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SETUP FOR MEASURING THE ELECTRICAL CONTACT RESISTANCE OF "METAL – THERMOELECTRIC MATERIAL" STRUCTURE

The paper presents a method for determining the electrical contact resistance of "metal - thermoelectric material" structure based on a probe method with thermostating of one side of the contact structure under study. The design of the experimental setup for implementing such a measurement method is described. An example of the results of studies on the contact resistance of "nickel – Bi-Te based extruded thermoelectric material" structure is presented. Bibl. 27, Figs. 8, Tabl. 1.

Key words: electrical contact resistance, probe method, measurement, Peltier effect, thermoelectric energy converters.

Introduction

Thermoelectricity finds more and more practical applications in various fields [1–8]. According to estimates [9], the market of thermoelectric products today is more than 900 million US dollars and grows by approximately 9% every year. More than 60 million thermoelectric modules are produced.

The use of thermoelectricity for waste heat recovery is particularly attractive [10]. Almost all technological processes in industry, as well as the production of electrical energy, are associated with the use of fuels, including nuclear, to produce thermal energy. Most of this energy in industry, after the implementation of technological processes, is dissipated into the environment by gaseous or liquid coolants. In thermal engines, only 25–40% of thermal energy is converted into mechanical energy. The remaining more than 50% is given to the environment, which leads to its thermal pollution and disruption of the Earth's thermal balance.

At the same time, the main obstacle to the widespread practical use of thermoelectric recuperators is their relatively high cost, primarily due to the high cost of the thermoelectric material from which they are made.

The cost of thermoelectric energy converters can be reduced by tens of times and approach the cost required for wide practical applications of thermoelectricity due to their miniaturization. However, attempts to create miniature modules encounter the increasing influence of contact resistances, which significantly reduce the quality of the modules [9, 11–15].

To develop technologies for manufacturing contact structures with optimal contact resistance values, it is necessary to conduct a number of experimental studies, which are possible only with the availability of accurate methods and modern equipment for measuring the parameters of thermoelectric materials and modules based on them [16–20].

Measuring the resistance of contact structures in thermoelectric energy converters is also extremely important. At the same time, as the analysis of the literature shows [21, 22], such equipment has not yet been created. The developed methods for measuring electrical contact resistance in thermoelectricity can be divided into those based on measuring the characteristics of "packet" contact structures, which are a series of thermoelements with series-connected contact resistances; probe and microelectronic methods, which require the manufacture of test structures.

In [23], the probe method of measuring the electrical contact resistance of "metal – thermoelectric material" structure is considered and the results of the analysis of possible measurement errors and computer optimization of this method to achieve their minimum values are presented. It is shown that deviations from isothermal conditions in the sample, caused by the influence of the Joule and Peltier effects, can lead to very significant (over 100%) errors in measurements, and it is proposed to use samples with a rectangular cross-section and thermostating their wider side to reduce them.

The purpose of this work is as follows. Based on the analysis of the features of physical processes that occur when measuring the electrical contact resistance of "metal-thermoelectric material" structure by the probe method, to develop a design of the setup for implementing this measurement method with thermostating of the investigated contact structure.

1. Description of the method for measuring the electrical contact resistance and the design of the measuring setup

A physical model of the probe method for measuring the electrical contact resistance of "metal – thermoelectric material" structure is shown in Fig. 1.

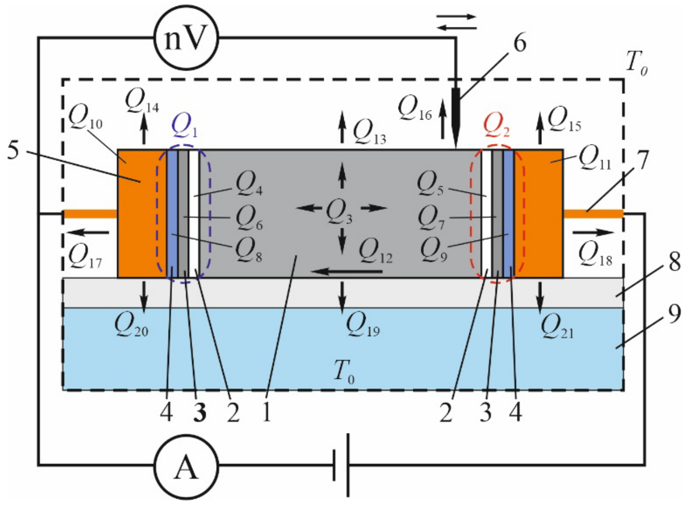

Fig. 1. Physical model of measuring the electrical contact resistance of "metal-thermoelectric material" structure by the probe method with thermostating of the sample:
1 – thermoelectric material sample; 2 – transient contact layer;
3 – metal anti-diffusion coating; 4 – solder; 5 – metal contact plates;
6 – movable potential probe; 7 – current leads; 8 –electrical insulator; 9 – thermostat

The structure under study consists of a sample of thermoelectric material 1 with a metal (e.g., nickel) coating 3 applied to its ends, a transient contact layer 2, a solder layer 4, and metal (e.g., copper) contact plates 5. On the side surface of the sample, a probe in the form of a pointed plate or needle is located, which measures the potential distribution along the sample when a direct electric current of magnitude *I* is passed through it. To minimize the influence of the Peltier and Joule effects, the sample is glued on one side to an electrical insulator with high thermal conductivity (for example, beryllium oxide ceramics) and placed on a thermostated surface.

In Fig. 1: T_0 – ambient temperature; Q_1 – the Peltier heat absorbed at "metal-thermoelectric material" contact; Q_2 – the Peltier heat released at "thermoelectric material-metal" contact; Q_3 – the Joule heat released in the bulk of thermoelectric material sample; Q_4, Q_5 – the Joule heat released on transient contact layers; Q_6, Q_7 – the Joule heat released in the bulk of metal anti-diffusion coatings; Q_8, Q_9 – the Joule heat released in the bulk of solder layers; Q_{10}, Q_{11} – the Joule heat released in the bulk of metal contact plates; Q_{12} – heat flux from the hot to cold contact of "metal-thermoelectric material" structure; Q_{13}, Q_{14}, Q_{15} – heat transfer from the lateral sample surface and metal contact plates to the environment by radiation and convection; Q_{16}, Q_{17}, Q_{18} – heat transfer from the lateral sample surface and metal contact plates to the environment through potential probe and current leads; Q_{19}, Q_{20}, Q_{21} – heat transfer from the lateral sample surface and metal contact plates to thermostat.

The value of the electrical contact resistance of "metal – thermoelectric material" structure is determined by the formula

$$\rho_c = \frac{S}{I} \cdot \Delta U \,, \tag{1}$$

where ρ_c – specific electrical contact resistance of "metal-thermoelectric material" structure, ΔU – voltage drop on the contact, S – contact area.

As shown by computer object-oriented simulation using the Comsol Multiphysics application package [24, 25], when using thermostating even on only one side of the sample, the non-isothermality in it is significantly reduced (Fig. 2). The error in determining the contact resistance caused by such non-isothermality will not exceed 2% [23].

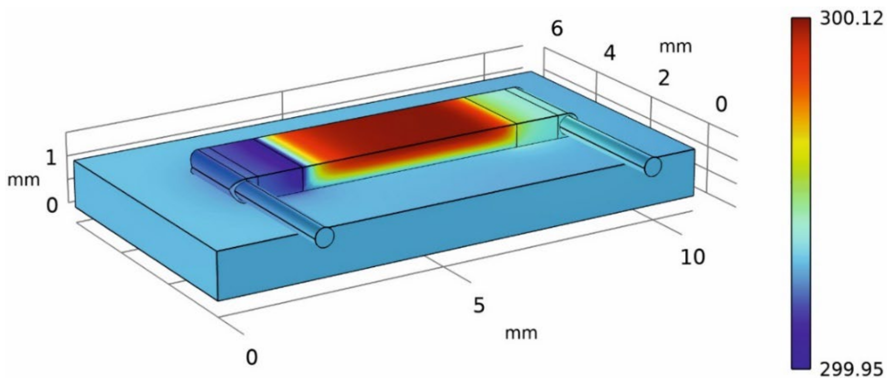

Fig. 2. Temperature distribution in the studied contact structure during thermostating of one of its surfaces (for the case of a sample with a length of 5 mm and a cross-section of 0.5 mm by 2 mm, at a current of 0.5 A; contact resistance 10^{-6} Ohm·cm^2).

The design of the measuring unit of the setup developed at the Institute of Thermoelectricity (Ukraine) for implementing the probe method of measuring the electrical contact resistance of a "metal – thermoelectric material" structure with thermostating of the sample is shown in Fig. 3.

On the movable coordinate table of the microscope, in a round niche, a base metal (from D16T) plate 6 is installed. A copper pedestal 5 is attached to it with screws, for installing a beryllium plate with samples 4 of the studied contact structures glued to it. To increase productivity, several samples can be glued to the plate (usually 2–4 pieces). Current leads from the samples are soldered to a separate small terminal block (not shown in the figure), which is fixed on a copper pedestal in the immediate vicinity of the crystals. The small terminal block, in turn, is connected by conductors to the common terminal block, to which the remaining elements of the measuring circuit are connected.

The beryllium ceramic plate 4 is pressed against the copper pedestal 5 using two overlay plates with four screws. To improve the thermal contact of the plate with the pedestal, thermally conductive grease is used, and the samples are glued to the ceramics with a thermally conductive adhesive on a special fixture – to ensure the accuracy and identity of their installation.

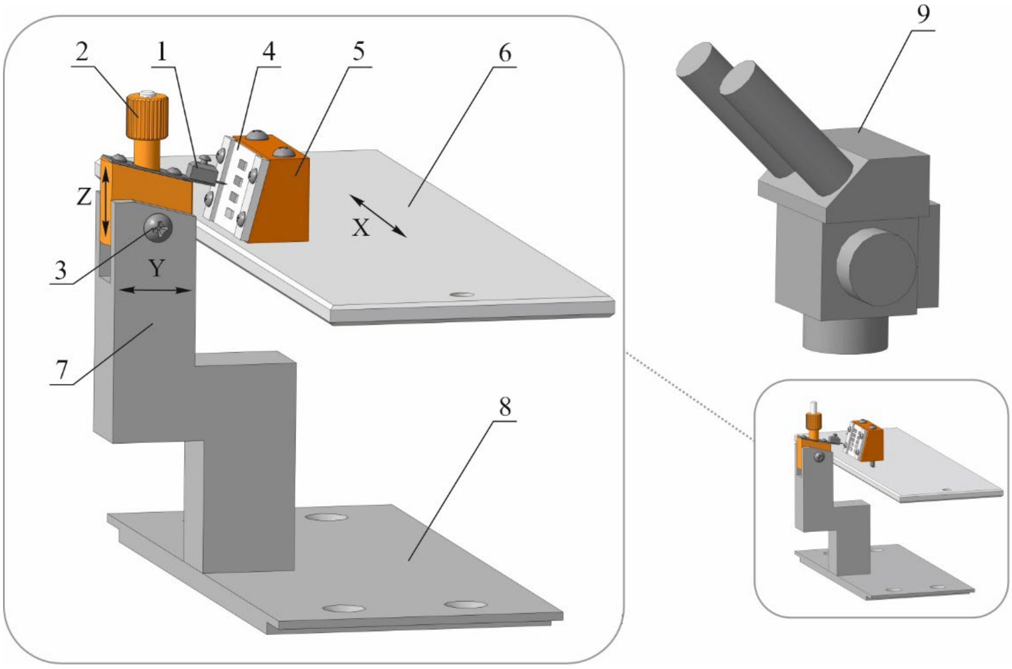

Fig. 3. Design of the measuring unit of the setup for measuring the electrical contact resistance of "metal–thermoelectric material" structure: 1 – measuring probe; 2 – screw for vertical movement of the probe; 3 – side screw for rigid fixation of the probe; 4 – beryllium ceramic plate to which the samples under study are glued; 5 – copper pedestal; 6 – metal base attached to the microscope coordinate table; 7 – bracket; 8 – metal base attached to the lower fixed cast-iron frame of the microscope; 9 – microscope

A plate 8 is attached to the lower fixed cast-iron frame of the microscope, and an intermediate bracket 7 is attached to it – for carrying the probe assembly 1 (fixed in the X and Y coordinates) to the working area of the bench. This bracket 7 contains a mechanism for vertical movement (Z axis) of the probe assembly. The movement "up – down" is carried out using a screw 2, and its rigid fixation is carried out using a side screw 3. The probe itself is a replaceable, sharply sharpened steel needle, up to 0.3 mm in diameter, which is held in place by a screw. The probe also has a spring damper along the axis of the probe needle, preventing the crystal surface from being damaged when contact is made.

For convenience in observing the movement of the probe and bringing it to the sample, the platform of the pedestal with the samples is inclined to the axis of view of the inspection microscope 9 at an angle of 15°. The axis of the probe is inclined horizontally at the same angle, so that it is perpendicular to the plane of the sample.

The voltage drop when a stabilized direct current passes through the sample is measured by moving the probe and touching the surface of the sample at points along its centerline in height.

Reliable contact with the sample surface, while minimizing the penetration of the probe needle tip into its surface, is ensured by an electronic sound alarm circuit, which is triggered when the needle tip touches the surface. When reliable contact is achieved, the sound alarm circuit is turned off using the "sound" toggle switch so that the electrical potentials from the circuit do not distort the measuring voltages.

The electrical connection diagram of the measuring unit is shown in Fig. 4.

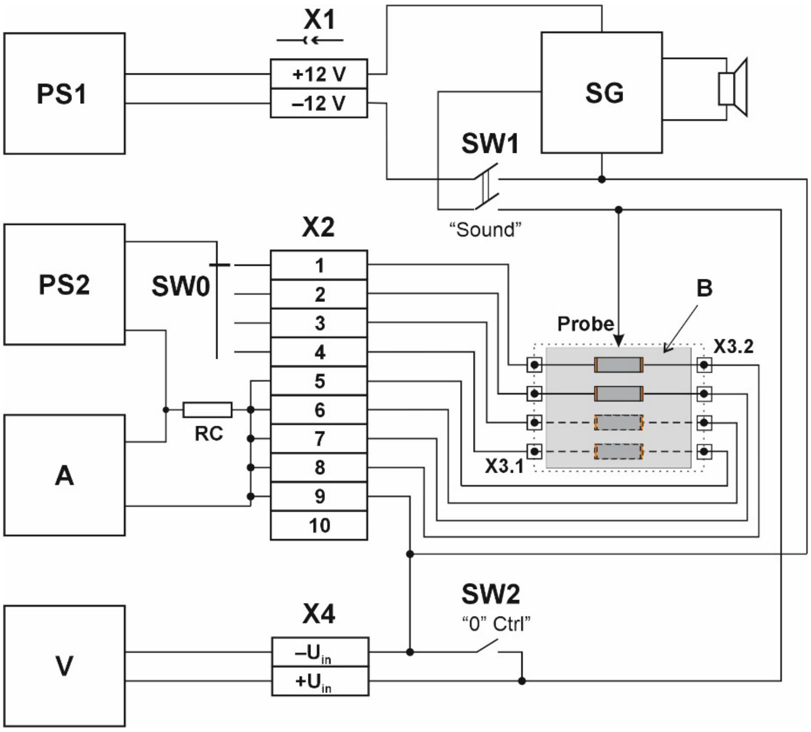

Fig. 4. Electrical connection diagram of the setup for measuring the electrical contact resistance of "metal – thermoelectric material" structure:
PS1 – power supply unit of the sound alarm; SG – sound generator board; PS2 – DC source for crystals (IT5962A device); SW0 – switch for supplying current to crystals; RC – resistance coil P-321 for measuring current; A – device for measuring current through the crystal (M3500A multimeter); B – board with glued crystals; V – voltmeter for measuring voltage drop across the crystal; SW1 – switch for disabling the sound alarm; SW2 – switch for shortening the voltmeter input; X1–X4 – terminals

The appearance of the developed setup for measuring the electrical contact resistance of "metal – thermoelectric material" structure is shown in Fig. 5.

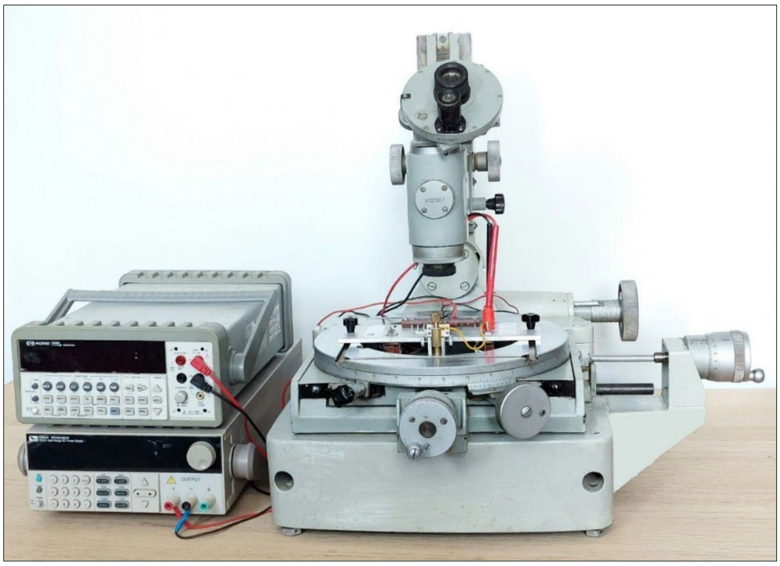

Fig. 5. Appearance of the setup for measuring the electrical contact resistance of "metal – thermoelectric material" structure

2. Results of experimental studies

In the contact zone of the sample, when current passes, a voltage drop occurs on: a copper bus with a thickness of 0.25 mm, which is soldered to the ends of the sample; a solder layer between the copper bus and a nickel layer with a thickness of 3–5 μm; an anti-diffusion contact layer of nickel with a thickness of 2–2.5 μm; a contact resistance between the surface of the anti-diffusion layer of nickel and the surface of the thermoelectric material; a layer of thermoelectric material between the surface of the nickel contact layer and the tip of the movable probe. The total voltage drop across the solder and nickel layers at the specified layer thicknesses and a current of 0.5 A does not exceed 0.1 μV, so when determining the contact resistance, such a voltage drop can be neglected. The voltage drop across the 0.25 mm thick copper bus is taken into account as a correction.

The total voltage drop between the copper busbar and the point of contact of the point probe to the surface of the thermoelectric material sample is equal to the sum of the voltage drop across the contact resistance and the voltage drop across the section of the thermoelectric material sample from the location of the contact layer and the location of the point probe Accordingly, the total value of the contact resistance is determined as the difference between the total resistance (between the copper bus and the point of contact of the point probe with the sample surface, taking into account the correction) and the ohmic resistance of the corresponding section of the thermoelectric material. Therefore, formula (1) for determining the specific contact resistance can be rewritten as

$$\rho_c = \frac{S}{I}\cdot\left[\left(U - U_M\right) - \frac{l_{Ni} - l_1}{l_2 - l_1}\left(U_2 - U_1\right)\right], \tag{2}$$

where S is the cross-sectional area of the sample, I is current through the sample, U_M is the correction for the voltage drop on copper, l_{Ni} is the coordinate of the nickel layer, l_1 is the coordinate of the probe on the material (position 1), l_2 is the coordinate of the probe on the material for determining electrical conductivity (position 2).

Thus, the determination of the specific contact resistance is reduced to geometric measurements of the exact position of the nickel layer, the exact position of the probe tip at position 1 (at a distance of approximately 100–150 μm from the nickel layer), the exact position of the probe tip at position 2 (at a distance of approximately 400 μm from position 1), the thickness and width of the sample, and electrical measurements of the current and voltage on the probe at positions 1 and 2.

To reduce the measurement error of the specific contact resistance, a precision nanovoltmeter was used for electrical measurements. The total error of electrical measurements when determining the contact resistance on the setup does not exceed 0.5%.

A series of experimental studies of the contact resistance of the "nickel – Bi-Te based extruded thermoelectric material" structure were conducted at the Institute of Thermoelectricity using the developed measuring setup.

The initial blanks of extruded thermoelectric materials had a cylindrical shape. Samples were cut out in the form of disks with a thickness of 5.0 mm, which, after applying a nickel layer to them, were cut on a wire-cutting machine ALTEC-13009 [26, 27] into plates with a width of 2.0 mm. Copper buses with a width of 2.0 mm and a thickness of 0.25 mm were soldered to the ends of such plates with a nickel layer. Following that, the plates with soldered copper buses were again cut on a wire cutting machine into samples with a thickness of 0.5 mm. As a result, the obtained samples have the shape of parallelepipeds 5 mm long with a cross section of 0.5×2.0 mm and with copper contact buses soldered on the ends.

To create a contact anti-diffusion layer of nickel on the surface of thermoelectric material samples, an electrochemical method of deposition from an electrolyte was used. The thickness of the nickel layer was 2.5 μm. To achieve the correct geometric shape of the samples and remove roughness from their surface, before applying nickel, the samples were polished on an abrasive, after which their thickness and width were measured. Prior to applying the nickel layer, the contact surface of the samples was etched to remove defects that arose during the dimensional processing of the surfaces. Fig. 6 shows an enlarged image of the surface of one of the samples after surface etching (microscope magnification ×400).

The samples processed in this way were glued with the wide side using thermally conductive adhesive to the surface of a plate made of thermally conductive beryllium oxide ceramics (Fig. 7).

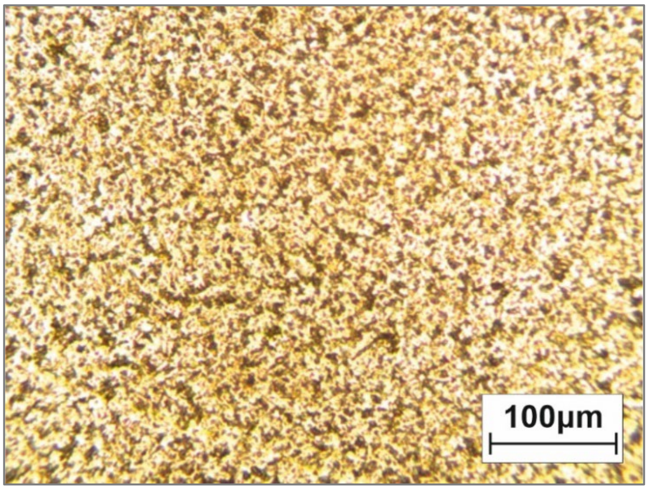

Fig. 6. Surface of a sample of extruded n-type thermoelectric material after grinding on micropowder with a grain size of less than 1 μm and etching

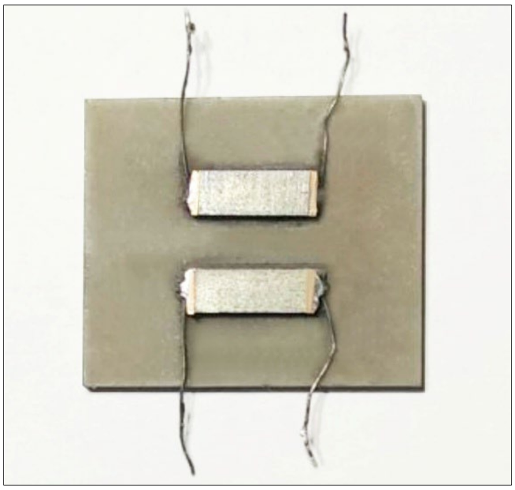

Fig. 7. Beryllium oxide substrate with samples for contact resistance measurement

The ceramic substrate with the samples was inserted and fixed in the measuring cell of the device for measuring contact resistance (Fig. 8).

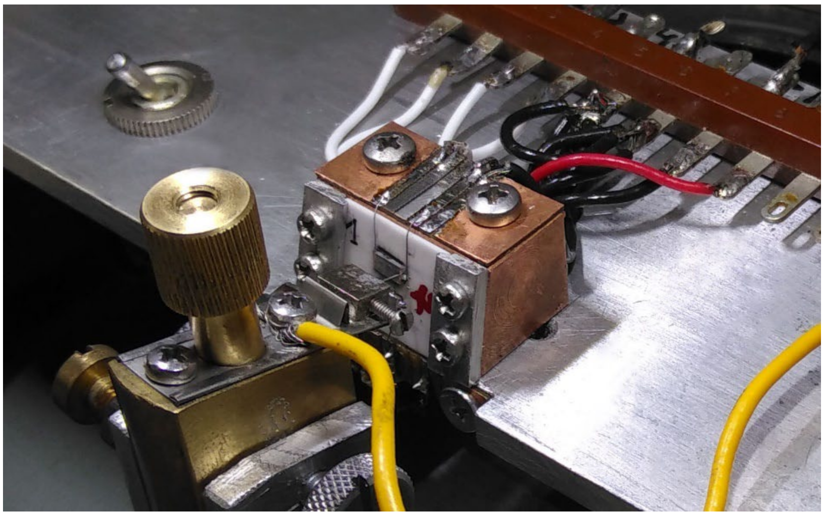

Fig. 8. Measuring cell with test samples placed on a ceramic substrate

The results of contact resistance measurements on 8 samples of extruded thermoelectric material, the surface of which was treated with diamond micropowders of different fractions before electrochemical deposition of the nickel layer, are given in Table 1.

From the analysis of the obtained contact resistance values, a relationship between the surface roughness of the thermoelectric material and the contact resistance values follows. The minimum contact resistance values are achieved for the polished surface of thermoelectric materials.

Table 1

Results of measuring the electrical contact resistance of "nickel – Bi-Te based extruded thermoelectric material" structure

No.	Thermoelectric material conductivity type	Fraction of diamond micropowders with which the surface of the thermoelectric material was treated, microns	The value of specific contact resistance, 10^{-6} Ohm cm^2
1	n-type	40/28	8.1
2	p-type	40/28	5.0
3	n-type	20/14	4.0
4	p-type	20/14	3.0
5	n-type	5/3	1.4
6	p-type	5/3	1.3
7	n-type	<1.0	0.8
8	p-type	<1.0	0.9

The obtained contact resistance values at minimum surface roughness values are on a par with known analogues.

Conclusions

1. A detailed physical model of the process for determining the electrical contact resistance of "metal – thermoelectric material" structure was considered. Using computer simulation it was shown that the error in determining the electrical contact resistance caused by the nonisothermality of the investigated contact structure can be considerably reduced by means of thermostating one of its sides and under such conditions will not exceed 2%.
2. The design of the experimental setup developed at the Institute of Thermoelectricity (Ukraine) for implementing such a measurement technique is described. An example of the results of studies of the contact resistance of "nickel – Bi-Te based extruded thermoelectric material" structure is given. A relationship has been established between the surface roughness of a thermoelectric material and the values of specific contact resistance – the minimum values of specific contact resistance are achieved for the polished surface of thermoelectric materials.

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УСТАНОВКА ДЛЯ ВИМІРЮВАННЯ ЕЛЕКТРИЧНОГО КОНТАКТНОГО ОПОРУ СТРУКТУРИ «МЕТАЛ – ТЕРМОЕЛЕКТРИЧНИЙ МАТЕРІАЛ»

У роботі наведено методику визначення електричного контактного опору «метал – термоелектричний матеріал» на основі зондового методу з термостатуванням однієї зі сторін досліджуваної контактної структури. Описано конструкцію експериментальної установки для реалізації такої методики вимірювань. Наведено приклад результатів досліджень контактного опору структури "нікель – екструдований термоелектричний матеріал на основі Bi-Te". Бібл. 27, рис. 8, табл. 1.

Ключові слова: електричний контактний опір, зондовий метод, вимірювання, ефект Пельтьє, термоелектричні перетворювачі енергії.

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