

Physics of Flash Techniques

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ABSTRACT: In 1960 Parker et al first time described the flash method to determine thermal diffusivity, heat capacity and thermal conductivity. In the meantime flash methods became acknowledged as excellent methods to measure thermal diffusivity. Specific heat preferably is measured with calo-

rimeters. During the last years flash methods increasingly became used for applications subjected to thermal conductivity measurement. This can be drawn back to their applicability in a wide temperature range (about -150°C up to 2000°C and more), to the low uncertainties of measurement results (~1% of the measured diffusivity), and to the

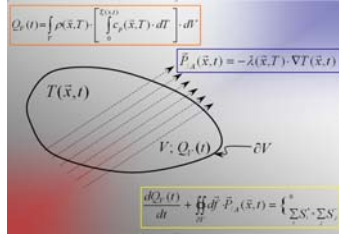
availability of mathematical tools to compute diffusivity from the detected temperature response of the specimen. Thermal conductivity can be calculated from thermal diffusivity, thermal density and heat capacity with the formula: $\lambda(T) = a(T) \times c_p(T) \times \rho(T)$. Compared to alternatives to measure thermal conductivity results from this method show

significantly lower uncertainties by a factor of ~2. Even anisotropic materials as well as partly inhomogeneous materials became measurable by flash methods. Last but not least time needs and costs for flash measurements are significantly lower than costs for most alternative methods for conductivity measurement. This poster introduces in

possibilities and limits of flash methods. More details particularly to the uncertainty of flash result in accordance to the recommendations of the GUM (guide to express uncertainty in measurement results: ENV 13005) are published in <http://phox.at/Download.php>.

PHENOMENOLOGY:

Theoretical considerations to flash techniques start with a heat balance of an arbitrary body and a deduction of the thermal conductivity equation from this. The situation of spatial heat propagation in an inhomogeneous heated area is illustrated on the right. The material properties needed to describe the physical situation are the thermal conductivity λ specific heat c_p , and the density ρ . One has to notice that these properties are dependent on the local composition of a material (thus on the spatial position x) and from the local temperature at the time of observation $T(x,t)$. The heat content $Q_V(t)$ of a volume V is calculated as shown. In case of absence of any thermal source S' or sink S'' any change of the heat content of this volume balances equal to θ with the integral heat flux $P_{A,i}(x,t)$ through the surface of this volume.



$$T(x,0) = f(x) = \sum_{n=0}^{\infty} V_n \cdot \cos\left(\frac{n \cdot \pi \cdot x}{h}\right); \quad T(x,0) = T_0 + \frac{P_A}{\rho \cdot c_p \cdot g} \cdot [1]_g$$

$$\int_0^h \cos\left(\frac{n \cdot \pi \cdot x}{h}\right) \cdot \cos\left(\frac{m \cdot \pi \cdot x}{h}\right) \cdot dx = \begin{cases} \frac{h}{2} \cdot \delta_{nm}; & \forall m; n \neq 0 \\ h; & \forall m = n = 0 \end{cases}$$

In this layer an initial temperature increase ΔT_i occurs. The mathematical description is done with the step-function $[1 - \Theta(g-x)]$. Consider that $\Theta(g-x) \equiv 0 \forall x < g$ and is not defined $\forall x < 0$ from physical reasons! After a sufficiently long time an average temperature increase over the whole sample ΔT_{∞} achieves.

$$V_0 \cdot h = \int_0^h T_0 \cdot dx + \int_0^h \frac{P_A}{\rho \cdot c_p \cdot g} \cdot [1]_g \cdot dx \quad V_n \cdot \frac{h}{2} = \int_0^h T_0 \cdot \cos\left(\frac{n \cdot \pi \cdot x}{h}\right) dx + \int_0^h \frac{P_A}{\rho \cdot c_p \cdot g} \cdot [1]_g \cdot \cos\left(\frac{n \cdot \pi \cdot x}{h}\right) \cdot dx$$

$$V_0 = T_0 + \frac{P_A}{\rho \cdot c_p \cdot h} \quad V_n = \frac{2 \cdot P_A}{\rho \cdot c_p \cdot h} \cdot \frac{\sin\left(\frac{n \cdot \pi \cdot g}{h}\right)}{\frac{n \cdot \pi \cdot g}{h}} = \frac{2 \cdot P_A}{\rho \cdot c_p \cdot h} \quad \forall g \ll h$$

$$V_n = \frac{2 \cdot P_A}{\rho \cdot c_p \cdot h} \quad \forall g \ll h$$

BASIC ASSUMPTIONS:

The local heat flux is determined by the local gradient of the temperature field $\nabla T(x,t)$ and the thermal conductivity of the medium filling this area (Fourier's law). The balance equation is given in the figure too. Under the assumption:

1. of a conservative system
2. of homogeneity of the thermally conducting medium (no spatial dependence of the material properties: thermal conductivity λ , specific heat c_p , and the density ρ)
3. and of the neglect of any non linearity

the balance equation leads to Fourier's thermal conductivity equation. This equation defines the interrelationship between the four thermo physical basic properties: $\lambda(T)$, $c_p(T)$, $\rho(T)$ and the thermal diffusivity $a(T)$. In general the thermal diffusivity $a(T)$ defines the so called transport coefficient of the thermal conductivity problem. Its meaning is conforming to the interpretation of the diffusivity coefficient of a diffusion problem.

$$\frac{dQ_V(t)}{dt} = \frac{\partial}{\partial t} \left[\int_V dV \cdot \rho(\vec{x}, T) \cdot \int_{c_p} \tau_{i,j}(\vec{x}, T) \cdot dT \right]$$

$$\frac{dQ_V(t)}{dt} = \int_V dV \cdot \rho(\vec{x}, T) \cdot c_p(\vec{x}, T) \cdot \frac{\partial T(\vec{x}, t)}{\partial t}$$

$$\int_V dV \cdot \rho(\vec{x}, T) \cdot c_p(\vec{x}, T) \cdot \frac{\partial T(\vec{x}, t)}{\partial t} = \int_V dV \cdot \lambda(\vec{x}, T) \cdot \nabla T(\vec{x}, t) \cdot \vec{df}$$

Conservative system
Linear description
Material:
Homogeneous
Isotropic

$$\int_V dV \cdot \rho(\vec{x}, T) \cdot c_p(\vec{x}, T) \cdot \frac{\partial T(\vec{x}, t)}{\partial t} = \int_V dV \cdot \lambda(\vec{x}, T) \cdot \nabla T(\vec{x}, t) \cdot \vec{df}$$

↓ Gauß Integral Law

$$\frac{\partial T(\vec{x}, t)}{\partial t} = \left[\frac{\lambda(T)}{\rho(T) \cdot c_p(T)} \right] \cdot \Delta T(\vec{x}, t) = a(T) \cdot \Delta T(\vec{x}, t)$$

↓ D'

$$\frac{\partial T(x, t)}{\partial t} = \left[\frac{\lambda(T)}{\rho(T) \cdot c_p(T)} \right] \cdot \Delta T(x, t) = a(T) \cdot \frac{\partial^2 T(x, t)}{\partial x^2}$$

$$T(x, t) = X(x) \cdot \Theta(t) \rightarrow X(x) \cdot \frac{d\Theta(t)}{dt} = a(T) \cdot \Theta(t) \cdot \frac{d^2 X(x)}{dx^2}$$

$$T(x, t) = T_0 + \Delta T_{\infty} \cdot \left[1 + 2 \cdot \sum_{n=1}^{\infty} \cos\left(\frac{n \cdot \pi \cdot x}{h}\right) \cdot e^{-\frac{a \cdot \pi^2 \cdot n^2 \cdot t}{h^2}} \right]$$

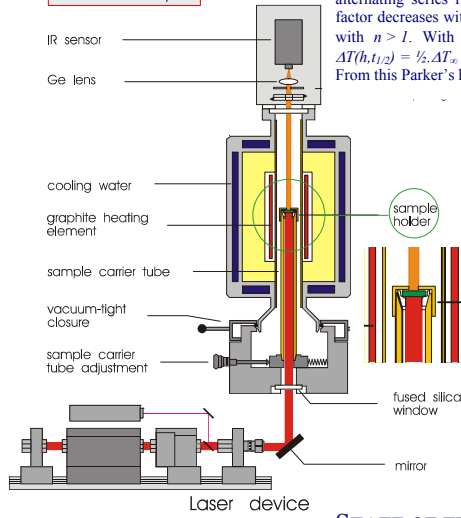
$$\Delta T(x, t) = \Delta T_{\infty} \cdot \left[1 + 2 \cdot \sum_{n=1}^{\infty} \cos\left(\frac{n \cdot \pi \cdot x}{h}\right) \cdot e^{-\frac{a \cdot \pi^2 \cdot n^2 \cdot t}{h^2}} \right]$$

$$\Delta T(h, t) = \Delta T_{\infty} \cdot \left[1 + 2 \cdot \sum_{n=1}^{\infty} (-1)^n \cdot e^{-\frac{a \cdot \pi^2 \cdot n^2 \cdot t}{h^2}} \right]$$

$$\frac{\Delta T(h, t_{1/2})}{\Delta T_{\infty}} \stackrel{!}{=} \frac{1}{2} = 1 - 2 \cdot e^{-\frac{a \cdot \pi^2 \cdot n^2 \cdot t}{h^2}}$$

$$a(T) = -\frac{\ln(1/4) \cdot h^2(T)}{\pi^2 \cdot t_{1/2}(T)}$$

Notice that $T(x,0)$ is the sum of the initial temperature T_0 and the temperature increase ΔT_{∞} . To calculate $\{V_n\}$ Fourier's serial expansion of $T(x,0)$ is used. Distinguishing $n=0$ from $n \neq 0$ gives V_0 and V_n . In case of the calculation of $\{V_n\}$ the approximation $[\sin(\xi)/\xi] \approx 1$ (possible for small arguments) is done. This requires that the thickness h of the thermal impact layer is significantly smaller than the thickness h of the specimen. This is fulfilled when the duration of the heat impact is significantly shorter than the time till a temperature increase at the top side of the sample occurs. With the Fourier-Coefficients the solution of the flash relevant thermal conductivity problem can be formulated by means of both $T(x,t)$ and $\Delta T(x,t)$. At the top side of the specimen ($x=h$) a simple alternating series results. Consider that the exponential factor decreases with n^2 . Thus Parker neglected all terms with $n > 1$. With $\Delta T(h, t_{1/2}) = \Delta T_{\infty}$ and the definition $\Delta T(h, t_{1/2}) = 1/2 \cdot \Delta T_{\infty}$ a simple analytical expression results. From this Parker's half-time-formula arises.



EXPERIMENTAL:

A scheme of a Laser Flash is shown on the left. Specimens are embedded under thermally insulated conditions. Nevertheless non negligible thermal interactions with the sample holder and the gas background occur. The laser pulse disrupts these conditions insulated conditions as well. The measurement starts at the required temperature under quasi-isothermal conditions. But after the heat impact no adiabatic conditions can be realised. Thus the IR-sensor detects a transient $T(t)$ -curve (left side) which captures the response of the material to the heat impact and its thermal interaction with the environment as well.

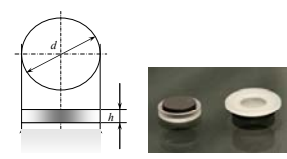
THEORY OF FLASH TECHNIQUES:

To be able to describe the basics of flash techniques analytically, simple experimental set-ups are realised. Aim is to reduce the complexity of the experimental situation to a transient but one dimensional phenomenon. Therefore disk-like samples (e.g. like a thin coin) are used. This leads to the differential equation for $T(x,t)$ as formulated on the right side. $T(x,t)$ can be separated in a product $T(x,t) = \Theta(t) \cdot X(x)$. Algebraic transformation leads to two ordinary differential equations and to the separation constant C . From the second law of thermodynamics C has to be set negative (consider that this is the only way to avoid an infinite temperature increase). By definition $c := -C$ is set. For the time-dependent equation this leads to a negative eigenvalue and to a monotonically decreasing solution $\Theta(t)$. For the spatial equation this leads to two complex eigenvalues and to two oscillating eigenfunctions summarised to $X(x)$. From theory the general solution $T(x,t) = \Theta(t) \cdot X(x)$ contains three constants U , V and c . They become specified by two boundary conditions and one initial condition.

$$\frac{1}{\Theta(t)} \cdot \frac{d\Theta(t)}{dt} = a(T) \cdot \frac{1}{X(x)} \cdot \frac{d^2 X(x)}{dx^2} = C$$

$$\frac{d\Theta(t)}{dt} - C \cdot \Theta(t) = 0; \quad \frac{d^2 X(x)}{dx^2} - \frac{C}{a(T)} \cdot X(x) = 0$$

$$T(x, t) = e^{-ct} \cdot \left[U \cdot \sin\left[\frac{c}{a} \cdot x + V \cdot \cos\left[\frac{c}{a} \cdot x\right]\right] \right]$$



Boundary conditions: In case of flash experiments adiabatic boundaries are applied. This means that an ideal thermal insulation of the considered volume is assumed. After an initial heat impact (e.g. laser pulse) no further heat exchange between the specimen and its thermal environment occurs. Thus any heat flux vanishes at the surfaces of the considered volume. As mentioned above this is formulated mathematically by a vanishing temperature gradient $\nabla T(x,t)$ at any surface. In case of a thin and flat body - as illustrated - one can neglect the barrel of the specimen and restrict the boundaries to the bottom and the top surface. Thus the sinusoidal part of the solution vanishes and the constant c degrades into an infinite sequence of numbers $\{c_n\}$ fulfilling the boundary condition of a vanishing heat flux at the top side of the specimen. Consequently the remaining constant V degrades in an infinite series $\{V_n\}$ corresponding to $\{c_n\}$. Thus the solution of the differential equation becomes an infinite series.

$$\left. \frac{\partial T(x,t)}{\partial x} \right|_x=0} = 0 \Rightarrow \frac{\partial T(x,t)}{\partial x} \Big|_{x=0} = \frac{\partial T(x,t)}{\partial x} \Big|_{x=h} = 0$$

$$\left. \frac{\partial T(x,t)}{\partial x} \right|_{x=0} = 0 \rightarrow U = 0$$

$$\left. \frac{\partial T(x,t)}{\partial x} \right|_{x=h} = 0 \rightarrow \sqrt{\frac{c}{a}} \cdot h = n \cdot \pi \rightarrow c_n = \frac{n^2 \cdot \pi^2 \cdot a}{h^2}$$

$$T(x, t) = \sum_{n=0}^{\infty} V_n \cdot \cos\left(\frac{n \cdot \pi \cdot x}{h}\right) \cdot e^{-\frac{a \cdot \pi^2 \cdot n^2 \cdot t}{h^2}}$$

Initial conditions: The series of $\{V_n\}$ will be specified by the initial condition. Mathematically the simplest initial condition is described by a Dirac delta-function $\delta(t - 0)$. Experimentally this requires an initial heat impact with an infinitesimal short duration at the bottom side of the specimen at $t = 0$. This heat impact is deposited in an infinitesimal thin layer with the cross section A of the specimen and the thickness g .

$$Q = \rho \cdot c_p \cdot [A \cdot g] \cdot \Delta T_i = P_A \cdot \delta(t - 0) \cdot A; \rightarrow \Delta T_i = \frac{P_A}{\rho \cdot c_p \cdot g}$$

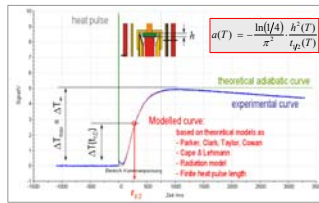
$$T(x,0) = T_0 + \Delta T_i = f(x)$$

$$T(x,0) = T_0 + \frac{P_A}{\rho \cdot c_p \cdot g} \cdot [1 - \Theta(g-x)]$$

$$[1 - \Theta(g-x)] = [1]_g$$

$$P_A \cdot A = Q = \rho \cdot [A \cdot h] \cdot c_p \cdot \Delta T_{\infty}$$

$$\Delta T_{\infty} = \frac{P_A}{\rho \cdot c_p \cdot h}$$



STATE OF THE ART:

Parker's theory was improved continually. But it is still a basis to estimate the *Equipment Specific Uncertainty (ESU)* and the standard uncertainty of the method. Therefore the recommendations of the GUM (guide to express uncertainty in measurement results: ENV 13005) are applied. Advanced techniques regard to specific heat transfer mechanisms and materials. As a result voluminous samples as well as foils, insulating and highly conducting materials, but also pastes, foams, glasses and even melts became measurable. Thermal diffusivities are measurable within a range of $\sim 0.5 \times 10^6 \text{ m}^2/\text{s}$ till $\sim 1000 \times 10^6 \text{ m}^2/\text{s}$. This is illustrated in the image downwards. For example diffusivity results from a reference graphite material are shown. The ESU is approximately 1% of the measured data. The standard uncertainty $u_c(a)$ could be estimated in a magnitude of $\sim 2.5\%$ of the diffusivity data (conf. Int. = 95%; coverage factor $k = 2$).

