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Thermal Conductivity Measurements of Metal Hydride Compacts Developed for High-Power Reactors

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Thermal conductivity measurements on porous metal hybride compacts were performed to supplement the limited existing data. These materials are proposed for reactors that are designed for heat-pump applications requiring high specific powers. Previous computational studies have shown that the effective thermal conductivity k_{eff} is a crucial optimization parameter. If it is too low, the reactors themselves limit the system performance. It is sufficiently high, external thermal resistances dominate and overdesign of the materials is unnecessary and unavoidably increases parasitic thermal losses. In this study nine samples were tested using the comparative method and careful attention was paid to ascertaining and propagating all errors into the final data reported.

Nomenclature

C_p = heat capacity
F_Q = ratio of parasitic thermal capacity to the parasitic capacity of the metal hydride, Eq. (5)
f = mass fraction
h_c = contact conductance
k = thermal conductivity
k_d = thermal conductivity of fully dense material
k_{eff} = stagnant thermal conductivity
k_{pd} = thermal conductivity of powder beds
L = length
M = metal
m = mass
P_{H2} = hydrogen gas pressure
Q_{mh} = parasitic capacity of the metal hydride
Q_w = parasitic thermal capacity of the porous metal hydride compact
T = temperature
$T_{\text{III,O}}$ = temperature at III,O, Fig. 5
$T_{\text{II,L}}$ = temperature at II,L, Fig. 5
$T_{\text{II,O}}$ = temperature at II,O, Fig. 5
$T_{\text{I,L}}$ = temperature at I,L, Fig. 5
T_1 = temperature at thermocouple location 1
T_2 = temperature at thermocouple location 2
T_3 = temperature at thermocouple location 3
T_4 = temperature at thermocouple location 4
T_5 = temperature at thermocouple location 5
t = shell thickness
U_h = estimated uncertainty for h_c
U_{kc} = estimated uncertainty for SS304 calibrated sample
U_L = estimated uncertainty for sample length
U_T = estimated uncertainty at 95% confidence limit for temperature measurement
x = heat flow direction
Δx = spacing between thermocouple 1 and 4
ΔT = temperature swing
ξ = particle diameter
ρ = density

Subscripts
I = meter bar
II = sample
III = Cu sink
mh = metal hydride

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Introduction

METAL hydrides have been considered for use in heat pumps, air conditioners, thermal compressors for cryocoolers, and hydrogen storage beds for many years.[1–5] Despite rapid kinetics, technological applications must deal with decrepitation of most metal hydrides into micron-sized powders after several absorption/desorption cycles. The thermal conductivity of powder beds ($k_{\text{pb}} \sim 10^{-1}$ W/m K) is much lower than that of fully dense materials ($k_d \sim 10^{1}$ W/m K), mainly as a result of contact resistances between particles. To realize the potential of the rapid kinetics, materials fabrication must focus on lowering the internal thermal resistance of metal hydride reactors to increase k_{eff}.

The concept of porous metal hydride (PMH) compacts was introduced by Ron et al.[6] and Congdon.[7] In the approach of Ron et al., metal hydride and aluminum powders were mixed, compacted, and sintered. The thermal conductivity of such compacts was reported to be from 1 to 10 W/m K for mass fractions of aluminum between 0.1 and 0.5. The disadvantages with this process are that sintering must be performed under high pressure and the parasitic thermal losses because of aluminum are significant ($C_p \sim 907$ J/kg K). In the method of Congdon, an organic binder contains powders. However, long-term structural stability of PMH compacts using an organic binder is questionable given the large and rapid reactor temperature fluctuations. Measured data of k_{eff} are not available for this composite.

In previous work,[3,8] the authors proposed an alternative technique in which metal hydride particles are plated with thin copper shells and cold-compacted with low melting point metal binder. The plating process is based on the work described in Park and Kirchheim.[9] Preparation of these materials

is straightforward and does not require high pressure. The authors have demonstrated excellent long-term structural integrity. The major objective of this paper is to report experimental thermal conductivity data obtained for such PMH compacts.

Preparation of PMH Compacts

The rare-Earth intermetallic hydride, $LaNi_5$ (purity greater than 99.5%, from Japan Metal and Chemical Company), was used in all the work reported here. It has a nominal crystalline density of 8.3 g/cm^3, a heat absorption of 32 kJ/gmole of H_2, a plateau pressure of 301 kPa at 30°C, and a hydrogen uptake capacity of 1.1 wt%.[10–12] Figure 1 shows both the hydrogen pressure/atom ratio (P_{H2} vs H/M) and the van't Hoff (P_{H2} vs $-1/T$) diagrams.

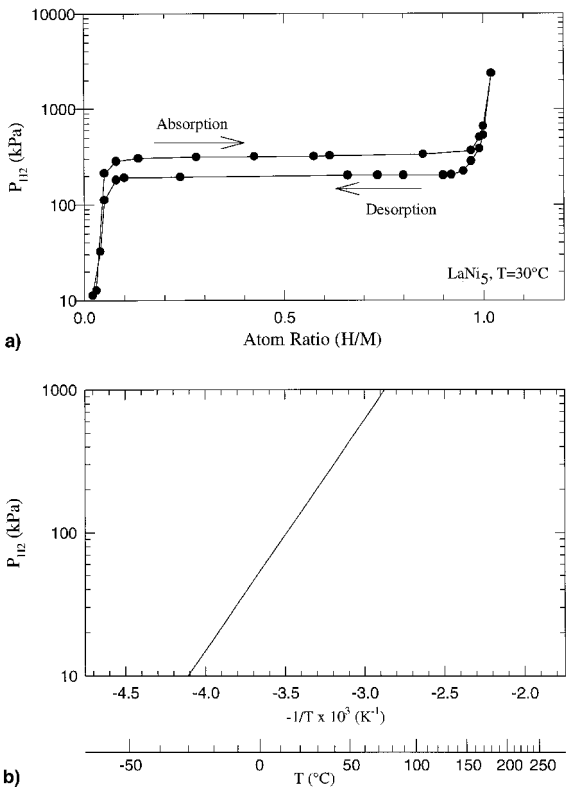

Fig. 1 Thermodynamic properties of $LaNi_5$: a) H_2 pressure vs atom ratio (H/M), and b) H_2 pressure vs negative inverse temperature.

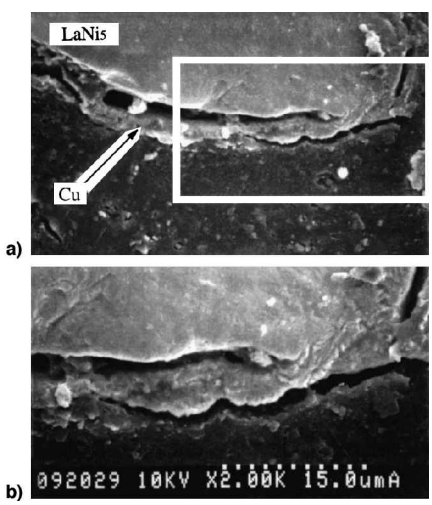

Fig. 2 Scanning electron micrograph photography of copper-plated $LaNi_5$: a) $LaNi_5$ encapsulated with copper, and b) 2X enlarged portion of the boxed area. (The scale shown in the bottom corresponds to the top.)

Table 1 Preparation summary for the nine tested samples

Sample	F, kpsi	f, mass fraction	$m_{LaNi5+Cu}$ g	m_{Sn} g	m_{final} g	L, mm	D, mm
1	27.0	0.05	1.999	0.106	2.090	5.38	9.56
2	27.0	0.10	1.999	0.222	2.200	5.49	9.56
3	27.0	0.15	2.001	0.351	2.330	5.66	9.57
4	36.0	0.05	1.998	0.105	2.091	5.19	9.58
5	36.0	0.10	2.000	0.225	2.215	5.36	9.57
6	36.0	0.15	1.998	0.350	2.341	5.47	9.54
7b	45.0	0.05	1.999	0.108	2.090	5.10	9.60
8	45.0	0.10	1.999	0.220	2.209	5.24	9.56
9	45.0	0.15	2.001	0.354	2.347	5.39	9.56

An optimal copper plated particle is shown in Fig. 2. It has an approximately 1 μm thickness and a small void region between the copper and hydride. Trials were done to evaluate the effect of temperature, mixing speed, and concentration. Using the selected process, nine PMH compacts were prepared as follows:

1) As-received $LaNi_5$ was ball-milled and sieved. Particles collected were distributed from 25 to 45 μm. The powder was washed thoroughly with ethanol to remove all fines. The powder remaining was resieved mechanically and vacuum dried.

2) Three grams of $LaNi_5$ particles were immersed in 100 cm^3 of the plating solution at room temperature for 2 min. The plating solution, containing 1.5-g $CuSO_4$ and 0.8-ml H_2SO_4 per 100 cm^3, was contained in a Pyrex® vessel and vigorously agitated by a glass stirring rod and Pyrex paddle at 1250 rpm. The plated powder samples were removed and washed over filter paper with ethanol and vacuum dried.

3) The plated powder was mixed with Sn powder (−325 mesh) and cold-pressed in a 0.953-cm (3/8-in.) die at three compaction pressures. The green pellets were sturdy. Only a small fraction of the initial sample was lost during pressing (<−0.6% average). Table 1 is a summary of the nine samples that were tested.

Experimental Setup and Calibration

The comparative method was adopted for thermal conductivity measurements.[13–15] In this method the test sample is placed between a reference material with known thermal conductivity and a constant temperature sink (Cu was used). A heat flux is induced through the sample and the apparatus allowed to achieve steady state. In the absence of convective or radiative losses and using a meter bar of sufficient length, one-dimensional Fourier analysis can be applied. This method is useful for materials with thermal conductivities in the $0.2 < k < 200$ W/m K range.[16] The disadvantage of this method when thermocouple wells cannot be placed within the sample, as was the case here (nondestructive testing), is that the interfacial contact resistance between the sample and reference must be calibrated.

The experimental apparatus consists of the meter bar and embedded heater, constant temperature sink, and sample. The apparatus was enclosed in a vacuum chamber (<10^{-3} torr). Figure 3 shows the experimental apparatus. The meter bar has a diameter of 0.953 cm (0.375 in.), giving a cross-sectional area of 0.7125 cm^2, and is constructed of 304 stainless steel (SS). A literature search was conducted to determine the thermal conductivity of SS304. The data obtained are plotted in Fig. 4.[16–20] Prior research[20] concluded that SS304 is a good reference material because it shows relatively small scatter in the thermal conductivity. A value of 14.9 ± 0.4 W/m K (for sample temperatures 20–35°C) was used, with the uncertainty propagated in the data reduction.

Matched thermocouples (Omega), copper–constantan, with a wire diameter of 5.08×10^{-3} cm (0.002 in.) were used after lacquering and spacing with epoxy beads. Heat sink grease was placed into the wells (well i.d. = 0.05 cm) with a hypodermic

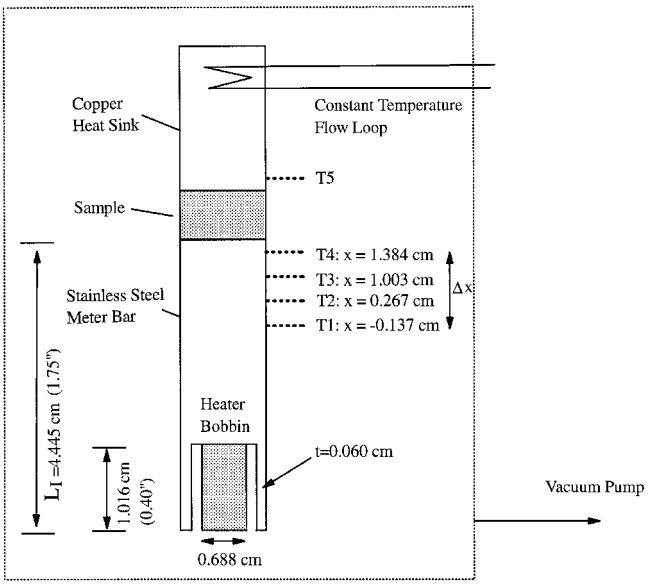

Fig. 3 Experimental apparatus.

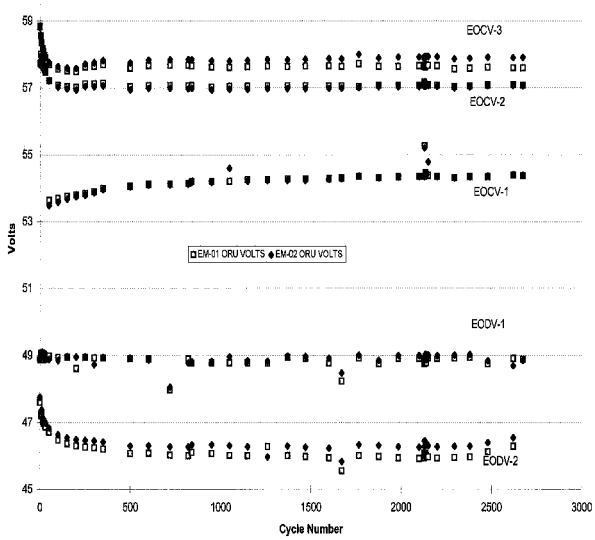

Fig. 4 Thermal conductivity data from published literature.

needle prior to inserting the thermocouples. The thermocouple wells were drilled on a micropress to the center of the meter bar. A micrometer stage was used to measure the thermocouple well spacing. The wells adjacent to the contact faces were placed to within 0.025 cm (0.01 in.), allowing the temperatures to be taken as the interfacial temperatures with negligible error. The following text briefly describes the relevant experimental procedure used to calibrate the thermal resistance of the interface:

1) Both faces of the interface were coated with ~0.03 cm^3 of silicon heat sink compound to reduce the contact resistance and achieve reproducible results.

2) Power, temperature, and vacuum readings were made at intervals spanning at least 3 h. Temperature readings were made by direct voltage measurement of the thermocouple junction potential with respect to reference junctions placed in an ice-bath. The ability of the ice-bath to maintain correct temperature within ±0.01°C was verified using a calibrated platinum resistance temperature detector probe. The junction voltages were converted to temperature using linear interpretation between National Institute of Standards and Technology values of the standard voltages for type *T* thermocouples.

3) A reference sample was aligned between the meter bar and sink and a constant pressure was applied.

Figure 5 shows the idealized apparatus for analysis when a sample is in place. The contact conductance h_c was estimated using a SS304 sample and uncertainty in the estimate quantified. It should be noted that the estimated value of h_c is deduced from a system of two solid–solid interfaces. Possible systematic uncertainty introduced by a shift in h_c from solid-to-porous interfaces is not known.

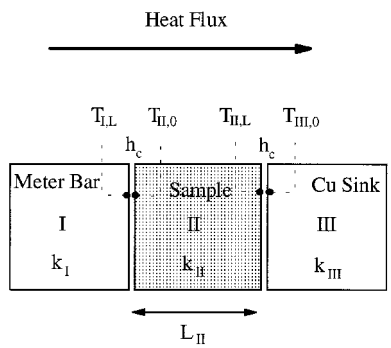

Fig. 5 Idealization of apparatus: two interfaces.

Based on the conservation of energy, the data are reduced by expressing h_c in the following form:

$$h_c = \frac{2k_I k_{II} \left.\frac{dT}{dx}\right|_I}{k_{II}T_{III,O} - k_{II}T_{I,L} - L_{II}k_I \left.\frac{dT}{dx}\right|_I} \tag{1}$$

where it is assumed that the values of h_c for both interfaces are the same. Letting $k_I = k_{II} = k_c$ (test sample is the same as the meter bar) gives

$$h_c = \frac{2k_c \left.\frac{dT}{dx}\right|_I}{T_{III,O} - T_{I,L} - L_{II}\left.\frac{dT}{dx}\right|_I} = \frac{2k_c \frac{(T_4 - T_1)}{\Delta x}}{T_5 - T_4 - L_{II}\frac{(T_4 - T_1)}{\Delta x}} \tag{2}$$

where $\Delta x = 1.521$ cm and $L_{II} = 0.993$ cm, respectively. The uncertainty U_h in this data reduction equation can be expressed as[21]

$$U_h = \sqrt{\left(\frac{\partial h}{\partial T_1}U_T\right)^2 + \left(\frac{\partial h_c}{\partial T_4}U_T\right)^2 + \left(\frac{\partial h_c}{\partial T_5}U_T\right)^2 + \left(\frac{\partial h_c}{\partial k_c}U_{k_c}\right)^2 + \left(\frac{\partial h_c}{\partial L_{II}}U_L\right)^2} \tag{3}$$

The data obtained are shown in Table 2. An average $\bar{h}_c$ of 17,730 ± 7100 W/m^2 K was obtained where the stated uncertainty is the 95% confidence limits of the estimated $\bar{h}_c$. As expected, the uncertainty accompanying the measurement of h_c is quite substantial. Nevertheless, the uncertainty was propagated through when the sample data were reduced. For some samples the influence is rather small. For other samples the error was large. However, the overall consistency of the data will provide some measure of validity to the resulting conclusions.

Table 2 Calibrated contact conductance

T_1, °C	T_4, °C	T_5, °C	h_c, W/m^2 K	U_{hc}, W/m^2 K
31.170	28.125	25.750	15,540	4,440
30.775	27.725	25.375	16,620	5,050
31.625	28.625	26.425	24,280	10,710
32.025	29.075	26.750	14,460	3,990
——	——	——	$\bar{h}_c$ = 17,730 ± 7,100	6,047

Results and Discussion

In this section we present the final data from this study and compare it with that of Ron,[6] which is the only other set of data known to the authors. The primary focus of these measurements was to ascertain whether k_{eff} for the preparation process described is adequate to meet optimized performance criteria developed from an analytical model of transient compressible porous flow in a coupled reactor heat-pump configuration.[22] Briefly, the data show that the measured green values are indeed sufficient. Although final trials to quantify k_{eff} for activated samples have not been completed, calorimetric studies from several prototype reactors indicate that the green values here are reasonable estimates.[3,8] Because of the complicated morphology of the materials in this study and the difficulty in developing correlations for powder beds undergoing large volumetric strains during absorption/desorption and the effect of hydrogen in the actual problem,[23] no attempt is made here to model the data themselves.

Conservation of energy gives the following equation used to calculate thermal conductivities for samples 1–9, with the value of $\bar{h}_c$ determined in the previous section

$$k_{\text{II}} = \frac{L_{\text{II}} k_{\text{I}} [(T_4 - T_1)/\Delta x]}{T_5 - T_4 - 2(k_{\text{I}}/\bar{h}_c)[(T_4 - T_1)/\Delta x]} \tag{4}$$

The uncertainty $U_{k\text{II}}$ in this data reduction equation was obtained using a symbolic mathematical package and $U_{k\text{I}}$, U_T, $T_{L\text{II}}$, and U_{hc} propagated. The expression is quite complex and was coded in Fortran for use. Based on Eq. (4) and the expression of $U_{k\text{II}}$, the thermal conductivity and associated uncertainty of k_{eff} of the nine samples was determined. The results are given in Table 3 for each trial. The confidence interval for the mean of each set of three experimental trials at the 95% confidence level is also given in Table 3.

It is well-known that thermal conductivity affects the overall system performance in metal hydride sorption heat pumps. However, prior calculations by the authors have showed asymptotic behavior in the parameter k_{eff}. These include comprehensive transient fully compressible coupled flow calculations as well as a more simplified thermodynamic analysis.[22,24] Increasing k_{eff} dramatically improves system performance to a point. Values of $k_{\text{eff}} > 5$–10 W/m K appear to be optimal.[22] Based upon the data in Table 3 these values are achievable.

A second and equally important gauge of reactant suitability is the amount of parasitic thermal mass in the final composite. The authors believe that the most meaningful comparison between PMH compacts prepared by different processes with different constituents is based on heat capacity weighted mass fraction of the inactive (nonmetal hydride) constituents. This is because the inactive material is detrimental from the standpoint of being a parasitic thermal reservoir in many applications of metal hydrides. We define F_Q as

$$F_Q = \frac{Q_w}{Q_{\text{mh}}} = \frac{m_w C_{p,w} \Delta T}{m_{\text{mh}} C_{p,\text{mh}} \Delta T} \tag{5}$$

This is the ratio of parasitic thermal capacity Q_w to the parasitic capacity of the metal hydride Q_{mh}. The ratio $Q_w/(Q_w + Q_{\text{mh}})$ is not used because the parasitic thermal capacity of the metal hydride is inherent to the active material. Note that F_Q may exceed unity. A value of unity simply means that the parasitic thermal effect of the inactive material equals that of the irreducible part caused by the hydride itself. Values of F_Q that are small compared with unity are desired for heat pump operation. To express the data of Ron[6] in terms of F_Q, the authors have assumed that his PMH compacts consisted exclusively of aluminum, metal hydride, and open pore space. In this case

$$F_Q = \frac{f_{\text{Al}}}{1 - f_{\text{Al}}} \frac{C_{p,\text{Al}}}{C_{p,\text{mh}}} \tag{6}$$

In our process two different inactive constituents (Cu and Sn) are present with f_{Sn}, a known parameter. For this situation

$$F_Q = \frac{f_{\text{Cu}} C_{p,\text{Cu}} + f_{\text{Sn}} C_{p,\text{Sn}}}{f_{\text{mh}} C_{p,\text{mh}}} \tag{7}$$

Neglecting the enclosed void space we can write the remaining mass fractions in terms of the particle radius ξ and shell thickness t

$$f_{\text{Cu}} = \rho_{\text{Cu}}(1 - f_{\text{Sn}}) \left\{ \frac{1}{[\xi/(\xi + t)]^3(\rho_{\text{mh}} - \rho_{\text{Cu}}) + \rho_{\text{Cu}}} - \frac{1}{(\rho_{\text{mh}} - \rho_{\text{Cu}}) + [\xi/(\xi + t)]^3 \rho_{\text{Cu}}} \right\} \tag{8}$$

$$f_{\text{mh}} = \rho_{\text{mh}}(1 - f_{\text{Sn}}) \left\{ \frac{1}{(\rho_{\text{mh}} - \rho_{\text{Cu}}) + [(\xi + t)/\xi]^3 \rho_{\text{Cu}}} \right\} \tag{9}$$

Table 3 Thermal conductivity of samples 1–9

Sample	k_{II}, W/m K	$U_{k\text{II}}$, W/m K
1	2.9	0.2
	2.9	0.2
	2.4	0.1
	2.7 ± 0.7	——
2	3.0	0.2
	3.2	0.2
	3.3	0.2
	3.2 ± 0.4	——
3	3.1	0.2
	3.0	0.2
	3.3	0.2
	3.1 ± 0.4	——
4	2.9	0.2
	3.0	0.2
	3.2	0.2
	3.0 ± 0.4	——
5	3.4	0.2
	4.1	0.3
	4.2	0.3
	3.9 ± 1.1	——
6	5.2	0.4
	4.9	0.3
	5.7	0.4
	5.3 ± 1.0	——
7b	3.7	0.2
	3.9	0.2
	3.8	0.2
	3.8 ± 0.2	——
8	5.0	0.3
	5.2	0.4
	5.4	0.4
	5.2 ± 0.5	——
9	6.2	0.5
	6.6	0.5
	6.5	0.5
	6.4 ± 0.5	——

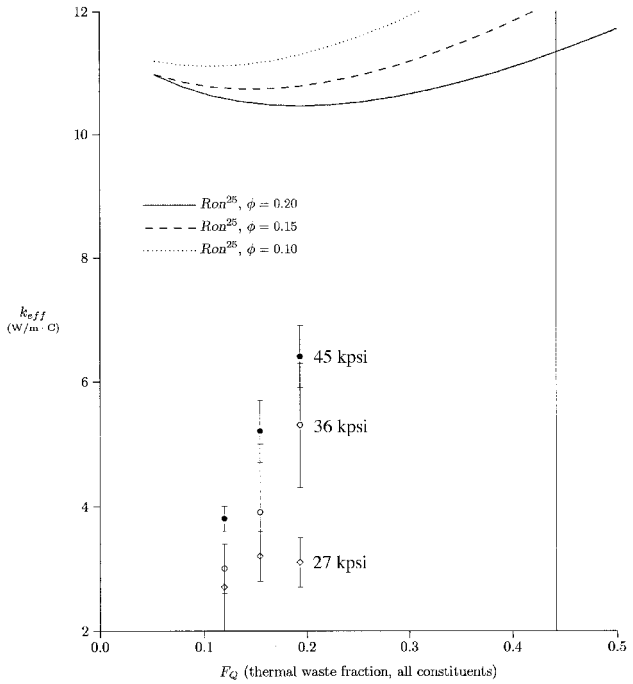

Fig. 6 Stagnant thermal conductivity under vacuum for metal hydride reactants prepared using the PMH Process (Cu-encapsulated $LaNi_5$ with Sn binder). Data from a recommended correlation for another PMH material (compacted aluminum and metal hydride powders) are plotted for illustration. Metal hydride reactors for use in heat pump applications are optimized by ensuring $k_{eff} \geq 5$ (W/m K) while minimizing F_Q as much as possible.

The following values for the densities were used: $\rho_{mh} = 8.231$, $\rho_{Cu} = 8.940$, and $\rho_{Sn} = 7.310$ g/cm^3, respectively. The following values of C_p were used: $C_{p,mh} = 418$, $C_{p,Cu} = 385$, and $C_{p,Sn} = 227$ J/kg K, respectively. Using Eqs. (7–9) it is possible to estimate the uncertainty U_{FQ} of F_Q in the nine samples. This was done assuming $t < 2$ μm. In this case, F_Q does not exceed 0.3.

Figure 6 shows a comparison between the present work and the data of Ron.[6,25] The results of the experiments reported here are presented with the associated uncertainty in F_Q and k_{eff}. Ron's data[6,25] are based upon his correlations. The uncertainty in his results was not stated. As can be seen, f_{Sn} needs to be at least 0.1 to achieve good values of k_{eff} and the compaction pressure must be more than 36 kpsi. It is important that the plating thickness of Cu be carefully controlled to minimize F_Q while maintaining good values of k_{eff}. A secondary consideration in the development of an adequate composite structure is the need to maintain long-term integrity and ensure sufficient permeability. The vertical line in Fig. 6 corresponds to the F_Q reported in the experiments of Ron,[26] where $f_{Al} \approx 0.18$.

Summary

Cu-encapsulated PMH compacts made with $LaNi_5$ were prepared and green values of k_{eff} obtained using the comparative method. A careful error analysis was performed to quantify the uncertainties in the method. The mass fraction of binder and compaction load were varied. k_{eff} was found to increase linearly with both parameters when $f_{Sn} \geq 0.10$. Values of k_{eff} ranging from 2.7 ± 0.7 to 6.4 ± 0.5 W/m K were measured. The measurements indicate that k_{eff} can be made sufficiently high for technological applications. The use of Sn as a binder, with its much lower parasitic mass, in conjunction with properly controlled encapsulation thickness resulted in values of F_Q lower than comparable PMH techniques.

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