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Experimental Estimation of the Degree of Adiabaticity of a Sample when Measuring the Thermal Diffusivity by the Temperature Waves Method

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Abstract—A method for experimental determination of the degree of adiabaticity of a sample when measuring the thermal diffusivity by the temperature waves method is described. Analysis of the thermal diffusivity of ARMCO iron at different temperatures is performed as an example. It is experimentally proven that the two-dimensional thermal model gives an adequate description of the temperature-wave propagation in a sample of finite sizes.

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INTRODUCTION

The temperature waves method has been actively used for more than a half a century in scientific laboratories around the world as the most precise method for the analysis of thermophysical properties of condensed substances up to the highest temperatures [1–4]. An important specific feature of this method is its internal controllability, noted more than once by L.P. Filippov [2, 3]. The internal controllability is the possibility of using temperature waves of different frequencies. However, frequency is an important parameter that determines the applicability of the adiabatic approximation in the case of plane waves. Nowadays, there is technical feasibility of relatively simple experimental determination of conditions for the adiabatization of a sample and finding frequencies at which one-dimensional adiabatic theory of plane temperature waves can be used. The aim of the present work is to describe the corresponding method of analysis.

PLANE TEMPERATURE WAVES METHOD

The plane temperature waves method (PTWM) has gained wide application after the work by O.A. Kraev and A.A. Stel'makh [5]. In that work, the propagation of a temperature wave through an infinite plate of thickness l was considered. The wave was excited with the help of an amplitude-modulated heat flux directed normally to the plate and acting on the first plane surface of the plate. The heat flux was harmonically modulated. The corresponding calculation has shown in [4, 5] that φ, the phase shift of the temperature of the second surface of the plate measured relatively to the phase of the main flux acting on the first surface, depends on the thermal diffusivity a of the material of the plane and on parameters of the heat exchange with the ambient medium. The result can be represented in the form

$$a = \frac{\omega l^2}{k^2}, \tag{1}$$

where ω is the angular frequency of the temperature wave. The parameter k is the similarity number typical for temperature waves [2–5]. The number k has no explicit physical meaning and characterizes the phase incursion of the temperature wave in a sample. In the given problem, the parameter k is found by the following formula [4, 5]:

$$\varphi = \arctan\left(\frac{\mathrm{Bi}_1\mathrm{Bi}_2(b_1 - b_2) - 2\frac{k}{\sqrt{2}}(\mathrm{Bi}_1 + \mathrm{Bi}_2)b_4 - k^2(b_1 + b_2)}{\mathrm{Bi}_1\mathrm{Bi}_2(b_1 + b_2) + 2\frac{k}{\sqrt{2}}(\mathrm{Bi}_1 + \mathrm{Bi}_2)b_3 - k^2(b_1 - b_2)}\right), \tag{2}$$

where Bi_1 and Bi_2 are the Biot numbers characterizing the heat exchange of the first and second sample surfaces with the ambient medium [4, 5]. Parameters b_1, b_2, b_3, and b_4 are defined by following formulas:

$$b_1 = \sinh\left(\frac{k}{\sqrt{2}}\right)\cos\left(\frac{k}{\sqrt{2}}\right), \quad b_2 = \cosh\left(\frac{k}{\sqrt{2}}\right)\sin\left(\frac{k}{\sqrt{2}}\right),$$
$$b_3 = \cosh\left(\frac{k}{\sqrt{2}}\right)\cos\left(\frac{k}{\sqrt{2}}\right), \quad b_4 = \sinh\left(\frac{k}{\sqrt{2}}\right)\sin\left(\frac{k}{\sqrt{2}}\right).$$

It is seen that even the solution to the one-dimensional problem contains many parameters. Therefore, it is of practical interest to look for variants of an experiment in which the number of measured parameters is minimal.

In the first place, we may assume that the heat exchange at both surfaces of the plate is equal ($\mathrm{Bi}_1 =$

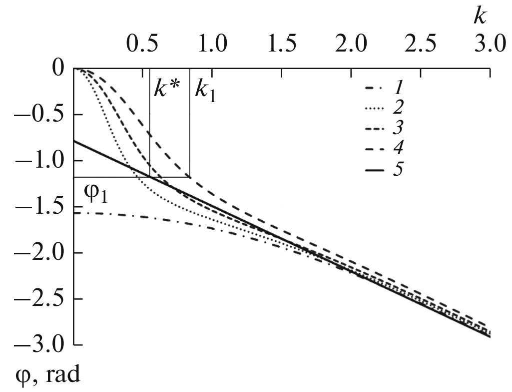

Fig. 1. Phase shift φ of temperature oscillations of the second surface of a plane sample vs. parameter *k* for Bi = (*1*) 0, (*2*) 0.05, (*3*) 0.1, and (*4*) 0.2, and (*5*) approximation (3).

$\mathrm{Bi}_2 = \mathrm{Bi}$). Then, it is easy to plot the dependence $\varphi(k)$ in which the heat exchange characteristic Bi plays the role of a parameter (Fig. 1). It follows from (1) and (2) that, at small phase shifts φ, small variations in the heat exchange parameter leads to large variations in *k*. However, with an increase in the frequency ν and, therefore, *k*, all dependences $\varphi(k)$ mutually approach (Fig. 1) the dependence corresponding to the adiabatic mode (Bi = 0) and group near it. From the practical point of view this result is very important, because it means that, with an increase in ν, it is less and less necessary to know the heat exchange parameters: the heat exchange does not influence results of estimation of thermal diffusivity. From the physical point of view, adiabatization takes place in the temperature waves method because with an increase in frequency, the temperature gradient in the sample increases, which leads to an increase in the fraction of the heat flux penetrating the sample [3].

The authors of [4, 5] suggested the approximation of $\varphi(k)$ by the formula (see Fig. 1)

$$\varphi = -\left(\frac{\pi}{4} + \frac{k}{\sqrt{2}}\right), \tag{3}$$

which at

$$k > 1.6 \tag{4}$$

describes dependences (2) with an error of 1% at

$$\mathrm{Bi} < 0.1. \tag{5}$$

This means that measuring the thermal diffusivity can be reduced to estimating φ and calculating the thermal diffusivity of a plate by (3) and (1) without regard for parameters of the heat exchange between the sample and the ambient medium. The frequency range corresponding to (4) is the range of the internal controllability of the PTWM.

Further, a two-dimensional problem was considered [6, 7], which, with increasing frequency, also reduces to one-dimensional adiabatic result (3). In this case, condition (4) should be replaced with

$$k > 2.5.$$

Then, formula (3) approximates the solution of the two-dimensional heat exchange problem with an error of 1%.

This result underlies the thermal diffusivity measuring method in the case when the lateral sizes of the heat flux acting upon the sample and the lateral sizes of the sample are bounded. The corresponding experiment conditions were established theoretically as a result of solving the heat equation and require experimental testing.

THE METHOD FOR EXPERIMENTAL ESTIMATION OF THE APPLICABILITY OF THE ONE-DIMENSIONAL ADIABATIC APPROXIMATION

The experimental estimation of the validity of the one-dimensional adiabatic model is based on measuring the thermal diffusivity, using temperature waves of different frequencies. The idea of the method can be explained if we regard the dependence $\varphi(k)$ presented in Fig. 1. In the experiment, phase φ is measured. Let's assume that, at sufficiently low frequencies of temperature waves, shift φ_1 is detected (see Fig. 1). Let's also assume that the heat exchange is characterized by value Bi = 0.2. Originally, this value is unknown. Therefore, *k* is estimated from adiabatic dependence (3), represented by curve *5* in Fig. 1, by value *k**. This value is smaller than true value k_1 (see Fig. 1) that could be found if the value of Bi was known. Therefore, the value a^* that is calculated by (1) will be higher than the true thermal diffusivity of the sample, a_1.

If the heat exchange of the sample with the ambient medium is not strong, then, possibly, *k** will be higher than the true value (for example, if Bi = 0.05; see Fig. 1).

With increasing frequency of the temperature wave, the difference between *k** and k_1 will decrease and, therefore, a^* will approach true value a_1. When the frequency of the temperature wave is sufficiently high, estimated value of a^* within the experimental error will coincide with a_1: $a^* \approx a_1 = \mathrm{const}$ (the range of internal controllability). The value of the thermal diffusivity that is calculated by (1) will be independent of the frequency of the wave and, it will be possible to infer in which mode the variations proceed from the dependence $a(\omega)$. If the values of the thermal diffusivity are frequency-independent, then the adiabatic regime takes place; if there is a frequency dependence, the adiabatic regime has not been established yet.

EXPERIMENTAL RESULTS

Experimental tests on establishing the adiabatic regime was performed on ARMCO iron. We have reliable data about it's properties known from publications [8, 9]. Measurements were performed on a device with laser excitation of the temperature wave [4]. The ILGN-701 (LG-43) continuous laser had the operating emission wavelength of 10.6 μm and a power of 60 W. In the given case, it is important that the device employ a highly stable amplitude modulator of laser radiation making it possible to vary the frequency of the temperature wave by means of software [10–12]. Time to switch among the range of wave's frequencies did not exceed 2 s, which made it possible to perform measurements for a comparatively short time in a wide range of frequencies of temperature waves at a practically constant sample temperature.

The sample had the form of a cylinder with a diameter of 10 mm and a height of 0.935 mm. Amplitude-modulated laser radiation was incident onto a front plane surface of the sample. A VR5/20 thermocouple with electrodes 50 μm in diameter was welded to the sample near the central point of the opposite (back) surface. Each electrode was butt-welded to the sample independently without globule formation [13]. This thermocouple was also applied for measuring the mean sample temperature and oscillations of the sample temperature. An FD-3A photo-diodeaimed at the central part of the back surface of the sample was used to assess oscillations of the sample's temperature. Thus, with the help of a thermocouple and a photo sensor, the phase φ of temperature oscillations of the second surface could be measured and, thereby, the thermal diffusivity could be found.

The sample was placed into a vacuum chamber preliminary vacuumed to a pressure of 10^{-3} Pa; then the chamber was filled with 6.0 grade helium to an excess pressure of 10^5 Pa. The mean sample temperature was varied with the help of a special heater: an electric resistance furnace.

The experiment was conducted as follows: the sample was heated to a certain temperature, which later was maintained constant. Then the phase φ of the temperature oscillations of the back surface of the sample was evaluated at several frequencies of temperature waves. Frequencies were varied with a modulator in the range from $\nu_1 = 2$ to $\nu_2 = 20$ Hz with a step of 1 Hz. Then the thermal diffusivity was calculated by formulas (1) and (3).

Figure 2 shows the results of the analysis of thermal diffusivity of iron at $\Theta_1 = 390$ K. According to reference data [8], the thermal diffusivity of iron at this temperature is $a_1 = 18 \times 10^{-6}$ m^2/s. At this temperature, the signal from the photo sensor is very weak; therefore, the thermal diffusivity was estimated only from the thermocouple data. The experiment has shown that, at low frequencies of temperature waves, the calculation by formulas (1) and (3) gives overestimated values of thermal diffusivity. With an increase in the frequency, the calculated value decreases and, at $\nu = 7$ Hz, settles at the level close to the reference one. The further increase in the frequency does not lead to variation in the calculated value of thermal diffusivity. With a further increase in frequency the amplitude of the signal decreases so that the results of calculation of thermal diffusivity lose stability.

Figure 2 presents the dependence of the parameter k. As we see from the figure, this parameter at the frequency $\nu = 7$ Hz equals to 1.6. Measurements of thermal diffusivity at higher frequencies is accompanied with a higher value of k. In this case, in the given frequency range, $a \approx$ const, which means that conditions for the adiabaticity have been reached. Generally speakeing, the obtained results agree with the corresponding theoretical assessment of the adiabatic approximation's applicability [6, 7].

Temperature, Θ_1, corresponds to relatively low temperatures at which the radiative heat transfer is not very intense. It is of greater interest to estimate the frequency dependence of thermal diffusivity at higher temperatures. Figure 3 presents the results for $\Theta_2 = 1536$ K. The reference value in this case is $a_2 = 7.3 \times 10^{-6}$ m^2/s. At the given temperature, thermal diffusivity was measured by both thermocouple and photo sensor. At Θ_2 the calculated values of thermal diffusivity at low frequencies were relatively high. However, with increasing fre-

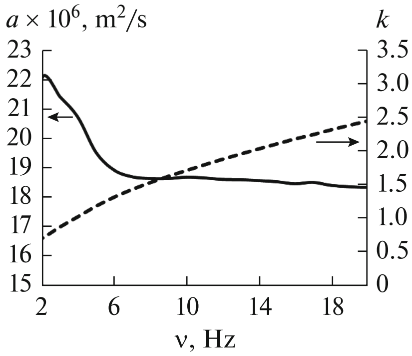

Fig. 2. Results of measurements of thermal diffusivity of ARMCO iron at different frequencies at 390 K.

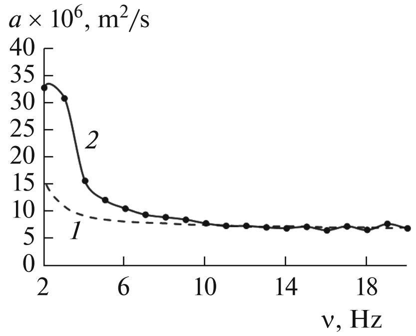

Fig. 3. Results of measurements of thermal diffusivity of ARMCO iron at different frequencies at 1536 K by (*1*) a thermocouple and (*2*) a photo sensor.

quency, the calculated values of a decreased and, at $\nu = 10$ Hz, stabilized close to the reference value level.

Thus, with an increase in the frequency, the system passes to the adiabatic regime and the parameters of heat exchange do not influence the results of thermal diffusivity measurements. Indeed, the experimental values of frequencies (and parameter k) of temperature waves corresponding to the adiabatic regime proved to be close to values given by the two-dimensional model [6, 7]. In other words, the model considered in those works adequately describes the propagation of temperature waves in real systems. Such a model was used for measuring the high-temperature thermal diffusivity of intra-rare-earth alloys [14].

The results of measurements with a thermocouple and a photo sensor up to the frequency of 20 Hz are in a good agreement with one another (Fig. 3). It should be noted that the thermocouple is an inertial element of the thermal system, thus with increasing frequency, the phase delay of the thermoelectric signal must increase. This should lead to the value of thermal diffusivity calculated by (1) reduction, However, in the frequency range below 20 Hz, this phenomenon is not observed. The question of thermocouple sensors applicability for temperature oscillations measurements has been discussed in literature more than once. The theoretical estimation made by L.P. Filippov evaluates the upper threshold frequency for a thermocouple with a diameter of 50 μm of about 10 Hz [2]. Our measurements have shown that, when measuring the thermophysical properties of iron, this thermocouple can also be used at higher frequencies up to 20 Hz.

CONCLUSIONS

The two-dimensional thermal model [6, 7] gives us an adequate description of the process of temperature-wave propagation in a sample of finite sizes. At sufficiently high frequencies of temperature waves, it becomes possible to apply an adiabatic variant of the PTWM for measuring thermal diffusivity. The results obtained in [6, 7] can serve as a basis for an operating procedure for measuring the thermal diffusivity of condensed bodies at high temperatures.

When measuring thermal properties of metals, e.g., iron, a thermocouple with a diameter of 50 μm welded to a sample without junction can be used to measure the phase of temperature oscillations in a sample in a range of frequencies from 0 to 20 Hz.

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REFERENCES

1. Drabble, J.R. and Goldsmid, H.J., *Thermal Conduction in Semiconductors*, Oxford: Pergamon, 1961.
2. Filippov, L.P., *Izmerenie teplovykh svoistv tverdykh i zhidkikh metallov pri vysokikh temperaturakh* (Measurement of the Thermal Properties of Solid and Liquid Metals at High Temperatures), Moscow: Mosk. Gos. Univ., 1967.
3. Filippov, L.P., *Izmereniya teplofizicheskikh svoistv veshchestv metodom periodicheskogo nagreva* (Measurements of Thermophysical Properties of Substances by the Method of Periodic Heating), Moscow: Energoatomizdat, 1984.
4. Ivliev, A.D., *High Temp.*, 2009, vol. 47, no. 5, p. 737.
5. Kraev, O.A. and Stel'makh, A.A., in *Issledovaniya pri vysokikh temperaturakh* (Research at High Temperatures), Novosibirsk: Nauka, 1966, p. 55.
6. Pozdeev, A.N., Ivliev, A.D., Kurichenko, A.A., and Morilova, L.V., *Inzh.-Fiz. Zh.*, 1987, vol. 52, no. 5, p. 856.
7. Morilov, V.V., Ivliev, A.D., and Pozdeev, A.N., *J. Eng. Phys.*, 1990, vol. 59, no. 2, p. 1026. 8. 1, p. 636.
8. *Selected Values of the Thermodynamic Properties of the Elements*, Metals Park, OH: Amer. Soc. Metals, 1973.
9. Zinov'ev, V.E., *Teplofizicheskie svoistva metallov pri vysokikh temperaturakh. Spravochnik* (Thermophysical Properties of Metals at High Temperatures: Handbook), Moscow: Metallurgiya, 1989.
10. Vekshina, O.A., Vekshin, I.M., Kurichenko, A.A., and Ivliev, A.D., *Meas. Tech.*, 2010, vol. 53, no. 6, p. 668.
11. Meshkov, V.V., and Ivliev, A.D., in *Teplofizicheskie issledovaniya i izmereniya v energosberezhenii pri kontrole, upravlenii i uluchshenii kachestva produktsii, protsessov i uslug. Mater. VII Mezhdun. teplofiz. shkoly* (Thermophysical Studies and Measurements in Energy Conservation in the Control, Management, and Improvement of the Quality of Products, Processes, and Services: Proc. VII Int. Heat-Phys. School), Tambov, 2010, vol. 2, p. 56.
12. Meshkov, V.V., and Ivliev, A.D., in *Mater. Mezhdun. nauch.-tehn. konf. "Sovremennye metody i sredstva issledovanii teplofizicheskikh svoistv veshchestv"*(Proc. Int. Sci.-Tech. Conf. on Modern Methods and Means of Research of Thermophysical Properties of Substances), St. Petersburg, 2010, p. 84.
13. Kulakov, M.V. and Makarov, B.I., *Izmerenie temperatury poverkhnosti tverdykh tel* (Measurement of the Surface Temperature of Solids), Moscow: Energiya, 1979.
14. Ivliev, A.D., Kurichenko, A.A., and Vekshin, I.M., *High Temp.*, 2016, vol. 54, no. 2, p. 206.

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