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## AN EXPERIMENTAL DEVICE FOR MEASUREMENT OF THERMAL CONDUCTIVITY OF DIAMOND MONOCRYSTALS

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An experimental device and technique for measurement of thermal conductivity in diamond monocrystals are described. The temperature dependence of thermal conductivity of natural and synthetic diamonds is presented for the region 300-600°K.

There have been few studies of the thermal conductivity of diamond. The question has been considered in only a small number of Soviet and foreign articles. This is due to the methodological difficulties arising in thermophysical measurements on very small (~1 mm) highly conductive specimens such as diamond crystals. We note that the thermal conductivity  $\lambda$  of pure nitrogen-free type IIa diamond crystals is 3-5 times that of copper at room temperature [1, 2].

Almost all known data on the thermal conductivity of diamond have been obtained with natural crystals of relatively large size and regular geometric form at low temperatures, while many practical applications of natural and synthetic diamonds require knowledge of thermal conductivity at moderate or high temperatures.

The present study will describe an experimental device for determination of the thermal conductivity of small diamond crystals 1 mm in size, of any geometric form, in the temperature range 300-600°K.

The operation of the device is based on a method described in [3] and utilized in [4] to determine the ratio of the thermal conductivity of type I and IIa natural diamonds to that of copper. The method is based on measurement of constriction thermal resistance [5], developed as a result of change in thermal flux when heat is transmitted into the specimen through an area whose radius is small in comparison to the linear dimensions of the specimen. The value of this resistance is given by the formula

$$R_t = \frac{H}{\pi\lambda r}, \quad (1)$$

where H is a dimensionless function, the value of which depends on the ratio between linear specimen dimensions and the radius of the area through which heat is introduced r. For specimens with dimensions (h, height; a, radius) satisfying the condition  $h \approx a > 10r$  the value

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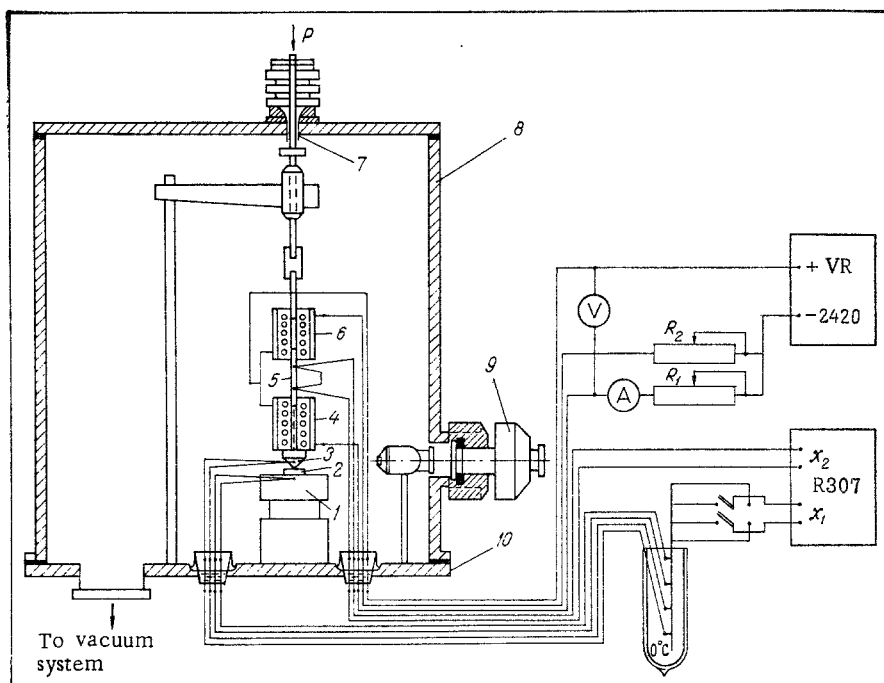


Fig. 1. Diagram of experimental device for measurement of the thermal conductivity of a diamond monocrystal.

of  $H$  may be taken as unity with satisfactory accuracy [3]. The temperature difference which develops in this case is independent of geometric form and specimen size, and at constant thermal flux is determined solely by the thermal conductivity of the material and the radius of the circular contact area, i.e.,

$$\Delta T = \frac{Q}{\pi \lambda r}. \quad (2)$$

Consequently,  $\lambda$  may be determined from Eq. (2) for given  $Q$ ,  $r$ , and  $\Delta T$ .

When using this method with materials having high  $\lambda$  values, it is very important to maintain a reliable contact with low thermal resistance of constant magnitude at the point where heat is introduced into the specimen. If a spherical diamond tip is pressed against the diamond crystal to be studied with sufficient force to produce elastic deformation, then an effective circular contact zone with low thermal resistance is formed between them [4]. This fact was in the design of our device, a diagram of which is shown in Fig. 1.

The thermal probe (thermoprobe) through which heat is introduced into the specimen is provided with a replaceable spherical diamond tip 3. The thermoprobe has two heaters: the main heater 4, which produces a constant thermal flux, and the auxiliary heater 6, which compensates for loss of heat from heater 4. Both heaters are thermally insulated on their exterior surfaces and provided with metallic screens. The heaters are separated by a thin wall German silver tube 5, to which the junctions of a differential Chromel - Copel thermocouple are attached.

The temperature sensors 2 on the specimen surface are two copper-Constantan thermocouples with a thermoelectrode diameter of  $\sim 70 \mu$ . The hot junction of the upper thermocouple is attached to the thermoprobe tip in the direct vicinity of the spherical diamond. The junction of the lower thermocouple is located in the center of the surface of heat sink 1, on which the specimen 2 is located. The thermocouple cold junctions are maintained at a constant temperature of  $0^\circ\text{C}$ . In measuring temperature differences these two thermocouples may be connected as one differential thermocouple. The heat sink 1 is located on steel plate 10 and contains vacuum leads for the thermocouples, a heater power supply, and an illuminating bulb. The heater electrical supply circuits and thermocouple connections are also shown in Fig. 1. The thermoprobe in the upper portion of the device ends in a steel rod 7, which is connected to the hydraulic system by a bellows vacuum seal. Pressure on rod 7 is continuously variable and is monitored by a reference manometer. The contact spot diameter is determined by an optical system including a microscope and an MPV-1 measurement device 9 (the illuminating bulb is not shown in the diagram).

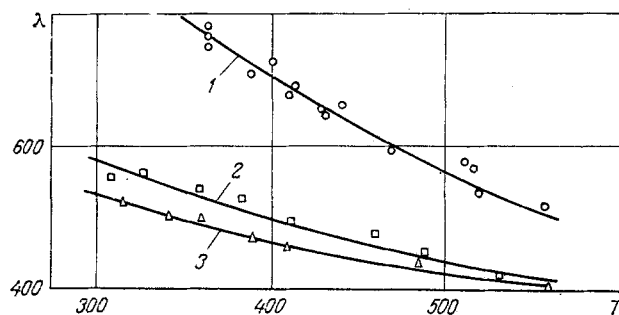


Fig. 2. Thermal conductivity  $\lambda$  [W/(M $\cdot$ °K)] versus temperature for natural (1) and synthetic (2, 3) diamond monocrystals.

The heaters are fed with dc current from a stabilized voltage source, the heater current being controlled by rheostats  $R_1$  and  $R_2$ . The power introduced into the specimen is measured by the current and voltage drop across the main heater, measured by an ammeter and a voltmeter.

The working space of the device is enclosed in a steel shell with lid 8 and connected to a vacuum system which creates a vacuum with residual pressure of  $\sim 5 \cdot 10^{-5}$  mm Hg.

The diamond crystal to be studied is first ground to obtain two plane-parallel surfaces. Then, after careful chemical processing a titanium film is vacuum deposited on one of these surfaces. The same method is used to deposit a gold film on the surface of heat sink 1. The prepared crystal is then located on the heat sink so that the titanium-coated surface is located above the thermocouple junction. The epitaxial titanium film on the diamond in contact with the gold permits a significant reduction in contact resistance at the metal-diamond interface [6]. The thermoprobe with the diamond tip is installed so that the contact spot is located in the center of the upper polished crystal surface.

The quantities  $\Delta T$ ,  $r$ , and  $Q = Iv$  appearing in Eq. (2) are measured when a stationary state with zero indication on the differential thermocouple located between the heaters is reached.

The device was tested with a specimen of known thermal conductivity. The specimen used was a type Ia natural diamond in the form of a rectangular parallelepiped with dimensions  $2.5 \times 2.5 \times 3.5$  mm<sup>3</sup>. The diamond type was determined by IR absorption spectroscopy. The thermal conductivity of the type Ia natural diamond was initially obtained by the conventional stationary method of longitudinal thermal flux and then by the method described above. The two values of  $\lambda$  thus obtained agree with each other and with the literature data for diamonds of this type [7, 8].

Temperature dependences of thermal conductivity for diamond monocrystals are presented in Fig. 2. Curve 1 is for a natural diamond, while curves 2 and 3 are for industrial synthetic diamonds with dimensions of  $1.25 \times 1.30 \times 1.15$  and  $1.5 \times 1.5 \times 1.15$  mm<sup>3</sup>, respectively. Both synthetic diamonds were cubooctahedrons with preferentially developed cube facets.

The error in thermal conductivity measurements by this method comprises  $\pm 12\%$ . Since diamonds have the greatest known thermal conductivity of any material, absolute values of the measured temperature drop are small, as a consequence of which the greatest contribution to error (to 10%) is produced by measurements of the temperature difference on the specimen. For materials with lower thermal conductivity, the accuracy of determining  $\lambda$  may be significantly higher.

The major advantage of the apparatus described herein is that it permits measurement of thermal conductivity of small crystals of irregular geometric form. If required (for example, in control measurements), its construction permits thermal conductivity measurements by the conventional stationary method of longitudinal thermal flux on larger specimens of regular form. This is accomplished by a simple change of the tips between which the specimen is held.

Moreover, such a device may be used to determine the thermal conductivity of coatings and to study contact heat exchange between different surfaces as a function of the contact pressure, gaseous medium, and temperature.

## NOTATION

$\lambda$ , specimen thermal conductivity;  $T$ , temperature;  $R_t$ , thermal resistance of constriction;  $a$ , specimen radius;  $h$ , specimen height;  $r$ , radius of area of heat introduction;  $\Delta T$ , temperature difference;  $Q$ , power introduced into specimen.

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## EFFECT OF A PHASE TRANSITION ON HEAT AND MASS TRANSFER IN DISPERSED FLOWS

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New effects are discussed which are characteristic for steady heat and mass transfer in a dispersed medium when a phase change occurs at particle surfaces.

Transport processes in real dispersed media and flows encountered in power engineering, chemical engineering, and other applications are frequently accompanied by homogeneous and heterogeneous chemical transformations and phase transitions. Examples are flows of hot liquids or unheated vapor-gas mixtures containing dispersed elements of a different phase, flows of reacting mixtures in the presence of catalyst particles, drying of granular materials, certain granulation processes, etc.

Reactions and phase transitions are significantly affected by the average temperature and concentration gradients of admixtures and reagents, and by average heat and mass fluxes, which in principle can lead to a change in the relations connecting the indicated thermodynamic forces and fluxes as compared with the relations characteristic of dispersed media with inert particles. Therefore one should expect both that the coefficients in these relations will differ from those for a homogeneous heat- and mass-transfer medium by amounts depending on the nature and intensity of the phase transitions and chemical transformations occurring and that specific cross effects, such as the effect of thermal diffusion, will appear.

As far as we know, these effects have not been specially studied. Using the general theory from [1] we show their presence by an example of a very simple problem of steady heat and mass transfer of vapor in a system with evaporating particles in the limit when the kinetics of evaporation (sublimation) is limited by the rate at which heat is supplied to the surface and can be described approximately in the temperature range of interest by a linear function

$$Q_r(T) = -LQ_c(T), \quad Q_c(T) = \alpha_1 + \alpha_2 T. \quad (1)$$

In addition, in order to concentrate our attention on the fundamental aspects of the matter and to simplify the calculations as much as possible, we make the same assumptions as

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