A New Laser Flash System for Measurement of the Thermophysical Properies

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1. Introduction

Over the past few decades, the flash method [1] has 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 entails 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 using an infrared detector. By placing the sample into a tube furnace, temperature-dependent measurements can easily be carried out as well.Since introduction of the method by Parker et al. [1], various improvements to it have been made. New evaluations have been developed which account for 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 determination of the thermal diffusivity *a* and the specific heat c_p. Once the bulk density of the material is known, a direct thermal conductivity determination is possible according to:

$$(T) = (T)\; c_p(T)\; a(T)\;.$$

The new NETZSCH model LFA 457 MicroFlash combines modern technology with state-of-the-art data processing techniques [5]. The fully automated top loading flash system with an Nd:YAG laser on the bottom and an infrared detector on top allows for easy sample loading and flexible sample geometries while providing an optimum signal-to-noise ratio. The specially designed 500 kHz data acquisition system allows measurement of critical samples such as thin films or high-conductivity materials. The system is fully software-controlled. The measured data is analyzed taking both heat loss and finite pulse effects into consideration. Standard constituents of the analysis software are the improved Cape-Lehman mode [5], the possibility of considering for radiative heat transfer, and non-linear regression routines for the characterization of two- and three-layer systems.

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 enlargement optics system which adjusts the beam diameter to the required sample diameters. From the enlargement optics system, the laser pulse is guided via a mirror through a window into the vacuum-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 adjusted to the actual sample dimensions (square samples, disk-shaped samples with various diameters, etc.).

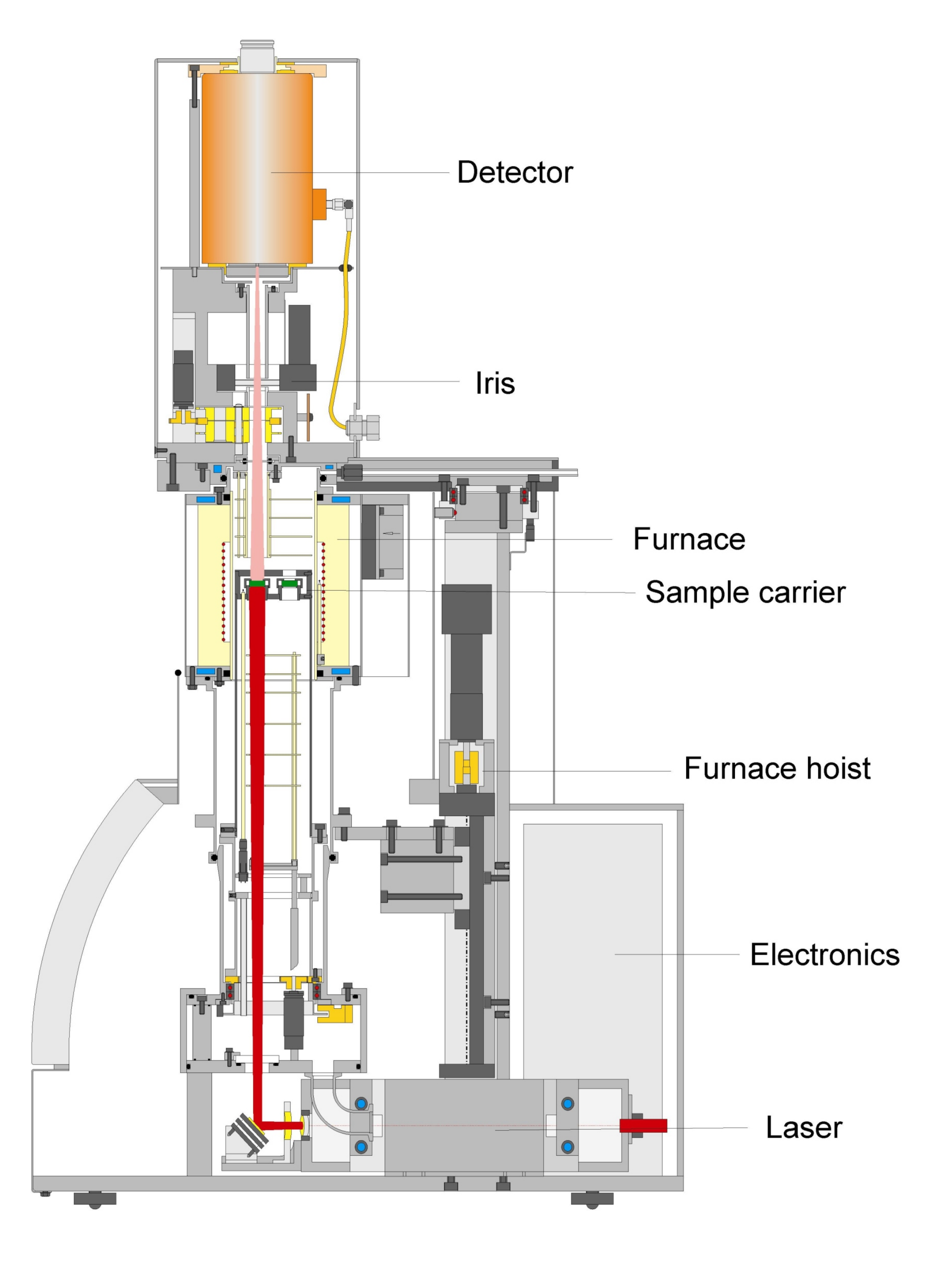

Figure 1. NETZSCH LFA 457 MicroFlash (1100°C-Version)

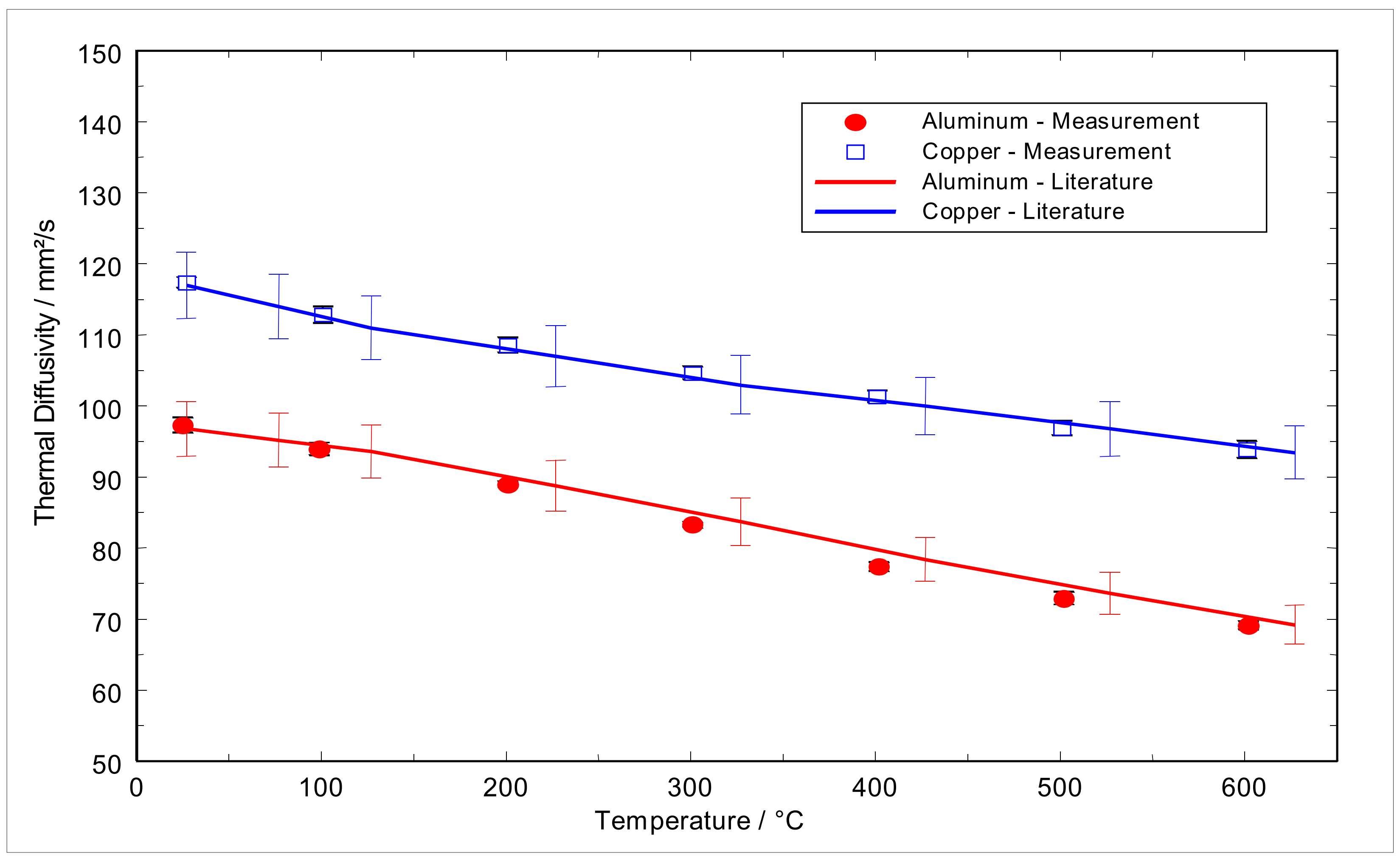

Figure 2. Thermal diffusivity of copper and aluminum between room temperature and 600°C (measurement results and literature values).

3. Results and discussion

Shown in figure 2 are the thermal diffusivity results on copper and aluminum between room temperature and 600°C, together with literature values [6]. 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 (symbols) and the values from the certificate (line with error bars) are generally within approx. 3 %. This is within the stated uncertainty of this standard reference material.

Presented in figure 4 are the measurement results of an NR/BR rubber mixture between -125 and 75°C. The specific heat used for calculating of the thermal conductivity was measured by an additional DSC test. It can clearly be 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.

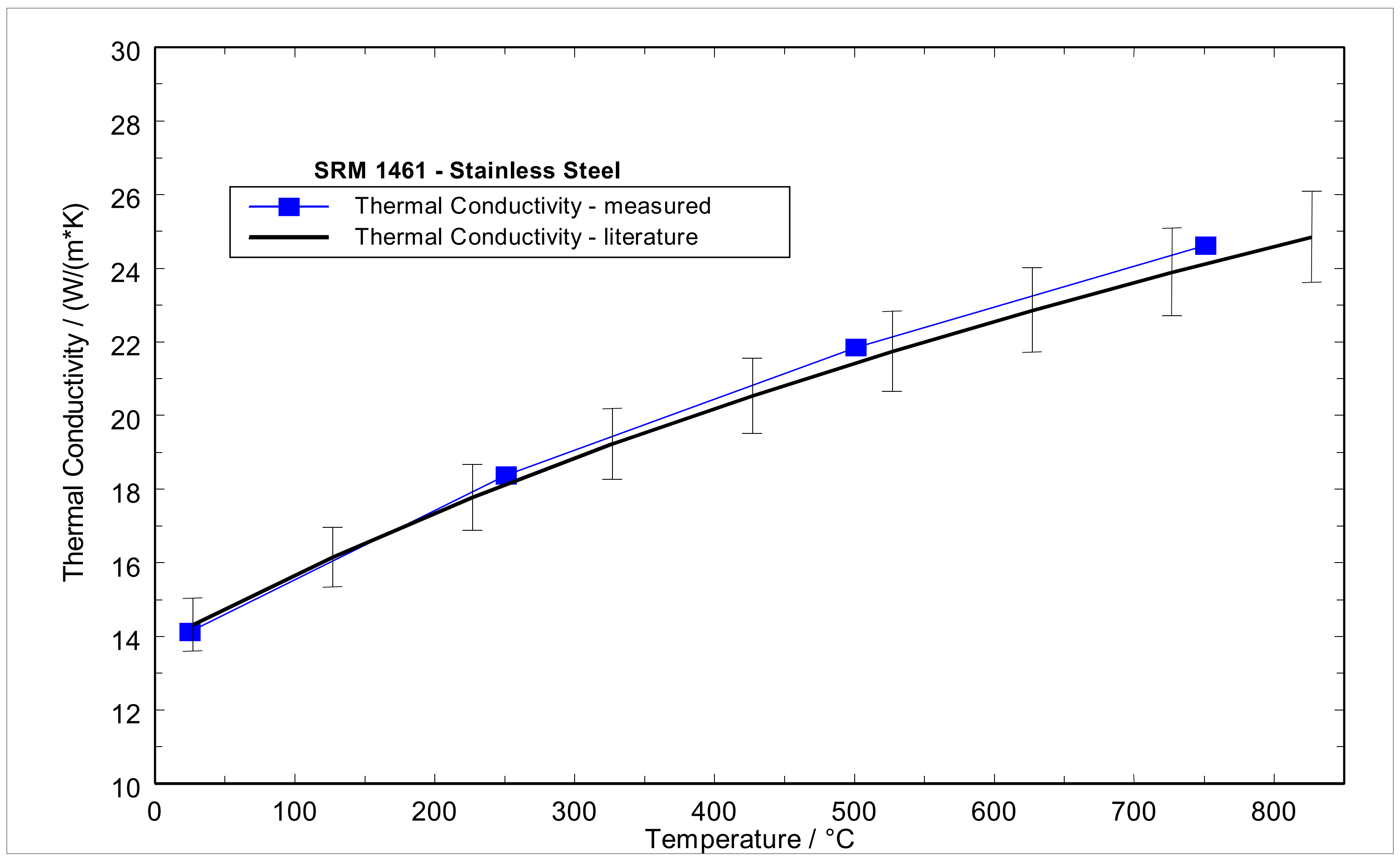

Figure 3.Thermal conductivity of NIST SRM 1461 stainless steel (comparison between measurement results and the values from the certificate).

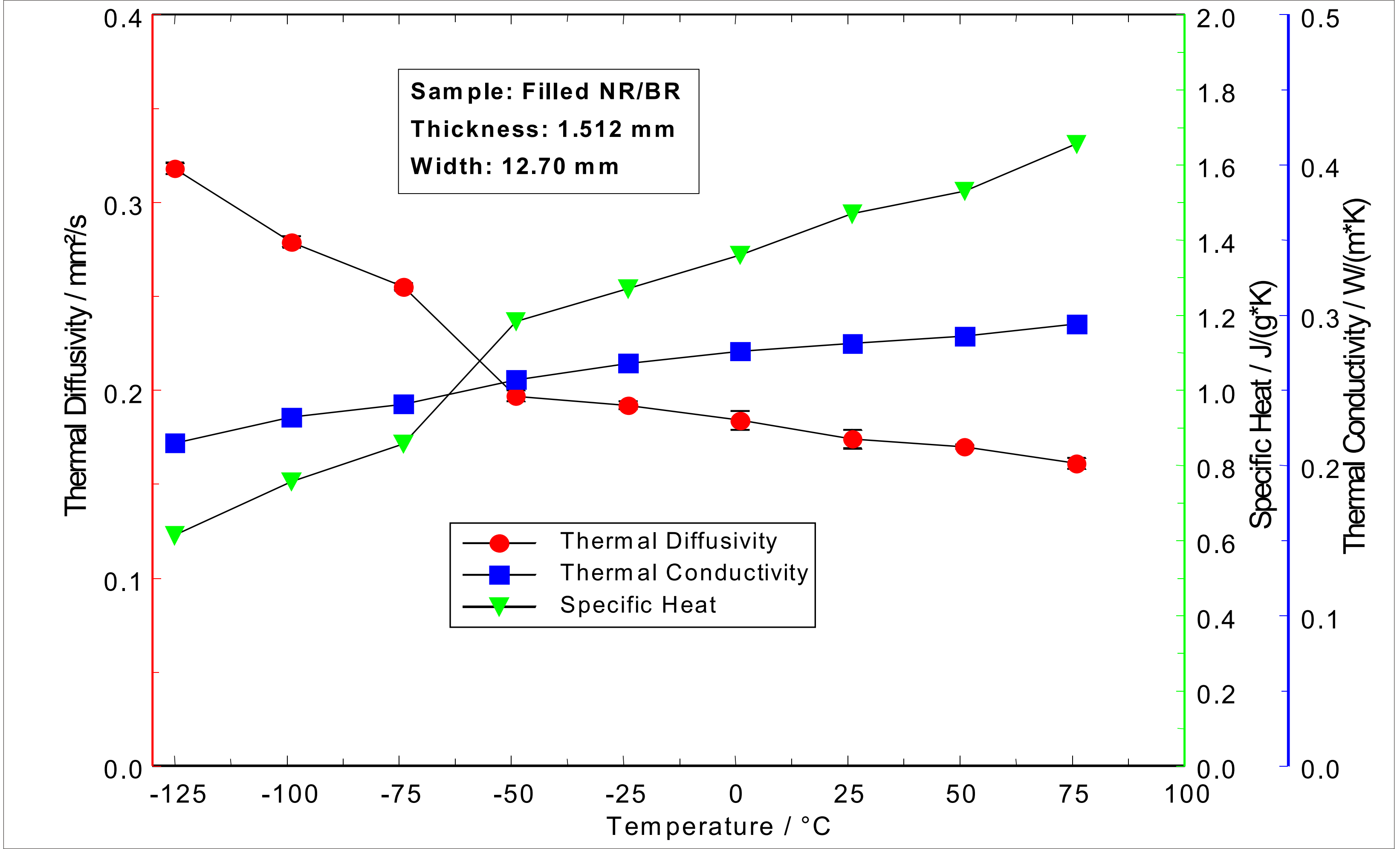

Figure 4. Specific heat, thermal diffusivity and thermal conductivity of an NR/BR rubber mixture between -125 and 75°C

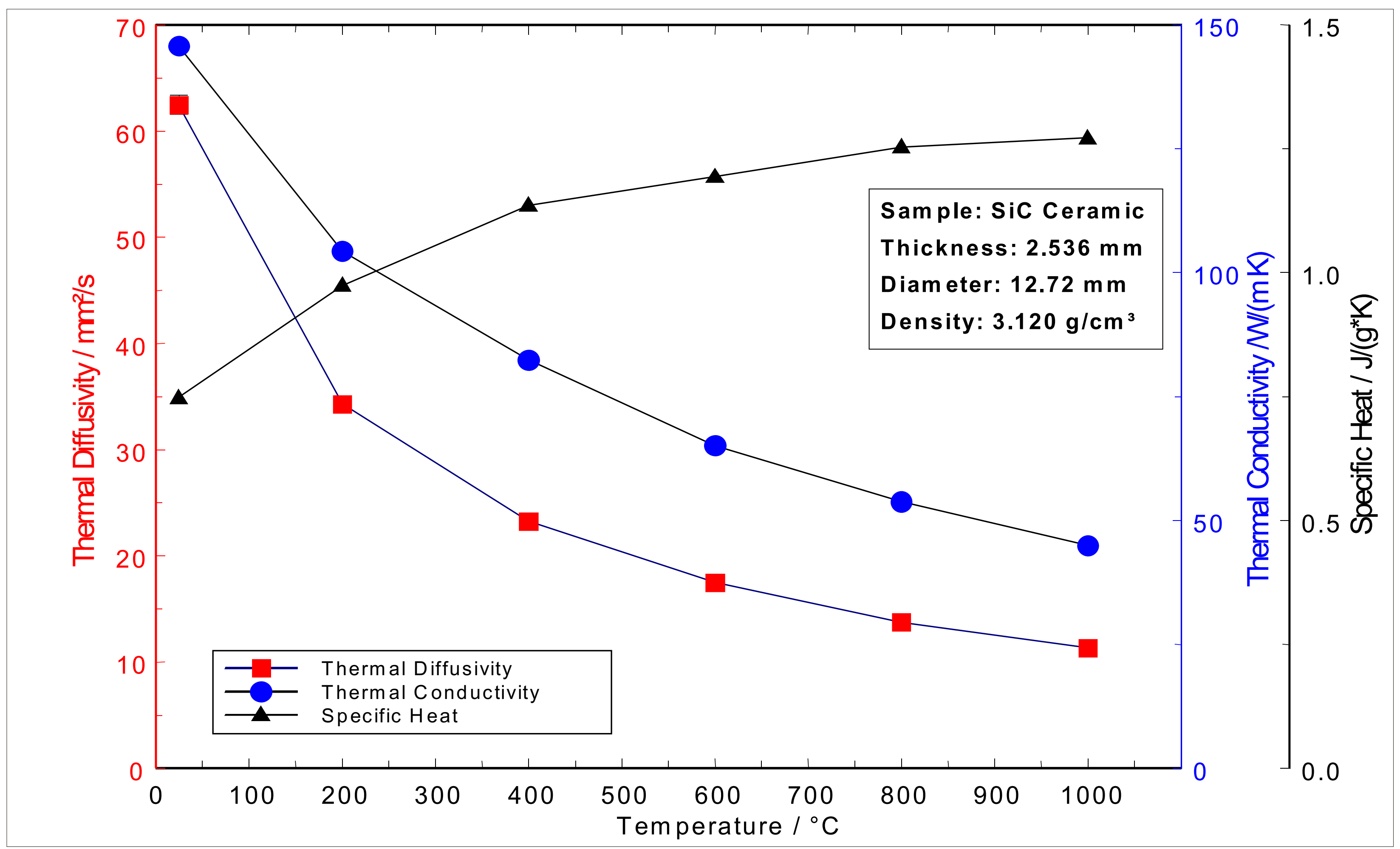

Figure 5. Specific heat, thermal diffusivity and thermal conductivity of a silicon carbide (SiC) ceramic between room temperature and 1000°C.

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

1. W. J. Parker, R. J. Jenkins, C. P. Butler, G. L. Abbott, 1961 *J. Appl. Phys.* ,Vol. 32, 1679-1684
2. R. D. Cowan, 1963 *J. Appl. Phys.*, Vol. 34, 926-929
3. T. Azumi, Y. Takahaski, 1981, *Rev. Sci. Instr.*, Vol. 52, 1411-1413
4. J. A. Cape, G. W. Lehman, 1963 *J. Appl. Phys.* ,Vol. 34, 1909-1913
5. J. Blumm, J. Opfermann, 2002 *High Temp.-High Press.*, Vol. 34, 515-5216.
6. Y. S. Touloukian, R. W. Powell, C. Y. Ho, M. C. Nicolaou, Thermophysical Properties of Matter, Vol. 10, Thermal Diffusivity, IFI Plenum, New York-Washington, 1973
7. J. G. Hust, A. B. Lankford, National Bureau of Standards, Certificate, Standard Reference Materials 1460, 1461 and 1462, 1984