Flash Methods to Examine Diffusivity and Thermal Conductivity of Metal Foams

A Comparison with Data of a Comparative Set-Up

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ABSTRACT

The analysis of the standard uncertainty of thermal conductivity results obtained by a comparative set-up motivated the use of a laser flash device to determine the thermal conductivity of metallic foam materials. In particular a Magnesium alloy is investigated. To meet the requirements of flash techniques coplanar samples are prepared. Therefore the surface near open porosity is filled with a ceramic paste (e.g. SiC). A finite element model is generated to study the influences of the preparation method to the measurement results. This enables to separate the conductivity behaviour of the foam structure from the inhomogeneity effects of the prepared sample. Results from flash based diffusivity measurements are used to examine thermal conductivity using $\lambda = \rho \, x \, c_p \, x \, a$. Data are compared with those obtained from a comparative set-up. For both methods a detailed analysis of the uncertainty of the measurement results is done. Results from both methods are within the overlapping uncertainty levels of both methods. A significant improvement of the uncertainty level of conductivity data obtained from laser flash data could be confirmed.

INTRODUCTION

Metallic foams represent a specific class of materials in engineering and design. Therefore one has to know their basic thermo-physical properties: thermal expansion, heat capacity and thermal conductivity. In former measurement campaigns a comparative set-up was used. It manages the relatively high thermal conductivity of ~10 W/m.K in a sufficiently wide temperature range of [T_R, ~400°C]. But the method suffers from its dependency of a highly accurate temperature measurement of a set of 6 thermocouples minimum. Therefore an alternative technique to perform conductivity data was searched. Here a method to measure thermal diffusivity of foamy materials with a laser flash [1-6] is described. The requirement of a coplanar specimen is met by filling the surface near porosity with a ceramic paste. The thermal conductivity is calculated from diffusivity data using $\lambda = \rho \times c_p \times a$.

Conductivity results are compared with those obtained from the comparative set-up. The standard uncertainty of results from both methods is estimated in accordance to the recommendations of the GUM (guide to express uncertainty in measurement results) [7]. To regard to the influences of the filler material in the open surface porosity a finite element (FE) - based simulation of the transient and spatial heat propagation was done. The superposition of the transient spatial heating of the ceramic filler and the metallic foam and its consequences to the IR detection of the LF were analysed. As a result the transient temperature response of the metallic body itself can be used to evaluate its thermal diffusivity and conductivity as well.

EXPERIMENTAL AND FE-CONCEPT

Preparation Method: Technique and Consequences

Of a coplanar prepared cylindric specimen as shown in Figure 1 any open porosity near the top and bottom surface of the specimen is filled with a ceramic paste (e.g. SiC). Its thermal conductivity is significantly lower than those of the metallic body. In comparison to the filling with a high conductivity paste or the closing of the top and the bottom side of the sample with a high-conductivity foil this showed the best results. Before flashing – but after the drying of the paste – the sample is grinded. The IR detector sees a coplanar specimen which is non-transparent for the laser beam. But the ceramic filler material in the surface near porosity influences the heat transfer in the metallic structure. And from principal it is in conflict to the assumption of a homogeneous material. A superposition of the transient temperature responses of both the metallic structure and the ceramic filler occurs.

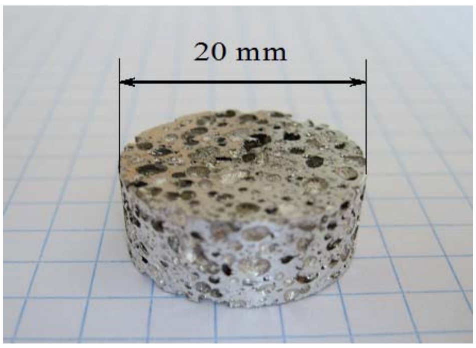

Figure 1: LFA sample of a Mg – foam material

The IR device detects an integral of the spatial thermal radiation. This causes an average transient temperature response and a shift in the half time. To evaluate the thermal diffusivity of the foam material its specific half time must be known. Thus the different runtimes of the heat pulses at the rear side of the specimen in the metal and in the ceramic paste must be separated.

Comparative Set-Up versus Laser Flash

Methods and the basic equations to determine thermal conductivity (Comparative Set-Up) [8] and thermal diffusivity (Laser Flash) [9] are formulated in equations (1) and (2) respectively. In accordance to the GUM (Guide to Express Uncertainty in Measurement Results: ENV 13005) [7] the corresponding mathematical models to estimate the Equipment Specific Uncertainties *ESU* are given there too. The used symbols are explained in TABLE 1.

As shown in Figure 2 in case of a comparative conductivity measurement the dependency of thermal conductivity data strongly correlates to the uncertainty of the input estimate of the temperatures $u^2(\Delta T)$. This significantly results from the dependency of the $ESU(\lambda)$ from $u^2(\Delta T)$. Under optimum conditions ($\lambda_S \simeq \lambda_R$) it is minimum 10% of the measured conductivity. By contrast flash results show a rather low *ESU* – mostly less than 1% of the measured diffusivity data (Figure 3). To consider effects from the measured samples the standard deviation of the individual measured results SDV^2 is added to the ESU^2 (Gaussian theory) to estimate the standard uncertainty u^2_c. To derive the $ESU^2(a)$ Parkers law is applied. In general specific theories are used to examine thermal diffusivity $a(T)$ from the detected $T(t)$ curve. The accuracy of the value $a(T)$ strongly depends on the fit-quality from this theoretical approach [10 - 15]. In the theoretical model to estimate the standard uncertainty $u^2_c(a)$ the standard deviation $SDV^2(a)$ is used to capture effects from the fit-quality.

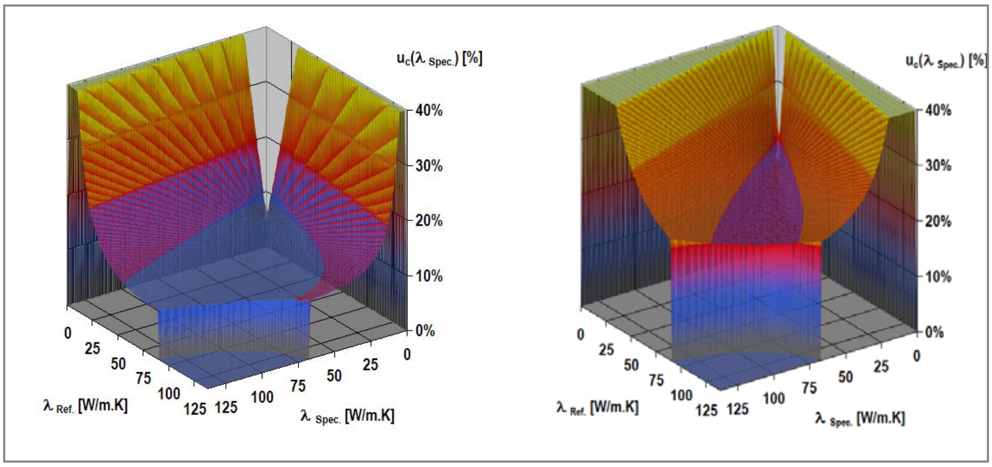

Figure 2. Comparative device: Equipment Specific Uncertainty *ESU* of thermal conductivity data.
LEFT: Uncertainty of the temperature input $u(\Delta T)$ = 1K. $u(\lambda)$ = 3%; $u(\Delta l)$ = 0,1mm.
RIGHT: Uncertainty of the temperature input $u(\Delta T)$ = 3K. $u(\lambda)$ = 3%; $u(\Delta l)$ = 0,1mm.

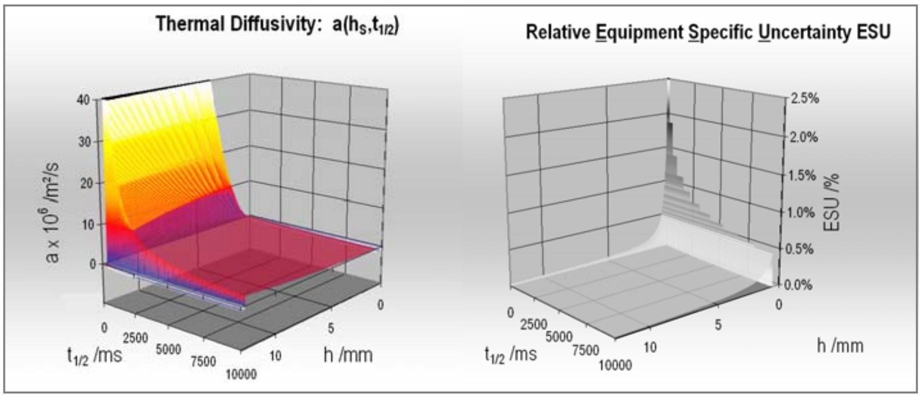

Figure 3. Expected uncertainties from Laser Flash data:
LEFT: Diffusivity data in dependence from sample thickness and half time
RIGHT: Equipment Specific Uncertainty *ESU* of thermal diffusivity data.

The Mg foam materials show a diffusivity a ~20 x 10^{-6} m²/s. To examine the transient *T(t)*-response about 5000 temperature values are detected within the required observation time: (~10000 ms). With respect to versatile sample dimensions of h ~ 10 mm and ϕ ~ 20 mm this results to an *ESU(a)* < 1% of the measured value.

TABLE 1. Mathematical Symbols

Comparative set-up	
λ_R; λ_S	thermal conductivity of the reference / sample
Δl_S	distance between thermocouples in the sample superscript u; l indicates upper / lower reference body
Δl_R	distance between thermocouples in a reference body superscript u; l indicates upper / lower reference body
ΔT_S	temperature difference between thermocouples in the sample superscript u; l indicates upper / lower reference body
ΔT_R	temperature difference between thermocouples in a reference body superscript u; l indicates upper / lower reference body
Flash device	
a	thermal diffusivity
h	height of a laser flash specimen
$t_{1/2}$	half time of the transient temperature response in a flash experiment
Others	
a	thermal diffusivity
u	uncertainty of an individual input estimate
$u_C(x)$	standard uncertainty of an output estimate x - defined as a function of serveral input estimates: $x = f(x_1, \ldots, x_i; \ldots x_n)$
ESU	Equipment Specific Uncertainty
SDV	standard deviation of a set of data

$$\lambda_S(T) = \frac{1}{2} \cdot \lambda_R(T) \cdot \frac{\Delta l_S}{\Delta T_S} \cdot \left[\frac{\Delta T_{R;h}}{\Delta l_{R;h}} + \frac{\Delta T_{R;l}}{\Delta l_{R;l}} \right]; \qquad u_c^{\,2}(\lambda) = ESU^2(\lambda) + SDV^2(\lambda_i) \tag{1}$$

$$ESU^2(\lambda) = \left(\frac{\Delta l_S}{2 \cdot \Delta T_S} \cdot \left[\frac{\Delta T_R^u}{\Delta l_R^u} + \frac{\Delta T_R^l}{\Delta l_R^l} \right] \right)^2 \cdot u^2(\lambda_R) +$$
$$+ \left(\frac{\lambda_R}{2 . \Delta T_S} \cdot \left[\frac{\Delta T_R^u}{\Delta l_R^u} + \frac{\Delta T_R^l}{\Delta l_R^l} \right] \right)^2 \cdot u^2(\Delta l_S) + \left(\frac{\lambda_R . \Delta l_S}{2 . \Delta T^2{}_S} \cdot \left[\frac{\Delta T_R^u}{\Delta l_R^u} + \frac{\Delta T_R^l}{\Delta l_R^l} \right] \right)^2 \cdot u^2(\Delta T_S) +$$
$$+ \left(\frac{\lambda_R . \Delta l_S}{2 . \Delta T_{S.}} \cdot \left[\frac{1}{\Delta l_R^u} + \frac{1}{\Delta l_R^l} \right] \right)^2 \cdot u^2(\Delta T_R) + \left(\frac{\lambda_R . \Delta l_S}{2 . \Delta T_S} \cdot \left[\frac{\Delta T_R^u}{\left(\Delta l_R^u\right)^2} + \frac{\Delta T_R^l}{\left(\Delta l_R^l\right)^2} \right] \right)^2 \cdot u^2(\Delta l_R)$$

$$a(T) \cong -\frac{\ln(1/4)}{\pi^2} \cdot \frac{h^2(T)}{t_{1/2}(T)}; \qquad u_c^2(a) = ESU^2(a) + SDV^2(a_i) \tag{2}$$

$$ESU^2(a) = a^2 \cdot \left[\frac{4 \cdot u^2(h)}{h^2} + \left(\frac{a \cdot \pi^2 \cdot}{\ln(1/4) \cdot h^2} \right)^2 \cdot u^2(t_{1/2}) \right]$$

FE Model and Simulation

To quantify the distortion of the specimen properties by the ceramic paste a simple finite element model was developed. A cubic, mono-sized porosity is assumed. Models are designed from a unit cell (8 nodes 3-D thermal solid elements). They differ from their ratio of characteristic size of a pore to the thickness of the remaining metallic walls of the sample. In dependence of the porosity of a sample or density respectively suitable sample geometry can be identified. As heat transfer mechanisms conductivity and radiation but no convection are considered.

DISCUSSION

FE Simulation of Foamy Samples

The images in Figure 4 show the transient temperature response of the Magnesium foam structure (lines with maximum values) and the SiC filler (lines with minimum values) for three characteristic types of samples with approximately the same height (~10 mm). Additionally the radiation dependent detector signal (lines between) is shown. Image *I* shows a sample with a pore size of 1 mm. Compared to the sample size this represents a quasi-homogeneous metallic body. No significant differences in the temperature responses of the metal, the SiC filler and the detector occur. Image *II* shows a typical sample with a pore size of 2-3 mm. The *IR*-detected temperature could be verified by measurement. It is significantly different from the real temperature response of the metallic structure. Thus the half time $t_{1/2}$ has to be corrected by a factor ~5. Image *III* shows a sample with only one open pore completely filled with SiC. The detector identifies the temperature response of the SiC!

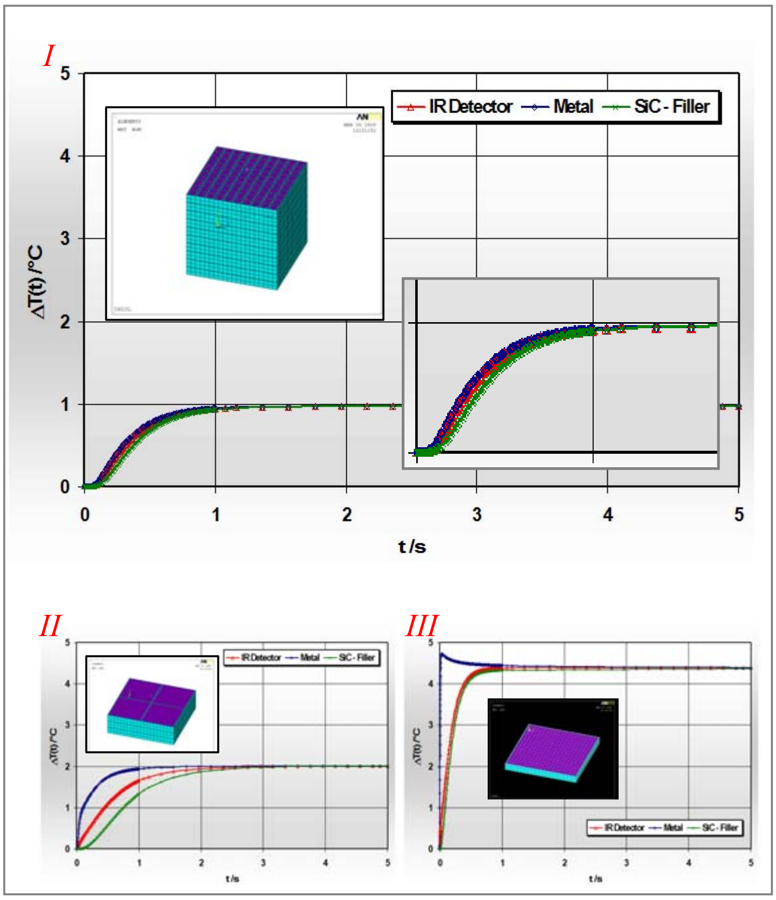

Figure 4. FE simulation of the transient temperature response of Mg-foam materials. Samples differ in their porosity. Porosity is filled with SiC.

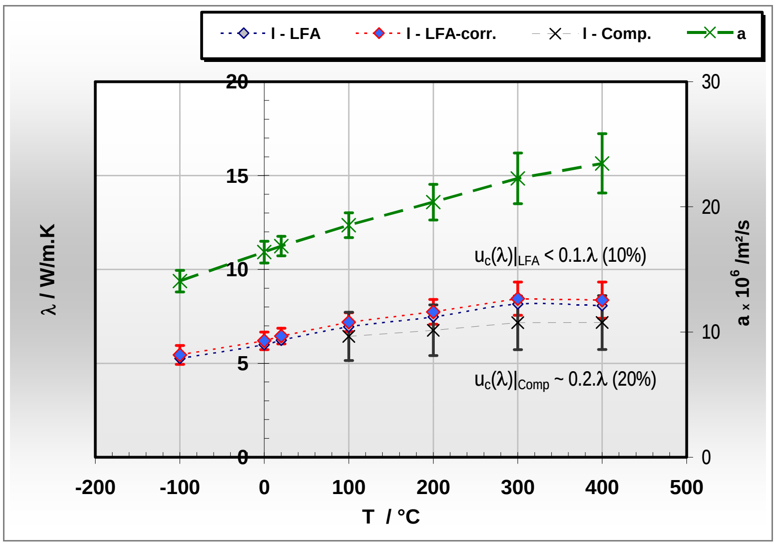

Figure 5. Thermal diffusivity and conductivity of a Mg-foam material. comparison between conductivity data from a comparative set-up and data calculated from flash based diffusivity data.

FE Based Correction of Flash Results in Comparison with Comparative Data:

A foam from a Magnesium-alloy as shown in the figure above was measured. The thermal conductivity of the Mg bulk is 156 W/mK, the attributed uncertainty is estimated with ± 5%. The bulk density is 1.738 g/cm³. The density of the foam at 20°C is $\rho_f = 0.411 \pm 0.015$ g/cm³ ~ 25% ρ_{MgA}. The radius R of the sample measures 20 mm and is approximately twice it´s height h. Thus the number of pores (mono-sized pores are assumed) in the volume can be calculated with $n_V = ½.n_S^{3/2}$ identifying n_S as the number of pores visible at the top surface of the sample. Equation $[R^2.\pi.h - n_V.\ ^4/_3.\pi.r^3].\ \rho_f = R^2.p.h.\rho_{MgA}$ gives the characteristic radius of the pores r – following to: $r = [R/n_S^{1/2}].[^3/_2.(\rho_{MgA} - \rho_f)]$. With n_S ~ 100 one estimates r ~ 2mm. This consequences a FE-model with 5 layers as illustrated in image *I*. From a first view no correction seems to be necessary. But the FE-analysis shows that a half time correction of about 10% should be done. The corrected diffusivity data and both the corrected and the un-corrected conductivity data as well are shown in the figure above. Corrected conductivity data are slightly higher. In comparison to conductivity results from a comparative set-up LFA based data are about 10% to 15% higher. LFA based data and Comparative data give results within the uncertainty levels of both methods. Comparative data were attributed with a constant uncertainty of 15% from their supplier. Therefore here is assumed that either a rough description of the *ESU* with an uncertainty of the temperature of ~2°C was used for the set-up or no statistics from a set of measured samples was done. To correct this $u_c(\lambda) = (2 \times ESU^2)^{1/2} \approx 20\%$ of λ is assumed. LFA based conductivity data show an uncertainty level (*95% Conf. Int.; k = 2*) from ~10%.

RESULTS

The self consistency of the numerical results and the agreement of the flash based conductivity results with those obtained from a comparative device have been proved. As a result for future characterisation of metal foam materials flash devices can be used. For the measured magnesium alloy an average uncertainty of diffusivity results is estimated to ~7%. In addition DSC and dilatometry are used to determine heat capacity and thermal density. Data from these methods are used to calculate thermal conductivity. For uncertainty of conductivity data less than 10% based on a confidential interval of *95%* (coverage factor $k = 2$) could be achieved.

The density of the measured foam material is about 24% of the bulk density. Thus the examined thermal conductivity data of the foam material of about 4% compared to those of the bulk material can be understood (comparison done at room temperature). To formulate the relation between the foam density and the thermal conductivity of the foamy structure was not scope of this work. But it could be shown that the number of visible pores at the top surface of a sample provides sufficient information to perform excellent measurement results.

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