A New Method for the Determination of the Specific Heat Capacity Using Laser-Flash Calorimetry Down to 77 K

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Abstract A new method for evaluation of the specific heat capacity in the temperature regime between 77 K and 330 K using laser-flash calorimetry is presented. Usually, laser-flash calorimetry is accomplished by performing an additional laser-flash measurement on a reference specimen with a known specific heat capacity and by comparing the maximum rear-side temperatures rises. In this study, the calibration is achieved by comparison of the rear-side temperature rise to specific-heat-capacity data determined by other methods in an adjacent temperature regime. Subsequently, the thus yielded proportional factor is used for the evaluation of the specific heat capacity from laser-flash measurements at temperatures where no specific-heat-capacity data are available. The reliability of this method is shown by performing measurements on a material with known specific heat capacity, aluminum oxide. Furthermore, the specific heat capacity and thermal conductivity of borosilicate crown glass (BK7) was determined experimentally.

Keywords Aluminum oxide · BK7 glass · Laser-flash · Low temperature · Specific heat capacity

1 Introduction

The laser-flash method is a standard method for the determination of the thermal diffusivity of solids [1]. One side of a thin specimen is heated by a short laser pulse, and the subsequent temperature rise at the opposite side of the specimen is recorded as a function of time. As the temperature rise is usually measured contact-free using an
infrared detector, this method is mostly used at temperatures above room temperature due to the rapidly declining sensitivity of infrared detectors at lower temperatures. In our study, we are using the temperature-dependent resistance of a thin gold strip which is sputtered onto the rear side of the specimen as an alternative temperature probe below room temperature [2,3].

Using the thermal diffusivity which was determined by the laser-flash method, the thermal conductivity can be calculated by multiplying the thermal diffusivity $a(T)$, the specific heat capacity $c_p(T)$, and the density $\rho(T)$ of the specimen

\[ \lambda(T) = a(T)c_p(T)\rho(T) \] (1)

Besides the thermal diffusivity, the specific heat capacity can also be obtained by the laser-flash experiment, which is also known as laser-flash calorimetry [1]. Not only the absolute rear-side temperature rise but also the amount of energy absorbed by the specimen must be known for calculation of the specific heat capacity. As the latter can hardly be determined experimentally, usually measurements on a reference material with known specific heat capacity and similar thermophysical properties are performed [4,5]. It is of great importance that not only the temperature of both specimens, the reference and sample material, is the same during the measurement but also the amount of energy that is absorbed by the specimens. However, one big problem in performing laser-flash calorimetry with a reference specimen is the fact that even when the surfaces of both specimens are coated with graphite, the spectral emissivities of the different materials are mostly unequal, for example, due to differences in surface roughness [6]. This problem could be solved using a calibrated surface, the so-called absorption disk, which is a thin layer of glassy carbon glued to the front side of the specimen [7]. One disadvantage of this method is the contact resistance between the absorption disk and the specimen which changes every time the absorption disk is glued to another specimen.

A new approach to determine the specific heat capacity over a wide temperature range with the laser-flash technique is to calibrate the maximum rear-side temperature rise using an averaged temperature-independent proportional factor. This factor is calculated by comparison of the maximum rear-side temperature rise to specific-heat-capacity data which is already available in an adjacent temperature range. Afterward, this calibration can be extrapolated to temperatures where no specific-heat-capacity data are available, but laser-flash measurements can be performed. An advantage of this method is that there is no need to perform laser-flash measurements on a reference specimen. This method is very promising as the gold strip temperature probe used in this study is quite suitable for this evaluation method, especially at low temperatures. In comparison to infrared detectors, the detector signal height during the laser-flash measurement is not influenced by environmental factors such as the different infrared optical properties of the specimen and reference.

Thus, the objective of this study is to test the method described above with our low-temperature laser-flash apparatus, to gather values for the specific heat capacity between 77 K and 300 K.