

METHODS OF SIMULTANEOUS MEASUREMENT OF HEAT CONDUCTIVITY, HEAT CAPACITY AND THERMAL DIFFUSIVITY OF SOLID AND LIQUID METALS AT HIGH TEMPERATURES

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Аннотация—Изложен опыт работы в направлении создания комплексных методов определения теплофизических свойств твердых и жидких металлов при высоких температурах. Описан метод продольных температурных волн, метод радиальных температурных волн и метод нагрева тонких проволок.

NOMENCLATURE

a ,	thermal diffusivity;
λ ,	heat conductivity;
c ,	specific heat capacity;
ρ ,	density;
α ,	heat-transfer coefficient;
σ ,	specific resistance;
T ,	temperature of sample;
T' ,	ambient temperature;
θ ,	temperature oscillation amplitude;
φ ,	temperature oscillation phase;
ω ,	circular frequency of oscillations;
R ,	radius of cylindrical sample;
S ,	cross-section of sample;
M ,	mass of sample;
Q ,	variable component of electronic heating power;
I ,	current intensity.

THE INTEREST shown by science and engineering in high-temperature studies of thermal properties of various materials justifies the attention which has recently been paid to the improvement of the existing experimental techniques and to the development of new methods of measurement. Of special interest are the investigations which deal with measurements of the whole set of main thermal parameters, i.e. heat conduc-

tivity, heat capacity and thermal diffusivity rather than with one property only. The study of the above set of parameters of one and the same object under similar conditions greatly shortens the measurement procedure and allows mutually co-ordinated and sometimes mutually controllable data to be obtained. Specially useful are the methods which allow the determination of a set of the parameters from the data of one experiment.

The present paper deals with the experience on development of simultaneous measurement methods accumulated at the Physical Department of the Moscow University. These investigations are included in the work of studying the thermal properties of solid and liquid metals at high temperatures [1].

The discussion starts with the methods which allow the set of properties of the material under investigation to be obtained from the results of one experiment. These methods are based on a great bulk of information which can be obtained and which is greater than that when only one parameter is measured. In this respect it is especially reasonable for measurements of a steady periodic process to use the so-called regular thermal regime of the third kind since it is this regime which gives a greater range of

information as compared to the steady-state and other transient regimes. In this case, indeed, the information sources are as follows: the constant component of temperature (mean temperature), amplitudes of temperature oscillation, phases of these oscillations, the frequency dependence of the amplitudes and phases for the case of harmonic oscillations or frequency structure of a composite periodic signal. The large body of information obtainable with the help of such a thermal regime makes it possible to perform various measurements including those which allow an internal cross-check because of the surplus of information [2]. In addition periodic processes have another advantage, namely multiple reproducibility of measurements.

It follows from the general consideration that a set of thermal properties can be measured only if in an experiment, in which space-time temperature variations are measured, at least one value with energy dimensions is also determined. These are the heat-transfer coefficient, power of an internal heat source, heat flux density (or the density of the energy source on the surface).

First let us discuss the methods based on surface heating.

A rather accurate measurement of heating power is possible when heating is produced by electron bombardment. This method of heating allows the power supplied to the sample to be determined directly by the electron current intensity and anode voltage.

Variable surface heating of samples at regular stages of the process leads to setting up of a periodic thermal regime when the temperature variation at each point of the body is purely periodic, and, proportionally to the distance from the heated surface, the phase of the temperature oscillation lags behind that at the surface. The amplitude of the oscillations also decreases. The oscillation process of this kind is similar to wave propagation and is, therefore, called temperature wave (though strictly speaking the process is not a wave since there is no wavefront nor directed energy flux when the temperature wave is propagating).

Plane, cylindrical and spherical waves can be distinguished depending on the symmetry of the isotherms of a temperature wave. Each type of these waves can be used to measure thermal parameters [1, 2]. For the present the first two types are used for simultaneous measurements.

The method of plane temperature waves, developed by the author in co-operation with A. N. Nurumbetov, uses electron heating of the bar base made of the metal to be studied. The temperature wave which is propagating along the bar axis can be practically considered as a plane one, since radial temperature gradients are relatively low. In accordance with this, the theory of axial waves is based on the solution of one-dimensional heat-conduction equation:

$$a \frac{\partial^2 T}{\partial x^2} = \frac{\partial T}{\partial t} + v(T - T'), \quad (1)$$

The application of the solution of equation (1) for definition of the thermal diffusivity of solids is well known and is the basis of the Ångström method of temperature waves and its variations.

Since in equation (1) two values a and v are unknown, v must be eliminated from the solution of (1) when determining the thermal diffusivity. This can be achieved by using the results of measurements (1) both of the amplitudes and phases of temperature oscillations or (2) the amplitudes at various frequencies or (3) the phases at various frequencies. However there is one more possibility of eliminating v , namely the use of such regimes of temperature waves when the term of the external heat transfer in equation (1) serves only as a small correction. These conditions can be satisfied for temperature waves with relatively short periods [3, 4]. In this case thermal diffusivity of the material can be determined independently from the ratio of temperature oscillation amplitudes at two points

$$a = \frac{l^2 \omega}{2[\ln(\theta_1/\theta_2)]^2} \quad (2)$$

or from the phase difference of these oscillations

$$a = \frac{l^2 \omega}{2(\Delta\phi)^2} \quad (3)$$

where l is the distance between the points, ω is the cyclic frequency of oscillations (the amplitude of temperature oscillations at the second end of the bar is considered to be very low).

Thus, to define thermal diffusivity we need half the amount of information required in the usual variants of this method. On the other hand the excess information (information about amplitudes and phases) can be used for the internal control of the experiment. The method of short temperature waves has also some other advantages, in particular, the length of the bar under investigation can be here much shorter than usually employed, i.e. some 3–5 cm. In the work described, this particular thermal regime is used.

As mentioned above, heat conductivity can be determined simultaneously with thermal diffusivity when the amount of heat (its variable component) supplied to the sample base is known. Then heat conductivity can be determined by the formula

$$\lambda = \frac{Q \exp[-\sqrt{(\omega/2a)l}]}{\sqrt{(\omega/a)} \pi R^2 \theta} \quad (4)$$

where Q is the amplitude of the variable component of heating power; θ is the amplitude of temperature oscillations at a point which is at a distance of l from the heating end. Heat conductivity can be thus determined by the method of axial temperature waves if the power and the amplitude of temperature oscillations for one point of the sample are known.

From the corresponding boundary condition in addition to formula (4), formula (5) is obtained for phase difference between periodic power variation and temperature oscillations at the same point of the sample at a distance l from the end of the bar.

$$\Delta\varphi = (\pi/4) + \sqrt{(\omega/2a)l}. \quad (5)$$

This formula can be applied instead of (3) for the determination of thermal diffusivity. Formulae (4) and (5) thus allow two values a and λ to be determined from the readings of one temperature probe, i.e. of a thermocouple placed at a

certain distance from the heating end of the sample being examined. The use of a great number of thermocouples gives excess information which can be used for internal control of the results.

The arrangement used for the study of the set of thermal parameters by the method of axial temperature waves is similar to that described in references [4] and [5] and we shall give here only a brief summary. A sample of 5–15 mm dia. and a few centimetres long is placed in the vacuum chamber along the axis of the cylindrical heater. An electron-emitting cathode is placed opposite the upper base of the sample. Negative potential of several hundred volts is applied to the cathode. The sample under investigation serves as the anode, its potential being zero (the sample is grounded). The anode-cathode voltage is periodically switched on and off by a special modulating device; the periods of modulation are within the range of 5–20 s. A Chromel–Alumel thermocouple serves as a temperature-sensing element, its leads being welded to the sample surface at a distance of several millimetres from the base. To obtain additional information about space-time temperature distribution, several temperature probes, i.e. constantan or Alumel leads about 3 mm apart are welded to the surface of the sample. A common thermoelectrode made of the same wire is welded to the lower end of the sample. Each temperature probe in pair with this electrode makes a differential thermocouple with the sample itself as an intermediate metal. Since there are practically no temperature oscillations on the lower end of the sample, these differential thermocouples register temperature oscillations at each point where the probes are welded. The set of thermocouples and temperature probes is surrounded by a screen protecting them from the anode current.

The constant component of thermoelectromotive force of temperature probes is compensated and measured by means of a potentiometer scheme, the variable one is applied to the entry of a photoelectric amplifier, the signal

from the exit of which is registered by a self-recording potentiometer. On the diagram tape of this potentiometer are also recorded the moments of switching on and off of the anode voltage. The curves of temperature oscillations are treated by the methods of harmonic analysis. The reproducibility of measurements of temperature oscillation amplitude (the first harmonics) is about 1 per cent, the error of phase determination ranges from 0.5 to 1 degC.

The investigation of regular sources of errors due to the deviations of the experimental conditions from the ideal ones (when the above formulae are valid) which includes the determination of corrections for heat transfer from the lateral surface of the sample, estimation of the error due to non-uniformity of the base heating and distortion of the temperature field by the thermocouples, leads to the conclusion that the role of all these factors is small and that they can be accounted for by small corrections.

From the analysis of the measurements [5] one can conclude that the maximum error in the determination of thermal diffusivity in phase variant of the method is 4–8 per cent when the readings of one thermocouple are used (Equation 5).

The obtained result can be more accurate when the readings of several temperature probes are used. In this case thermal diffusivity can be determined by the modified formula (3)

$$a = \frac{\omega}{2(d\varphi/dl)^2} \quad (6)$$

where $d\varphi/dl$ is the tangent of the angle of slope of the straight line $\varphi(l)$. The value $d\varphi/dl$ can be usually defined accurate to 2 per cent which allows determination of thermal diffusivity within an accuracy of about 4 per cent.

Similarly the information can be used relevant to the oscillation amplitudes at the points of probe location. Here thermal diffusivity is found from modified formula (2)

$$a = \frac{\omega}{2(d \ln \theta/dl)^2} \quad (7)$$

$d \ln \theta/dl$, the tangent of the slope angle of the

corresponding straight line can be defined within the accuracy of about 2 per cent which gives the error in thermal diffusivity determination of about 4 per cent. Figures 1 and 2 illustrate the

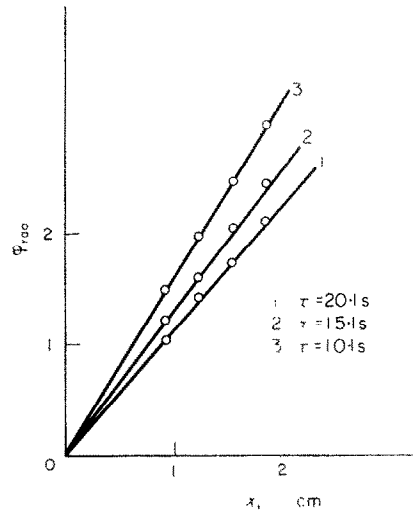


FIG. 1. Experimental dependence of phase difference between temperature oscillations and variation of heating power on the position of thermocouples.

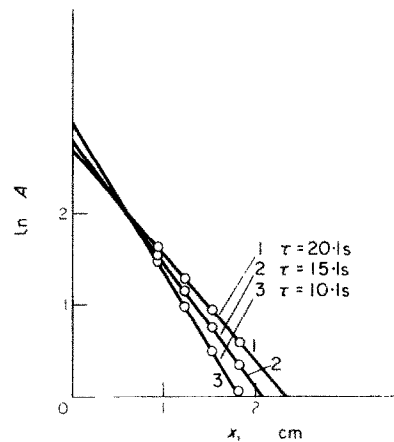


FIG. 2. Experimental dependence of the logarithm of temperature oscillation amplitude upon the position of thermocouples.

experimental dependence of $\varphi(l)$ and $\ln \theta$ on l (In Fig. 2 the intersection of curves is due to various amplification.)

The error in heat conductivity determination also appears to be somewhat different for the

case when only one thermocouple is used or when the information from the temperature probes is used together with it. The error is from 6 to 10 per cent in the first case and about 4 per cent in the second one.

The above methods of measurement were checked by broadly varying the conditions of the experiment, namely, modulation frequencies, heating power, the sizes of the samples. The results obtained in phase and amplitude variants of the method are compared. In all cases a good reproducibility of the results was obtained as illustrated by the data of Table 1 where measured

Table 1. The typical series of measurements obtained by the method of longitudinal temperature waves (Armco iron)

t	w	τ	a	a	λ	λ
(°C)	(W)	(s)	(cm ² /s)	[3]	$\frac{\text{cal}}{\text{cm s deg}}$	[6]
300	2.52	15.2	0.121	0.125	0.127	0.131
		20.1	0.119		0.125	
370	6.25	10.1	0.105		0.122	
		15.2	0.107	0.110	0.122	0.121
		20.2	0.106		0.120	

values of a and λ are presented for Armco iron. At present these methods are used in regular investigations of the set of thermal properties for metals at about 1000°C.

The second of the described methods is based upon the application of temperature waves of cylindrical symmetry. According to this method the lateral surface of the cylindrical sample (the variant with external heating) or internal surface of the hollow cylinder (internal heating) are exposed to variable electron heating [6, 7]. The theory of the method leads to the conclusion that the expression for the amplitude of temperature oscillations on the external surface of the cylindrical sample with internal heating or on the internal one of the similar sample with external heating (in the particular case on the axis of a solid cylinder) can be presented as follows

$$\theta = \theta_0 f(x, \xi) \quad (8)$$

where $x = R_2\sqrt{(\omega/a)}$, $\xi = (R_1/R_2)$ (R_1 and R_2 are the internal and external radii of the cylinder) and

$$\theta_0 = Q/Mc\omega. \quad (9)$$

For phase difference between temperature oscillations at the same point and power oscillations (more precisely for their first harmonics) the equation is obtained of the form

$$\varphi = \varphi(x, \xi) \quad (10)$$

The functions $f(x, \xi)$ and $\varphi(x, \xi)$ equal for the variants with external and internal heating are expressed in terms of the Kelvin (Thomson) functions and can be easily tabulated.

From the nature of relations (8) to (10) it follows that by this method thermal diffusivity a of the sample can be determined directly from the difference of phases φ , i.e. from the readings of one temperature probe. The knowledge of a in its turn allows estimation of heat capacity c from (9) by the results of measurements of oscillation amplitude θ of the same probe. Thus to calculate the values of thermal diffusivity, heat capacity and, consequently, of heat conductivity, it is necessary to record the temperature oscillations at one point of the sample (similar to the case of the first method described). It is typical of these methods that the values of $f(x, \xi)$ for optimum range of x variation appear to be very little different from unity, which makes the determination of heat capacity in this method almost independent of thermal diffusivity determination.

The experimental device designed for the above measurements has been described previously [7, 8]. In the present paper only a short summary is given. In the variant with external heating, a tungsten cylindrical spiral with the sample placed along the axis served as the electron-emitting cathode. Several hundred volts negative voltage was from time to time supplied to the cathode with the help of a modulating device, the anode sample being earthed. The temperature-sensing element, i.e. a thermocouple was inserted through a hole drilled along

the axis of the sample. In the variant with internal heating the cathode was placed along the axis inside the sample, while the thermocouple was welded to the external surface.

The variant with internal heating was also used for the study of liquid metals. In this case metal filled the space between two coaxial thin-walled tubes of tantalum of 24 and 8 mm dia., respectively.

To minimize the danger of convective mixing the metal was separated by a number of thin horizontal tantalum plates placed with 1 cm space between them.

The constant component of thermoelectromotive force of the thermocouple which corresponded to the mean temperature was compensated and measured by a potentiometer circuit. The variable component was amplified by a photoelectric amplifier and registered by an auto-type electronic potentiometer.

In the experiment we have estimated and taken into account the sources of errors due to the changes in heat transfer during the process of temperature oscillations, to the end effects of the cylinders, distortions of readings of thermocouples and to the non-uniformity of heat generation over the cylinder height. In the experiments with liquid metals additional errors were taken into account namely those due to the influence of the crucible walls and convective mixing.

The analysis of the errors which are due to inaccuracy of measurements leads to the conclusion that the maximum error in heat capacity determination by this method does not exceed 3-4 per cent, and that of thermal diffusivity, 4 per cent.

The above methods of measurements were carefully checked by studying the reproducibility of the results over a wide range of variations of experimental conditions, namely the periods of oscillation, heating power, sizes of samples; moreover comparison was made of the measurements by means of internal and external heating. Further, the data on thermal diffusivity were confirmed by measurements using information

about amplitudes or phases of temperature oscillations at two points of the cylinder being at various distances from the axis [9, 10]. All the cases show good agreement between the experimental data which is illustrated in Tables 2 and 3.

Table 2. The comparison of the results of measurements of heat capacity by the methods with external and internal heating (molybdenum 850° C)

External heating			Internal heating		
τ	w	c	τ	w	c
Period of heating	Power of periodic heating	Heat capacity	Period of heating	Power of periodic heating	Heat capacity
(s)	(W)	$\left(\frac{\text{cal}}{\text{g deg}}\right)$	(s)	(W)	$\left(\frac{\text{cal}}{\text{g deg}}\right)$
13.2	1.68	0.0770	13.2	2.45	0.0781
	3.36	0.0762		10.9	0.0746
26.4	4.30	0.0758	26.4	2.45	0.0733
	8.20	0.0747		7.80	0.0746
mean value		0.0759	mean value		0.0744
maximum deviation		1.7 per cent	max. deviation		1.4 per cent
Reported data		[22]			0.0735

Table 3. The typical series of measurements obtained by the method of radial temperature waves (liquid lead, 635° C)

τ	w	a	c
Period of heating	Periodically changing power	Thermal diffusivity	Heat capacity
(s)	(W)	(cm ² s)	cal/g deg
13.2	12.9	0.107	0.0338
	24.0	0.104	0.0336
26.4	12.9	0.105	0.0352
	24.0	0.109	0.0342
mean value		0.106	0.0342
maximum deviation		3 per cent	2.6 per cent

The above methods of measurements were used to obtain the set of properties for liquid tin and lead up to the temperatures about 1000°C (see Figs. 3 and 4). At present installations are

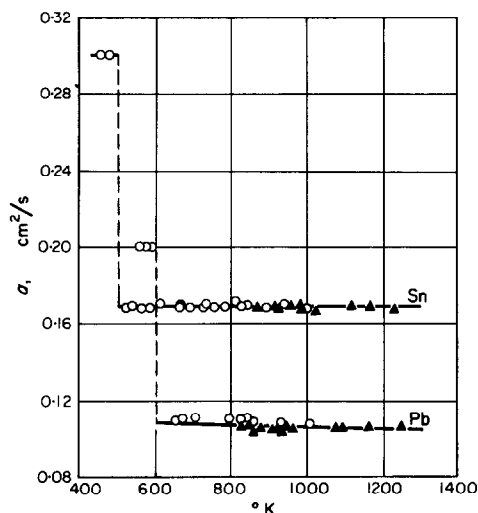


FIG. 3. The results of measurements of thermal diffusivity of solid and liquid tin and lead.

○ "two points" method [9], [10];
▲ complex method.

also developed for higher temperatures; to register temperature oscillations a contactless photo-electric method is used.

In the above two methods the set of thermal properties of metals can be determined from the results of one experiment. Another method of performing complex investigations involves various measurements of the same sample by means of the same device. In this direction great possibilities are revealed when the sample is heated by electric current passing through it. We consider the methods developed for thin samples (wire, foil) to be the most convenient. The application of such methods allows measurements at very high temperatures (up to 3000°C) by comparatively simple means. The equivalent devices are of small size, the amount of the power consumed is also small. Further the methods are described for determination of the set of properties which are based on the combination of measurements of heat conductivity under the conditions of steady heating and those of heat capacity by the method of pulsating heating.

There are some methods for the determination of heat conductivity in thin samples heated by a current. All of them are based on the study of temperature distribution along the sample in combination with power measurements.

In the methods considered, use is made of the study of small artificially produced temperature

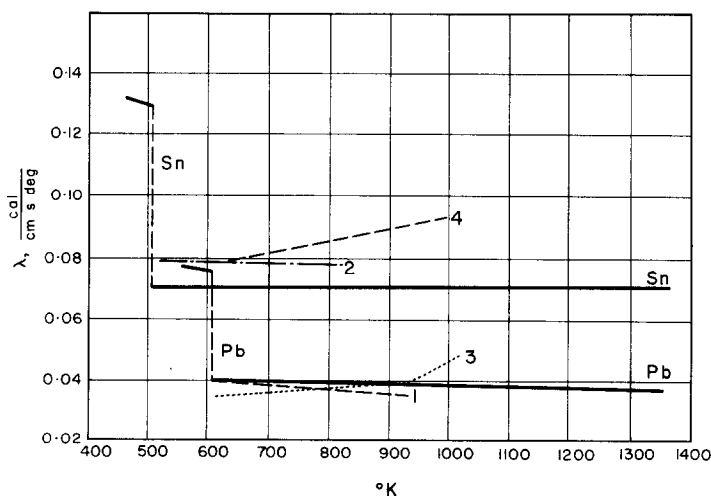


FIG. 4. The results of determination of heat conductivity of solid and liquid lead (smoothed curves) by the authors and other investigators (1, 2—reference [21]; 3, 4—reference [22]).

distortions of the sample at the section where initially the temperature was constant. These distortions are produced by means of a rider being automatically hung on the central section of the sample [11]. Under these conditions, the temperature distribution along the sample is of exponential nature, the index of the exponent being directly dependent on the heat conductivity of the sample. Such a way of heat conductivity determination which is close to the method of Jain and Kriesnhan [12, 13] is convenient, first of all, because the influence of the local temperature variations due to slight non-uniformity of the sample being studied can easily be eliminated by subtracting from the curve of temperature variation values from a similar curve obtained in a test without the rider. This method also uses a differential optic pyrometer specially designed to measure temperature distribution [14, 15], the readings of which instrument are proportional to the difference between the real temperatures of small sections of the samples being compared.

Thanks to the use of objectives which can be moved along the sample, the pyrometer can measure curves of temperature variation over the section of about 25 mm long. The pyrometer provides reproducibility of readings over the temperature range from 1000 to 3000°C with the accuracy of up to 0.1 degC; when temperature difference is about 20 degC the correction for non-linearity of the scale does not exceed 1 per cent. The basic arrangement of the pyrometer is as follows: the radiation beams from two optical channels of the pyrometer are alternatively cut by a disk so that the receiver of radiation, i.e. photomultiplier, receives signals alternately from one or the other section of the sample. Thus the variable component of the signal at the output of the registering system of the pyrometer appears to be the larger, the larger the temperature difference being studied. This temperature difference, however, is not determined by the measurement of the signal at the exit, but by the following compensation method: in one of the optical channels there is a photo-

metric wedge by moving which a zero value of the variable signal is set at the exit of the pyrometer, which corresponds to the equality of radiation fluxes. The measured temperature difference is thus compared with the readings on the scale of the photometric wedge.

Therefore the determination of heat conductivity consists in obtaining the curve of temperature distribution along the wire being studied or a strip of foil with the rider or without it, in measuring current I and voltage (potentiometer scheme is used to do this) and in determination of the mean temperature of the wire (the temperature is found by the resistance of the wire or with the aid of a disappearing filament micro-pyrometer). Heat conductivity is determined by the formula [11]

$$\lambda = \frac{4I^2\sigma}{S^2T\kappa^2} \left[1 - \frac{1}{2} \frac{d \ln(T^2/I)}{d \ln T} \right] \quad (11)$$

κ is the coefficient of the index of the exponent for temperature distribution along the sample

$$\kappa = \frac{d \ln \theta}{dx} \quad (12)$$

In Fig. 5 the experimental curves $\ln \theta$ are plotted versus x .

The second term of formula (11) in brackets is a small correction which takes into account the

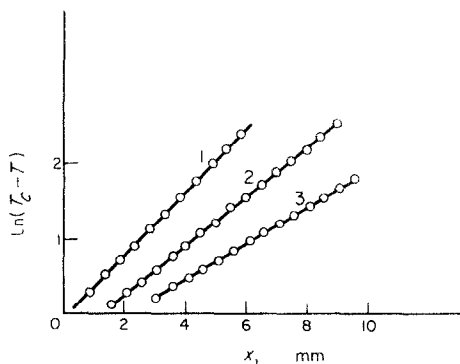


FIG. 5. Temperature distribution along tungsten wire near rider (1, 2100 K; 2, 1860 K; 3, 1600 K)

temperature dependence of the specific resistance and the integral emissivity of the sample.

To complete the experiment on determination of heat conductivity it is necessary to estimate the value of the specific resistance which in its turn requires measurements of the real temperatures. To perform these measurements the samples of absolutely black body may be used which are made of the material being studied. For the samples of foil, tubes with narrow orifices or slots can be used; for wire, spirals with closely wound turns are possible.

The analysis of the errors in heat conductivity determination including the estimation of both random and regular errors leads to the conclusion that the maximum error in heat conductivity measurements is about 7 per cent.

The discussed methods of heat conductivity measurements were studied in detail. A good agreement was established between the results obtained for wires of different diameters and for foil. Also a good reproducibility was found in experiments with various diaphragms of the pyrometer, with light filters and without them, with various riders and so on [11, 16]. Figure 6 gives the results of measurements of heat con-

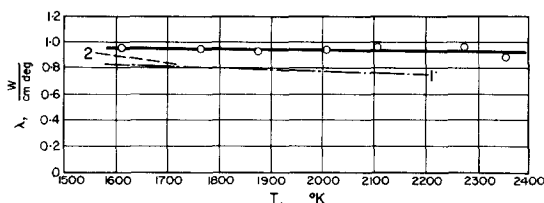


FIG. 6. The results of determination of heat conductivity of molybdenum by the authors and other investigators (1—reference [23]; 2—reference [24]).

ductivity for molybdenum obtained by the above method.

As already mentioned, to measure heat capacity of the same samples the described methods use pulsating heating, i.e. heating by electric current with both constant and variable components. In this case the heat-conductivity value

can be found from the amplitude θ of temperature oscillations of the sample by the formula

$$c = \frac{Q}{M\omega\theta} \quad (13)$$

There are various ways for determination of temperature oscillation amplitude [2]. In the case when the specific heat is determined simultaneously with heat conductivity, the photoelectric method of registering θ used in references [17, 18] is convenient. By this method a photoelectric registering part of a differential pyrometer can be used for measurements. The value of the oscillation amplitude is found by the formula

$$\theta = \frac{v_{\sim}}{v} \cdot \frac{T^2}{\alpha} \quad (14)$$

where v_{\sim} is a variable component of the voltage at the exit of the measuring scheme which corresponds to the temperature oscillation θ , v is a constant component corresponding to the mean temperature T (for its measurement a differential cascade is made at the output of the registering circuit, which compensates direct currents independent of T). The value of the constant α is equal to hv/k (h and k are Planck's and Boltzmann's constants, ν , frequency of radiation) when monochromatic radiation is registered. When dealing with a wide spectrum range the value α should be found from the experiment by the temperature dependence of the constant component v

$$\alpha = \frac{\partial \ln v}{\partial(1/T)} \quad (15)$$

The maximum error of heat capacity measurements accounting for both regular and random errors is from 4 to 7 per cent for the devices of classes 2 and 1, respectively, which are used to register v_{\sim} and v and can be considerably reduced when the devices of higher class of accuracy are used. The reproducibility of the measurement results which characterizes random errors is 0.5–1.5 per cent even at rather

essential changes of the experimental conditions (experiments with light filters and without them, with photomultipliers of various types and photocell, at various amplification, etc.). The results of temperature oscillation measurements by the photo-electric method agree within the same accuracy with the results of measurements performed for the same samples by the method of thermionic emission [18].

The measuring system for simultaneous determination of heat conductivity and heat capacity is at present used for regular studies over the temperature range from 1000 to 3000 C.

The above methods certainly do not exhaust the possibilities of simultaneous investigations. In particular, the development of the method of alternating induction heating [19, 20] and the non-stationary variant of the Kohlraush method [2] is rather promising.

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Abstract The results of the work on development of methods for simultaneous determination of thermal properties of solid and liquid metals at high temperatures are presented. The methods of longitudinal temperature waves, of radial waves and of heating of thin wires are described.

Résumé—On présente les résultats d'une étude sur le développement de méthodes pour la détermination simultanée des propriétés thermiques des solides et des métaux liquides à température élevée. Les méthodes des ondes de température longitudinales, des ondes radiales et du chauffage de fils fins sont décrites.

Zusammenfassung—Es wird über die Ergebnisse zur Entwicklung von Methoden zur gleichzeitigen Bestimmung thermischer Eigenschaften fester und flüssiger Metalle bei hohen Temperaturen berichtet. Die Methoden der Längs- und Radialtemperaturwellen und der beheizten dünnen Drähte werden beschrieben.