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Determination of the Thermal Diffusivity of Electrically Non-Conductive Solids in the Temperature Range from 80 K to 300 K by Laser-Flash Measurement

F. Hemberger · A. Göbel · H.-P. Ebert

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Abstract The adoption of the popular laser-flash method at temperatures far below 300 K is restricted by the weak signal-to-noise ratio and the limited spectral bandwidth of the commonly used mercury cadmium tellurite (MCT) infrared (IR) detector used as a non-contacting temperature probe. In this work, a different approach to measure the temperature rise in pulse heating experiments is described and evaluated. This method utilizes the change of the temperature-dependent electrical resistance of a thin strip of sputtered gold for the detection of a temperature rise as it was proposed by Kogure et al. The main advantage of this method at lower temperatures is the significantly higher signal-to-noise ratio compared to the commonly used IR detectors. A newly developed laser-flash apparatus using this detection method for the determination of the thermal diffusivity in the temperature range from 80 K to 300 K is presented. To test the accuracy of the new detection method, the thermal diffusivity of a borosilicate crown glass (BK7) specimen at 300 K was determined and compared to results derived with a MCT detector. Good agreement of the derived thermal diffusivity values within 3 % was found. The thermal diffusivity of BK7 and polycrystalline aluminum nitride (AlN) was measured at temperatures between 80 K and 300 K by a laser-flash method to test the functionality of the apparatus. Finally, the thermal conductivity was calculated using values for the specific heat capacity determined by temperature modulated differential scanning calorimetry (MDSC). Comparisons with literature data confirm the reliability of the experimental setup.

F. Hemberger (✉) · A. Göbel · H.-P. Ebert
Functional Materials for Energy Technology, Bavarian Center for Applied Energy Research (ZAE Bayern), Am Hubland, 97074 Würzburg, Germany
e-mail: hemberger@zae.uni-wuerzburg.de

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1 Introduction

1.1 General Description of the Laser-Flash Method

The laser-flash method, which was first introduced by Parker et al. [1] in 1961, is a frequently used method for the determination of the thermal diffusivity of solids. It allows a fast and reliable evaluation of thermal transport properties by means of a relative simple setup and specimen preparation. Typically one side of a disc-shaped specimen is heated by a short laser pulse, and the subsequent temperature rise at the opposite side is measured as a function of time using an IR detector. A calibration of the temperature probe is not necessary because the thermal diffusivity is calculated from the relative temperature rise. In Fig. 1a, schematic diagram of the laser-flash experiment is depicted.

The evaluation of a laser-flash experiment in order to achieve the thermal diffusivity incorporates the solution of the heat equation with appropriate boundary conditions in order to gain an analytical model which can be matched to the experimental data. The simplest model, which was introduced by Parker et al. [1], assumes that the entire surface of an isotropic specimen is uniformly heated by the laser pulse and that the heat flow through the specimen can be considered as one dimensional. Furthermore, it is presumed that the laser pulses are infinitesimally short and that there are no heat losses at the sample surfaces. This simplified model was improved by Cowan [2] by inclusion of heat losses at the sample front and back side. Afterwards, several models which consider also finite pulse lengths were presented, for example, by Azumi and Takahashi [3] and Cape and Lehman [4]. Non-local heat transport processes in diathermic media can also be considered in the evaluation of the thermal diffusivity [5]. If not mentioned otherwise, the model used in this work for the evaluation of the measured data is the model presented by Cape and Lehman [4].

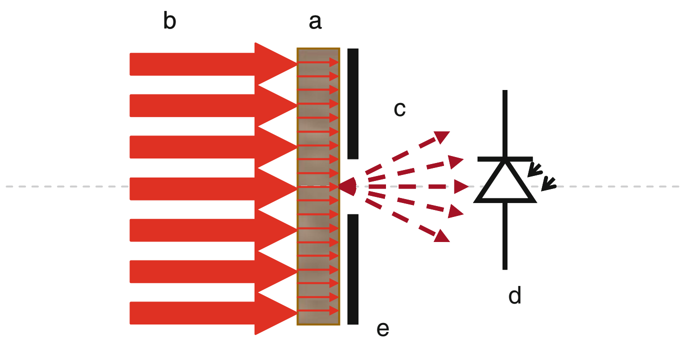

Fig. 1 Principle of a typical laser-flash setup: one side of a disk-shaped specimen (a) is heated with a laser pulse (b). The thermal radiation (c) emitted from the center of the opposite side of the specimen is detected as a function of time with an IR detector (d) and an optical diaphragm (e)

1.2 State of the Art

For time-dependent contact-free temperature probing, an IR detector is used in the vast majority of laser-flash equipment. This is caused by the laser-flash method being mostly used at room temperature and above. However, commercially available IR detectors are not suitable for low-temperature laser-flash measurements due to their degrading signal-to-noise ratio and limited spectral responsivity for temperatures far below 300 K. The lowest temperature at which reliable laser-flash measurements were performed using an MCT detector was 150 K [6]. Up to now, for temperatures below 150 K, no commercial laser-flash apparatuses are available.

Schulz et al. [7] performed low-temperature laser-flash measurements using a liquid helium cooled zinc-doped germanium IR detector which is sensitive down to 77 K. The alternative use of thermocouples is limited because of their heat capacity which influences not only the temperature field on the back side of the specimen but also the response time of the temperature probe [8]. The use of an intrinsic thermocouple, in which the specimen itself is part of the electric circuit and thereby the heat capacity of the temperature probe is widely reduced, was investigated by Heckman [8]. Fischer [9] discussed the suitability of micro-thermocouples based on semiconducting materials in laser-flash experiments. Finally, a promising alternative temperature detection method for laser-flash experiments based on a thin electrically conducting strip placed on the specimen surface was presented by Kogure et al. [10].

1.3 Objective of this Work

The objective of this work is the establishment and evaluation of a laser-flash apparatus for experiments in the temperature range between 80 K and 300 K. For detection of the time-dependent temperature rise, the temperature-dependent resistance change of a thin gold strip is used as described by Kogure et al. [10]. This detection method is very promising, not only because the heat capacity of a thin metal strip is very low in comparison to the heat capacity of micro-thermocouples but also because there is no need for special measuring equipment besides a Wheatstone bridge circuit and a digital storage oscilloscope. This temperature probe has so far only been used by Kogure et al. [10] on a polyethylene specimen. However, Kogure et al. hardly compared their results using a gold strip as temperature probe to conventionally determined thermal diffusivity values. In the work presented here, the thermal diffusivity and the corresponding thermal conductivity were determined in the temperature range from 80 K to 300 K and compared to available literature data in order to evaluate the reliability of the newly designed measurement setup. Two specimens, the first one made of borosilicate crown glass (BK7) and the second one made of polycrystalline aluminum nitride (AlN) were selected as test materials. At room temperature these two samples cover a wide range of thermal conductivity of about $1\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ (BK7) to about $230\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ (AlN). In order to test the reliability of the temperature detection by the resistance change of a thin gold strip, a comparison measurement with the commonly used non-contact temperature probing by an IR detector was performed on the same specimen.

2 Experimental Setup

2.1 General Overview

A schematic drawing of the newly designed low-temperature laser-flash apparatus is shown in Fig. 2. The experiment is performed in a cryostat, consisting of a Dewar filled with liquid nitrogen (LN_2) and a vacuum chamber containing the specimen and the optic components. By default, the atmosphere in the chamber consists of gaseous nitrogen to prevent freezing of moist air in the test equipment and to improve

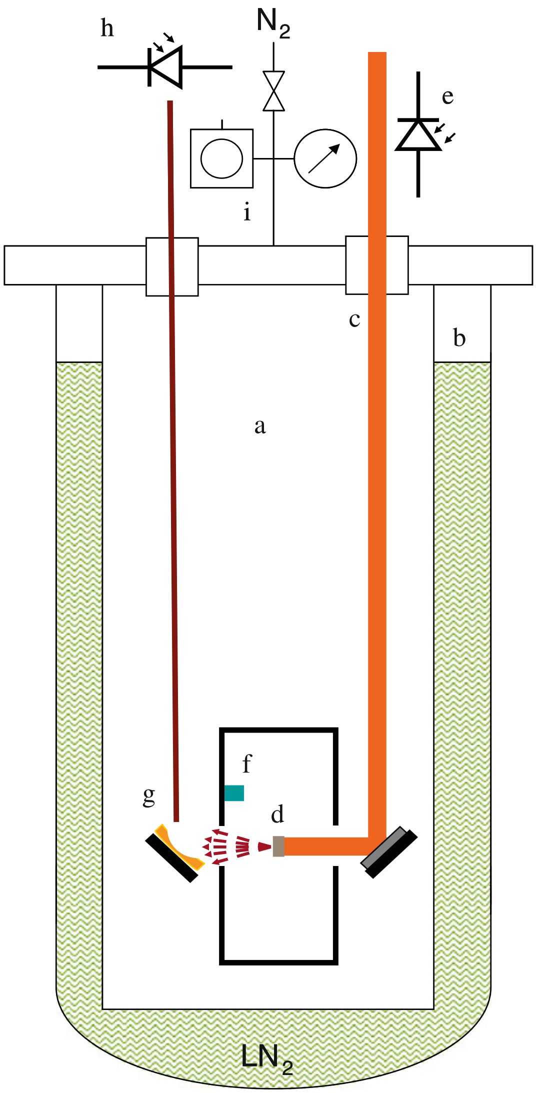

Fig. 2 Layout of the low-temperature laser-flash apparatus: The assembly consists of a vacuum chamber (a) surrounded by a LN_2 cryostat (b). The laser beam is fed through a window (c) to the surface of the specimen (d). The length and shape of the laser pulse is recorded by the pulse detector (e). The specimen itself is surrounded by a chamber with an electrical heater controlled by a Si diode temperature probe (f). For conventional temperature detection, the radiation of the backside of the specimen is transmitted by a concave gold coated mirror and an IR fiber (g) to the MCT detector (h). The atmosphere inside of the apparatus is controlled by an assembly of a vacuum pump, a pressure gauge, and an inlet for gaseous nitrogen (i)

the thermal coupling of the specimen to the measuring environment. In order to perform laser-flash measurements at temperatures above 80 K, the surroundings of the specimen are equipped with an electrical heater. To establish a constant temperature, a temperature controller (LTC-60, Leybold) and a Si diode are used. An additional Pt 100 resistance probe is used to measure the temperature close to the specimen. A Nd:YAG laser (Starweld 40, rofin) is used with a wavelength of 1064 nm which produces single pulses of lengths between 0.3 ms and 20 ms and accordingly energies between 1 J and 20 J. In order to guarantee a homogeneous energy dissipation on the specimen surface, the laser is running in multimode. Before the laser pulse is diverted to the specimen, it is focused by a beam concentrator to a diameter of about 15 mm to increase the energy density. The IR radiation emitted by the specimen backside is focused on an optical polycrystalline IR fiber (CeramOptec) by a concave mirror and thus transmitted to the LN_2-cooled MCT detector. The core of the fiber has a diameter of 400 μm and consists of AgCl; the clad with a diameter of 500 μm is made of AgBr. The starting time and the shape of the laser pulse are detected by a photodiode. The measured signal is recorded by a digital storage oscilloscope (PM3350, Philips) which is triggered by the signal of the photodiode.

Usually, repeated measurements were averaged in order to increase the signal-to-noise ratio and to minimize the influence of possible signal drift of the detector on the analysis results. The software used for evaluation of the measured data incorporates all the analytical models mentioned before in the introduction.

2.2 Gold Strip Temperature Detection System

An alternative approach used in this work is the detection of the temperature rise by measuring the temperature-dependent resistance increase of a thin gold strip which is prepared by magnetron sputtering (Polaron SC 7640, Quorum Technologies Inc.) on the back side of the specimen [1]. Obviously, the detection of the temperature rise via the resistance change of a thin gold strip can only be applied easily to electrically non-conductive specimens.

The sputtered gold strips were approximately 300 μm wide, 6 mm long, and less than 100 nm thick which results in an electrical resistance of several 10 Ω for the strips. The ends of the strips are provided with contact pads made of silver conductive paint which are electrically contacted by means of two spring-mounted contact pins. In order to minimize the influence of these contact pins on the laser-flash measurement, the contact area and the contact pressure are as small as possible. In Fig. 3a, schematic drawing of the specimen holder and the electrical contacts of the gold strip is depicted.

In order to detect the time-dependent resistance increase and therefore the temperature rise, the gold strip is integrated in a Wheatstone bridge, see Fig. 4. This circuit is commonly used for the precise detection of small resistance changes. The change of the bridge voltage U_B is directly proportional to the change of the resistance of the gold strip R_{GF} provided that the resistance change is much smaller than the sum of R_{GF} and R_3. The sensitivity of the Wheatstone bridge circuit is maximized by matching the value of R_3 equal to R_{GF} and by adjusting the ratio of R_1 to R_2 to set U_B equal to zero prior to the experiment.

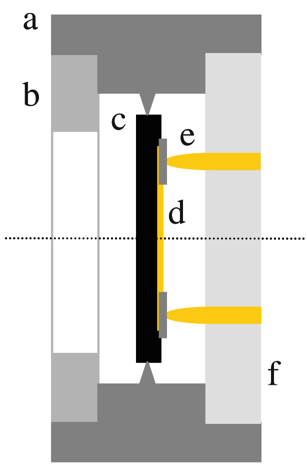

Fig. 3 Cross section of the specimen holder (a) with a diaphragm for shaping the laser pulse (b), a specimen supported by three spikes (c), a sputtered gold strip with contact pads made of silver conductive paint (d), a spring-loaded contact pin (e), and a support for the contact pins made of Teflon® (f)

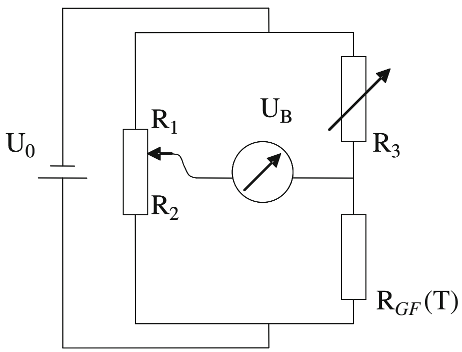

Fig. 4 Wheatstone bridge circuit for the determination of the temperature-dependent change of R_{GF}

Since only the relative temperature rise has to be measured in laser-flash experiments, neither the exact resistance of the gold strip nor its linear temperature coefficient must be known. In the same way, the influence of the resistance of the feed cable to the specimen in the cryostat is compensated by adjusting the bridge voltage equal to zero.

Typical values of 1 V for U_0 cause a heat dissipation of 5 mW or less in the gold strip during the measurement. As the gold strip has very good thermal contact with the specimen, the temperature rise of the strip caused by self-heating is negligible. Due to the small amount of gold used, the influence of the heat capacity of the strip on the laser-flash measurements can be neglected.

3 Specimens

3.1 Borosilicate Crown Glass (BK7)

BK7 can be manufactured with high homogeneity and is a standard material for optical applications in the visible spectral range due to its chemical stability, good scratch resistance, and low impurity content [11]. An intercomparison on the thermal properties of BK7 glass manufactured by the Schott AG (Mainz, Germany) was initiated by the Thermophysics Working Group (www.ak-thermophysik.de) in 2001. The results were presented at the 16th European Conference on Thermophysical Properties in 2002. The same specimen that was used for this purpose was also used in this work for low-temperature laser-flash measurements. Its diameter was measured as 20 mm, the thickness as 1.183 mm, and its density was calculated as $2460\,\text{kg} \cdot \text{m}^{-3}$. No values for the thermal diffusivity or the thermal conductivity of BK7 at temperatures below room temperature could be found in publications until this work.

For detection of the time-dependent temperature rise at the rear side of the specimen, a thin gold strip was sputtered onto the surface. The electrical resistance of this strip was determined to be 18 Ω at room temperature. The front side of the specimen was coated with a thin layer of 5 μm to 10 μm of graphite to improve absorption of the laser pulse energy. For temperature probing with the MCT detector, the opposite side of the specimen was also coated with graphite in order to increase the emission of radiation.

3.2 Polycrystalline Aluminum Nitride (AlN)

The second investigated specimen was a polycrystalline AlN ceramic. High values for the thermal conductivity of up to $319\,\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ for pure single crystals of AlN at room temperature are reported [12]. However, the thermal conductivity of commercially available AlN can be significantly lower due to impurities, mainly oxygen, which increase the thermal resistance as they are additional scattering centers for the lattice phonons. The specimen examined in this work was prepared by Bickermann et al. [13] by a special physical vapor transport (PVT) growth process which allows the fabrication of polycrystalline AlN samples with a very low oxygen content of only 100 ppm. The investigated specimen consists of single crystals with a dimension of about 0.5 mm each. The mean density of the specimen was determined as $3203\,\text{kg} \cdot \text{m}^{-3}$, the thickness as 1.019 mm, and the diameter as 12.9 mm.

In order to prepare an electrical conducting gold strip by a sputtering technique on the AlN-specimen surface, it was necessary to bridge the small gaps between the single crystals. For this, a thin intermediate layer of silicon dioxide was applied on the backside of the specimen by the dip-coating technique. The thickness of the applied layer could be determined to approximately 300 nm by use of a scanning electron microscope. A comparative laser-flash measurement at room temperature using an IR detector showed no influence of this additional intermediate layer on the determined values of the thermal diffusivity. The gold strip finally sputtered on top of this layer has an electrical resistance of 50 Ω at room temperature. The front side of the specimen was coated with a thin layer of 5 μm to 10 μm of graphite to improve absorption of the laser pulse energy. For temperature probing with the MCT detector, the opposite side of the specimen was also coated with graphite in order to increase the emission of radiation.

4 Results

4.1 Comparison of the Temperature Detection Methods

The thermal diffusivity of the BK7 glass specimen was determined at 300 K by two independent laser-flash experiments: first, using the standard detection method of probing the temperature rise on the backside of the specimen with the MCT detector; and second, using the proposed resistance detection method. In Fig. 5, both resulting laser-flash curves can be found; the signals were normalized for an easier comparison. In the measurement curve retrieved by the MCT detector, the typical instantaneous temperature shift for a semi-transparent specimen at time zero can be seen; due to the low absorption coefficient of gold of approximately 3 % at 10 μm, this peak is not present in the measurement curve derived with the resistance method. In order to evaluate the data retrieved by the MCT detector, the analytical model described by Hofmann et al. [5] was used, and the thermal diffusivity of the specimen was determined to be $(0.540 \pm 0.027)\,\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$. An evaluation according to Cape and Lehman [4] of the measurement data gained by the resistance detection method yields a value of $(0.523 \pm 0.026)\,\mathrm{mm}^2 \cdot \mathrm{s}^{-1}$ for the thermal diffusivity. The good agreement of the results within 3 % demonstrates the reliability of the new detection method.

It is also noticeable that the signal-to-noise ratio in the data retrieved with the resistance temperature probe is significantly better than by measurements with an IR detector. This results from the output signal of the IR-detection system being quite sensitive to fluctuations of the room temperature and electromagnetic disturbances in the vicinity.

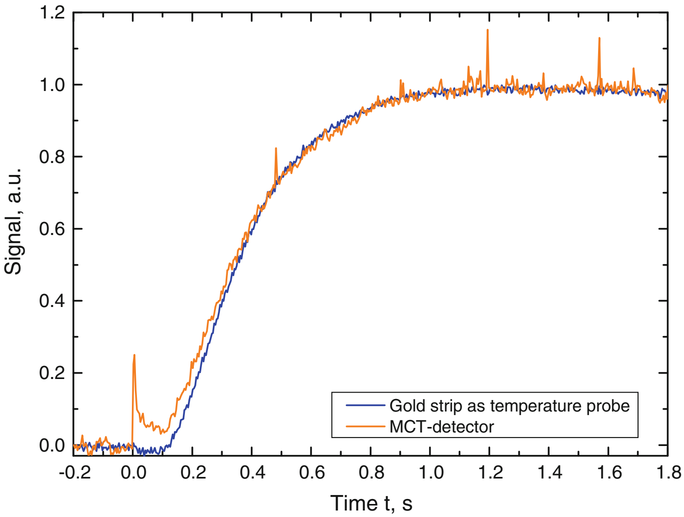

Fig. 5 Comparison of both temperature detection methods using a specimen of BK7 glass at 300 K

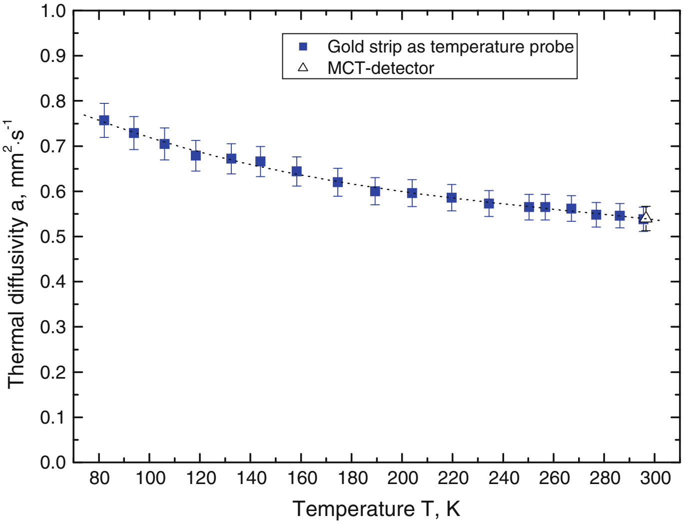

Fig. 6 Thermal diffusivity of BK7 glass as a function of temperature, measured by laser-flash method. *Dotted line* is added as a guide to the eye

4.2 Low-Temperature Thermal-Diffusivity Measurements on BK7 and AlN

The thermal diffusivity of both specimens was determined by the laser-flash method using a laser pulse of 0.3 ms duration in the temperature range between 80 K and 300 K.

Figure 6 shows the obtained thermal diffusivity values for the BK7 glass specimen. A comparative value determined for the same specimen at 300 K by a laser-flash experiment using an MCT detector is also depicted.

Figure 7 shows the obtained thermal diffusivity values for the polycrystalline AlN specimen. A comparative value determined at the same specimen at 300 K by a laser-flash experiment using a MCT detector is also presented.

The relative uncertainty of the thermal diffusivity values of 5 % was evaluated considering the uncertainty in determination of the specimen thickness, the uncertainty in the determination of the exact starting time of the laser pulse, and the inaccuracy due to the non-linear fit used for deriving the thermal diffusivity from the measured data [14].

A comparison of the thermal diffusivity values determined at room temperature with both temperature detection methods on the BK7 and the AlN specimens can be found in Table 1.

4.3 Specific Heat Capacity

The specific heat capacity of BK7 glass and the polycrystalline AlN specimen was measured by the MDSC technique (Q2000 by TA Instruments Inc.) in the range of 190 K to 300 K with a relative uncertainty of 5 %. Using literature data, an extension down to 80 K was made. As no literature values of the specific heat capacity of BK7

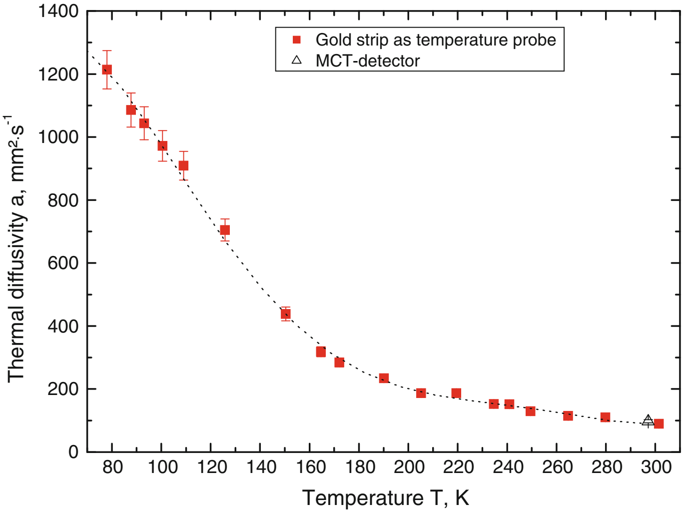

Fig. 7 Thermal diffusivity of a polycrystalline AlN specimen as a function of temperature, measured by laser-flash method. *Dotted line* is added as a guide to the eye

Table 1 Thermal-diffusivity values of the BK7 and AlN specimens at room temperature

Temperature probe	BK7		AlN	
	Temperature (K)	Thermal diffusivity ($mm^2 \cdot s^{-1}$)	Temperature (K)	Thermal diffusivity ($mm^2 \cdot s^{-1}$)
Gold strip	296 ± 1	0.538 ± 0.027	302 ± 1	89.8 ± 4.5
MCT detector	297 ± 1	0.540 ± 0.027	297 ± 1	97.8 ± 4.9

glass for low temperatures are available, the specific heat capacity of fused silica glass taken from Touloukian et al. [15] was used for extrapolation as the shape of the temperature dependence is comparable for both glass species. The relative uncertainty of the extrapolated values for the specific heat capacity of BK7 was expanded to 10 % due to the extrapolation process and the unavailability of uncertainty values for the specific heat capacity of fused silica glass in the extrapolated range.

For the specific heat capacity of the AlN specimen in the temperature range from 80 K to 190 K, literature data taken from Touloukian et al. [15] were used as our measured values for temperatures between 190 K and 300 K coincide very well with these literature values. According to our measurement uncertainty, the uncertainty of these literature data was estimated to 5 %.

The measurement results and the data achieved from the literature are shown in Fig. 6.

4.4 Calculated Thermal Conductivity

The thermal conductivity as a function of temperature can be calculated from measurement results or literature data for the thermal diffusivity, specific heat capacity, and

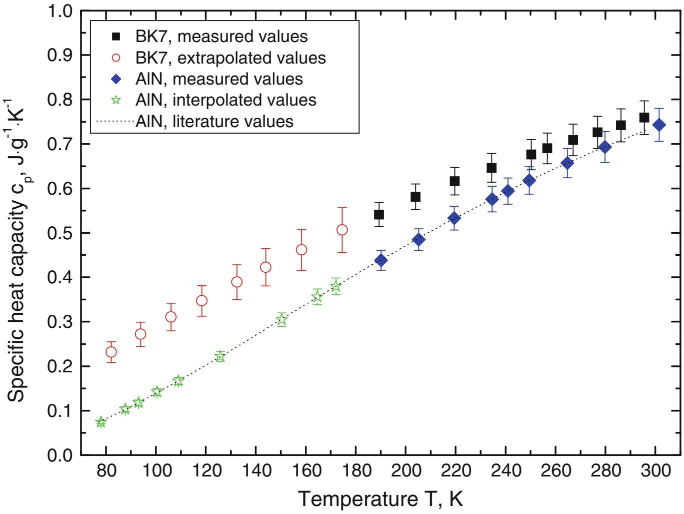

Fig. 8 Specific heat capacity of the investigated specimens. Depicted are measurement results, the extrapolated values for BK7, and literature data for the specific heat capacity of AlN (Touloukian et al. [15])

material density by simple multiplication of the values. The temperature dependence of the density can be neglected in this calculation because the magnitude of the thermal expansion of glasses and polycrystalline aluminum nitride is in the investigated temperature range smaller than 0.1×10^{-6} and 2×10^{-6} [16,17], respectively. The relative uncertainty in the determination of the density of the investigated specimens was 3 %.

The relative uncertainty of the calculated results for the thermal conductivity was evaluated according to the rules of error propagation considering the uncertainties of the specific heat capacity, density, and thermal diffusivity [14].

The values of the calculated thermal conductivity of BK7 glass are shown as a function of temperature in Fig. 9. At 296 K, the thermal conductivity is (1.011 ± 0.078) $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$. The relative uncertainty of the thermal conductivity is 7.7 % for $T > 190$ K and 11.6 % for $T < 190$ K due to the larger relative uncertainty of the extrapolated specific heat capacity values. By comparison to the temperature dependence of the specific heat capacity shown in Fig. 8, it can be seen that the temperature dependence of the thermal conductivity is mostly influenced by the temperature dependence of the specific heat capacity as was expected for an amorphous material. Within the stated relative uncertainty, the value for the thermal conductivity of BK7 at room temperature determined in this work coincides with literature data [16].

Figure 10 shows the calculated values of the thermal conductivity of the AlN specimen. The maximum thermal conductivity of the examined specimen of about (500 ± 39) $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ can be found at 125 K. At 297 K, the thermal conductivity is (229 ± 18) $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$. The relative uncertainty was calculated to be 7.7 % over the whole temperature range. A comparison of these results to values for the thermal conductivity reported by Slack et al. [12] is critical, due the fact that the investigated

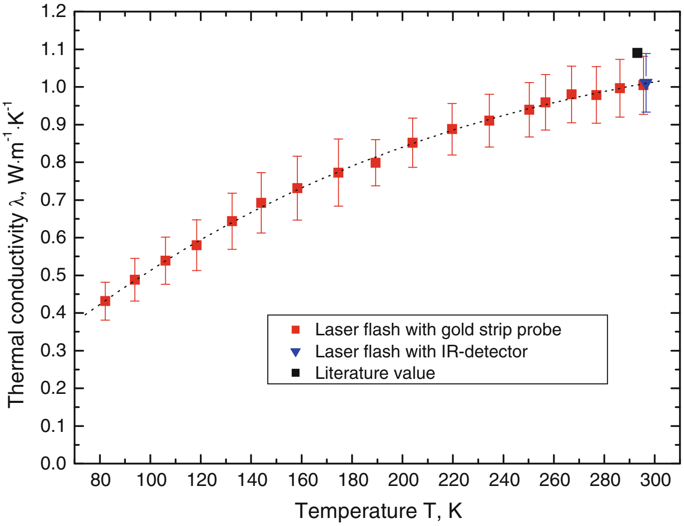

Fig. 9 Calculated values of the thermal conductivity of the investigated BK7 specimen, plotted as a function of temperature. For comparison, a literature value is also depicted [16]. *Dotted line* is added as a guide to the eye

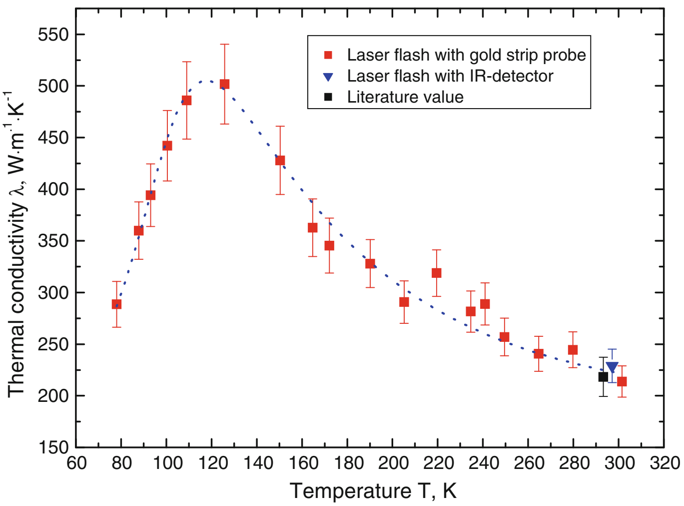

Fig. 10 Temperature dependence of the calculated thermal conductivity of the polycrystalline aluminum nitride specimen. For comparison, a literature value is also depicted [13]. *Dotted line* is added as a guide to the eye

specimens are not comparable with respect to oxygen content and crystal size. The value of the thermal conductivity of AlN at room temperature which was reported by Bickermann et al. [13] and which is also depicted in Fig. 10 was determined in the year 2004 by laser-flash and MDSC measurement on the same specimen investigated in this work, but with different measurement equipment. Therefore, a reasonable agreement of results was expected.

5 Conclusions

A newly designed low-temperature laser-flash apparatus for the determination of thermal diffusivity in the temperature range between 80 K and 300 K has been established. A key feature of the new apparatus is the measurement of the temperature rise at the back side of the specimen by the resistance change of a thin gold strip, which has been sputtered onto the specimen surface. The main advantages of this temperature probe are its applicability at lower temperatures and a significantly better signal-to-noise ratio, as the sensitivity of commonly used MCT detectors declines rapidly with decreasing temperature. Therefore, it is now possible to use a laser pulse with a shorter length and less power compared to the temperature probing by an MCT detector. This leads to better applicability of the laser-flash method to specimens with high thermal diffusivity and to less heating of the specimen by the laser pulse during the experiment.

At room temperature both temperature probes result in comparable values of thermal diffusivity. Furthermore, the use of a thin gold strip as a temperature probe for investigation of the semitransparent specimen has the advantage of avoiding direct radiation between the surfaces of the specimen due to the low absorption coefficient of gold in the appropriate wavelength range.

However, up to now it is not possible to perform measurements with the new detection method on electrically conducting or porous specimens. The investigation of electrically conducting specimens by use of an insulation intermediate layer is an objective of future work.

In order to test the reliability of the new experimental setup, the thermal diffusivity of a polycrystalline AlN specimen and an optical BK7 glass was determined in the range between 80 K and 300 K. Values for the thermal diffusivity of the investigated specimens were determined in the range from $0.5\,\text{mm}^2 \cdot \text{s}^{-1}$ up to $1200\,\text{mm}^2 \cdot \text{s}^{-1}$ with a relative measurement uncertainty of 5 %. Afterwards, the thermal conductivity of the investigated specimen was calculated using measurement results by the MDSC technique and literature data for the specific heat capacity. The range of the calculated values for the thermal conductivity of the investigated specimens was between $1\,\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ and $500\,\text{W} \cdot \text{m}^{1} \cdot \text{K}^{-1}$ with a relative uncertainty from 7.7 % to 11.6 %. The calculated values coincide satisfactorily with the available literature data.

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