

ANALYSIS OF FLASH METHOD PHYSICAL MODELS FOR THE MEASUREMENT OF THERMAL DIFFUSIVITY OF SOLID MATERIALS

Henrique Massard, Cláudio S. C. Pinto, Paulo Couto, Helcio R. B. Orlande and Renato M. Cotta

Federal University of Rio de Janeiro – UFRJ
Department of Mechanical Engineering – POLI/COPPE
P.O. Box: 68503, Cidade Universitária
Rio de Janeiro, RJ 21945-970, Brazil

Abstract. The flash method, proposed by Parker, Butler, Jenkins and Abbott from the U.S. Navy Radiological Defense Laboratory in 1961, is the most popular method for measuring the thermal diffusivity of solids. In this method, the front surface of a small sample is subjected to a very short burst of radiant thermal energy. The resulting temperature rise on the opposite surface of the sample is measured and the thermal diffusivity is computed from the temperature rise versus time data. Also, the specific heat can be computed from the measured data, thus allowing for the calculation of the thermal conductivity. Several theoretical models are available for the flash method, which include adiabatic boundary conditions, heat losses, surface coating effects, among other aspects. In this paper, some of these models are briefly discussed and compared, as applied to the identification of the thermal diffusivity of 1020 carbon steel. The Netzsch Nanoflash LFA 447/1 of LTTC/COPPE/UFRJ was used for the measurements.

Keywords. Flash Method, Physical Models, Thermal Diffusivity Measurement.

1. Introduction

In 1961, Parker *et al.* have published their pioneering work on the development of a method for the identification of the thermal diffusivity of solid materials. Although not specifically devised to, the proposed method also permits the identification of the specific heat of materials. In the method developed by Parker *et al.* (1961), a small and thin specimen is subjected to a high-intensity short-duration radiant energy pulse. The energy of the pulse is absorbed on the front surface of the specimen and the resulting rear surface temperature rise is recorded, as illustrated in Fig. (1). The thermal diffusivity value was calculated by Parker *et al.* (1961) from the specimen thickness and the time required for the rear surface temperature rise to reach 50% of its maximum value. Generally, the temperature rise in the specimen is small, so that the physical properties can be assumed as constant during the test. Therefore, if the thermal diffusivity of the specimen is to be determined over a temperature range, the test procedure must be repeated at each temperature of interest. Parker *et al.* (1961) named their test as the *Flash Method*. Such a method is particularly advantageous because of the simple specimen geometry, small specimen size requirements, rapidity of measurement and handling, with a single apparatus, of materials having a wide range of thermal diffusivity values over a large temperature range.

The Flash Method became an ASTM standard in 1992. The essential components of the apparatus are the flash source, sample holder, environmental enclosure (optional), temperature response detector, and recording device. The flash source may be a pulse laser, a flash or Xenon lamp, or other device capable to generate a short duration pulse of substantial energy. The duration of the energy flash should be less than 2 % of the time required for the rear face temperature rise to reach one half of its maximum value. The pulse hitting the specimen's surface must be spatially uniform in intensity. Most pulse lasers exhibit hot spots and a substantially higher intensity in the center region of the beam than in the periphery. For this reason, systems using unmodified beams directly from a pulse laser should use beams somewhat larger in diameter than the largest diameter of the samples to be tested (ASTM 1461-01, 2001). Most commonly used lasers are ruby (visible red), Nd:glass, and Nd:YAG (near infrared). Properly engineered Xenon flash sources, when properly focused, can provide a lower cost and lower maintenance alternative to lasers for many applications.

An environmental control chamber is required for measurements above and below room temperature. This chamber must be gas or vacuum tight if operation in protective atmosphere is desired. The enclosure shall be fitted with a window, which has to be transparent to the flash source. A second window is required if optical detection of the rear surface temperature rise is used. In such cases, it is recommended that the optical detector be shielded from direct exposure to

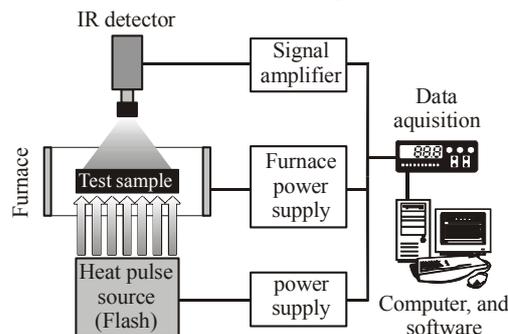


Figure 1. Flash method.

the energy beam with the use of appropriate filters. The furnace may be horizontal or vertical. The specimen support shall be loosely coupled thermally to the specimen. Specimen supports may be constructed to house one or several samples at a time, with the latter providing substantial improvements in data and testing speed (ASTM 1461-01, 2001).

The detector can be a thermocouple, infrared detector, optical pyrometer, or any other means that can provide a linear electrical output proportional to a small temperature rise. It shall be capable of detecting a 0.05 K change above the specimen's initial temperature. The detector and its associated amplifier must have a response time substantially smaller than 2 % of the half time value (ASTM E-1461-01, 2001). Nowadays, Flash method apparatus do not make use of thermocouples for the measurement of the rear surface temperature rise, because of their slow response, intrusive character and fin effect. Infrared detectors became popular as a reliable and accurate sensor for small temperature variations, within the time scale required for the Flash Method. The Flash Method is a primary method for the identification of thermal diffusivity by itself; hence, it requires no calibration. However, actual execution of the measurement is subject to random and systematic errors. It is therefore important to verify the performance of the apparatus in use, in order to establish the extent that these errors may affect the measurement data. This can be accomplished by testing one or several materials with well-known thermal diffusivity. So far, there are no Standard Reference Materials (SRM) available for thermal diffusivity. However, a large amount of data is available in the literature on a number of industry-accepted reference materials that have been used for verification purposes. They include the POCO Graphite and the Electrolytic Iron, which are used as reference materials for thermal conductivity (ASTM E-1461-01, 2001).

The Flash Method can also be used for the identification of the specific heat of the specimen, by comparing its temperature rise with that of a sample of a reference material (Parker *et al.*, 1961). Although this is a very attractive extension of the method, one must exercise extreme caution in performing it, as the opportunity for errors abounds (ASTM E-1461-01, 2001). In the course of an ordinary thermal diffusivity test, the amount of energy absorbed by the sample is important only to the extent that it will generate a sufficient rear surface signal (Parker *et al.*, 1961). On the other hand, the absorbed energy must be controllable, repeatable and accurately known for the identification of the specific heat. In this case, the temperature rise of the reference material is used to compute the absorbed energy. There are several conditions that must be satisfied for this process to be valid (ASTM E-1461-01, 2001), such as: (i) The energy source must be able to reproduce the pulse energy for fixed pulse settings; (ii) The detector must maintain its sensitivity during the reference sample and the specimen tests; (iii) The reference sample must be similar in size to the specimen; (iv) Both the specimen and the reference sample must be coated with a thin uniform graphite layer, in order to ensure that the emissivity of the two samples are the same; (v) Both the reference sample and the specimen must be homogeneous and isotropic; (vi) The reference sample and the specimen must be tested strictly at the same temperature and in the same environment. Preferably the test must be conducted with the reference sample and the specimen side-by-side and with very small time intervals between them; (vii) Verification of the process is to be done by using the same reference sample as unknown as was used for calibration; (viii) The same test must be performed with three different thicknesses in order to obtain a measure of the implied accuracy. After identifying the thermal diffusivity and the specific heat with the Flash Method, the thermal conductivity can be obtained from the definition of thermal diffusivity. In order to do so, the specimen's density can be calculated from its mass and volume measurements (ASTM E-1461-01, 2001).

In order to obtain the thermal diffusivity from the time required for the rear surface temperature to reach 50% of its maximum variation, Parker *et al.* (1961) used a one-dimensional heat conduction model, neglecting heat losses and assuming that the energy input was instantly deposited within a small depth of the specimen. In order to cope with experimental conditions where heat losses cannot be neglected and with a finite pulse duration, other authors proposed alternative mathematical models for the heat conduction problem in the specimen, such as those described in reference (Cowan, 1961 and 1963; Cape and Lehman, 1963; and Clark and Taylor, 1975). In fact, the finite pulse width effect occurs strongly when thin samples of high thermal diffusivity are tested, while heat losses become dominant at high temperatures when testing thick samples (ASTM E-1461-01, 2001). Recently, more involved models dealing with the coupled conduction-radiation heat transfer within the specimen were proposed for semi-transparent materials (Mehling *et al.*, 1998; and Lazard *et al.*, 2004). We note that other models can be found in the literature for the Flash method. However, the authors are of the opinion that those addressed here are sufficiently general to cope with the different factors affecting the temperature variation at the rear surface of the specimen.

The objective of this paper is to present a comparison of some of the mathematical models available for the identification of thermal diffusivity with the Flash Method, by using experimental data obtained for specimens made of 1020 carbon steel. The experimental data were obtained with the Flash Method apparatus Netzsch LFA-447 of the Laboratory of Heat Transmission and Technology of COPPE/UFRJ. We present below a discussion of the models available from the references cited above. The experimental apparatus is thus described and the results obtained with the different models available within the software accompanying the apparatus are compared.

2. Mathematical Models

2.1. The Model of Parker *et al.* (1961)

Parker *et al.* (1961) developed the earliest mathematical model for the identification of thermal diffusivity with the Flash Method. The authors assumed that the entire radiant energy was absorbed instantaneously (zero pulse width) in a very thin layer of the sample material facing the heat pulse source, and that the material properties were constant over

the small temperature step of the measurement. In addition, they assumed that heat conduction in the sample was one-dimensional and that heat losses could be neglected. Under these conditions, the temperature at the rear face ($x = L$) of the solid at any time can be written as follows:

$$T(L, t) = \frac{Q}{\rho C_p L} \left[1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp\left(-n^2 \pi^2 \frac{\alpha t}{L^2}\right) \right] \quad (1)$$

where Q is the heat flux resulting from the light pulse, while ρ , C_p and α are the density, the specific heat, and the thermal diffusivity of the solid, respectively. Equation (1) can be written in a dimensionless format, by using the following non-dimensional parameters:

$$\Theta = T(L, t) / T_m \quad (2)$$

$$\tau = \alpha t / L^2 \quad (3)$$

where T_m is the maximum temperature rise at the rear surface [$T_m = T(L, t \rightarrow \infty)$]. By substituting Eqs. (2) and (3) into Eq. (1), we obtain:

$$\Theta(\tau) = 1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp(-n^2 \pi^2 \tau) \quad (4)$$

Figure (2) shows the variation of the non-dimensional temperature Θ as a function of τ , as given by Eq. (4). According to Parker *et al.* (1961), the thermal diffusivity can be determined from Eq. (4) and Fig. (2), at the point where the non-dimensional temperature Θ is equal to 0,5. At this point, the non-dimensional time τ is equal to 0,1388, and from Eq. (4) it follows that:

$$\alpha = 0.1388 L^2 / t_{0,5} \quad (5)$$

where $t_{0,5}$ is the time required for the back surface temperature to reach half of the maximum temperature rise. Therefore, the thermal diffusivity α can be directly determined from Eq. (5) by measuring the time where the non-dimensional temperature Θ equals 0.5 ($t_{0,5}$). It is important to observe that it is not necessary to know the amount of energy absorbed in the front surface in order to determine the thermal diffusivity with this model, which depends only on the shape of the temperature vs. time curve [Fig. (2)] and the thickness of the solid.

Parker *et al.* (1961) compared the data obtained from Eq. (5) with other values available at the time, obtaining good agreement and maximum deviations of the order of $\pm 5\%$. However, this model is somewhat limited in applicability, because heat losses are neglected and an instantaneous pulse heating in the front surface of the solid is assumed. The finite pulse width effects are more pronounced during the test of thin samples of materials with high thermal diffusivity. The heat loss effects are more significant when testing thick samples at high temperatures.

2.2. The Model of Cowan (1961 and 1963)

The first paper dealing with heat losses in the Flash Method was actually published before the pioneering work of Parker *et al.* (1961). Cowan, in July 1961, published a paper proposing a new method (which he did not denote as the Flash Method) for the identification of thermal diffusivity. Such a method was essentially identical to the Flash Method proposed by Parker *et al.* (1961). However, it is worthwhile noticing that Cowan's paper (1961) was submitted for publication on January 03, 1961, while Parker *et al.* (1961) submitted their paper for publication on September 29, 1960. While Cowan's focus was on the mathematical model and the theoretical aspects for the identification of thermal diffusivity by using amplitude and/or phase measurements of the surface temperature variation (1961), Parker's focus was on the practical implementation of the Flash Method with a simple mathematical model that was appropriate for their experimental conditions (1961).

Cowan's model (1961) is one-dimensional, with instantaneous absorption of the energy from the source on one of the sample surfaces. Heat losses at both sample surfaces are temperature dependent, as a linearized radiation boundary condition. Cowan (1961) examined the energy beam either as a square wave or as sine wave.

Later in 1963, Cowan (1963) used the mathematical model derived in 1961 to address the heat loss effects on the Flash method. When heat is lost from the sample surface, the temperature vs. time curve reaches its maximum at a lower temperature when compared to the adiabatic case. After the peak, the solid temperature falls due to the cooling effect provided by the heat loss, as illustrated in Fig. (3). The expression for the rear surface temperature derived by Cowan (1963) is:

$$\Theta(\tau) = \frac{Q}{\rho C_p L} \left[\exp(-a\tau) + 2 \sum_{n=1}^{\infty} (-1)^n \exp(-n^2 \pi^2 \tau) \right] \quad (6)$$

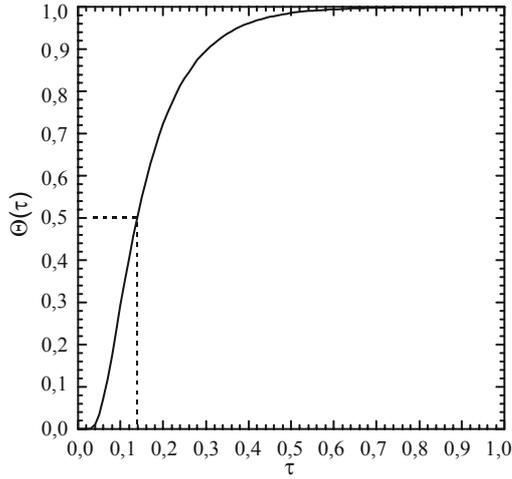


Figure 2. Thermal diffusivity measurement according to Parker *et al.* [2].

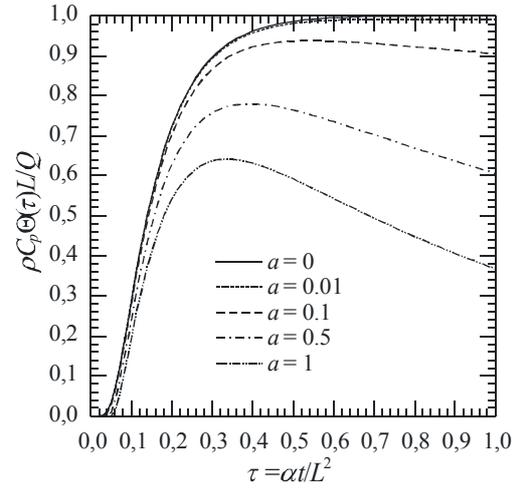


Figure 3. Heat losses effects on the rear surface temperature according to Cowan (1961 and 1963).

where τ is defined by Eq. (3) and $\Theta(\tau) = T(L, \tau) - T_0$. The non-dimensional parameter a in the first term on the right hand side of Eq. (6) accounts for the heat losses at the solid, and it is given by:

$$a = L \left[\frac{1}{k} \left(\frac{dW}{dT} \right)_{x=0} + \frac{1}{k} \left(\frac{dW}{dT} \right)_{x=L} \right] \quad (7)$$

where W is the is heat loss per unit of area (W/m^2) at the surfaces at $x = 0$ and $x = L$, and k is the thermal conductivity. It is clear that if there are no heat loss from the solid surfaces, $a = 0$, Eq. (6) reduces to Eq. (4) of Parker's (1961) model.

Cowan's model (1961 and 1963) requires the knowledge of the value of a at least approximately, for maximum accuracy. In the case of radiation losses only, a rough knowledge of the sample emissivity ε may give a with sufficient accuracy from:

$$a \approx 2.3 \times 10^5 \{ [1 + (T_L / T_0)^3] (T / 1000)^3 (\varepsilon L / k) \} \quad (8)$$

As Equation (6) correlates Θ and τ as a function of a , an alternative approach for the thermal diffusivity estimation is to experimentally measure the ratios:

$$\Theta(5t_{0.5}) / \Theta(t_{0.5}) \text{ or } \Theta(10t_{0.5}) / \Theta(t_{0.5}) \quad (9)$$

and then find the value of τ for a given heat loss estimation. When there is no heat loss from the sample ($a = 0$), the rear surface temperature remains constant after reaching its maximum and the ratio $\Theta(5t_{0.5}) / \Theta(t_{0.5})$ [or $\Theta(10t_{0.5}) / \Theta(t_{0.5})$] becomes 2. The non-dimensional time τ in Eq. (6) then becomes 0.1388 as in the Parker (1961) analysis.

2.3. The Model of Cape and Lehman (1963)

In 1963 Cape and Lehman (1963) presented a two-dimensional model for the Flash method, by assuming the sample with the form of a cylinder with radius r_0 and thickness L . Heat conduction through the sample was assumed as axially symmetric. The pulse energy absorbed by the sample, initially at the temperature T_0 , was assumed to be a source-term in the energy equation. The source-term was a function of the radial and longitudinal coordinates and of the time variable. Heat losses through the specimen surfaces were taken into account as a linearized radiation term.

Cape and Lehman's (1963) model is given by:

$$\frac{1}{\alpha} \frac{\partial \delta(r, x, t)}{\partial t} = \frac{\partial^2 \delta}{\partial r^2} + \frac{1}{r} \frac{\partial \delta}{\partial r} + \frac{\partial^2 \delta}{\partial x^2} + \frac{q(r, x, t)}{k} \quad \text{in } 0 < x < L, 0 < r < r_0, t > 0 \quad (10)$$

$$-\frac{\partial \delta}{\partial x} + v_x \delta = 0 \quad \text{at } x = 0, \text{ in } 0 < r < r_0, t > 0 \quad (11)$$

$$\frac{\partial \delta}{\partial x} + v_x \delta = 0 \quad \text{at } x = L, \text{ in } 0 < r < r_0, t > 0 \quad (12)$$

$$\frac{\partial \delta}{\partial r} + v_r \delta = 0 \quad \text{at } r = r_0, \text{ in } 0 < x < L, t > 0 \quad (13)$$

$$\delta = 0 \quad \text{for } t = 0, \text{ in } 0 < x < L, 0 < r < r_0 \quad (14)$$

where

$$\delta(r, x, t) = T(r, x, t) - T_0 \quad (15)$$

$$v_{x,r} = \frac{4\sigma\epsilon_{x,r}T_0^3}{k} \quad (16)$$

where, in Eq. (16), σ is the Stefan-Boltzman constant and ϵ is the surface emissivity.

Cape and Lehman (1963) solved Eqs. (10) to (14) analytically (Ozisik, 1993), by assuming that the pulse energy was uniform over the surface and that it was fully absorbed at the boundary $x = 0$. Cape and Lehman (1963) presented results obtained with the analytical solution for different levels of heat loss and for pulses in the form of a step function and of a triangular variation.

2.4. The Model of Clark and Taylor (1975)

Later in 1975, Clark and Taylor (1975) proposed a correction procedure for the identification of thermal diffusivity, based on Cape and Lehman's (1975) mathematical model. Instead of analyzing the cooling part of the temperature rise curve as Cowan (1961 and 1963), Clark and Taylor (1975) procedure was based on the analysis of the heating part of the curve (*i.e.*, before the back surface temperature reached its maximum). In accordance with the Clark and Taylor (1975), the previous correction procedures for radiation heat losses in the flash method required the accurate knowledge of the amount of heat lost or used the cooling part of the curve. A cooling curve correction scheme is difficult from the experimental point of view. Such is the case because a common occurrence in the flash method is the spatial non-uniformity of the laser beam. The non-uniform heating effects are more significant after the maximum temperature rise, when heat conduction within the sample becomes three-dimensional. The problem of losses through the sample holder is also more significant for times larger than that corresponding to the maximum temperature.

Clark and Taylor (1975) determined five points on the time scale corresponding to five different dimensionless temperatures Θ as measured on the temperature rise curve. These times were $t_{0.2}$, $t_{0.3}$, $t_{0.4}$, $t_{0.7}$ and $t_{0.8}$. They developed a theoretical relationship between the dimensionless time τ in Eq. (3) and the following three sets of time ratios:

$$\Theta(t_{0.7})/\Theta(t_{0.3}), \Theta(t_{0.8})/\Theta(t_{0.4}), \text{ and } \Theta(t_{0.8})/\Theta(t_{0.2}) \quad (17)$$

The three values resulting from these ratios are very close and are averaged to compute a more accurate value of thermal diffusivity.

2.5. The Model of Mehling *et al.* (1998)

With the development of commercial and research apparatuses based on the Flash Method, the original method that applied only to solid opaque materials was gradually extended to non-homogeneous and semi-transparent materials, as well as liquids (ASTM E-1461-01, 2001). Mehling *et al.* (1998) presented a mathematical model for semi-transparent, non-absorbing and non-scattering materials.

The measurement of the thermal diffusivity with the Flash Method generally requires an opaque graphite coating to absorb the flash pulse and to increase emission in the infrared range (for the measurement of the rear surface temperature variation with an infrared detector). For semi-transparent optically thin materials, the use of a highly reflecting coating below the graphite one permits the use of conventional methods for the identification of the thermal diffusivity, such as those described above (Mehling *et al.*, 1998). This reflecting coating is usually obtained by vapor deposition of gold, which is an expensive and time-consuming process (Mehling *et al.*, 1998). In order to avoid the use of the gold coating, the radiation heat transfer within the sample needs to be taken into account in the mathematical model used for the Flash method.

Mehling *et al.* (1998) proposed a model where radiation is applicable to a sample of a non-participating medium. In their model, in addition to heat conduction within the sample, a direct radiative heat transfer between the front and rear surfaces is taken into account in a one-dimensional formulation. Since the sample medium is considered as non-participating, the radiative heat transfer only depends on the boundary temperatures. The model proposed by Mehling *et al.* (1998) is given by:

$$\frac{1}{\alpha} \frac{\partial \delta(x,t)}{\partial t} = \frac{\partial^2 \delta}{\partial x^2} \quad \text{in } 0 < x < L, t > 0 \quad (18)$$

$$-\frac{\partial \delta}{\partial x} + v\delta(0,t) + v\eta[\delta(0,t) - \delta(L,t)] = 0 \quad \text{at } x = 0, t > 0 \quad (19)$$

$$\frac{\partial \delta}{\partial x} + v\delta(L,t) + v\eta[\delta(0,t) - \delta(L,t)] = 0 \quad \text{at } x = L, t > 0 \quad (20)$$

where δ and ν are defined by equations (15) and (16), respectively. The factor η appearing in equations (19) and (20) take into account the location and width of the totally transparent spectral regions.

Note that in the definition of Mehling's model (1998) the initial condition was not included. Mehling *et al.* (1998) do not present the initial condition used for their model, but we believe that it was the same used by Parker *et al.* (1961), which considers that the energy pulse is instantaneously absorbed in a thin layer below the irradiated surface.

Mehling *et al.* (1998) show that their model is capable of capturing the jump in the rear surface temperature, resultant from the radiative heat transfer between the sample surfaces.

2.5. The Model of Lazard *et al.* (2004)

In a recent paper, Lazard *et al.* (2004) presented a model for coupled conduction-radiation in a sample of an absorbing, emitting and anisotropically scattering medium. With such model, the greatest restriction of the medium be non-participating of Mehling's model (1998) was overcome.

Experimental results were presented by Lazard *et al.* (2004) for non-scattering materials, such as glass. The use of the proposed model also avoids the use of the expensive gold coating, but it requires the estimation of two additional parameters, as compared to the purely conductive model.

Before we present below the experimental apparatus used in this work, as well as the results obtained, one final word regarding the different models is in order. It is a very difficult task to choose the best model for the identification of thermal diffusivity, specially when we consider models/methods like those of Parker *et al.* (1961), Cowan (1961 and 1963) or Clark and Taylor (1975), which do not rely on the best agreement between the experimental results and those obtained with the estimated parameters, but only on the temperature experimental data at selected times. Generally, nowadays (see, for example, Lazard *et al.*, 2004) statistically based techniques (Beck and Arnold, 1977; Ozisik and Orlande, 2000) are used for the identification of the unknown parameters appearing in the mathematical model used for the experimental conditions under picture. With this approach, the analysis of the residuals (difference between measured and estimated temperatures), as well as of statistical parameters like the correlation coefficient, permit a qualitative/quantitative evaluation of the model used to correlate the experimental data. Generally, the model most appropriate for a specific experiment is the one which yields the smallest and non-correlated residuals, which in turn result in a large correlation coefficient (Beck and Arnold, 1977).

3. Experimental Apparatus and Analysis of Experimental Data

In this section, the characteristics of the equipment used for the identification of the thermophysical properties of 1020 carbon steel (the Netzsch Nanoflash LFA 447/1) is discussed. The LFA 447/1 is a tabletop instrument that works with a high power Xenon-Flash lamp in the temperature range of room temperature to 200°C, and it has an integrated sample changer for 4 samples. Figure (4) shows a schematic of the instrument systems. A high-power Xenon flash lamp is surrounded by a parabolic mirror (reflector). The Xenon flash lamp is able to supply a pulse energy up to 10 J (up to 5 J/cm²), selectable by the measurement software, in the wavelength range of 150 nm to 2000 nm. The software also allows the energy pulse length to be adjusted to 0.1, 0.2 or 0.4 ms.

The integrated sample changer allows up to 4 different sample positions. Each sample is moved automatically into the measurement position. The sample positioning is controlled by the measurement software. The integrated sample changer uses interchangeable sample holders for rounded (\varnothing 10 mm, \varnothing 12.7 mm and \varnothing 25.4 mm) or squared samples (10 mm x 10 mm and 8 mm x 8 mm). The sample changer is surrounded by glass windows for protection of the flash lamp and of the IR detector.

The LFA 447/1 uses an InSb-IR detector in the wavelength range of 2000 to 5000 nm cooled by liquid nitrogen, which allows a non-contact fast measurement of the temperature rise in the back of the sample. The close connection to the preamplifier system permits a fast data acquisition (500 kHz, 12 Bit) and a measurement of 2000 points per shot

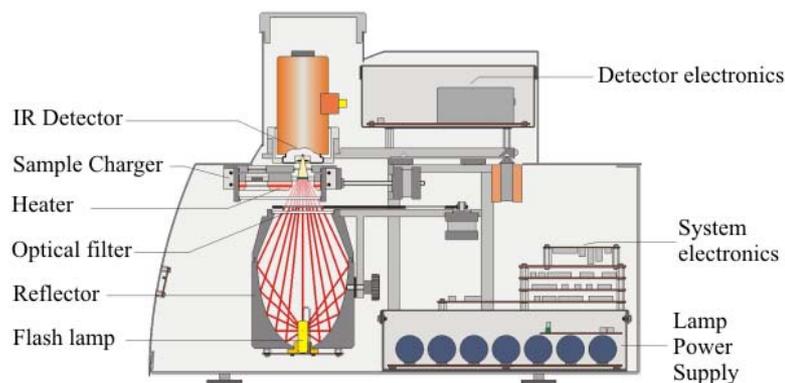


Figure 4. Schematics of the Netzsch Nanoflash LFA 447/1.

(detector signal). An integrated furnace (heater) maintains the sample temperature stable during measurement. The heater is integrated in the automatic sample changer, which has a low thermal mass allowing fast heating/cooling rates. Sample temperature measurement (thermocouple) is realized in the sample carrier. A Julabo Chiller is used to cool the surrounding components.

The LFA 447/1 is capable of thermal diffusivity measurements in the range of 0.01 mm²/s up to 1000 mm²/s, with an accuracy of 3-5% for most materials. The specific heat accuracy is 5-7%. This allows the calculation of the thermal conductivity in the range of 0.1 W/mK to 2000 W/mK with an accuracy of 3-7% for most materials.

The analysis of the experimental data is performed with a software called Proteus, provided by Netzsch, which includes different mathematical models, such as: Adiabatic (Parket *et al.*, 1961), Cape-Lehmann (Cape and Lehmann, 1963), Cowan (Cowan, 1961 and 1963), and Radiation (Mehling *et al.*, 1998).

One of the experimental effects that strongly influence the diffusivity identification is the finite pulse duration, as compared to the heat diffusion time. In reality, the laser beam has a short but finite pulse duration. The intensity distribution of the beam within the pulse duration is normally at a maximum. This pulse intensity function can influence the duration of the temperature distribution inside the test piece. If this pulse duration is long, in comparison to the heat transport time that is needed to reach 50% of the maximum temperature change at the back of the test piece, then this effect has to be taken into account when determining the thermal diffusivity. In Netzsch-LFA software, this pulse correction can be taken also into account according to Lechner and Hahne (1993). If the pulse duration is comparable to the time necessary for heating the back of the test piece, one has to carry out a correction of the finite pulse width. The mathematical model used in the Netzsch-LFA software is the following approximation of a laser-pulse (Lechner and Hahne, 1993):

$$pulse = \begin{cases} 0 & ; \text{for } t < delay \\ [1 - \exp(-t / \tau_1)] \exp(-t / \tau_2) & ; \text{for } delay \leq t \leq t_{end} \\ [1 - \exp(-t_{end} / \tau_1)] \exp(-t_{end} / \tau_2) \exp[-(t - t_{end}) / \tau_3] & ; \text{for } t_{end} < t \end{cases} \quad (21)$$

where τ_1 , τ_2 , and τ_3 , are time constants automatically determined to fit the pulse, and *delay* is the time delay between the beginning of the measurement and the beginning of the pulse shot, automatically determined by the software. The rise of the laser impulse is determined by the time constant τ_1 , the following slow decay by the time constant τ_2 . The parameter t_{end} determines the point in time of the fast decay, which takes place with the time constant τ_3 .

4. Results and Discussion

In this section the models used by the LFA 447/1 analysis software are applied to the identification of the thermo-physical properties of 1020 carbon steel.

Three rounded samples were manufactured for testing. The samples surfaces are flat and parallel to provide an accurate measurement for the sample thickness, and to improve the one-dimensional heat transfer characteristic for the tests. To determine the sample thickness, a micrometer was used, and measurements were performed to at least three significant figures, at four different points on the sample. The average value of the four measurements was considered as the thickness value for the tests.

Prior the measurements, the samples were coated with a graphite spray in order to enhance the absorption of the Xenon light pulse energy and the emission of IR radiation to the temperature detector. The graphite coating greatly increases the resulting signal-to-noise ratio when the surface is highly reflective, as with metals or ceramics with a metal film. The sample mass was measured with a precision scale. The bulk density of the sample was calculated by dividing the sample mass by the apparent sample volume calculated from the measured sample dimensions.

The 1020 carbon steel was tested against an alumina sample reference for the specific heat estimation. Both reference and test samples were coated side-by-side and at the same time to provide a similar coating thickness. Table 1 shows the dimensions and densities of the test and reference samples used in this investigation.

Table 1. Samples dimensions and densities.

Sample	Diameter [mm]	Thickness [mm]	Density [g/cm ³]
1020 carbon steel	12,650 ± 0,005	2,950 ± 0,005	7,85 ± 0,03
Alumina*	12,650	3,002	3,92

* Values provided by NETZSCH-Gerätebau GmbH

The alumina reference sample and 1020 carbon steel test samples were loaded into the sample holder and tested between 25° up to 200°C, with a 25°C temperature step. A total of eight measurements were performed at each temperature level in the test sample and in the reference sample during two successive days of experimental investigation.

Figures 5 and 6 show the results for the thermal diffusivity measurements of the 1020 carbon steel according to the four models available in the LFA 447/1 Nanoflash analysis software. Figure 5 presents the data for the different models without energy pulse correction, while Fig. 6 shows the data with pulse correction. In each figure the mean value of four

measurement data is shown for each temperature. The uncertainty (ε) bar is shown relative to the mean value at each temperature, and it was obtained for a 99% confidence interval ($\varepsilon = \pm 2,576 \sigma$). Table 2 summarizes the mean standard deviation (σ) obtained for each model available in the analysis software, with and without pulse correction. The standard deviation at each temperature was obtained from the following expression:

$$\sigma = \sqrt{\left[n \sum Y^2 - (\sum Y)^2 \right] / n^2} \tag{22}$$

where Y is the measured parameter and n is the total number of measurements at each temperature step.

The thermal diffusivity curves seem to exhibit the same behavior as a function of temperature for all the models. This is because the raw experimental data used to obtain the thermal diffusivity (IR sensor output signal) is the same for every model. The Mehling *et al.* (1998) model presents the largest measurement uncertainties, as it can be observed in Figs. 5 and 6. Also, from Tab. 2, not much difference was found on the results considering pulse correction and no pulse correction, but the Mehling *et al.* (1998) model presents the highest mean standard deviation. This is because this model was developed for semi-transparent, non-absorbing and non-scattering materials. The 1020 carbon steel is an opaque and highly absorbing material.

The optimum sample thickness (L) depends on the thermal diffusivity (α) of the material. In the analysis methods used by the LFA 447/1 Nanoflash, the time taken for the back surface to reach half its maximum temperature ($t_{0,5}$) should be at least three times longer than the length of the light pulse (100 μ s minimum) and not longer than about 3 seconds. The optimum $t_{0,5}$ is in the range of about 10-500msec. The Parker analysis gives $\alpha = 0,1388 L^2/t_{0,5}$ (see section

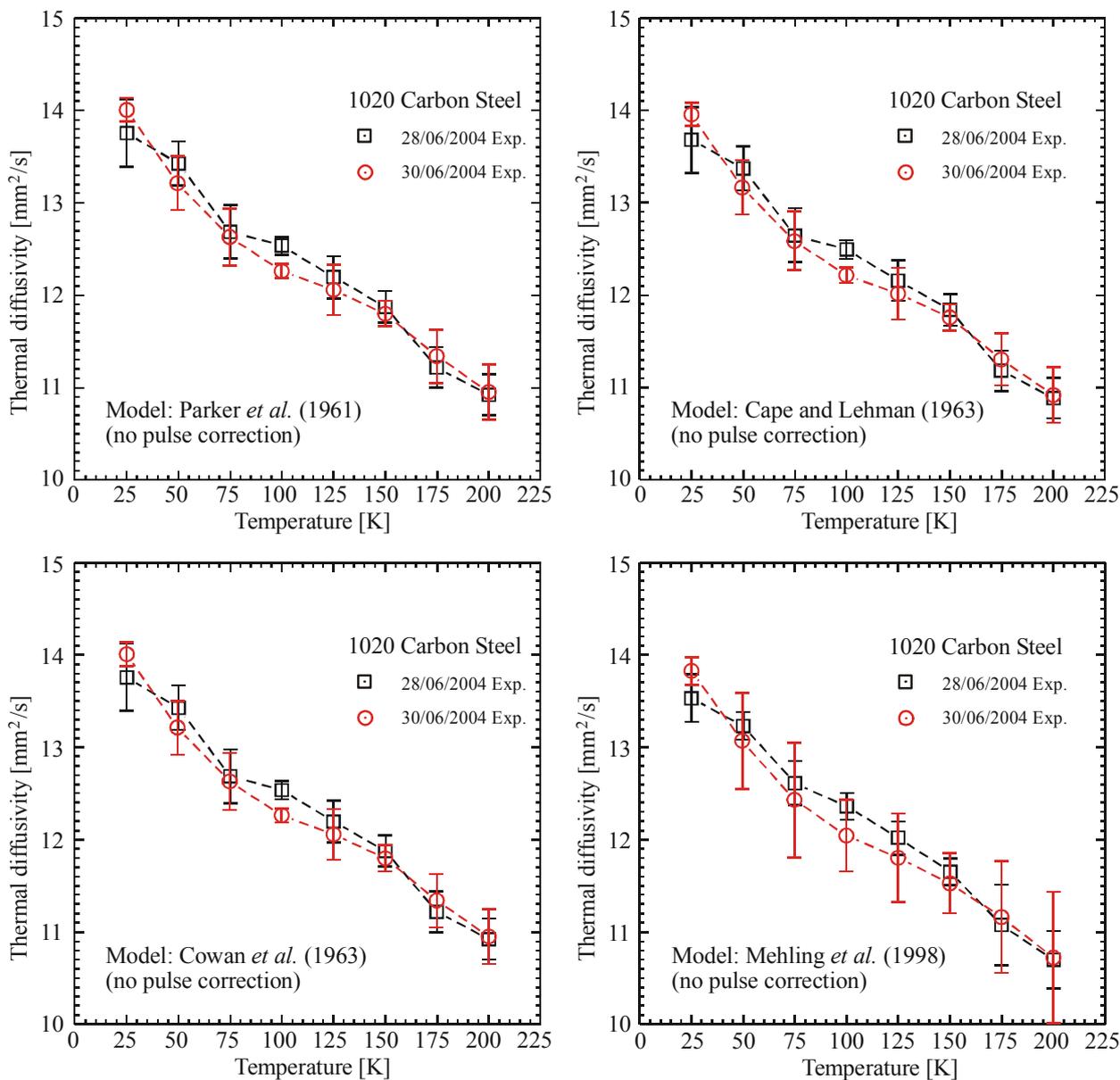


Figure 5. Thermal diffusivity experimental data for carbon steel (no energy pulse correction).

2.1). Therefore, combining the Parker analysis formula and the range values for $t_{0,s}$, the optimum value for the sample thickness ranges from $0,27\alpha^{1/2}$ to $1,90\alpha^{1/2}$. If the sample is too thick, it may be difficult to obtain a large enough signal on the temperature rise curve, even with amplifier gains at highest settings. From the thermal diffusivity data shown in Figs. 5 and 6, the optimum values for the sample thickness are in the range from 0,89 mm at higher temperatures to 7,01 mm at lower temperatures, confirming that the chosen sample test thickness (3 mm) was sufficiently enough to assure the accuracy of the analysis.

Table 3 shows a comparison between the experimental values obtained for the thermal diffusivity of 1020 carbon steel at 25°C and values listed by Callister, Jr. (2000). The identified values in this work are in excellent agreement with those presented in the literature.

Table 2. Thermal diffusivity standard deviations.

Model	σ (no pulse correction)	σ (with pulse correction)
Parker <i>et al.</i> (1961)	0,088 mm ² /s	0,088 mm ² /s
Cape and Lehman (1963)	0,088 mm ² /s	0,089 mm ² /s
Cowan <i>et al.</i> (1963)	0,088 mm ² /s	0,088 mm ² /s
Mehling <i>et al.</i> (1998)	0,137 mm ² /s	0,134 mm ² /s

5. Conclusions

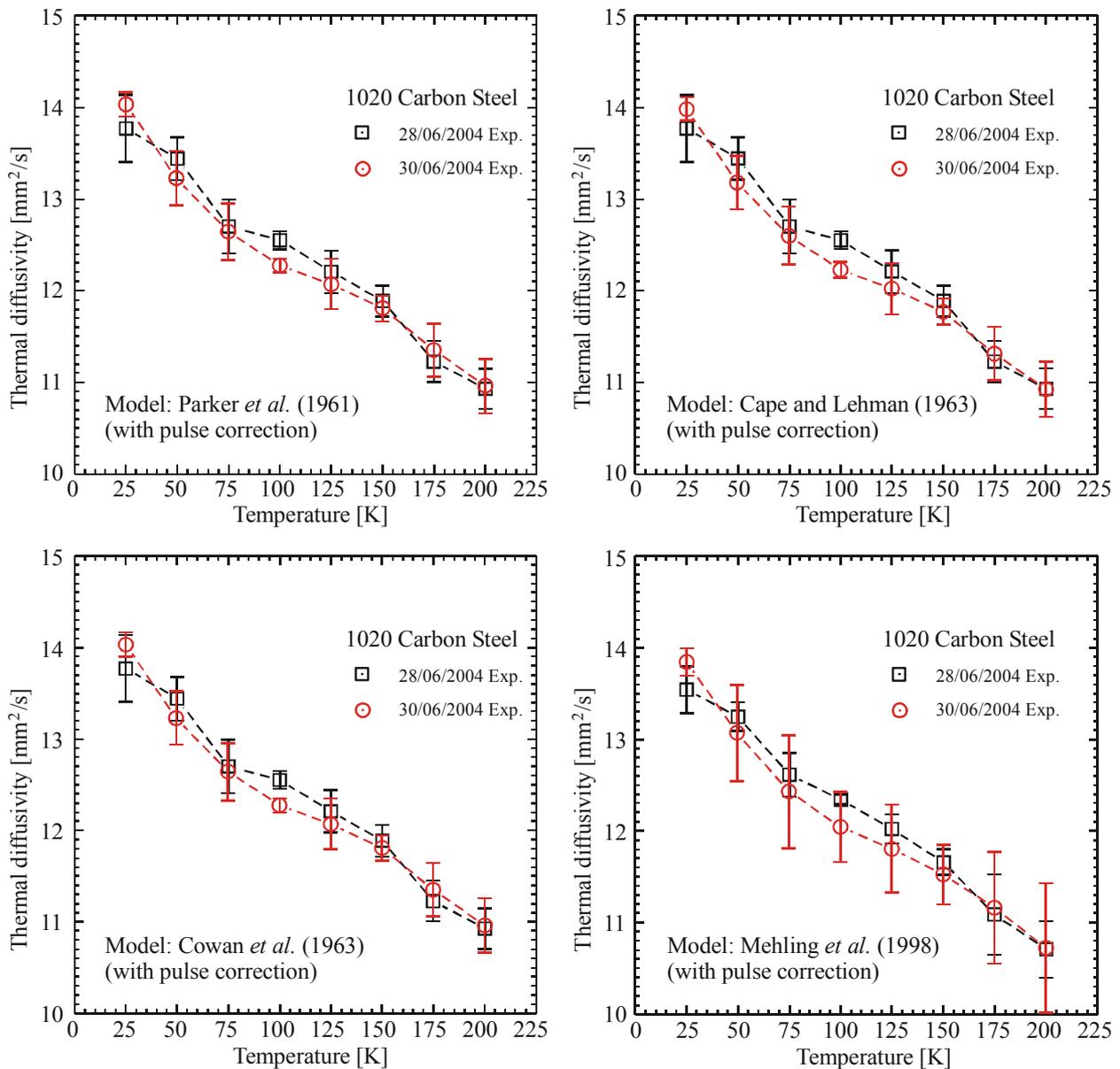


Figure 6. Thermal diffusivity experimental data for 1020 carbon steel (with energy pulse correction).

Table 3. Experimental data and literature values for thermal diffusivity at 25°C.

Models	Thermal diffusivity, α [mm ² /s]	
	Experimental	Literature*
Parker <i>et al.</i> (1961)	– pulse corr.	13,8 ± 0,4
	+ pulse corr.	13,8 ± 0,4
Cape and Lehman (1963)	– pulse corr.	13,7 ± 0,4
	+ pulse corr.	13,8 ± 0,4
Cowan <i>et al.</i> (1963)	– pulse corr.	13,8 ± 0,4
	+ pulse corr.	13,8 ± 0,4
Mehling <i>et al.</i> (1998)	– pulse corr.	13,5 ± 0,3
	+ pulse corr.	13,5 ± 0,3

*Callister, Jr (2000).

An analysis of the analytical models used in the Flash Method to identify the thermal diffusivity of solid materials was performed in this work. The models were used to identify the thermal diffusivity of a 1020 carbon steel test sample between 25°C and 200°C, on a Netzsch LFA 447/1 Flash Method apparatus. The experimental procedure was compatible to ASTM E1461-01 standard to ensure accuracy and traceability of the results.

For the 1020 carbon steel, within the temperature range of the tests conducted in this work, no significant difference was observed on the thermal diffusivity identified with the different mathematical models available in the analysis software of the Flash apparatus. An error analysis was performed in the experimental results, and a maximum error of 3,8 % was found at higher temperatures for the Mehling *et al.* (1998) model. An average error of less than 3 % was found over the entire range of temperature and models used in this work.

The values for thermal diffusivity obtained here are in excellent agreement with those available in the literature.

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