

# **Thermal Diffusivity, Specific Heat, and Thermal Conductivity Measurement Using NETZSCH LFA 447 Nanoflash™**

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Thermal diffusivity, specific heat, and thermal conductivity samples can be measured by the flash method utilizing a NETZSCH LFA 447 Nanoflash™ instrument. This instrument and method conform to ASTM E1461-07, “Standard Test Method for Thermal Diffusivity by the Flash Method”.

## Thermal Diffusivity

Transient heat transfer problems occur when the temperature distribution changes with time. The fundamental quantity that enters heat transfer situations not at steady-state is the thermal diffusivity. It is related to the steady-state thermal conductivity through the equation

$$D = \frac{\lambda}{c_p \rho} \quad (1)$$

where

D = thermal diffusivity

$\lambda$  = thermal conductivity

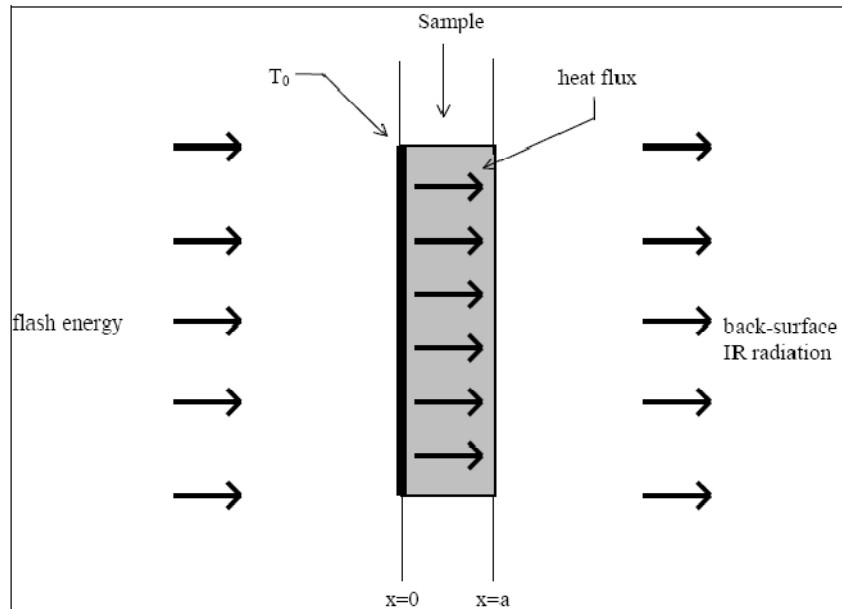
C<sub>p</sub> = specific heat

$\rho$  = density

Thermal properties of materials are measured by experimentally establishing a heat flow boundary value problem, solving the theoretical equations, and then measuring the necessary temperatures or heat fluxes to determine the thermal property by matching to the theoretical solution. Thus, the easiest theoretical way to measure the thermal conductivity is to set up a steady-state, linear flow of heat through the material and apply Fourier's equation. This approach has led to the development of a number of methods for measuring the thermal conductivity including the guarded hot plate and linear rod methods. These methods are time consuming and can be susceptible to errors arising from non-realization of the assumed boundary or steady-state conditions. The flash methods of measuring thermal diffusivity remove the steady-state condition at the expense of measuring temperature as a varying function of time.

The measurement of the thermal diffusivity of a material is usually carried out by rapidly heating one side of a sample and measuring the temperature rise curve on the opposite side. The time that it takes for the heat to travel through the sample and cause the temperature to rise on the rear face can be used to measure

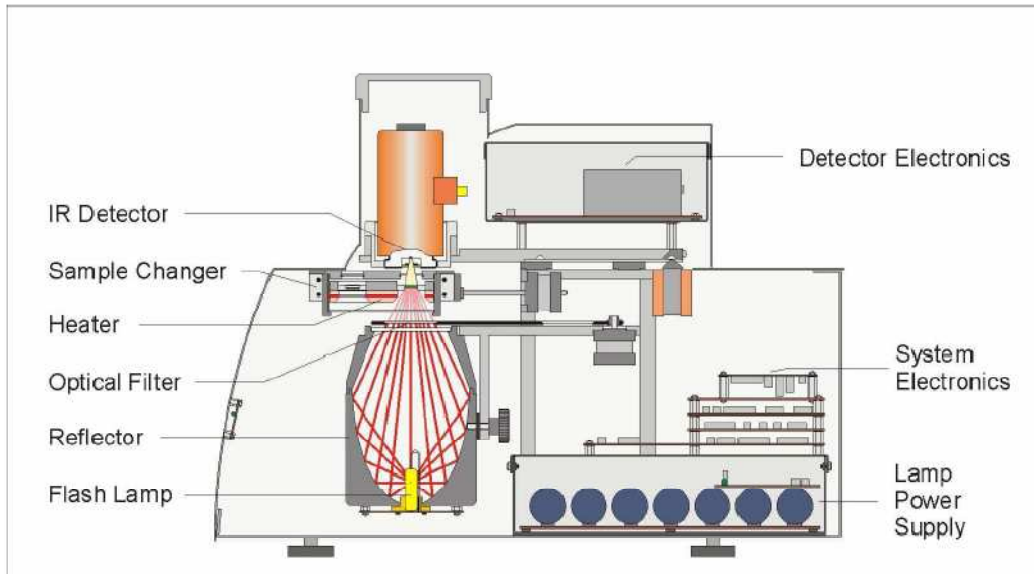
the through-plane diffusivity and calculate the through-plane thermal conductivity if the specific heat and density are known. The through-plane measurement is depicted in Figure 1.



**Figure 1**

### Through-Plane Method and Analysis

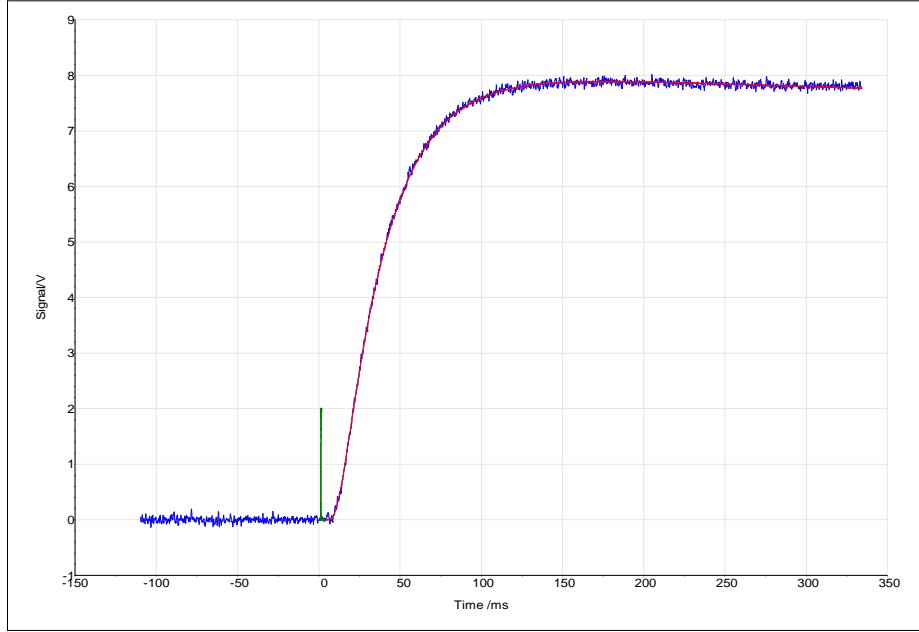
The basic method is sketched in Figure 2. The sample is normally a disk with a standard diameter of 12.7 mm and a thickness ranging from about 0.1 to 3 mm, although disks and square samples of various sizes can also be accommodated. With the NETZSCH LFA 447 Nanoflash™ system, the sample is aligned between a Xenon Flash Tube/Reflector assembly (wavelength: broadband, visible to near IR, approximately 170, 280 and 500  $\mu$ s pulse widths) and an indium antimonide (InSb) IR detector in a furnace. A Type K thermocouple in contact with the sample tray controls the sample and its surroundings at any temperature between 20 and 300°C. Once the sample has been stabilized at the desired temperature, the flash lamp is fired several times over a span of a few minutes and the necessary data is recorded for each “shot”. The flash energy strikes and is absorbed by the front surface of the sample, causing a heat pulse to travel through the samples’ thickness. The resulting sample temperature rise is fairly small, ranging from about 0.5 to 2 degrees C. This temperature rise is kept in the optimum range by adjustable filters between the flash lamp and the furnace. The sample lies in the field of view of the IR detector and the temperature rise signal vs. time is amplified and recorded with a high speed A/D converter. Figure 3 is an example of an actual temperature rise curve.



**Figure 2**

The instrument is fully automated to control all systems and record, analyze and report the thermal diffusivity, specific heat and the calculated thermal conductivity.

Samples that do not naturally have a high value of emissivity or absorptivity are coated with a graphite film before testing. The graphite increases the energy absorbed on the flashed side and increases the temperature signal on the back side of the sample. Some materials are transparent to infrared or visible radiation and can be coated with a metal film approximately  $1000\text{\AA}$  on both sides. This prevents penetration of the flash energy into the sample on the front side, and on the back side it prevents the viewing of the IR detector into the sample.



**Figure 3**

In the through-plane measurement depicted in Figure 1, the sample can be thought of as part of a sheet of material infinite in two dimensions but of finite thickness. Before the flash, the temperature of the sample and surroundings is at some uniform temperature that can be taken as zero. Immediately after the flash, the front surface of the sample is at some higher temperature,  $T_0$ . The heat flux lines are parallel and directed through the sample; there is no heat flow in the plane of the sample. The boundary conditions consist of radiation heat transfer from the front and rear faces into a surrounding space at the initial zero temperature. There have been many solutions of this boundary/initial value heat equation problem in different forms. The first to apply the solution to the flash diffusivity measurement was Parker et al.<sup>1</sup> Using the notation of Koski<sup>2</sup>, the resulting equation for the rear face temperature as a function of time is

$$T(a, t) = 2T_f \sum_{n=1}^{\infty} \frac{\gamma_n^2 (\gamma_n^2 + L^2) \cos \gamma_n}{(\gamma_n^2 - L^2)(\gamma_n^2 + L^2 + 2L)} \exp(-\gamma_n^2 Dt/a^2) \quad (2)$$

where

$T_f$  = final adiabatic sample temperature

$D$  = thermal diffusivity

$t$  = time

$a$  = sample thickness

$L$  = heat loss factor (Biot number)

Each  $\gamma_n$  is found by solving the transcendental equation

$$\tan \gamma_n = \frac{2\gamma_n L}{\gamma_n^2 - L^2} \quad (3)$$

The finite width of the flash energy pulse,  $t_p$ , can affect the measurement of thin and/or high diffusivity samples because the heating of the front surface can no longer be considered instantaneous relative to the time for the heat to diffuse through the sample. To include this effect in the analysis, the pulse width is folded into the results by convoluting Equation (2) with the pulse shape. If there is no heat loss, i.e.  $L=0$ , and the finite pulse can be ignored, then Equation (2) leads to the Parker expression

$$D = \frac{0.1388a^2}{t_{50}} \quad (4)$$

where  $t_{50}$ , the half rise time, is the time for the back face temperature to reach 50% of its maximum value. The constant value of approximately 0.139 is known as the Fourier number. Equation (4) is a very useful expression for calculating optimum sample thickness and expected rise times if the approximate diffusivity is known.

Equation (2) can be used to generate the temperature rise curve if the diffusivity and the loss factor are known. The analysis of the flash data requires the inverse of this operation; the temperature rise curve is known and the diffusivity is to be determined. Depending on the sample and measurement conditions, the measured temperature-versus-time curves of the detector are analyzed using the NETZSCH LFA Analysis software. The software has several different models that can be selected including combinations of facial and radial heat loss, finite pulse effect, semi-transparent materials, and two and three layer systems. The models are based on non-linear regression routines that take the entire measurement curve (normally 2000 points) into consideration by adjusting the unknown diffusivity and heat loss factor until the error between the theoretical and measured temperature rise curves is minimized.

### Specific Heat Capacity

The specific heat capacity (or specific heat) of a material is defined as the amount of energy required to raise a unit mass of material by one unit of temperature at constant pressure,

$$C_p = \frac{Q}{m\Delta T}$$

where:

$C_p$  = specific heat

$m$  = mass

$\Delta T$  = change in temperature

Q = energy

Specific heat can be measured with the flash method by comparing the temperature rise of the sample to the temperature rise of a reference sample of known specific heat tested under the same conditions<sup>1</sup>. This temperature (voltage) rise is recorded during the diffusivity measurement, so the specific heat can be calculated from the same data with a suitable calibration. Assuming that the flash energy and its coupling to the sample remain essentially unchanged between samples:

$$Q = \text{absorbed energy} = (mC_p\Delta T)_{ref} = (mC_p\Delta T)_{sample}$$

and:

$$C_{P_{sample}} = \frac{(mC_p\Delta T)_{ref}}{(m\Delta T)_{sample}} = \frac{(mC_p\Delta V)_{ref}G_{sample}}{(m\Delta V)_{sample}G_{ref}} \quad (5)$$

A reference sample is measured at each temperature of interest to calibrate the change in output voltage ( $\Delta V$ ) of the IR detector (or thermocouple for low temperature measurements) resulting from the absorbed energy. The measured  $\Delta V$  divided by the detector amplifier gain (G) will be proportional to the temperature rise ( $\Delta T$ ), as long as the temperature rise is small. This calibration gives the “absorbed energy” or a calibration factor in terms of the mass, specific heat,  $\Delta V$  and G for reference sample measurement. When a sample of unknown specific heat is measured, the “absorbed energy” divided by the product of the mass and  $\Delta V/G$  of the test sample, Equation 5, gives the test sample specific heat.

The measured  $\Delta V$  will be affected by heat loss during the measurement, so in order to use ratios of the measured  $\Delta V$  in Equation 5, the heat loss factor L (see Equation 2) of the reference and test sample should be similar. If the heat loss factors are significantly different, ratios of the theoretical  $\Delta V$  are used. The theoretical  $\Delta V$ , or final adiabatic sample temperature  $T_f$ , can be calculated from Equation 2 once the diffusivity and loss factor L have been determined as described above.

Testing has shown that the flash energy is stable from pulse to pulse within about  $\pm 2\%$ , so the average of several pulses at each temperature is used in the calculations. The absorptive efficiency of the front surface of the samples to the flash energy and the radiative efficiency of the back surface to the IR detector are controlled by coating the reference and test samples with the same thin layer of graphite.

### Thermal Conductivity and Thermal Resistance

The sample thermal conductivity can be calculated with Equation (1), with measurements of the diffusivity and specific heat as described above, and with a measurement of the sample bulk density. The bulk density is normally calculated from the measured sample volume (calculated from the measured sample dimensions) and mass.

The thermal resistance is defined as

$$R = \frac{a}{\lambda} \quad (6)$$

where :

R = thermal resistance

a = sample or layer thickness

## References

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