Québec à Trois-Rivières, Canada, IEA Task 22. (Kinetics, Capacity and Engineering Thermal Properties section)
  - Dr. Martin Dornheim, Helmholtz-Zentrum Geesthacht, Germany, IEA Task 22. (Engineering Thermal Properties sections)
  - Dr. Renju Zacharia, Université du Québec à Trois-Rivières, Canada, IEA Task 22. (Thermodynamics section)
  - Dr. Tom Autrey and Dr. Scot Rassat, Pacific Northwest National Laboratory, Richland WA, USA. (Thermodynamics section)
  - Dr. Maximilian Fichtner, Karlsruher Institute for Technology, Germany, IEA Task 22. (Engineering Thermal and Mechanical Properties sections)
  - Dr. Robert Bowman Jr., Consultant to U.S. Department of Energy. (Cycle Life)
  - Dr. Ewa Rönnebro, Pacific Northwest National Laboratories, USA. (Engineering Thermal Properties sections)
  - Dr. Bart van Hassel, United Technologies Research Center, USA. (Engineering Thermal Properties sections)
  - Dr. Lars Röntzsch, Fraunhofer Institute for Manufacturing Technology and Advanced Materials, Dresden, Germany. (Engineering Thermal Properties sections)
  - Dr. Michel Latroche, Institut de Chimie et des Matériaux de Paris Est CNRS, France, IEA Task 22. (Engineering Thermal Properties sections)
  - Dr. Patricia De Rango, Institut Néel CNRS, Grenoble, France, IEA Task 22. (Engineering Thermal Properties sections)
  - Dr. Mike Veenstra, Ford Motor Co., USA. . (Engineering Thermal Properties sections)

- Key Accomplishments -

2007 – 2011:

- ***Task 1: Final Introduction section 100% complete.***
- ***Task 2: Final Kinetics section 100% complete***
- ***Task 3: Updated Capacity section, with review of Spillover by Tom Gennett and Phil Parilla (NREL) and Channing Ahn (CalTech) 100% complete.***
- ***Task 4: Final Thermodynamic section 100% complete.***
- ***Task 5: Final version Cycle Life 100% complete.***

2011 – 2012:

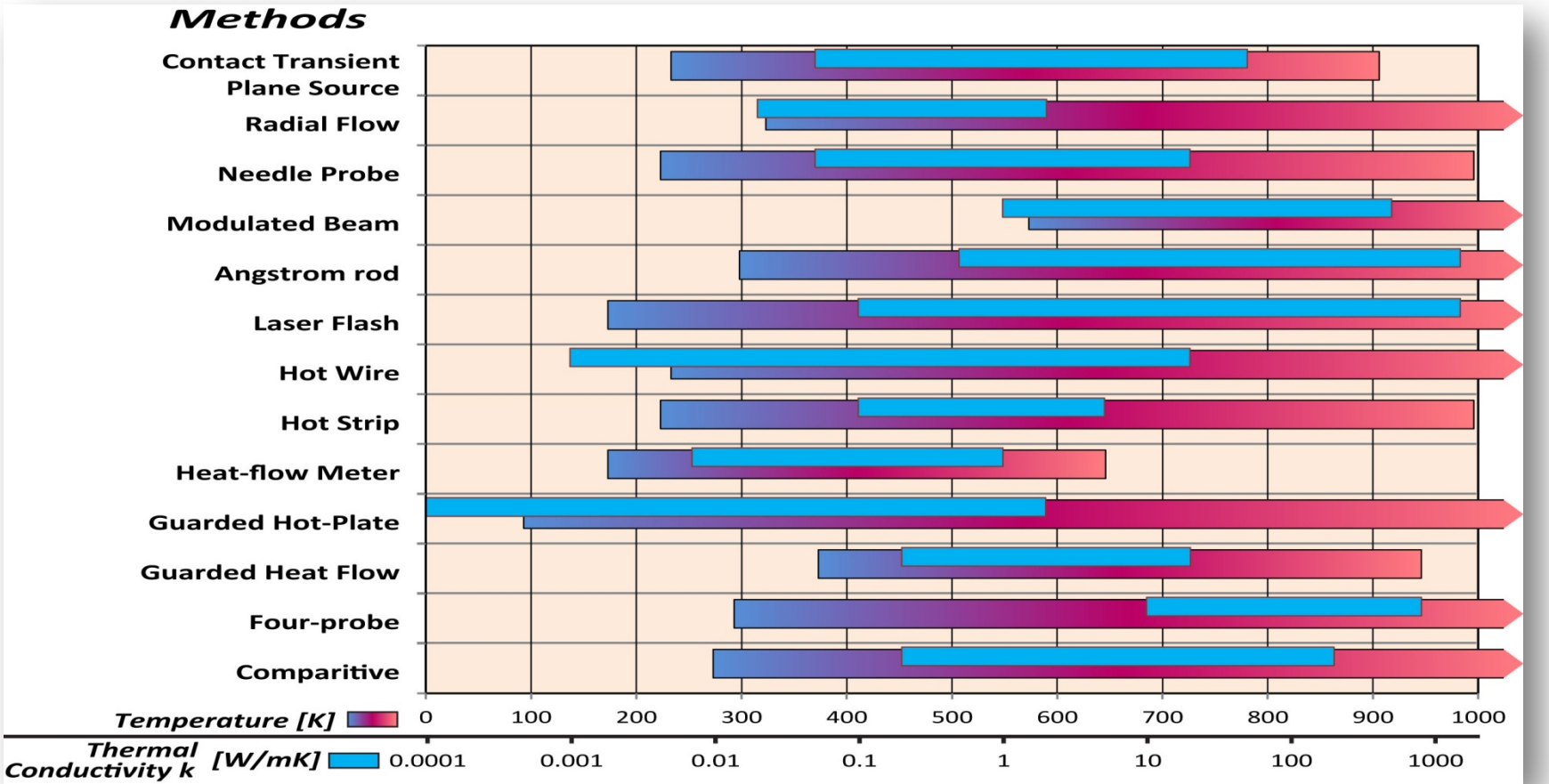
- ***Task 6: Engineering Thermal Properties section work in full progress.***
- ***Task 7: Engineering Mechanical Properties section started.***
- ***Reviewed Tasks 1-5 Document Delivered to DOE March 2012.***
- ***Final integrated Tasks 1-5 Document currently being posted to DOE website for world-wide access.***
- ***Project reviewed by Hydrogen Storage Tech Team, August 18, 2011***
- ***Additional US and International Contributors: Maximilian Fichtner, Ewa Rönnebro, Daniel Dedrick, Bart van Hassel, Lars Röntzsch, and Michel Latroche, contributions developed through collaborations within IEA Task 22.***

Please download the current Best Practices document from:

http://www1.eere.energy.gov/hydrogenandfuelcells/pdfs/best_practices_hydrogen_storage.pdf

- Technical Accomplishments -

Section 6: Measurements of Engineering Thermal Properties of Hydrogen Storage Materials

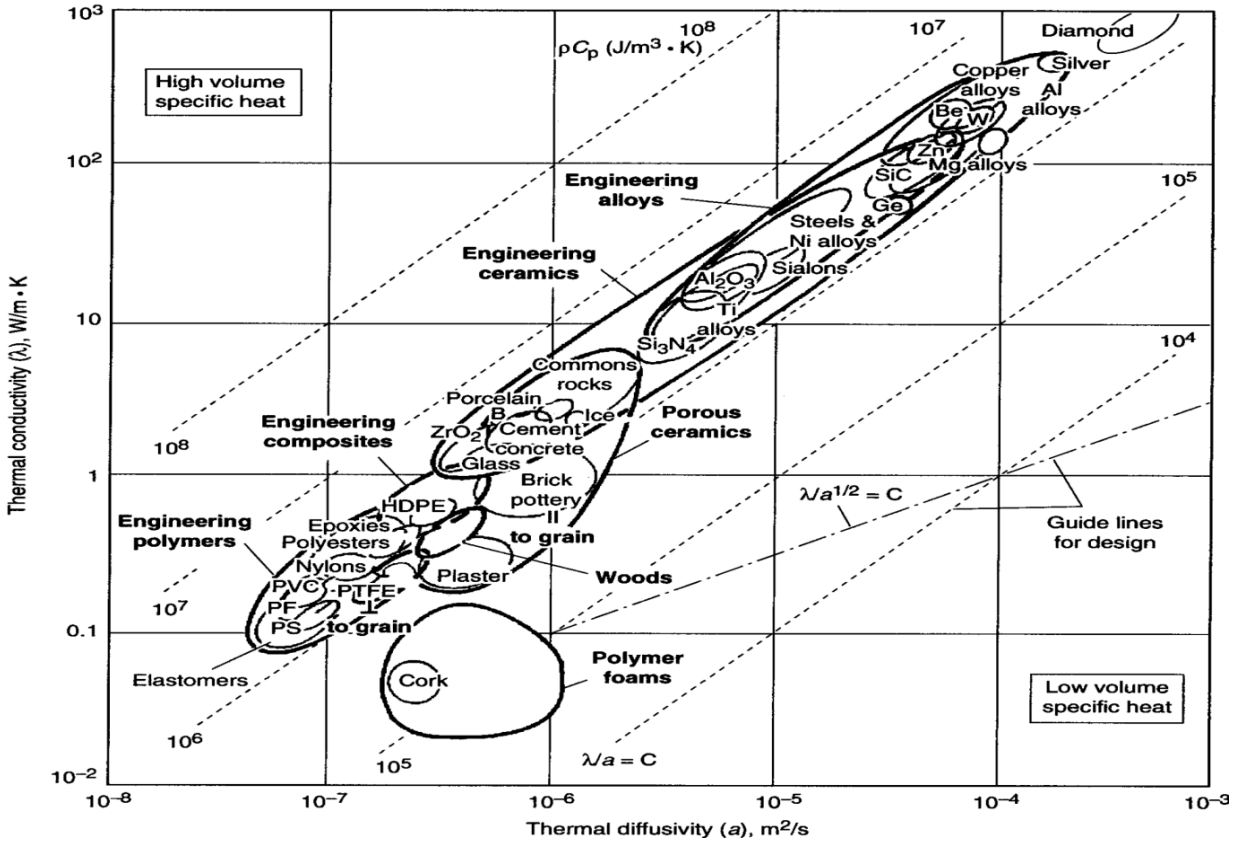


Typical measurement methods for differing ranges of Thermal Conductivity and measurement temperatures.

(ranges may vary for specifically designed measurement systems)

Adapted from: Evitherm.org article, "Measurement Methods", http://www.evitherm.org/
Wikipedia article "Thermal Conductivity", http://en.wikipedia.org/wiki/Thermal_conductivity

Thermal Conductivity & Diffusivity for Common Materials



The material property governing the flow of heat through a material at steady state is the thermal conductivity *k*.

The property governing transient heat flow is the thermal diffusivity, *a*.

They are related by: $a = k/\rho C_p$

where ρ is the density and C_p the specific heat.

The quantity ρC_p is the volumetric specific heat.

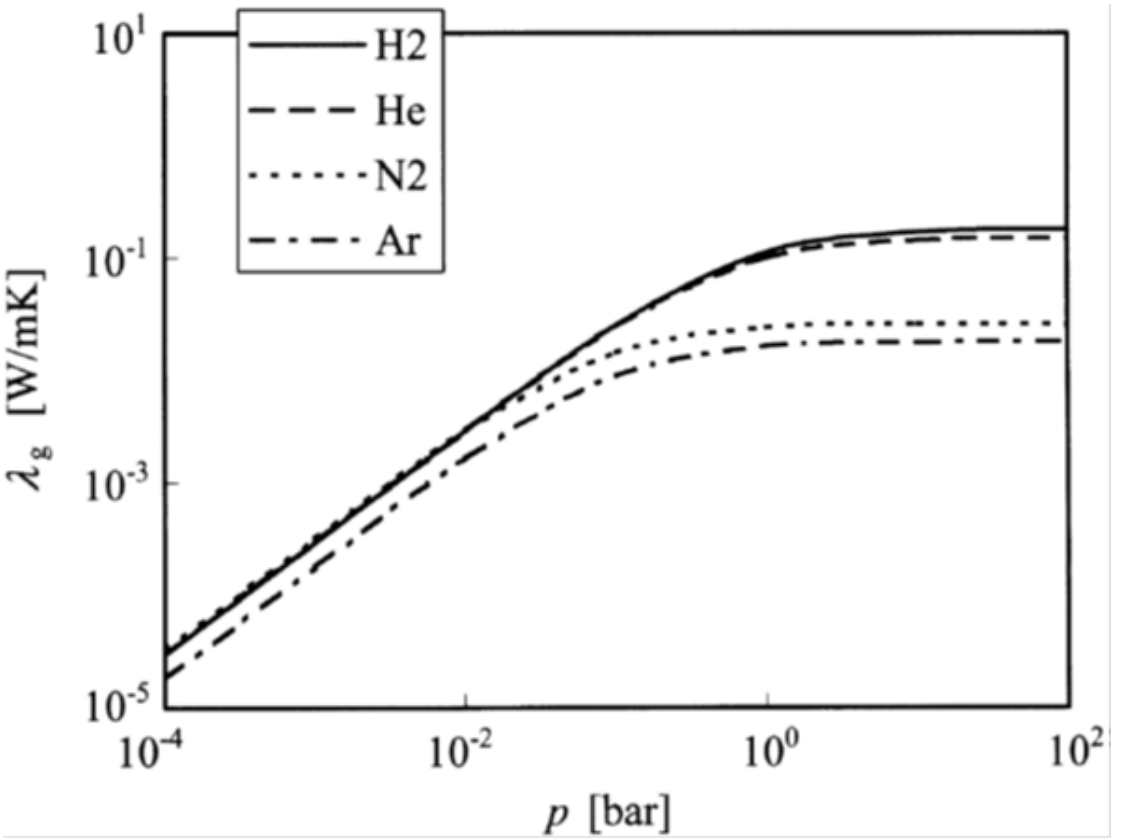
All three properties vary with temperature.

Room temperature thermal conductivity *k* vs. thermal diffusivity a. Contours are volume specific heat, ρC_p .

- **Both Thermal Conductivity and Diffusivity vary by 5 orders of magnitude depending on the materials.**
- **Hydrogen storage materials also cover a wide range of intrinsic Thermal Conductivity values (metal alloys to polymers and oxides)**

"Thermal Properties of Metals", Introduction to Thermal Properties ASM Ready Reference: (#06702G), (2002), Fran Cverna, Technical Editor.

Considerations for Real Hydrogen Storage Systems



Effect of the pressure on the thermal conductivity of different gases.

- **Heat transfer in an operating hydrogen storage system must take the conductive and convective heat transfer of the gas into account.**

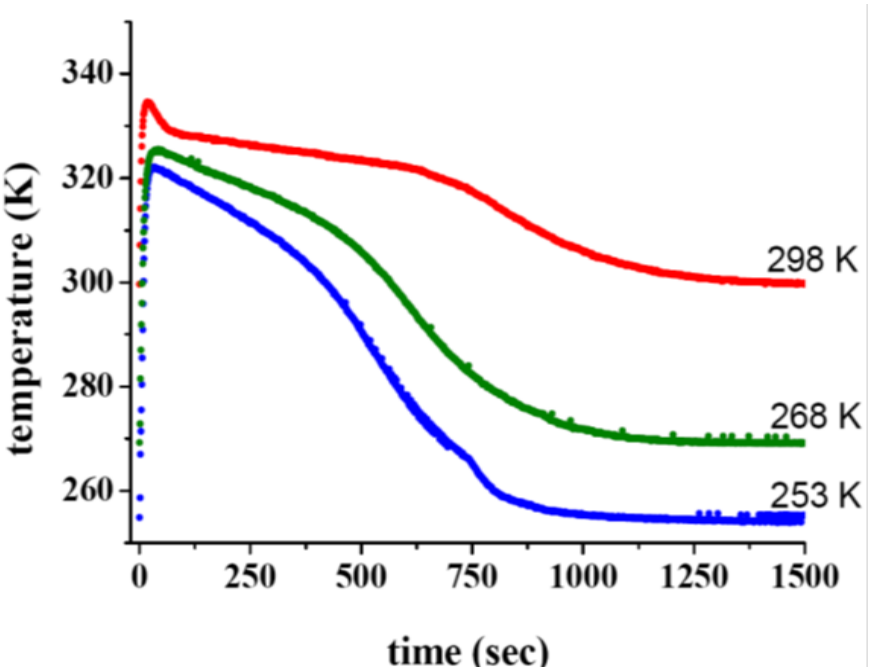
Asakuma, Y., Miyauchi, S., Yamamoto, T., Aoki, H., Miura, T., "Homogenization method for effective thermal conductivity of metal hydride bed", Int. J. of Hydrogen Energy, 29 (2004) p.209 – 216.

Hydrides: Enthalpy of Reaction & Heat Transfer

Small hydride test bed (150 g AB_5). Designed as a buffer tank for Room Temperature start up of PEM fuel cell.

Temperature (K)	253	268	298
t_{90} (sec) small sample (0,5g)	234	76	149
t_{90} (sec) buffer tank (150g)	427	442	750

Rates of hydrogen absorption of small sample 0.5 g vs. 150 g bed.



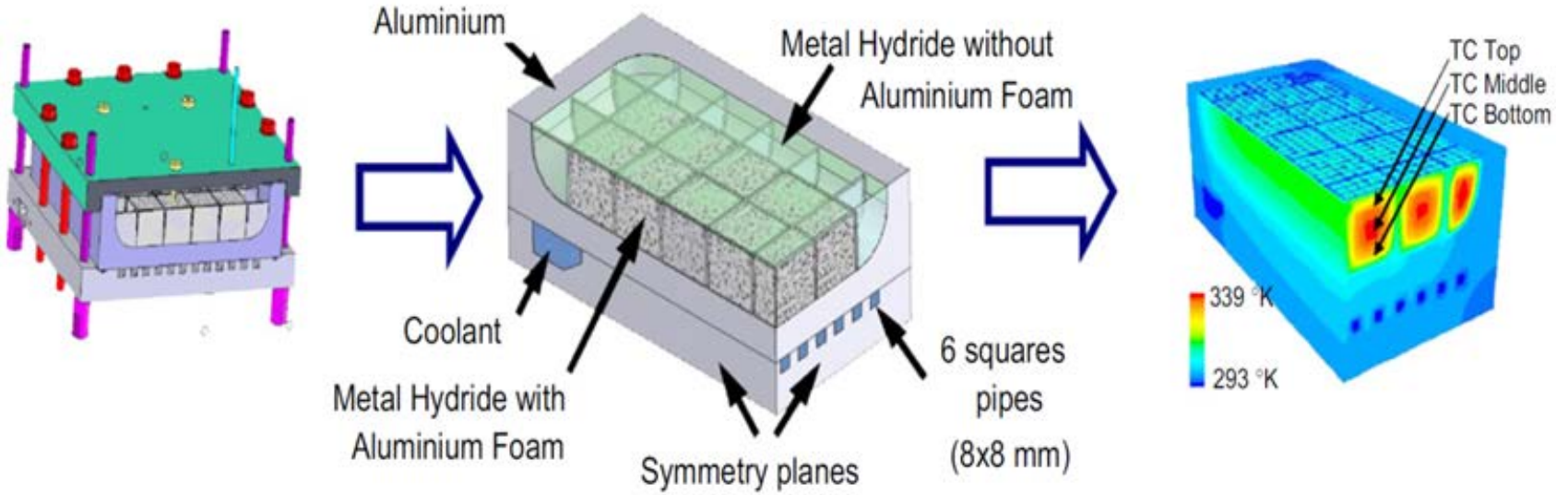
Internal temperature as a function of time for hydride buffer tank upon filling at 50 bar H_2. Temperature measured from thermocouple in center of hydride material, tank in temperature controlled water bath at temperatures indicated on each curve.

- **Without sophisticated means of heat transfer, the heat of hydride formation will cause a significant rise in temperature, greatly reducing absorption rates.**

Challet, S., Latroche, M., Heurtaux, F., "Development of a La0.55Y0.45Ni5 metal hydride pilot buffer tank for fuel cell car", WHEC 16 / 13-16 June (2006) – Lyon France.

Sophisticated Methods and Models of Heat Transfer for Hydrogen Storage are Being Developed World-Wide

Example: System Design

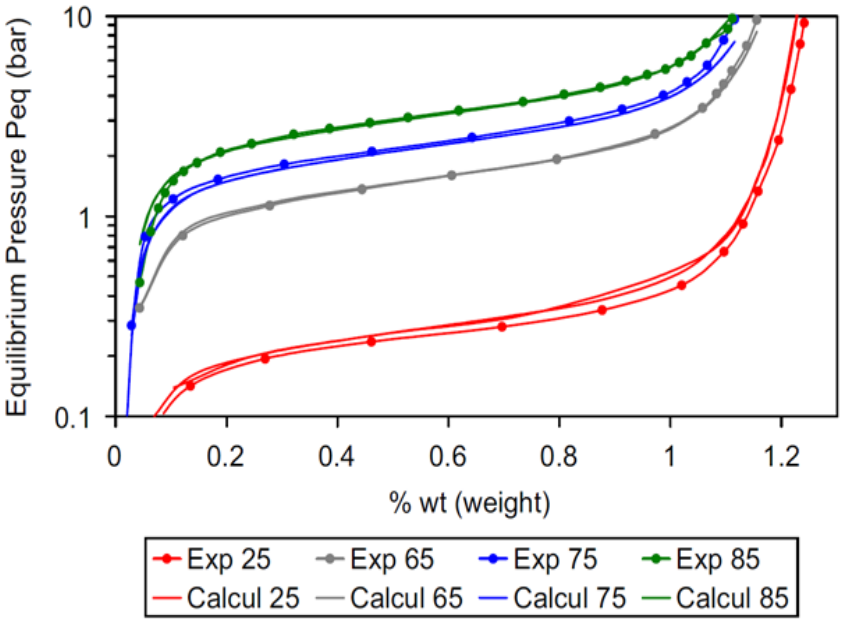


A metal-hydride storage bed (left), its modeled geometry (center), and calculated temperature contours of temperatures (right) during absorption.

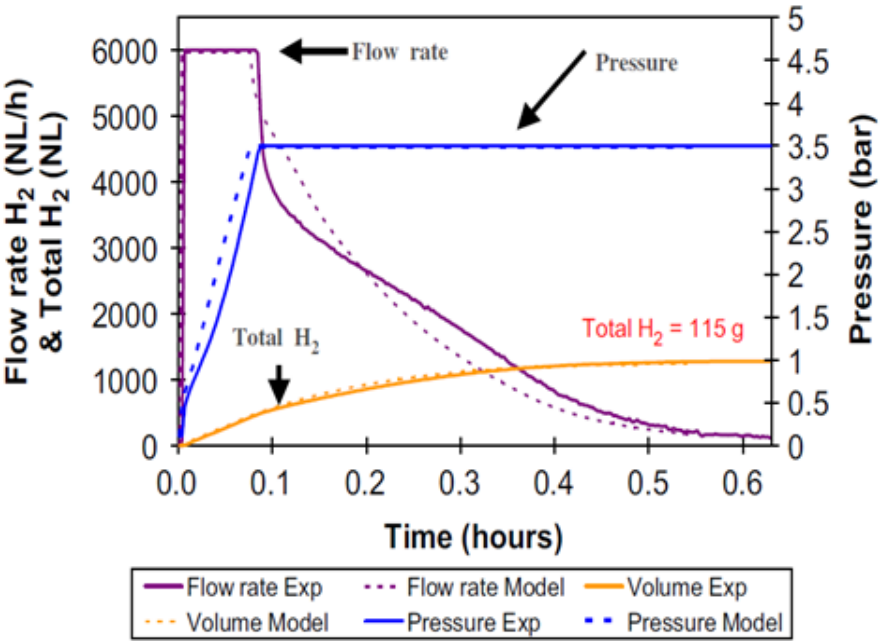
- **Advanced hydrogen storage systems will likely involve several different heat transfer solutions.**
- **These may entail complex combinations of materials, components, geometries, and gas flows that need to be modeled and tested.**

Botzung, M., Chaudourne, S., Gilliaa, O., Perret, C., Latroche, M., Percheron-Guegan, A., and Marty, P., "Simulation and experimental validation of a hydrogen storage tank with metal hydrides", Int. J. of Hydrogen Energy, 33 (2008) p. 98 – 104.

Example: Materials & System Modeling



Experimental and Modeled PCI curves of $MmNi_{5-x}Sn_x$ at 25, 65, 75 and 85°C.



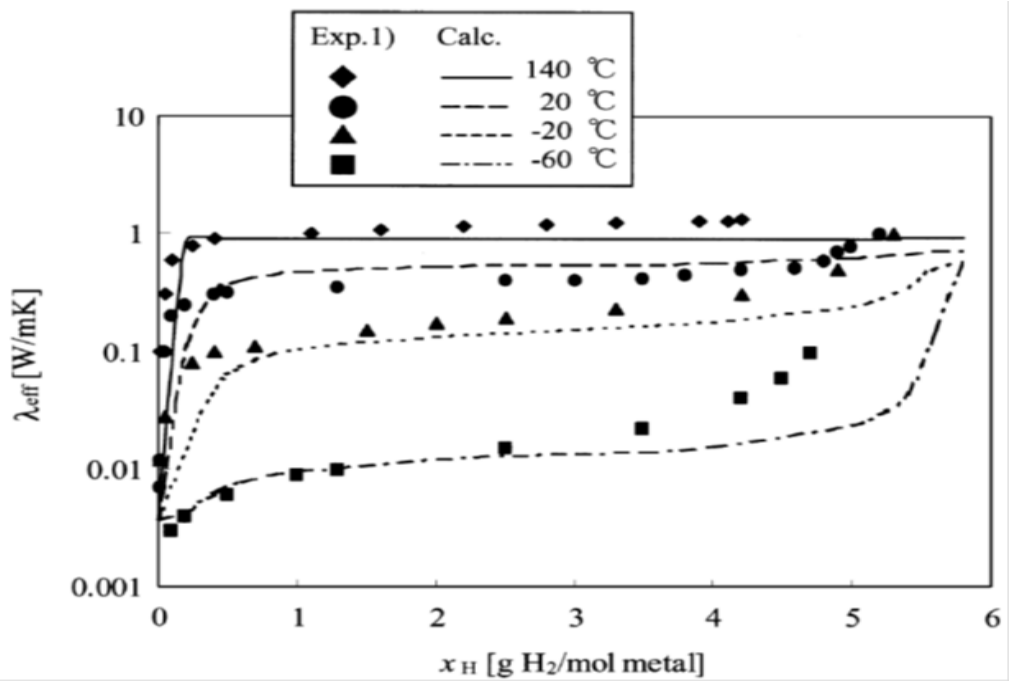
Experimental results (solid) vs. model (dashed) results for system (previous slide).

- **The accurate determination of the effective thermal conductivity of storage materials, additives, and system components is critical for modeling advanced systems.**
- **This data is aiding both materials development and systems design.**

Botzung, M., Chaudourne, S., Gilliaa, O., Perret, C., Latroche, M., Percheron-Guegan, A., and Marty, P., "Simulation and experimental validation of a hydrogen storage tank with metal hydrides", Int. J. of Hydrogen Energy, 33 (2008) p. 98 – 104.

Measuring and Modeling Thermal Conductivity

Example: Effective Thermal Conductivity

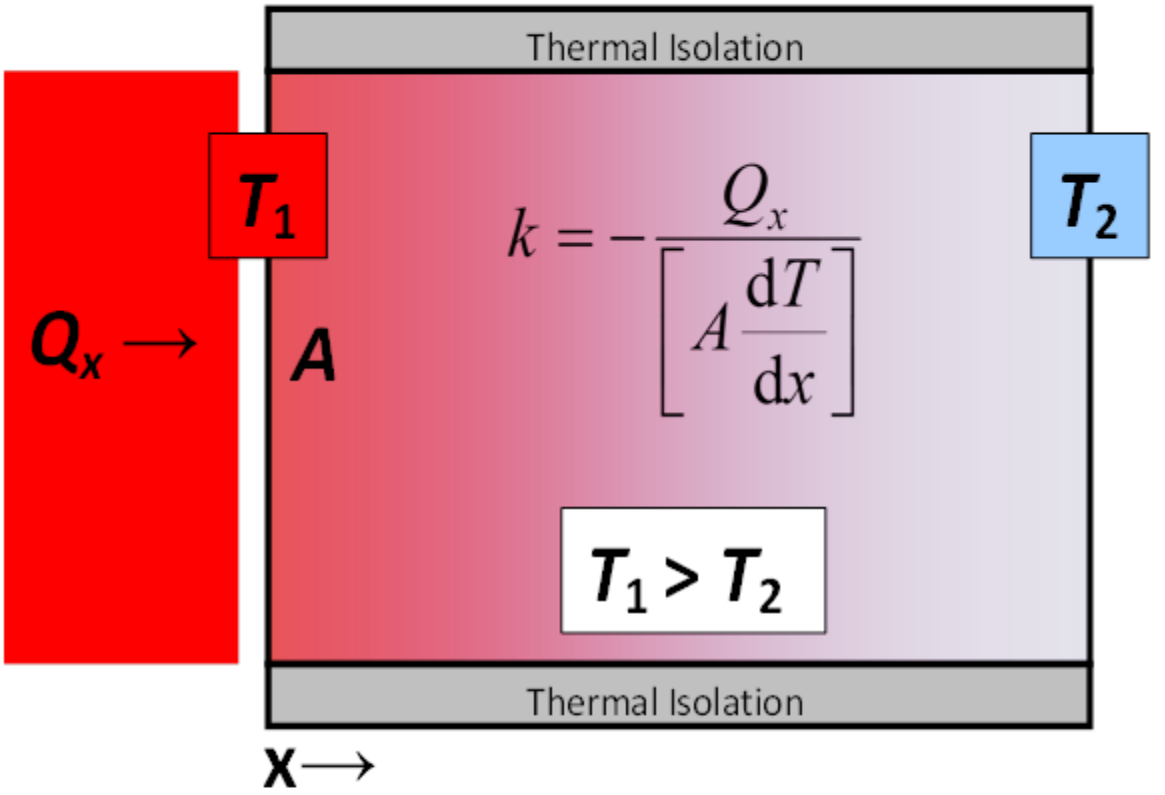


Relation between the effective thermal conductivity and hydrogen concentration for $LaNi_{4.7}Al_{0.3}$: (symbols) experimental data, (lines) homogenization method.

- **A homogenization method has been used to develop a numerical model describing thermal conductivity as a function of gas type, gas pressure and bed temperature.**
- **The method represents the microstructure within the a hydrogen storage bed.**
- **This may be a powerful tool for estimating effective thermal conductivity within hydride beds by considering microscopic behaviors such as the pulverization and change of the contact area between particles at different hydrogen concentrations.**

Asakuma, Y., Miyauchi, S., Yamamoto, T., Aoki, H., Miura, T., "Homogenization method for effective thermal conductivity of metal hydride bed", Int. J. of Hydrogen Energy, 29 (2004) p.209 – 216. Hahne, D, Kallweit, J., "Thermal conductivity of metal hydride materials for storage of hydrogen: experimental investigation", Int J Hydrogen Energy (1998) 23(2) p.107–14.

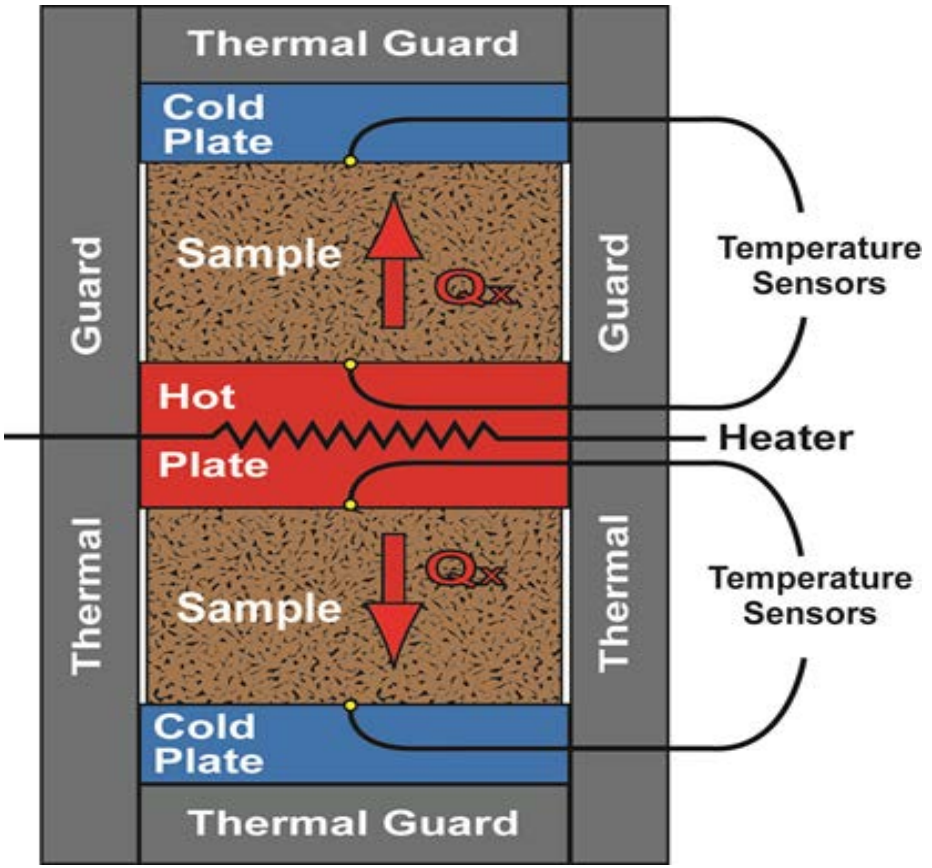
One Dimensional Heat Transfer & Thermal Conductivity



Schematic diagram a simple thermal conductivity measurement of a solid.

- **In Theory: The measurement of Thermal Conductivity is greatly simplified by one-dimensional (or uni-axial) heat flow.**
- **In Practice: This is difficult to achieve completely.**

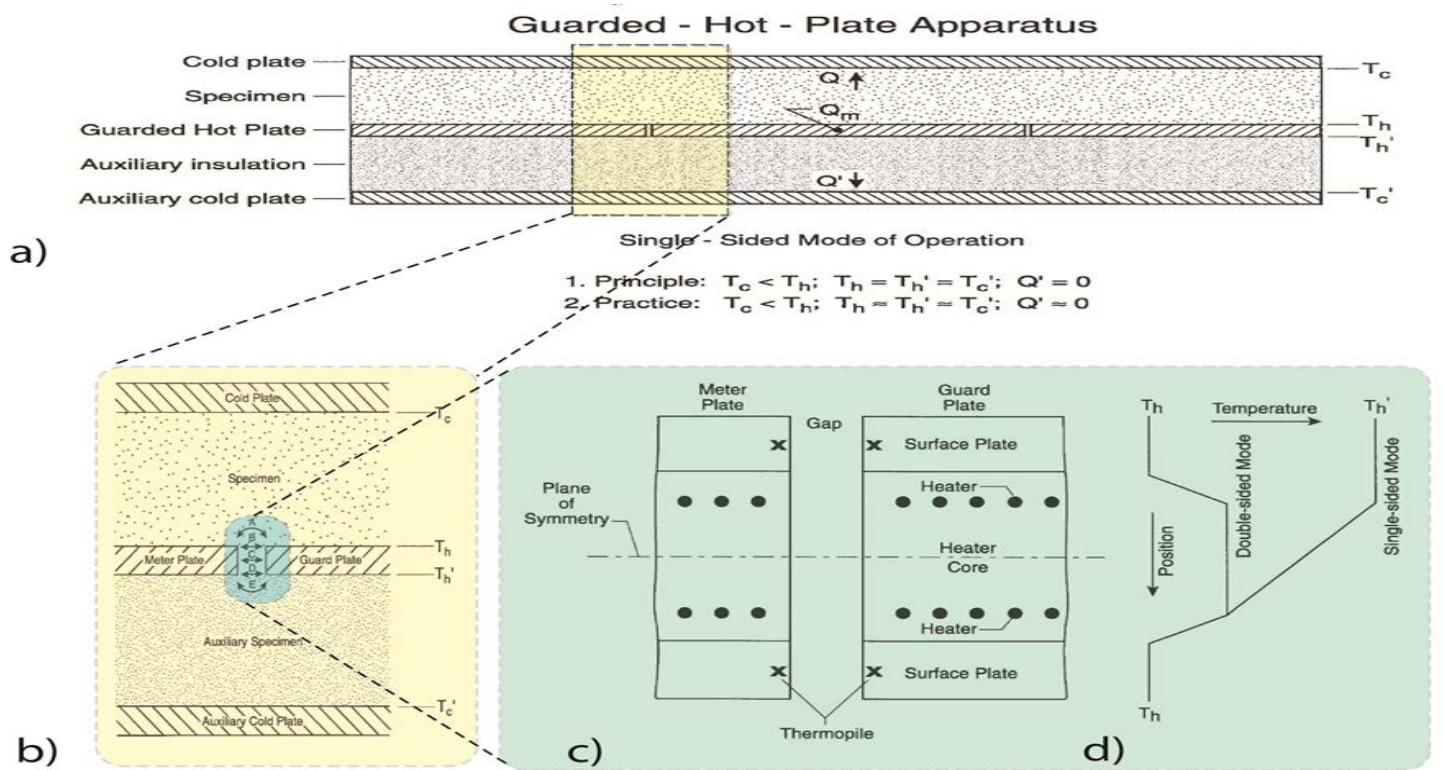
Example Measurement Method: Guarded Hot Plate Method (ASTM C 177)



Schematic of basic guarded hot plate design.

- **The Guarded Hot Plate is a well established method for measuring the thermal conductivity of low conductivity powder materials.**

Details of Error Reduction in Design of Advanced Guarded-Hot Plate Instruments



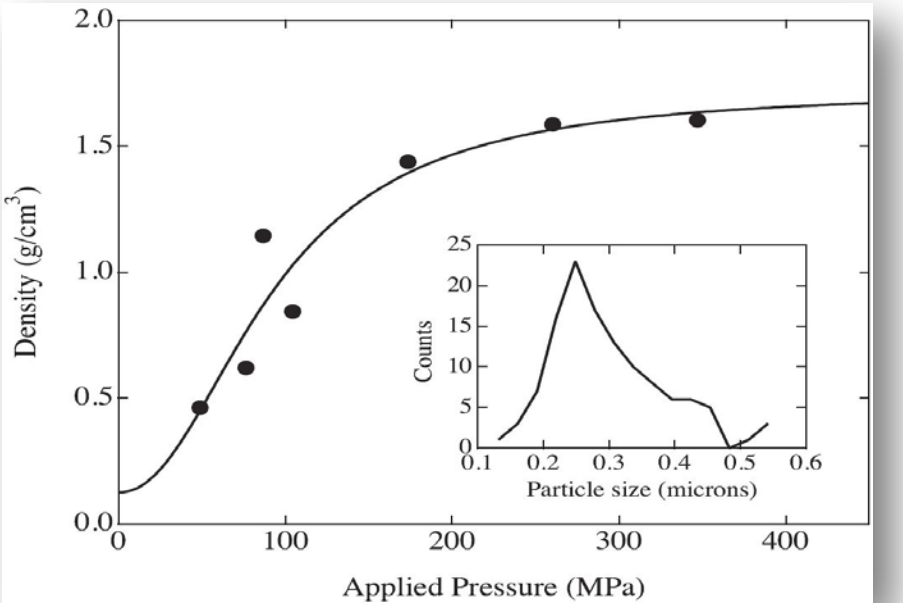
a) Schematic diagram of an advanced single-sided mode of operation Guarded-Hot-Plate apparatus, b) heat flows at gap region, c) hypothetical Guarded-Hot-Plate with distributed heat source, and d) Corresponding temperature profile for double- and single-sided mode of operation.

- **Heat leaks lead to error in the determination of Thermal Conductivity.**
- **Today's equipment is expected to employ advanced methods for reducing such sources of error.**

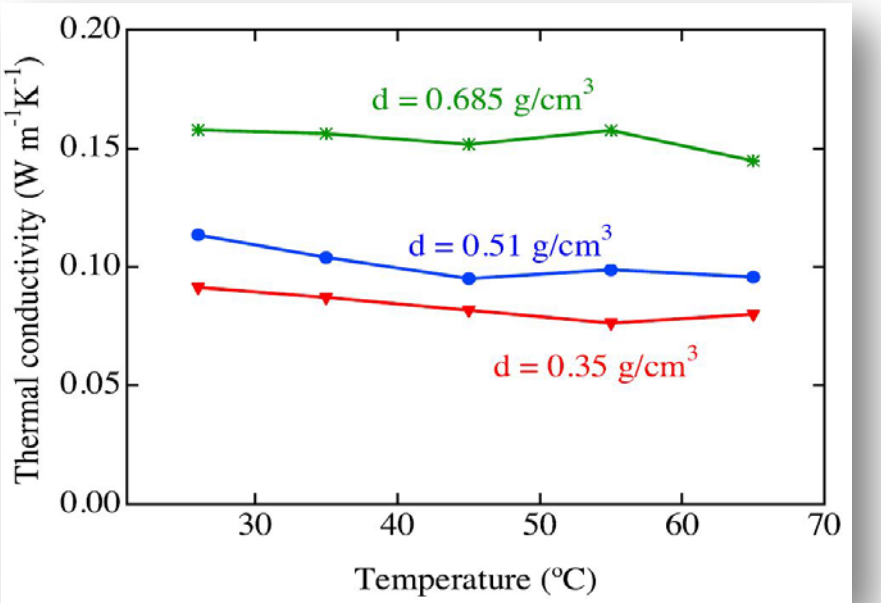
Adapted from"Standard Practice for Using a Guarded-Hot-Plate Apparatus or Thin-Heater Apparatus in the Single-Sided Mode", ASTM International, Designation: C 1044 – 98, Reapproved (2003), standard downloaded from www.bzfxw.com

Thermal Conductivity of Physisorption Materials

Example Study MOF-5:



Density of pellets versus forming pressure. Inset: Particle size distribution of powder.



Thermal conductivity of MOF-5 pellets vs. of increasing density vs. temperature.

Thermal Conductivity: $k = \alpha \rho C_p$

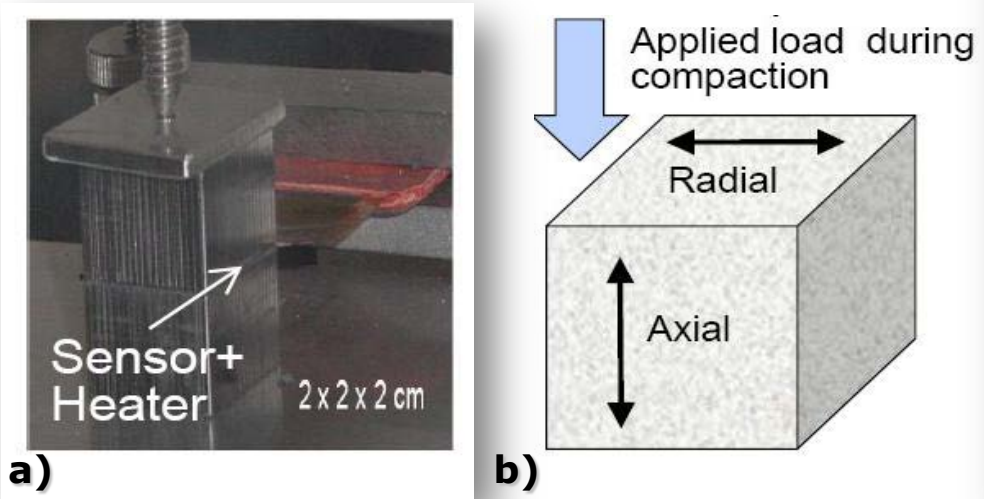
where α is the measured thermal diffusivity, ρ is the density, and C_p *is heat capacity measured by DSC.*

- **Optimal compaction: ρ = 0.5 g/cm^3 yields 350% increase in volumetric H_2 density and modest 15% reduction in gravimetric excess capacity.**
- **Thermal diffusivity measurements: xenon flash thermal diffusivity instrument, 12 mm diameter 2mm thick sample pellets under a N_2 atmosphere.**
- **Thermal conductivity increased slightly with density, but below single crystal value.**

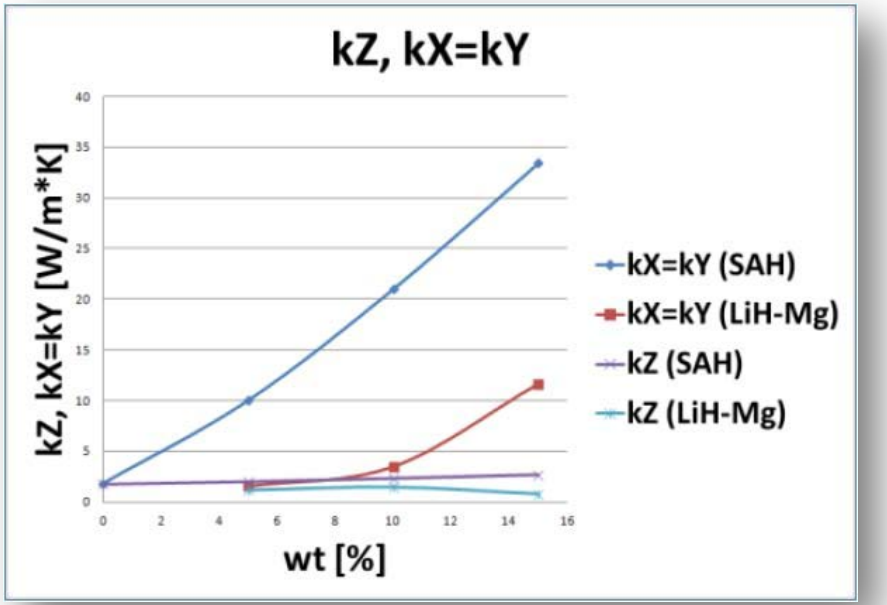
J.J. Purewal, D. Liu, J. Yang, A. Sudik, D.J. Siegel, S. Maurer, U. Muller, "Increased volumetric hydrogen uptake of MOF-5 by powder densification", International Journal of Hydrogen energy 37 (2012) 2723-2727.

Issue: Anisotropy in Thermal Conductivity

Example Study;



a) Thermal measurement system and b) Material compaction process.

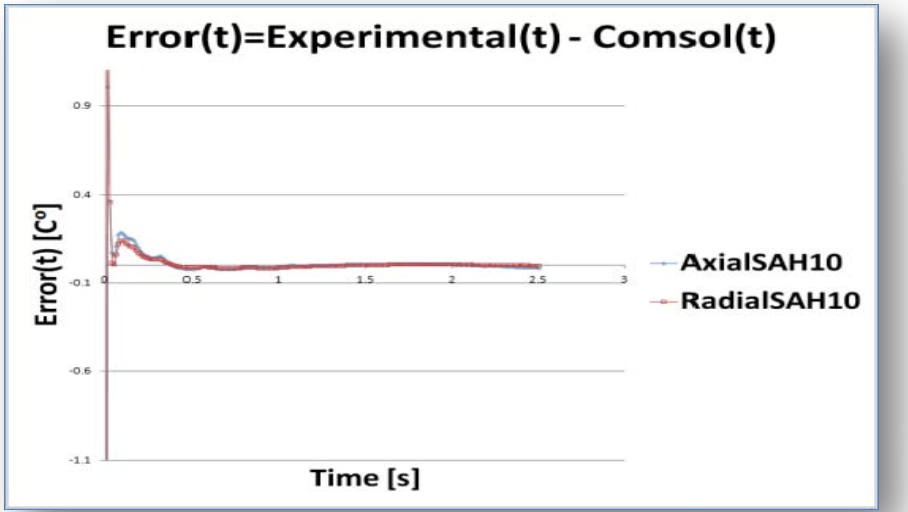


Thermal conductivity of sodium alanate as a function of cycle and phase.

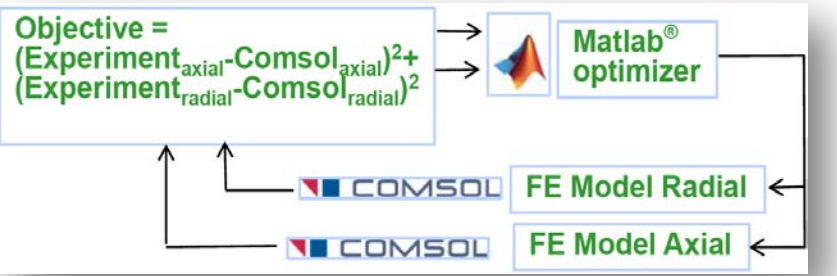
- **Example: 3 materials; Ti-doped sodium aluminum hydride (SAH), an 8:3 mixture of Lithium Hydride and Magnesium Amide (LAMH), and a Metal Organic Framework (MOF-5) were mixed with expanded natural graphite and uni-axially pressed to compact the material into cube shaped samples.**
- **Thermal conductivity was measured using a Hot Disk Thermal Constants Analyzer.**

Fedchenia, I.I., and van Hassel, B.A., "Solution of Inverse Thermal Problem for Assessment of Thermal Parameters of Engineered H2 Storage Materials", paper, Comsol Conference, Boston (2011). This material is based upon work supported by the U.S. Department of Energy under Contract No. DE-FC36-09GO19006

Example: Parametric Modeling of Measurements



The difference between Comsol model and experimental measurements.

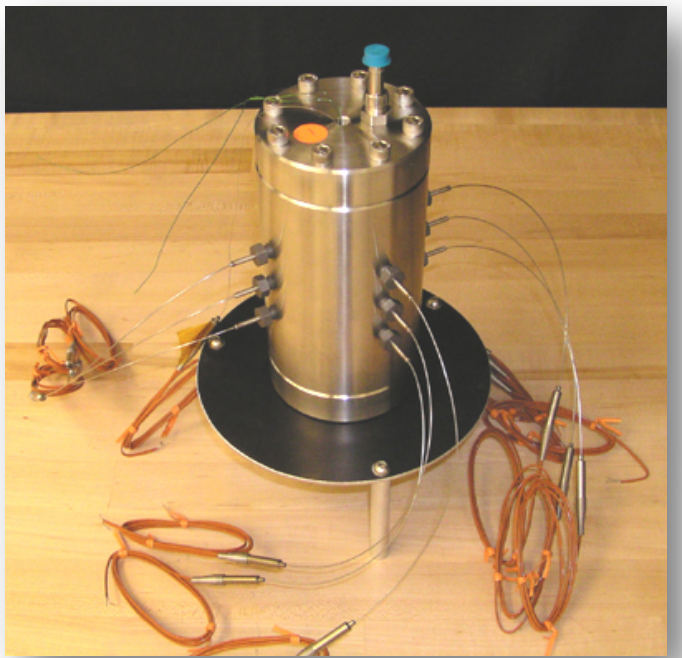


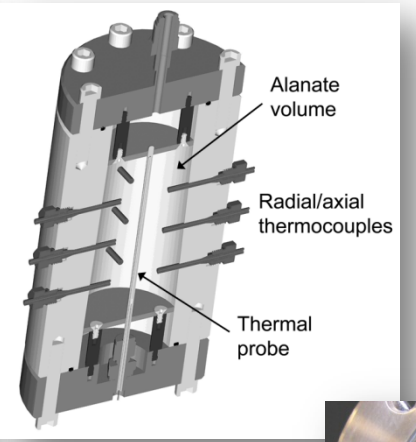
Parametric Modeling: Matlab optimizer (backward) and Comsol (forward) Solvers

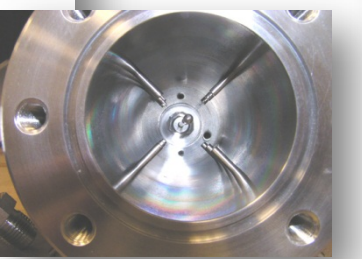
- Analytical solutions for the hot disc in infinite media becomes prohibitive for material exhibiting anisotropic properties.
- It also does not allow inclusion of the heat transfer coefficient for the boundary between the sensor and the material.
- This becomes important for the materials modified by the applied stress. Not accounting for this leads to the wrong relation between the heat applied from the hot disc and the heat transfer properties of the material.
- Also, only thermal diffusivity can be estimated from the analytical model, heat capacity must be measured in a separate experiment.
- A (Comsol) physical model was used in this example for material parameters identification.

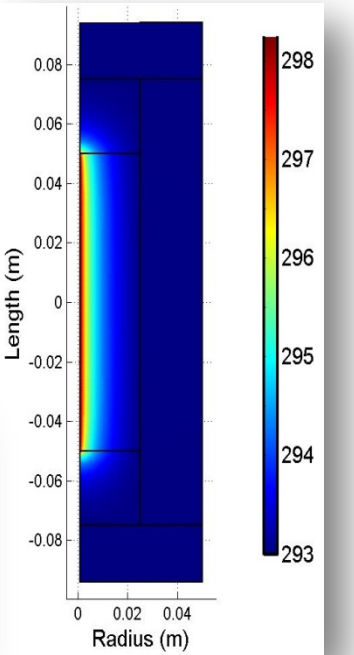
Fedchenia, I.I., and van Hassel, B.A., "Solution of Inverse Thermal Problem for Assessment of Thermal Parameters of Engineered H2 Storage Materials", paper, Comsol Conference, Boston (2011). This material is based upon work supported by the U.S. Department of Energy under Contract No. DE-FC36-09GO19006

Example: In-Situ Measurement System







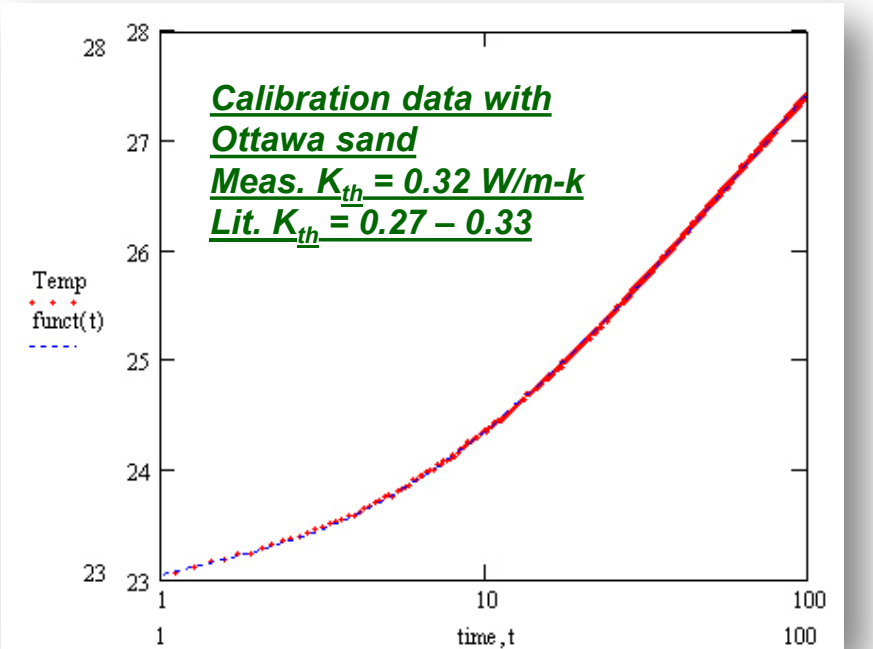


Instrumented chamber to measure: Thermal conductivity and wall resistance as a function of phase, temperature, cycle, morphology, and pressure.

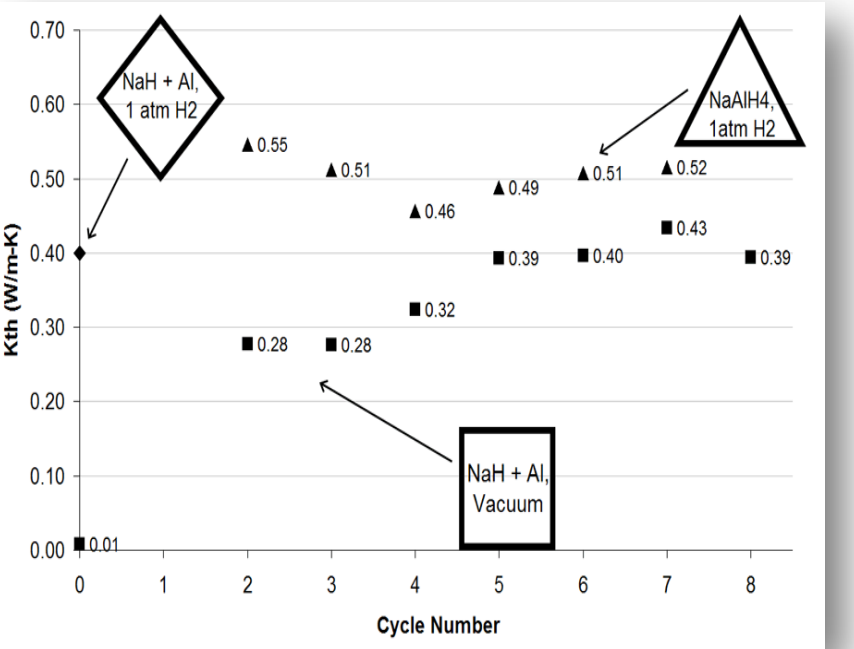
- **Ideally one would like to measure the thermal conductivity of hydrogen storage materials "In-situ", preferably, under different hydrogen pressures, temperatures, hydrogen contents, and after progressive cycling.**
- **This significantly narrows the field of methods that are applicable to such measurements..**

K. Gross et al. "Hydride Development for Hydrogen Storage", DOE Hydrogen and Fuel Cells Annual Merit Review, Berkeley, CA May 19-22, 2003. Dedrick, D.E., Kanouff, M.P., Replogle, B.C. and Gross, K.J., "Thermal properties characterization of sodium alanates", J. Alloys and Compounds, Vol. 389, (2005) p. 299–305.

Example: Results of In-situ Measurements



Calibration against a standard material.

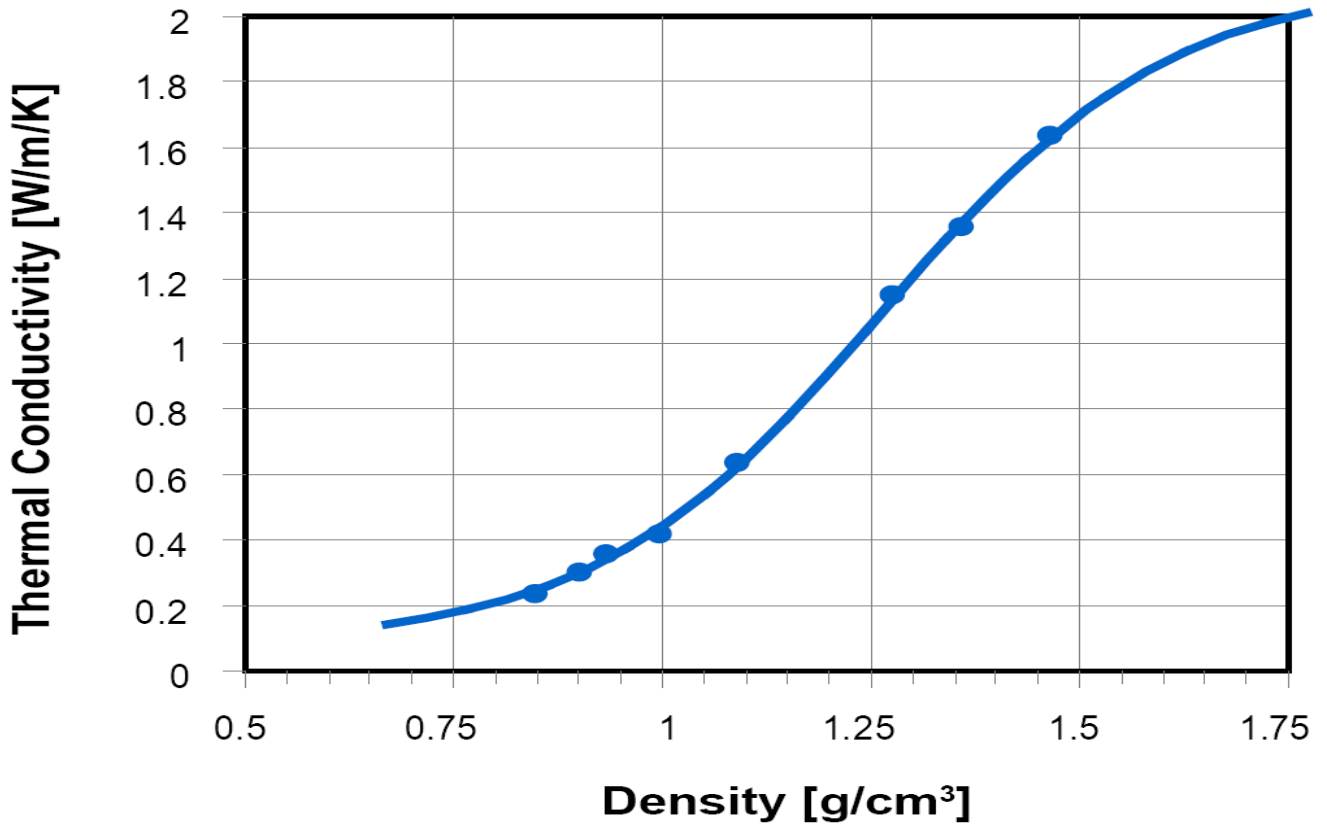


Thermal conductivity of sodium alanate as a function of cycle and phase.

- **Hydrogen gas enhances the thermal conductivity.**
- **The variation with cycle stems from morphology changes experienced during decomposition/recombination.**

Dedrick, D.E., Kanouff, M.P., Replogle, B.C. and Gross, K.J., "Thermal properties characterization of sodium alanates", J. Alloys and Compounds, Vol. 389, (2005) p. 299–305.

Example: Enhancement of Thermal Conductivity



Thermal Conductivity of Sodium Alanate Pellets as a Function of Density

- Thermal Conductivity of sodium alanate increased by almost a factor of 7 with a 39% reduction in volume through compaction at 40kpsi.

Bart van Hassel, Daniel A. Mosher, Jose Miguel Pasini, Mikhail Gorbounov, John Holowczak, Steve Hay, John Khalil, Fanping Sun, Xia Tang and Ron Brown, United Technologies Research Center (UTRC), "Advancement of Systems Designs and Key Engineering Technologies for Materials-Based Hydrogen Storage", FY 2010 Annual Progress Report, DOE Hydrogen Program

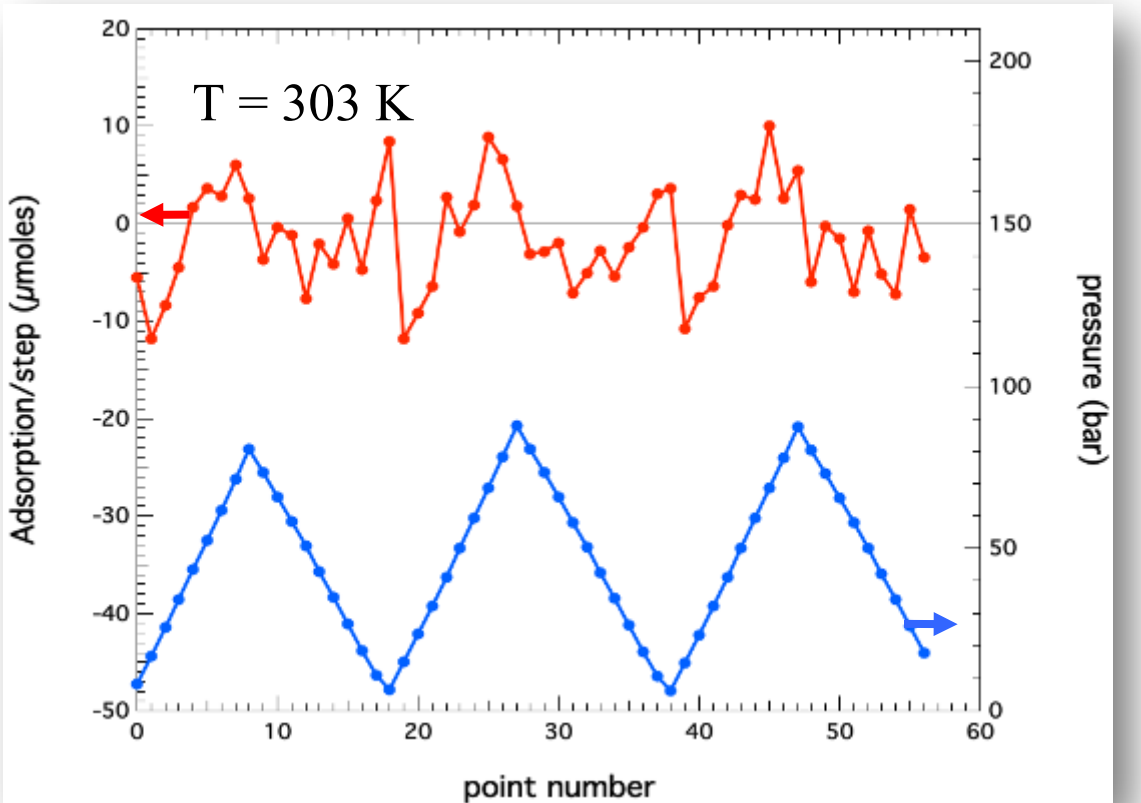
DOE Special Request

PI: Dr. Phil Parilla, NREL

- **Add a more in-depth analysis of source and propagation of errors specifically in making Physisorption PCT measurements.**
- **Provide an in-depth evaluation of differential pressure method of making PCT or Capacity measurement specifically with respect to high-surface area materials.**
- **The results of this work will be written up and incorporated into the "Best Practices" document.**
- **Improve communication of standardized terminology and reporting practices of materials and system storage capacity**
- **Both the analysis of source and propagation of errors and the evaluation of the differential pressure method are currently underway with progress being communicated with the Best Practices project.**

Collaboration: Phil Parilla, NREL
Reducing Error in Physisorption Measurements

The true performance of the system is determined by measuring zero adsorption on blank or empty sample holder



- **With empty container, get calibration constants with standard procedures using He gas**
- **Then measure empty container with H2 for 'adsorption'**
- **This tests both the hardware and the data analysis**
- **Accumulated error here was about 70 micromoles in 56 steps**

P.A. Parilla, HSCoE Technical Exchange Meeting, 2/17/10

- Future Work -

- ***Engineering Thermal Properties***
  - First draft of the Engineering Thermal Properties section will be completed.
  - Final draft will be reviewed by experts in the field.
  - Reviewer's edits and comments of the will incorporated into the final document.
- ***Engineering Mechanical Properties***
  - Outline of the first draft of the Mechanical Properties section will be completed.
  - *First draft of the Mechanical Properties section will be completed.*
  - *Final draft will be reviewed by experts in the field.*
  - *Reviewer's edits and comments of the will incorporated into the final document.*
- ***NREL Collaboration***
  - Integrate results from NREL's work on the analysis of source and propagation of errors and the evaluation of the differential pressure method into the Best Practices document (Phil Parilla).
  - Integrate procedures and methodologies learned from NREL's project on Spillover investigations and characterization/validation into Best Practices (Tom Gennett and Phil Parilla).
- ***Gas Permeability Measurements of Hydrogen Storage Materials***
- ***Compatibility Measurements of Containment Materials***

FY2012 / *Future Work*

- Project Summary -

- ***Relevance:*** To fill the need for a best practices guide for the measurement of critical performance properties of advanced hydrogen storage materials.
- ***Approach:*** Create a reference resource of best methods and caveats in measuring Target-based properties: General Introduction to Hydrogen Storage Materials and Measurements, Kinetics, Capacity, Thermodynamic, Cycle Life, Engineering Thermal Properties, and Engineering Mechanical Properties Measurements.
- ***Accomplishments:*** Task 1, 2, 3, 4, and 5 completed. Task 6 and 7 in progress.

  Achieving a high-level of participation from experts in the field.
- ***Collaborations:*** Official collaboration with NREL, multiple co-authors and International collaboration through IEA task 22 as well as industry.
- ***Future Work:*** FY2012 - Finalize Task 6 (Engineering Thermal Properties), commence Task 7 (Engineering Mechanical Properties), and Integration of applicable NREL work.
- ***Document:***

http://www1.eere.energy.gov/hydrogenandfuelcells/pdfs/best_practices_hydrogen_storage.pdf

Thank You!

H2 Technology Consulting LLC

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