A New Laser Flash System for Measurement of the Thermophysical Properties

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1. Introduction
In recent decades, the laser flash method [1] has been developed into one of the most widely used techniques for measuring the thermal diffusivity and thermal conductivity of various kinds of solids, powders and liquids. This technique allows heating the front side of a small, usually disk-shaped plane-parallel sample by a short energy (laser) pulse. The temperature rise on the rear surface is measured versus time. The rapid temperature drop on the surface, for example, due to laser heat loss [2] and finite pulse effects [3]. Some analysis methods are already available which simultaneously take both of these into account in the optimum way [4]. Furthermore, modern systems allow for easy sample loading and flexible sample geometries through an enlargement optics. Positioning of the laser can be controlled by the software via the voltage level of the capacitor bank and/or via a filter system positioned in the outlet area of the laser system. The laser pulse is deployed through an enhancement optics system which adjusts the beam diameter to the required sample diameters. From the enhancement optics system, the laser pulse is guided via a mirror through a window into the vacuum light tight sample chamber. Inside the sample chamber is an automatic sample changer for up to 3 samples. The samples are positioned in easily user-interchangeable sample carriers which can be adapted to the actual sample dimensions (square samples, disk-shaped samples with various diameters, etc.).

2. Experimental
Presented in figure 1 is the schematic design of the NETZSCH LFA 457 MicroFlash (measurement part). Positioned in the base of the device is the head of an Nd:YAG laser. The laser has a pulse length of 330 µs and a pulse energy output of up to 15 J/pulse. Power is supplied to the laser by a capacitor bank positioned in a separate box. The power output of the laser can be controlled by the software via the voltage level of the capacitor bank and/or via a filter system positioned in the outlet area of the laser system. The laser pulse is deployed through an enhancement optics system which adjusts the beam diameter to the required sample diameters. From the enhancement optics system, the laser pulse is guided via a mirror through a window into the vacuum light tight sample chamber. Inside the sample chamber is an automatic sample changer for up to 3 samples. The samples are positioned in easily user-interchangeable sample carriers which can be adapted to the actual sample dimensions (square samples, disk-shaped samples with various diameters, etc.).

3. Results and discussion
Shown in figure 2 are the thermal diffusivity results on copper and aluminum between room temperature and 600°C (measurement results and literature values). The literature values taken have a stated uncertainty of 3-4%. Comparing the measurement results with the literature values, it can be seen that the deviations between the results are less than the uncertainty of the literature values. Figure 3 shows the result of a direct thermal conductivity determination (measurement of room temperature bulk density, specific heat and thermal diffusivity and calculation of the thermal conductivity) of an NIST certified thermal conductivity standard SRM 1461 stainless steel. The test results are compared with the values listed in the NIST certificate [7]. As can be seen, the deviations between the test results and the literature results are generally within 5% of the stated uncertainty. This is well within the stated uncertainty of this standard reference material. Presented in figure 4 are the thermal diffusivity results on an NR/BR rubber mixture between -125 and 75°C. The specific heat used for calculating the thermal conductivity was measured by an additional DSC test. It can be clearly seen that the thermal diffusivity decreases over the entire temperature range. Between -75 and -50°C, a step is visible in the thermal diffusivity. The specific heat increases over the entire temperature range. Also here, a step is visible in the same temperature range as the step in the thermal diffusivity results. Both steps can be explained by a glass transition in the rubber material. The thermal conductivity does not show any effects of the glass transition; a nearly linear increase was obtained over the entire temperature range.

Figure 1. NETZSCH LFA 457 MicroFlash (1100°C-Version)

Figure 2. Thermal diffusivity of copper and aluminium between room temperature and 600°C (measurement results and literature values).

Figure 3. Thermal conductivity of NIST SRM 1461 stainless steel (comparison between measurement results and the values from the certificate).

Figure 4. Specific heat, thermal diffusivity and thermal conductivity of an NR/BR rubber mixture between -125 and 75°C.

Figure 5. Specific heat, thermal diffusivity and thermal conductivity of a silicon carbide (SiC) ceramic between room temperature and 1000°C.

Depicted in figure 5 are the thermophysical properties of an SiC (silicon carbide) ceramic between room temperature and 1000°C. The thermal diffusivity and specific heat were determined in the LFA 457. Using the measured data, the thermal conductivity was calculated by multiplying the thermal diffusivity, specific heat and room-temperature bulk density. The thermal diffusivity values decrease over the entire temperature range. The specific heat increases, as can be expected from the Debye theory. The thermal conductivity decreases over the entire temperature range, as well. However, it is at a very high level (nearly 150 W/(m*K)) at room temperature, which is typical for polycrystalline SiC ceramics.

Conclusion
A new laser flash device was developed for the characterization of a wide range of materials in solid and liquid state. Tests on standard materials have proven the reliability of the new system. The measurement results presented on a wide range of materials demonstrate the capability of the instrument in various application fields.

Literature