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MEASUREMENT OF THERMOPHYSICAL PARAMETERS OF ANISOTROPIC MATERIALS

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In papers [1—3] very practical pulse methods of measuring thermophysical parameters of isotropic samples with the help of a point or a line heat source were shown. However, for measurements on anisotropic materials a plane source of heat had to be used.

In this paper relations will be shown, which make the use of a point or a line source for measurements on anisotropic materials possible.

INTRODUCTION

Measurements of thermal properties of materials are rather difficult and slow. Some methods have been devised trying to avoid these difficulties.

The pulse methods [1—3, 5] are timesaving and their accuracy is for many applications sufficient. They can be applied to measurements on thin films and on samples of undefined shape [2, 3] and they render the calculation of specific heat, thermal conductivity and thermal diffusivity from the results of a single measurement possible.

The thermal properties of many natural as well as synthetic materials appear to be anisotropic. In such cases the results from the papers [1—3] cannot be used. Therefore, measurements on anisotropic samples usually performed with a plane source of heat, and are difficult to perform, as it is not easy to obtain a negligible heat capacity of the source and a good contact with the sample.

Formulae enabling the calculation of thermophysical parameters of anisotropic materials from the results of pulse measurements with the help of a point or a line source of heat will be found in this paper.

HEAT CONDUCTION IN ANISOTROPIC MEDIUM

The specific heat c, the thermal conductivity λ, defined by the Fourier law and the thermal diffusivity k, defined by the relation

$$k = \frac{\lambda}{c\gamma}, \qquad (1)$$

(where γ is the density), will be called thermophysical parameters (properties). All three parameters are in an isotropic case scalar quantities. If the thermal parameters are anisotropic, then the directions of the vectors of the heat current and of the temperature gradient are generally not the same. Their relation can be written as

$$\bar{i} = -\bar{\bar{\lambda}} \cdot \text{grad}\, T. \tag{2}$$

$\bar{\bar{\lambda}}$ is the thermal conductivity tensor. According to [4] it is symmetrical. Its graphic image is an ellipsoidal surface. In a coordinate system with the axes identical with the axes of the ellipsoid the non-diagonal components of the tensor are zero, i. e. three components, the so called main thermal conductivities, give its complete description. Similarly, it is sufficient to know the three main thermal diffusivities.

HEAT CONDUCTION FROM A PULSE HEAT SOURCE

Let us suppose a homogenous infinite heat conducting sample, e. g. a solid or a viscous liquid. Let the function F in the equation

$$\frac{\partial T}{\partial t} = \frac{\lambda}{c\gamma} \nabla^2 T + \frac{1}{c\gamma} F(x, y, z, t) \tag{3}$$

express the pulse-like development of a known amount of heat Q in a very small volume round the point O(0, 0, 0). The temperature distribution in the point $P(x, y, z)$ will be

$$\Delta T = \frac{Q}{8\pi^{3/2}\, \lambda (kt)^{3/2}} \exp\,(r^2/4kt); \tag{4}$$

$$r^2 = x^2 + y^2 + z^2,$$

as it results from the solution of the (3) equation.

From the maximum of the function (3) in the point P and the corresponding time

$$t_m = \frac{r^2}{6k} \tag{5}$$

the thermal parameters can be calculated.

$$k = \frac{r^2}{6t_m}; \quad \lambda = 1{\cdot}23 \times 10^{-2} \frac{Q}{rt_m \Delta T}; \quad c = 0{\cdot}0738 \frac{Q}{r^3 \gamma \Delta T}. \tag{6}$$

We get similar results if the heat source is a line, identical with the z axis and Q is the heat quantity supplied by a unit length of source:

$$k = \frac{r^2}{4t_m}; \quad \lambda = 2{\cdot}92 \times 10^{-2} \frac{Q}{t_m \Delta T}; \tag{7}$$

$$c = 0{\cdot}117 \frac{Q}{r^2 \gamma \Delta T}, \quad r^2 = x^2 + y^2.$$

The increase in temperature as well as the time of its maximum is the same in every point on the $|r| =$ const. surface, i. e. this surface is every moment an isotherm. In an anisotropic material the isotherm gets deformed and as it can be demonstrated it is the surface of an ellipsoid.

The equation of heat conduction in anisotropic materials for a coordinate system mentioned above is [4]

$$\frac{\partial T}{\partial t} = k_1 \frac{\partial^2 T}{\partial x^2} + k_2 \frac{\partial^2 T}{\partial y^2} + k_3 \frac{\partial^2 T}{\partial z^2} + \frac{1}{c\gamma} F(x, y, z, t) \tag{8}$$

After a transformation of coordinates in the form

$$\xi = x \left(\frac{k}{k_1}\right)^{1/2}, \quad \eta = y \left(\frac{k}{k_2}\right)^{1/2}, \quad \zeta = z \left(\frac{k}{k_3}\right)^{1/2} \tag{9}$$

with an arbitrary k we have

$$\frac{\partial T}{\partial t} = k \left(\frac{\partial^2 T}{\partial \xi^2} + \frac{\partial^2 T}{\partial \eta^2} + \frac{\partial^2 T}{\partial \zeta^2}\right) + \frac{1}{c\gamma} F_1(\xi, \eta, \zeta, t). \tag{10}$$

The only formal difference between the equations (10) and (3) is in the function F_1. If it could be formed with the same symmetry with respect to the ξ, η, ζ coordinates, as the function F with respect to the x, y, z coordinates, the solution of the equation (10) would have the same form as that of the equation (3). It happens in the case of negligible dimensions cf the heat source, i. e. if it can be regarded as a point or a line. Thus the solution of the equation (10) for an infinite space is

$$\Delta T = \frac{Q}{8\pi^{3/2}\, \lambda (kt)^{3/2}} \exp\left[-\frac{\xi^2 + \eta^2 + \zeta^2}{4kt}\right]. \tag{11}$$

Because of an arbitrary k, λ does not mean any real thermal conductivity and ξ, η, ζ are unknown coordinates.

The transformations (9) can be expressed in a more useful form. We shall choose for this purpose the points

$$P_x(x, 0, 0) \equiv P_\xi(\xi, 0, 0)$$
$$P_y(0, y, 0) \equiv P_\eta(0, \eta, 0)$$
$$P_z(0, 0, z) \equiv P_\zeta(0, 0, \zeta)$$

with the condition $x = y = z$. As the thermal parameters are anisotropic, the temperature increase in the points P_x, P_y, P_z, i. e. ΔT_1, ΔT_2, ΔT_3, as well as the time of their maximum t_{m1}, t_{m2}, t_{m3} are different. The ratio of the warmings-up is

$$\frac{\Delta T_i}{\Delta T_j} = \left(\frac{k_i}{k_j}\right)^{3/2}, \quad i, j = 1, 2, 3. \tag{12}$$

The scalar quantity k from the relations (9) can be expressed as a tenzor

$$\bar{\bar{k}}_{\xi\eta\zeta} \equiv \begin{pmatrix} k & 0 & 0 \\ 0 & k & 0 \\ 0 & 0 & k \end{pmatrix}. \tag{13}$$

This tensor can be regarded as the thermal diffusivity tensor, transformed into the $0, \xi, \eta, \zeta$ coordinate system. If the condition

$$k^3 = k_1 k_2 k_3 \tag{14}$$

is fulfilled, i. e. (14) is an invariant of the $\bar{\bar{k}}$ tensor [7], the choice of k preserves the used physical units, including the unit of volume.

Let us define a new quantity

$$\Delta T = \left(\prod_{i=1}^{n} \Delta T_i\right)^{1/n} \tag{15}$$

with $n = 3$ in the case of the point and $n = 2$ for the line heat source. It is easily seen that for this ΔT the relation

$$\frac{\Delta T_i}{\Delta T} = \left(\frac{k_i}{k}\right)^{3/2}, \quad i = 1, 2, 3 \tag{16}$$

is correct.

The (12) and (16) relations make it possible to express the transformations by the maximal warmings-up ΔT_i, measured in the pionts P_x, P_y, P_z.

Now the specific heat c may be calculated by means of the results of a measurement in the point $P_x(x, 0, 0) \equiv P_\xi(\xi, 0, 0)$, the (9), (16) and the evidently valid relation

$$c = 0{\cdot}0738 \frac{Q}{\gamma \varrho^3 \Delta T_\varrho}, \quad \varrho^2 = \xi^2 + \eta^2 + \zeta^2. \tag{17}$$

$$c = 0{\cdot}0738 \frac{Q}{\gamma \xi^3 \Delta T_1} = 0{\cdot}0738 \frac{Q}{\gamma x^3 \left(\frac{k}{k_1}\right)^{3/2} \Delta T_1};$$

$$c = 0{\cdot}0738 \frac{Q}{\gamma x^3 \Delta T}. \tag{18}$$

Obviously the x can be replaced by y or z coordinates.

It is not necessary to measure in points with the same distance from the source. In the expression

$$c = 0{\cdot}0738 \frac{Q}{\gamma x' y' z' (\Delta T'_1 \Delta T'_2 \Delta T'_3)^{1/3}} \tag{19}$$

the x', y', z' coordinates are arbitrary.

The three warming-up measurements may be realized successively. If the heat pulses are not of the same magnitude, the following relation is to be used:

$$c = \frac{0{\cdot}0738}{\gamma} \frac{1}{x' y' z'} \left(\frac{Q_1}{\Delta T'_1}\right)^{1/3} \left(\frac{Q_2}{\Delta T'_2}\right)^{1/3} \left(\frac{Q_3}{\Delta T'_3}\right)^{1/3}. \tag{20}$$

The results for the line heat source:

$$c = 0{\cdot}117 \frac{Q}{\gamma x^2 \Delta T}, \tag{21}$$

$$c = 0{\cdot}117 \frac{Q}{\gamma x' y' (\Delta T'_1 \Delta T'_2)^{1/2}}, \tag{22}$$

$$c = \frac{0{\cdot}117}{\gamma} \frac{1}{x' y'} \left(\frac{Q_1}{\Delta T'_1}\right)^{1/2} \left(\frac{Q_2}{\Delta T'_2}\right)^{1/2}. \tag{23}$$

The time of the maximum of the function (11) in the point $P_x(x, 0, 0) \equiv P_\xi(\xi, 0, 0)$ is

$$t_{m1} = \frac{\xi^2}{6k} = \frac{x^2}{6k_1},$$

as it results from the relations (5) and (9). The formula for the calculation of thermal diffusivity is

$$k_1 = \frac{x^2}{6t_{m1}}. \tag{24}$$

Similarly for k_2, k_3.

The relation (6) for the calculation of thermal diffusivity is valid for anisotropic samples. The same statement could be proved with respect to the relation (7) for k. It follows from these facts that it is possible to measure and calculate thermal diffusivity correctly for every direction.

Formulae for the calculation of thermal conductivity components result from the relation (1) after substituting the (18) and (24) expressions, e. g. for the direction Ox

$$\lambda_1 = 1{\cdot}23 \times 10^{-2} \frac{Q}{x t_{m1} \Delta T}, \tag{25}$$

similarly for the other directions. Obviously the ΔT is defined by the expression (15).

Results for the line source:

$$\lambda_1 = 2{\cdot}92 \times 10^{-2} \frac{Q}{t_{m1} \Delta T}, \tag{26}$$

$$\lambda_2 = 2{\cdot}92 \times 10^{-2} \frac{Q}{t_{m2} \Delta T}.$$

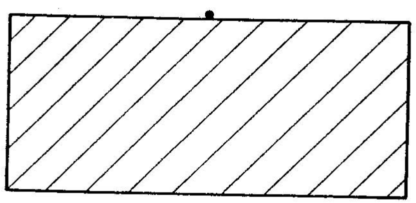

Fig. 1. Thin wire on the surface of the sample as a heat source.

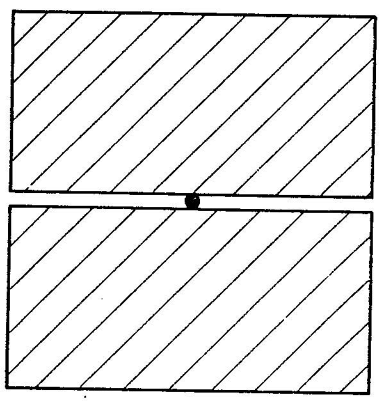

Fig. 2. Thin wire between two „semiinfinite" samples as a heat source.

The measurement is particularly influenced by the nearest vicinity of the place where the heat source and the indicator are located. The valididy of the derived relations is not influenced by the finite dimensions of the sample, if they are not smaller than a certain minimum. According to Krempaský [1, 2] for isotropic materials the distance from the point source — or from the point on the line source opposite the indicator — to the surface must be $R \geqq 3r$. The error is then less than 1%. The same condition is valid for anisotropic samples, if r is replaced by ϱ. But as ϱ is usually unknown it is necessary to choose an appropriately greater diameter. For example $R = 4r$ is sufficient up to the ratio of $\lambda_{max}/\lambda_{min} = 3{\cdot}2$.

As the heat spreads from the source radially (more exactly see [4]), i. e. it does not pass through any of the $x0y$, $y0z$, $z0x$ planes, the heat conduction in „semiinfinite" samples bounded with one of those planes can be calculated similarly as in infinite samples. Therefore, measurements on an isolated surface of the material are possible. The amount of heat Q is to be doubled in calculations.

The heat spreads from a line source in planes, perpendicular to the line, i. e! similarly as in an isolated thin foil from a point source. This fact permits the measurement of thermal properties of thin films in directions lying in the plane of the film, with the help of a point source.

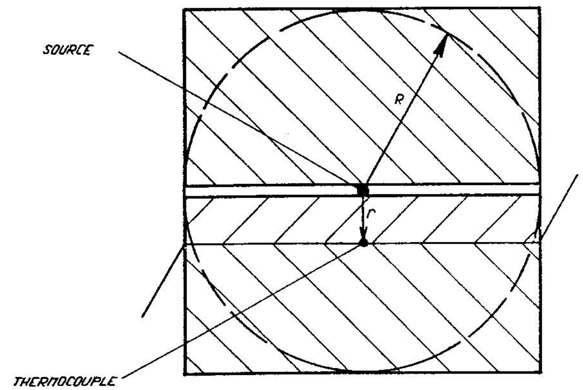

Fig. 3. The fundamental arrangement of the measurement.

The structure of the greatest part of practically interesting materials is defined with regard to the main axes of the thermal conductivity tensor. In crystalline materials, except those belonging to the monoclinic and triclinic systems, the necessary directions for measurement result from the symmetry elements.

In monoclinic crystals one direction should be identical with the twofold axis or with the normal to the plane of symmetry. If the remaining coordinate axes are the axes of the crystal, perpendicular to $0z \equiv b$, the thermal diffusivity tensor is

$$\bar{\bar{k}} \equiv \begin{pmatrix} k_{aa} & k_{ac} & 0 \\ k_{ca} & k_{cc} & 0 \\ 0 & 0 & k_3 \end{pmatrix}. \qquad (27)$$

Its components as well as those for the triclinic crystal can be found by constructing the tensor ellipsoid. A system of equations in the form

$$k_{aa}a_n^2 + k_{bb}b_n^2 + k_{cc}c_n^2 + (k_{ab} + k_{ba})\, a_n b_n + (k_{ac} + k_{ca})\, a_n c_n + (k_{bc} + k_{cb})\, b_n c_n = 1, \qquad (28)$$

where $k_{ij} = k_{ji}$, should be used. From measurements along the coordinate

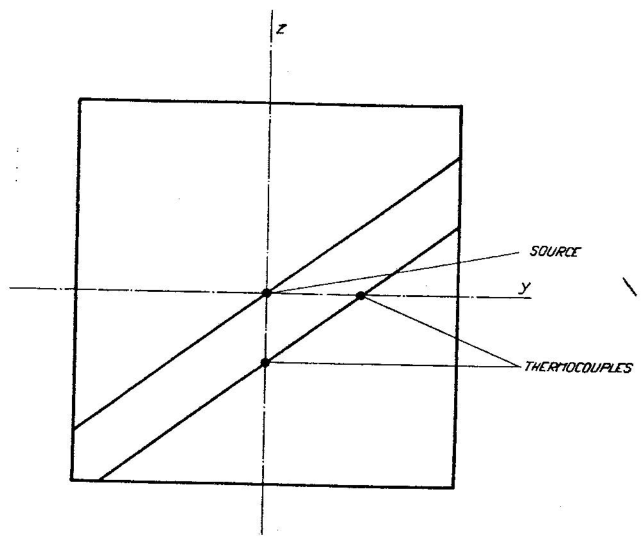

Fig. 4. Measurement in two directions.

axes and in further three directions, e. g. lying in the $a0b$, $b0c$, $c0a$ planes the k_{ij} components can be calculated, as $1/k_{ij}^{1/2}$ is the length of the radiusvector of a point on the ellipsoid and thus it gives the a_n, b_n, c_n coordinates.

For the calculation of k in every direction, or for the transformation to another coordinate system the formula

$$k_{kl} = \sum_{i=1}^{3} \sum_{j=1}^{3} c_{ik} c_{jl} k_{ij} \qquad (29)$$

is valid [4, 6, 7].

ARRANGEMENT OF MEASUREMENTS

The simplest realization of the line heat source is a thin wire, pressed to the surface of the sample or between two „semiinfinite" samples through which an electric current passes. (Fig. 1, 2). The arrangement from Fig. 2, where the heat transfer from the surface of the sample does not need to be taken into account, can be recommended.

A satisfactory realization of the point source is more difficult. On metals and semiconductors a point pressed to the surface can be used. The heat pulse is achieved by Peltier's effect and Joule's heat. Similarly a knifeedge can be employed as a line source.

An excellent heat contact between the parts of the sample pressing the indicator as well as between the indicator and sample is inevitable.

The influence of the heat capacity of the source and of its heat conduction is discussed in detail in Krempaský's paper [2].

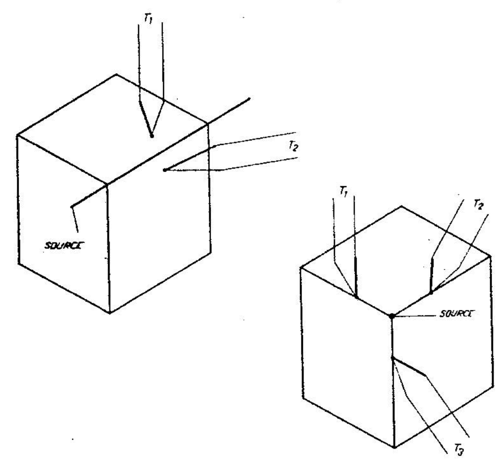

Fig. 5. Measurement on the surface of the sample.

The coefficients can be measured one by one on samples, cut perpendicularly to the chosen direction (Fig. 3). If for the measured material the relation $\lambda_1 = \lambda_2 \neq \lambda_3$ is valid, a line source is suitable, perpendicular to the z-axis, with indicators in the z-axis and perpendicularly to it (Fig. 4). Fig. 5 shows the surface measurement of more components on an appropriately cut sample.

Other arrangements are dealt with in detail in papers [2, 3].

CONCLUSIONS

A solution of the differential equation of the heat conduction in anisotropic materials with regard to pulse measurements of thermophysical parameters has been shown in the paper. Principles of measurement and formulae for calculation of thermal conductivity and thermal diffusivity components as well as of specific heat have been included. The use of line or point heat sources makes the measurements in comparison to the flat source considerably easier.

The choice of necessary directions for measurement and the way of obtaining the entire description of thermal diffusivity (and thermal conductivity) of single crystals have been shown.

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