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Finite Pulse Time Effects in Flash Diffusivity Measurements

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ABSTRACT

Analytical short-time solutions of one-dimensional heat conduction problems associated with flash thermal diffusivity measurements are given for the case of triangular pulse shape. The heat conduction problem is solved by the use of Laplace transforms taking advantage of the fact that the convolution of the temperature response curve at the back face of the sample with a triangular pulse function reduces to a simple multiplication in the Laplace domain. Instead of using the usual inversion, an expansion of the transform, followed by a term-by-term inversion, yields the solution in the time domain. This leads to a rapidly converging series for short times. Even using only one term, the relative error from the exact solution does not exeed 0.1 % at the double half-rise time. The given solutions describe the rear-face temperature response after illumination of the front surface of a mono-layer slab with a triangular shape pulse for the case of heat losses from the sample faces as well as for adiabatic conditions. Numerical approximations are given for less common higher functions appearing in the analytical solutions.

INTRODUCTION

During the thirty years passed since the first publication by Parker et al. (1961), the laser flash method has become a standard method for the determination of thermal diffusivity. The main advantage in comparison with other commonly used stationary or instationary measurement methods is the economy with regard to measuring time and sample size and a temperature range which well exeeds 1000°C.

The experiment is performed by illuminating the front surface of a flat, thin sample for a very short time and recording of the temperature versus time history at the back face of the sample. The thermal diffusivity is obtained by comparison of the sampled data with an analytical solution of the heat conduction equation given by Parker et al. (1961):

$$\theta/\theta_\infty = 1 + 2\sum_{n=1}^{\infty}(-1)^n \cos\frac{n\pi x}{l}\exp(-n^2\pi^2 Fo) \tag{1}$$

where θ_∞ is the maximum temperature rise at the rear face, l the thickness of the sample and Fo the Fourier-number which denotes a dimensionless time defined by $Fo = t/t_0$, where t_0 is the characteristic rise time $t_0 = l^2/a$. The evaluation of a measurement is easily done by use of the relation $t_0 = t_{1/2}/0.1388$, where $t_{1/2}$ is the time needed to reach $\theta_\infty/2$.

Eq. 1 is derived assuming adiabatic conditions during the measurement and infinite pulse width of irradiation. Often these ideal conditions can't be achieved: samples of low thermal conductivity suffer a remarkable heat-loss even at temperatures well below 1000°Cwhereas good conducting samples are affected by finite pulse time. Therefore these influences have to be taken into account at the derivation of the theoretical solution . This work was done in the late sixties and early seventies by different authors (Cape and Lehmann, 1963, Heckman, 1973, Taylor and ClarkIII, 1973, 1975).

The effect of non-ideal measurement conditions was treated as heat-loss correction and finite pulse width correction respectively and applied by the use of standard tables in order to retain the simple $t_{1/2}$-method and because extensive numerical evaluation equipment was not a matter of course at that time.

Eq. 1 can be expressed as

$$\theta/\theta_\infty = \frac{2}{\sqrt{\pi Fo}}\sum_{n=0}^{\infty}\exp\left(\frac{-(n+1/2)^2}{Fo}\right) \tag{2}$$

which is a well known identity in the literature on Laplace transformation (Carslaw and Jaeger, 1959, Doetsch, 1972). The main difference between these two series is the convergence behaviour: Eq. 1 converges only slowly at short times, whereas Eq. 2 has a slow convergence behaviour at longer times. Since heat-loss has little influence on the thermal response of the sample at short times, Eq. 2 is well suited for the evaluation of measurements affected by heat-loss (James, 1980). Moreover using the first term of Eq. 2 yields a simple evaluation method for the thermal diffusivity, as was outlined by Takahashi et al. (1988). Nevertheless only a few publications concerning this method appeared in recent years. James (1980) derived an analytical solution based on Eq.2 for flash measurements affected by heat-losses, Gembarovič et al. (1990) described a least squares fitting technique.

Analytical solutions taking finite pulse time effects into consideration were related to Eq. 1. The aim of this paper is to give *short time* solutions for the heat conduction equation describing the thermal response of a mono-layer slab illuminated by a triangular pulse under adiabatic conditions or affected by heat-loss respectively.

THEORY

For the derivation of the solutions given in the following section we consider a mono-layer slab of thickness l of homogenious material characterized by its thermal diffusivity $a = \lambda/\rho\, c_p$, where λ is the thermal conductivity and $\rho\, c_p$ the heat capacity per unit volume. The initial temperature $\theta(x, 0)$ is set to zero for simplicity. In the idealized case of measurement, the front surface of the sample at $x = 0$ will be illuminated by an infinitesimal short heat pulse $q\,\delta(t)$, where $\delta(t)$ denotes Diracs delta-function and q the energy deposited per unit area. The heat diffusion within the slab shall be one-dimensional and without any heat-loss from the sample surface. After having reached a stationary temperature distribution, the sample will have the uniform temperature $\theta_\infty = q/\rho\, c_p l$.

The effect of heat-loss is described by a constant heat-transfer coefficient α. It is assumed that there won't be any radial heat-loss in order to keep the problem one-dimensional. Furthermore the temperature rise $\theta_\infty = \theta(x, \infty)$ has to be small enough for a linearisation of radiative heat-losses to be valid to describe the physical situation.

The form of the finite heat pulse of most commonly used flash sources is approximated by a triangular pulse shape throughout literature (Taylor and ClarkIII, 1973), so finite pulse time will be treated in this way.

Basic equations

Let

$$\begin{aligned} \beta_1 &= 1/b \\ \beta_2 &= -1/b\,(1-b) \\ \beta_3 &= 1/(1-b) \end{aligned} \tag{3}$$

where b denotes the fraction of the pulse duration t_Δ at which the apex occurs (see Fig. 1).

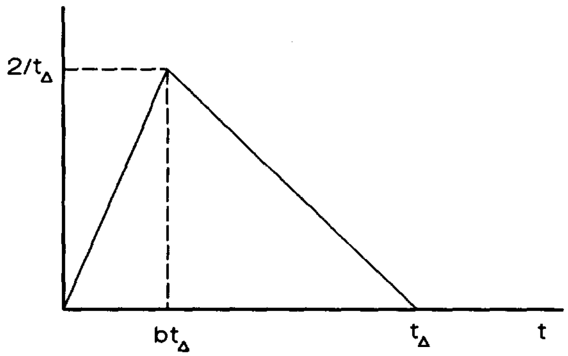


Figure 1: Triangular pulse as a function of *Fo*

Given a linear system (i.e. the sample in this case) with a known transfer function g(t), which is the response to an excitation in the form of a delta-function δ (t), the response to any arbitrary excitating function f(t) is mathematically described by the convolution of these two functions:

$$f(t) * g(t) \;=\; \int_0^t f(t-\tau)g(\tau)\,d\tau \Big/ \int_0^\infty f(\tau)\,d\tau \tag{4}$$

One of the basic statements of Laplace transformation is the convolution theorem

$$f(t) * g(t) \quad \circ\!\!-\!\!\!-\!\!\bullet \quad F(s)\,G(s) \tag{5}$$

showing that a convolution in the time domain reduces to a simple multiplication in the Laplace domain. To describe the influence of the finite pulse time in flash measurements, we have to derive the solution to our heat conduction problem in the Laplace domain, $\Theta(x,s)$ and to multiply this by the transform $F(s)$ of a triangular pulse denoted by $f(t)$ in the time domain. Finally the product has to be retransformed in order to get the temperature response in the time domain.

Then the triangular pulse and its transform are given by

$$f(t) \;=\; \frac{2}{t_\Delta^2}\begin{cases} \beta_1 t & 0 \le t \le b\,t_\Delta \\ \beta_3(t_\Delta - t) \;\text{ for } & b\,t_\Delta < t \le t_\Delta \\ 0 & t > t_\Delta \end{cases}$$

$$\circ\!\!-\!\!\!-\!\!\bullet \tag{6}$$

$$F(s) \;=\; \frac{2}{s^2 t_\Delta^2}\begin{cases} \beta_1 - [\beta_1(1+st)e^{-st}] \\ \beta_1 + \beta_2 e^{-sbt_\Delta} + [\beta_3(1+s(t-t_\Delta)e^{-st}] \\ \beta_1 + \beta_2 e^{-sbt_\Delta} + \beta_3 e^{-st_\Delta} \end{cases}$$

The factor $2/t_\Delta^2$ is chosen to represent $f(t)$ in a unit form, which removes the denominator in Eq. 4. The terms in square brackets yield a time shift about t in the time domain. Therefore they are only important in the case of the convolution of a function $g(t)$, which does not vanish at $t = 0$:

$$\lim_{t \to +0} g(t) \neq 0. \tag{7}$$

In the case of the functions treated here, g(t) does vanish, so the bracketed terms don't have to be taken into account.

The convolution of the unit pulse function $f(t)$ with any arbitrary function g(t), which vanishes at $t = 0$, yields the general solution, expressed in terms of the dimensionless Fourier-number *Fo*:

$$f(Fo) * g(Fo) = \frac{2}{t_0^2 Fo_\Delta^2} \begin{cases} \beta_1 h(Fo) & \text{for } 0 \leq Fo \leq b\,Fo_\Delta, \\ \beta_1 h(Fo) + \beta_2 h(Fo - b\,Fo_\Delta) & \text{for } bFo_\Delta \leq Fo \leq Fo_\Delta, \\ \beta_1 h(Fo) + \beta_2 h(Fo - b\,Fo_\Delta) + \beta_3 h(Fo - Fo_\Delta) & \text{for } Fo \geq Fo_\Delta \end{cases} \quad (8)$$

with

$$h(Fo) \quad \circ\!\!-\!\!-\!\!\bullet \quad \frac{G(st_0)}{s^2 t_0^2}. \quad (9)$$

To get the solution of the heat conduction equation in the Laplace domain, $\Theta(x, s)$, we have to solve

$$\frac{\partial^2 \theta}{\partial x^2} = \frac{1}{a}\frac{\partial \theta}{\partial t} \quad \circ\!\!-\!\!-\!\!\bullet \quad \frac{d^2\Theta}{dx^2} = \frac{s}{a} = \left(\frac{\xi}{l}\right)^2 \quad (10)$$

which is done by the general solution

$$\Theta = C_1 e^{\xi/l} + C_2 e^{-\xi/l}. \quad (11)$$

The boundary conditions are

$$\dot{q} = -\lambda\frac{\partial \theta}{\partial x} = \mathrm{q}\delta(t) - \alpha\theta \quad \circ\!\!-\!\!-\!\!\bullet \quad -\lambda\frac{d\Theta}{dx} = \mathrm{q} - \alpha\Theta \quad (12)$$

for $x = 0$ and

$$\dot{q} = -\lambda\frac{\partial \theta}{\partial x} = -\alpha\theta \quad \circ\!\!-\!\!-\!\!\bullet \quad -\lambda\frac{d\Theta}{dx} = -\alpha\Theta \quad (13)$$

for $x = l$, which describes the measurement situation of an infinitesimal short pulse absorbed in the front-face plane at $x = 0$. The equation describing the temperature history at the rear face is derived from Eq. 11-13 to

$$\Theta(\xi, Bi)/\Theta_\infty = 2\,t_0 \frac{\xi e^{-\xi}}{(\xi + Bi)^2 - (\xi - Bi)^2 e^{-2\xi}} \quad (14)$$

where Bi denotes the Biot-number $\alpha l/\lambda$. This can be expanded to the infinite sum

$$\Theta(\xi, Bi)/\Theta_\infty = 2\,t_0 \sum_{n=0}^{\infty} \frac{\xi\,(\xi - Bi)^{2n}}{(\xi + Bi)^{2n+2}}\, e^{-(2n+1)\xi} \quad (15)$$

which allows a term-by-term inversion to the time domain.

Adiabatic Conditions

In the case of adiabatic conditions the Biot-number has to be set to zero and Eq. 15 reduces to

$$\Theta(\xi)/\Theta_\infty = 2\,t_0 \sum_{n=0}^{\infty} \frac{e^{-(2n+1)\xi}}{\xi} \tag{16}$$

which is the Laplace transform corresponding to Eq. 2, since

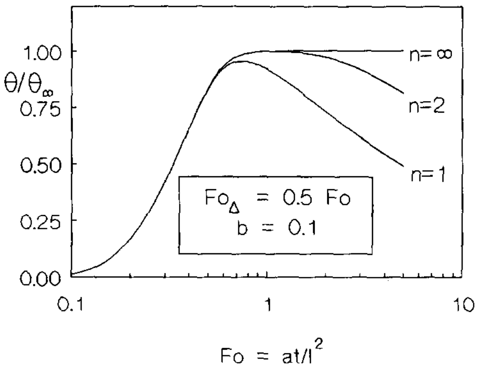
$$\frac{e^{-(n+1/2)^2/Fo}}{t_0\sqrt{\pi Fo}} \quad \circ\!\!-\!\!\bullet \quad \frac{e^{-(2n+1)\xi}}{\xi} \tag{17}$$

The convolution of Eq. 16 with the triangular pulse function is performed by inserting

$$h(Fo) = 16\,t_0^2\,Fo^{3/2} \sum_{n=0}^{\infty} \mathrm{i}^3\mathrm{erfc}\left(\frac{n+1/2}{\sqrt{Fo}}\right) \tag{18}$$

into Eq. 8. $h(Fo)$ contains the third integral of the complementary error function $\mathrm{i}^3\mathrm{erfc}\,(Fo)$, which can easily be computed by the use of rational approximations (see section Numerical Approximations).

Fig. 2 shows the thermal response for the case $b = 0.1$ and $Fo_\Delta = 0.5\,Fo$. The first term of the solution given ahead and the sum of the first two terms are also plotted. It should be remarked that even the first term gives an approximation to the true solution which can be used up to the double half rise time without exeeding a relative error of 0.1 %. The range of use is independent of the pulse time length.



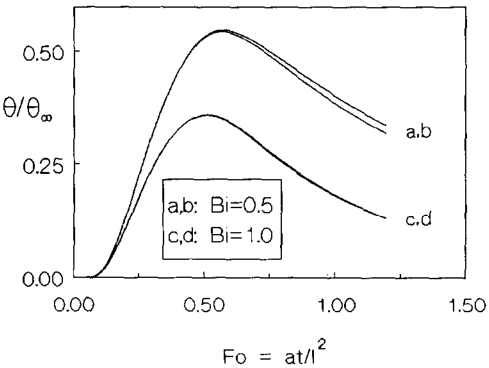


Figure 2: Thermal response curve for $b = 0.1$, $Fo_\Delta = 0.5\,Fo$ and adiabatic conditions, showing the true solution and different approximations

Figure 3: Thermal response curve for combined heatloss and finite pulse time effects with $Fo_\Delta = 0.5 Fo$, $b = 0.1$ and $Bi = 0$ or 1

Heat-loss

The solution of the heat conduction problem in the presence of heat-loss from the sample surfaces is described by Eq. 15. Let

$$\Theta_{hl,n} = \frac{\xi}{(\xi + Bi)^2} e^{-(2n+1)\xi} \tag{19}$$

Then the n-th term of the sum in Eq. 15 can be rewritten to

$$\frac{2t_0}{(2n+1)!}\left(\frac{\mathrm{D}_n}{2} + Bi\right)^{2n} (\mathrm{D}_{Bi})^{2n}\Theta_{hl,n} \tag{20}$$

where D_ν is a symbolic notation for the differential operator $\partial/\partial\nu$.

Taking finite pulse time effects into account, $\Theta_{hl,n}$ has to be replaced by

$$\Theta_{fpt,n} := \frac{\Theta_{hl,n}}{s^2} \tag{21}$$

Using

$$\mathrm{D}_\nu F(s,\nu) \quad \bullet\!\!-\!\!\!-\!\!\circ \quad \mathrm{D}_\nu f(t,\nu) \tag{22}$$

and

$$F(cs) \quad \bullet\!\!-\!\!\!-\!\!\circ \quad \frac{1}{c} f\left(\frac{t}{c}\right) \tag{23}$$

the n-th term of the solution is transformed back to the time domain to

$$h_n = \frac{2t_0}{(2n+1)!}\left(\frac{\mathrm{D}_n}{2} + Bi\right)^{2n} (\mathrm{D}_{Bi})^{2n}\theta_{fpt,n}. \tag{24}$$

The generating function $\theta_{fpt,n}$ is

$$\begin{aligned}\theta_{fpt,n} = {} & \frac{2}{Bi^3}[(1 - \eta Bi - Bi^2 Fo)\mathbf{F}_\eta - \\ & -(1 + \eta Bi)\mathrm{erfc}\left(\frac{\eta}{\sqrt{Fo}}\right) + 2Bi\sqrt{\frac{Fo}{\pi}}e^{-\eta^2/Fo}]\end{aligned} \tag{25}$$

with

$$\mathbf{F}_\eta = e^{2\eta Bi + Bi^2 Fo}\mathrm{erfc}\left(\frac{\eta}{\sqrt{Fo}} + Bi\sqrt{Fo}\right) \tag{26}$$

and $\eta = n + 1/2$. Finally inserting $h(Fo) = \sum_{n=0}^{N} h_n$ into Eq. 8 yields the equation describing the time response curve.

Since even the second term of the solution is a somewhat lengthy expression, the application of the derived formulas may be restricted to the first term, which is obtained by inserting

$$\begin{aligned}h_0 \;=\; & \frac{4t_0}{Bi^3}[(1 - Bi/2 - Bi^2 Fo)\mathbf{F}_{1/2} \\ & -(1 + Bi/2)\mathrm{erfc}\left(\frac{1}{2\sqrt{Fo}}\right) + 2Bi\sqrt{\frac{Fo}{\pi}}e^{-1/4Fo}]\end{aligned} \tag{27}$$

into Eq. 8. The effect of finite pulse time combined with heat-loss on the response curve is shown in Fig. 3. The lower curve, which was calculated with $Bi = 1.0$ shows no deviation between the complete solution (c) and the approximation by one term (d) in a *Fo*-range exeeding the range of interest for the evaluation of measurements. The two curves begin to spread in the vicinity of $Fo = 1.5$. For a lower Biot-number (curves a and b) one term is sufficient to describe the thermal response curve up to its maximum.

NUMERICAL APPROXIMATIONS

The solutions of the heat conduction equation given in the preceeding sections contain repeated integrals of the complementary error function, namely erfc(x) and i^3erfc(x). Whilst numerical approximations for erfc(x) are given in every comprehensive work on numerical mathematics, the third integral is only available in the tabular form (Abramowitz and Stegun, 1965). To ease the numerical implementation, rational approximations will be given for these functions.

Rational functions of the form

$$f(x) = \sum_{n=0}^{N} a_n x^n / \sum_{m=0}^{M} b_m x^m \tag{28}$$

are very often used for the computation of functions in a certain interval. The main advantage is the short execution time combined with a high precision. The function i^3erfc(x) was evaluated by rational Chebyshev approximation (Cody et al., 1968). The values given for erfc(x) are taken from Cody (1969).

It may be remarked that the function $\mathbf{F}_\mu$ defined in Eq. 12 can be transformed to

$$\mathbf{F}_\mu = e^{-\mu^2/Fo} e^{\xi^2} \mathrm{erfc}\xi, \tag{29}$$

where $\xi = \mu/\sqrt{Fo} + bi\sqrt{Fo}$. Therefore the approximations given in Table 1 for the complementary error function can be simplified by the factor e^{ξ^2} to avoid computer overflow.

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NOMENCLATURE

λ	thermal conductivity
ρ	density
c_p	specific heat capacity
a	thermal diffusivity
l	sample thickness
q	energy deposited per unit area
θ	excess temperature
θ_∞	equilibrum excess temperature
t	time
t_0	specific rise time l^2/a
$t_{1/2}$	time needed to reach $\theta_\infty/2$
Fo	Fourier-number t/t_0
t_Δ	duration of triangular pulse
b	fraction of pulse width at which apex occurs
β_i	weighting factors
α	heat transfer coefficient
Bi	Biot-number $\alpha l/\lambda$
s	variable in the Laplace domain
ξ	$\sqrt{st_0}$
Θ, Θ_∞	Laplace transforms of θ, θ_∞
$\mathbf{D}_\nu$	differential operator ∂/∂_ν

$$\mathrm{erfc}(x) = 1 - x \sum_{j=0}^{2} p_j x^{2j} / \sum_{j=0}^{2} q_j x^{2j}, \quad |x| \le 0.5$$

j	p_j		q_j	
0	2.13853 32237	(01)	1.89522 57241	(01)
1	1.72227 57703	(00)	7.84374 57083	(00)
2	3.16652 89065	(-01)	1.00000 00000	(00)

$$\mathrm{erfc}(x) = e^{-x^2} \sum_{j=0}^{4} p_j x^{j} / \sum_{j=0}^{4} q_j x^{j}, \quad .46875 \le x \le 4.0$$

j	p_j		q_j	
0	7.37388 83116	(00)	7.37396 08908	(00)
1	6.86501 84849	(00)	1.51849 08190	(01)
2	3.03179 93362	(00)	1.27955 29509	(01)
3	5.63169 61891	(-01)	5.35421 67949	(00)
4	4.31877 87405	(-05)	1.00000 00000	(00)

$$\mathrm{erfc}(x) = \frac{e^{-x^2}}{x} \left\{ \frac{1}{\sqrt{\pi}} + \frac{1}{x^2} \sum_{j=0}^{1} p_j x^{-2j} / \sum_{j=0}^{1} q_j x^{-2j} \right\}, \quad x \ge 4.0$$

j	p_j		q_j	
0	-1.24368 544	(-01)	4.40917 061	(-01)
1	-9.68210 364	(-02)	1.00000 000	(00)

$$\mathrm{i}^3\mathrm{erfc}(x) = \sum_{j=0}^{3} p_j x^{j} / \sum_{j=0}^{3} q_j x^{j}, \quad x \le 0.8$$

j	p_j		q_j	
0	4.47996 17307	(-01)	4.76431 04987	(00)
1	-7.68596 61317	(-01)	4.49348 48660	(00)
2	4.71586 42365	(-01)	2.65932 61331	(00)
3	-1.03965 92405	(-01)	1.00000 00000	(00)

$$\mathrm{i}^3\mathrm{erfc}(x) = \frac{e^{-x^2}}{x^4} \sum_{j=0}^{3} p_j x^{-2j} / \sum_{j=0}^{3} q_j x^{-2j}, \quad 0.8 \le x \le 5.0$$

j	p_j		q_j	
0	5.90340 26845	(-03)	8.37175 91605	(-02)
1	3.48975 98579	(-02)	9.12762 34378	(-01)
2	3.27974 61253	(-03)	2.43284 27517	(00)
3	-2.15551 39741	(-04)	1.00000 00000	(00)

$$\mathrm{i}^3\mathrm{erfc}(x) = \frac{e^{-x^2}}{8x^4} \left\{ \frac{1}{\sqrt{\pi}} + \frac{1}{x^2} \sum_{j=0}^{2} p_j x^{-2j} / \sum_{j=0}^{1} q_j x^{-2j} \right\}, \quad x \ge 5.0$$

j	p_j		q_j	
0	-4.22018 15431	(-01)	1.49601 54263	(-01)
1	-6.05352 55981	(-01)	1.00000 00000	(00)
2	1.51677 23483	(00)		

Table 1: Rational Approximations for erfc(x) and i^{3}erfc(x)