Thermal conductivity of composite materials in the range from 7 to 80 K used in cryogenic engineering

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Abstract. A method and a measuring device designed to determine the thermal conductivity of existing and newly created composite materials used in superconducting magnets and other objects of cryogenic technology are considered. The results of studies of the temperature dependence of the modern advanced materials thermal conductivity in the range from 7 to 80 K are shown.

Composite materials are widely used in cryogenic engineering. They have unique physical and operational properties and make it possible to manufacture structural parts in the process of creating the material itself [1]. The development of modern composite materials for the needs of cryogenic technology requires a large number of measurements of the thermal conductivity of materials, which most manufacturers, as a rule, do not indicate. This is due to the fact that the laws “On technical regulation” and “On standardization” abolished the obligation to comply with state (national) standards of manufactured products, giving them the status of “voluntary application”. However, these laws are gradually being supplemented with exceptions that abolish this “voluntariness” status in several of the most critical areas of government regulation, including nuclear energy [2]. At present, both domestic and foreign enterprises supplying various composite materials to the market do not have the obligation and ability to carry out final control over the colossal number of existing properties, especially in the cryogenic temperature range. In this regard, a measuring installation was created, designed to obtain the values of thermal conductivity of composite materials. The obtained values make it possible to confirm or obtain new data on the temperature dependence of the thermal conductivity of existing and newly created materials.

The modern most accurate methods for measuring thermal conductivity use stationary methods of a plate or cylinder [3;4]. The cryogenic measuring device created at the BINP SB RAS is based on the method of a stationary one-dimensional temperature field in a plate. To calculate the thermal conductivity \( \lambda \), the classical formula was used:

\[
\lambda = \frac{Q_0 h}{(T_H - T_X) S_0},
\]
where: $Q_0$ - heat flow in the sample; $h, S_0$ — thickness and cross-sectional area of the sample; $T_H$ and $T_X$ - the temperature of its heated and cooled surfaces.

A diagram of the plate method is shown in figure 1.

Figure 1. Schematic diagram of the method: 1 - heat sink, 2 - test sample, 3 - heater, 4 - adiabatic screen.

A sample of the test material in the form of a plane-parallel plate is installed between the surfaces of the heater and the heat sink. The stationarity of the temperature $T_0$ of the sample is ensured by the stabilization of the temperature $T_X$ of the heat sink. Heat flux $Q_0$ in the sample is set by an electric heater with a known power. To exclude heat transfer on the bottom and side surfaces of the heater, an adiabatic screen is used, the temperature of the $T_A$ of which is maintained equal to the temperature of the heater $T_H$. In this case, the value of the heat flux directed into the sample is equal to the power of the $P_H$ heater and equation (1) takes the form

$$\lambda = \frac{P_H h}{(T_H - T_X)S_0}.$$  

(2)

The reliability of the thermal conductivity values obtained by this method depends on the measurement errors of the quantities included in (2). At the same time, their main source is the discrepancy between the measured values of the $P_H$ power and the actual values of the heat flux $Q_0$, due to non-observance of the conditions of adiabaticity and stationarity. If the difference $\Delta T_{AH}$ between the temperature $T_A$ of the adiabatic screen and the heat pump of the sample heater is not zero, then in the general case heat fluxes arise between them, due to the thermal conductivity of wires ($Q_\lambda$), convention ($Q_\alpha$) and thermal radiation ($Q_\varepsilon$) [4]. In this case, a heat flux equal to

$$Q_0 = P_H \pm (Q_\lambda + Q_\alpha + Q_\varepsilon) = P_H \pm (\lambda_{AIP} S_{IP} / l + \alpha F_H + 4 \varepsilon \sigma T_H^3 F_H) \Delta T_{AH},$$  

(3)

where $P_H$ - power of the sample heater, $\lambda_{AIP}$, $S_{IP}$ and $l$ - thermal conductivity, cross-sectional area and length of wires going from the heater to the screen; $\alpha$ and $F_H$ - heat transfer coefficient on the open surface of the heater and its area; $\varepsilon$ is the reduced emissivity in the heater-screen system, $\sigma$ is the Stefan-Boltzmann constant.

Under vacuum conditions, the thermal conductivity of the residual gas, as well as the convective component of heat transfer between the heater and the screen, can be neglected ($\alpha = 0$ and $P_a = 0$).

When implementing the method, it is also important to observe the condition of stationarity, at which the heat flux $Q_V$ absorbed or released when the average temperature $T_0$ of the sample changes is negligible compared to the heat flux $Q_0$ coming from the heater. In this case, the dependence $\delta_V$ of the error on the rate $v$ of the sample temperature change has the form:

$$\delta_V = cv / Q_0 = c \rho \pi r^2 h v / Q_0.$$  

(4)
Here \( c, m, \rho, r, h \) are the specific heat, mass, density, radius and thickness of the sample. For a given value of \( \delta V \), formula (4) makes it possible to establish the requirements for temperature stability depending on the characteristics of the sample.

The installation for the implementation of the considered method was created on the basis of a KG-15 / 150-1 cryostat with liquid helium (figure 2), into which its thermal block is immersed (figure 3). The unit contains a main heater 9, surrounded by an adiabatic screen 6, which ensures the elimination of heat losses of the heater, and a heat sink 3 with a heater 4, which sets the required temperature of the sample 8. The clamping stop 10 and rods 5 provide tight contact of the main heater with the sample and heat sink. In addition, indium was used together with APIEZON cryogenic vacuum grease to improve contact. For temperature measurements, we used sensors 7, 11 and 12 based on DT-670C-CU silicon diodes (“Lake Shore” firm). The entire device was placed in vacuum chamber 2.

![Figure 2. External view of the cryogenic installation of the INP SB RAS.](image)

Heaters of sample 9, heat sink 4, and adiabatic shield 6 were connected to a three-channel power supply “AKIP-114/2” 15 with digital control. The power released by the sample heater, as well as the signals from the temperature sensors, were measured with a multichannel multimeter 13 based on an AD7794 ADC. The entire measurement process was recorded using a personal computer 14.

Calculation of the measurement error on such an installation in the range of thermal conductivity from 0.05 to 5 W / (m \cdot K) and temperature values from 7 to 80 K, provided that the temperature of the adiabatic screen differs from the heater temperature by no more than 0.02 K, and the instability of the sample temperature within 30 minutes does not exceed 0.05 K, shows that its value is less than 5\%.

Investigations of the installation error using standard thermal conductivity samples made of KV quartz glass and plexiglass (PMMA) with a diameter of 30 mm and a thickness of 10 mm confirmed the calculated result [5]. The resulting error estimate complies with the requirements of GOST 8.511-84 (GSI. State special standard and state verification scheme for measuring instruments for thermal
conductivity of solids in the temperature range from 4.2 to 90 K.) for measuring instruments for thermal conductivity in the specified temperature range.

![Figure 3. Thermal measuring installation unit: 1 - flange, 2 - vacuum chamber, 3 - heat sink, 4 - heat sink heater, 5 - fixing rod, 6 - adiabatic screen with heater, 7 - heat sink temperature sensor, 8 - sample, 9 - main heater, 10 - clamping stop, 11 - heater temperature sensor, 12 - adiabatic screen temperature sensor, 13 - multichannel multimeter, 14 - computer, 15 - power supply.](image)

Using a measuring setup, the temperature dependence of the thermal conductivity of a number of structural materials used in the manufacture of superconducting magnets based on aluminum oxide (Al2O3), boron nitride (BN), gadolinium oxysulfide (Gd2O2S), gadolinium oxide (Gd2O3), single-walled carbon nanotubes (composition: TUBALL 78%, metal impurities 12%), Resin without filler (Ed-20) was studied. The materials measured were PEEK (Polyetheretherketone) and cylindrical fiberglass. The latter are a multilayer composite material based on epoxy / ether resin, fiberglass and fiberglass from domestic manufacturers and the PRC. The results of measurements of the thermal conductivity of these composite materials are shown in table 1.

**Table 1.** Thermal conductivity of composite materials.

| №  | Sample material       | Thermal conductivity, W/(m K) |
|----|-----------------------|-------------------------------|
|    |                       | 7K                            | 80K                           |
| 1  | Ed20 / BN             | 0.082                         | 0.562                         |
| 2  | Ed 20/TUBALL          | 0.073                         | 0.157                         |
| 3  | Ed 20/Gd₂O₃           | 0.057                         | 0.225                         |
| 4  | Ed 20                 | 0.083                         | 0.157                         |
The results obtained during a series of measurements are the basis for the development of a technology for the manufacture of new composite materials in the design of superconducting magnets and other objects of cryogenic technology with given values of thermal conductivity and the nature of its temperature dependence in the region of cryogenic temperatures. The obtained results play an important role in the design of modern cryogenic and superconducting devices. The measuring installation created for this purpose allows, in addition, to carry out the incoming control of the compliance of the data on the thermal conductivity of materials received from manufacturers with its actual values.

References
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|   | Description                        | Thermal Conductivity | Density |
|---|------------------------------------|----------------------|---------|
| 5 | Ed 20/Gd$_2$O$_2$S                | 0.118                | 0.676   |
| 6 | Ed 20/Al$_2$O$_3$                 | 0.051                | 0.296   |
| 7 | PEEK                              | 0.021                | 0.175   |
| 8 | Cylindrical fiberglass (China)     | 0.078                | 0.335   |
| 9 | Cylindrical fiberglass (Russia)    | 0.068                | 0.303   |