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Evaluation of the Thermal Diffusivity of Thin Specimens from Laser Flash Data

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Abstract Thermal-diffusivity data obtained by a pulse technique are substantially in error if the sample thickness becomes too small. In this case the inevitable spike caused by the trigger pulse for the laser and the thermogram of the specimen are interacting, and a superposition of both signals is recorded, whose evaluation with standard algorithms systematically underestimates the thermal diffusivity of materials. Therefore, this incorrect calculation corresponding to the spike-thermogram overlay was investigated for general tendencies, and a calculation method transferring the experimental data obtained for small thicknesses into representative thermal diffusivity values for the investigated material will be introduced.

Keywords Laser flash · Pulse technique · Sample thickness · Spike-thermogram overlay · Thermal diffusivity

Nomenclature

$t_{0.5}$	Characteristic time of increase, ms
α_s	Saturation value of thermal diffusivity in $\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$ = the maximum of measured/calculated diffusivity for each class of compounds
α_m	Measured/calculated diffusivity at given L, $\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$
Δ	Deviation of α_m from α_s, %
L	Sample thickness, mm
T	Initial sample temperature,°C
T_max	Maximum of temperature rise on the backside of the sample, °C

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1 Introduction

The flash method [1] has been established as the standard test method for the determination of the thermal diffusivity and is used to measure values of the thermal diffusivity of a variety of solid materials [2] over a large temperature range [3,4]. The method can be considered as an absolute method of measurement, since no reference standards are required.

In the laser-flash method, a laser pulse is used as a heating source to introduce a high-intensity short duration energy pulse on the front side of a small disc-shaped specimen. The introduced heat spreads over the whole sample, and an infrared detector or any other device, which provides a linear electrical signal proportional to the temperature rise, detects the increase of temperature on the backside. The signal is recorded as a function of time starting at the initial temperature T. Competing with thermal diffusion through the material heat loss by radiation [5], convection and surface effects occur, causing the measured temperature to pass typically through a maximum ($T_{\max}$). This maximum is used for the determination of the characteristic time of increase $t_{0.5}$ [1], at which the intensity of the signal has reached half of its maximum value.

$$T/T_{\max} = 0.5 = 1.37\alpha\pi^2 t_{0.5}/L^2 \tag{1}$$

where $t_{0.5}$ is the characteristic time of increase. The thermal diffusivity α for a sample thickness L is calculated as follows:

$$\alpha_{0.5} = 0.138785\, L^2/t_{0.5} \tag{2}$$

Exact algorithms are used to fit the derived signal and to correct for a finite pulse width and loss of heat. Various procedures of correction are given in [6–8]. These algorithms are used to solve very complex numerical equations and result in the final formula for the time of increase $t_{0.5}$, which is valid when the detector signal has reached the prescribed relative height of the maximum.

Modern flash equipment such as the LFA 457 Micro Flash, equipped with a Nd:YAG laser and a liquid-nitrogen cooled InSb infrared detector provided by Netzsch Gerätebau GmbH, fulfil all technical requirements.

The obtained thermograms are fitted automatically and solved for $t_{0.5}$ by the determination of the maximum and minimum in relation to the baseline implementing pulse correction, different heat transfer models, and radial heat loss corrections.

Nevertheless, the thermal-diffusivity data obtained by this laser-flash method are substantially in error if the sample thickness becomes too small and/or significant coating thicknesses are applied. The optimum specimen thickness depends on the magnitude of the estimated thermal diffusivity and should be chosen so that the time to reach the maximum temperature is in the range between 20 ms and 200 ms [2] (see Fig. 1).

This means that specimens should always be thick enough to be a representative for the test material and that too small sample thicknesses can be a source of error especially in the measurement of highly conducting materials.

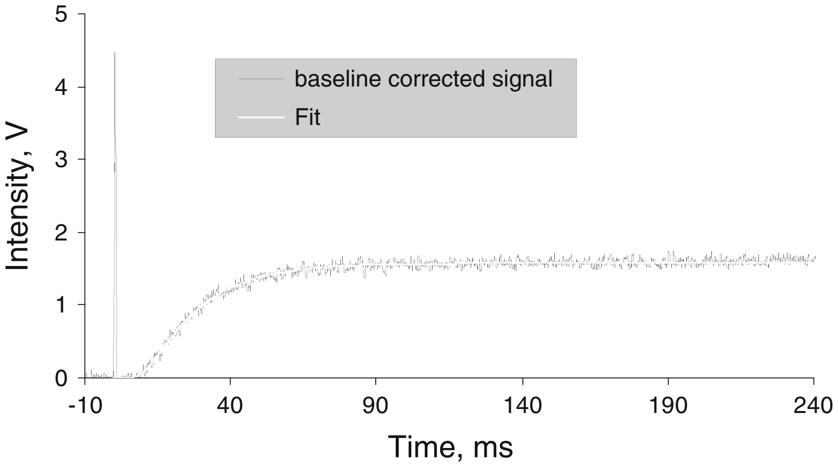

Fig. 1 Thermogram of a specimen sufficiently separated from the spike

In Fig. 1 the spike at the beginning of the measurement curve is an induction-caused electrical potential resulting from the trigger pulse of the laser. If the sample thickness becomes too small, a superposition of the introduced potential and the thermogram of the specimen takes place. This behavior is illustrated in Fig. 2a. Furthermore, surface effects and/or influences of coatings can have a strong impact on the test result.

An automated software analysis is used to fit this more or less distorted signal and to calculate the characteristic time of increase $t_{0.5}$ in an incorrect way, as long as standard evaluation routines are applied. This behavior is illustrated in Fig. 2b. The maximum of the fitted curve is in comparison to the recorded signal shifted to larger time values, and the calculation of the thermal diffusivity generally results in too small values. In such a case an increase of sample thickness is required to improve the quality of the data. A special definition of the fitting range (part of the instrument software) or other correction routines are sometimes very helpful but limited in application and only successful if the whole thermogram is not distorted.

Often it is not possible to increase the thickness of the sample. Nevertheless, accurate diffusivity values of these thin component parts for the purpose of process control and quality assurance are desired. Therefore, this incorrect calculation corresponding to the spike-thermogram interaction and/or coating influences was investigated at room temperature as a function of the sample thickness L for several metals with different thermal diffusivities and analyzed for general tendencies within defined experimental conditions.

2 Experiment and Results

For all experiments cylindrical-shaped specimens (12.7 mm in diameter) with plane parallel polished surfaces have been used. The tests were carried out after coating the

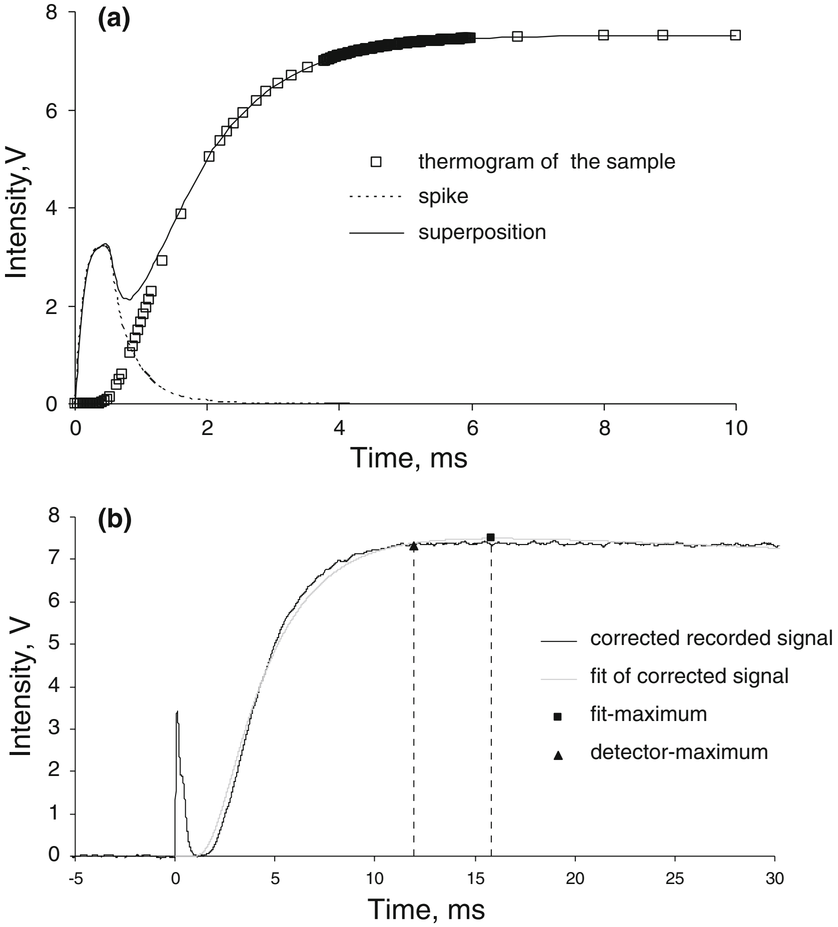

Fig. 2 (**a**) Illustration of spike-thermogram overlay and the resulting recorded superposition and (**b**) discrepancy in the derived maxima of the recorded signal and the corresponding software automated fit for a 2 mm copper–diamond composite

samples with graphite on the Netzsch Model LFA 457 Micro Flash equipped with a liquid-nitrogen cooled InSb infrared detector and a Nd:YAG laser.

The experimental conditions listed in Table 1 have been found to be the best applicable standard parameters within a wide temperature range covering most metals and their alloys. For all diffusivity calculations the Cowan model [9] including a pulse correction was used and each data point is the average value of five single measurements.

The miscalculated/measured thermal diffusivity α_m caused by the influence of the coating and the spike as a function of sample thickness for several metals, covering a wide range of diffusivities is summarized in Fig. 3. At larger sample thicknesses, the diffusivity values remain constant and are within ±1 % of literature data. To describe the constancy of the measured diffusivity for large specimen thicknesses and to enable

Table 1 Experimental parameters

Laser voltage	1730 V
Beam diameter	12.7 mm
Filter transmission	100 %
Signal duration	12 half times
Number of signal acquisition points	12000
Baseline stability threshold	1.5 V
Amplifier gain	127
Purge gas	Nitrogen
Gas flow	60 mL · min^{-1}
Sample temperature	(25.8 ± 0.2) °C

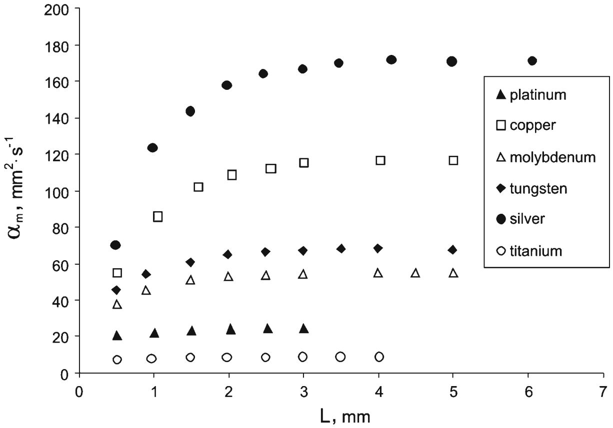

Fig. 3 Tendencies in miscalculation of the thermal diffusivity as a function of sample thickness for selected metals, covering a wide diffusivity range (without optimization of the fitting range or other corrections)

a detailed mathematical analysis, a so-called saturation value α_s is introduced, which is equal to the maximum of thermal diffusivity for each class of compounds.

For all specimens starting at a certain thickness, the spike and thermogram superposition has to be taken into account and the obtained thermal diffusivity is decreasing when the thickness L is reduced below a threshold value. The magnitude is, on the one hand, directly correlated to L and, on the other hand, a function of α_s. The meaning is, that for samples with high diffusivity, the curvature describing the decrease is much steeper than for samples with small diffusivity.

3 Analysis and Modeling

To enable a direct comparison of all curves, a normalization was introduced as well as a percent deviation Δ between α_s and α_m expressed as

$$\Delta = ((\alpha_s - \alpha_m)/\alpha_s)100 \tag{3}$$

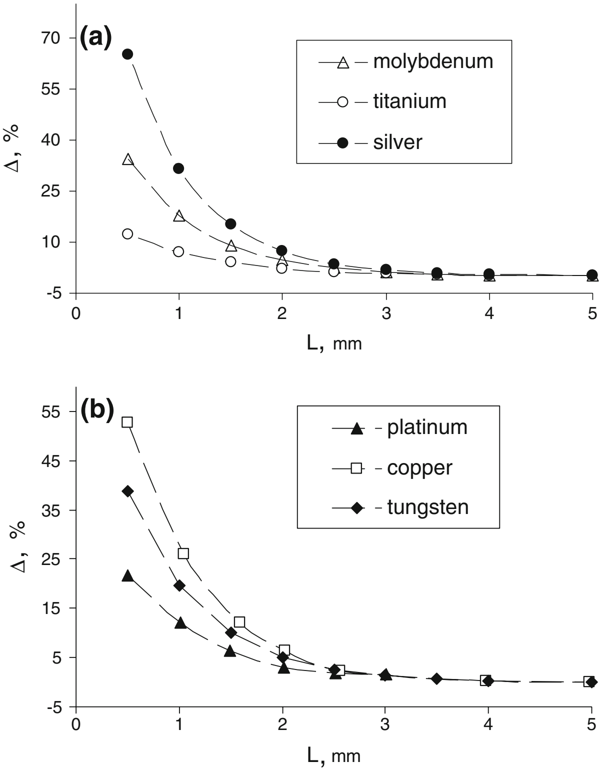

Fig. 4 (**a**) Deviation of measured diffusivity from the saturation value α_S as a function of sample thickness L for molybdenum, titanium, and silver and (**b**) deviation of measured diffusivity from the saturation value α_S as a function of sample thickness L for platinum, copper, and tungsten

Plotting Δ versus L shows the usual relationship between all curves (see Fig. 4a,b).

The mathematical expression for this thickness-dependent relationship is given by

$$\Delta = A\mathrm{e}^{(cL)} \tag{4}$$

Applying this kind of equation to fit the Δ versus L relationships for the investigated materials, six individual solutions for Eq. 4 with independent values for A and c are obtained.

To generalize Eq. 4, it was necessary to express A as a function of α_s. Therefore, the derived values of A for molybdenum, tungsten, platinum, copper, silver, and titanium are plotted versus the corresponding α_s, and we obtain the following relationship between A and α_s expressed by Eq. 5 and illustrated in Fig. 5:

$$A = 5.8275\alpha_\mathrm{s}^{0.6087} \tag{5}$$

The final step is to express the factor c in Eq. 4 as a function of α_s. Therefore, we apply the same procedure and plot c versus α_s. The correlation of all data points as a function of α_s is given by the following equation and plotted in Fig. 6:

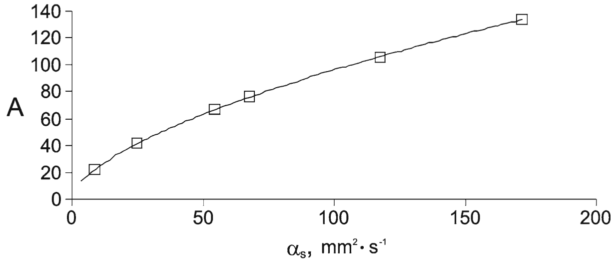

Fig. 5 Correlation of the *A*-values in Eq. 4 as a function of α_s described by Eq. 5

$$c = -0.1069 \quad \ln\alpha_\mathrm{s} - 0.8991 \tag{6}$$

Substituting *A* and *c* in Eq. 4 by Eqs. 5 and 6 and rearranging the resulting expression leads to the final generalized Eq. 7 to calculate Δ for a sample with known α_s at certain thicknesses *L*.

$$\Delta = 5.8275\alpha_\mathrm{s}^{(0.6087-0.1069L)}\mathrm{e}^{-0.8991L} \tag{7}$$

Substituting Δ in Eq. 7 by $\{((\alpha_\mathrm{s} - \alpha_\mathrm{m})/\alpha_\mathrm{s})100\}$ and extracting α_m gives the direct relation between the measured diffusivity α_m at a certain sample thickness *L* and the real diffusivity α_s;

$$\alpha_\mathrm{m} = -0.058275\alpha_\mathrm{s}^{(1.6087-0.1069L)}\mathrm{e}^{-0.8991L} + \alpha_\mathrm{s} \tag{8}$$

Equation 8 was tested for accuracy and is strictly valid for sample thicknesses of 1 mm to 6 mm starting at a thermal diffusivity α_s of 8 $\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$ up to 400 $\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$.

But even down to a 0.5 mm thickness, acceptable results for samples with small and moderate thermal-diffusivity values are obtained. This fact is quite unexpected, because at very small specimen thicknesses the influence of the graphite coating on the thermal-diffusivity determination by the formation of a multilayer system is no longer negligible.

Equation 8 can be rearranged to show the underlying relationship between α_s and the other variables/constants. The resulting equation is similar to a polynomial equation:

$$\alpha_\mathrm{s} + d\alpha_\mathrm{s}^{b} + c = 0 \tag{9}$$

$$\begin{gathered} d = -0.058275\mathrm{e}^{-0.8991L} \\ b = -0.1069L + 1.6087 \\ c = -\alpha_\mathrm{m} \end{gathered}$$

No closed-form solution to this equation is possible, but it is easily solved by numerical methods.

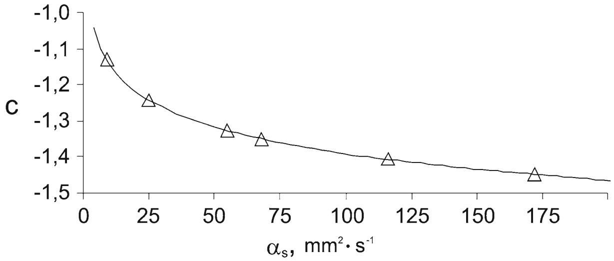

Fig. 6 Correlation of c-values in Eq. 4 as a function of α_s expressed by Eq. 6

4 Discussion and Conclusion

A numerical solution of Eq. 8 and the direct correlation for a series of α_m values as a function of L with the corresponding α_s is shown in Fig. 7. The line for 1 % and, respectively, the lines for 5 %, 10 %, and 20 % are marking the thickness range at which the difference between α_s and α_m is reaching the corresponding magnitude.

The graphic allows finding the α_s value for a measured α_m at a certain sample thickness L in a quick way. Another interpretation of Fig. 7 is the direct correlation of the required minimum thickness of a sample and the expected α_s to stay within the range of instrumental uncertainty (1 %) of thermal-diffusivity determination by the laser-flash method.

Furthermore, the equation is not only describing the behavior of conventional metals, but also composite materials satisfy this relationship. This fact is demonstrated in Fig. 8, where in the case of a copper–diamond composite and a silver–diamond composite, the theoretical tendencies of the deviations between calculated and measured values are compared for some sample thicknesses.

Finally it can be concluded that the derived equation enables an estimation of the error in calculation and the recalculation of thermal-diffusivity values for thin specimens or material parts from the experimental data. But it has to be explicitly expressed

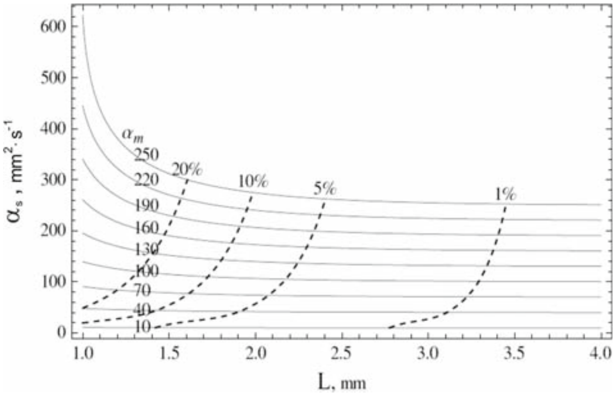

Fig. 7 Numerical solution of Eq. 8 for selected α_m values

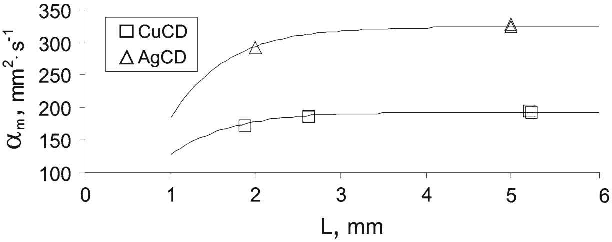

Fig. 8 Comparison of measured diffusivity for a copper-diamond composite (CuCD) and silver–diamond composite (AgCD) and the modeled curve of miscalculation as a function of *L*

that the derived correlations are only valid for the used experimental conditions and a certain thickness of the graphite coating. The applicability to experiments with other laser energies has not yet been investigated.

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