MEASURING THE THERMOPHYSICAL PROPERTIES OF MATERIALS IN THE 20-300°K TEMPERATURE RANGE

Yu. V. Bol'shakov, L. L. Vasil'ev, and F. M. Pozvonkov

UDC 536.21:536.2.083

The thermophysical properties of polymethylmethacrylate and phenopolyurethane were measured by quasisteady heating of plant specimens.

Methods of measuring the thermophysical properties of materials are based on either steady or transient heating.

Speedy measurement is the main advantage of transient methods [1-8], while the thermophysical properties of materials (thermal conductivity, thermal diffusivity, and specific heat) can be determined compositely by quasisteady methods.

An apparatus for determining the thermophysical properties of materials by a quasisteady method has already been described in the technical literature [7, 8]; a cylindrical specimen is placed inside an adiabatic shell which also serves as a constant-power heat source. The test range of temperatures there is 4-300°K. In many cases, however, a plate specimen appears more convenient to use for such measurements.

Here the authors apply a quasisteady method of composite thermophysical measurements to an infinitely large plate heated from a constant-power source.

The calculation formulas for this problem are:

$$\lambda = \frac{q(x_2^2 - x_1^2)}{2R\Delta T}, \tag{1}$$

$$a = \frac{dT}{d\tau} \cdot \frac{1}{2\Delta T}(x_2^2 - x_1^2), \tag{2}$$

$$c = \frac{\lambda}{a\gamma}. \tag{3}$$

The basic apparatus for determining the thermophysical properties of materials by this method is shown schematically in Fig. 1.

The cryostat housing 7 containing a vat with liquid helium 6, a vat with liquid nitrogen 5, and the shielded specimen 4, is evacuated to $1 \cdot 10^{-5}$-$1 \cdot 10^{-7}$ torr by means of a diffusion pump 2 through a nitrogen trap 3 and also with a mechanical pump 1.

The liquid helium is poured in from the Dewar flask 12 through vacuum tubing with a siphon valve. The vapor forming here is fed through vapor recovery system into a gas tank. Helium is compressed in a rubber chamber 10 connected to the Dewar flask and to that recovery system through vacuum-grade rubber tubing with clamps.

The liquid nitrogen is poured in from the Dewar flask 11 through a similar arrangement, with the forming vapor exhausted into air.

Institute of Heat and Mass Transfer, Academy of Sciences of the Belorussian SSR, Minsk. Translated from Inzhenerno-Fizicheskii Zhurnal, Vol. 24, No. 6, pp. 1039-1044, June, 1973. Original article submitted May 30, 1972.

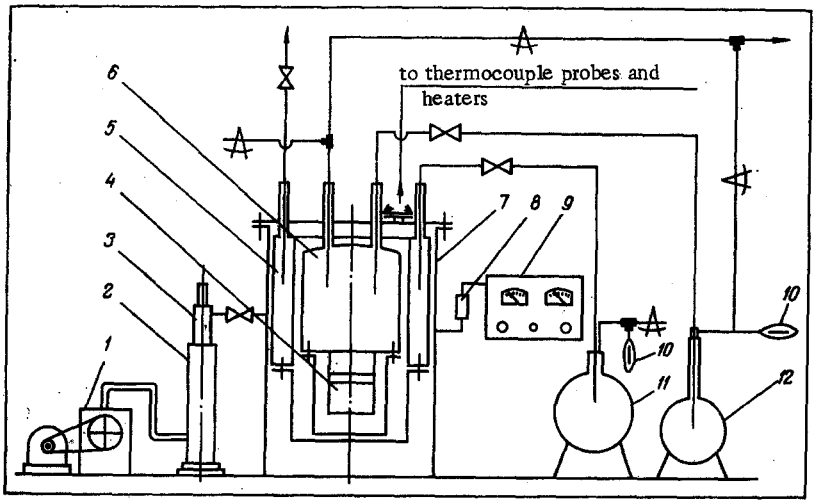

Fig. 1. Schematic diagram of the apparatus for studying the thermophysical properties of materials: 1) prevacuum pump; 2) diffusion pump; 3) nitrogen trap; 4) specimen; 5) vat with liquid nitrogen; 6) vat with liquid helium; 7) cryostat housing; 8) ion tubes for vacuum indication; 9) vacumeter; 10) chamber; 11) Dewar flask for nitrogen; 12) Dewar flask for helium.

The vacuum is checked with a thermocouple and ion tubes mounted on the cryostat housing. A model VIT-2 vacumeter for the $1 \cdot 10^{-1}$-$1 \cdot 10^{-7}$ torr range serves as a secondary measuring instrument. A vacuum level of $1 \cdot 10^{-6}$ torr is maintained by means of an adsorbent cartridge containing activated carbon and located on top of the vat with liquid nitrogen.

In order to satisfy the initial and the boundary conditions in the problem of heating an infinitely large plate from a constant-power source, it is necessary to ensure the absence of any radial temperature gradient inside the specimen and any temperature gradient along its x = 0 section, i.e.,

$$\frac{\partial T(0,\tau)}{\partial x} = 0.$$

The measuring circuit designed to satisfy these requirements is shown schematically in Fig. 2. The absence of a radial temperature gradient is ensured by means of a shield with a longitudinal temperature profile corresponding to that of the specimen. The condition $\partial T(0, \tau)/\partial x = 0$ is achieved by holding the base of the copper shield and the base of the specimen at the same temperature.

Specimen 1 of the test material, 20 mm thick and 50 mm in diameter, is surrounded by a shield 2 of the same material and, through a bearing ball 6, is pressed by base 5 against the chassis 3, the latter being in intimate contact with the liquid helium bath. The specimen and the shield are separated from the chassis by several layers of insulation in which a differential thermocouple D_1 has been installed.

Heater coils H_2 and H_3 of manganin wire 0.1 mm in diameter and with a resistance of 100 Ω each have been mounted on the surfaces of the specimen and the shield which face the chassis. A heater coil H_1 with a resistance of 150 Ω has been mounted on the chassis.

Thermocouple D_1 is used for the automatic control of adiabatic conditions between the specimen end-face and the chassis. In case of a temperature unbalance between them, thermocouple D_1 transmits a signal through a model F-116 photocompensating amplifier to a power amplifier 7, from which heater H_1 is energized. Heater H_2 is energized from a constant-power source within the 0.1-1.0 W range throughout the experiment.

Similarly, the differential thermocouple D_3, together with the heater coil H_4 mounted on the copper base of the shield and energized from the second power amplifier, both maintain a zero temperature difference between the base of the shield and the base of the specimen. The power level of heater H_3 is adjusted during the experiment so as to ensure the same temperature in the middle part of shield and specimen.

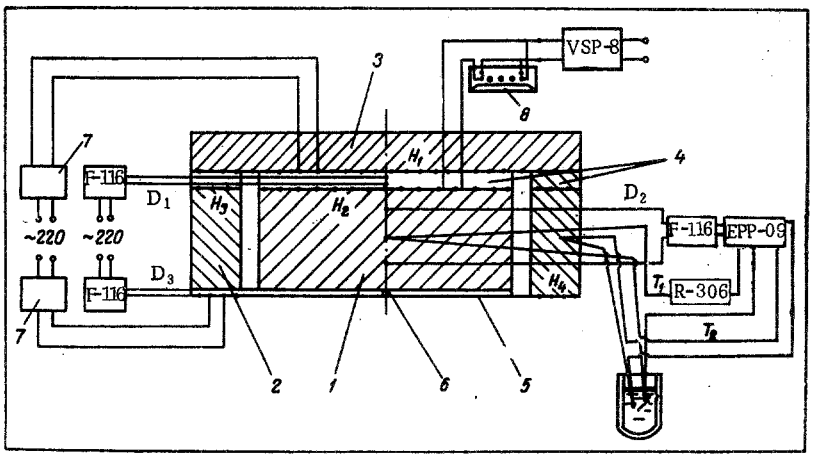

Fig. 2. Schematic diagram of the measuring and the temperature recording circuit: 1) specimen; 2) shield; 3) thermal insulator; 4) insulation; 5) base of shield; 6) bearing ball; 7) power amplifier; 8) wattmeter; H_1, H_4) compensating trim heaters; H_2) main heater of the specimen; H_3) heater for the shield; D_1, D_2, D_3) differential thermocouples; T_1, T_2) absolute thermocouples.

The temperature at the center of the specimen is measured with an absolute thermocouple T_1, while the temperature difference between two points at the respective distances x_1 and x_2 from it is measured with a differential thermocouple D_2. The thermocouples are made of gold–cobalt and copper wires 0.1 mm in diameter. The auxiliary junction of the absolute thermocouple is at the temperature of liquid nitrogen.

A signal from the absolute thermocouple is compensated with a model R-306 low-resistance potentiometer (accuracy class 0.015) and recorded by a model ÉPP-09 automatic potentiometer (accuracy class 0.5) on the 0-1 mV scale. A signal from the differential thermocouple is recorded by both potentiometers simultaneously.

The constant-power source (heater H_2) is energized from a model VSP-30 stabilized voltage supply. The heater circuit current is measured with a model M-104 milliammeter and the voltage drop across the heater is measured with a model M-1109 voltmeter.

The experiment was performed as follows. After the pressure inside the cryostat had been reduced to $1 \cdot 10^{-2}$ torr, liquid nitrogen was poured into both the operating and the standby chamber. The adsorbent lowered the cryostat pressure further down to $(2-3) \cdot 10^{-6}$ torr and maintained it at that level throughout the test. While the specimen was cooled down to 80°K, which required 3-8 h, the operating chamber was purged of liquid nitrogen and filled with liquid helium. At that time the cryostat pressure dropped by approximately one order of magnitude, while the specimen temperature dropped to 5-10°K within 5-7 h.

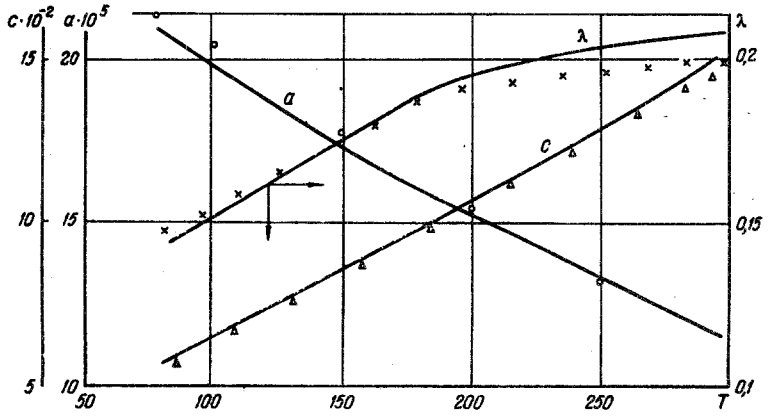

Fig. 3. Temperature-dependence of the thermophysical properties of polymethylmethacrylate (acrylic glass).

TABLE 1. Thermophysical Properties of Phenopolyurethane

T	λ	$a \cdot 10^7$	c_p	T	λ	$a \cdot 10^7$	c_p
30	0,011	40,0	75,0	170	0,02666	64,4	787,5
40	0,012	30,0	112,5	180	0,02766	61,8	918,75
50	0,1316	24,5	162,5	190	0,0286	59,4	981,25
60	0,0144	18,0	205,0	200	0,02935	57,6	1043,75
70	0,0156	11,0	256,0	210	0,0303	56,0	1106,25
80	0,0169	4,5	306,0	220	0,03111	54,4	1162,5
90	0,018	98,5	360,0	230	0,0323	53,0	1212,5
100	0,0187	92,6	420,0	240	0,0327	51,5	1262,5
110	0,0198	87,4	478,0	250	0,0311	50,0	1310,0
120	0,021	82,2	537,5	260	0,0337	48,8	1360,0
130	0,0223	77,8	592,5	270	0,0343	47,6	1212,5
140	0,233	73,8	656,2	280	0,0347	46,0	1456,25
150	0,0245	70,0	718,5	290	0,035	45,0	1510,0
160	0,0255	67,0	787,5	300	0,0353	44,5	1550,0

A basic difficulty in conducting this experiment was the establishment and the maintenance of adiabatic heating conditions in the cryostat, i.e., conditions under which all the generated power would be spent on just raising the temperature of the specimen, without any heat loss through the lateral or the end surfaces. Following the suggestions in [7], we used a photoelectric device with a continuous-duty electronic output stage for precise thermo control.

In the experiment we measured the thermophysical properties of polymethylmethacrylate with a density $\gamma = 1180$ kg/m^3 and grade PPU-308N phenopolyurethane with a density $\gamma = 49$ kg/m^3.

The test accomplished two purposes: the apparatus performance was thus checked out, and new data were obtained.

The results for polymethylmethacrylate in the 75-300°K temperature range are shown in Fig. 3, together with data from [7], for comparison. According to the graph, both sets of data agree closely within the 75-200°K temperature range.

The discrepancy of up to 10% between our test data and the results in [7] at temperatures above 200°K can be explained by our inability to maintain a less than 6-4°K temperature difference between shield and specimen. The test results prove the necessity of controlling the shield temperature with more accuracy, especially at temperatures above 200°K, because the thermal emissivity of materials increases with rising temperature.

The test results for phenopolyurethane with a density $\gamma = 49$ kg/m^3 cover the 20-300°K temperature range and are presented in Table 1.

This test was performed under a vacuum of $(2\text{-}3) \cdot 10^{-6}$ torr inside the cryostat cavity and at thermal flux densities $q = 0.1$ or 0.2 W/m^2. The level of thermal flux density was selected so as to ensure a temperature difference of not more than 5-10°K between two locations in the specimen, based on the hypothetically linear temperature dependence of the thermophysical properties [3].

The difference between the values for phenopolyurethane obtained in these two test runs did not exceed 6%.

The maximum relative error in determining the thermophysical properties of these materials by the described test method over the 20-300°K temperature range did not exceed 7%.

NOTATION

q	is the thermal flux density, W/m^2;
R	is the specimen thickness, m;
ΔT	is the temperature difference between two locations in the specimen, °K;
x_1 and x_2	are the distances from the base of the specimen to the respective location of temperature measurement, m;
$dT/d\tau$	is the heating rate for the specimen, °K/sec;
γ	is the density of the specimen material, kg/m^3;
λ	is the thermal conductivity, W/m·deg;
a	is the thermal diffusivity, m^2/sec;
c	is the specific heat, J/kg·deg;
T	is the temperature, °K.

LITERATURE CITED

1. G. M. Kondrat'ev, Regular Heating Mode [in Russian], GITTA (1954).
2. G. M. Kondrat'ev, Thermal Measurements [in Russian], Moscow–Leningrad (1957).
3. E. S. Platunov, in: Heat and Mass Transfer [in Russian], Vol. 7, Moscow–Leningrad (1966).
4. G. N. Dul'nev and A. F. Chudnovskii, in: Heat and Mass Transfer [in Russian], Vol. 7, Moscow –Leningrad (1966).
5. G. N. Dul'nev, E. S. Platunov, V. V. Kurenin, and S. E. Burovoi, in: Heat and Mass Transfer [in Russian], Vol. 7, Moscow–Leningrad (1966).
6. R. N. Mustafeev, Priborostroenie, 7, No. 3 (1964).
7. L. L. Vasil'ev and Yu. E. Fraiman, Thermophysical Properties of Poor Heat Conductors [in Russian], Minsk (1967).
8. A. D. Shnyrev and V. F. Barsukov, in: Heat and Mass Transfer at Low Temperatures [in Russian], Minsk (1970).