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STUDYING THE THERMOPHYSICAL CHARACTERISTICS OF NONMETALLIC COATINGS

Yu. A. Zagromov and V. V. Kulikov

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A method is described for experimental determination of the thermophysical characteristics of nonmetallic coatings. Experimental data for one such coating is presented.

The wide use of nonmetallic coatings in different areas of technology to protect metal from corrosion, intensify radiant heat transfer, and provide thermal insulation confronts researchers with the task of measuring the thermophysical properties of these coatings. The methodological aspects of this problem are traceable to two basic difficulties. First, obtaining measurements within the broad range of working pressures and temperatures of the coatings is fairly complex in itself. Second, most well-known methods generally require the measurement of temperature at two points. Of course, such measurements cannot be obtained in thin coatings, since the thermal resistance of the coating in this case is comparable with the thermal resistance of the contact of the temperature transducer. This produces a substantial measurement error [1].

In connection with this, we believe nonsteady methods are promising. Such methods make it possible to obtain all necessary data with regard to temperature measurement at a single point on the specimen (the temperature of the metallic substrate).

We propose a variant of the method in [2] that will more accurately determine the thermal conductivity, diffusivity, and volume specific heat of thin coatings applied to a metal substrate with a known volume specific heat.

In the heating of the surface of a coating and the thermally insulated surface of a metal substrate by a constant heat flux with the condition $\lambda_m > \lambda$, the change in substrate temperature is described in dimensionless form by the following relation [3]

$$\frac{\theta_m(Fo)}{Ki} = \frac{KFo}{1+K} - \frac{K(3+K)}{6(1+K)^2} - 2 \sum_{n=1}^{\infty} \frac{(\mu_n^2 + K^2) \cos \mu_n \exp(-\mu_n^2 Fo)}{\mu_n^2 (\mu_n^2 + K^2 + K)}, \quad (1)$$

where μ_n are the roots of the characteristic equation $\tan \mu_n = -\mu_n/K$.

When $Fo > 1.5$, quasisteady heating begins and the series in Eq. (1) can be ignored [3]. In this case, Eq. (1) is written in dimensionless form as

$$\vartheta_m(\tau) = \frac{q\tau}{c\rho l + c\rho l_m} - \frac{q l K (3 + K)}{6\lambda (1 + K)^2}, \quad (2)$$

from which, knowing the empirical relation $\Phi_m(\tau)$ (Fig. 1), we can determine the thermophysical characteristics of the coating:

$$\lambda = \frac{qlK(3+K)}{6b(1+K)^2}, \quad (3)$$

$$a = \frac{l^2(3+K)}{6\Delta\tau(1+K)}, \quad (4)$$

$$c\rho = \frac{ql}{\alpha l} - \frac{c\rho l_m}{l}. \quad (5)$$

The experimentally found value of $K = c\rho l / c\rho l_m$ is inserted in (3) and (4).

The error of λ and a depends mainly on the accuracy of the measurement of b and $\Delta\tau$, which will be represented in dimensionless form to make the analysis easier.

When $Fo > 1.5$, Eq. (1) is the equation of a straight line in the dimensionless coordinates θ and Fo which cuts off segments B and ΔFo , equal to $B = KiK(3+K)/6(1+K)^2$, $\Delta Fo = 3 + K/6(1+K)$, on the θ and Fo axes, respectively.

Figure 2 shows the change in the absolute quantity $B = f_1(Ki, K)$ compared to a similar relation for the case when the specimen is heated on the side of the substrate. When $K = 3$, both variants of the method are equivalent. However, when K is low, B , as ΔFo (Table 1), is an order greater with heating on the coating side. Thus, this variant is preferable for thin coatings.

Methodological errors may arise due to the finiteness of the thermal conductivity of the metal substrate and heat loss from its surface, the nonuniformity of the temperature field, and the dependence of the thermophysical properties on temperature. The last two of these considerations can be eliminated through a proper selection of specimen dimensions and heating time.

The effect of the thermal conductivity of the metal substrate was evaluated on the basis of a comparison of Eq. (2) with the solution of the similar problem when $\lambda_m \neq \infty$, the quasi-steady part of which appears as follows after certain transformations

$$\Phi_m = \frac{q\tau}{c\rho l + c\rho l_m} + \frac{qlK(3+K)}{6\lambda(1+K)^2} \left[1 + \frac{\beta(3K+1)}{K(K+3)} \right], \quad (6)$$

where $\beta = \frac{l_m}{\lambda_m} / \frac{l}{\lambda}$.

The error of the thermal conductivity is found from the relation

$$\delta\lambda = \left(\frac{\beta(3K+1)}{K(K+3)} \right) / \left(1 + \frac{\beta(3K+1)}{K(K+3)} \right) 100\%. \quad (7)$$

Figure 3 shows results of calculation of $\delta\lambda$ compared to similar estimates for the case of heating on the substrate side taken from [2]. It can be seen that the thermal conductivity error, given the same values of β and K , is much less for the proposed variant of the method. It can be seen from (6) that the finiteness of λ_m does not distort the measurement of volume specific heat. The heat conductivity error is equal to the right side of Eq. (7).

In conducting tests in a vacuum, the error due to radiative heat loss from the substrate surface can be ignored, since the amount by which the specimen temperature exceeds the ambient temperature is negligible.

Practical application of the method using the traditional contact technique of heating, although presenting no serious technical difficulties, poses several problems worth noting. Due to the considerable roughness of the coating surface, the thermal resistance of the contact between it and the heater, being an uncontrolled quantity, leads to a significant error — especially in measurements in a vacuum [4]. An increase in the compressive load may lead to deformation of the coating and a change in its physical properties. The use of various types of high-heat-conducting lubricants is also unsuitable for thin coatings.

To realize the method, we used a contactless method of heating the specimen with a flow of electrons. This avoided the above problems. Figure 4 shows the experimental unit. Specimens 1 in the form of 16-mm-diameter disks were connected to thermocouples in a movable holder

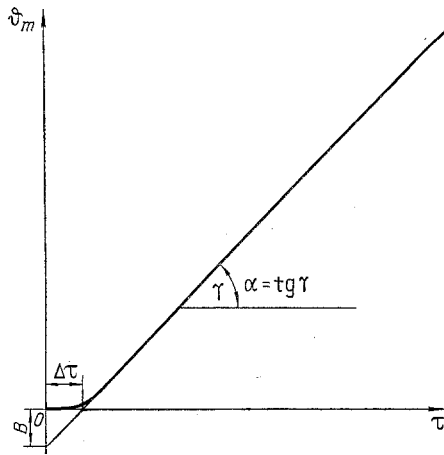


Fig. 1

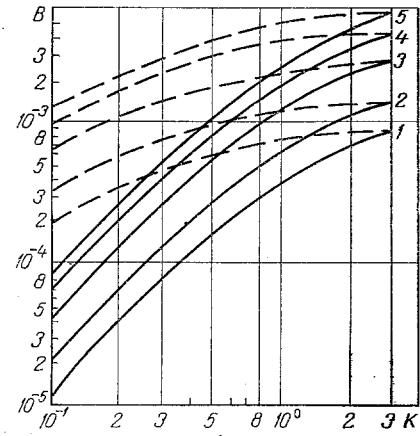


Fig. 2

Fig. 1. Toward the derivation of Eqs. (3), (4), and (5), θ_m , °C; τ , sec.

Fig. 2. Graph of dependence of absolute value of dimensionless complex B on K and Ki: 1) $Ki = 4.5 \cdot 10^{-3}$; 2) $7.5 \cdot 10^{-3}$; 3) $1.5 \cdot 10^{-2}$; 4) $2.25 \cdot 10^{-2}$; 5) $3 \cdot 10^{-2}$; dashed curves — heating on coating side; solid curves — heating on substrate side.

TABLE 1. Dependence of Absolute Value of Dimensionless Complex ΔFo on K

K	0,1	0,2	0,3	0,5	0,8	1,0	2,0	3,0
Heating on coating side								
ΔFo	0,469	0,444	0,423	0,388	0,352	0,333	0,278	0,25
Heating on substrate side								
	0,03	0,056	0,077	0,111	0,148	0,166	0,222	0,25

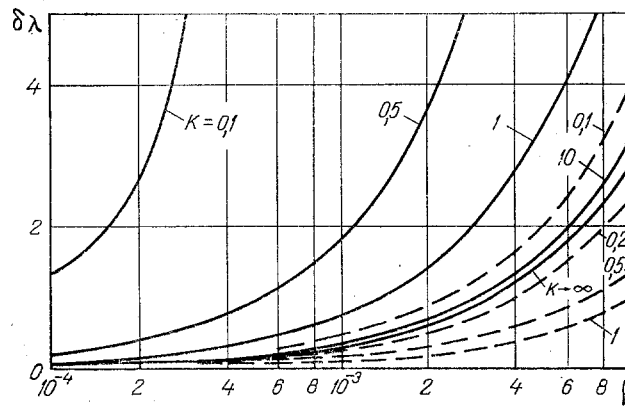


Fig. 3. Dependence of thermal conductivity error on ratio of thermal resistances of metal substrate and test coating: dashed curves — heating on coating side; solid curves — heating on substrate side. $\delta\lambda$, %.

2 placed inside the thermostatted vacuum chamber 3. The specimens were heated with a flow of accelerated, fully absorbed electrons by using an electron gun with an electrostatic deflector.

The electrons emitted by the cathode 4 are accelerated by the voltage applied to the anode 5 and deflected 90° by the field of the plates of the deflector 6, which completely eliminates heating of the specimen by radiation from the incandescent cathode. Then, passing through the suppressor grid 7, the electrons strike the specimen, which is grounded. The

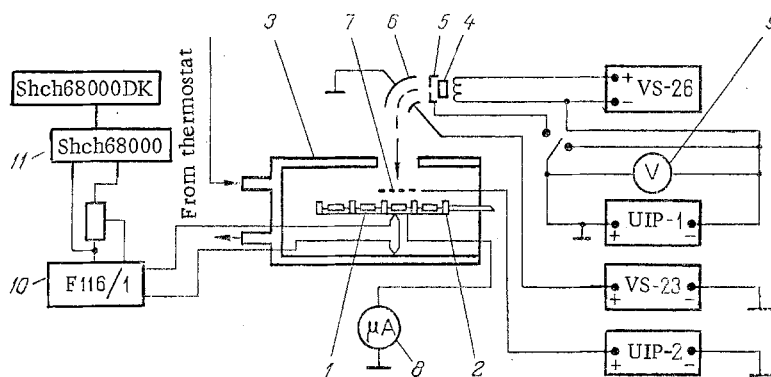


Fig. 4. Basic diagram of experimental unit.

TABLE 2. Experimental Dependences of Thermal Conductivity ($W/m \cdot ^\circ C$) and Volume Specific Heat ($J/m^3 \cdot ^\circ C$) on Temperature ($^\circ C$)

t	Heating on coating side ($l = 0,1 \cdot 10^{-3}$ m)		Heating on substrate side [2] ($l = 0,7 \cdot 10^{-3}$ m)	
	λ	$cp \cdot 10^6$	λ	$cp \cdot 10^6$
20	0,110	1,52	0,113	1,3
40	0,115	1,58	0,117	1,33
60	0,117	1,62	0,124	1,38
80	0,120	1,67	0,127	1,42

specimen current was measured with a microammeter 8, while the energy of the electrons was measured with voltmeter 9. The signal from a differential Copper-Constantan thermocouple (ϕ 0.1 mm), the cold junction of which was attached to the wall of the thermostatted volume and electrically insulated from the ground, enters photoelectric amplifier 10 and then passes through a resistance box to the input of digital voltmeter 11 and digital printer 12, making it possible to record the change in temperature with an interval of 0.04 sec. During the measurements, the cold junction is at a constant temperature equal to the initial temperature of the specimen.

We used an indirect-heat lanthanum boride cathode in the electron gun. Studies have shown that, compared to a direct-heat tungsten cathode, it gives a more uniform current density. It also has a fairly low operating temperature and is little affected by the atmosphere.

The proposed variant of the method was checked experimentally on specimens of polymethylsiloxane filled with zinc oxide. The specimens were of the same composition as those in [2] but had a thickness of 10^{-4} m. The spray-coating of the coating surface beforehand with a layer of bismuth of the order of 200-500 Å thickness ensured absorption of electrons with energies of 400-600 eV without significantly affecting the physical properties of the coating [5]. The specimen ends were also coated during the vacuum deposition to permit the charge to flow unhindered to the substrate. The resulting experimental data agrees well with the data in [2] for a similar coating (Table 2).

The experiment time was 1-2 sec for the specimens at a heat flux $q = (500-600) W/m^2$ and the heating temperature did not exceed $1^\circ C$.

In conclusion, we should note that it is best to use electronic heating for thin coatings having substantial porosity, since the pores may be spray-coated to a substantial depth when the metal is vacuum deposited. This may in turn lead to a change in the thermophysical properties of the material. In this case, it is better to use heating with a light flow.

NOTATION

$\theta = \theta(\tau)/t_0$, dimensionless temperature; $Ki = ql/\lambda t_0$, Kirpichev's criterion; $\theta(\tau) = t(\tau) - t_0$, amount by which temperature exceeds initial temperature t_0 ; τ , time; q , heat flux; l , thickness; λ , thermal conductivity; a , diffusivity; cp , volume specific heat. Index m , quantities pertaining to metal substrate of specimen.

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