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METHODS AND EQUIPMENT FOR THE PREPARATION OF THERMOELECTRIC MATERIAL SAMPLES FOR MEASURING THEIR PROPERTIES BY THE ABSOLUTE METHOD

The importance of high-quality preparation of the studied samples of thermoelectric materials for measuring their properties using a complex absolute method is shown. The requirements for the samples under study are given, as well as a description of the methods and equipment for producing samples that meet these requirements. Bibl. 27, Figs. 11.
Key words: measurement, electric conductivity, Seebeck coefficient, thermal conductivity, figure of merit, absolute method.

Introduction

The importance of improving methods and equipment for measuring the properties of thermoelectric materials cannot be overestimated. High requirements for the accuracy of measuring equipment are put forward both when solving the problems of finding new and optimizing known thermoelectric materials to increase their thermoelectric figure of merit [1-3], and when choosing the optimal thermoelectric material for specific practical applications of thermoelectric energy converters [4-13].

One of the best for determining the thermoelectric properties of materials (electrical conductivity, Seebeck coefficient, thermal conductivity and figure of merit Z) is the complex absolute method [14-16]. It is widely used in creating standards and has important advantages: measurements of σ, α, κ, Z are performed simultaneously on the same sample, which reduces errors; small samples can be used for measurements; thermoelectric parameters are found from classical formulae without making amendments.

Papers [17-26] give the results of comprehensive research conducted at the Institute of Thermoelectricity of the National Academy of Sciences of Ukraine and the Ministry of Education and Science of Ukraine, aimed at developing methods for minimizing the errors of the absolute method. The result of these studies is the creation of "ALTEC-10001" measuring equipment (Fig. 1), the accuracy of which in determining the figure of merit is 3-5 times higher than the accuracy of measurement when using other methods, in particular, the Harman method [27].

Fig. 1. "ALTEC-10001" installation for measuring the thermoelectric properties of materials by the complex absolute method, developed at the Institute of Thermoelectricity of the National Academy of Sciences and the Ministry of Education and Science of Ukraine.

From the analysis of possible measurement errors, it follows that it is necessary to ensure high requirements for the sample being measured - the accuracy of its dimensions, the correctness of its shape, the quality of its surface, uniformity, etc. It is extremely important to create high-quality electrical and thermal contacts on the end surfaces of the sample and install thermocouples on its side surface. However, ensuring compliance with these requirements is not always given due attention, although deviations from them lead to such significant errors that the use of all necessary methods to improve measurement accuracy may become useless.

Therefore, *the purpose of this work* was to develop special methods and equipment for preparing samples for measurements.

1. Requirements for the studied samples of thermoelectric material

Fig. 2 shows a diagram of the complex absolute method taken as a basis when creating the “ALTEC-10001” installation. The thermoelectric parameters of the sample under study are determined from the formulae

$$\sigma = \frac{I}{U}\frac{l}{S}, \tag{1}$$

$$\alpha = \frac{E_\alpha}{T_h - T_c}, \tag{2}$$

$$\kappa = \frac{I_0 \cdot U_0}{T_h - T_c}\frac{l}{S}, \tag{3}$$

$$Z = \frac{\alpha^2 \sigma}{\kappa}, \tag{4}$$

where l is the distance between thermocouple probes; S is cross-sectional area of the sample; I, U are current through the sample and voltage drop between measuring probes when determining electrical conductivity; E_α is thermoEMF between identical legs of thermocouple probes; T_h and T_c are the “hot” and “cold” temperatures in the sample; I_0, U_0 are current and supply voltage of the reference heater.

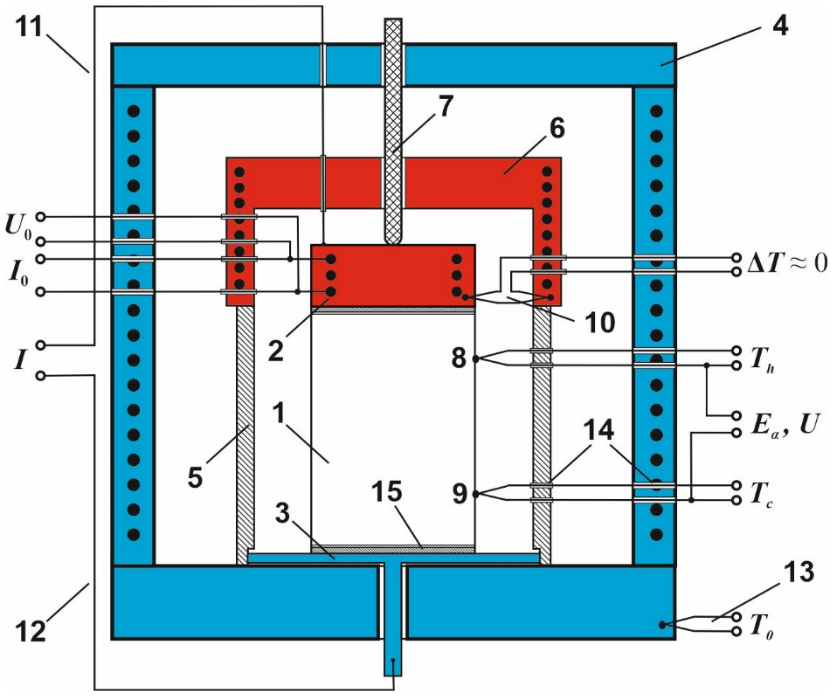

Fig. 2. Complex absolute method of measuring thermoelectric parameters of materials: 1 – sample under study; 2 – reference heater; 3 – mounting pad; 4 – thermostat; 5 – screen; 6 – screen heater; 7 – clamp; 8, 9 – measuring probes-thermocouples; 10 – zero thermocouple; 11, 12 – current leads of the sample; 13 – thermocouple of the thermostat thermoregulator; 14 – thermal keys; 15 – contact structures.

Papers [17, 18] examined in detail the influence of various physical factors on the accuracy of measurements of parameters of thermoelectric materials, primarily thermal conductivity and electrical conductivity, depending on the geometric dimensions of the samples and structural elements of the measuring cell. It has been shown that measurement errors can be significant, more than 50%, if special means are not used to eliminate them.

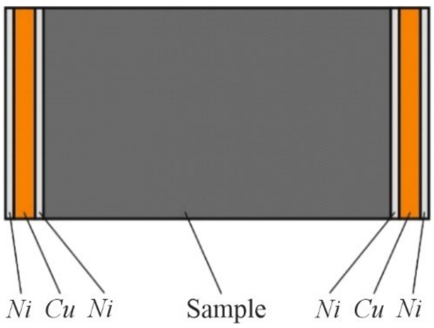

Fig. 3. Contact structures for measuring the thermoelectric properties of material by the absolute method.

It has been established that cylindrical samples with a diameter of 6-8 mm are optimal for ensuring minimal measurement errors and time to establish stationary conditions. In doing so, the length of the sample should be at least 2 times greater than the diameter of the sample, and the distance between the probes should not exceed ⅓ of the length of the sample.

In addition, as shown in [19], in order to achieve a one-dimensional distribution of electric current and heat flow in the sample, it is necessary to metallize the ends of the sample. The layers must be resistant to temperature effects in the entire working interval of measurements and have fairly good adhesion and anti-diffusion properties. A set of metal coatings was determined (Fig. 3), which ensures acceptable values of errors in thermal conductivity and electrical conductivity measurements. The optimal contact structure created on the ends of the sample consists of anti-diffusion nickel layers with a thickness of ~ 10 μm and a copper layer with a thickness of ~ 100 μm.

2. Production of thermoelectric material samples

The process of cutting thermoelectric material has its own specific features, so the direct use of modern serial equipment for cutting semiconductors is not always justified in relation to thermoelectric material.

The Institute of Thermoelectricity of the National Academy of Sciences and the Ministry of Education and Science of Ukraine has developed a small-sized desktop machine "ALTEC-13009" for cutting thermoelectric material with free and bound abrasive.

The basis of the cutting tool is the replaceable frame 1 (Fig. 4), made of high-strength aluminum alloy. Tungsten wire 3 is wound on the frame. The distance between the wires is set by the grooves of the size bars 2. Diamond micropowder is applied to the surface of the wire.

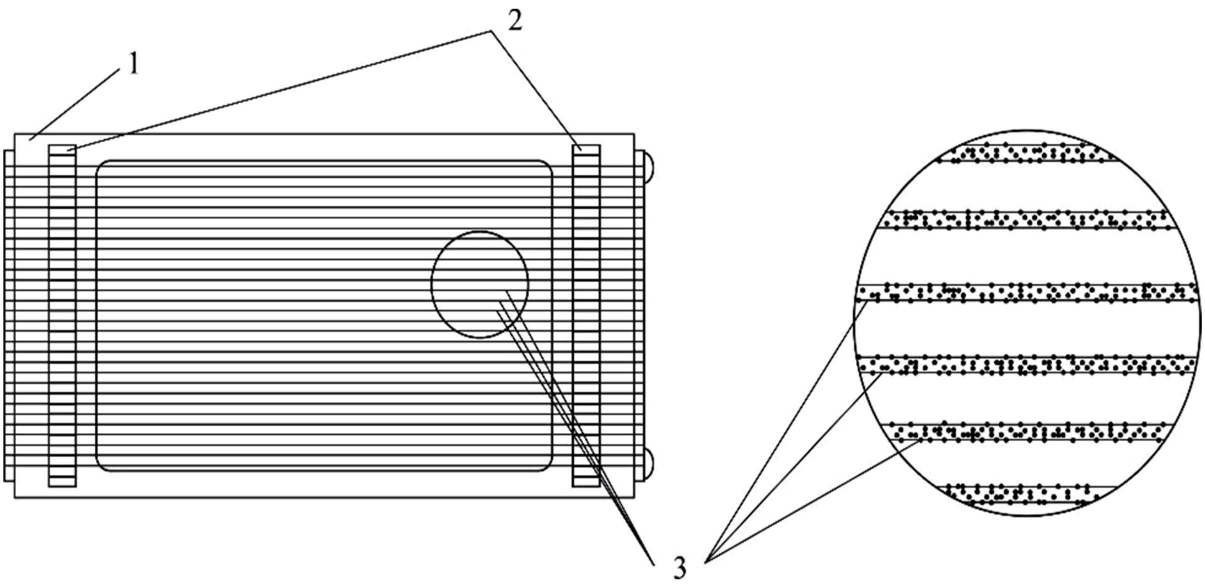

Fig. 4. Tool for cutting with wires with fixed diamond grains: 1 – tool, 2 – size bars; 3 – wires with fixed diamond grains.

A small-sized desktop machine is designed for producing rectangular-shaped samples from thermoelectric material in laboratory conditions (Fig. 5). The machine is schematically shown in Fig. 6.

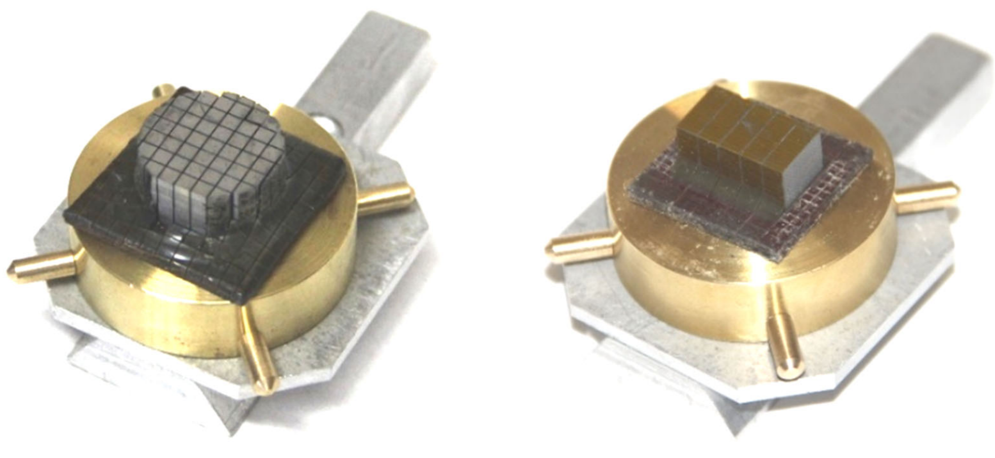

Fig. 5. Appearance of the result of cutting the thermoelectric material with the "ALTEC-13009" desktop machine.

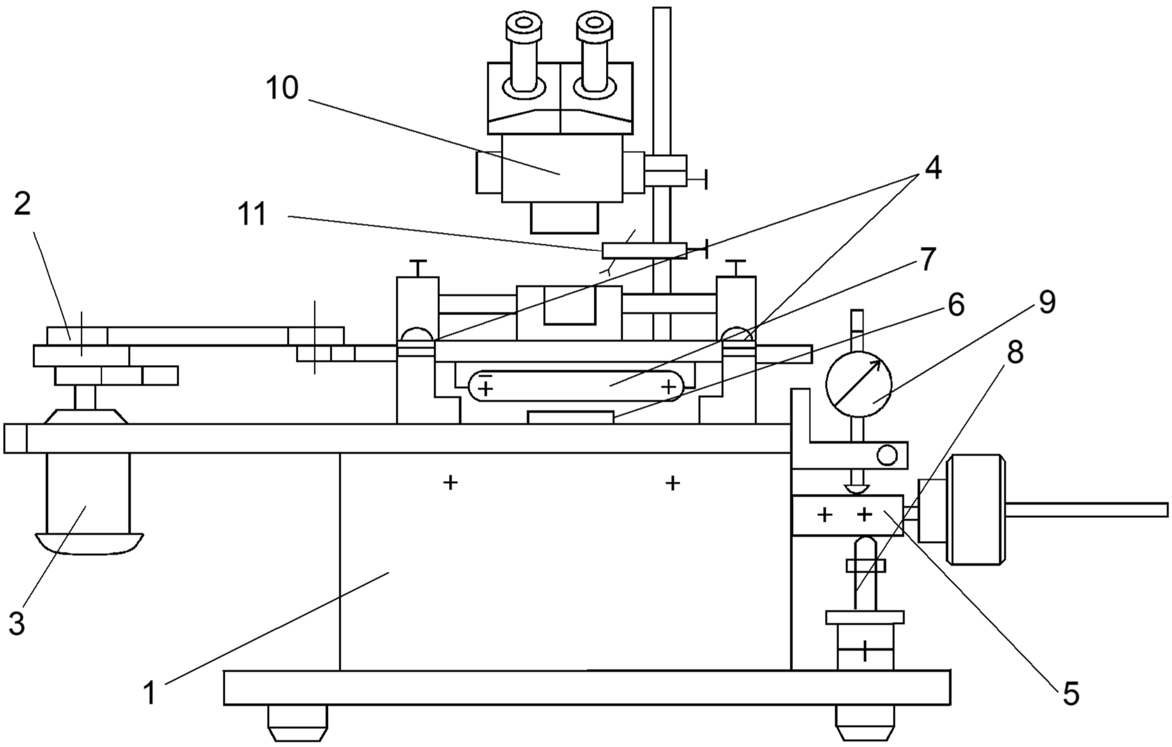

Fig. 6. Schematic of a small-sized desktop machine for cutting thermoelectric material: 1 – bed; 2 – drive unit; 3 – electric motor; 4 – carriage fastening unit; 5 – mechanism for raising and lowering the table; 6 – material to be cut; 7 – cutting tool; 8 – system for regulation and control of cutting depth; 9 – indicator; 10 – microscope; 11 – coolant supply.

The working tool of the machine is a frame with wires arranged parallel to it. Guides installed on the frame set the required distance between the wires and, accordingly, the dimensions of the samples. The machine allows cutting under conditions of small deforming influences. This achieves minor violations of the surface layers of the material.

The frame is secured to the movable carriage using two clamping nuts. Using the same nuts, the cutting wires are aligned parallel to the direction of movement of the tool. Bearing sliding of the carriage guides ensures the accuracy and ease of their reciprocating movement.

The machine consists of a carriage with a cutting tool 7, the reciprocating movement of which is carried out by the drive unit 2 from the electric motor 3; carriage fastening units 4; mechanism for raising and lowering the table 5 with a counterweight for adjusting the pressure on the edge of the cutting tool; system for control of cutting depth 8, indicator 9 for controlling the cutting depth; device for supplying coolant 11. The beginning and end of the cutting process is controlled by the indicator 9. The appearance of the small-sized desktop machine "ALTEC-13009" is shown in Fig. 7.

The error when cutting a thermoelectric material with a free abrasive is 0.01 mm with a depth of violation of the surface layer of 5-15 μm; when using a cutting tool coated with diamond abrasive - 0.02 mm with a depth of violation of the surface layer of 10-25 microns.

To create round samples, first a workpiece in the form of a polyhedron is created on the “ALTEC-13009” machine (Fig. 8), which is manually brought to a cylinder using the grinding equipment shown in Fig. 9.

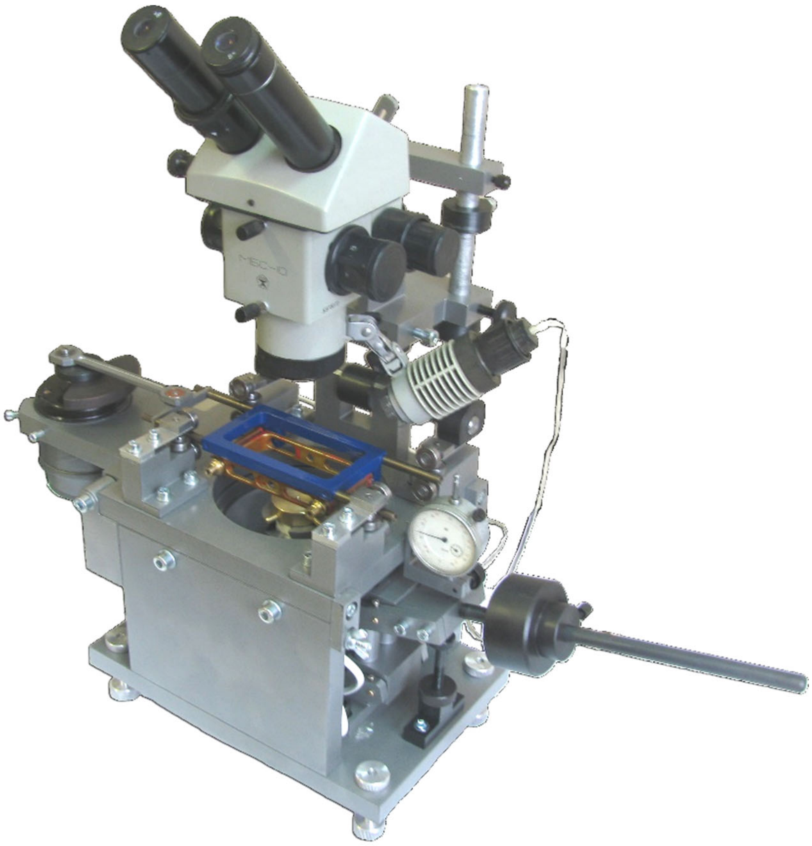

Fig. 7. Appearance of the small-sized desktop machine "ALTEC-13009".

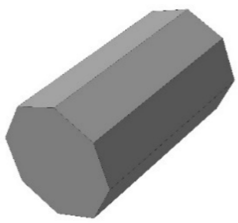

Fig. 8. A workpiece of thermoelectric material in the form of a polyhedron.

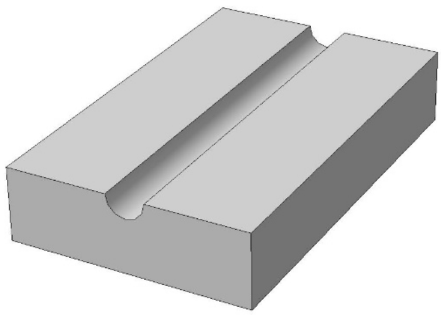

Fig. 9. Equipment for grinding the side surface of a multifaceted workpiece.

The accuracy of the geometric dimensions of the sample is controlled by an instrument microscope 10 with a resolution of 0.001 mm.

3. Creation of contact structures on the end surfaces of the studied samples

Before applying metal coatings, the surfaces of the ends of the sample should be freed from damaged deformed layers formed when cutting the material. Depending on the cutting methods, the depth of the damaged layers varies. An example of the effect of the cutting method on the depth of the damaged layer is given in Table 1.

Damaged layers significantly affect the value of contact electrical and thermal resistances and, therefore, the reproducibility and error of measurements. Therefore, before applying metal coatings, the damaged layers should be removed by mechanical processing followed by chemical or electrochemical etching.

Table 1

Effect of cutting method on the depth of deformed layers of thermoelectric material based on Bi_2Te_3.

№	Cutting method	Depth of deformed layers *h*, µm	
		n-type	*p*-type
1.	Electroerosion	2 – 5	5 – 10
2.	Wire with free abrasive	15 – 20	20 – 30
3.	Diamond disc	40 – 50	50 – 80

The appearance of the installation for creating contact structures on the measured samples is shown in Fig. 10.

The installation comprises: three vessels with electrolytes – for applying nickel and copper coatings, as well as preliminary etching of the surface of the sample; direct current source; measuring devices for controlling the amount of current during etching and applying coatings.

Fig. 10. Appearance of the installation for creating contact structures on measured samples of thermoelectric materials.

The process of creating a contact structure includes the following stages:

– mechanical treatment of the surface of the sample ends by grinding with powder with a fraction of up to 20 µm;

– placing the sample in equipment that protects the side surface of the sample and creates an electrical outlet from the side surface of the sample to the current source;

– electrochemical etching of sample ends;

– ultrasonic cleaning of the surface of sample ends;

– application of a nickel layer with a thickness of about 10 µm;

– application of a copper layer with a thickness of at least 100 μm;
– application of a nickel layer with a thickness of about 10 μm.

The composition of electrolytes is selected individually for each thermoelectric material. For example, for thermoelectric materials based on Bi_2Te_3, an aqueous solution of potassium hydroxide (KOH - 150 g/l) and sodium citrate ($NaC_3H_4(CO_2H)_3$ - 100 g/l) can be used for electrochemical etching; solution temperature 18-25 °C, current density Dk = 20 A/dm^2, anodes made of stainless steel. To apply nickel layers, an electrolyte composition is used: nickel (II) heptahydrate sulfate ($NiSO_4{\cdot}7H_2O$) – 150 g/l; potassium chloride (KCl) – 14 g/l; sodium sulfate dihydrate ($Na_2SO_4{\cdot}10H_2O$) – 70 g/l; magnesium sulfate heptahydrate ($MgSO_4{\cdot}10H_2O$) – 15 g/l; boric acid (H_3BO_3) – 25 g/l. Cathodic current density = D_k = 0.5-1.5 A/dm^2, electrolyte temperature = 18-25 °C; pH 4.5-5.5, electrolyte deposition rate ~ 10 μm/h. Nickel plates are used as anodes, the ratio of the anode area to the cathode area is ~ 2:1. Nickel plating is carried out with continuous filtration and intensive mixing of the electrolyte. The copper coating is applied with an electrolyte of the following composition: copper sulfate (II) pentahydrate ($CuSO_4{\cdot}5H_2O$) – 200 g/l; sulfuric acid (H_2SO_4 – 50 g/l. Electrolyte temperature = 18-25 °C, cathodic current density $D_к$ = 1-2 A /dm^2, electrolyte deposition rate ~ 10-15 μm/h, pure electrolytic copper anodes.

The estimated total time for applying the Ni (~10 μm) – Cu (~100 μm) – Ni (~10 μm) contact structure is about 8-10 hours.

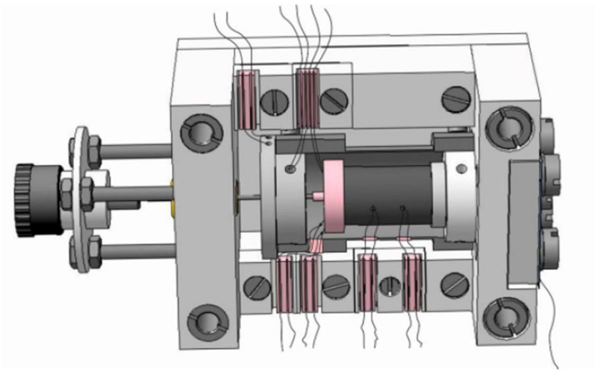

Fig. 11. Placement of the studied sample of thermoelectric material in the measuring cell of the "ALTEC-10001" installation.

The thermoelectric material sample prepared in this way is placed in the measuring cell of the "ALTEC-10001" installation (Fig. 11).

The specified sample preparation equipment provides the measurement conditions necessary to realize the capabilities of the complex absolute method, and is an integral part of the overall measurement strategy.

Conclusions

1. The requirements for the preparation of the studied samples of thermoelectric materials for measuring their properties by the complex absolute method are given.
2. Optimal for ensuring minimal measurement errors and time for establishing stationary conditions are cylindrical samples with a diameter of 6-8 mm, a length of at least 2 times the diameter of the sample and a distance between probes equal to ~ ⅓ of the length of the sample. A description of a small-sized desktop machine for cutting thermoelectric material is presented, allowing the production of samples of the required geometry.
3. To achieve a one-dimensional distribution of electric current and heat flow in the sample, it is necessary to metallize the ends of the sample. The optimal contact structure created on the ends of the sample consists of anti-diffusion nickel layers with a thickness of ~ 10 μm and a copper layer with a thickness of ~ 100 μm. A description of the installation for galvanic application of the necessary contact structures and the method of its use are given.

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МЕТОДИ ТА ОБЛАДНАННЯ ДЛЯ ПІДГОТОВКИ ЗРАЗКІВ ТЕРМОЕЛЕКТРИЧНОГО МАТЕРІАЛУ ДО ВИМІРЮВАНЬ ЇХ ВЛАСТИВОСТЕЙ АБСОЛЮТНИМ МЕТОДОМ

Показана важливість якісної підготовки досліджуваних зразків термоелектричних матеріалів до вимірювань їх властивостей комплексним абсолютним методом. Наведено вимоги до досліджуваних зразків, а також опис методів та обладнання для виготовлення зразків, які задовольнятимуть цим вимогам. Бібл. 27, рис. 11.

Ключові слова: вимірювання, електропровідність, коефіцієнт термоЕРС, теплопровідність, добротність, абсолютний метод.

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