

ANALYSIS OF METROLOGICAL CONDITIONING OF THERMAL DIFFUSIVITY MEASUREMENTS APPLYING MODIFIED ÅNGSTRÖM'S METHOD AT SCANNING MODE OPERATION

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Abstract

A technique of the temperature dependent thermal diffusivity investigation has been analysed. The technique is based on the modified Ångström method utilising periodic temperature oscillations in a slab specimen. The additional modification consists of imposing linear temperature changes – application of linear scans. This scanning mode procedure enables investigation of the thermal diffusivity over a certain temperature interval. The thermal diffusivity is calculated independently from two relations based on the measured amplitude attenuation and the measured phase shift of the temperature oscillation passing through the investigated specimen. The paper discusses the problem of the thermal diffusivity identification uncertainty. As a result correction factors for the thermal diffusivity data derived from simplified formulae have been determined. The procedure of the thermal diffusivity measurement has been verified by numerical modelling. Comparison between the initial and response signals with illustration of the linear scan imposed to a basic oscillation with reference to a classical procedure of subsequent steps, illustration of the approximation error analysis methodology and results in the form of a thermal diffusivity correction factor, comparison between the uncorrected and corrected applying in numerical modelling of the temperature oscillation experiment are presented in the paper.

Keywords: *thermal diffusivity, thermophysical properties, temperature oscillation, modified Ångström technique*

1. Introduction

Periodic heating or so-called temperature (thermal) wave technique is probably the oldest one transient technique for investigation of thermophysical properties. Initially described by Ångström in 1861 in application for investigation of metal bars and extended within limits of certain heat conduction problem models [5, 14] it for many years was treated as a standard thermophysical property measurement method. However, in view of a growing application of mostly laser flash and pulse techniques (comp. e.g. [8, 10]) in the last decades it has been limited in its general application. Nowadays we can observe renaissance of the temperature oscillation technique [3] especially in specific domains like microscopic measurements, studies of thermal processes [6, 7] and investigation of special materials [2]. It is due to some advantages of the thermal wave method like possibility of application for different materials [12], the unique potential for a cross-validation by comparing results obtained from the amplitude and phase analyses [1, 15], possibility of multiproperty investigation, spectral Fourier studies and thermal cycling measurements [15], possibility of application with different instrumentation [5, 7].

One of the Ångström's method modifications was developed for investigation of bad thermal conductors studied as plates [1, 2]. The thermal diffusivity of such specimens is obtained in cross-planar direction applying solutions of a certain heat conduction problem with a temperature oscillation applied over a certain steady mean temperature [4]. In the present paper an extension of the above-described modification is analysed. This extension is based of introducing a scanning mode measurement procedure that means that measurements are performed with oscillations applied to a linearly changed mean temperature.

2. Theory

Assuming that the temperature oscillation amplitude – is relatively small:

$$\theta_{\max} \ll T_m \quad (1)$$

and that the oscillation frequency is high enough to confine several oscillations within a single ramp of a steady or a linearly changed temperature:

$$f = \frac{1}{\tau_{\Omega}} \gg \frac{1}{\tau_{i+1} - \tau_i}, \quad i = 0, 1, \dots, k, \quad (2)$$

where:

θ_{\max} - amplitude of the temperature oscillation (“temperature wave”, “thermal wave”),

T_m - certain mean temperature (temperature level),

τ_{Ω} - oscillation period,

the problem can be treated as superposition of two linear problems of heat conduction. The first one refers to the temperature oscillations, the second to linear temperature changes in time. The individual problems are referred as regular heating regime of the third and second type respectively [10].

If we focus our attention on the temperature differences with respect to the initial temperature T_0 :

$$\theta(x, \tau) = T(x, \tau) - T_0, \quad (3)$$

which is uniform over the object i.e. a plate of thickness l at $\tau = 0$, than mathematical formulation of component problems is given by the governing equation:

$$\frac{\partial \theta}{\partial \tau} = a \frac{\partial^2 \theta}{\partial x^2}, \quad a = \frac{\lambda}{\rho c_p}, \quad (4)$$

where:

T - temperature,

x - space coordinate,

τ - time,

θ - temperature changes referred to T_m ,

x - space coordinate,

τ - time,

a - thermal diffusivity (TD),

λ - thermal conductivity (TC),

ρ - thermal conductivity (TC),

c_p - specific heat,

with the initial condition:

$$\theta(x, 0) = 0 \quad \text{at } 0 \leq x \leq l \quad \text{for } \tau = 0, \quad (5)$$

and two sets of boundary conditions (BC), one for harmonic temperature changes problem (Fig. 1):

$$\frac{\partial \theta(0, \tau)}{\partial x} = 0, \quad \theta(l, \tau) = A_0 \sin(2\pi f \tau - \varepsilon), \quad (6)$$

and one for the linear temperature changes problem:

$$\frac{\partial \theta(0, \tau)}{\partial x} = 0, \quad -\lambda \frac{\partial \theta(l, \tau)}{\partial \tau} = T_0 + b \tau. \quad (7)$$

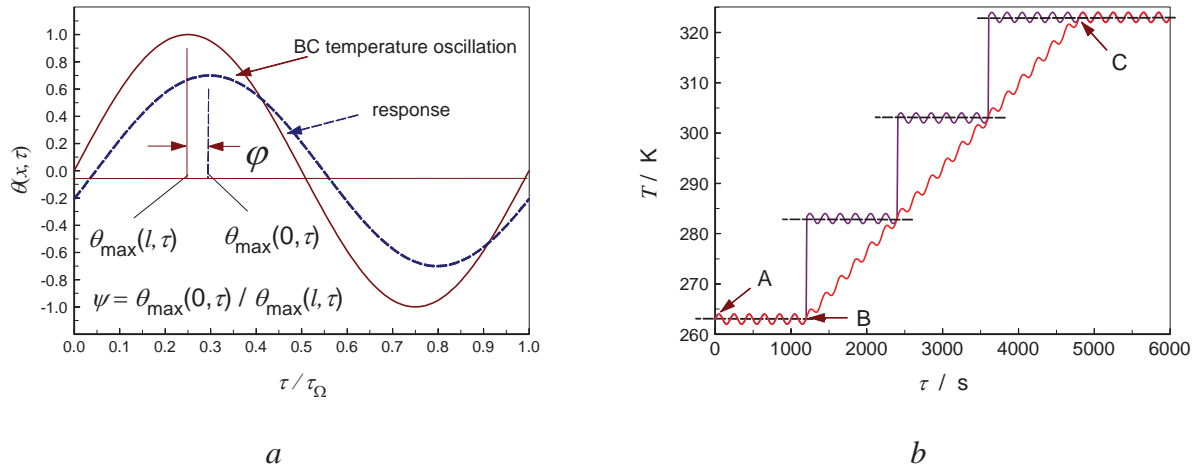


Fig. 1. Comparison between the initial and response signals (a) with illustration of the linear scan imposed to a basic oscillation with reference to a classical procedure of subsequent steps (b)

The solution of the BC (6) problem is [4]:

$$\begin{aligned} \theta(x, \tau) = & A_0 \psi \sin(2\pi f \tau - \varphi - \varepsilon) + \\ & + 4\pi k \sum_{n=0}^{\infty} \frac{(-1)^n (2n+1) [4l^2 \omega \cos \varepsilon + a(2n+1)^2 n^2 \sin \varepsilon]}{16l^4 \omega^2 + a^2 \pi^4 (2n+1)^4} \exp\left[-\frac{a(2n+1)\pi^2}{4l^2} \tau\right] \cos\left[\frac{(2n+1)\pi x}{2l}\right], \end{aligned} \quad (8)$$

where (Fig. 1.a):

$$\psi(x) = \sqrt{\frac{\cosh 2kx + \cos 2kx}{\cosh 2kl + \cos 2kl}}, \quad (9)$$

$$\varphi(x) = \arg\left[\frac{\cosh kx(1+i)}{\cosh kl(1+i)}\right], \quad (10)$$

$$k = \sqrt{\frac{\pi f}{a}} = \sqrt{\frac{\pi}{a \tau_\Omega}}, \quad (11)$$

while for the BC given by Eq. (7) we get [4]:

$$\begin{aligned} \theta(x, \tau) = & b\tau + \frac{b(x^2 - l^2)}{2a} + \\ & + 16 \frac{bl^2}{a\pi^3} \sum_{n=0}^{\infty} \frac{(-1)^n}{(2n+1)^3} \exp\left[-\frac{a(2n+1)^2 \pi^2}{4l^2} \tau\right] \cos\left[\frac{(2n+1)\pi x}{2l}\right]. \end{aligned} \quad (12)$$

The series terms with exponential components in (8) and (12) represents transients that die away as $\tau \rightarrow \infty$. The representative solution of the steady oscillations over a linearly changed temperature expressed by superposition of the two appropriate components is:

$$\theta(x, \tau) \cong A_0 \psi \sin(2\pi f \tau - \varphi - \varepsilon) + b\tau + \frac{b(x^2 - l^2)}{2a}. \quad (13)$$

3. Thermal diffusivity identification procedure

Knowing the parameter l and having obtained the amplitude of temperature response attenuation ψ and the response phase lag φ one can evaluate the parameter k (or directly the thermal diffusivity a) by solving nonlinear Eqs (9) and (10). These two solutions should be the same that creates opportunity for the procedure validation. However, because the solutions can not be expressed in direct form, and because numerical routine in the case of Eq. (10) is badly conditioned, appropriate simplifications of (9) and (10) are usually applied [1, 2]. These approximations lead to the following formulae:

$$a \cong \frac{\pi f l^2}{\ln^2 \frac{2}{\psi}} = \frac{\pi l^2}{\tau_\Omega \ln^2 \frac{2}{\psi}}, \quad (14)$$

$$a \cong \frac{\pi f l^2}{\varphi^2} = \frac{\pi l^2}{\tau_\Omega \varphi^2}, \quad (15)$$

the first of which is the ‘‘amplitude’’ one and the second is the ‘‘phase’’ second respectively. It is interesting that Eq. (15) has the same form as that obtained for the semi-infinite solid problem solution (comp. e.g. [4, 14] 16]). However these solutions are valid only under the assumption that the frequency f is high enough i.e.:

$$k l > K_{\min} \Leftrightarrow f > \frac{K_{\min}^2 a}{\pi l^2}, \quad (16)$$

where K_{\min} is a certain parameter defining limits for the approximation validity.

The thermal diffusivity can be determined from amplitude and phase measurements. In both cases the two temperature signals are analysed: one corresponding to the thermally treated surface $\theta(\tau, x=l)$ and the response signal $\theta(\tau, x=0)$. The two signals are usually approximated by the following function:

$$f(\tau) = A \sin(2\pi f \tau + B) + C + D \tau; \quad n \tau_\Omega \leq \tau < (n+1)\tau_\Omega, \quad n = 0, 1, \dots, N, \quad (17)$$

where:

A, B, C, D - the identified coefficients in the course of the applied curve fitting procedure,

n - indexes of the following time periods,

N - total number of the analysed periods,

and parameters ψ and φ are calculated from:

$$\psi = \frac{A_{x=0,n}}{A_{x=l,n}}, \quad \varphi = B_{x=l,n} - B_{x=0,n}, \quad (18)$$

where:

$x=l$ - denotes the input temperature oscillation with a linear offset,

$x=0$ - denotes the response signal.

As it follows from Eq. (17) the procedure is applied within time intervals corresponding to every consecutive oscillation period n . Next, the thermal diffusivity can be derived:

- directly from simplified relations (14) and (15),
- after correction of the Eqs (14) and (15) results applying differential formulae,
- from numerical solution of nonlinear Eqs (9) and (10) expressed as [12]:

$$\frac{\sqrt{2}}{\psi} = \sqrt{\cosh\left(2\sqrt{\frac{\pi f}{a}} \cdot l\right) + \cos\left(2\sqrt{\frac{\pi f}{a}} \cdot l\right)}, \quad (19)$$

$$tg\varphi = tg\left(\sqrt{\frac{\pi f}{a}} \cdot l\right) \tanh\left(\sqrt{\frac{\pi f}{a}} \cdot l\right) \Leftrightarrow \varphi = \arctg\left\{tg\left(\sqrt{\frac{\pi f}{a}} \cdot l\right) \tanh\left(\sqrt{\frac{\pi f}{a}} \cdot l\right)\right\}, \quad (20)$$

with reference to $kl = l\sqrt{\frac{\pi f}{a}}$ or a regarded as unknown parameter.

Analysing Eq. (13) one can propose another identification procedure based on the last right hand term utilisation. In such a case the thermal diffusivity is obtained from:

$$a = \frac{bl^2}{2(C_{x=l,n} - C_{x=0,n})}. \quad (21)$$

This procedure corresponds to the thermal diffusivity identification applying regular heating regime of a second kind methodology (comp. e.g. [9, 10]). However, in real measurements results based on this strategy could be strongly affected by the temperature bias caused by heat losses not accounted for in the above described mathematical model.

Summing up, the following advantages of the proposed modification of the temperature oscillation technique by imposing linear temperature changes can be indicated:

- the new procedure does not affect the original high thermal (temperature) resolution measurements possibility,
- the high thermal resolution TD data can be gathered not only at certain temperatures but also within a certain temperature interval (Fig. 1.b),
- the original chance for a cross-validation of the results by comparing the “amplitude” and “phase” data is supplemented by opportunity of TD calculation from Eq. (21) at scanning mode sequences.

4. Analysis of TD identification procedure performance

There are several aspects of implementation of the above-described technique in practice. First of all the effects of the initial transients prior to regular heating regime segments needs to be discussed. Regular heating regime conditions, in that case linear (2-nd kind) and periodic (3-rd kind), are disturbed at the oscillation start and at every change of the imposed temperature offset step (Fig. 1.b, points A, B and C). The dynamics of the heat transfer stabilisation process is described by time dependent terms in Eqs (8) and (12) respectively. Comparing the appropriate exponents in exponential terms one can get:

$$\frac{a(2n+1)\pi^2}{4l^2} < \frac{a(2n+1)^2\pi^2}{4l^2} = \frac{a(2n+1)\pi^2}{4l^2}(2n+1), \text{ for } n = 1, 2, \dots \quad (22)$$

This means that the transient caused by the offset temperature heating /cooling rate change dies faster than the basic one. Only the first terms of the compared series are of the same range. The same the conclusion can be drawn that the modification does not affect the original procedure metrological limits.

The second problem concerns applying simplified formulae (14) and (15) for the TD identification when analysing the experimental results. In such a case one can avoid solving non-linear problems given by Eqs (19) and (20). From the last two the second one is badly conditioned in numerical means. It is because of a periodic character of the involved functions. However, when simplified formulae are utilised the experiment needs to be optimised for the condition (16). When defining certain K_{min} values¹ the appropriate limitations of the identification accuracy need to be kept in mind. Moreover, regarding possible temperature dependence of the thermal diffusivity the kl parameter changes related to that phenomenon should be taken into account. This is the reason

¹ That could be different between the “phase” and the “amplitude” procedures.

for a more thorough examination of the approximation error behaviour.

In our case the error related to utilisation of the simplified formulae (14) and (15) has been analysed using a differential approximation. The method is illustrated in Fig. 2. It is based on the assumption of asymptotic convergence of a certain approximate function $f_{app}(\xi)$ to the exact one $f(\xi)$ with increasing ξ :

$$\Delta\eta = f(\xi) - f_{app}(\xi) \xrightarrow{\xi \rightarrow \infty} 0, \quad (23)$$

where:

ξ - independent variable,

η - dependent variable.

In such a case the differential $d\xi$ can be expressed as:

$$d\xi = [f^{-1}(\eta)] d\eta = \frac{d\eta}{f'(\xi)}, \quad (24)$$

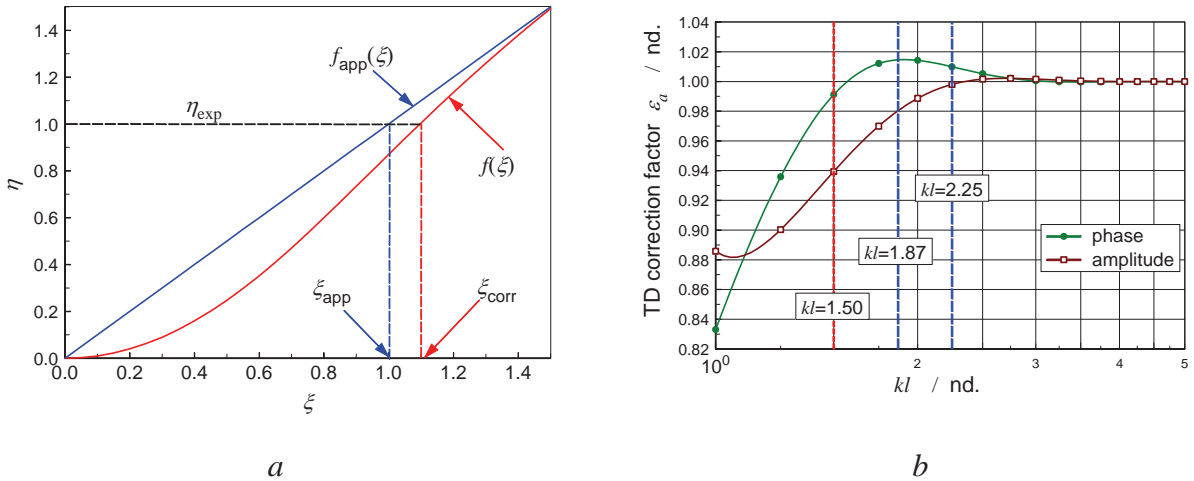


Fig. 2. Illustration of the approximation error analysis methodology (a) and results in the form of a thermal diffusivity correction factor (b) for the case when simplified formulae (14) and (15) for the TD calculation are applied (depicted curves correspond to the “phase” problem given by Eq. (20))

Tab. 1. Correction factors ε_a for the TD obtained from simplified formulae recalculation

kl	ε_a		kl	ε_a		kl	ε_a	
	phase	amplitude		phase	amplitude		phase	amplitude
1	0.8330	0.8858	1.7	1.0010	0.9648	2.4	1.0070	1.0006
1.05	0.8580	0.8817	1.75	1.0121	0.9700	2.45	1.0061	1.0011
1.1	0.8806	0.8828	1.8	1.0136	0.9746	2.5	1.0053	1.0015
1.15	0.9011	0.8870	1.85	1.0144	0.9788	2.55	1.0045	1.0018
1.2	0.9195	0.8931	1.9	1.0147	0.9825	2.6	1.0038	1.0020
1.25	0.9357	0.9003	1.95	1.0146	0.9858	2.65	1.0032	1.0021
1.3	0.9504	0.9081	2	1.0142	0.9887	2.7	1.0026	1.0021
1.35	0.9631	0.9162	2.05	1.0136	0.9912	2.75	1.0021	1.0021
1.4	0.9741	0.9241	2.1	1.0128	0.9933	2.8	1.0017	1.0020
1.45	0.9834	0.9319	2.15	1.0119	0.9952	2.85	1.0013	1.0020
1.5	0.9912	0.9394	2.2	1.0109	0.9967	2.9	1.0010	1.0019
1.55	0.9977	0.9465	2.25	1.0099	0.9980	2.95	1.0007	1.0017
1.6	1.0029	0.9531	2.3	1.0089	0.9991	3.00	1.0005	1.0016
1.65	1.0069	0.9592	2.35	1.0079	0.9999	3.05	1.0003	1.0014

and the error due to exchange of the initial problem of the independent variable ξ identification:

$$\eta_{\text{exp}} - f(\xi) = 0, \quad (25)$$

by its approximation:

$$\eta_{\text{exp}} - f_{\text{app}}(\xi) = 0, \quad (26)$$

can be corrected as follows:

$$\xi_{\text{corr}} = \xi_{\text{app}} + \left. \frac{d\eta}{f'(\xi)} \right|_{\xi=\xi_{\text{app}}} = \xi_{\text{app}} + \frac{\eta_{\text{exp}} - f(\xi_{\text{app}})}{f'(\xi_{\text{app}})},$$

where:

exp - denotes a certain (experimental) ordinate value,

app - denotes solution of the approximate problem given by Eq. (26),

corr - denotes corrected value of the solution.

Assuming $\xi=kl$ and taking:

$$\eta = \begin{cases} \sqrt{\cosh(2kl) + \cos(2kl)} & \text{for amplitude analysis,} \\ \arctg\{tg(kl)\tanh(kl)\} & \text{for phase analysis,} \end{cases} \quad (27)$$

for problems (19) and (20) respectively and defining:

$$\delta_{kl} = \frac{(kl)_{\text{corr}} - (kl)_{\text{app}}}{(kl)_{\text{app}}}, \quad (28)$$

one can get from Eq. (11):

$$a_{\text{corr}} = \frac{1}{(1 + \delta_{kl})^2} a_{\text{app}} = \varepsilon_a a_{\text{app}}. \quad (29)$$

where:

δ_{kl} - is the relative difference of the corrected solution with reference to the approximate one,

ε_a - correction factor for the TD recalculation according to Eq. (29).

The parameter ε_a corresponds to the TD identification uncertainty while applying simplified formulae (14) and (15) respectively. The correction factor values obtained using the above-described methodology are depicted in Fig. 2.b and shown in Table 1. These values can also be applied for correction of the TD calculated from (14) and (15). In such a case an onerous procedure of solving non-linear problems (19) and (20) can be avoided. From comparison of numerical solutions of exact problems (19) and (20) with corrected results obtained from (14) and (15) it follows that this correction procedure ensures the accuracy of about 2% while $kl > 1.0$, and better than 1 % while $kl > 1.5$.

According to [2] the recommended value for K_{min} is 1.5. However, for K_{min} strictly equal to 1.5 the “amplitude” thermal diffusivity identified when applying Eq. (14) is overestimated by about 6.5 % (Fig. 2.b). Applying the obtained values of ε_a (Tab. 1) one can get new limits for K_{min} :

$$K_{\text{min}, 2\%} = 1.87; \quad K_{\text{min}, 1\%} = 2.25, \quad (30)$$

where 2% and 1% - denotes the corresponding theoretical accuracy of identification for both: amplitude and phase formulae.

5. Numerical test of the TD correction procedure

The numerical modelling has been performed to illustrate the proposed technique of the thermal diffusivity measurement performance. In the course of simulation both effects of the

linear scans and TD data correction according to Eq. (25) have been investigated. The assumed model was referred to real experiments performed while the temperature oscillation procedure has been validated experimentally [12]. This is why certain thermophysical parameters have been assumed for the modelled media. The model took into consideration the phase transition within the narrow region over 273 K with a substantial change in the thermophysical properties. The details concerning the model geometry, applied temperature input and media properties can be found elsewhere [13]. The temperature programme of a virtual experiment was composed of a steady oscillation over 263 K, oscillation with a linear scan from 262 K up to 323 K and finishing segment of a steady oscillation over 323 K. The oscillation period was equal to 120 s and the temperature change rate of the linear ramp equal to 1 K/min. Combination of these parameters with thermophysical properties of the modelled media resulted in kl equal to 1.55 in the low temperature region and equal to 3.91 in the high temperature region (comp. Fig. 3.a). The final results obtained applying FEM Comsol software are shown in Fig. 3. The identified TD values are in agreement with the expected ones. Moreover, the overestimation of the amplitude TD results in the low temperature region fully agrees with the result of the parameter ε_a estimation: the TD relative difference of 6.4% is almost as has been expected for the $kl=1.55$ (comp. Fig. 2.b and Tab. 1). Performance of the applied correction procedure is illustrated in Fig. 3.b.

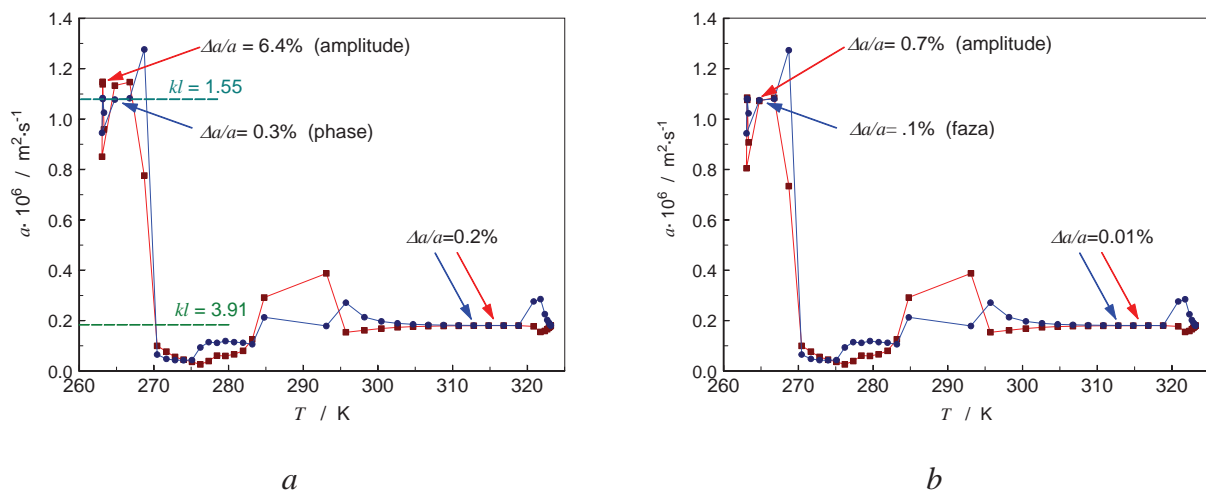


Fig. 3. Comparison between the uncorrected (a) and corrected applying Tab. 1 data for ε_a (b) results of a TD identification in numerical modelling of the temperature oscillation experiment

6. Conclusion

The performed analysis has been focused on the metrological conditioning of the modified temperature oscillation technique of the thermal diffusivity measurement. The improvement is based on imposing linear temperature changes, similar to those known from DSC techniques, onto the temperature oscillations. The proposed modification facilitates investigation of materials exhibiting temperature dependence of thermophysical properties. The studies resulted in more precise definition of limits for application of a simplified procedure of the thermal diffusivity calculation. Moreover, by developing the correction procedure the limits of a standard formulae application have been extended. The same optimisation of real experiments has been made easier. The outcomes of the theoretical analysis have been validated by numerical simulation. The obtained results will be applied in investigation of materials exhibiting strong dependence of thermophysical properties including phase changes like ice accretion structures and heat shock absorbers.

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