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MEASURING THE THERMAL RESISTANCE OF A “METAL – THERMOELECTRIC MATERIAL” CONTACT STRUCTURE USING A COMPREHENSIVE ABSOLUTE METHOD FOR DETERMINING PARAMETERS OF THERMOELECTRIC MATERIALS

The paper discusses the possibility of measuring the thermal resistance of a “metal – thermoelectric material” contact structure using a comprehensive absolute method for determining the thermoelectric properties of materials. It describes the measurement technique and provides the results of studies of possible measurement errors obtained by constructing a physical model as close as possible to real conditions and computer simulation. The influence of radiation, heat loss through conductors, and other factors on the accuracy of measurements is determined. The conditions for minimizing measurement errors are established. Bibl. 32, Figs. 10, Table 1.

Key words: thermal contact resistance, measurement, computer simulation, accuracy, thermoelectric power converters.

Introduction

One of the tasks of modern thermoelectricity is the miniaturization of thermoelectric energy converters, which will significantly reduce the cost and expand the possibilities of their practical use. The main obstacle to this is the relatively large values of contact resistance, since, as is known, the influence of contact resistance on the efficiency of a thermoelectric energy converter increases as it becomes miniaturized [1 – 14].

Therefore, the development of methods and equipment for studying contact structures in thermoelectric energy converters, the creation of a technology for their manufacture and its optimization is an important and urgent task.

In doing so, as literature analysis shows [15, 16], the accuracy and reliability of methods for measuring thermal contact resistance, as well as methods for measuring electrical contact resistance, require significant improvement to implement in practice the possibilities for reducing contact resistances provided by theoretical studies.

There are a number of methods for measuring thermal contact resistance. These methods are based on measuring the steady-state heat flux passing through the sample in a certain direction. The basics of the method are set out in the international standard ASTM D5470-06 [17]. Thus, in [18], a standard

method is described, which is based on the use of a reference sample with a previously known thermal conductivity as a heat flux meter.

To determine the thermal contact resistance, a slightly modified comprehensive absolute method for determining the thermoelectric properties of materials and the corresponding measuring equipment developed at the Institute of Thermoelectricity (Ukraine) [19 – 28] can be used. To use this method, reference samples are not required, since the heat flux through the structure under study is determined by the electrical power of the heat source, and all possible heat losses are minimized.

The purpose of this work was to analyze the possibilities of measuring the thermal resistance of the "metal – thermoelectric material" contact structure using a comprehensive absolute method and to study the influence of deviations from the ideal physical model of such a method on the accuracy of measurements.

1. Physical model and main sources of measurement errors

The simplest model of the comprehensive absolute method, taken as a basis for developing the methodology for determining the thermal resistance of the “metal – thermoelectric material” contact structure, is shown in Fig. 1. It contains a sample of thermoelectric material 1 with metal anti-diffusion coatings 2 applied to its end surfaces, metal plates 3, ceramic contact plates 4, transient contact layers 5, 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 temperature gradient in the sample. The contact structure shown in the figure is similar to that commonly used in thermoelectric energy converters (metal anti-diffusion coatings, metal connecting plates, and a ceramic base).

The given model does not take into account heat exchange with the environment, as well as heat transfer by thermocouples and heater conductors.

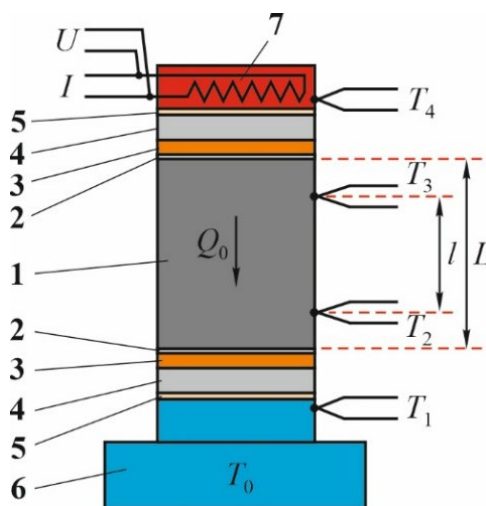


Fig. 1. The simplest physical model of the process of measuring thermal contact resistance by the comprehensive absolute method: 1 – sample of thermoelectric material; 2 – metal anti-diffusion coatings; 3 – metal contact plates; 4 – ceramic contact plates; 5 – transient contact layers; 6 – thermostat; 7 – electric heater.

For such a model, the thermal conductivity κ of a 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 flux through the sample, taken equal to the electric power of the heater; T_2 and 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 heater temperature T_4 , the thermostat temperature T_1 , and the total length of the sample L are additionally measured. Assuming that the thermal resistance of the contact structures on both ends of the sample is the same, its value can be determined by the formula

$$R_T = \Delta T_{cont.} \frac{S}{W} = \frac{1}{2} [(T_4 - T_1) - \Delta T_L] \frac{S}{W}, \quad (2)$$

where $\Delta T_{cont.}$ is the temperature drop across the studied contact structure; ΔT_L is the total temperature difference across the thermoelectric material sample (Fig. 2).

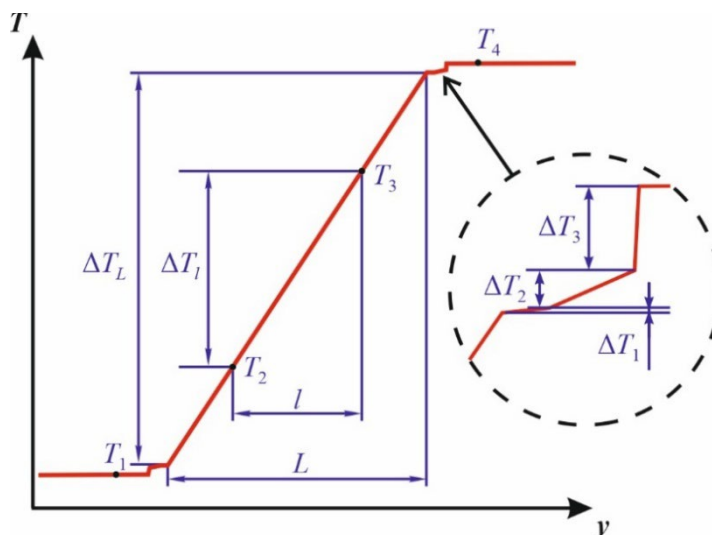


Fig. 2. Temperature distribution along the axis of the studied contact structure ΔT_1 – temperature difference across the sample of thermoelectric material at points located at a distance l from each other; ΔT_L – total temperature difference across the sample; L – sample length; ΔT_1 – temperature difference across the metal plate; ΔT_2 – temperature difference across the ceramic plate; ΔT_3 – temperature difference across the transient contact layer between the ceramic plate and the heater.

Since

$$\Delta T_L = \frac{W}{\kappa} \frac{L}{S}, \quad (3)$$

and all the quantities necessary for calculating the thermal conductivity of the sample κ are determined simultaneously with the quantities for calculating the thermal resistance of the contact structure, formula (2) can be rewritten as

$$R_T = \frac{1}{2} \left[(T_4 - T_1) - \frac{L}{l} (T_3 - T_2) \right] \frac{S}{W}. \quad (4)$$

Although the model shown in Fig. 1 is far from real measurement conditions, its advantage is the ability to determine the desired values from simple mathematical expressions based on Fourier's law. In this case, finding the values of the desired values is achieved either by taking into account all errors, or by taking special measures to minimize their influence on the measurement results.

To find possible errors in determining thermal resistance, it is important to know the instrumental errors of all measuring instruments and methodological errors, which must be minimized to acceptable values.

Instrumental errors occur when measuring the values of the cross-section of the sample S , its length L , the distance between the thermocouples l , the current I and voltage U of the heater, the EMF of the thermocouples $T_1 - T_4$. In this case, the total value of instrumental errors when using modern measuring equipment is usually less than 0.2% (Table 1).

Table 1

Instrumental errors in measuring thermal contact resistance by the absolute method

Measured values	Resolution of measuring instruments	Typical measurement errors
S, l, L	10^{-3} mm	± 0.01 %
I	10^{-6} A	± 0.001 %
U	10^{-7} V	± 0.001 %
T_1, T_2, T_3, T_4	10^{-7} V	± 0.1 %
		$\Sigma < 0.2$ %

Methodological errors are a consequence of deviations from the simplest physical model, primarily from the conditions of adiabaticity and one-dimensionality of heat flux through the studied contact structure.

The advantage of the absolute method is the possibility of their minimization. To find ways of minimization, a more realistic physical model should be considered, which takes into account the causes of the most significant errors (Fig. 3).

In particular, to reduce heat transfer by radiation, the sample and the reference heater are surrounded by a gradient radiation screen with a heater, the power of which is selected such that the temperature distributions along the sample and the screen are the same. In addition, all conductors of thermocouples and the reference heater are led outside through so-called thermal switches, which are assemblies made of electrical insulators with the highest possible thermal conductivity, for example, beryllium oxide. They are made in the form of tubes, rings or plates. Through the holes in them, conductors are passed, which are brought into thermal contact with the electrical insulator. The latter, in turn, is in thermal contact with the gradient radiation screen. In this case, the temperature difference along the conductors approaches zero, the heat flux through them is minimized and, therefore, the magnitude of the errors is minimized. A similar thermal switch is also used in the clamping mechanism for fixing the sample.

This approach allows us to retain the simplest expression (4) when finding the desired value of thermal resistance RT .

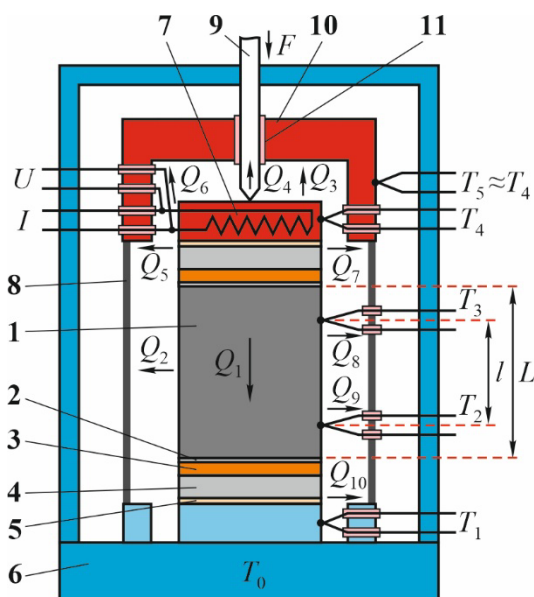


Fig. 3. A real physical model of the process for measuring thermal contact resistance by the comprehensive absolute method: 1 – thermoelectric material sample; 2 – metal anti-diffusion coatings; 3 – metal contact plates; 4 – ceramic contact plates; 5 – transient contact layers; 6 – thermostat; 7 – electrical heater; 8 – screen; 9 – clamping mechanism; 10 – screen heater; 11 – thermal switches.

In Fig. 3: Q_1 – heat flowing from the sample to the thermostat; Q_2 – heat losses due to radiation from the sample surface; Q_3 – heat losses due to radiation from the heater surface; Q_4 – heat losses through clamping mechanism; Q_5 – heat losses due to radiation from the surface of contact structures; Q_6 – heat losses through current and potential conductors of the heater; $Q_7 - Q_{10}$ – heat losses through conductors of thermocouples; T_0 – thermostat temperature.

The thermal conductivity equation for finding temperature distributions in a sample and other elements of a physical model is given below

$$\nabla(-\kappa_i \cdot \nabla T) = Q_i, \quad (5)$$

where κ_i are thermal conductivities of elements of the physical model.

The boundary conditions that determine the heat transfer by radiation between the structural elements of the measuring cell can be written as

– lateral surface

$$q = \varepsilon_1(G_1 - \sigma T^4), \quad (6)$$

– lateral surface of the reference heater

$$q = \varepsilon_2(G_2 - \sigma T^4), \quad (7)$$

– upper surface of the reference heater

$$q = \varepsilon_2(G_3 - \sigma T^4), \quad (8)$$

– the internal surface of the gradient radiation screen

$$q = \varepsilon_3(G_4 - \sigma T^4), \quad (9)$$

– internal surfaces of the gradient radiation screen heater

$$q = \varepsilon_4(G_5 - \sigma T^4), \quad (10)$$

– external surfaces of the gradient radiation screen heater

$$q = \varepsilon_4(G_6 - \sigma T^4), \quad (11)$$

– external surface of the gradient radiation screen

$$q = \varepsilon_3(G_7 - \sigma T^4), \quad (12)$$

– thermostat surfaces in the gap between the sample and the gradient radiation screen.

$$q = \varepsilon_5(G_8 - \sigma T^4), \quad (13)$$

– internal surfaces of the thermostat

$$q = \varepsilon_5(G_9 - \sigma T^4). \quad (14)$$

The outer surfaces of the thermostat are maintained at a temperature of T_0

$$T = T_0. \quad (15)$$

In formulae (6) – (14): $\varepsilon_1, \varepsilon_2, \varepsilon_3, \varepsilon_4, \varepsilon_5$ – are the radiation coefficients of the sample, reference heater, screen, screen heater, and thermostat, respectively; σ is the Stefan-Boltzmann constant; G is the heat flux generated by the irradiation of each surface.

$$G = G_m + F_{amb}\sigma T_{amb}^4. \quad (16)$$

where G_m is the magnitude of radiation from other elements of the measuring cell and the sample; F_{amb} is the field of view factor, equal to that part of it that is not affected by other surfaces; T_{amb} is the temperature at distant points in the directions included in F_{amb} .

2. Computer model

The solution of the problem in the form (5) with boundary conditions (6) – (16) was performed using the COMSOL Multiphysics application package [29], which uses the finite element method for calculations [30, 31].

The coefficient G_m , depending on the relative position of the surfaces, is calculated by introducing into the computer model an additional variable J , which is given by the equation

$$J = (1 - \varepsilon) \{ G_m(J) + F_{amb}\sigma T_{amb}^4 \} + \varepsilon\sigma T^4. \quad (17)$$

which is solved jointly with thermal conductivity equation.

A typical view of the finite element method mesh when modeling the absolute method is given and the temperature distribution in the sample and the structural elements of the measuring cell is shown

in Fig. 4.

The solutions obtained by computer simulation do not have the same versatility as analytical ones, but still make it possible to solve specific optimization problems for measuring devices designed to study samples of a given geometric shape and size in a given temperature range.

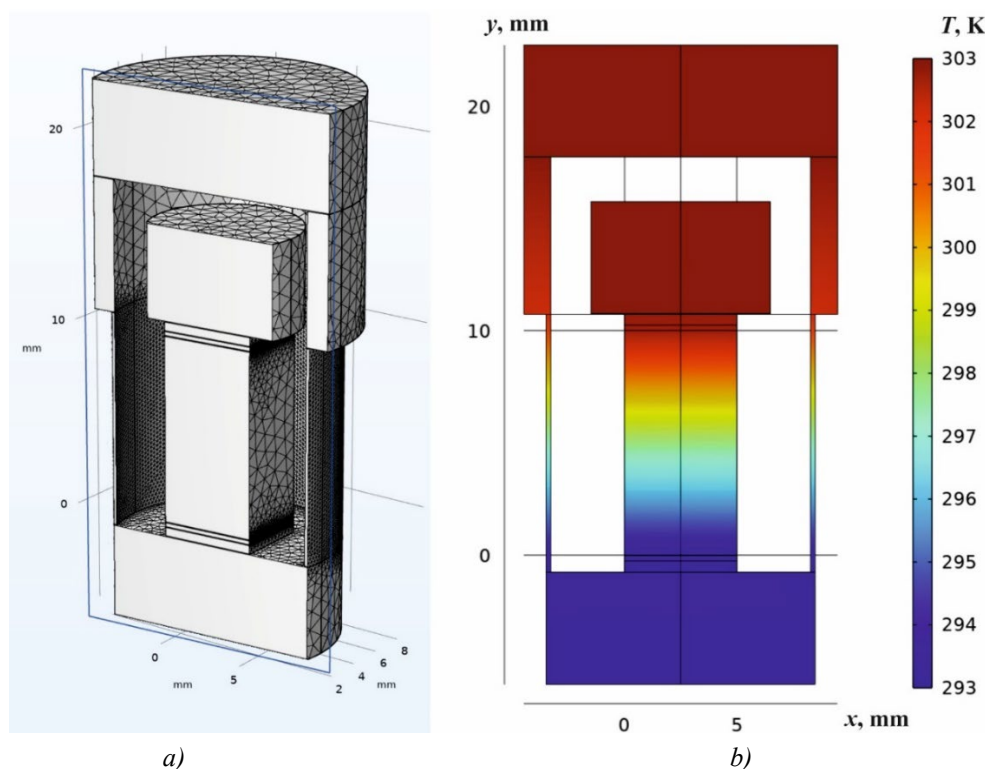


Fig. 4. Computer model of the process of measuring thermal contact resistance by the comprehensive absolute method: a – image of the finite element method mesh in COMSOL Multiphysics; b – typical temperature distribution in the studied contact structure and structural elements of the measuring cell.

3. Results of computer studies of the main sources of measurement errors

3.1. Errors in determining the heat flux through the studied structure

Using computer simulation, the dependence of measurement errors on the emissivity coefficients of the sample, gradient radiation screen, sample and screen heaters, thermostat; thermostat temperature and other parameters was investigated. The results of the studies are given below. Calculations were performed for typical sample sizes used for measurements of σ , α , κ and Z by the comprehensive absolute method – length 10 mm, cross-sectional area $5 \times 5 \text{ mm}^2$. The diameter of the reference heater is 8 mm, the inner diameter of the screen is 12 mm; thermal conductivity of the sample is $2 \text{ W/(m}\cdot\text{K)}$, of the reference heater, screen heater and thermostat – $400 \text{ W/(m}\cdot\text{K)}$, screen – $15 \text{ W/(m}\cdot\text{K)}$; thermostat temperature is from -50 to 300°C . If necessary, computer simulation makes it possible to reproduce these results for other temperature ranges and sample sizes.

The main obtained dependences of the errors in determining the heat flux through the studied contact structure on various factors are given in Figs. 5-7. Thus, Fig. 5 shows the dependence of δW on the emissivity of the sample and structural elements of the measuring cell at a thermostat temperature of 100°C . As can be seen from the figure, to achieve minimal errors, the emissivity coefficients of the surface of the gradient radiation screen and the screen heater should be large, and those of the sample, reference heater and thermostat should be small. Increasing the absorption coefficient of the screen is

achieved by blackening or by using rings for additional over-radiation and approaching the absorption coefficient of absolutely black bodies. The lateral surfaces of the sample and the reference heater, as well as the surface of the thermostat in the gap between the sample and the screen, should be polished or a thin layer of shiny electrical insulating material with a low emissivity should be applied to them.

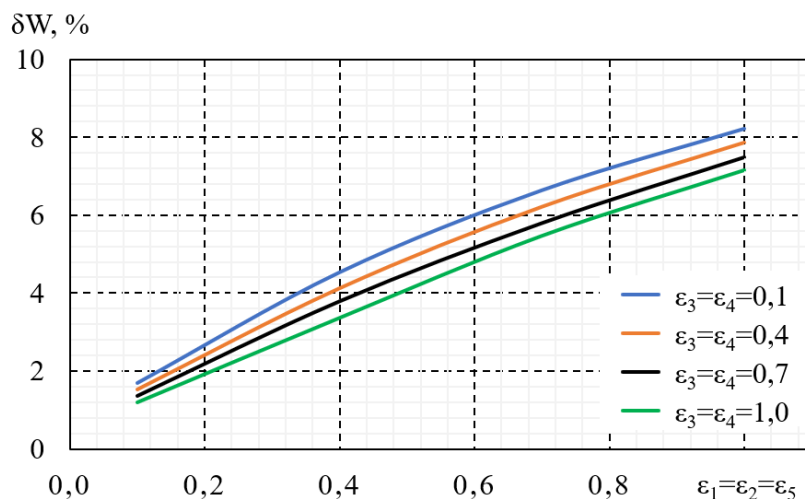


Fig. 5. Dependence of the error in determining the heat flux through the studied contact structure on the emissivity of the sample and the structural elements of the measuring cell ($\epsilon_1, \epsilon_2, \epsilon_3, \epsilon_4, \epsilon_5$ – emissivity coefficients of the sample, reference heater, screen, screen heater and thermostat, respectively).

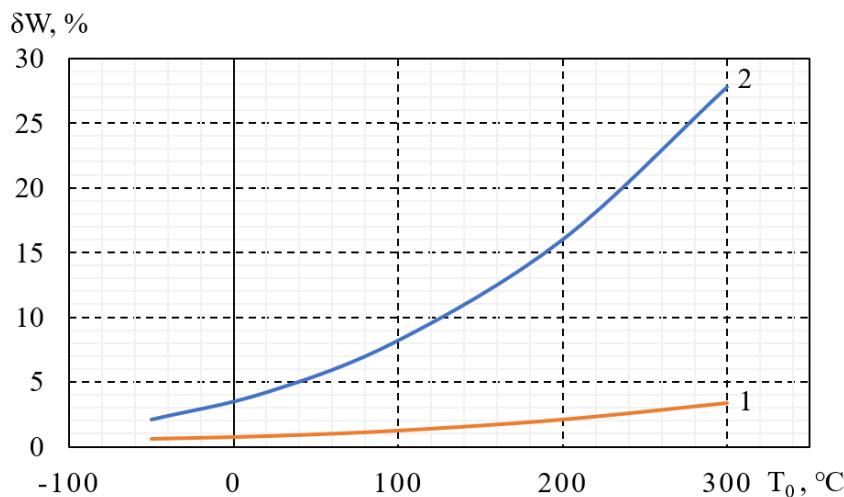


Fig. 6. The dependence of the error in determining the heat flux through the studied contact structure on temperature: 1 – $\epsilon_1 = \epsilon_2 = \epsilon_5 = 0.1, \epsilon_3 = \epsilon_4 = 1.0$; 2 – $\epsilon_1 = \epsilon_2 = \epsilon_5 = 1.0, \epsilon_3 = \epsilon_4 = 0.1$.

The dependence of the error in determining the heat flux through the studied contact structure on the thermostat temperature is shown in Fig. 6. Two cases are shown:

- 1) $\epsilon_1 = \epsilon_2 = \epsilon_5 = 0.1, \epsilon_3 = \epsilon_4 = 1.0$ – most favorable when the emissivity coefficients of the sample surface, reference heater, and thermostat are small, and those of the gradient radiation screen and screen heater are large;
- 2) $\epsilon_1 = \epsilon_2 = \epsilon_5 = 1.0, \epsilon_3 = \epsilon_4 = 0.1$ – most unfavorable when the emissivity coefficients of the

sample surface, reference heater, and thermostat are large, and those of the gradient radiation screen and screen heater are small.

From Fig. 6 it is seen that at a temperature of $T_0 = -50\text{ }^{\circ}\text{C}$ the errors in determining the heat flux through the studied structure do not exceed 3.5 % and increase with a rise in temperature to 28 % at $T_0 = 300\text{ }^{\circ}\text{C}$. However, by choosing the optimal values of the emissivity of the physical model elements, they can be reduced to the level of 0.7 – 3.4 %.

Fig. 7 shows the dependence of the error in determining the heat flux through the studied contact structure, caused by heat losses due to radiation, on the temperature difference across the sample (at $T_0 = 20\text{ }^{\circ}\text{C}$; $\varepsilon_1 = \varepsilon_2 = \varepsilon_5 = 1.0$, $\varepsilon_3 = \varepsilon_4 = 0.1$). As expected, the errors increase with increasing temperature difference.

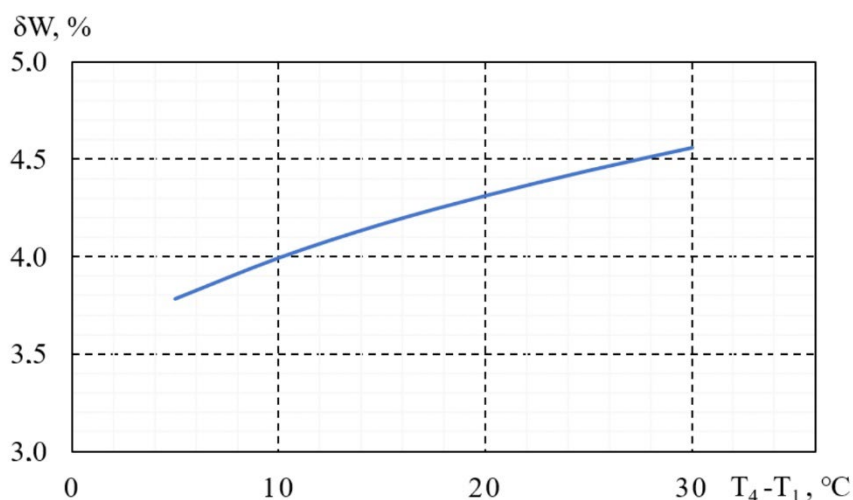


Fig. 7. Dependence of the error in determining the heat flux through the studied contact structure on the temperature difference between the heater and the thermostat ($T_0 = 20\text{ }^{\circ}\text{C}$; $\varepsilon_1 = \varepsilon_2 = \varepsilon_5 = 1.0$, $\varepsilon_3 = \varepsilon_4 = 0.1$).

Computer simulation also established that heat losses through thermocouple conductors, current and potential conductors of the heater, as well as through the clamping mechanism in total can reach ~ 3 % (at a temperature of 300 °C), however, the use of thermal switches allows these losses to be reduced to ~ 0.5 %.

3.2. Errors in determining the temperature difference on the contacts

Fig. 8 shows the dependence of the temperature drop across the thermal resistance of the studied contact structure on the temperature difference between the heater and the thermostat. The dependence was obtained for a typical contact structure for thermoelectric energy converters, consisting of a nickel anti-diffusion coating with a thickness of 10 μm , a copper connecting plate with a thickness of 0.25 mm, and a ceramic plate with a thickness of 0.5 mm. The thermal resistance of the transient contact layer between the ceramic plate and the heater is $8 \cdot 10^{-5}\text{ K}\cdot\text{m}^2/\text{W}$, the thermostat temperature is 20°C.

To increase the accuracy of measurements, it is desirable that the temperature drop across the thermal resistance be as large as possible. However, this leads to increased heat loss due to radiation, as shown above. In addition, in this case, the influence of the temperature dependence of the thermal conductivity of the thermoelectric material sample, which is quite significant in certain temperature ranges, will also increase (Fig. 9).

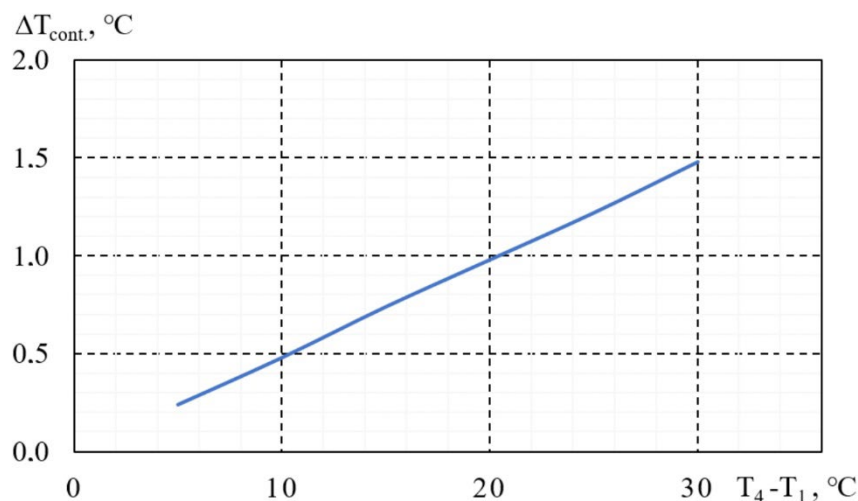


Fig. 8. Dependence of the temperature drop across the thermal resistance of the studied contact structure on the temperature difference between the heater and the thermostat ($T_0 = 20\text{ }^\circ\text{C}$).

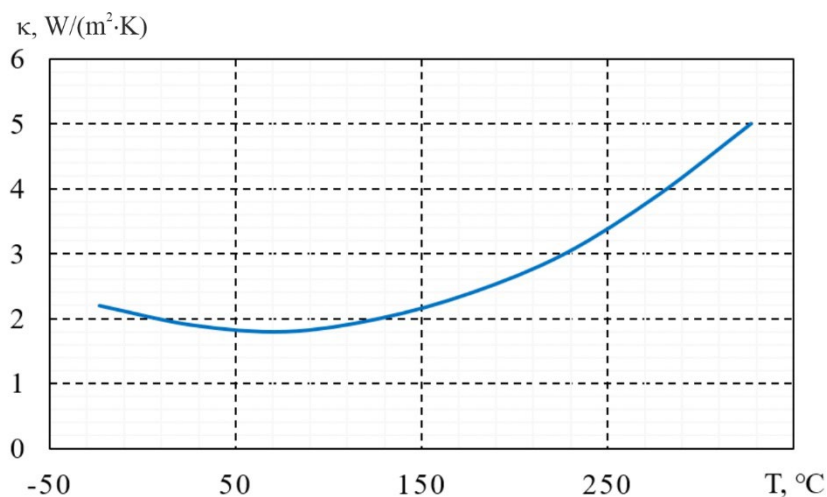


Fig. 9. Temperature dependence of thermal conductivity of a sample of thermoelectric material $(\text{Bi}_2\text{Te}_3)_{0.25}(\text{Sb}_2\text{Te}_3)_{0.72}(\text{Sb}_2\text{Se}_3)_{0.03}$, doped with lead, for generator thermoelectric energy converters (electrical conductivity at a room temperature – $2 \cdot 10^5 \text{ Ohm}^{-1} \cdot \text{m}^{-1}$) [32].

Taking into account the temperature dependence of the thermal conductivity of a thermoelectric material sample in the computer model made it possible to assess its impact on the accuracy of determining the temperature drop across the thermal resistance of the studied contact structure with an increase in the temperature difference between the heater and the thermostat (Fig. 10).

As can be seen from Fig. 10, the error in determining the temperature drop across the thermal resistance grows rapidly with a rise in temperature difference between the heater and the thermostat. The optimal temperature difference from the point of view of sufficient resolution and minimal measurement errors is 10 – 15 $^\circ\text{C}$.

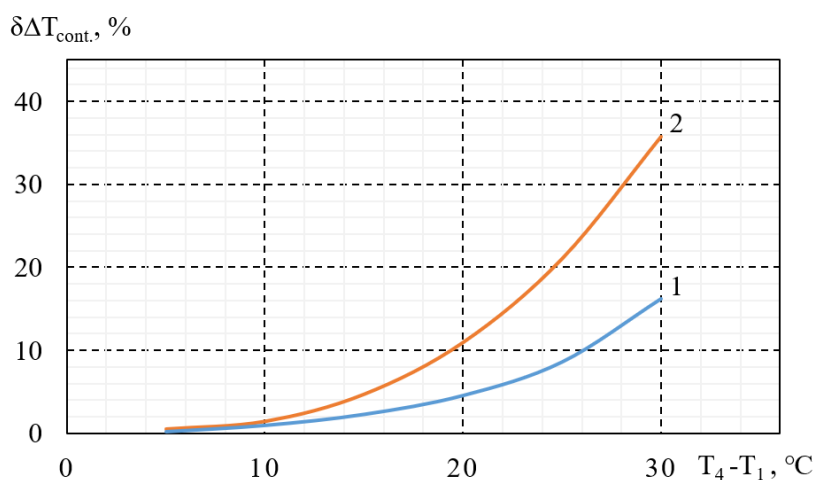


Fig. 10. Dependence of the error in determining the temperature drop across the thermal resistance of the studied contact structure on the temperature difference between the heater and the thermostat: 1 – $T_0 = 20$ °C; 2 – $T_0 = 300$ °C.

In general, the obtained results of computer simulation confirm the possibility of measuring the thermal resistance of the "metal – thermoelectric material" contact structure using the comprehensive absolute method and are the basis for modifying the design of the measuring equipment "ALTEC-10001", developed at the Institute of Thermoelectricity, to implement the above measurement method.

Conclusions

1. The possibility of measuring the thermal resistance of the “metal-thermoelectric material” contact structure using the comprehensive absolute method for determining the thermoelectric properties of materials has been confirmed.
2. A detailed physical model of the process of measuring thermal contact resistance by the comprehensive absolute method was constructed and a computer model was developed on its basis to study the influence of various factors on the accuracy of measurements.
3. Using computer simulation, the dependence of measurement errors on the emissivity coefficients of the sample, gradient radiation screen, sample and screen heaters, thermostat; thermostat temperature and other parameters was investigated. It was found that to achieve minimal errors, the emissivity coefficients of the gradient radiation screen and screen heater surface should be large, and those of the sample, reference heater and thermostat should be small.
4. It was determined that at a temperature of $T_0 = -50$ °C, the errors in determining the heat flux through the structure under study do not exceed 3.5 % and increase with a rise in temperature to 28 % at $T_0 = 300$ °C. At the same time, by choosing the optimal values of the emissivity of the elements of the physical model, they can be reduced to the level of 0.7 – 3.4 %.
5. It has been established that heat losses through thermocouple conductors, current and potential conductors of the heater, as well as through the clamping mechanism in total can reach ~ 3 % (at a temperature of 300 °C), however, the use of thermal switches allows these losses to be reduced to ~ 0.5 %.
6. The influence of the temperature difference across the sample on the accuracy of determining the temperature drop on the thermal resistance of the contact structure under study was analyzed. It was found that the optimal temperature difference between the heater and thermostat in terms of sufficient resolution and minimal measurement errors is 10 – 15 °C.

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**ВИМІРЮВАННЯ ТЕПЛОВОГО ОПОРУ КОНТАКТНОЇ СТРУКТУРИ
«МЕТАЛ – ТЕРМОЕЛЕКТРИЧНИЙ МАТЕРІАЛ» ЗА ДОПОМОГОЮ
КОМПЛЕКСНОГО АБСОЛЮТНОГО МЕТОДУ ВИЗНАЧЕННЯ
ПАРАМЕТРІВ ТЕРМОЕЛЕКТРИЧНИХ МАТЕРІАЛІВ**

У роботі розглянуто можливість вимірювання теплового опору контактної структури «метал – термоелектричний матеріал» за допомогою комплексного абсолютного методу визначення термоелектричних властивостей матеріалів. Наведено опис методики проведення вимірювань та результати досліджень можливих величин похибок вимірювань, отримані шляхом побудови максимально наближеної до реальних умов фізичної моделі та комп'ютерного моделювання. визначено вплив випромінювання, втрат тепла по провідниках та інших факторів на точність вимірювань. Встановлено умови мінімізації похибок вимірювань. Бібл. 32, рис. 10, табл. 1.

Ключові слова: тепловий контактний опір, вимірювання, комп'ютерне моделювання, точність, термоелектричні перетворювачі енергії.

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