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Thermal Diffusivity and Conductivity in Low-Conducting Materials: A New Technique

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A comparative method is presented, suitable to measure both thermal diffusivity and conductivity of low-conducting solids. The repeatibility of the measurements of thermal conductivity is 3%, whereas for diffusivity is 6%. Data for some low-conducting materials are given, consistent with those reported in the literature.

KEY WORDS: low-conducting materials; thermal conductivity; thermal diffusivity.

1. INTRODUCTION

Recently a new method of measurement of thermal diffusivity in low-conducting materials [1, 2] has been presented with the aim of reducing, to negligible levels, the uncontrolled heat exchanges between specimen and environment. The method uses a vertical cylindrical specimen with the lower base in contact with a copper disk: this acts as a heat source when an electric current is switched on in a surrounding resistive coil. The high conductivity of copper ensures that, at any time, the instantaneous temperature is uniform throughout the disk and coincident with the temperature of the junction of a thermocouple inserted into the disk itself: such a feature would not necessarily be true for a thermocouple attached to a low-conducting material, because strong instantaneous thermal gradients could be presents in this case around the junction.

In the above method, the thermal expansion of the specimen is recorded by means of a capacitive cell: in this way, all the thermal field

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detectors (namely, the thermocouple inserted in the copper disk and the capacitive cell) are out of the specimen and, therefore, produce no perturbation on the thermal field present in it. The accuracy is remarkably high for materials with thermal expansion coefficient larger than 0.5×10^{-5} K^{-1}. Below this limit, the method fails.

The purpose of the present paper is to provide a method for low-conducting, low-expanding materials, allowing, again, an experimental arrangement in which the thermal field detectors are external with respect to the specimen under study.

2. PRINCIPLES OF THE EXPERIMENTAL METHOD

A schematic view of the apparatus, which is placed in a vacuum chamber, is shown in Fig. 1. The specimen P lies between two copper disks, D_1 and D_2. The lower copper disk works as a heat source: to achieve this it is surrounded by an insulating resistive wire J. Switching on the current generator to which J is connected, joule heat is transferred to D_1. By changing the current in J, it is possible to vary in an arbitrary way the heat supplied to the specimen. Since the thermal conductivity of copper is very high, the temperature of D_1 is essentially uniform, i.e., independent of the space coordinates throughout the disk: this means that the lower base of the specimen, which is in good thermal contact with D_1, can also be considered at a uniform temperature. We have noted that a good thermal

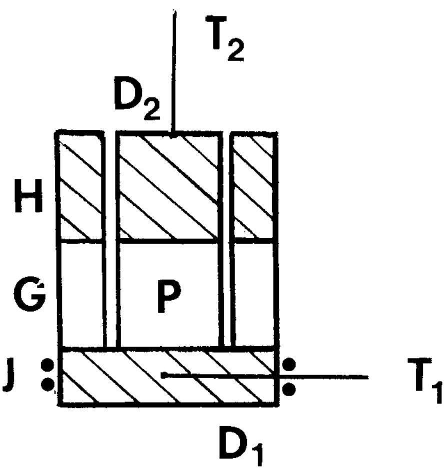

Fig. 1. Schematic of the experimental arrangement for thermal diffusivity and conductivity measurements.

contact is always easily realized by a thin layer of silver glue. A thermocouple T_1, inserted into D_1, measures the temperature of the heat source: this thermocouple does not produce uncontrolled heat exchanges with the environment because heat losses or gains, if any, through the thermocouple leads, are automatically included into the heat source (see Ref. 1).

The specimen P is surrounded by a hollow cylinder G, which is made of its same material. The distance between the specimen and the hollow cylinder is about 0.25 cm. During the measurement, the instantaneous temperature distribution on the inner surface of G is the same as that on the lateral surface of the specimen: therefore, G is a good thermal guard hindering heat losses from the lateral surface of P.

The specimen and its thermal guard support the copper disk D_2 and the holow copper cylinder H, respectively: H acts as the thermal guard of D_2. The temperature of D_2 is measured by a Chromel–Alumel thermocouple T_2 (0.0076 cm in diameter wire). We point out that the heat lost by radiation from the upper surface of D_2 can be rendered negligibly small if this surface is accurately polished: on the contrary, in the absence of D_2, the dispersion of heat from the upper surface of the specimen (usually nonmetallic, in this kind of measurement) would not be negligible at all. In this way, the only uncontrolled heat losses are produced by the thermocouple T_2, but these losses can be greatly reduced through the use of very small diameter leads. The copper disk D_2 also avoids the problem of attaching the thermocouple to the specimen; in such a case the distortion of the heat flux lines, produced by the termocouple junction, would greatly reduce the accuracy of the measurements.

As shown in the following section, the simultaneous recording of the temperatures of the two thermocouples allows the determination of both the thermal diffusivity of the sample and the ratio of the sample conductivity to copper conductivity.

3. MATHEMATICAL FRAMEWORK

Since the base of the specimen is uniformly heated and heat exchanges through the lateral surface are not allowed by the thermal guard, the problem is unidimensional. Let us introduce a coordinate system with the origin at the center of the lower base of the specimen. Let k and k' be the conductivities of the specimen and of copper, respectively, α and α' the corresponding thermal diffusivities, and b the length of the specimen: the solution of the Fourier equation for a specimen subjected to conditions of constant heating rates S and S' through the two bases, namely,

$$S = -k(\partial\theta/\partial z)_{z=0} \tag{1}$$

and

$$S' = -k(\partial\theta/\partial z)_{z=b} \tag{2}$$

is given by

$$\theta(z, t) = (kb)^{-1}(S - S')(\alpha t + \tfrac{1}{2}z^2) - Sz/k + \sum_n a_n \cos(n\pi z/b) \exp(-n^2\pi^2\alpha t/b^2) \tag{3}$$

This expression is an extension of the formula already used in Ref. 1. Let us introduce a new coordinate system z' with the origin at the center of the base of the upper copper disk D_2: the solution of the heat conduction equation in D_2, subjected to zero flux at the upper base of the disk $(z' = d)$ and with heat flux

$$S' = -k'(\partial\theta'/\partial z')_{z'=0} \tag{4}$$

at the lower base, is given by

$$\theta'(z', t) = (k'd)^{-1}S'[\alpha' t + \tfrac{1}{2}(z' - d)^2] + \sum_n c_n \cos(n\pi z'/d) \exp(-\alpha' n^2\pi^2 t/d^2) \tag{5}$$

where d is the length of the copper disk D_2.

To extend the solution to a general heating rate, the entire time interval of an experimental curve can be subdivided into many small intervals of equal width, each of them being characterized by constant values of S, S', depending, however, on the time interval itself.

Let us indicate by S_m and S'_m the heating rates referring to the interval between t_m and t_{m+1}, and by $\theta_m^{m+1}(z, t)$ and $\theta_m'^{m+1}(z', t)$ the corresponding temperature fields: one has

$$\theta_m^{m+1}(z, t) = (kb)^{-1}(S_m - S'_m)(\alpha t + \tfrac{1}{2}z^2) - S_m z/k + \sum_n a_{n,m} \cos(n\pi z/b) \exp(-\alpha n^2\pi^2 t/b^2) \tag{6}$$

$$\theta_m'^{m+1}(z', t) = (k'd)^{-1}\, S'_m[\alpha' t + \tfrac{1}{2}(z' - d)^2] + \sum_n c_{n,m} \cos(n\pi z'/d) \exp(-\alpha' n^2\pi^2 t/d^2) \tag{7}$$

Let us impose the conditions

$$\theta_0^1(z, 0) = 0 \tag{8}$$

$$\theta_0'^1(z', 0) = 0 \tag{9}$$

and (for $m > 1$)

$$\theta^{m}_{m-1}(z, t_m) = \theta^{m+1}_{m}(z, t_m) \tag{10}$$

$$\theta'^{m}_{m-1}(z, t_m) = \theta'^{m+1}_{m}(z, t_m) \tag{11}$$

Putting $\theta_{m+1}(z) = \theta^{m+1}_{m}(z, t_{m+1})$ and $\theta'_{m+1}(z') = \theta'^{m+1}_{m}(z', t_{m+1})$, one obtains after standard analysis, for $m > 1$,

$$\begin{aligned}\theta_{m+1}(z) = {} & (kb)^{-1}(S_m - S'_m)(\alpha t_{m+1} + \tfrac{1}{2}z^2) - S_m z/k \\ & - (b/k) \sum_{p=1}^{m} [\alpha t_p/b^2 + \tfrac{1}{2}G_z(t_{m+1} - t_p)](S_p - S'_p - S_{p-1} + S'_{p-1}) \\ & + (b/k) \sum_{p=1}^{m} M_z(t_{m+1} - t_p)(S_p - S_{p-1}) \\ & - \tfrac{1}{2}(b/k)(S_0 - S'_0)\, G_z(t_{m+1}) + (b/k)\, S_0 M_z(t_{m+1})\end{aligned} \tag{12}$$

and

$$\begin{aligned}\theta'_{m+1}(z') = {} & (k'd)^{-1}S'_m[\alpha' t_{m+1} + \tfrac{1}{2}(z' - d)^2] - \tfrac{1}{2}(S'_0 d/k')\, R_{z'}(t_{m+1}) \\ & - (d/k') \sum_{p=1}^{m} (S'_p - S'_{p-1})[\alpha' t_p/d^2 + \tfrac{1}{2}R_{z'}(t_{m+1} - t_p)]\end{aligned} \tag{13}$$

and for $m = 0$,

$$\begin{aligned}\theta_1(z) = {} & (kb)^{-1}(S_0 - S'_0)(\alpha t_1 + \tfrac{1}{2}z^2) - S_0 z/k \\ & - \tfrac{1}{2}(b/k)(S_0 - S'_0)\, G_z(t_1) + (b/k)\, S_0 M_z(t_1)\end{aligned} \tag{14}$$

and

$$\theta'_1(z') = (k'd)^{-1}S'_0[\alpha' t_1 + \tfrac{1}{2}(z' - d)^2] - (S'_0 d/2k')\, R_{z'}(t_1) \tag{15}$$

where the functions G_z, M_z and $R_{z'}$ are defined as follows:

$$G_z(t) = (4/\pi^2) \sum_n [(-1)^n/n^2] \cos(n\pi z/b) \exp(-\alpha n^2\pi^2 t/b^2) + 1/3$$

$$M_z(t) = (2/\pi^2) \sum_n [((-1)^n - 1)/n^2] \cos(n\pi z/b) \exp(-\alpha n^2\pi^2 t/b^2) + 1/2$$

$$R_{z'}(t) = (4/\pi^2) \sum_n (1/n^2) \cos(n\pi z'/d) \exp(-\alpha' n^2\pi^2 t/d^2) + 1/3$$

Now if $\theta^{\mathrm{Cu}}_{m+1}$ is the temperature measured by the thermocouple inserted into the copper disk at t_{m+1}, the boundary condition between the disk D_1 and the specimen P is [3]

$$\theta^{\mathrm{Cu}}_{m+1} - (\theta_{m+1})_{z=0} = S_m/H \tag{16}$$

where H depends on the nature of the thermal contact between specimen and copper. The boundary condition between D_2 and P is

$$(\theta_{m+1})_{z=b} - (\theta'_{m+1})_{z'=0} = S'_m/H \tag{17}$$

Using the parameters $\gamma = k/(Hb)$, $X = k'/k$, $X' = Xb/d$ and defining $Q_m = bS_m/k$, $Q'_m = dS'_m/k'$, the boundary conditions for $m > 0$ can be written as

$$\begin{aligned}\theta^{\mathrm{Cu}}_{m+1} &- \alpha b^{-2}(Q_m - X'Q'_m)t_{m+1} + b^{-2}\sum_{p=1}^{m}[\alpha t_p + \tfrac{1}{2}b^2 G_0(t_{m+1}-t_p)]\\ &\times [Q_p - Q_{p-1} - X'(Q'_p - Q'_{p-1})] - \sum_{p=1}^{m} M_0(t_{m+1}-t_p)(Q_p - Q_{p-1})\\ &+ \tfrac{1}{2}(Q_0 - X'Q'_0)\, G_0(t_{m+1}) - M_0(t_{m+1})Q_0 = \gamma Q_m\end{aligned} \tag{18}$$

and

$$\begin{aligned}b^{-2}&(Q_m - X'Q'_m)(\alpha t_{m+1} + \tfrac{1}{2}b^2) - Q_m\\ &- b^{-2}\sum_{p=1}^{m}[\alpha t_p + \tfrac{1}{2}b^2 G_b(t_{m+1}-t_p)][Q_p - Q_{p-1} - X'(Q'_p - Q'_{p-1})]\\ &+ \sum_{p=1}^{m} M_b(t_{m+1}-t_p)(Q_p - Q_{p-1}) - \tfrac{1}{2}G_b(t_{m+1})(Q_0 - X'Q'_0)\\ &+ M_b(t_{m+1})Q_0 - d^{-2}Q'_m(\alpha' t_{m+1} + \tfrac{1}{2}d^2) + \tfrac{1}{2}Q'_0 R_0(t_{m+1})\\ &+ d^{-2}\sum_{p=1}^{m}(Q'_p - Q'_{p-1})(\alpha' t_p + \tfrac{1}{2}d^2 R_0(t_{m+1}-t_p)) = X'\gamma Q'_m\end{aligned} \tag{19}$$

while, for $m = 0$, one has

$$\theta^{\mathrm{Cu}}_1 - \alpha b^{-2}(Q_0 - X'Q'_0)t_1 + \tfrac{1}{2}G_0(t_1)(Q_0 - X'Q'_0) - M_0(t_1)Q_0 = \gamma Q_0 \tag{20}$$

and

$$\begin{aligned}b^{-2}(Q_0 - X'Q'_0)(\alpha t_1 + \tfrac{1}{2}b^2) - Q_0 - \tfrac{1}{2}G_b(t_1)(Q_0 - X'Q'_0) + M_b(t_1)Q_0\\ - d^{-2}Q'_0(\alpha' t_1 + \tfrac{1}{2}d^2) + \tfrac{1}{2}Q'_0 R_0(t_1) = X'\gamma Q'_0\end{aligned} \tag{21}$$

Although the previous theory, for the sake of generality, has been formulated so as to include the effect of the contact layers between copper and specimen, we now use Eqs. (18)–(21) with $\gamma = 0$. This is in fact the appropriate choice of γ for a low-conducting specimen in contact with a high-conducting material like copper: measurements of γ, for such boundary conditions as published in Ref. 1, invariably lead to negligible values of this parameter. In these circumstances, assuming the copper diffusivity α' to be known, we are left with two unknown parameters, namely, α and X'.

The analysis of the experimental curves $\theta^{(1)}(t)$ and $\theta^{(2)}(t)$, as measured by the thermocouples T_1 and T_2, proceeds through the following steps:

(a) Subdivide the total time interval into N parts of equal width $\tau = t_{m+1} - t_m$, and for each t_{m+1} read the experimental value of the temperature $\theta^{\mathrm{Cu}}_{m+1} = \theta^{(1)}(t_{m+1})$ to be inserted in Eqs. (18) and (20).

(b) Impose a trial couple of values (α, X').

(c) Solve Eqs. (20) and (21) with respect to Q_0 and Q'_0.

(d) Solve Eqs. (18) and (19) with respect to the Q_m's and Q'_m's; a computer can be easily programmed for this operation, because having Q_0 and Q'_0, one immediately obtains $Q_1, Q_2, \ldots,$ and $Q'_1, Q'_2, \ldots,$ from (18) and (19) written for $m = 1$, $m = 2, \ldots,$ respectively,

(e) Determine through Eqs. (13) and (15) the corresponding function $\theta'_{\alpha, X'}(d, t)$.

(f) According to the least-squares principle, minimize with respect to α, X' the square sum

$$\Delta = \sum_i \left[\theta'_{\alpha, X'}(d, t_i) - \theta^{(2)}(t_i)\right]^2 \tag{22}$$

The values of α and X' at the minimum are taken as the experimental determinations of these two parameters.

4. EXPERIMENTAL PROCEDURE AND RESULTS

The experiment is performed when the signals of the two thermocouples T_1 and T_2 are stable. Switching on in the coil J a current of the order of 1 A for about 30 s, one obtains a time behavior like that shown in Fig. 2, referring to a specimen of plexiglass at room temperature. This material appears particularly suitable to check the method, because, owing to the relevant value of the thermal expansion coefficient, its thermal diffusivity can also be accurately determined by the use of the dilatometric technique [1, 2].

Each copper disk in the present experiment was 10 mm thick, and the specimen was 2 mm high, with a radius of 10 mm. The corresponding

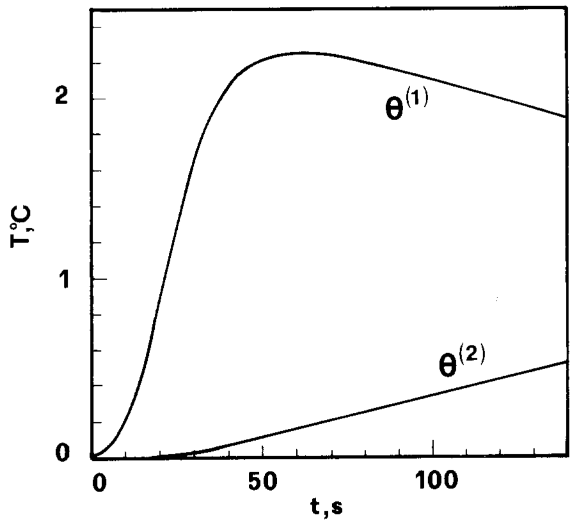

Fig. 2. Time behavior of the signals of the two thermocouples $\theta^{(1)}$ and $\theta^{(2)}$.

analysis of the two curves is summarized in Table I, where the behavior of Δ in the plane (α, X') clearly shows the existence of a minimum. In this analysis the value adopted for α' was $0.93\ \mathrm{cm^2 \cdot s^{-1}}$ [4], which was previously determined for the copper alloy employed to fabricate the two copper disks.

The mean values of α and X', as resulting from the average over 10 different measurements, are $\alpha = 0.95 \times 10^{-3}\ \mathrm{cm^2 \cdot s^{-1}}$ and $X' = 370$, with standard deviations of 6 and 3%, respectively. The value of α is fully consistent with the result given in Ref. 2 ($0.95 \times 10^{-3}\ \mathrm{cm^2 \cdot s^{-1}}$). As shown in Table II, also the corresponding value of $X = (d/d)X' = 1850$ is in good

Table I. Behavior of the Square Sum Δ, Given by Eq. (22), as a Function of the Thermal Diffusivity α and of the Reduced Conductivity Ratio $X' = k'b/(kd)$ for a Measurement on Plexiglass at Room Temperature

	α ($10^{-3}\ \mathrm{cm^2 \cdot s^{-1}}$)						
X'	0.80	0.85	0.90	0.95	1.00	1.05	1.10
360	0.10	0.12	0.16	0.21	0.28	0.35	0.43
365	0.11	0.09	0.09	0.12	0.16	0.21	0.26
370	0.16	0.11	0.09	0.07	0.10	0.12	0.15
375	0.26	0.18	0.12	0.11	0.09	0.10	0.11
380	0.41	0.30	0.21	0.16	0.12	0.11	0.10

Table II. Experimental Values of the Thermal Diffusivity α and of the Conducivity Ratio $X = k'/k$ at Room Temperature, Compared with the Literature Values α_0, X_0

	α ($cm^2 \cdot s^{-1}$)	X	α_0 ($cm^2 \cdot s^-$)	X_0
Fused silica	8.4×10^{-3}	292	7.8×10^{-3}	270
Zerodur	10.5×10^{-3}	243	—	—
Plexiglass	0.95×10^{-3}	1850	0.95×10^{-3}	1880

agreement with the data available from the literature. The same table includes the results for two typical low-conducting, low-expanding materials (fused silica and Zerodur, a glass ceramic with thermal expansion coefficient $= 0.5 \times 10^{-7}\ K^{-1}$), for which the dilatometric method is not suitable.

To compare these data with those reported in the literature, we present in the last column in Table II the values of X calculated with the copper conductivity taken from Ref. 5 and the conductivities of fused silica and plexiglass taken from Refs. 5 and 6, respectively. The third column in the table lists the values of diffusivity as taken from Ref. 5 for fused silica and from the dilatometric technique, Ref. 2, for plexiglass.

5. CONCLUSIONS

The method presented in this paper is simple and provides accurate measurements of the thermal transport parameters of low-conducting materials. The accuracy is connected with the minimization of the uncontrolled heat exchanges between specimen and environment, owing to the occurrence of the following circumstances:

(i) the specimen is not directly in contact with the thermal detectors (thermocouples);

(ii) the heat loss by radiation (usually high for nonmetallic materials) is eliminated from the two bases due to the presence of the copper disks and hindered from the lateral surface by the thermal guard surrounding the specimen.

In this way, the method ensures a remarkable correspondence between the boundary conditions existing in the experiment and those assumed in the mathematical solution of the heat diffusion equation. The failure of such a correspondence is the main source of errors in the measurements of thermal diffusivity. From this point of view, also our heat source is preferable to the laser beam usually employed in this kind of measurements, because in this way we avoid the uncertainties connected with the spatial distribution or with the intesity fluctuations of the beam: these are in fact responsible for large errors, estimated to be of the order of 10% [7].

A final comment concerns the value of be assigned to the copper diffusivity α'. The numerical analysis of a couple of experimental curves (such as in Fig. 2) shows that a relative change given to α' is correspondingly found in the resulting value of k'/k. Consequently, since the diffusivities of metals can be determined within 1% [4], the effect on X is expected to be lower than the uncertainty associated with the lack of repeatibility (see Section 4). The effect on α, on the other hand, is negligible because we find that a relative change as high as 10% in α' produces only a 0.5% change in the resulting value of α.

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