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Method and Mounting for Solid and Liquid Matter Thermal Diffusivity Investigations at High Temperatures[1]

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ABSTRACT

We present a description of a new method for condensed matter thermal diffusivity measurements. The method theory is based on the analysis of temperature waves propagation in the restricted bodies. The appropriate two-dimensional heat-conduction equation is solved within the framework of a linear approximation. The analytic solution received pointes out the relations between the thermal diffusivity of the investigated matter and the ocsillation phase of the surface temperature at the periodically heated field of the sample. We have shown, that this method can be used for matter thermal diffusivity measurements at the temperatures of a few thousands kelvin.

A new mounting for solid and liquid matter thermal diffusivity measurements has been elaborated on the basis of our method. The temperature wave in the sample is excitated by the amplitude modulated laser radiation. The sample's mean temperature is varied within the limits 300 - 1200 K with the help of a special heater. Sample's surface temperature ocsillations are transformed to an electrical signal by means of a photo-sensor. The signal oscillations phase shift with the attitude to the laser radiation amplitude oscillations is measured with the help of an original device, using quasi-optimal filtration.

KEY WORDS: heat-conduction equation, laser, liquid metal, quasi-optimal measuring instrument for signal parameters, solid metal, temperature-wave experimental method, thermal diffusivity.

1. INTRODUCTION

Thermal diffusivity $a(T)$ is an important thermophysical characteristic of matter. Its high-temperature measurements have as a rule lesser errors, than the heat-conduction ones. Therefore nowdays such measurements are carried out at a lot of laboratories. However it should be noted that only solid matter thermal diffusivity measurement methods are elaborated sufficiently enough, whereas there are no special methods for liquid matter thermal diffusivity measurements, appropriate for liquid metals investigations. Earlier only in the works, carried out under the leadership of Philippov and Novikov [1], liquid metals thermal diffusivity measurements have been realized. The measurements have been executed with the help of a method of radial temperature waves, propagating through the system: the first vertical side of the crucible, metal investigated, the second vertical side of the crucible. In order to decrease a natural convection intensity, there are horizontal tantalum plates in the space between the crucible sides. Such a sample manufacturing is very labour-consuming. A problem how to account the influence of the sample's construction features and its sizes instability on the results of the thermal diffusivity measurements, is also complicated. The sample thickness being large enough(6 mm), it is necessary to take into account the corrections caused by a radiative heat exchange. This method has also some another defects, but one should have in mind, that there have not been another means of measurements and that the first liquid metals thermal diffusivity measurement results have been obtained with the help of this method.

We present a new method for thermal diffusivity measurements, where the crucible sides are not elements of the heat scheme. This method can be success-

fully used for liquid matter thermal diffusivity measurements, including liquid metals investigations.

2. METHOD THEORY

Let on the centre of the plate surface of the sample, being a half-restricted cylinder with radius R(see Fig.1), placed into the camera with an inert gas, the modulated heat flux falls with radius b and surface density

$$q = \overline{q} + q_0 \exp(i\omega t),$$

where $\overline{q}$ is the constant component, q_0 - an amplitude of the variable component, ω - modulation frequency (temperature wave frequency), t - time. The heat flux excitates in the sample a temperature wave

$$T(r,z,t) = \overline{T}(r,z) + \Theta(r,z)\exp(i\omega t),$$

where r, z – current coordinates, counted off the central point of the sample surface, being influenced by the heat flux, $\bar{T}(r,z)$ - a constant component, $\Theta(r,z)$ - an amplitude of a variable component of the sample temperature.

Supposing, that sample's thermophysical characteristics don't depend on its coordinates and temperature, the temperature field in the sample is described by the following equation:

$$a\left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r}\frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2}\right) = \frac{\partial T}{\partial t}$$

with the boundary conditions:

$$\begin{cases} z=0: \quad -\lambda\dfrac{\partial T}{\partial z}+\sigma\epsilon\delta(T^4-T_1^4)+\dfrac{\lambda_c(T-T_1)}{L_1}=\begin{cases} q\,,\ 0\le r\le b\,, \\ 0\,,\ b<r\le R\,, \end{cases} \\ r=R: \quad \lambda\dfrac{\partial T}{\partial r}+\sigma\epsilon\delta(T^4-T_2^4)+\dfrac{\lambda_c(T-T_2)}{L_2}=0, \\ z=\infty: \quad T=\mathrm{const}(t)\,, \\ \lambda=\mathrm{const}(T)\,, \end{cases}$$

where a, λ - thermal diffusivity coefficient and heat conduction coefficient respectively, L_1 and L_2 - the distances from the sample surfaces to the nearest internal surfaces of the camera.

In approximation considered, the problem divides into two ones - for $\bar{T}$ and for $\Theta(r,z)$. Later we'll examine only quasi-stationary problem for the variable component of the temperature wave in the sample $\Theta(r,z)$. The approximation $\Theta(r,z) \ll \bar{T}(r,z)$ allows us to linearize corresponding boundary conditions, neglecting the terms, little in comparison with $\sim(\bar{T}^3\Theta)$.

And so there is the following problem:

$$\frac{\partial^2\Theta}{\partial r^2}+\frac{1}{r}\frac{\partial\Theta}{\partial r}+\frac{\partial^2\Theta}{\partial z^2}-\frac{i\omega}{a}\Theta=0\,,$$

$$\begin{cases} z=0: \quad -\dfrac{\partial\Theta}{\partial z}+\alpha_1\Theta=f(r)=\begin{cases} q_0/\lambda\,,\ 0\le r\le b\,, \\ 0\,,\ b<r\le R\,, \end{cases} \\ r=R: \quad \dfrac{\partial\Theta}{\partial r}+\alpha_2\Theta=0\,, \\ z=\infty: \quad \Theta=0\,. \end{cases}$$

Solution, received by the variables separation method [2,3], shows, that the shift of the sample's plate surface temperature oscillations phase with the attitude

to the heat flux oscillations phase depends on the coordinates of the surface point, sample's thermal diffusivity, sample sizes, heat flux radius and the value of heat exchange of the sample and the camera internal sides.

In spite of a lot of parameters unknown, it is principally possible to simply determine the sample's thermal diffusivity in the total case. In this purpose it is necessary to carry out a few measurements at various temperature waves frequencies, and then to solve appropriate system of equations. This approach is inconvenient technically: each measurement is accompanied by the error, and if you want the thermal diffusivity measurement error to be admissible, you need to provide a very high precision for measurements on the each ω.

To reduce the number of such measurements required and therefore to simplify the calculation procedure and to increase the precision, we have analysed the each problem parameter influence on the temperature wave parameters at the plate surface of sample ($z = 0$). So, at the sufficiently little neighbourhood of the central point of the sample's plate surface, temperature wave can be treated as an isophase (that is plate), thus in this space the wave parameters dependence on the radius r may be neglected.

Real sample has limited sizes, being not half-unrestricted, but, as the analysis has shown, if the height of sample is larger, than its diameter, the restricted cylinder can be considered as a half-unrestricted. And what is more, if the sample diameter is 2-3 times more than the heat flux diameter, parameter R is not important and the sample can be treated as a half-unrestricted body. Under these conditions the problem decision has insignificant dependence on the heat exchange value up to the the temperatures of a few thousands kelvin. This

decision can be approximated by the expression:

$$\varphi = \operatorname{arctg} \frac{\sin(b\beta/\sqrt{2}) + \cos(b\beta/\sqrt{2}) - \exp(b\beta/\sqrt{2})}{\sin(b\beta/\sqrt{2}) - \cos(b\beta/\sqrt{2}) + \exp(b\beta/\sqrt{2})},$$

where $\beta = \sqrt{\omega/a}$.

There are two measured parameters in this expression: φ - temperature oscillation phase shift of the central part of the sample's plate surface with respect to the heat flux oscillations, and b - the heat flux radius. By means of this formula thermal diffusivity can be calculated with the methodical error no more than a few procents. In Fig.2 $\varphi(\beta)$ dependence graphs for some possible values of b are shown.

The analysis of the measurements methodical errors has shown, that one of the essential contributies to these errors is due to the b -measurements error. This parameter control is technically complicated, therefore it is more convenient to measure by this method relative thermal diffusivity (its temperature dependence). During these measurements it is necessary for the value of b to be constant. Then the absolute values of $a(T)$ should be obtained from the well-known exact enough thermal diffusivity value for one of the points of the temperature interval investigated. As a rule, there is such a point at least for the solid phase of the investigated matter.

The heat conduction problem statement mentioned above is valid, strictly speaking, only for solids, as the corresponding equations for liquid samples should take into account the convection currents appearance possibility. However it is possible, that the convection does not appear. The axis z in Fig.1 coinciding with the gravity force direction, the sample's liquid matter is in the stable equilirium,

if there is no convection [4], that is the following inequality is executed:

$$\frac{\partial T}{\partial z} \leq 0 .$$

If the half-unrestricted sample surface temperature is varied according to the law of simple harmonic oscillations, this inequality provides the following estimation for the liquid sample temperature wave amplitude, preventing the appearance of the natural convection in the sanple:

$$|\Theta(r,z)| \leq \sqrt{\frac{2a}{\omega}} \left| \frac{\partial T(r,z)}{\partial z} \right| .$$

The measurements carring out, convection can appear also due to the reasons don't take into account above (for example, according to heterogeneity of the temperature field in the camera, created by the heater changing average temperature of the sample). It is advisable to analyse such processes for each concrete problem, not in the whole. It can be noted, however, that the temperature-wave method allows us to take into account the convection influence, carring out the measurements under different frequences, and to eliminate this influence.

3. EXPERIMENTAL MOUNTING

In Fig.3 the block-scheme of the new experimental mounting is shown. CO_2-laser radiation (radiating power 40 W) **1** is converted by a mechanical modulater **2** into a right-angled pulses sequence, then passes throuth a collimator **11**, and, being reflected from a mirrow **12**, has an influence on the sample surface **4**. The sample is placed to a vacuum camera **5**, provided with a system of vacuum pumps and an inert gas feeding **7** as well as with a system of heater power supply **6**, varying the sample's mean temperature. The heat radiation of the central part of the sample surface, having passed throuth its own collimator (which is not

shown in Fig.3), is converted with the help of a photodetector **8** into the electrical signal. Variable part of this signal parameters are estimated in a meter **10**, realizing quasi-optimal filtration. The device **3** locks the work of the modulator and meter. Sample's mean temperature is measured by a thermocouple **9**.

The heat flux radius b with the help of the collimator **11** can be varied within the limits (1.5-3) mm. The sample radius R 1s (5-8) mm, its height - (15-20) mm. Temperature wave frequency can be changed within the limits (2-35) Hz, permitting to investigate the matters with thermal diffusivities ranged $(2-90)\cdot 10^{-6} m^2/s$. The maximum sample temperature in the camera is 2100 K.

The measurement error analysis has shown, that relative thermal diffusivity measurement error at this mounting does not exceed 7%, resolution capacity being 5%. It should be mentioned, that there are some reserves of measurements precision increasing at this mounting.

With the help of elaborated mounting measurements of the thermal diffusivity of solid and liquid iron, steel and rare-earth metals has been carried out. Within the limits of measurement error our results coincide with the reliable literary data available. This fact confirms the validity of the approximations used when this measurements method was elaborated.

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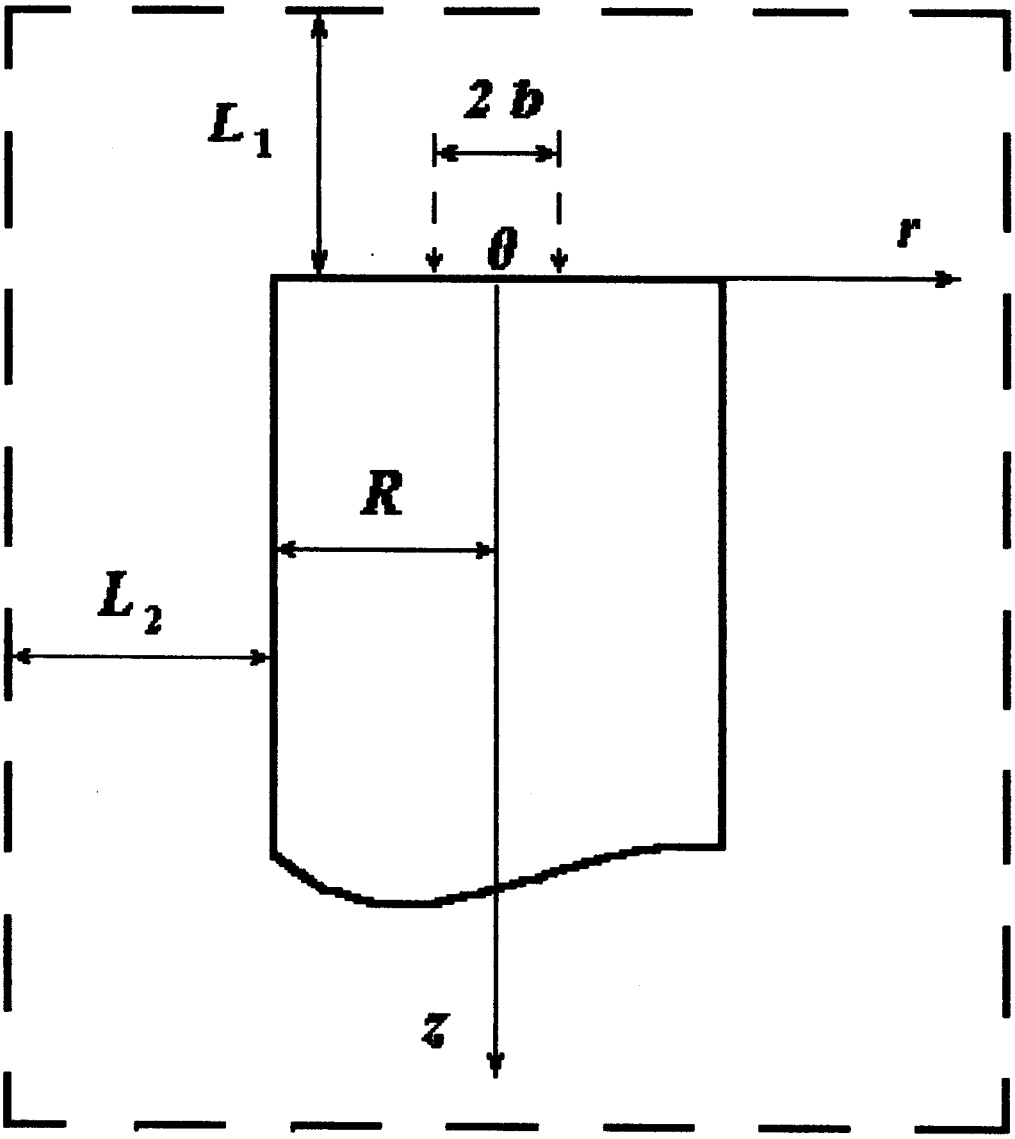
FIGURE CAPTIONS

Fig.1 To the heat conduction problem statement.

Fig.2 $\varphi(\beta)$ dependence under various b.

Fig.3 Solid and liquid matter thermophysical characteristics investigation mounting block-scheme.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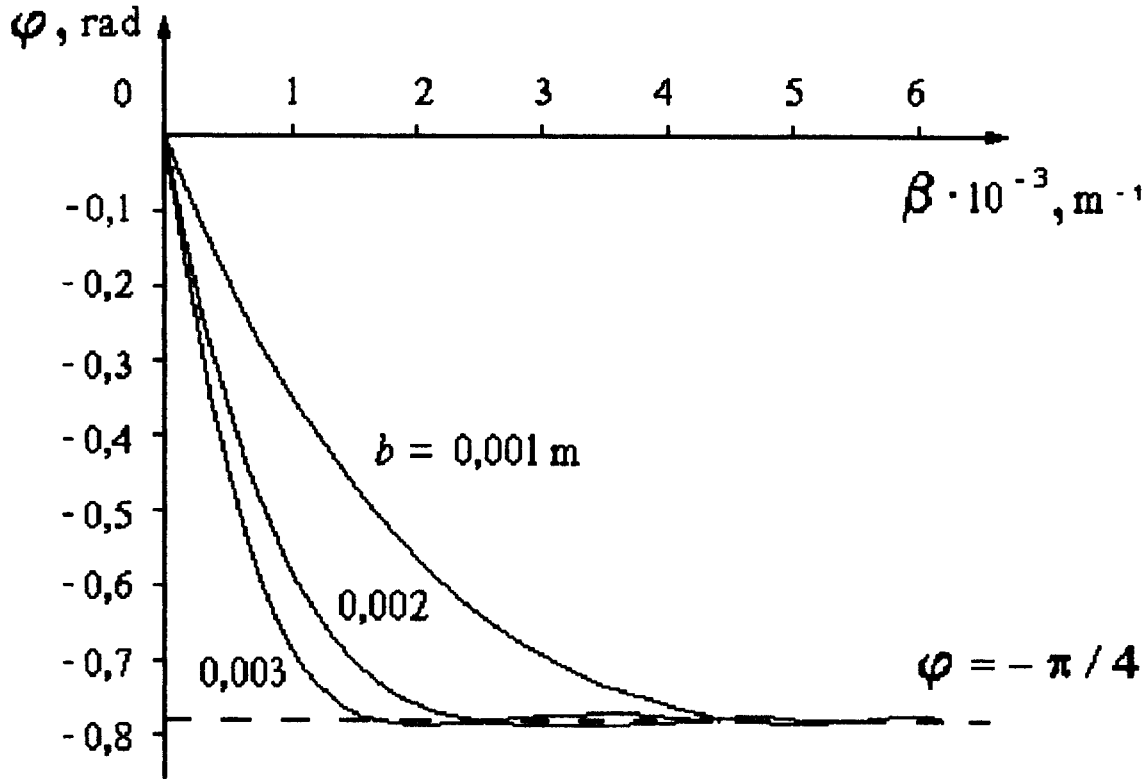
φ, rad
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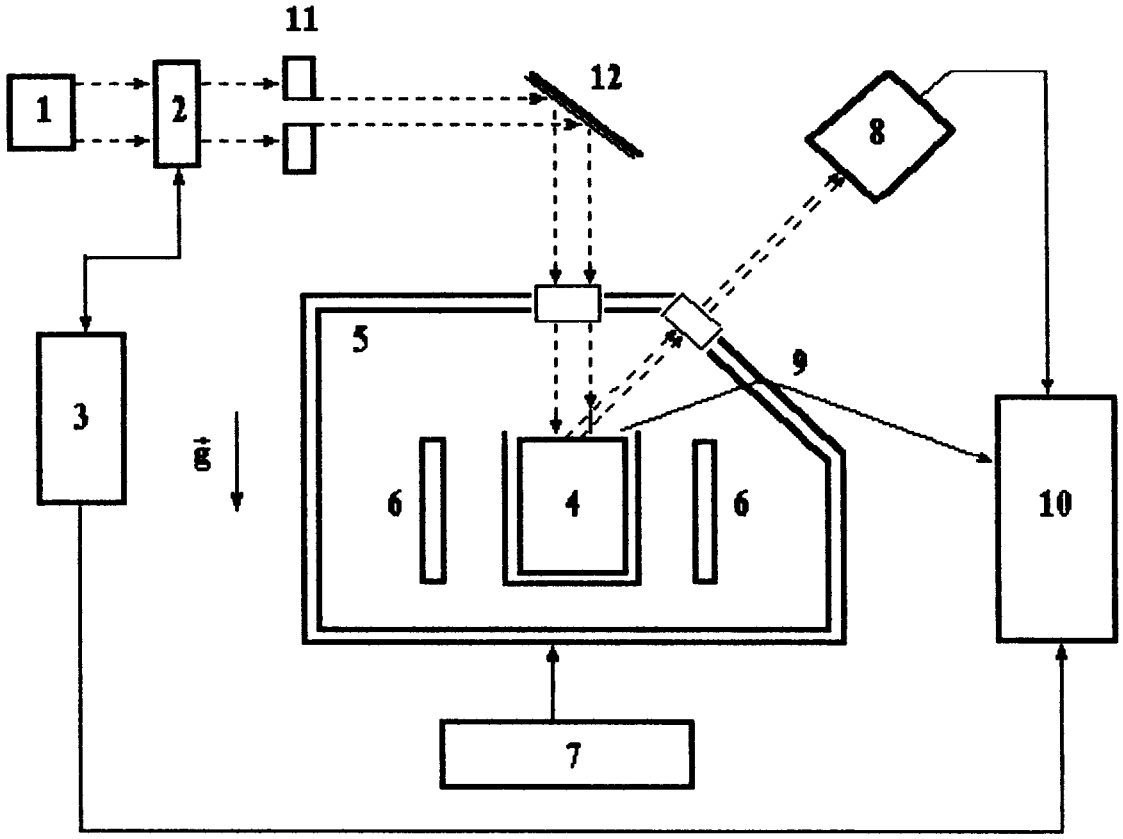
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