## ULTRASONIC METHOD FOR DETERMINING THE THERMAL

## DIFFUSIVITY OF MATERIALS

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A method is proposed for determining the thermal diffusivity of materials by recording the amplitude variation of resonance vibrations of a disk-shaped specimen of the investigated material subjected to heat flux.

We have previously [1, 2] discussed a method for determining the thermal diffusivity of materials by means of ultrasound. Ultrasonic vibrations are excited in a specimen in the shape of a thin circular plate at a frequency close to flexural resonance. Then one of the flat surfaces of the specimen is subjected to a heat pulse from a pulsed gas-discharge tube. The variation of the temperature field in the specimen and the concomitant variation of the elastic modulus cause a shift of the specimen resonance frequency, changing the amplitude of its vibrations. The thermal diffusivity of the specimen material is calculated from the rise time of the new amplitude value, and its heat capacity is determined from the amplitude increment. The main advantages of the method are speed, the use of small specimens, and the possibility of determining simultaneously a whole set of thermophysical and physicomechanical properties, including the thermal diffusivity, heat capacity, elastic modulus, Poisson ratio, and internal friction.

The given method is marked by inherent restrictions associated with the need to record very fast processes attributable to temperature equalization in the thickness of the specimen. It has been shown [1] that the time constant characterizing the amplitude variation is  $\tau_T = h^2/4\pi^2 a$ . For a specimen having thickness of 1 or 2 mm and made from a material with a high thermal conductivity the value of  $\tau_T$  ranges from fractions of a millisecond to a few milliseconds, creating experimental difficulties in connection with the generation of highintensity pulsed heat fluxes.

Accordingly, we have developed a version of the ultrasonic method for determining the thermal diffusivity of thin disks whereby it is possible ' increase the time constant of the temperature-equalization process in the specimen through the use of radiosymmetrical heating by a heat flux that varies in the radial direction. Due to the large thermal-equilibration time in this case it is possible to use ordinary resistance heaters, which have a comparatively slow response.

We investigate the variation of the resonance frequency of a disk specimen subjected to the action of a radiosymmetrical thermal perturbation (Fig. 1). The frequency shift in the given case is caused by two processes:

1) variation of the elastic modulus of the tested material with increasing temperature;

2) inception of thermal stresses in the sample due to heating nonuniformity.

If the heat flux is constant on the heating surface and its time variation is described by the step function  $q(r, t) = q_0 \mathbf{1}(t)\mathbf{1}(b-r)$  (Fig. 1), then with neglect of heat transfer away from the specimen surface the temperature field in the specimen is described by the expression [3]

$$\Theta(r, z, t) = \frac{q_0}{c_p \rho} \left\{ \frac{b^2}{|\Phi_{0,0}|^2} t + \sum_{k,n=0}^{\infty} B(n) \frac{(-1)^k}{|\Phi_{k,n}|^2} \cos\left(\frac{\pi kz}{h}\right) J_0\left(\frac{v_n r}{r_0}\right) \frac{1 - \exp\left\{-\left(\frac{\pi^2 k^2}{h^2} - \frac{v_n^2}{r_0^2}\right) at\right\}}{\left(\frac{\pi^2 k^2}{h^2} - \frac{v_n^2}{r_0^2}\right) at}, \quad (1)$$

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$$B(n) = \begin{cases} \frac{r_0 b}{v_n} J_1\left(\frac{v_n b}{r_0}\right) \text{ for } n \neq 0, \\\\ \frac{b^2}{2} \text{ for } n = 0; \end{cases}$$
$$\|\Phi_{k,n}\|^2 = A_k \int_0^{r_0} J_0^2\left(\frac{v_n r}{r_0}\right) r dr;$$

 $A_k = h$  for k = 0 and  $A_k = h/2$  for  $k \neq 0$ ;  $J_0$  is the Bessel function of order zero;  $\nu_n$  is the root of the equation  $J_0'(\nu_n) = 0$ ; and  $c_p$  is the specific heat of the specimen material. The prime on the summation sign over k and n signifies that the term with k = n = 0 is to be omitted.

The influence of the thermal stresses arising in the specimen for the temperature distribution (1) is estimated as follows. Inasmuch as the specimen surface is free of stresses and the specimen has a small thickness, the components  $\sigma_{ZZ}$ ,  $\sigma_{rZ}$ ,  $\sigma_{\varphi Z}$  of the stress tensor are small and can be neglected. In other words, a plane stressed state exists in the specimen. The other components of the stress tensor are described by the expressions [4]:

$$\sigma_{rr} = \alpha_{T} E_{0} \left\{ \frac{1}{r_{0}^{2}} \int_{0}^{r_{0}} \Theta(r, z, t) r dr - \frac{1}{r^{2}} \int_{0}^{r} \Theta(r, z, t) r dr \right\},$$
  

$$\sigma_{qq} = \alpha_{T} E_{0} \left\{ \frac{1}{r_{0}^{2}} \int_{0}^{r_{0}} \Theta(r, z, t) r dr - \frac{1}{r^{2}} \int_{0}^{r} \Theta(r, z, t) r dr - \Theta(r, z, t) \right\},$$
  

$$\sigma_{rq} = 0,$$
(2)

in which  $E_0$  is the Young's modulus at the test temperature.

The equation for the free vibrations of the specimen with additional stresses present in it has the following form in cylindrical coordinates [5]:

$$D\Delta^{2}\omega - \left(\overline{\sigma}_{rr} \frac{\partial^{2}\omega}{\partial r^{2}} + \frac{\overline{\sigma}_{\varphi\varphi}}{r^{2}} - \frac{\partial^{2}\omega}{\partial \varphi^{2}} + \frac{\overline{\sigma}_{rr}}{r} - \frac{\partial\omega}{\partial r} + \frac{\partial\overline{\sigma}_{rr}}{\partial r} - \frac{\partial\omega}{\partial r}\right) = \rho h \omega^{2} \omega, \qquad (3)$$

where w is the transverse displacement;  $\omega$  is the cyclic frequency of the vibrations;  $\Delta$  is the Laplace operator; and D is the flexural stiffness of the plate, calculated according to the formula  $D = \int_{-h/2}^{+h/2} \frac{E}{1-\sigma^2} z^2 dz$ . The quantities  $\bar{\sigma}_{rr}$  and  $\bar{\sigma}_{\varphi\varphi}$  are interpreted as the stress tensor components integrated with respect to the z coordinate. The equation for the free vibrations of a plate specimen prior to the action of heat flux has the form

$$D_0\Delta^2 w^{(0)} = \rho h \omega_0^2 w^{(0)}. \tag{4}$$

Here the index 0 refers to the values of the particle displacements w, natural frequencies  $\omega$ , and stiffness D before thermal perturbation. The solutions of Eq. (4) are well known [5, 6].



Fig. 1. Diagram of thermal treatment of specimen.



Fig. 2. Variation of the specimen vibration parameters due to thermal perturbation of the specimen. 1)  $\delta_1 A = B_2 \exp[-t/\tau_R]$ ; a) variation of resonance curve; b) amplitude variation  $\delta A$ , arbitrary units.



Fig. 3. Block diagram of instrumentation used to measure the thermal diffusivity of materials by means of ultrasound.

TABLE 1. Results of Control Measurements of Thermal Diffusivity by the Ultrasonic (US) Method

Material	Specimen dimen., mm		, kilz	1	Thermal diffusivity			
	diameter	thickness	Working frequency	τ <sub>R</sub> , sec	mean value C am 10-6 m <sup>2</sup> /sec	ethod	handbook data • 10- <sup>6</sup> m²/sec	discrepancy, To
Armeo iron	29,0	5,3	47,4	0,636	22.5	8,2	21.9 [9]	-2.7
Steel Kh18N9T	19,9	2,9	61,2	2,12	3,21	8,5	3,64 [9]	-11,8
Bismuth (99 <b>.</b> 995% pure)	23,4	4,0	20,2	1,43	6,52	8,4	6,42 [9]	1,6
Aluminum (99.99% pure)	35,2	4,0	28,4	0,215	97,6	3,8	88,5 [10]	10,3
Titanium alloy VT <del>.</del> 8	20,1 20,1	2,4 3,6	49,6 59,1	2,31 2,37	2,98 2,92	6,8 3,5	3,16[10] 3,26[10]	-5,7 -7,6
	1 1	1 I		1				1

We write Eqs. (3) and (4) in the operator form

$$\hat{L}_{0}\omega + \hat{L}_{1}\omega = \lambda \omega, \qquad (3a)$$

$$\hat{L}_{0}\omega^{(0)} = \lambda^{(0)}\omega^{(0)}. \qquad (4a)$$

Here  $\hat{L}_0 = \Delta^2$ , and  $\lambda^{(0)} = \rho h \omega_0^2 / D_0$  is the eigenvalue of the unperturbed equation. The perturbation operator  $\hat{L}_1$ , as implied by (3) and (4), is written in the form

$$\hat{L}_{1} = \frac{b_{E}}{D_{0}(1-\sigma^{2})} \left( \int_{-h/2}^{+h/2} z^{2} \Theta(r, z+h/2, t) dz \right) \Delta^{2} - \frac{1}{D_{0}} \left( \overline{\sigma}_{rr} - \frac{\partial^{2}}{\partial r^{2}} + \frac{\overline{\sigma}_{\varphi\varphi}}{r^{2}} - \frac{\partial^{2}}{\partial \varphi^{2}} + \frac{\overline{\sigma}_{rr}}{r} - \frac{\partial}{\partial r} + \frac{\partial\overline{\sigma}_{rr}}{\partial r} - \frac{\partial}{\partial r} \right),$$

where  $b_E = dE/d\Theta$  is the temperature coefficient of the elastic modulus.

Numerical estimates for the characteristic quantities E,  $\sigma$ ,  $\alpha_{T}$ , h, and  $r_{0}$  show that the second term in the equation is much smaller than the first, i.e.,  $\hat{L}_{1}w/\hat{L}_{0}w\ll 1$ . Consequently, the thermal perturbation may be regarded as small. Now, assuming that the solutions of Eq. (4) are known, we invoke linear perturbation methods to solve Eq. (3). In accordance with the general postulates of linear perturbation theory [7] the variation of the eigenvalue due to the perturbation has the form

$$\delta\lambda = \int_{V} w^{(0)} \hat{L}_{1} w^{(0)} dV / \int_{V} [w^{(0)}]^2 dV,$$

where V is the volume of the system. Then for the shift of the m-th natural flexural frequency of the sample, where m = 0, 1, ..., we obtain the expression

$$\frac{\delta f_m}{f_m^{(0)}} = -\frac{1}{\|w_m\|^2} \left[ \frac{\alpha_T E_0 q_0}{4 \pi^2 c_p \rho^2 f_m^2} \sum_{n=1}^{\infty} \frac{A_{m,n} \frac{r_0 b}{\nu_n} J_1\left(\frac{\nu_n b}{\cdot r_0}\right)}{\|\Phi_{n,0}\|^2} F_{n,0}\left(t\right) + \frac{q_0 b_E}{c_p \rho \left(1 - \sigma^2\right) D_0} \left\{ \frac{b^3 h^3 \|w_m\|^2}{12 \|\Phi_{0,0}\|^2} t - \sum_{k,n=0}^{\infty} \frac{4 h^3 B\left(n\right)}{\|\Phi_{n,2k}\|^2} M_{mn} F_{n,2k}\left(t\right) \right\} \right],$$
(5)

in which

$$\begin{split} \|w_{m}\|^{2} &= \int_{V} [w_{m}^{(0)}]^{2} r dr d\varphi; \\ F_{n,2k} &= \frac{1 - \exp\left\{-\left(\frac{4 \pi^{2} k^{2}}{h^{2}} + \frac{v_{n}^{2}}{r_{0}^{2}}\right) at\right\}}{\left(\frac{4 \pi^{2} k^{2}}{h^{2}} + \frac{v_{n}^{2}}{r_{0}^{2}}\right) a}; \\ A_{m,n} &= \int_{V} w_{m}^{(0)} \left[\frac{r_{0}}{rv_{n}} J_{1}\left(\frac{v_{n}r}{r_{0}}\right) - J_{0}\left(\frac{v_{n}r}{r_{0}}\right)\right] \Delta w_{m}^{(0)} r dr d\varphi - \int_{V} w_{m}^{(0)} \left[\frac{2r_{0}}{v_{n}r} J_{1}\left(\frac{v_{n}r}{r_{0}}\right) - J_{0}\left(\frac{v_{n}r}{r_{0}}\right)\right] \frac{\partial^{2} w_{m}^{(0)}}{\partial r^{2}} r dr d\varphi; \\ M_{mn} &= \int_{V} [w_{m}^{(0)}]^{2} J_{0}\left(\frac{v_{n}r}{r_{0}}\right) r dr d\varphi. \end{split}$$

The quantities  $A_{mn}$  and  $M_{mn}$  do not depend on the time.

The first sum in expression (5) describes the contribution of thermal stresses to the variation of the specimen natural frequencies, and the second sum describes the contribution of the change in flexural stiffness of the disk. It is evident from (5) that for a radially nonuniform heat flux the time variation of the resonance frequency can be represented by a sum of exponentials with different time constants. Some of the time constants characterize the equalization of the temperature field in the specimen thickness, while others describe its radial equalization. The largest "thickness" time constant is  $\tau_{\rm T} = h^2/4\pi^2 a$ , and the largest "radial" time constant is  $\tau_{\rm R} = r_0^2/\nu_1^2 a$ , where  $\nu_1 = 3.83$  is the first root of the equation  $J_1(\nu_1) = 0$ . The ratio  $\tau_{\rm R}/\tau_{\rm T} = (1.64 r_0/h)^2 \gg 1$ .

It is evident from Eq. (5) that the summed terms decay rapidly with increasing indices k and n, hence the equation can be limited to the first few terms of the sums. Retaining terms with n=1 and k=0 in (5), we obtain the relation

$$\frac{\delta f_m}{f_m^{(0)}} = gt - c \left(1 - \exp\left\{-t/\tau_R\right\}\right),$$
(5a)

in which g and c are constants.

The perturbation-induced variation of the natural frequency can be recorded on the basis of the amplitude variation of the specimen vibrations near the given frequency (Fig. 2a). If curve 1 is the resonance variation before thermal perturbation and  $f_{r_1}$  is the corresponding resonance frequency, then at a driving frequency  $f_k$  the vibration amplitude is equal to  $A_1$ . When the resonance curve changes to the form of curve 2 under the influence of thermal perturbation, the vibration amplitude assumes the value  $A_2$ . When the amplitude variations take place on the linear part of the slope of the resonance curve, we have

$$A_2 - A_1 = \delta A = (\Delta A / \Delta f_r) \delta f_r$$

$$\delta A = B_1 t + B_2 (1 - \exp\{-t/\tau_p\}), \tag{6}$$

where  $B_1$  and  $B_2$  are time-independent multipliers. Now the time variation of the amplitude has the form shown in Fig. 2b. It follows from Eq. (6) and Fig. 2b that the difference between the experimental curve (solid) and its asymptotic representation extended to zero (dashed) is described by the exponential function

$$\delta_1 A = B_2 \exp\{-t/\tau_R\},\,$$

from which, taking the logarithm, we can determine the time constant  $\tau_R$  and the thermal diffusivity of the material according to the slope of the resulting straight line.

A block diagram of the instrumentation used to implement the method is given in Fig. 3. The high-frequency oscillator 1 drives the piezoelectric emitter 2, which is attached to the rod-type acoustic line 3. The ultrasonic vibrations generated by the piezoelectric emitter propagate along the acoustic line and excite vibrations in the sample 4, which are recorded through the acoustic line 8 by the piezoelectric receiving transducer 9. The signals from the latter are amplified by the amplifier 10, and after detection by the detector 11 the envelope of the high-frequency signal is recorded on the screen of the electronic oscilloscope with memory 12. The thermal perturbation is created by the motion picture projector lamp 5 through the iris 7, and a step-change of the heat flux is realized by means of the shutter 6. A high-frequency oscillator of the type GZ-7A and an amplifier of the type USh-10 are used. The transducers are TsTS-19 lead zirconate—titanate piezoelectric ceramics with a diameter of 6 mm and thickness of 3 mm. The device used to attach the specimen to the acoustic lines and transducers has a construction identical to that described in [8]. The projector lamp has a power rating of 500 W.

The results of control measurements performed at room temperature are summarized in Table 1. The table indicates agreement of our results with handbook data, allowing for measurement errors. The rms scatter of the results of individual measurements is 4 to 8%. We note that the measurement results are not affected by the uniformity of the heat flux, because the latter determines only the values of the individual components in expression (6).

Thus, the given method can be used to measure the thermal diffusivity of materials. The possibility of determining, in addition to the thermal diffusivity, the values of the elastic constants by application of the procedure described in [8] puts the given method in the class of fast and efficient techniques for assessing the properties of new materials. The asset that the ultrasonic transducers and thermal perturbation source can be placed at a good distance from the measured object ensures the capability of conducting measurements in radioactive radiation fields at high temperatures. Calculations show that heat-transfer corrections can be disregarded up to values of the Biot number  $Bi \approx 0.5$ , which corresponds to temperatures up to 2000-2500°K for specimens 10 to 15 mm in diameter.

#### NOTATION

 $\tau_{\rm R}$ ,  $\tau_{\rm T}$ , time constants characterizing the temperature variation in the specimen; *a*, thermal diffusivity; h, r<sub>0</sub>, thickness and radius of specimen; r, z, instantaneous coordinates; t, time; q, heat flux; b/2, radius of heated section of specimen surface;  $\Theta$ , temperature variation of specimen due to heat flux;  $\rho$ , density of specimen;  $\sigma$ , Poisson ratio; E, Young's modulus;  $\sigma_{\rm rr}$ ,  $\sigma_{r\varphi}$ ,  $\sigma_{\varphi\varphi}$ , components of thermal stress tensor in specimen;  $\alpha_{\rm T}$ , thermal coefficient of linear expansion; D, flexural stiffness; w, vibrational displacement; b<sub>E</sub>, temperature coefficient of elastic modulus;  $\omega^{(0)} = 2\pi f^{(0)}$ , natural cyclic frequency of specimen vibrations;  $\delta A$ , recorded variation of vibration amplitude; Bi, Biot number.

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# EXPERIMENTAL STUDY OF THE THERMAL

# CONDUCTIVITY OF WEAKLY ABSORBING LIQUIDS

### IN LAYERS TRANSPARENT TO INFRARED RADIATION

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The thermal conductivities of a number of organic liquids are determined by a transient method under irregular thermal conditions.

A great deal of experimental work has been carried out in the last few years in connection with the influence of radiation on thermal-conductivity measurements in semitransparent media, a category including the majority of liquids. Measurements have most frequently been based on the most widely accepted steadystate methods (plane layers, coaxial cylinders, heated filaments). Theoretical analysis [1-5] and a considerable number of experimental investigations [3, 5-8] show that the influence of radiation expresses itself as a dependence of the thermal conductivity on the thickness of the layer used for the measurements. The molecular thermal conductivity is approximately obtained on extrapolating this relationship to zero layer thickness.

There are certain possible ways of eliminating the effect of radiation on the results of thermal-conductivity measurements; these include the so-called transient methods, especially those involving irregular thermal conditions. These methods are most frequently realized by the transient heating of a metal filament immersed in the liquid, with simultaneous recording of its temperature. The use of very thin filaments with a low intrinsic heat capacity enables the thermal conductivities to be measured quite rapidly, so that the region (layer) over which the measurement is carried out may be relatively small. For a broad class of weakly absorbing liquids, the thickness of this layer may be smaller than the mean free path of the photons characterizing infrared radiation. The radiative mechanism of heat transfer may then be almost entirely neglected, and the value of the thermal conductivity obtained under these specific conditions may be identified with the molecular thermal conductivity. The influence of radiative heat transfer then only appears as a loss of some of the heat from the surface of the heater and from the interior of the layer into which the transient temperature field penetrates. The total extent of these losses is small in the case of brief measurements and need not be taken into account.

The criterion of "transparency" as regards thermal radiation may be written in the form

$$l > l^*,$$

(1)

where

$$\overline{l} = \int \frac{1}{k_{v}} \cdot \frac{\partial u}{\partial T} \, dv \, \left/ \int \frac{\partial u}{\partial T} \, dv, \, l^{*} = (at)^{1/2}.$$

In order to estimate  $\overline{l}$  it is sufficient to limit consideration to monochromatic radiation of frequency  $\nu_0$  corresponding to the maximum of the Planck distribution function at the particular temperature. In this case  $\overline{l} = 1/k_{\nu_0}$ , and inequality (1) may be expressed in the form

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