The thermal conductivity is measured in this way from the delay in the temperature rise at the core relative to the block temperature. The temperature difference in the liquid and the heating rate are measured with an Elektronika-4 timer and class 0.001 R-345 potentiometer. The pressure was produced and measured by means of a loaded-piston gauge type MP-2500, class 0.05, and a set of standard gauges. The thermal conductivity was measured with various heating rates, which produced temperature differences from 2 to 8 K in the layer, which enabled us to check for and eliminate effects from natural convection. The reproducibility indicates that there was none. The working equation incorporates all the characteristic corrections. The relative standard deviation in the thermal conductivity was ±2.2%.

Table 1 gives the results, which show that there is a negative temperature coefficient, but a positive pressure one. The pressure effect increases with temperature, which is characteristic of all carboxylic-acid esters.

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DETERMINING METAL THERMOPHYSICAL PARAMETERS BY X-RAY DILATOMETRY WITH RAPID HEATING

E. N. Bludilin, M. E. Gurevich, A. F. Zhuravlev, Yu. V. Kornyushin, and V. N. Minakov

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A linear approximation has been used for the thermal-conduction equation on the basis of the difference between the surface and bulk temperatures in a new method of determining metal thermophysical characteristics. The working scheme is given along with measurements on iron-alloy specimens.

High-speed x-ray diffraction measurements are widely used with metals at heating rates over 10 K/sec [1, 2]. Recently, the informativeness has been increased by combining this with dilatometry on the same specimen, which is heated by passing a current through it [2]. The measured quantities give data on the temperature patterns, and we have proposed simple methods of determining the various thermophysical parameters without increasing the number of sensors or substantially complicating the operations [3].

Figure 1 shows the scheme. The cylindrical (planar) specimen is exposed to a monochromatic x-ray beam and the diffraction line is scanned across a fixed detector by means of a slot aperture with a period $\geq 10^{-2}$ sec. The lattice parameter is determined from the diffraction angle. At the same time, one measures the change in diameter in the working part by means of a dilatometer fitted with an electromechanical sensor [sensitivity $(\Delta\ell/\ell) \leq 10^{-6}$], and the current and potential drop across the working part are monitored. The surface temperature is monitored with a chromel-copel thermocouple 50 μm in diameter welded to the specimen.

The heating rate is chosen to produce a planar temperature distribution at the center and sharp drops in temperature at the points where the specimen is attached to the contacts

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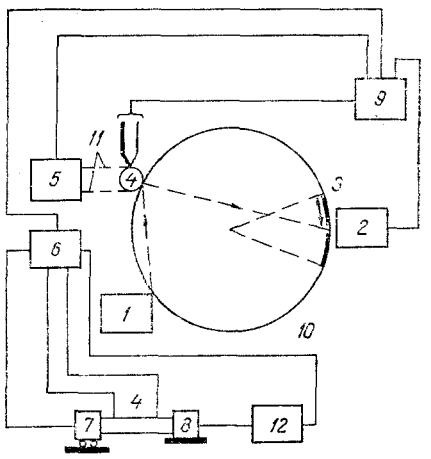

Fig. 1

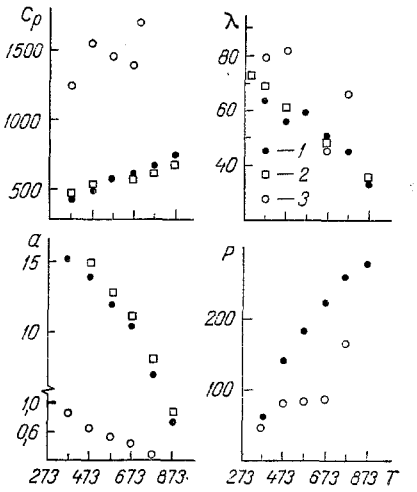

Fig. 2

Fig. 1. Apparatus for determining thermophysical parameters with high-speed radiography: 1) x-ray source; 2) detector; 3) slot stop; 4) specimen; 5) dilatometer; 6) unit for measuring current and voltage; 7) mobile contact; 8) fixed contact; 9) recording unit; 10) focusing circle; 11) dialtometer impellers; 12) power supply.

Fig. 2. Thermophysical parameters as functions of temperature: 1) Armco Fe; 2) especial-purity Fe [9]; 3) Fe–Ni–C. C_p, J/(kg·K); λ, W/(m·K); a, 10^{-6} m²/sec; P, 10^3 W/m²; T, K.

[4]. The length is such that heating rates over 30 K/sec produce this plateau over not less than 20% of the length, or over 70% of it at 400 K/sec.

The sensitivity and accuracy specifications have been defined from previous x-ray dilatometry. For example, the equilibrium thermal-vacancy concentrations in pure metals have been determined [5, 6] from measurements on the linear dimensions and lattice parameter made with an accuracy in relative measurements of about 10^{-5}. In our experiments, the focal radius was 52 mm, and when a standard was used, the error in measuring the lattice parameter was 10^{-5} nm.

The conduction equation here is [7]

$$(\nabla,\ \lambda\nabla T)+\sigma E^2=c\,\frac{\partial T}{\partial t}\,. \tag{1}$$

We consider the first boundary-value problem for a homogeneous specimen, where one can identify the deviation from the mean temperature in the cross section

$$\tau=T-\langle T\rangle \tag{2}$$

and which is such that

$$|\tau|\ll\langle T\rangle\,. \tag{3}$$

Under the usual conditions (homogeneous specimen, with no regions where c, λ, and σ differ by an order of magnitude), the characteristic heat-transfer time in the specimen is $R^2(c/\lambda)$, and the temperature difference $\langle T\rangle - T_S$ does not have time to change substantially, so

$$R^2\ll(\lambda/c)\Big/\left|\frac{\partial\ln(\langle T\rangle-T_S)}{\partial t}\right|, \tag{4}$$

which is the criterion for the quasistationary state. We neglect $\partial\tau/\partial t$ by comparison with $\lambda\Delta\tau$ and get from (1) in the approximation linear in τ that

$$\Delta\tau=\frac{c}{\lambda}\,\frac{\partial\langle T\rangle}{\partial t}-\frac{\sigma}{\lambda}\,E^2. \tag{5}$$

We examine the solutions for the plate and cylinder, as those are the shapes most commonly used. The cooling is by heat transfer to the environment.

For a plate uniform in properties, the solution to (5) is

$$\tau = \frac{1}{2}\left(\frac{c}{\lambda}\frac{\partial \langle T\rangle_z}{\partial t} - \frac{\sigma E^2}{\lambda}\right)(z^2 - h^2) - T_S - \langle T\rangle_z. \tag{6}$$

This satisfies $\tau|_{z=\pm h} = T_S - \langle T\rangle_z$, i.e., it describes the deviation in surface temperatures from the mean bulk value. For cooling, one solves (5) with E = 0.

The thermal diffusivity is given here by

$$a = -\frac{h^2}{3}\frac{\partial \langle T\rangle_z}{\partial t}/(\langle T\rangle_z - T_S). \tag{7}$$

The measured values are substituted into (7) to derive a and then the specific heat from

$$c = \sigma E^2 h^2 \Big/ \left(h^2\frac{\partial \langle T\rangle_z}{\partial t} + 3a(\langle T\rangle_z - T_S)\right). \tag{8}$$

The rate of heat loss from unit source is defined by $P = -\lambda\left(\frac{\partial \tau}{\partial z}\right)_{z=h}$, and (6) gives

$$P = 3\lambda(\langle T\rangle_z - T_S)/h \tag{9}$$

within the present accuracy.

For a cylinder, the above approximation involves neglecting the inhomogeneity in the temperature along the generator, which can be very accurate for electrical heating, and we write the solution to (5) as

$$\tau = \frac{1}{4}\left(\frac{c}{\lambda}\frac{\partial \langle T\rangle_\rho}{\partial t} - \frac{\sigma E^2}{\lambda}\right)(\rho^2 - R^2) + T_S - \langle T\rangle_\rho. \tag{10}$$

Then

$$c = (\sigma E^2 R^2 - 8\lambda(\langle T\rangle_\rho - T_S))/R^2\frac{\partial \langle T\rangle_\rho}{\partial t}. \tag{11}$$

On cooling,

$$a = -R^2\frac{\partial \langle T\rangle_\rho}{\partial t}/8(\langle T\rangle_\rho - T_S). \tag{12}$$

The rate of heat loss from unit surface is

$$P = 4\lambda(\langle T\rangle_\rho - T_S)/R. \tag{13}$$

These expressions and those for a planar specimen indicate how the geometry affects the heat transfer.

Equations (7) and (8) for a plate and (11) and (12) for a cylinder (wire) enable one to calculate the thermophysical characteristics from measured lattice-parameter changes (x rays) and changes in relative length (dilatometry) during heating and cooling.

To relate these measurements to the temperature change, we consider the thermal expansion in a nonuniformly heated specimen. For a flat specimen, the thickness change (in the plate) and the change in the lattice parameter are readily related to the difference between the averaged and surface temperatures:

$$\frac{1}{\alpha}\left(\frac{\delta h}{h} - \frac{\delta b}{b}\right) = \langle T\rangle_z - T_S, \tag{14}$$

on the basis that $\delta h/h$ corresponds to the temperature change in the entire thickness (as measured by dilatometry), while $\delta b/b$ corresponds to the surface temperature (x-ray treatment).

We use the expression for dilatation in an evenly heated cylinder [8] to get

$$\frac{3}{\alpha}\frac{1-\nu}{1+\nu}\left(\frac{\delta R}{R} - \frac{\delta b}{b}\right) = \langle T\rangle_\rho - T_S. \tag{15}$$

The measurements were made on Armco iron and an alloy containing 16% Ni, 18% C, and balance Fe, diameter 2 mm and length 50 mm. The diffraction angles were measured for the (211) line in monochromatic CrK_β radiation. The camera had been standardized on high-purity

iron. The thermophysical parameters were calculated from (11)-(13). Figure 2 shows the results, which may be compared with those from independent methods [9], with which they agree throughout the temperature range.

Temperature differences up to 3 K enable one to measure the thermal diffusivity with an error of less than 1% [10]. For our Armco iron 2 mm in radius used with heating (cooling) at 10^2 K/sec, the difference between the mean bulk temperature and the surface value was ~3.5 K for a specimen at 600 K. For poor conductors ($a < 10^{-6}$ m^2/sec), the differences are much larger. For example, with specimens of the above alloy under the same conditions, the difference was ~10 K. It is clear that the accuracy increases in proportion to the radius.

Such measurements can be used to determine metal thermophysical parameters with fast heating.

NOTATION

λ, thermal conductivity; c, specific heat; T, specimen temperature; σ, electrical conductivity; E, electric field strength in specimen; $\langle T\rangle$, mean bulk temperature independent of coordinates; τ, temperature deviation from mean; T_S, surface temperature; L, characteristic specimen size; 2h, plate thickness; $\langle T\rangle_z$, temperature averaged over layer; z, coordinate along axis perpendicular to plate plane; $a = \lambda/c$, thermal diffusivity; P, heat loss rate from surface; p, polar coordinate in cross section plane for cylindrical specimen $0 \leq \rho \leq R$; R, radius of cross section for cylindrical specimen; $\langle T\rangle_\rho$, temperature averaged over cylinder section; b, lattice parameter; α, linear-expansion coefficient; ν, Poisson's ratio; δ, variation symbol (small increment).

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