PULSE METHOD OF MEASURING THE THERMAL DIFFUSIVITY AND THE SPECIFIC HEAT OF SMALL SEMICONDUCTOR SPECIMENS

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A pulse method is described by which the thermal diffusivity and the specific heat can be measured with a bead microthermistor, a carbon bolometer, and a heater film. The thermal conductivities of several semiconductor and dielectric materials have been thus determined.

Transient methods of measuring the heat transfer coefficients offer certain advantages over steady-state methods. Among these advantages are fast measurement, easier protection against heat losses, and the feasibility of miniaturizing the test apparatus. All this applies also to pulse methods, which are now being rapidly developed. The gist of these methods is that one end of the specimen is heated by pulses from a special source. A temperature probe at the other end of the specimen records the signal change in time. With the rate of temperature rise and the magnitude of the thermal pulse known, one can calculate the thermophysical properties of the specimen; its thermal diffusivity (a) and its specific heat per unit volume ($c\rho$). The thermal conductivity is then found from the well known relation $\lambda = ac\rho$.

The authors have developed a method of measuring the thermal diffusivity and the specific heat of small specimens, the latter being heated from a miniature heater film which had been deposited on the surface of a standard specimen. Unlike the pulse methods where the test plate is heated from an external luminous or electronic source [1, 2], our method is treated in terms of a multilayer heating problem. A system of three adjoining layers is shown in Fig. 1. Layers 0 and II are made of standard material whose properties are known, while the properties of layer I are to be determined. On the contact surface between layers 0 and I (surface 0/I) is installed a heater film and on the I/II interface a film-type temperature probe. We first consider the ideal one-dimensional case, where the thicknesses of heater and probe are assumed negligible, while layers 0 and II are assumed so thick that the heat losses at the outer ends of the system become small. In other words, layers 0 and II are considered infinitely large. This is always valid within a given test time interval, if the thickness Δ of layers 0 and II satisfies the inequality

$$\frac{\Delta_{0,2}^2}{a_{0,2}} \gg \frac{d_1^2}{a_1}, \tag{1}$$

with the subscripts 0, 1, and 2 referring to layers 0, I, and II respectively. The problem concerning the transmission of a thermal δ-pulse through the system in Fig. 1 is solved by the operational method [3]. As a result, we obtain for the temperature T_1 at the I/II interface the following series

$$T_1 = \frac{D}{\sqrt{\pi t}} \sum_{n=1}^{\infty} K_1^{n-1} \exp\left[-(2n-1)^2 \frac{d_1^2}{4a_1 t}\right], \tag{2}$$

with $D = 2Q/(b_1(1 + g_0 + g_1 + g_0 g_1))$.

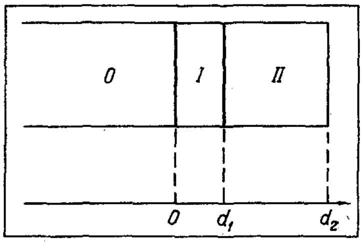

Fig. 1. Schematic arrangement of the standard specimens (0, II) and the test specimen (I).

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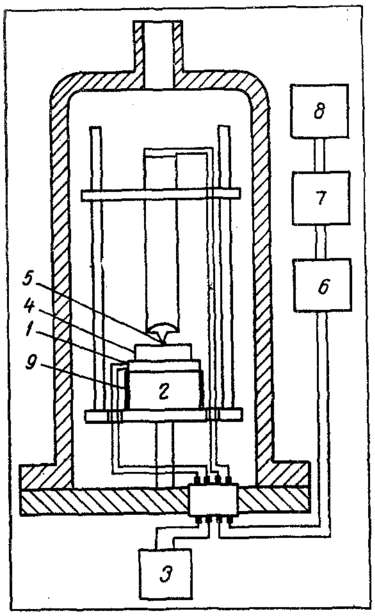

Fig. 2. Schematic diagram of the instrumentation.

If the thickness of layer II is zero and the heat transfer from the end surface of the test specimen is negligible, then the result appears the same as (2) but with $g_1 = 0$. It follows from the solution to (2) that the temperature curve passes through a maximum. In practice this maximum is rather flat and a precise determination of the time to reach that maximum ($t_{\max}$) is difficult. Much more conveniently, one determines graphically the time $t_{1/2}$ necessary for reaching half the maximum temperature level.

If only the first term of expression (2) is retained, then

$$t_{\max} = A_{\max}\frac{d_1^2}{a_1}, \tag{3a}$$

$$t_{1/2} = A_{1/2}\frac{d_1^2}{a_1}, \tag{3b}$$

with the numerical coefficients $A_{\max} = 0.5$ and $A_{1/2} = 0.135$.

Adding the second and the following terms in (2) for a determination of $A_{1/2}$ results in a small correction and affects the calculated value of thermal diffusivity according to formula (3b) only slightly when $A_{1/2} = 0.135$. Thus, with the addition of the second term in (2) we have $A_{1/2} = 0.135\,(1-0.02\,K_1)$. With the entire series in (2) added and with the maximum value $K_1 = 1$, moreover, the correction to $A_{1/2}$ based on the first term only does not exceed 3% [2]. The value of the thermal activity b_1 of a test specimen (b_0 and b_2 are assumed known) is determined from the temperature at any point on the ascending portion of curve (2) as, for example, from the maximum temperature $T_{\max}$:

$$T_{\max} = \sqrt{\frac{2A_{1/2}}{\pi t_{1/2}}}\,D\exp(-0.5). \tag{4}$$

The realization of this measurement scheme involves a few necessary corrections. First of all, the thermal pulse has a finite width. In order to make the errors arising from this circumstance negligible [4, 5], the pulse width is chosen much smaller than time $t_{1/2}$ (with a pulse width below $0.02\,t_{1/2}$ the correction to a_1 calculated according to (3b) is not more than 2%). Secondly, the heater film and the probe film have each a finite thickness. Regardless of the careful polishing of specimens, furthermore, there appear contact junctions with nonzero thermal resistances between the heater film and the probe film respectively and the specimen.

We can now consider the problem of heat transmission through the the system in Fig. 1 with layer II thermophysically equivalent to a contact junction and a temperature probe combination. Equivalency is to be interpreted here as an equality between the small thickness $\Delta_2 = d_2 - d_1$ and the thermal diffusivity a_2 of layer II and the respective parameters of that contact junction and temperature probe combination.

We will determine the temperature on the outer surface of layer II. For simplification, we let $b_1 = b_0$ ($g_0 = 1$). The result for temperature T_2 at the outer surface of layer II is represented as a series of decaying exponential terms:

$$T_2 = \frac{D}{\sqrt{\pi t}}\sum_{n=1}^{\infty}(-1)^n K_2^{n-1}\exp\left[-\frac{1}{4}\left(\frac{d_1}{\sqrt{a_1}} + (2n-1)\frac{\Delta}{\sqrt{a_2}}\right)^2\right]. \tag{5}$$

Retaining only the first term of series (5), we obtain $t_{1/2}$ as in (3b) with $A_{1/2} = 0.135$ replaced by $A_{1/2}$ as a function of $t_{1/2}$:

$$A_{1/2} = \frac{A_{1/2}^0}{\left[1 - \frac{R}{\sqrt{t_{1/2}}}\right]^2}, \tag{6}$$

where $R = (\Delta_2/\sqrt{a_0})\sqrt{A_{1/2}^0}$. If $d_1/\sqrt{a_1} \gg \Delta_2/\sqrt{a_2}$, then the correction to $A_{1/2}^0 = 0.135$ may be disregarded. Otherwise, one must experimentally determine $A_{1/2}$ as a function of $t_{1/2}$ at constant properties of the contact junction and temperature probe. A few corrections arise also from the consideration of the heat transfer between the outer end of layer II and the ambient medium [3] (when inequality (1) is not satisfied). The effect of end losses is negligible if

$$\frac{Hd_1}{\lambda_1} \leqslant 0.1.$$

Condition $Hd_1/\lambda_1 \cdot d_1/l \leq 0.05$ is sufficient for disregarding the heat transfer at the lateral specimen surfaces. Under the earlier specified conditions, the error in determining a_1 according to formula (3b) does not exceed 3-5%.

A schematic diagram of the instrumentation is shown in Fig. 2. The heater film I with an electrical resistance of approximately 1 Ω was produced by fusing silver paste on the surface of a standard 20 × 10 × 15 mm quartz base 2 with known thermophysical properties. As a source of thermal pulses we used discharges of a C = 650 μF capacitor 3 controlled by a thyristor switch. The energy of a discharge pulse $Cv^2/2$ did not exceed 1.5 J, the pulse width did not exceed 1 μsec. The temperature probe in the first test mode comprised a carbon bolometer not thicker than 15 μ and produced by a technology shown in [6], in the second test mode it comprised a high-sensitivity bead microthermistor 5 (diameter 0.1 mm). In the first case the bolometer was deposited on the surface of a thick standard quartz specimen ($d_2 - d_1 = 1.5$ cm) so as to satisfy condition (1). In the second case, the surface of the test specimen remained free except at the point of contact with the thermistor. Before installation into the apparatus, specimens 4 (with characteristic section dimensions from 3 × 3 to 5 × 5 mm and 1 to 5 mm thick) were carefully polished without destroying their plane-parallel geometry. Reliable thermal contacts were effected by adequate pressure and by filling the contact junctions with vacuum-grade oil or glycerine. In practice, under a vacuum of 10^{-2} to 10^{-3} mm Hg and T = 400°K, end and lateral heat losses could be disregarded over the entire range of thermal conductivity of the test specimens. The signal from the temperature probe was transmitted through a bridge circuit 6 with a model F-301-1 amplifier 7 and recorded by means of a model H-326-1 high-speed instrument 8. The total specimen temperature was measured with the aid of a separate heater 9 from room temperature up to 100°C. The thermal diffusivity was calculated by formula (3b). The specimen thickness was adjusted so as to make time $t_{1/2}$ less than 1.5 sec. When the thermistor was used and $t_{1/2}$ was less than 1.5 sec, $A_{1/2}$ depended strongly on $t_{1/2}$ and this relation could be established experimentally on standard materials. Within test accuracy, this relation could be described well by expression (6) with the value of R determined experimentally, and it was found unaffected by the thermal properties of the test specimens (ranging from quartz to bismuth). In the thermistor measurement $A_{1/2}$ did not deviate from the value $A^0_{1/2} = 0.135$ by more than 10% at $t_{1/2} \geq 1.5$ sec but by as much as 50% at $t_{1/2} = 0.75$ sec. With the use of a carbon bolometer $A_{1/2}$ became less dependent on $t_{1/2}$ (because of lower R values in (6)). The error in the thermal diffusivity of standard materials at room temperature, with $a = 2 \cdot 10^{-7}$-10^{-5} m²/sec, did not exceed 7% at $t_{1/2} \geq 1.5$ sec and reached 15% at $t_{1/2} = 0.5$ sec. After the thermal activity had been calculated, the specific heat was evaluated on the basis of formula (4). Here $T_{\max}$ was determined from a preliminary calibration of the temperature probe. The constant ratio $N = Q/v^2$ of the effective Q in a thermal pulse (per unit area of specimen surface) to the capacitor voltage v squared was also determined in preliminary measurements.

The formula for calculating the specific heat from thermistor measurements ($b_2 = 0$) follows from (4) and (3b):

$$\frac{c_1\rho_1}{c_0\rho_0} = B\frac{v^2}{c_0\rho_0 d_1 T_{\max}} - \sqrt{\frac{a_0}{a_1}}, \tag{7}$$

with the constant $B = 2\sqrt{2/\pi}\exp(-0.5)N$. The value of B is conveniently determined from tests with standard materials. The measurement error in the specific heat relative to the value according to formula (7) reached 15-20%.

We used this method (with a microthermistor) to test at room temperature several materials for which no or insufficient thermal diffusivity (and thermal conductivity) data are available.

On the basis of the relations derived here, the test conditions were set up so as to keep the error in the thermal diffusivity according to (3b) within 7% of the empirical formula (6). The thermal conductivity of cadmium sulfide and cadmium selenide single crystals, calculated from values of specific heat given in the literature, is 13 and 7.1 W/m·deg respectively. The difference between our measurements and the only available published data (20.2 W/m·deg for CdS [7] and 4.2 W/m·deg for CdSe [8]) may be due to different impurity levels and degrees of structural perfection in the specimens. Tests at temperatures up to 100°C indicate a slight drop in the thermal conductivity of CdSe at a rate of $2 \cdot 10^{-3}$ deg^{-1}. The values of thermal conductivity 0.46 W/m·deg for the polycrystalline Seignette salt specimen and 0.42 W/m·deg for the amorphized ternary $CdGeAs_2$ compound agree closely with the published data (0.42 W/m·deg for Seignette salt [8] and 0.5 W/m·deg for $CdGeAs_2$ [9]). Depending on the method of heat treatment, $CdGeAs_2$ appears in a random oriented or in an ordered crystalline form. In the latter case $\lambda = 6.5$-7.8 W/m·deg; i.e., the thermal conductivity is 15-20 times higher than of the amorphous form. Single crystals of naphthalene have a low thermal conductivity, which is characteristic of compounds with weak intermolecular forces. The bond anisotropy results in an anisotropy of the thermal conductivity in naphthalene: in the direction of high-energy bonds, i.e., parallel to the $\{100\}$ planes $\lambda_{II} = 0.55$ W/m·deg is 1.2 times higher than $\lambda_I = 0.46$ W/m·deg in the perpendicular direction. The well known published value $\lambda = 0.38$ W/m·deg [10] refers, evidently, to polycrystalline naphthalene specimens. Weaker intermolecular Van der Waals forces are responsible for the low value of thermal conductivity $\lambda = 0.17$ W/m·deg obtained in our tests for polycrystalline carbon tetrabromide.

NOTATION

λ	is the thermal conductivity;
a	is the thermal diffusivity;
c	is the specific heat;
b	is the thermal activity;
H	is the heat-transfer coefficient;
ρ	is the density;
T_1, T_2	are the temperature of the respective interface above ambient;
Q	is the heat content in a pulse, per unit area of specimen surface;
$g_0 = b_0/b_1$;	
$g_2 = b_2/b_1$;	
$g' = (g_0 + g_1)/(1 + g_0 g_1)$;	
$K_1 = (1-g')/(1 + g')$;	
$K_2 = (1-g_1)/(1 + g_1)$;	
$R = (\Delta_2/\sqrt{a_2}) \sqrt{A^0_{1/2}}$;	
l	is the specimen width;
C	is the capacitance of the capacitor;
v_p	is the voltage across the capacitor;
t_{max}	is the time to reach the maximum temperature;
$t_{1/2}$	is the time to reach half the maximum temperature;
N, A_{max}, $A_{1/2}$	are the constant coefficients;
$\Delta_{0,2}$	is the thickness of layers 0 and II;
d_1	is the thickness of layer I;
d_2	is the total thickness of layers I and II;
$\Delta_2 = d_2 - d_1$.	

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