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From Light Flash to Heat Transfer of Polymers

What is the magnitude of the thermal conductivity of a molten thermoplastic material in an injection mold? How efficient and direction-dependent is the thermal diffusivity of a fiber-reinforced thermoplastic? How can the heat transfer of thermosetting circuit boards in electronic components be optimized? What is the dependence of the thermal conductivity on the carbon black additive concentration in a rubber compound?

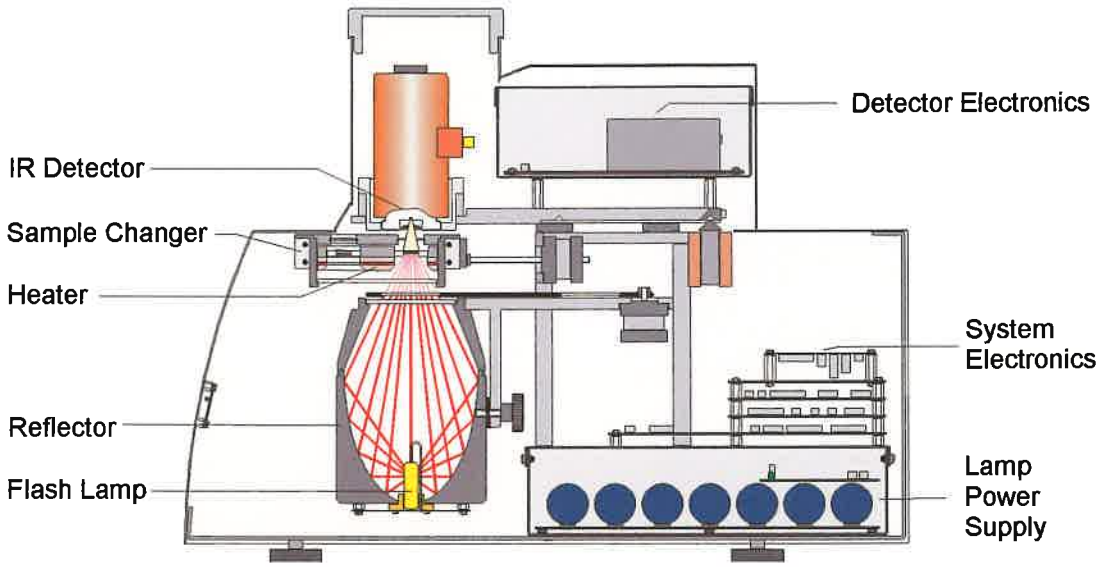

Fig. 1: Schematic design of the LFA 447 *Nanoflash*™

In order to answer questions like these, the thermophysical properties of the polymer must be known. Besides the melting, crystallization and reaction behavior, which are usually measured with a differential scanning calorimeter (DSC), the thermal diffusivity and thermal conductivity are of high practical interest.

Flash Method: fast, non-destructive and contact-free

The thermal diffusivity α can be measured with modern flash techniques. The front side of a plane-parallel sample is heated by a short light pulse. The resulting temperature rise on the rear surface is measured as a function of time [1]. If the specific heat c_p and density ρ are additionally known, the temperature-dependent thermal conductivity λ of the material can be calculated:

$$\lambda(T) = \alpha(T)\, c_p(T)\, \rho(T)$$

Presented in this work is the contact-free flash method using the new LFA 447 *Nanoflash*™ by NETZSCH-Gerätebau, Selb, Germany. Due to the temperature range, room temperature to 300°C, it is ideal for the polymer field, particularly for electronic packaging. With a suitable sample container, the thermal diffusivity can not only be measured on the solid polymer but also on the molten material.

This is, for example, important for injection molding, since modern 3-D simulation programs require reliable input parameters for the filling of the mold and for the temperature distribution in the mold.

Modern Flash Apparatus

The LFA 447 *Nanoflash*™ (fig. 1) [2] works according to national and international standards such as DIN EN 821, DIN 30905 or ASTM E-1461.
The light pulse is produced by means of a high-performance Xenon flash lamp placed within a parabolic mirror. A homogeneous illumination of the entire sample surface is achieved. Both the released energy of the flash lamp and the length of the heating pulse can be adjusted via the 32-bit MS® Windows™ software.
The flash lamp, sample and infrared detector are vertically arranged. The samples are located in an automatic sample changer, with which up to four samples can be measured in one measuring cycle. There are standard sample holders for testing round and square samples from 8 mm to 25.4 mm width. The furnace (RT to 300°C) is directly integrated into the sample changer, creating a small thermal mass and therefore fast heating and cooling times are possible. The sample thermocouple, which accurately measures the temperature, is positioned in the sample carrier. The measurement of the temperature rise, after the light pulse, is carried out with a liquid-nitrogen cooled InSb infrared detector. The non-contact measurement of the temperature rise guarantees an easy sample change and a short response time for the signal acquisition system
There are 15 different evaluation models, with and without correction, available to the user. These models can take into consideration heat losses from the side and from the front surfaces. Also, the analysis of 2- and 3-layer component systems, using non-linear regression, with or without pulse length correction, is possible.

The thermal diffusivity measuring range is 0.01 to 1000 mm²/s, with a reproducibility of approx. +/-3%.
In addition to the thermal diffusivity, by employing a comparative method, the specific heat can also be determined with the *Nanoflash*™. Pyroceram is often used as a calibration standard. For the specific heat, a reproducibility of +/-5% is achieved. If the bulk density is known, a direct determination of the thermal conductivity is possible. The thermal conductivity range is 0.1 to 2000 W/mK.

Heat transfer in the Semi-crystalline Thermoplastic Polypropylene (PP)

Figure 2 depicts the thermal diffusivity (red), thermal conductivity (blue) and specific heat (green) of PP as a function of temperature. From room temperature to the onset of melting at 150°C (extrapolated onset of the melting peak from a DSC measurement), the thermal conductivity decreases significantly from approx. 0.098 to 0.075 mm²/s. After melting, it reaches an almost constant value of 0.085 mm²/s at 250°C. As expected, the specific heat increases in two steps: from 1.5 J/gK at room temperature to 2.2 J/gK at 90°C and, during softening and melting, from 2.3 J/gK to 2.8 J/gK at 250°C. Prior to and after melting, the resulting thermal conductivity shows an increase from 0.14 W/mK to 0.22 W/mK (250°C).

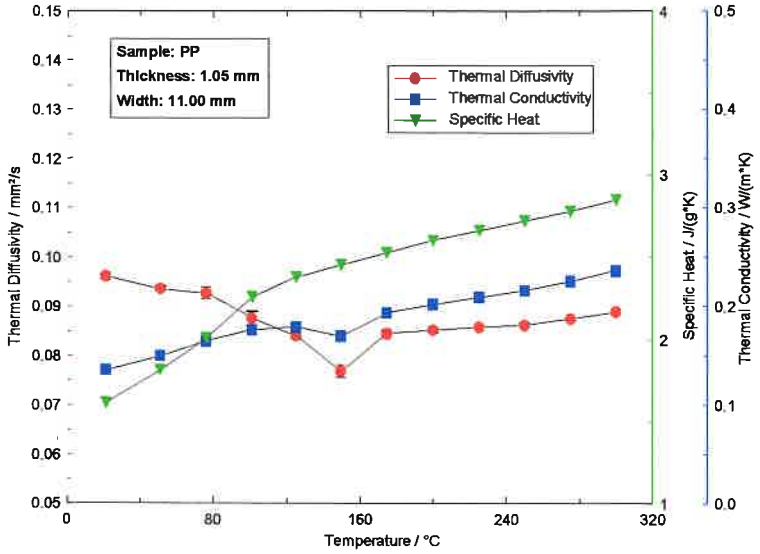

Fig. 2: Thermal diffusivity (red), thermal conductivity (blue) and specific heat (green) of polypropylene as a function of temperature

Direction-dependent thermal conductivity of copper fiber-reinforced PP

For fast heat removal in electronic components, the use of thermally conductive plastics for housings and chassis is becoming increasingly important. By employing special sample holders, the flash method can be used for direction-dependent analysis of the thermal diffusivity and conductivity of anistropic materials. As can be seen in the measurement example (figure 3), the thermal conductivity for a copper fiber-reinforced PP in the fiber direction (in-plane) at 200°C is higher by a factor of four compared to the thermal conductivity perpendicular to the fiber (normal).

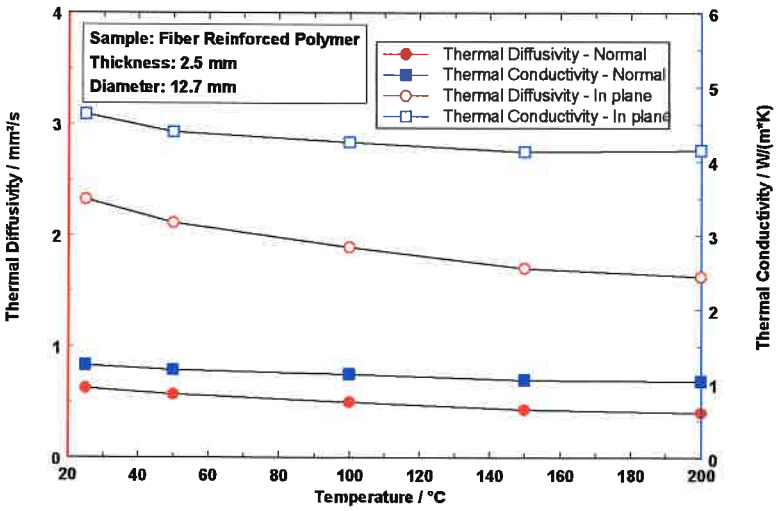

Fig. 3: Thermal diffusivity (red) and thermal conductivity (blue) of copper fiber-reinforced polypropylene as a function of temperature, perpendicular to the fiber direction (filled symbols, normal) and in the fiber direction (unfilled symbols, in-plane)

Influence of Carbon Black on the Thermal Diffusivity in a Rubber Compound

Finally, we would like to demonstrate that the flash method also offers a fast solution for the determination of the effect of the filler content on the thermal conductivity of a polymer. As an example, figure 4 shows the linear correlation of the thermal diffusivity, measured at room temperature, and the carbon black content of a rubber mixture.

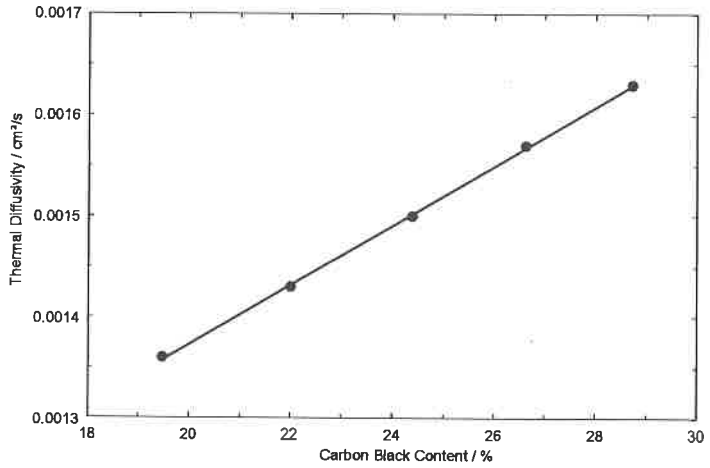

Fig. 4: Thermal diffusivity of carbon black filled rubber mixture at room temperature

Summary and Prospects

The LFA 447 *Nanoflash*™ has been designed as an effective, easy-to-use- and highly accurate system, particularly for the investigation of polymers and composites.

If a low-temperature range (e.g. for rubbers, elastomers) or for temperatures higher than 300°C (e.g. for high-temperature resistant composites), is of interest, the new LFA 457 *Microflash*™ would be the right choice. This instrument works with a laser system and two furnaces: –125°C to 500°C and room temperature to 1100°C.

Literature:

[1] Blumm, J.: Methoden zur Ermittlung der Wärmeleitfähigkeit; in: LaborPraxis, November 2002, S. 66-69.
[2] NETZSCH Instruments Inc., Burlington, MA, USA: Instruction Manual LFA 447 *Nanoflash*™, 7/2003

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