TEMPERATURE WAVE METHOD FOR HEAT-INSULATING MATERIALS

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The article presents a new method of solving the inverse boundary-coefficient heat conduction problem with the pulse-period action of the heat flux on one of the surfaces of a flat body. The method is based on the use of a numerical Fourier analysis for processing temperature fluctuations recorded on the opposite surface of the body.

Methods for identifying thermophysical properties of materials based on the theory of a regular regime of the third kind (temperature waves) have been known for more than one hundred years [1] and find ever increasing use in engineering practice [2-7]. Among their basic advantages are high informativeness, self-controllability, and the possibility of a continuous repeated experimental cycle. Besides, these methods make it possible to perform measurements at small temperature drops across the specimen thickness, which enables us "to work" in the linear region even with a sharp temperature dependence of the properties of the materials under investigation.

As a rule, the temperature wave method is successfully used to determine thermophysical properties of good conductors of heat, for example, metals [3, 4]. Utilization of this method for the investigation of properties of heat-insulating materials at high temperature poses a number of additional problems. First, even in conducting experiments in vacuum due to a low thermal conductivity of the material at high temperatures one cannot disregard the parameters of external heat transfer by thermal radiation. Under these conditions for determining the thermal conductivity coefficient the solution of only the coefficient inverse problem is not sufficient. It is necessary to solve the inverse problem of the combination boundary-coefficient type [8]. With a traditional approach [9] its solution requires additional information obtained by conducting the experiment in at least one more frequency regime.

Second, because of a low thermal diffusivity of heat-insulating materials and natural limitations on the minimal thickness of the specimen, which are defined by the material structure, one has to excite temperature waves in the low frequency range $\sim$0.01-0.1 Hz, which hampers the analog filtering of a legitimate signal.

Third, when investigating heat-insulating materials produced from semitransparent dielectrics (SiO_2, Al_2O_3, etc.), the applied photoelectric recording of temperature fluctuations is to be performed from the radiation in that portion of the infrared region, which corresponds to the nontransparency region of material (usually 5-7 μm). This tends to decrease the sensibility of the recording scheme and to deteriorate the signal/noise ratio. The situation is further aggravated when it is necessary to measure small fluctuations of the temperature of the specimen surface with its large mean value.

The present work proposes a new approach, which enables us to overcome the above-listed difficulties. It is not confined to investigating the first harmonic of temperature fluctuations alone, but is based on their classical representation in the form of a harmonic progression. According to this approach, the thermal pulse-period action is performed on one of the surfaces of a specimen with such a relative pulse duration of rectangular pulses that at least two temperature waves with different frequencies are simultaneously excited in the specimen. Recording of several temperature fluctuation harmonics at once is possible due to the creation of a computer-based automated experimental plant and application of modern achievements in the field of digital signal processing [10]. This makes it possible, along with digital filtering of a heavily noised legitimate signal, to perform its harmonic analysis.

The problem of propagation of flat small-amplitude temperature waves in a plate of finite thickness heated up to the temperature T_0 reduces to the following system of dimensionless equations and boundary conditions:

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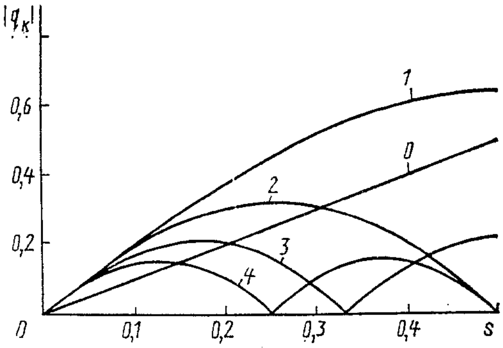

Fig. 1. Dependence of the amplitudes $|q_k|$ of the first five harmonics of the rectangular pulse Fourier series on the relative pulse duration s (the curve number corresponds to the harmonic number).

$$\mathrm{Pd}\,\frac{\partial\theta}{\partial t}=\frac{\partial^2\theta}{\partial x^2}\,, \tag{1}$$

$$-\frac{\partial\theta}{\partial x}\bigg|_{x=0}+\mathrm{Bi}\,\theta=q\,(t), \tag{2}$$

$$-\frac{\partial\theta}{\partial x}\bigg|_{x=1}-\mathrm{Bi}\,\theta=0, \tag{3}$$

where q is a 2π-periodic function of time, which, as a result of its Fourier series expansion, will be represented in the form

$$q\,(t)=\mathrm{Re}\sum_{k=0}^{\infty}q_k\exp ikt. \tag{4}$$

The temperature fluctuations on the surface x = 1 will be written

$$\theta\,(t)=\mathrm{Re}\sum_{k=0}^{\infty}G_k\,(\mathrm{Pd},\ \mathrm{Bi})\,q_k\exp ikt. \tag{5}$$

The analytical expressions for the frequency characteristic $G_k(\mathrm{Pd}, \mathrm{Bi})$ of the system (1)-(3) are well known [2].

To record small temperature fluctuations on the surface x = 1 one measures with a sensor its signal, proportional to these fluctuations and determined by L measurements performed on each of N periods at discrete times. Taking advantage of the fast Fourier transform algorithm for the spectral analysis of the measured signal, we will represent it in the following manner:

$$U\,(t_l)=\sum_{k=0}^{L-1}U_k\exp ikt_l. \tag{6}$$

The practical choice of the parameters L and N for the optimal assessment of the spectrum $\{U_k\}$ can be made by turning to recommendations of a well-developed theory of digital signal processing [10, 11].

It is assumed everywhere that the measuring circuit does not introduce amplitude-phase distortions in the working frequency range. When they are present they are taken into account by common methods. It is evident that the coefficients of expansions (5) and (6), when $k \neq 0$ and there are no noises, must coincide within the factor A, independent of k. If we are to relate the heat flux modulation and the temperature fluctuations to time, then with a proper reference point the factor A may be considered real. If we are not to make a time relation, then $A = |A| \exp i\varphi$, where φ determines the phase shift between the corresponding harmonics of expansions (5) and (6).

In the case k = 0 the consistency between (5) and (6) within $|A|$ can be ensured by excluding from the measured signal the portion of its constant component, which corresponds to the heater temperature. Thus, after the values of U_k have been determined using digital filtering, the problem of measuring thermal diffusivity with the least-squares method reduces to the problem a multiple nonlinear regression

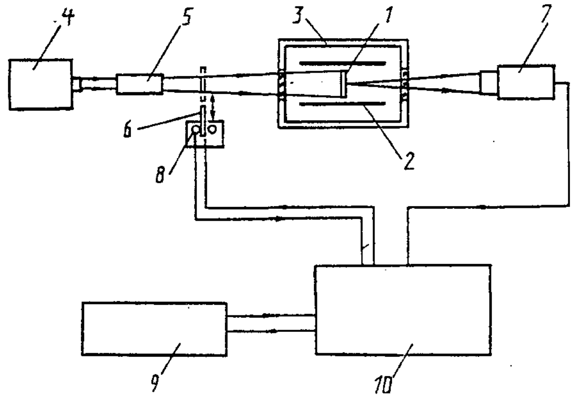

Fig. 2. Schematic diagram of the plant: 1) specimen, 2) heater; 3) vacuum chamber, 4) laser, 5) beam-expanding device, 6) cutoff device, 7) pyrometer, 8) optron pair, 9) Élektronika-60 minicomputer, 10) CAMAC modules.

$$\min_{\mathrm{Pd,Bi},A} \sum_{n=1}^{N} \sum_{k \in K} V_k \, |G_k(\mathrm{Pd,\ Bi})\, q_k - AU_k^n|, \tag{7}$$

where the number of nonzero values of V_k is larger than unity for the regression's nonconfluence.

A series of experiments conducted on the plant described below has shown that it is the white noise that makes a major contribution to the random error in determining the amplitude of the recorded fluctuations. The white noise spectral density does not depend on frequency, therefore the random error variance in U_k is independent of k. On the other hand, with increasing frequency the amplitude characteristic of the system (1)-(3) decreases rapidly, since

$$|G_k(\mathrm{Pd,\ Bi})| \sim \exp(-\sqrt{k}). \tag{8}$$

Under these conditions with increasing k the signal-noise ratio rapidly deteriorates. Therefore in choosing a form of the function q(t) one should seek to ensure commensurability of the amplitudes of different harmonics.

Figure 1 gives the dependence on the relative pulse duration of the amplitudes of the first five harmonics of the Fourier-series of the rectangular pulse, which is easiest to realize in practice. It can be easily seen that with a short relative pulse duration the amplitudes of all the harmonics of the heat flux turn out comparable. In view of (8) we obtain that the amplitude of the fourth harmonic of temperature fluctuations is approximately by an order of magnitude smaller than their first harmonic amplitude, i.e., the first four harmonics can be quite accessible for simultaneous recording.

The necessary signal/noise ratio can be ensured by decreasing the relative duration of pulses with a simultaneous increase of the density of the heat flux, which periodically affects one of the surfaces in the specimen. However, this method is practically limited owing to an increase in the temperature drop on the specimen on account of the presence of the constant component $\Delta\theta = q_0/(2+\mathrm{Bi})$ and the possible manifestation of nonlinear effects. We note that the emergence of nonlinear effects is easily controlled in the process of measuring by the presence in the measured signal of the third or the fourth harmonics at specially assigned values of relative duration of thermal pulses, respectively, s = 0.33 (3) or s = 0.25. Since these harmonics are absent in the heat flux spectral analysis with the indicated relative pulse durations (Fig. 1), their statistically significant emergence against the background of noise is possible solely because of nonlinear effects. Such a control may be of prime importance in investigating thermal diffusivity of composite materials, in which the emergence of nonlinear effects with increasing temperature may be related to the onset of decomposition of a binder.

It is necessary to point out that simultaneous recording of several temperature fluctuation harmonics in one experiment creates a considerable excess of information. This opens up prospects of investigating more sophisticated models, taking into account the radiative component of heat transfer [5-7, 12].

The proposed method is realized on the plant, whose schematic diagram is shown in Fig. 2. The plant is equipped with an automation system based on the Élektronika-60 minicomputer and modules made in the CAMAC standard. In addition to controlling the experiment the computer performs collection, digital filtering, and preprocessing of experimental data.

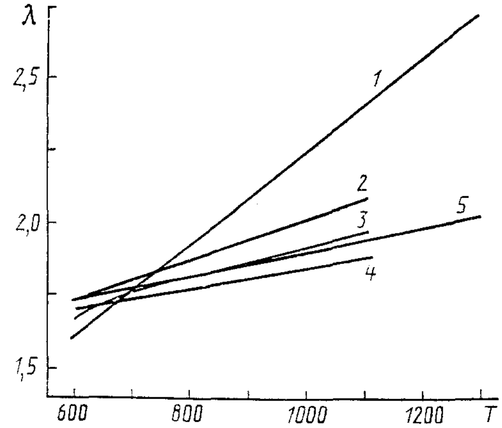

Fig. 3. Temperature dependence of the thermal conductivity coefficient of quartz glass: 1) [14]; 2) [15]; 3, 4) [12]; 5) present work. λ, W/(m·K); T, K.

The investigated specimen 1, having the form of a disk 30 mm in diameter, is fixed on molybdenum needles in the center of an alundum pipe heated by the external cylindrical molybdenum heater 2 to the necessary temperature. The heater provides heating of the specimen up to 2000 K. The entire construction is located in the vacuum water-cooled chamber 3, where a vacuum of the order of 10^{-3} Pa is created. The CO_2-laser 4 generating radiation at a wavelength of 10.6 μm with a power of 45 W is used as the source of the heat flux, periodically affecting the front surface of the specimen. With the two-lens beam-expanding device 5 the laser radiation is uniformly distributed over the specimen surface. Π-shaped radiation pulses of the prescribed relative duration are formed using the cutoff device 6, controlled from the automation system.

Temperature fluctuations on the opposite surface of the specimen were recorded by various sensors. When investigating porous quartz ceramics, in particular, the infrared pyrometer 7 was used, which records radiation at a wavelength of 5.35 μm. The signal from the sensor arrives at a 14-digit analog-to-digital converter of the automation system. It is at the same point that the signals from the optron pair 8 mounted on the cutoff device arrive, which enables us to make a time relation of the temperature fluctuations to the variations of the heat flux.

With the aim of comparing the method, proposed in the given work, and the methods realized earlier based on the theory of the regular regime of the third kind, a special series of experiments is conducted. As an investigated material we have chosen high-purity highly scattering quartz ceramics with pore dimensions ~1 μm and porosity about 50%. High scattering permitted the reduction to a minimum of the radiation component of heat transfer, which, according to estimates, did not exceed 1% at the maximal temperature of measurements performed in vacuum. The specimen was 1.56 mm thick. The amplitude of the recorded temperature fluctuations was 4 K, their period was chosen equal to 8 sec.

For the sake of comparison, from the traditional variants of the temperature wave method we have chosen the most widespread phase method [9], easily realized on the described plant. It consists of measuring a phase shift between the first harmonics of variations of a periodic heat flux established on one of the specimen surfaces and temperature fluctuations, recorded on the opposite surface, with alternate conduction of two experiments respectively at the frequencies ω and 2ω. With the aim of obtaining the maximal amplitude of the first harmonic the periodic flux was established in the form of rectangular pulses with the relative pulse duration $s = 0.5$.

It is difficult to directly compare the errors of determining the thermal diffusivity using the compared approaches, since their inherent systematic errors are different, and a calculation of random errors requires the acceptance of various statistical hypotheses. Therefore we choose $K = \{1, 2\}$ and will consider the constant A real. Apart from this we assume that A depends on k. The latter means that we deliberately deteriorate the precision of the proposed method, in fact, neglecting the information on the relationship between the amplitudes of the first and the second harmonics. Such information may prove to be important, since $Pd \sim \ln |U_1/U_2|$, while $Pd \sim \varphi^2_k$. Hence, for determining the thermal diffusivity the relationship of the amplitudes is less critical to the errors in measurements.

The efficiency function, meeting the described procedure, has the form

$$\min_{\mathrm{Pd,Bi}} \sum_{n=1}^{N} \sum_{k=1,2} \left[\operatorname{arctg} \frac{\operatorname{Im}\{G_k(\mathrm{Pd},\ \mathrm{Bi})\, q_k\}}{\operatorname{Re}\{G_k(\mathrm{Pd},\ \mathrm{Bi})\, q_k\}} - \operatorname{arctg} \frac{\operatorname{Im} U_k^n}{\operatorname{Re} U_k^n} \right]^2 . \tag{9}$$

It can be shown that (9) coincides with (7) when A_k is substituted for A and the weights are properly prescribed. With a new efficiency function it is possible to make a correct comparison of the error of determination of the thermal diffusivity by the proposed and known methods, conducting experiments with the same parameters of digital filtering at the same temperatures and values of thermal action. In the proposed method the relative pulse duration $s = 0.25$ was chosen from the condition of attaining the maximum of the second harmonic amplitude of the temperature fluctuations.

Owing to the identical experimental conditions the systematic errors of both methods are mainly the same, except for the error, associated with the nonlinearity of the problem, whose assessment requires special consideration. Besides, in the new method there is a decrease in the systematic error due to the nonisothermicity of the specimen (it is half as large here). Comparison of the results of calculating the random errors of measurements shows that in the proposed variant it is lower. Thus, the mean square deviation for the value of the thermal diffusivity coefficient obtained from the results of 30 observations at the average specimen temperature 1260 K is 25% smaller and comprises 0.5%. Therefore, the proposed approach even in its truncated "phase" variant, not permitting the realization of minimal errors, apart from a substantial decrease of experimental time, reduces systematic and random errors.

Comparison of the temperature dependence of the thermal conductivity of the quartz ceramics investigated with the temperature dependence of the true thermal conductivity of quartz glass can be a metrological check of the proposed method. This appears possible, since the thermal conductivity coefficient on condition that the material structure does not depend on temperature can be represented in the form

$$\lambda_{\mathrm{cer}}(T) = \gamma \lambda_{\mathrm{glass}}(T). \tag{10}$$

The relation (10) is valid on account of the fact that from the possible mechanisms of heat transfer in porous ceramics [13] in the conducted experiments only the heat transfer through a glass matrix is realized.

Figure 3 compares the temperature dependences of the true thermal conductivity of quartz glass, given in [12, 14], with the results of investigations of the thermal conductivity of quartz ceramics of various porosity, recalculated by Eq. (10) and obtained in the present work and the work [15]. To obtain the values of λ_{glass} with such a recalculation the constant γ was determined by comparing the recalculated results with the averaged data [12] for glass at a temperature of 600 K, where the contribution of radiation to heat transfer may be disregarded. It is evident that the data thus obtained are basically in good agreement, the exception is provided by the results of [14]. This confirms a high precision of the proposed method and the reliability of the data obtained in the present work.

NOTATION

λ, thermal conductivity coefficient; T, specimen temperature; T_0, heater temperature; $\theta = (T - T_0)/T_0$, dimensionless temperature; t, dimensionless time; q, dimensionless heat flux density; x, dimensionless coordinate; ω, angular frequency of variation of the incident heat flux density; Pd, Predvoditelev number; Bi, Biot number; φ, phase difference between the fluctuation harmonics of the heat flux density and the temperature of a point in the specimen; k, harmonic number; G, frequency characteristic; V_k, weight of the k-th harmonic; U, value of the measured signal of temperature fluctuations; K, subset of natural numbers; N, number of fluctuation periods; n, period number; L, number of measurements in a period; l, measurement number; s, relative pulse duration; γ, constant of proportionality between thermal conductivities of quartz glass and quartz ceramics.

REFERENCES

1. A. J. Angstrom, Ann. Physik, No. 14, 513 (1861).
2. L. P. Filippov, Measurements of Thermophysical Properties of Substances by a Method of Periodic Heating [in Russian], Moscow (1984).

3. A. A. Kurichenko, A. D. Ivliev, and V. E. Zinov'ev, Teplofiz. Vys. Temp., **24**, No. 3, 493-499 (1986).
4. A. D. Ivliev and V. E. Zinov'ev, Teplofiz. Vys. Temp., **18**, No. 3, 532-539 (1980).
5. J. F. Schats and G. Simmons, J. Appl. Phys., **43**, No. 6, 2586-2591 (1972).
6. P. V. Prosuntsov and S. V. Reznik, Inzh.-fiz. Zh., **49**, No. 6, 977-982 (1985).
7. G. Ya. Belov, Teplofiz. Vys. Temp., **16**, No. 4, 755-760 (1978).
8. O. M. Alifanov, Reverse Heat Transfer Problems [in Russian], Moscow (1988).
9. G. I. Petrunin and R. I. Yurchak, Vest. MGU, Fizika. Astron. **12**, No. 5, 613-614 (1971).
10. J. S. Bendat and A. G. Piersal, Random Data: Analysis and Measurement, Wiley, New York (1986).
11. V. S. Korolyuk, I. I. Portenko, A. V. Skorokhod, and A. F. Turbin, Handbook on the Probability Theory and Mathematical Statistics [in Russian], Moscow (1985).
12. O. A. Sergeev and A. A. Men', Thermophysical Properties of Semitransparent Materials [in Russian], Moscow (1977).
13. A. V. Lykov, Heat and Mass Transfer: Reference Book [in Russian], Moscow (1978), pp. 344-357.
14. V. K. Bityukov, V. A. Petrov, and S. V. Stepanov, Teplofiz. Vys. Temp., **21**, No. 6, 1106-1114 (1983).
15. A. G. Romashin and Yu. E. Pivinskii, Ogneupory, No. 9, 58-63 (1968).