Adapted Laser-Flash Method for Estimating Thermal Diffusion Properties of a Porous Material Surrounded by Compact Layers

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ASSOCIAZIONE ITALIANA PROPRIETÀ TERMOFISICHE

Bari, 20th September 2024

editors

Laura Fedele
Umberto Berardi
Sergio Bobbo
Alessandro Bortolin
Gianluca Cadelano
Giovanni Ferrarini

PROCEEDINGS

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2024

Bari

XXVII AIPT

Associazione Italiana Proprieta' Termofisiche

09.00 – 09.30 **Apertura dei lavori**

09.30 – 10.30 ***Assemblea Soci AIPT***

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Approvazione bilanci
Rinnovo cariche sociali
Analisi di consuntivo ECTP2023
Varie ed eventuali

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Adapted Laser-Flash Method for Estimating Thermal Diffusion Properties of a Porous Material Surrounded by Compact Layers

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Abstract

Composite structures obtained by superimposing layers of different materials find application in various technological fields. The number of layers and the characteristics of the constituent materials are chosen based on different needs, e.g. for thermal protection of metal components or to preserve high mechanical strength by reducing the overall weight of the structure. For reasons related to production methods, some layers are not available separately from others and the study of their thermal properties cannot be achieved using methods conceived for homogeneous samples. For the evaluation of the properties of these non-free-standing layers, some modifications of the traditional laser-flash method are necessary, especially regarding measurements post-processing.

In the present work, the measurements on three-layer samples were carried out using the classic laser-flash technique, but the analysis of the thermal response of the sample was carried out with a significantly different approach compared to that proposed by Parker and for which the method introduced by Cowan allows heat losses to be considered. The non-normalized thermal response that is measured on the rear surface of the sample was analyzed with a 1D heat diffusion analytical solution, considering both the heat losses on the front and rear surfaces of the sample and the finite pulse effect.

The tested samples are three layers of the same material (Ti6Al4V) but with a different structure. In fact, the two external layers are compact, while the intermediate one is made up of sintered powder of various porosities, which is deposited through additive manufacturing. Since the thermal properties of the internal porous layer are of great interest (in particular its apparent thermal conductivity), the article describes the procedure adopted to estimate them and discusses their identifiability. Furthermore, using the thermal conductivity data available in the literature, a first validation of the adapted laser-flash method is also provided.

1. Introduction

The laser flash method, since its invention about 60 years ago [1-3], has been widely used for the determination of thermal diffusivity.

There are several advantages that have made this technique very popular, to name just a few the speed with which measurements can be carried out, the very small size of the samples and the multiplicity of materials that can be measured.

The original method, as is well known, consists in sending a flash of energy, produced for example by a laser, onto the base surface of a sample of cylindrical shape, recording on the opposite face the increase in temperature over time via a sensor (thermocouple or detector IR). In its original version, proposed by Parker [1], the thermal diffusivity can be calculated starting from the thermal transient recorded as:

$$\alpha = 1.38 \cdot \frac{L^2}{\pi^2 \cdot t_{1/2}} = 0.139 \cdot \frac{L^2}{t_{50\%}} \qquad (1)$$

where:
α is the thermal diffusivity [m^2/s]
L is the thickness of the sample [m]
$t_{1/2}=t_{50\%}$ is the time required to reach half the maximum temperature rise [s].

The method proposed by Parker allows the thermal diffusivity to be accurately determined only if the hypotheses underlying the model are respected when carrying out the measurement. Among these of special relevance are both the hypothesis that the thermal transient that follows the absorption of the energy released by the flash in a thin layer of the sample is adiabatic and the hypothesis that the pulsed energy is released instantaneously.

An adiabatic measurement, as assumed by Parker, cannot be obtained experimentally, even when operating in high vacuum conditions. The presence of radiative losses causes the experiment to deviate from this hypothesis more evidently the higher the measurement temperature and the thicker the sample.

For this reason, at the end of the transient, the maximum temperature reached is lower than that obtainable for the same energy pulse in the adiabatic case. Consequently, the $t_{50\%}$ term in Eq. (1) is underestimated and the thermal diffusivity overestimated. In addition, the thermogram does not present a stable plateau over time once the maximum increase in temperature has been reached, but the temperature decreases over time in a more marked manner the higher the temperature of the sample.

Under these conditions the previous Eq. (1) is no longer accurate and therefore to use the laser flash method in the years following its invention, various models have been developed to take radiation losses into account [4–10].

Regarding the effect of the finite time of the pulse, the “non-instantaneous" energy release results in a slower temperature increase with a consequent increase in the $t_{50\%}$ term in Eq. (1) which leads to an underestimation of the thermal diffusivity value. This effect, which is particularly relevant with highly diffusive materials and very thin samples, has also been widely studied and corrective methods have been proposed in the literature [7, 11–13].

Aware of these problems, in the present work the measurements were performed using the laser-flash technique in its classic version, but the analysis of the thermal transient was carried out in significantly different way.

First of all, the thermal transient recorded on the rear surface of the sample was not normalized using the maximum recorded temperature whose value, as previously mentioned, is influenced by heat losses. The thermal response to the energy pulse, as described below, was then analyzed using a 1D heat diffusion analytical solution considering both heat losses from the front and rear surface of the sample and the effect of the finite time of the pulse.

The samples used to test the model are three-layer samples of Ti6Al4V in which the two external layers are massive while the intermediate one is made up of sintered powder deposited through additive manufacturing.

The thermal properties of the intermediate layer, which cannot be obtained as a free-standing sample, are of particular interest. The article describes the procedure adopted to estimate these properties and discusses their identifiability.

2. *Methodology*

Over the course of several decades, the flash technique has consolidated itself as an important method for the estimation of thermal diffusion properties of metals and alloys. In this period, in addition to the improvements of the experimental apparatus consistent with the technical evolution of the hardware components, significant modifications of the methods for determining the thermal properties of interest have been proposed. In fact, compared to the Parker method mentioned in the previous section, in which only a few experimental data were used to determine the thermal diffusivity, nowadays, thanks to the greater capacity for in silico data reduction, more complex estimation algorithms, which typically involve the entire thermogram recorded, are gaining ground.

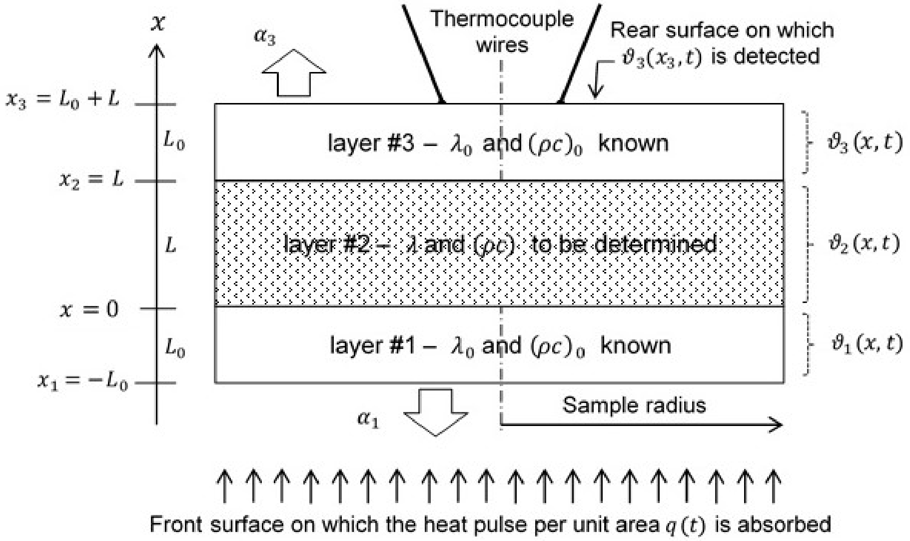

Figure 1. Geometric scheme of the sample and arrangement of the layers.

In the case studied, due to the configuration of the samples, the original Parker method cannot be used, and the estimation of the thermal diffusion properties necessarily requires a non-linear procedure. In fact, the material under test is made up of a porous intermediate layer characterized by a dispersed solid matrix (sintered powder), surrounded by two layers with the same chemical composition but with a compact structure.

The geometric scheme in Fig. 1 shows how the external layers have the same nominal thickness L_0, the extent of which can be compared with the thickness L of the porous layer.

The property estimation method that was used is based on two different models, which will be called direct and inverse in the following. Based on the assigned boundary conditions, the direct model provides, starting from the provisional values assigned to the properties to be estimated, the temperature distribution as a function of time within the sample volume. The inverse model is instead arranged on a recursive algorithm, based in our case on least squares, which proposes at each iteration a new set of values of the thermal properties of the material under test. Once convergence is achieved, the property values obtained are those that minimize the difference between the measured temperature values and those calculated by the direct model for the entire observed transient.

From a general point of view, theoretical treatments and applications of the direct model are reported in the literature cited in the introduction, while the complete theory for the inverse model can be found, for example, in [14]. As regards the direct model, the 1D analytical solution for a three-layer sample is reported in [15, 16], and is obtained using the Laplace transform and its inverse, considering the heat losses by radiation, the contact thermal resistance between the layers and the finite duration of the thermal pulse.

Although this solution can be applied directly to the case in question, it is however possible to formulate some simplifying assumptions and hypotheses as follows:

- according to the 1D model, the heat losses are imposed only at the front and rear surfaces ($x=x_1$ and $x=x_3$ in Fig. 1). The radiative exchanges involving these surfaces were linearized [9] and calculated at the initial temperature T_0 of the sample (equivalent film coefficient), assuming the same emissivity ε_0 (T_0) for both surfaces:

$$\alpha_1 = \alpha_3 = \alpha(T_0) = 4\sigma\varepsilon_0(T_0)\, T_0^3 \tag{2}$$

  where σ is the Stefan – Boltzmann constant,
- the contact resistances between the intermediate layer and the surrounding ones were not considered because the powder sintering process aims to create a porous structure mechanically connected to the interfaces with the compact layers ($x=0$ and $x=x_2$ in Fig. 1). The presence of defects on these interfaces (typically bubbles) could be considered in the model as thermal contact resistances, but the estimated values of the properties of the porous layer could not however be purified from this effect. Therefore, perfect thermal contact between the layers was assumed and therefore the temperature at the interface between two layers (Fig. 1) is the same:

$$\vartheta_1(0,t) = \vartheta_2(0,t), \;\; \vartheta_2(x_2,t) = \vartheta_3(x_2,t) \tag{3}$$

- Regarding the finite duration of the heat pulse, its effect on the thermal response of the non-pulsed surface was determined a priori. The response versus time q(t) of the laser system used was approximated by the normalized distribution H(t) suggested by Larson and Koyama [11], such that:

  $$q(t) = Q_0 H(t) = Q_0\,(t/t_p^2)\exp(-t/t_p) \qquad (4)$$

  where Q_0 represents all the energy per unit surface area uniformly absorbed in a very thin sublayer of the front surface of the sample and t_p is the instant of time at which the peak energy release occurs which for the laser device used (Nd:YAG laser) is close to 0.2 ms. By adopting the theoretical values available in the literature for the thermal properties of the sintered powder [17-19], some comparisons were carried out considering or neglecting the finite duration of the thermal pulse. Both due to the relatively high thickness of the specimen and the total energy release time, greater than 99.9% in 2 ms, the effect of the finite-pulse time was found to be negligible compared to the case in which the pulse is approximated by the Dirac function δ(0). Consequently, the finite duration of energy pulse was not considered (t_p -->0), so

  $$q(t) = Q_0\delta(0) \qquad (5)$$

Based on these assumptions and considering both the set temperature T_0 of the vacuum chamber and the initial condition $\vartheta_j(x,t)=T_j(x,t)-T_0=0$ for each layer and assuming as boundary conditions

$$-\lambda_0(T_0)\frac{\partial\vartheta_1(x_1,t)}{\partial x} = Q_0\delta(0) - \alpha_0(T_0)\vartheta_1(x_1,t) \qquad (6)$$

$$-\lambda_0(T_0)\frac{\partial\vartheta_3(x_3,t)}{\partial x} = \alpha_0(T_0)\vartheta_3(x_3,t) \qquad (7)$$

the increase in temperature on the non-pulsed rear surface reported in [16] can be written as

$$\vartheta_3(x_3,t) = 8\frac{Q_0}{C_0L_0}\sum_{k=1}^{\infty} D_k^{-1}\exp\left(-\mu_k^2\frac{\lambda_0}{C_0L_0^2}t\right) \qquad (8)$$

where $C_0=(\rho c)_0$, λ_0, and L_0 are respectively the volumetric heat capacity, the thermal conductivity and the thickness of the surrounding layers, while the quantity Q_0 represents the energy absorbed per unit area of the pulsed surface. The dimensionless term D_k in the sum of Eq. (8) can be expressed as

$$\begin{aligned} D_k = {} & \psi_1\chi_1 cos(\psi_1\mu_k) + 2\psi_2\chi_2 cos(\psi_2\mu_k) + \psi_3\chi_3 cos(\psi_3\mu_k) + \\ & +(2\beta_0^2/3)\{\ 2[\chi_1 sin(\psi_1\mu_k) - 2\chi_2 sin(\psi_2\mu_k) + \chi_3 sin(\psi_3\mu_k)] \\ & \quad - \mu_k[\psi_1\chi_1 cos(\psi_1\mu_k) + \psi_3\chi_3 cos(\psi_3\mu_k)]\ \} + \\ & \quad + 2(\beta_0/\mu_k^2)\{\chi_1[cos(\psi_1\mu_k) + \psi_1\mu_k sin(\psi_1\mu_k)] + \chi_3[cos(\psi_3\mu_k) + \\ & \quad + \psi_3\mu_k sin(\psi_3\mu_k)]\} \end{aligned} \tag{9}$$

in which

$$\beta_0 = \alpha(T_0)L_0/\lambda_0, \tag{10}$$

$$\chi_1 = 2 + 1/\sqrt{\eta\,\kappa} + \sqrt{\eta\,\kappa},\ \ \chi_2 = \sqrt{\eta\,\kappa} - 1/\sqrt{\eta\,\kappa},\ \ \chi_3 = 2 - 1/\sqrt{\eta\,\kappa} - \sqrt{\eta\,\kappa} \tag{11}$$

$$\psi_1 = 2 + \frac{L}{L_0}\sqrt{\kappa/\eta}, \qquad \psi_2 = \frac{L}{L_0}\sqrt{\kappa/\eta}, \qquad \psi_3 = 2 - \frac{L}{L_0}\sqrt{\kappa/\eta} \tag{12}$$

where the quantities to be estimated $\kappa=\lambda/\lambda_0$ and $\eta=C/C_0=((\rho c))/(\rho c)_0$ represent the thermal conductivity and volumetric heat capacity of the sintered powder, both dimensionless using the similar properties of the adjacent layers.

The terms μ_k in the Eq. (9) are the k-th eigenvalue obtained from the solution of the following equation:

$$\begin{aligned} & \chi_1 sin(\psi_1\mu_k) + 2\chi_2 sin(\psi_2\mu_k) + \chi_3 sin(\psi_3\mu_k) - \{2(\beta_0/\mu_k)[\chi_1 cos(\psi_1\mu_k) + \\ & \chi_3 cos(\psi_3\mu_k)] + (\beta_0^2/\mu_k^2)[\chi_1 sin(\psi_1\mu_k) - \ sin(\psi_2\mu_k) + \chi_3 sin(\psi_3\mu_k)]\} = 0 \end{aligned} \tag{13}$$

The maximum temperature increase detectable on the rear surface corresponds to the asymptotic value ϑ_∞ achieved in the case of an adiabatic sample, defined as

$$\vartheta_\infty = \frac{Q_0}{2C_0L_0+CL} = \frac{Q_0}{C_0L_0}\left(2 + \frac{L}{L_0}\eta\right)^{-1} \tag{14}$$

Through this asymptotic value, the thermal response given by Eq. (8) can be normalized, becoming independent of the amount of the absorbed heat Q_0

$$\Theta_3(x_3,t)=\frac{\vartheta_3(x_3,t)}{\vartheta_\infty}=8\left(2+\frac{L}{L_0}\eta\right)\sum_{k=1}^{\infty}D_k^{-1}\exp\left(-\mu_k^2\frac{\lambda_0}{C_0L_0^2}t\right) \qquad (15)$$

Since the expected duration of the transient is well above one second, the sample cannot be assumed adiabatic and heat losses involving the surrounding layers make it arduous to experimentally determine ϑ_∞. In fact, compared to the asymptotic value of Eq. (14), the maximum temperature increase detectable on the non-pulsed surface is smaller the greater the heat losses. Moreover, the peculiar arrangement of the material under test inside the specimen makes it impossible to perform an a posteriori correction for the effect of thermal losses on the estimated properties [9-10].

For this reason, it was decided not to normalize the response recorded on the rear surface of the sample and among the quantities to be estimated, in addition to the dimensionless parameters κ and η previously introduced, the quantity of heat Q_0 absorbed on the pulsed surface of the sample was added. The estimate of this last quantity is not of particular interest but is necessary due to the configuration of the sample being studied.

As regards the inverse model, the theory for an equivalent case is detailed in [20], and therefore only some aspects concerning the identifiability of the parameters to be estimated are recalled here. Fundamental help is given by the so-called sensitivity coefficients, defined through first partial derivatives of the dependent variable with respect to the parameters to be estimated. In this case the dependent variable is the temperature increase given by Eq. (8) and its derivatives with respect to the parameters κ, η and Q_0 were calculated numerically at each time step and for each iteration.

The sensitivity coefficients provide important information in the estimation problem. The sensitivity coefficient matrix is composed of as many rows as the time intervals observed and in this case of three columns (as many as the parameters to be estimated) and allows us to highlight the possible linear dependence between the parameters to be estimated. In fact, for the simultaneous identification of the parameters the product between the sensitivity matrix and its transpose gives a square matrix whose determinant must always be different from zero, as happens in the proposed case. Furthermore, for the parameters to be estimated, the magnitude of these coefficients allows their estimation uncertainty to be established, the higher the magnitude of the coefficient, the lower the estimation uncertainty.

3. First results and discussion

The described method was applied to several samples with layers arranged according to the geometry shown in Fig. 1, but with different thicknesses of the sintered powder layer [20]. Since the method requires the estimation of the energy input that induces the thermal transient, some preliminary results are reported below to investigate this aspect. The samples tested are two, characterized by the same overall thickness and by some differences in the thickness of the layers, as reported in Tab. 1.

Table 1. Layer thicknesess

Sample	Thickness L_0 of the surrounding layers (mm)	Thickness L of the sintered powder (mm)
#1	0.61	1.81
#2	0.50	2.03

For the second sample, which has a greater thickness of sintered powder than the first, the tests were conducted by modifying the input energy assigned to the laser and increasing it by approximately 25%. The aim was both to verify that the estimated values of the properties of the sintered powder were independent of the flash energy and to verify that the estimated values for the absorbed energy Q_0 were consistent with the energy used for the pulse.

Both samples were studied from room temperature to 800 °C by taking measurements approximately every 200 °C within this range. In this temperature range the values of the thermal diffusion properties of the compact Ti6Al4V were taken from the literature [19] as well as those relating to the sintered powder with the same composition. In the literature the latter are only available starting from a temperature of 200 °C and are obtained based on the properties of the compact material assuming three-dimensional aggregation schemes for the sintered powder [17-18].

Regarding the radiative heat losses approximated by Eq. (2), the emissivity $\varepsilon_0 (T_0)$ of the compact Ti6Al4V surfaces (both the front and rear ones) was here assumed independent of the temperature and considered equal to 0.3.

The estimated values of thermal conductivity and volumetric heat capacity of the sintered powder are shown in Figs. 3-4 using the rhomboid and circular markers respectively for samples #1 and #2. The error bars in the figure refer to the estimated standard deviation for the bilateral t-distribution for a confidence level of 95%. In both figures the solid lines represent the trend obtained considering the estimated values for both samples while the dashed lines refer to what was found in the literature for the sintered powder and the dotted lines to the properties of the compact material.

The comparison between the values obtained from the measurements and those available in the literature shows good agreement. It is also interesting to note that the properties of the compact material are as expected higher, by approximately seven times for thermal conductivity and two times for volumetric heat capacity in a temperature-independent manner.

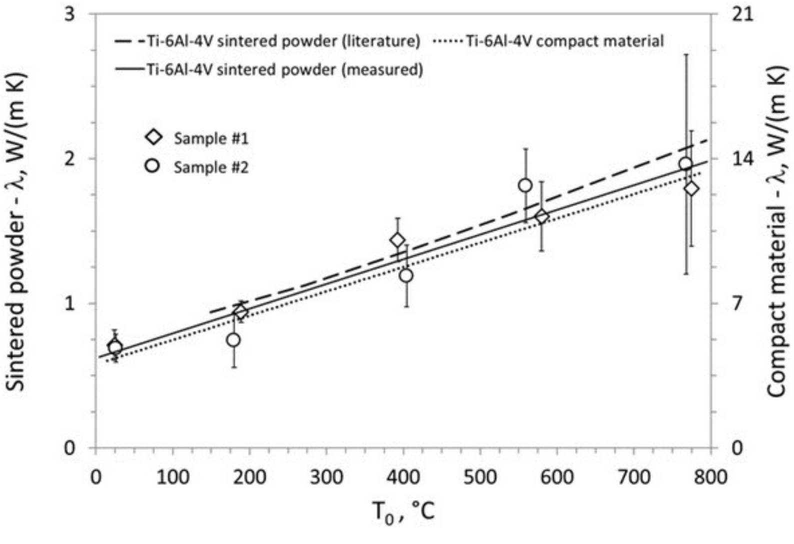

Figure 2. Trend of the estimated thermal conductivity as a function of temperature and comparison with literature data for the compact material and for the sintered powder.

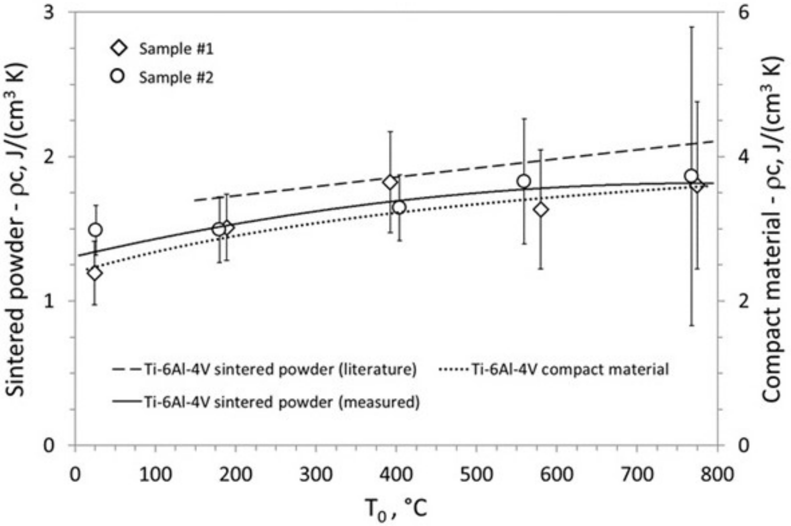

Figure 3. Estimated volumetric heat capacity versus test temperature for the sintered powder and comparison with literature data and compact material.

Figure 4 shows the estimated values of the energy Q_0 that is absorbed per unit surface area of the samples. Since for sample #2 the energy pulse used is higher, the trend of the estimated values of the absorbed energy in Fig. 4 seems to confirm the validity of the calculations. In fact, as can be seen in Fig. 4, the ratio between the estimated values of the energy Q_0 for the two samples is almost constant at all test temperatures T_0. Examining the results obtained it is possible to verify that the energy absorbed by sample #2 is approximately 15% higher compared to an increase in flash energy of approximately 25%. This difference can reasonably be explained by considering both the efficiency of the laser system and the fact that the flash energy value is not a measured quantity. What is considered most important here is that these ratios are approximately independent of the test temperature.

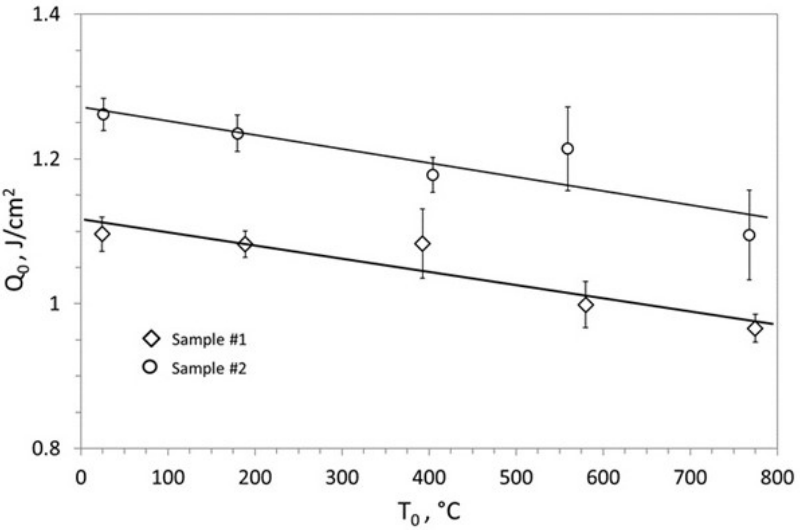

Figure 4. Estimation of the energy absorbed on the front surface of the samples as a function of the test temperature.

As regards the identifiability of this parameter, it is less simple to explain the trend as a function of temperature with a negative slope found in the two cases examined. Among the extreme temperature values investigated, the relative variation of Q_0 is about 15% for both samples. We can list three possible reasons: the inverse model, the assumptions about quantities influencing the heat losses, in particular the temperature-independent emissivity, and the behavior of the measuring equipment when the temperature varies. The good agreement between the expected and estimated values for the thermal properties of the sintered powder allows us to exclude at the moment the first two reasons listed as both would affect these results. As regards the apparatus, the optical path of the laser beam is mainly external to the measurement chamber. Lenses and mirrors are kept at room temperature, except for the optical window on the measuring chamber which allows the passage of the laser beam. The temperature dependence of the optical properties of this window could explain the reduction in laser beam intensity, but this has yet to be demonstrated.

References

[1] W.J. Parker, R.J. Jenkins, C.P. Butler, G.L. Abbott, “Flash method of determining thermal diffusivity, heat capacity, and thermal conductivity”, J Appl Phys, 32 (9) pp.1679–1684, 1961.

[2] F. Righini, A. Cezairliyan, “Pulse method of thermal diffusivity measurements (a review)”, High Temperature-High Pressure, 5 pp. 481-501, 1973.

[3] K.D. Maglic, A. Cezairliyan, V.E. Peletsky, Compendium of Thermophysical Property Measurement Methods, 2, Plenum Press, 1992.

[4] R.D. Cowan, “Pulse method of measuring thermal diffusivity at high temperatures”, J Appl Phys, 34 (4) pp.926–927, 1963.

[5] D.A. Watt, “Theory of thermal diffusivity by pulse technique”, J Appl Phys, 46 (2) pp.714–719, 1975.

[6] L.M. Clark, R.E. Taylor, “Radiation loss in the flash method for thermal diffusivity”, J Appl Phys, 17 (2) pp.231–240, 1966.

[7] R.C. Heckman, “Finite pulse-time and heat-loss effects in pulse thermal diffusivity measurements”, J Appl Phys, 44 (4) pp.1455–1460, 1973.

[8] J.A. Cape, G.W. Lehman “Temperature and finite pulse time effects in the flash method for measuring thermal diffusivity”, J Appl Phys, 34 (7) pp.1909–1913, 1963.

[9] L. Dusza, “Combined solution of the simultaneous heat loss and finite pulse corrections with the laser flash method”, High Temperature-High Pressure, 27/28 (5) pp. 467-473, 1995.

[10] A.R. Mendelsohn, “The effect of heat loss on the flash method of determining thermal diffusivity”, Appl Phys Lett, 2 (1) pp.19–21, 1963.

[11] K.B. Larson, K. Koyama, “Correction for finite-pulse-time effects in very thin samples using the flash method of measuring thermal diffusivity”, J Appl Phys, 38 (2) pp.465–474, 1967.

[12] J. Xue, X. Liu, R. Taylor, “The effects of a finite pulse time in the flash thermal diffusivity method”, Int J Thermophys, 14 (1) pp.123–133, 1993.

[13] T. Azumi, Y. Takahashi, “Novel finite pulse-width correction in flash thermal diffusivity measurement”, Rev Sci Instrum, 52 (9) pp.1411–1413, 1981.

[14] J. V. Beck, K. J. Arnold, Parameter estimation in engineering and science, Wiley, New York, pp. 340-341, 1977.

[15] H.J. Lee, Thermal Diffusivity in Layered and Dispersed Composites, PhD thesis, Purdue University, Lafayette, IN, USA, 1975.

[16] J.N. Sweet, Data Analysis Methods for Flash Thermal Diffusivity Experiments, Sandia National Laboratories, Albuquerque, NM, USA, 1989. https://doi.org/10.2172/6312039

[17] N.K. Tolochko, M.K. Arshinov, A.V. Gusarov, V.I. Titov, T. Laoui, L. Froyen, "Mechanisms of selective laser sintering and heat transfer in Ti powder" Rapid Prototyp J, 9 (5) pp. 314–326, 2003.

[18] M. Shapiro, V. Dudko, V. Royzen, Y. Krichevets, S. Lekhtmakher, V. Grozubinsky, et al., "Characterization of Powder Beds by Thermal Conductivity: Effect of Gas Pressure on the Thermal Resistance of Particle Contact Points", Part. Part. Syst. Charact., 21 pp. 268–275, 2004.

[19] M. Boivineau, C. Cagran, D. Doytier, V. Eyraud, M.H. Nadal, B. Wilthan, et al., "Thermophysical properties of solid and liquid Ti6Al4V (TA6V) alloy", Int J Thermophys, 27 (2) pp. 507–529, 2006.

[20] M. Galati, E. Campagnoli, V. Giaretto, L. Iuliano, "Modelling the thermal behavior of Ti6Al4V sintered powder bed in electron beam powder bed fusion (EB-PBF)", Procedia CIRP, 118 pp. 664–669, 2023.