THE USE OF THE FLAT PLATE METHOD TO INVESTIGATE THE THERMOPHYSICAL PROPERTIES OF HIGHLY EFFICIENT HEAT INSULATION AT LOW TEMPERATURES

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This paper describes the design and technical features of a calorimetric device for the investigation of the thermophysical properties of highly efficient insulation in the temperature range 4.2°–373° K.

The most efficient low-temperature insulation at present is the vacuum-multilayered type, consisting of a set of radiation screens separated by spacers with the lowest possible thermal conductivity situated in a vacuum. The mechanism of heat transfer through such insulation is very complex and at present has no exact mathematical solution in view of the large number of interdependent variables. Hence, it is of great importance to determine experimentally the thermal conductivity of various types of vacuum-multilayered insulation in relation to the following factors: temperature of boundary walls, thickness of insulation, and mechanical load on the insulation.

Of the existing three types of calorimeters—spherical, cylindrical, and flat—the last is most suitable for the proposed purpose in view of the following main advantages: a) the specimen can be fairly large in the form of disks of varying diameter; b) it can easily be mounted for the test or replaced by another; c) the thickness of the specimen can be varied in a wider range, and each side can be in contact with a hot and a cold wall; d) during the experiment a uniform load can be applied to the specimen or the packing density can be changed.

In choosing the parameters and type of calorimeter we used the experience gained in work on calorimeters designed for similar purposes and described in [1–5]. A recently published paper [6] describes a calorimeter which is similar in its characteristics to the one proposed. We think that the design of the proposed calorimeter is simpler, the question of maintaining pressure stability is tackled in a new way, and edge effects are reduced to a minimum.

Calorimeter. The calorimeter was designed and constructed in the Low-Temperature Physico-Technical Institute of the Academy of Sciences of the Ukrainian SSR in 1963–1964. It has the following main characteristics:

1. It can be used to determine the thermal conductivity of multilayered, powdered, fibrous, or cellular insulation with a heat flux density of 15–50,000 $\mu W/cm^2$ through the specimen.

2. The cold wall can have discrete temperatures in the range 4.2°–240° K, depending on the boiling point of the cryogenic liquid used.

3. The hot wall can have any temperature from 273° to 373° K and discrete temperatures in the range 77°–240° K or, if an electric heater is used, any temperature in the range 77°–273° K. During the experiment the temperature of the hot wall can be varied and accurately maintained.

4. The specimen can be tested at a residual gas pressure of 13.3 to $13.3 \cdot 10^{-6}$ N/m^2.

5. A mechanical load of 0 to $1 \cdot 10^5$ N/m^2 can be applied to the specimen during the experiment.

6. The specimen can be up to 60 mm thick. The distance between the hot and cold walls can be varied during the experiment and set to within ±0.1 mm.

Figure 1 shows a schematic section of the calorimeter, the main parts of which are the cold wall, hot wall, nitrogen screen, and vacuum chamber.

The cold wall consists of a control vessel 1 of capacity 700 cm^3 (outer diameter 100 mm) and a guard vessel 2 of 5500-cm^3 (outer diameter 230 mm) capacity. The heat flux through the specimen is determined from the rate of evaporation of the cryogenic liquid from the control vessel 1. The guard vessel 2 presents stray heat flow into the control vessel and reduces edge effects. Tube 3 (26 × 0.3 1Kh18N9T) is used to fill the control vessel with liquid, to lead off the evaporated gas, and to receive the mechanical load transmitted through the specimen to the cold wall. To prevent heat transmission to the control vessel by radiation through tube 3 a copper spring screen 4 suspended on a wire is mounted in tube 3 where the guard vessel joins it. Inside the control and guard vessels there are copper compensators 5 to prevent thermal stratification of the liquid at low heat fluxes. The copper compensators in the guard vessel also act as heat bridges when the level of the liquid is reduced. Tube 6 facilitates the exhaustion of the space between vessels 1 and 2.

The hot wall is in the form of a vessel 7 of capacity 6000 cm^3, the top of which forms the working stage, on which the specimen 8 is laid. The height of the hot wall can be adjusted through 60 mm by means of the screw 9, and the distance between the cold and hot walls is measured by the displacement indicator 10. The temperature of the hot wall is kept constant by pumping thermostated liquid through vessel 7 or by filling it with a coolant.

The nitrogen screen 11 of 4500-cm^3 capacity serves to reduce the rate of boiling off of cryogenic liquids from the guard vessel, especially in the case where liquid hydrogen or helium is used. Vessel 11, like vessels 1, 2, and 7, is made of 1.2-mm thick M1 copper.

The vacuum chamber accomodates the cryogenic vessels 1, 2, and 11 and the working stage 7 with the

specimen 8 and consists of two halves—a top half 12 and a bottom half 13. For mounting the specimen the top half of the chamber together with the cryogenic vessels is removed. The VN-2MG forepump and the N-5S diffusion pump in conjunction with the cryogenic surfaces of vessels 1, 2, and 11 provide a vacuum of up to $13.3 \cdot 10^{-6}$ N/m^2 in the chamber. The vacuum in the chamber is measured by means of a VIT-1A vacuum gauge with LT-2 and LM-2 thermocouple and ionization manometric elements. Various gases at pressures of 13.3 to $13.3 \cdot 10^{-6}$ N/m^2 can be admitted to the chamber through a leak.

The temperature distribution over the specimen 8 and the guard ring 14 is measured by means of copper-constantan thermocouples and a P-306 potentiometer with an M 17/3 galvanometer.

Maintenance of constant pressure and measurement of small gas flows. Unless special measures are taken, the gas pressure above the cryogenic liquid in the control vessel will undergo changes due to the barometric pressure and this will lead to errors in measurement of the rate of its boiling off. The effect of this error is greater, the more efficient the investigated insulating material. To maintain constant gas pressure in the control vessel a membrane manostat, as shown in Fig. 2, is mounted between the control vessel and the device for measuring the gas flow. The lower cavity of the manostat is filled with air to a fixed pressure and hermetically sealed and the whole manostat is immersed in a thermostated bath. Variations in barometric pressure are hardly transmitted to the control vessel at all, since the diameter of the opening of the manostat nozzle is 1–2 mm and that of the membrane is 70 mm.

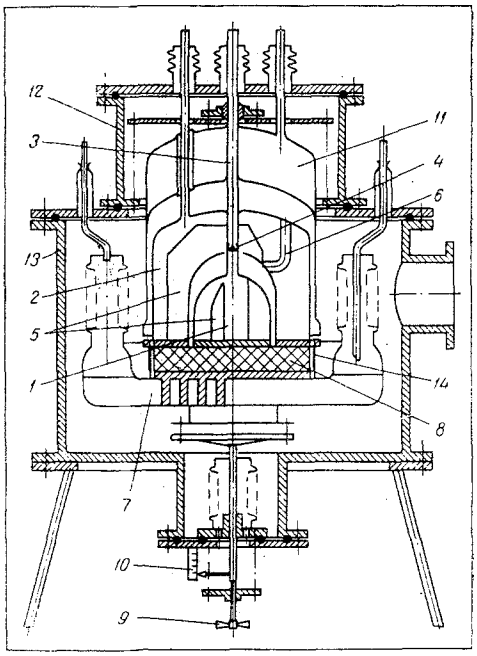

Fig. 1. Schematic section of calorimeter.

The pressure in the guard vessel is maintained slightly higher (usually 150 N/m^2) than in the control vessel by means of a bubbler to avoid condensation of gas evaporated from the control vessel on the walls of the drainage tube.

Figure 2 shows the system for measurement of small flows of gas from the control vessel. The gas flow is measured from the rate at which it displaces liquid with a low coefficient of viscosity from a graduated volume. To reduce the measurement error the liquid level in the graduated and compensation vessels is kept the same during the measurements.

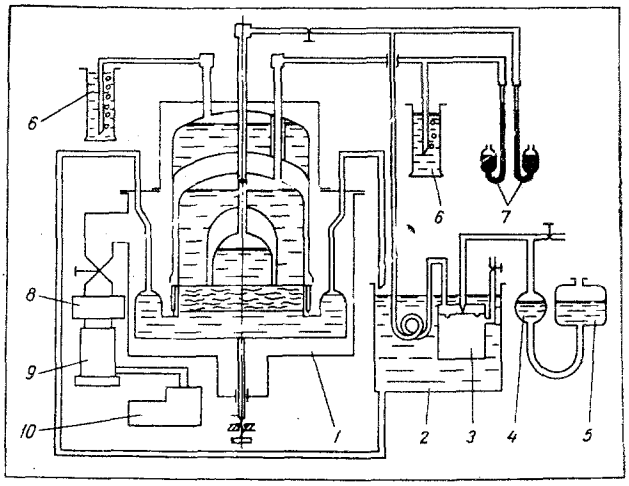

Fig. 2. Measuring system. 1) Calorimeter; 2) thermostat; 3) manostat; 4) calibrated vessel; 5) compensation vessel; 6) bubbler; 7) oil manometer; 8) nitrogen trap; 9) N-55 diffusion pump; 10) VN-2MG forepump.

Large gas flows are measured with rheometers or GSB wet gas meters.

Edge effects. In the determination of the thermal conductivity on a flat instrument it is assumed that the heat flux through the part of the specimen under the control vessel is uniform. Radiative heat transfer between the edges of the specimen and the hot surfaces of the instrument, which are at different temperatures, leads to distortion of the temperature gradient near the edges of the specimen. Owing to the great anisotropy of multilayered insulation these temperature distortions are transmitted into the specimen. One of the purposes of the guard vessel is to reduce the deviations of the heat flux through the central part of the specimen from a unidimensional flux. On the attainment of a particular thickness, depending on the kind of investigated material and its packing density, the guard part of the specimen may be inadequate. In most experiments, when the hot wall did not have to be moved, we used a foam plastic guard ring, the temperature of which over its height was close to the temperature distribution in the specimen, as Fig. 3 shows. The gap between the ring and the specimen was 1 mm. An aluminized polyethylene terephthalate film was cemented to the inner surface of the ring with the aluminum towards the specimen. The material of the specimen was riffled polyethylene terephthalate film aluminized on one side and 12.5 μ thick. The height of the specimen was 40 mm. The

Effective Thermal Conductivities of Insulating Materials

Material	Thickness of specimen, mm	Temperature of hot wall, °K	Temperature of cold wall, °K	Packing density, screens/cm	Effective thermal conductivity, μW/cm·deg	Vacuum in chamber, N/m^2
A	30	300	77/20	20	0.85/0.56	$1.3 \cdot 10^{-5}$
B	30	300	77/20	28	0.68/0.475	$1.3 \cdot 10^{-5}$
C	44	300	77/20	—	138/86.7	$5 \cdot 10^{-5}$

packing density was 27 layers/cm. Longitudinal windows 2–3 mm high were present on the hot end of the ring to facilitate exhaustion of the specimen.

Stray heat inflow. To determine the stray heat inflow to the control vessel we kept the temperature of the hot wall, like that of the cold, equal to the boiling point of the liquid nitrogen. Between the hot wall and guard vessel there was a copper ring. The experiments showed that when the pressure difference between the guard and control vessels was of the order of 150 N/m^2, the stray heat inflow was practically zero.

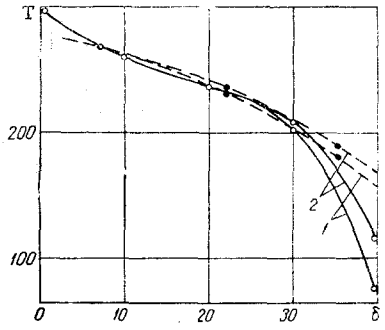

Fig. 3. Temperature distribution over height of specimen (solid line) and guard ring (broken line). 1) Boundary temperatures 300°–20° K. 2) 300°–77° K.

It should be noted that the percentage content of nitrogen in the guard and control vessels must be the same. If not, there will be either an increase in the stray heat inflow or there will be partial condensation of the gas evaporating from the control vessel.

Accuracy of measurements. The over-all accuracy of measurement of thermal conductivity on the described apparatus is ±5% from an estimate based on measurement of the volume of evaporating gas, maintenance of the pressure in the measuring vessel, and measurement of the temperature of the hot wall. The accuracy of measurement also depends on the thermal conductivity of the tested materials and increases with its increase.

However, the greatest inaccuracy in the results of the tests lies in the reproducibility of the specimens themselves. Since the materials used as spacers are not subject to thorough control as regards thickness and content of binder, there is a difference of up to 150% in the thermal conductivities for specimens of the same packing density and thickness, but prepared from different batches of material. Tests of the same specimen on two similar apparatuses gave practically the same results.

From the experiments we obtained numerous data regarding the thermal conductivity of various types of insulating materials, from cellular to vacuum-multilayered. We devoted particular attention to the latter. The table gives the data for the following materials:

A—crimped polyethylene terephthalate film aluminized on one side and 12.5 μ thick;

B—annealed aluminum foil 14 μ thick separated by SBR-M glass paper spacers 40 μ thick with elementary fibers of diameter 5–7 μ;

C—PSB foam plastic.

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