

DETERMINATION OF THERMOPHYSICAL PROPERTIES OF SOLIDS AT HIGH TEMPERATURES BY MEASURING PHASE CHARACTERISTICS OF A PLANE TEMPERATURE WAVE AT TWO FREQUENCIES

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The authors propose and substantiate a procedure to determine thermal diffusivity and the ratio of integral emissive power to thermal conductivity of solids by measuring the phase delay of a plane temperature wave at two frequencies. The method is applicable for a wide range of heat loss parameters and does not need calorimetric measurements. Errors are calculated and the domains of applicability of the given procedure are pointed out.

A method of plane temperature waves is widely employed in thermophysical properties (TPP) measurements of solids in a high-temperature range [1]. The general problem in all modifications of the method is the difficulty of TPP determination from theoretical considerations. Relations obtained in [2-9] represent experimentally measured phase shifts and amplitudes of a temperature wave as a function of thermal diffusivity, the specimen thickness, the modulation frequency of the heat flux, and heat losses — parameters dependent, in their turn, on TPP. In the general case, it is impossible to obtain analytically the inverse dependences relating the substance properties of a specimen with measured quantities.

The approximate dependences [3, 10-12] of thermal diffusivity on phase shifts of a plane temperature wave may be used only in those cases, when heat transfer in the system may be neglected or reduced to a small correction that requires use of thin (from tenths of a millimeter to 1 mm) specimens with relatively high thermal conductivity and creation of high modulation frequencies of the heat flux (tens and hundreds Hz). Thus, these formulas are not applicable for poor heat conductors with a considerable "representative" element in the form of a specimen (composites, disperse materials, rocks) as well as in the cases when a high modulation frequency fails to be attained or is not reasonable because of decreasing the temperature signal amplitude.

The approximate relations [13, 14] may be applied only for refractory metals [13] and high-temperature ceramics [14] but their use is limited by a narrow range of heat loss parameters to be evaluated which requires additional information on TPP, for instance, from literature.

The main drawback of the above methods is the possibility to determine only thermal diffusivity. In order to determine a set of TPP (thermal diffusivity, thermal conductivity, heat capacity), it is necessary to conduct a separate calorimetric experiment, the main problem of which, i.e., measurement of the amplitude of specific heat flux oscillations, arises due to low reliability of calibration measurements with standard substances [16] or due to a complicated procedure of an account of the radiation emitted by the probe onto the specimen if electrocompensation methods are used [17].

The foregoing generates the thought about development of such methods of TPP determination which, firstly, could be used in a wide range of heat loss parameters and, consequently, for a wide class of substances (from metals to poor heat conductors) and, secondly, would not need heat flux measurement. One such methods has been developed by the authors and involves phase delay measurement of the temperature wave in a specimen at two frequencies.

The proposed method to determine thermal diffusivity and an integral emissive power-to-thermal conductivity ratio of solids employs the expression obtained in [2] for the phase shift δ_0 between the fluctuations of the heat flux modulated with

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a cyclic frequency ω , which uniformly irradiates one of the surfaces of an infinite homogeneous and isotropic specimen with thickness l , and the temperature fluctuations on the opposite side of the specimen. If angles δ_0 are measured at two frequencies ω_1 and ω_2 ($\omega_2 > \omega_1$), then we may write the following system of equations

$$\begin{aligned} \gamma_1 &= \frac{\beta\alpha^2\Gamma_2^{(1)} + 2\alpha B\Gamma_3^{(1)} + 2B^2\Gamma_1^{(1)}}{\beta\alpha^2\Gamma_1^{(1)} + 2\alpha B - 2B^2\Gamma_2^{(1)}}, \\ \gamma_2 &= \frac{\beta\alpha^2\Gamma_2^{(2)} + 2\alpha\mu B\Gamma_3^{(2)} + 2\mu^2 B^2\Gamma_1^{(2)}}{\beta\alpha^2\Gamma_1^{(2)} + 2\alpha\mu B - 2\mu^2 B^2\Gamma_2^{(2)}}, \\ \gamma_1 &= \operatorname{tg} \delta_0(\omega_1), \quad \gamma_2 = \operatorname{tg} \delta_0(\omega_2), \\ \beta &= \frac{r}{(1+r)^2}, \quad \mu = \left(\frac{\omega_2}{\omega_1}\right)^{1/2} = \left(\frac{\tau_1}{\tau_2}\right)^{1/2}, \\ \Gamma_1^{(1)} &= \operatorname{tg} B + \operatorname{th} B, \quad \Gamma_1^{(2)} = \operatorname{tg} \mu B + \operatorname{th} \mu B, \\ \Gamma_2^{(1)} &= \operatorname{tg} B - \operatorname{th} B, \quad \Gamma_2^{(2)} = \operatorname{tg} \mu B - \operatorname{th} \mu B, \\ \Gamma_3^{(1)} &= \operatorname{tg} B \operatorname{th} B, \quad \Gamma_3^{(2)} = \operatorname{tg} \mu B \operatorname{th} \mu B, \\ \alpha &= l(A_0 + A_l), \quad r = \frac{A_l}{A_0}, \\ A_x &= \left[\frac{\left(\frac{\partial\Phi}{\partial T}\right)}{\lambda} \right]_{T=T(x; 0)}; \quad x = 0, l, \\ B &= l \left(\frac{\omega}{2a}\right)^{1/2}. \end{aligned} \tag{1}$$

From the first equation of system (1), we find an expression for a heat losses parameter α :

$$\alpha = \frac{1}{2} [(A_1^2 + 4A_2^2)^{1/2} - A_1], \tag{3}$$

where

$$A_1 = \frac{2B(\gamma_1 - \Gamma_3^{(1)})}{\beta(\gamma_1\Gamma_1^{(1)} - \Gamma_2^{(1)})}; \quad A_2 = \frac{2B^2(\gamma_1\Gamma_2^{(1)} + \Gamma_1^{(1)})}{\beta(\gamma_1\Gamma_1^{(1)} - \Gamma_2^{(1)})}. \tag{4}$$

Substituting (3) and (4) into the second equation of system (2), we obtain the transcendental equation

$$f(B) = \gamma_2 - \frac{\beta\alpha^2\Gamma_2^{(2)} + 2\alpha\mu B\Gamma_3^{(2)} + 2\mu^2 B^2\Gamma_1^{(2)}}{\beta\alpha^2\Gamma_1^{(2)} + 2\alpha\mu B - 2\mu^2 B^2\Gamma_2^{(2)}} = 0. \tag{5}$$

Solving this equation by a numerical method allows us to determine B and calculate thermal diffusivity from relation (2). It is worthy noting that $f(B)$ is a periodical function and that only one root of Eq. (5) corresponds to a real experiment. In order to determine the necessary root B in Eq. (5), we used the secant iteration method [15]. As the first two iterations, the B values calculated by approximate relations [3, 11] were used. Computer-aided numerical simulation of the experiment proved that the procedure works. The accuracy of the root B determination was not worse than $2 \cdot 10^{-6}$.

In the mentioned works [3-14], consideration has been given to heat transfer from specimen surfaces only by radiation. The proposed procedure allows thermal diffusivity determination not only for a wide range of heat losses but also for other types of heat transfer. Since heat loss parameters are simply excluded, they are not to be evaluated, which for most of the heat transfer types represents a rather complicated problem. The last circumstance is of importance in those cases when a chamber with a specimen is to be filled with an inert gas under pressure, therefore an account of convective heat transfer becomes an urgent problem.

If heat is transferred from specimen surfaces only by radiation, then using the relations for heat loss parameters, α and r [2], we may calculate the ratio of integral emissive power ϵ to thermal conductivity λ of the specimen substance

$$\frac{\varepsilon}{\lambda} \approx \frac{\alpha}{4\sigma T^3(0; 0) l(1+r)}, \quad r \approx \frac{T^3(l; 0)}{T^3(0; 0)}. \quad (6)$$

Here $\sigma = 5.668 \times 10^{-8} \text{ W}/(\text{m}^2 \cdot \text{K}^4)$ is the Stefan–Boltzmann constant. Substituting the obtained root B into (4) and (3), we may determine α .

We have been also concerned with methodical errors in TPP determination which may be evaluated only, because of the complexity of (5), by a numerical method allowing a choice of the α and B ranges and the ratio $k_1 = \tau_1 / \tau_2$ of modulation periods which correspond to minimal errors in determination of thermal diffusivity and an integral emissive power-to-thermal conductivity ratio.

Using data [5] on the ranges of thermal diffusivity, thermal conductivity and integral emissive power of solids, we may calculate, using (2) and (6), the limits within which parameters α and B vary: $0.2 \leq B \leq 20$, $10^{-4} \leq \alpha \leq 20$.

The ratio K of modulation periods was varied within $1.25 \leq K \leq 10$.

Errors were computed by a numerical method as follows. In the first part of the program, a solution to the direct problem was specified, i.e., for a given pair of α and B values at different K calculation was made, by the formula [2], of the angles $\delta^0(\omega_1)$ and $\delta^0(\omega_2)$ of the phase shift between the specific heat flux fluctuations and the temperature fluctuations on the rear surface of the specimen at modulation frequencies ω_1 and ω_2 ($\omega_2 = K\omega_1$). Next, one of the experimentally measured quantities (specimen thickness l , modulation periods τ_1 and τ_2 , mean temperature $T(0;0)$, phase shifts $\delta_0(\omega_1)$ and $\delta_0(\omega_2)$) was changed by 1%, with other quantities remaining unchanged. Then the angles $\delta_0(\omega_1)$ and $\delta_2(\omega_2)$, corresponding to the experimental value changed by 1%, were calculated and the mentioned iteration secant method was applied to solve an inverse problem, i.e. to calculate σ and β . The changes in α and β , as compared to their initial values, were assumed to be equal to those in ε/λ and thermal diffusivity, respectively, caused by 1% variation of one of the quantities measured in the experiment.

The calculations have demonstrated that the changes in thermal diffusivity caused by errors of 1% in measurements of mean temperature $T(0; 0)$, thickness l , modulation periods τ_1 and τ_2 do not depend practically on α , β and the ratio K of modulation periods and amount, respectively, to ~ 0.001 , ~ 2 and $\sim 1\%$. For the ratio ε/λ , those values are ~ 3 , ~ 1 , and $\sim 0.001\%$.

The method has proved to be more sensitive to the errors in measurements of phase shifts $\delta_0(\omega_1)$ and $\delta_0(\omega_2)$, with TPP variation being very highly dependent on K . Variation of $\delta_0(\omega_2)$ causes a somewhat larger change in TPP than $\delta_0(\omega_1)$ variation does.

Figure 1 shows the domain boundaries of the parameters α and B (in dependence on the ratio K of modulation frequencies), within which a 1% change in the phase shift $\delta_0(\omega_2)$ causes a change of thermal diffusivity by less than 3% (in the majority of the known devices, realizing the method of plane temperature waves, an error in thermal diffusivity measurement is about 3-5%). The domains given above lie below the present curves.

As is seen, with increasing the ratio K the ranges of applicable α and B values expand. Consequently the experiment, using the suggested procedure, needs a frequency shift as large as possible. However there are constraints owing to the facts that an amplitude of the temperature signal falls with increasing frequency $\omega_2 = K\omega_1$ (approximately proportionally to ω_2 [1]) and that each subsequent increase of K entails a progressively decreasing expansion of the domain of adequate values of α and B .

As it follows from Fig. 1, for thermal diffusivity measurement by the proposed method the following α and B ranges are most convenient:

$$\begin{aligned} B \geq 2,3, \quad \alpha \leq 3, \quad K = 2, \\ B \geq 2, \quad \alpha \leq 15, \quad K = 4, \\ B \geq 1,9, \quad \alpha \leq 20, \quad K = 10. \end{aligned} \quad (7)$$

Here an error in the thermal diffusivity measurement ceases to change with B .

Figure 2 represents the ranges of α and B values (bound with curves), within which a 1% change of the phase shift $\delta_0(\omega_2)$ entails a change of ε/λ by less than 20% (just such error in integral emissive power measurement is considered to be sufficiently satisfactory [18]). As in the previous case, the ranges of applicable α and B values expand with an increase in the ratio K of modulation periods.

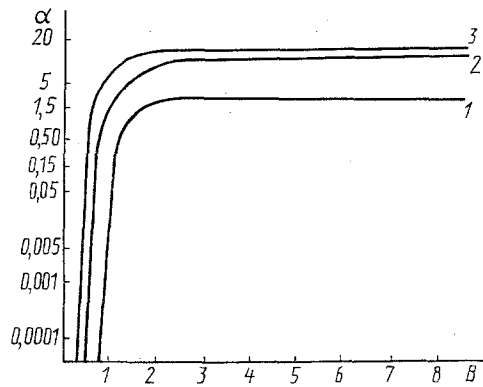


Fig. 1. Domain boundaries within which a 1% change in the phase shift $\delta_0(\omega_2)$ causes a change in thermal diffusivity by less than 3%: 1) $K = 2$; 2) 4; 3) 10; α , B , dimensionless.

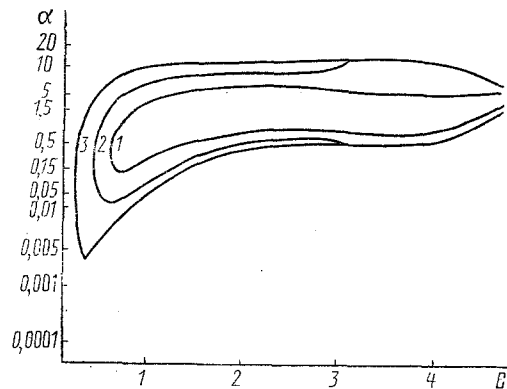


Fig. 2. Domain boundaries within which a 1% change of the phase shift $\delta_0(\omega_2)$ entails a change in ϵ/λ by less than 20%; 1) $K = 2$; 2) 4; 3) 10.

Also, it is seen that the domain of adequate α values is bound both from below and from above and narrows with an increase in the parameter B .

Analysis of the calculation results reveals that for combined determination of thermal diffusivity a and integral emissive power-to-thermal conductivity ratio ϵ/λ the following α and B ranges are most convenient:

$$\begin{aligned}
 2,3 \lesssim B \lesssim 3,8, \quad 0,75 \lesssim \alpha \lesssim 4, \quad K = 2, \\
 2 \lesssim B \lesssim 4, \quad 0,5 \lesssim \alpha \lesssim 8, \quad K = 4, \\
 1,9 \lesssim B \lesssim 4, \quad 0,4 \lesssim \alpha \lesssim 15, \quad K = 10.
 \end{aligned} \tag{8}$$

As it follows from (8), the main drawback of this method lies in relative narrowness of the ranges in which α and B can be varied.

The calculated α and B domains, within which TPPs are determined with an error less than that a priori given, make it possible to evaluate, provided specimen thermal conductivity λ is known, a temperature range applicable for measurements by formula (6). Indeed, having determined in Fig. 2 the α values at their minimum and maximum (at the given B and K), corresponding to an error of 20% in ϵ/λ determination, we may calculate (with the integral emissive power ϵ and specimen thickness l being known) that temperature range within which ϵ/λ is measured with an error less than 20%. For instance, for a substance with $\lambda = 1 \text{ W/(m}\cdot\text{K)}$ ($B = 1$; $\epsilon = 0.5$, $K = 2$; $l = 10^{-3} \text{ m}$) this range is $800^\circ\text{C} \leq t \leq 2600^\circ\text{C}$. Analogously,

we may evaluate that temperature range in which thermal diffusivity may be determined with an error less than 3% (with an error of 1% in $\delta_0(\omega_2)$ determination). For instance, for $\lambda = 10 \text{ W/(m}\cdot\text{K)}$, $B = 1$, $K = 2$, $\varepsilon = 0.5$, $l = 10^{-3} \text{ m}$ this range is $t \leq 3700^\circ\text{C}$.

Thus, measurement of phase shifts of a plane temperature wave at two frequencies makes it possible to exclude heat loss parameters and to determine thermal diffusivity for a wide class of substances (from metals to poor heat conductors) not only in the case of radiative heat transfer but also for other types of heat transfer. The most optimal ranges of α and B are the ranges specified in (7).

The proper choice of α , B , and K will lead to satisfactory accuracy of estimation of the ratio ε/λ . For all-round determination of thermal diffusivity and ε/λ , the ranges of (8) are most applicable. In all cases it is desirable to create the shift K of modulation frequencies as maximum as possible.

NOTATION

l , specimen thickness, m; ω , cyclic modulation frequency, rad/sec; a , thermal diffusivity, m^2/sec ; B , dimensionless parameter; $\delta_0(\omega_1)$ and $\delta_0(\omega_2)$, angles of phase shift at modulation frequencies ω_1 and ω_2 ; A_0 , A_l , α , r , β , dimensionless heat loss parameters; τ_1 , τ_2 , modulation periods, sec; $K = \mu^2$, ratio of modulation periods; Φ , heat flux from a specimen surface, W/m^2 ; λ , thermal conductivity, $\text{W}/(\text{m}\cdot\text{K})$; $T(0; 0)$ and $T(l; 0)$, mean temperatures of rear and heated sides, K ; B_0 and B_1 , first approximations for parameter B determination; ε , integral emissive power.

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