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EQUIPMENT FOR DETERMINING THE THERMAL CONDUCTIVITY OF THERMOELECTRIC MATERIALS AND THE THERMAL RESISTANCE OF CONTACT STRUCTURES USING THE ABSOLUTE METHOD

The paper presents a methodology for determining the thermal resistance of contact structures in thermoelectric energy converters based on the absolute method of thermal conductivity measurement. A modified design of the "ALTEC-10001" equipment, developed at the Institute of Thermoelectricity (Ukraine), is described for implementing this methodology. An example of research results on the thermal resistance of a contact structure consisting of a thermoelectric material with an anti-diffusion nickel coating, a copper plate, and a ceramic plate is provided. Bibl. 19, Fig. 6, Tab. 1.

Key words: thermal contact resistance, thermal conductivity, measurement, thermoelectric energy converters, absolute method.

Introduction

One of the main obstacles to the miniaturization of thermoelectric energy converters, and consequently, a significant reduction in their cost and an expansion of their practical applications, is the relatively high values of contact resistances. This is due to the fact that the influence of contact resistance on the efficiency of a thermoelectric energy converter increases as its size decreases [1-5]. Therefore, the development of methods and equipment for studying contact structures in thermoelectric energy converters, the creation of manufacturing technologies, and their optimization are important and relevant tasks.

At the same time, as the literature analysis shows [6, 7], the accuracy and reliability of methods for measuring contact thermal resistance, as well as methods for measuring electrical contact resistance, require significant improvement to practically implement the potential reduction of contact resistances predicted by theoretical studies.

The fundamentals of methods for measuring thermal contact resistance, based on the measurement of a steady-state heat flow passing through a sample in a specific direction, are outlined in the international standard ASTM D5470-06 [8]. For instance, in [9], a standard method is described that relies on using a reference sample with a predetermined thermal conductivity as a heat flow sensor.

To determine the thermal contact resistance, a slightly modified comprehensive absolute method for measuring the thermoelectric properties of materials can be used, along with the corresponding automated measurement equipment "ALTEC-10001," developed at the Institute of Thermoelectricity (Ukraine) [10-16]. This method does not require reference samples, as the heat flow through the studied structure is determined by the electrical power of the heat source, while all possible heat losses are minimized.

The goal of this work is to develop a modified design of the "ALTEC-10001" equipment for performing measurements of thermal resistance in contact structures for thermoelectric energy converters using a complex absolute method, based on the analysis of the characteristics of the physical processes involved in these measurements.

1. Description of the method for measuring thermal resistance in contact structures for thermoelectric energy converters and the design of the measuring equipment

The detailed physical model of the complex absolute method, which was used as the basis for developing the technique for determining thermal contact resistance and investigating the accuracy of this method, is shown in Fig. 1. It includes a sample of thermoelectric material (1) with metal anti-diffusion coatings (2) applied to its end surfaces, metal plates (3), ceramic contact plates (4), transitional contact layers (5), a thermostat (6), a reference heat source – an electric heater (7), as well as thermocouples T_1–T_4 for measuring the temperatures of the heater, thermostat, and the temperature gradient within the sample.

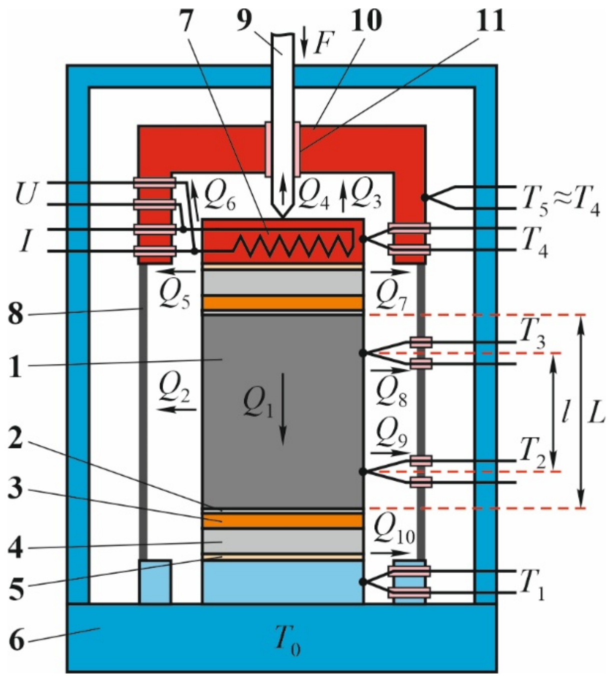

Fig. 1. The physical model of the process of measuring the thermal conductivity of thermoelectric materials and the thermal resistance of contact structures using the absolute method:
1 – thermoelectric material sample; 2 – metal anti-diffusion coatings;
3 – metal contact plates; 4 – ceramic contact plates; 5 – transitional contact layers;
6 – thermostat; 7 – electric heater; 8 – shield; 9 – clamping mechanism;
10 – shield heater; 11 – thermal switches

In Fig. 1: Q_1 – heat transferred from the sample to the thermostat; Q_2 – heat losses due to radiation from the surface of the sample; Q_3 – heat losses due to radiation from the surface of the heater; Q_4 – heat losses through the clamping mechanism; Q_5 – heat losses due to radiation from the surface of the contact structures; Q_6 – heat losses through the current and potential conductors of the heater; Q_7 – Q_{10} – heat losses through the thermocouple conductors; T_0 – temperature of the thermostat. The contact structure shown in the figure is similar to the one typically used in thermoelectric energy converters (metal anti-diffusion coatings, metal switching plates, and a ceramic base).

The thermal conductivity κ of the thermoelectric material sample is determined by the formula:

$$\kappa = \frac{W}{T_3 - T_2}\frac{l}{S}, \quad (1)$$

where: $W = I \cdot U$ is the heat flow through the sample assumed to be equal to the electrical power of the heater; T_2 i T_3 are the temperatures on the lateral surface of the sample at points located at a distance l from each other; S is the cross-sectional area of the sample.

To determine the thermal resistance R_T of the investigated contact structure, the temperature of the heater T_4, the temperature of the thermostat T_1 , and the total length of the sample L are additionally measured. Assuming that the thermal resistance of the contact structures at both ends of the sample is identical, its value can be determined using the formula:

$$R_T = \frac{1}{2}\left[\left(T_4 - T_1\right) - \frac{L}{l}\left(T_3 - T_2\right)\right]\frac{S}{W}. \quad (2)$$

As demonstrated by computer-based object-oriented modeling conducted using the Comsol Multiphysics platform [17, 18], the primary source of errors in determining both the thermal conductivity of the sample material and the thermal resistance of contact structures is heat loss due to radiation from the surface of the reference heater and the sample. It has been determined that at a temperature of T_0 = -50°C, the errors in determining the heat flux through the studied structure do not exceed 3.5% and increase to 28% at T_0 = 300°C. However, by selecting optimal values for the emissivity of the elements in the physical model, these errors can be reduced to a range of 0.7% – 3.4%.

Additionally, all thermocouple and reference heater conductors are routed outside through so-called thermal switches, which are nodes made of electrical insulators with the highest possible thermal conductivity, such as beryllium oxide. These thermal switches are designed in the form of tubes, rings, or plates. Conductors pass through openings in these elements and are brought into thermal contact with the electrical insulator. The insulator, in turn, is in thermal contact with the gradient radiation shield. In this configuration, the temperature difference along the conductors approaches zero, minimizing heat flow through them and, consequently, reducing measurement errors. A similar thermal switch is also used in the clamping mechanism to secure the sample. Overall, heat losses through the thermocouple conductors, current and potential conductors of the heater, as well as through the clamping mechanism, do not exceed 0.5% when such thermal switches are applied.

These results formed the basis for the modified design of the measuring unit of the "ALTEC-10001" equipment, developed at the Institute of Thermoelectricity (Ukraine). This modification was implemented to apply the proposed methodology for determining the thermal resistance of contact structures in conjunction with measuring the thermal conductivity of the thermoelectric material, as shown in Fig. 2.

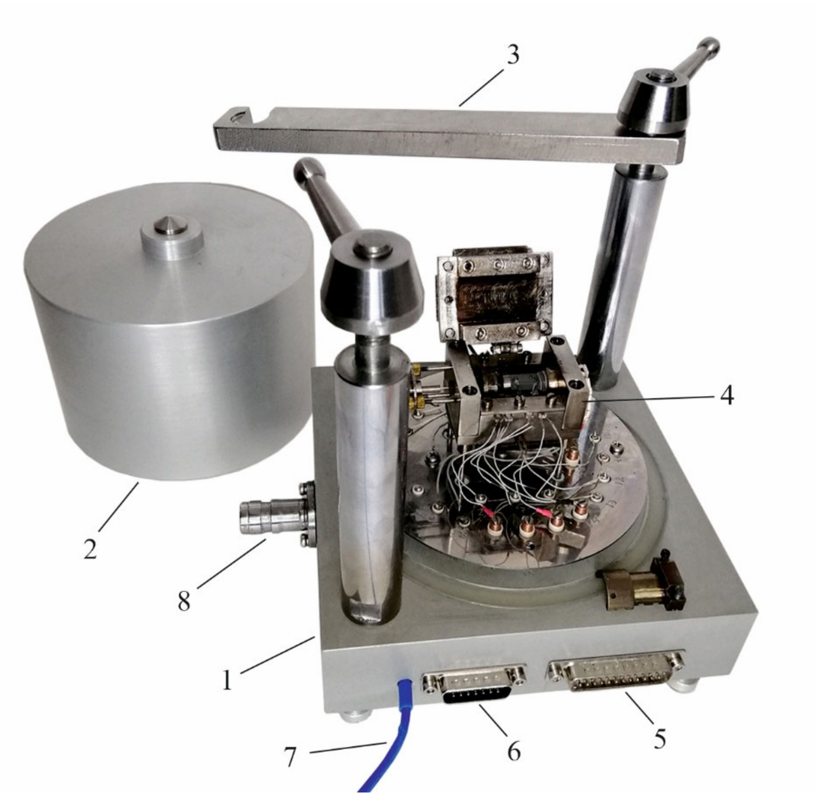

Fig. 2. Measuring unit of the "ALTEC-10001" equipment, developed at the Institute of Thermoelectricity (Ukraine): 1 – metal base; 2 – vacuum cap; 3 – clamping of the cap; 4 – measuring cell-thermostat; 5, 6 – connectors for connecting to the measurement control unit; 7 – cold junctions of the thermocouples; 8 – fitting for connecting to the vacuum pump

The diagram of the measuring cell-thermostat, in which the sample – the investigated contact structure – is placed, is shown in Fig. 3. The temperature gradient in the sample is created using the reference heater 3, which is pressed against the sample by a thin-walled tube 6.

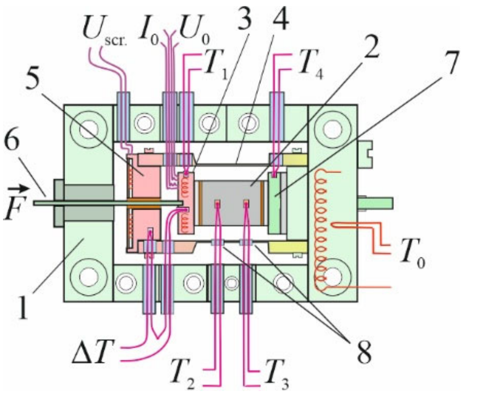

Fig. 3. Diagram of the measuring cell: 1 – thermostat housing; 2 – investigated contact structure; 3 – reference heater; 4 – shield; 5 – shield heater; 6 – clamping; 7 – mounting platform; 8 – thermal switches

To reduce heat losses from the surface of the sample and the reference heater, a gradient radiation shield 4 with a shield heater 5 is used, as well as thermal switches 8 on all the electrodes of the sample and the reference heater.

Heating elements and the thermocouple junction (T_0), used for stabilizing the temperature of the measuring unit by the thermostat, are embedded within the walls of the thermostat.

Measuring probes are embedded into the lateral surface of the sample at a certain distance from each other. These probes are made from a material with high thermal conductivity and chemical inertness relative to the sample material, such as platinum. The probes contain working junctions of the measuring differential thermocouples (T_2 and T_3). These junctions are in reliable thermal and electrical contact with the probe housings.

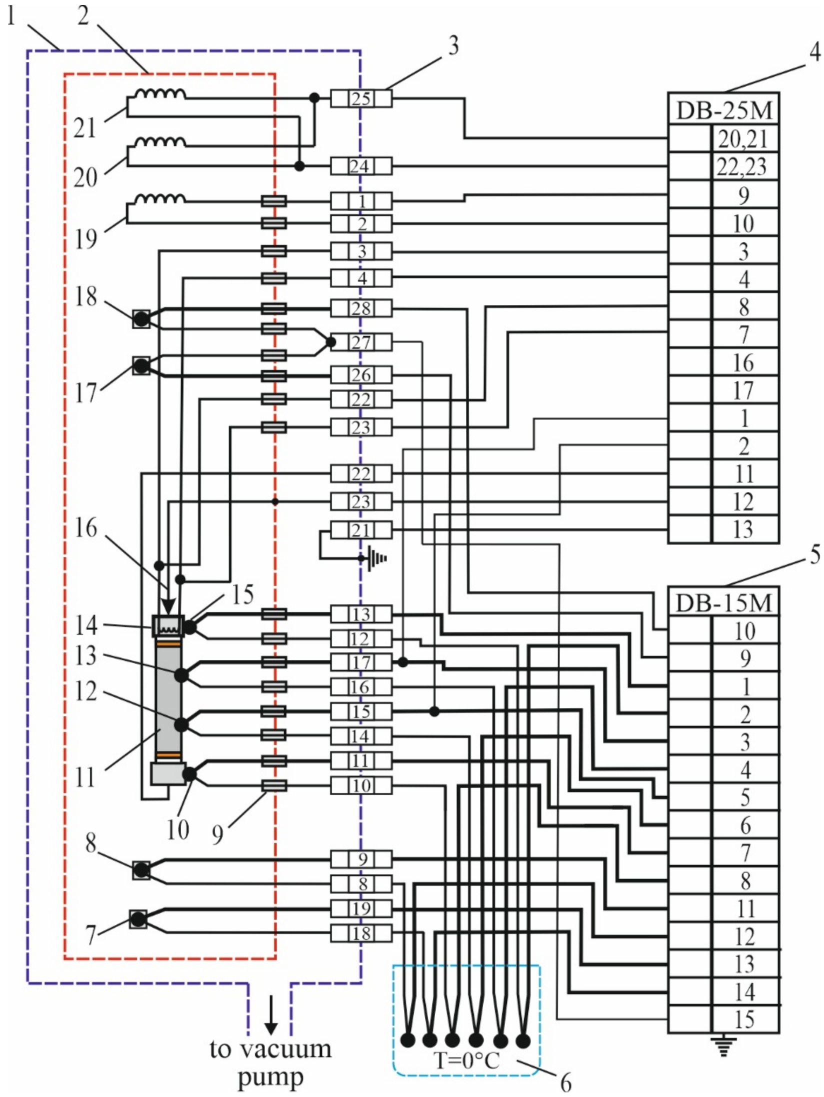

Fig. 4. Electrical circuit of the measuring unit: 1 – measuring unit; 2 – thermostat; 3 – hermetic outputs; 4 – DB-25M connector; 5 – DB-15M connector; 6 – Dewar vessel; 7 – thermocouple for the thermostat temperature controller; 8 – thermocouple for the overheating protection system; 9 – thermal switches; 10, 12, 13, 15 – thermocouples; 11 – sample; 14 – Reference heater; 16 – clamping; 17, 18 – Thermocouples for determining the temperature gradient between the sample heater and the shield; 19 – shield heater; 20, 21 – thermostat heaters

The working solders of the two additional thermocouples, necessary for determining the thermal resistance of the contact structure, are located in the reference heater (T_1) and in the mounting platform

(T_4). The free ends of all thermocouples are placed in the Dewar vessel (at a temperature of 0°C).

The electrical circuit of the measuring unit is shown in Fig. 4. The electrical outputs of the measuring unit 1 are connected to the measurement control system via two connectors 4 and 5, and the corresponding cables.

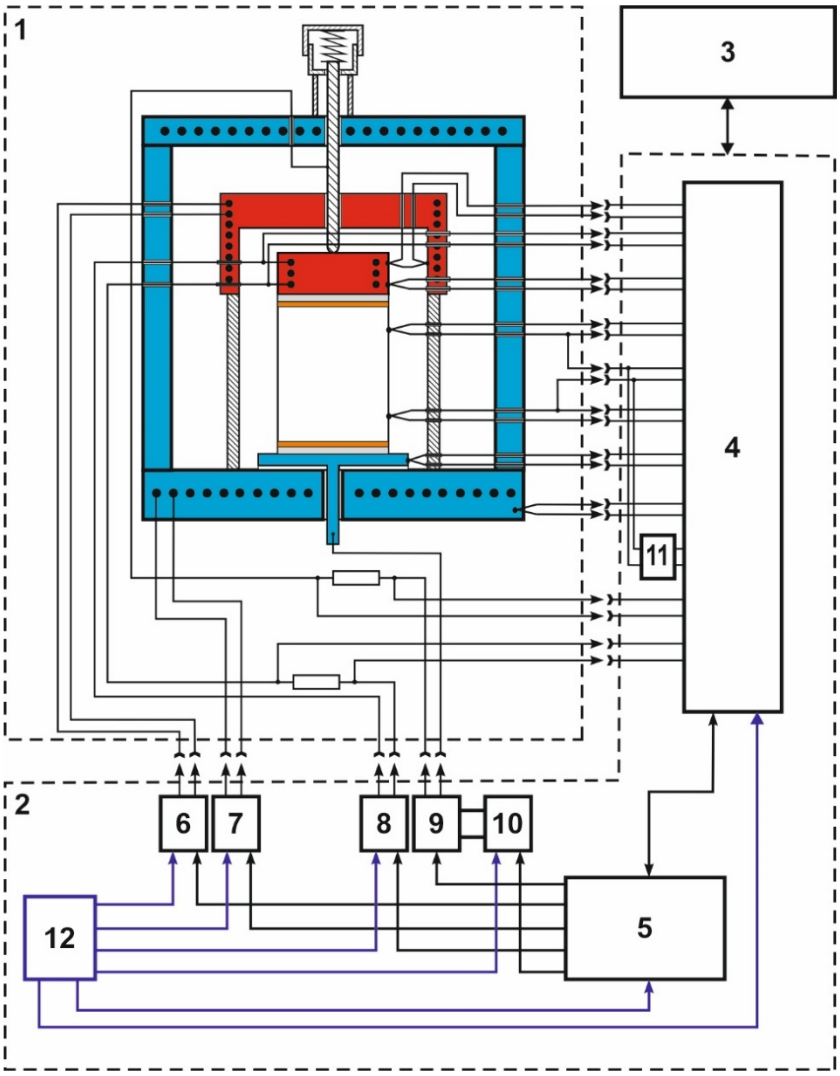

Fig. 5. The block diagram of the measurement control system: 1 – measuring unit; 2 – measurement control unit; 3 – computer; 4 – analog-to-digital converter; 5 – controller; 6, 7 – switches; 8, 10 – adjustable voltage sources; 9 – electronic switch; 11 – synchronous detector; 12 – power supply unit

The measurement control system consists of a control unit and a computer with the software "SThEM," developed by the Institute of Thermoelectricity of the National Academy of Sciences and the Ministry of Education and Science of Ukraine, in collaboration with the scientific and production enterprise "Tereks" (Ukraine). The block diagram of the system is shown in Fig. 5.

The measurement control unit contains:

– Means of setting and maintaining the temperature of the measuring thermostat over a wide temperature range (temperature controller, power supply unit, reference thermocouple, etc.);

– Adjustable power supply unit for passing current through the sample, current switch;

– Adjustable power supply unit for the reference heater;

– Means of maintaining zero temperature gradient between the reference and shield heaters (temperature controller, power supply unit, reference zero thermocouple, etc.);

– High-precision voltage meter;

– The ability to set the required switching on/off cycle for the power supply units and the moments for recording measurement results of all measurement channels (temperatures of the "hot" and "cold" probes, voltage drop between the probes, current and voltage through the sample, current and voltage of the reference heater);

– The ability to transmit measurement results to a computer for further processing, construction of graphs and tables, and the creation of a sample passport.

The system uses a 24-bit 8-channel analog-to-digital converter (ADC) with differential inputs, which has a voltage measurement range of ± (5 μV - 2.5 V). The differential inputs of the ADC allow for high-precision voltage measurements in the electrical circuits of various units with different power sources.

The system also includes intelligent switches 6 and 7 with their own protection circuits against short circuits and overcurrent. The use of such switches ensures high reliability of the setup, preventing it from malfunctioning in the event of issues in the power circuits. The use of modern field-effect transistors, manufactured using MOSFET technology, with low resistance in the open state, reduces heat dissipation, which made it possible to avoid the use of heat sinks. These switches are capable of switching electrical power up to 600 W.

A regulated stable voltage source 8 is used to power the reference heater with a stable voltage. It is capable of providing a power supply to the reference heater of up to 10 W.

The electronic switch 9 allows for controlling the current through the sample, changing the current direction, and supplying alternating current to the sample. This enables the measurement of the electrical conductivity of the thermoelectric material when the studied contact structure does not contain insulation layers. The switch is constructed in an H-bridge configuration based on powerful MOSFET transistors. The current through the electronic switch can be adjusted within the range of 0.01 to 8.0 A. The regulated stable current source 10 provides a current through the sample within the range of 0.05 to 4.0 A. The synchronous detector 11 allows for accurately detecting alternating voltages and measuring the pulse voltage values across the sample. The synchronous detector is constructed with a key scheme, featuring various discrete inputs that, when controlled, enable different operating modes during the passage of pulsed current through the sample.

The programmable controller 5 contains two PID PWM temperature regulators, the discrete outputs of which ensure the automatic or manual operation of the system according to the set cycle diagrams and operating algorithms, as well as an input for processing the signal received from the ADC.

The power supply unit 12 is built on the basis of a power toroidal transformer and linear voltage regulators without the high-frequency noise typical of switch-mode power supplies, which improves the accuracy of the measurements.

The measurement block is connected to a personal computer 3 via a USB channel, where measurement cycles are set, necessary calculations are performed, graphs are constructed, and measurement protocols are generated.

2. Experimental Research Results

A series of experimental studies on the thermal resistance of contact structures and the thermal conductivity of thermoelectric materials included in their composition were conducted. The structure under investigation is shown in Fig. 6. It consisted of a *Bi-Te*-based thermoelectric material 1 in the form of a rectangular parallelepiped with a length of 10 mm and a cross-section of 5x5 mm. On its end face, an anti-diffusion nickel coating with a thickness of 3-5 μm was applied using a typical technology for manufacturing thermoelectric energy converters, and copper plates 2 with a thickness of 0.25 mm and ceramic plates 3 with a thickness of 0.63 mm made of aluminum oxide were soldered. To mount the measuring probes-thermocouples into the side surface of the sample, holes 4 were made in the sample at certain distances from each other. The length of the sample L and the distance between the probes l were determined using an instrumental microscope with a resolution of 0.001 mm. The equipment described in detail in [19] was used to manufacture the samples.

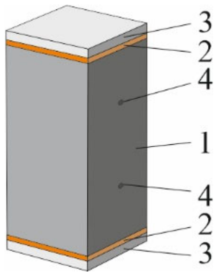

Fig. 6. The investigated contact structure: 1 – thermoelectric material; 2 – copper plates; 3 – ceramic plates; 4 – holes for thermocouple probes

The sample of the contact structure under investigation was mounted in a measurement cell with different variants of heat transfer between the end surfaces of the sample and the surfaces of the reference heater and the mounting platform of the thermostat: "dry" contact, kerosene, and silicone thermal conductive paste.

The measurement process was controlled using the "SThEM" software in manual mode. A temperature gradient of approximately 10°C was created on the sample using the reference heater. At the same time, the thermostat provided power to the screen heater in such a way that the temperatures of the screen heater and the sample were equal. After establishing steady-state conditions, the temperatures $T_1 – T_4$ and the electrical power of the reference heater W were recorded. The values of thermal conductivity and thermal contact resistance were calculated using formulas (1) and (2), respectively. The measurement results (at a temperature of T_0 = 20°C) are presented in Table 1.

Table. 1.

The results of the measurements of thermal conductivity of thermoelectric materials and thermal resistance of contact structures with different heat transfer variants

No.	Heat transfer between the end surfaces of the investigated structure and the surfaces of the reference heater and thermostat.	Thermal conductivity of a thermoelectric material, W/(m·K)	The value of the specific thermal contact resistance of the contact structure, 10^{-5} K·m²/W
1	"Dry" contact	2.07	9.41
2	Kerosene	2.04	5.43
3	Silicone thermal conductive paste	2.11	8.51

As seen in the table, the best results are achieved with the heat transfer method that uses kerosene to improve the thermal contact between the surfaces. However, in practice, its application is problematic. At the same time, the use of silicone thermal conductive grease only differs from the "dry" contact by approximately 10%. The deviations in the measured values of the thermal conductivity of the thermoelectric material sample are within 3.5%, which corresponds to the technical specifications of the

"Altec-10001" equipment for the case of using samples with a square cross-section.

The developed equipment will be useful for further research on contact structures of different compositions, their optimization, and the creation of manufacturing technologies.

Conclusions

1. The detailed physical model of the process of measuring thermal conductivity of thermoelectric materials and thermal resistance of contact structures for thermoelectric energy converters has been considered. Through computer modeling, it has been shown that the measurement errors of these quantities are primarily determined by the amount of heat loss from the surface of the reference heater and the sample. These losses, in the temperature range from -50 to 300°C, can be instrumentally reduced to a level of 0.7 – 3.4%.
2. The modified design of the "ALTEC-10001" equipment, developed at the Institute of Thermoelectricity (Ukraine), for implementing the proposed methodology for determining the thermal resistance of contact structures is described. The results of studies on the thermal resistance of the contact structure, consisting of thermoelectric material with an anti-diffusion nickel coating, copper and ceramic plates, for various variants of heat transitions between the surfaces of the ceramic plates and the surfaces of the reference heater and thermostat, are presented.

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ОБЛАДНАННЯ ДЛЯ ВИЗНАЧЕННЯ ТЕПЛОПРОВІДНОСТІ ТЕРМОЕЛЕКТРИЧНИХ МАТЕРІАЛІВ ТА ТЕПЛОВОГО ОПОРУ КОНТАКТНИХ СТРУКТУР АБСОЛЮТНИМ МЕТОДОМ

У роботі наведено методику визначення теплового опору контактних структур для термоелектричних перетворювачів енергії, що базується на абсолютному методі вимірювання теплопровідності. Описано модифіковану конструкцію обладнання «АЛТЕК-10001», розробленого в Інституті термоелектрики (Україна), для реалізації такої методики. Наведено приклад результатів досліджень теплового опору контактної структури, що складається з термоелектричного матеріалу з антидифузійним нікелевим покриттям, мідної та керамічної пластин. Бібл. 19, рис. 6, табл. 1.

Ключові слова: тепловий контактний опір, теплопровідність, вимірювання, термоелектричні перетворювачі енергії, абсолютний метод.

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