Flash Techniques to Measure Thermal Diffusivity and Thermal Conductivity of Metal-Foam-Materials

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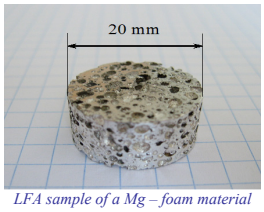
INTRODUCTION:

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 (LF) 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 $\lambda = \rho_x \, c_{p\,x} \, a$.

EXPERIMENTAL:

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. To flash a coplanar the metallic body the sample is grinded after the drying of the paste.



LFA sample of a Mg – foam material

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. 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). To regard to the influences of the filler material in the open surface porosity a 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.

FEM 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.

A superposition of the transient temperature responses of both the metallic structure and the ceramic filler occurs. 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.

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 proofed. 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 lass than 10% based on a confidential interval of *95%* (coverage factor $k = 2$) could be achieved.

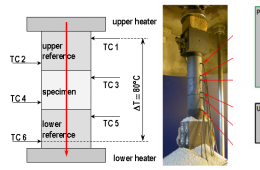
COMPARATIVE SET-UP VERSUS LASER FLASH :

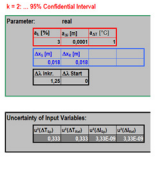
Methods and the basic equations to determine thermal conductivity (Comparative Set-Up) and thermal diffusivity (Laser Flash) are illustrated. In accordance to the GUM (Guide to Express Uncertainty in Measurement Results: ENV 13005) the mathematical models to estimate the Equipment Specific Uncertainties *ESU* are given. The dependency of the *ESU* in case of a comparative conductivity measurement $ESU(\lambda)$ strongly correlates to the uncertainty of the input estimate of the temperatures $a_{\Delta T}$. It is about 10% of the measured conductivity under optimum conditions. By contrast flash results show a rather low *ESU* – mostly less than 1% of the measured diffusivity data. 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. 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.

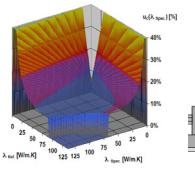
$$\lambda_S(T) = \frac{1}{2} \cdot \lambda_R(T) \cdot \frac{\Delta l_S}{\Delta T_S} \cdot \left[\frac{\Delta T_{R,h}}{\Delta l_{R,h}} + \frac{\Delta T_{R,l}}{\Delta l_{R,l}} \right]$$

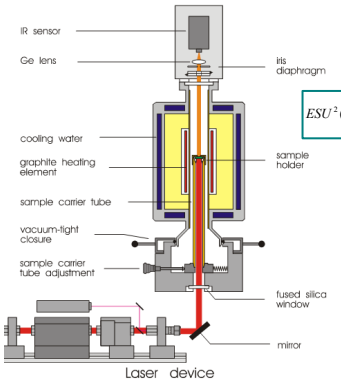
$$ESU^2(\lambda) = \left(\frac{\Delta l_S}{2 \cdot \Delta T_S} \cdot \left[\frac{\Delta T_R^u}{\Delta l_R^u} + \frac{\Delta T_R^l}{\Delta l_R^l} \right] \right)^2 \cdot u^2(\lambda_R) + \left(\frac{\lambda_R}{2 \cdot \Delta T_S} \cdot \left[\frac{\Delta T_R^u}{\Delta l_R^u} + \frac{\Delta T_R^l}{\Delta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

$$u_c^2(\lambda) = ESU^2(\lambda) + SDV^2(\lambda)$$







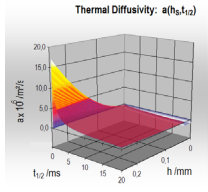


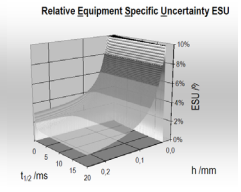
$$a(T) \equiv -\frac{\ln(1/4)}{\pi^2} \cdot \frac{h^2(T)}{t_{1/2}(T)}$$

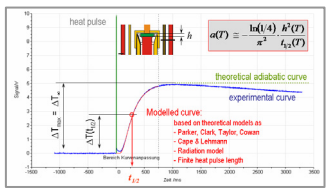
$$ESU^2(a) = a^2 \cdot \left[\frac{4 \cdot u^2(h)}{h^3} - \frac{a \cdot \pi^2 \cdot u^2(t_{1/2})}{\ln(\frac{1}{4}) \cdot h^2} \right]$$

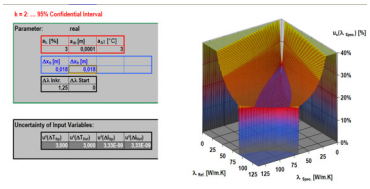
$$u_c^2(a) = ESU^2(a) + SDV^2(a)$$

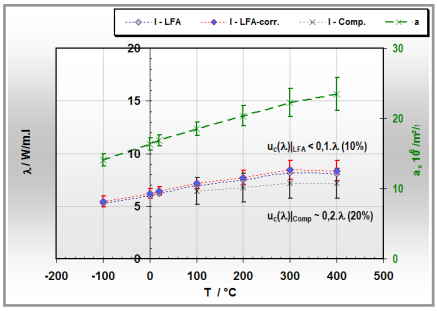
Mg foam materials show a diffusivity $a \sim 20 \times 10^{-6}$ m²/s. To model the transient $T(t)$-response 5000 temperature values are detected within the required observation time: (~10000 ms). With respect to versatile sample dimensions of $h \sim 10$ mm and $\phi \sim 20$ mm this results to an $ESU(a)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

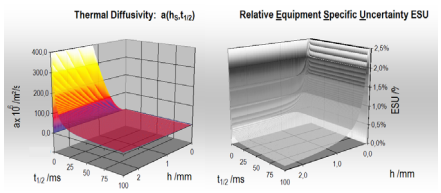


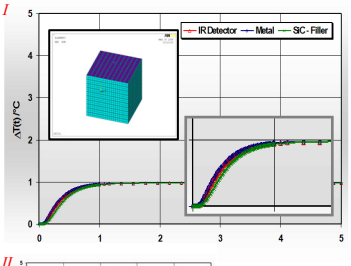


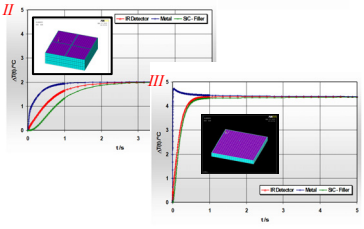


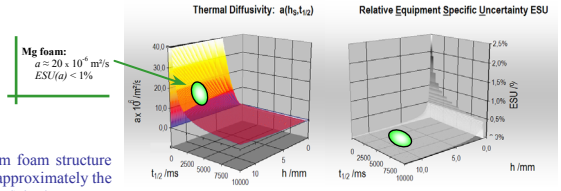












FEM SIMULATION OF FOAMY SAMPLES:

The images (left) show the transient temperature response of the Magnesium foam structure (blue) and the SiC filler (green) for three characteristic types of samples with approximately the same height (~10 mm). Additionally the radiation dependent detector signal (red) is shown.
Image *I* shows a sample with a pore size of 1 mm. Compared to the sample size this represents a type 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!

FEM BASED CORRECTION OF FLASH RESULTS IN COMPARISON WITH COMPARATIVE DATA:

A foam from a Magnesium-alloy as shown in the figure above was measured. Its density 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 $[R^2.\pi.h - n_V \cdot {}^4/_3.\pi.r^3]$. $\rho_f = R^2.p.h.\rho_{MgA}$ gives the characteristic radius of the pores r – following to: $r = [R/n_S^{1/2}].[^3/_2.(\rho_{MgA}-\rho_f)]$. With $n_S \sim 100$ one estimates $r \sim 2$mm. 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 $u_c(\lambda) = (2 \times ESU^2)^{1/2} \approx 20\% \times \lambda$ is assumed. LFA based conductivity data show an uncertainty level (*95% Conf. Int.*; $k = 2$) from ~10%.

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