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# METHODS FOR ASSURING HIGH QUALITY ELECTRIC AND THERMAL CONTACTS WHEN MEASURING PARAMETERS OF THERMOELECTRIC MATERIALS

*A computer model is created that allows studying the errors in the measurement of electric conductivity and thermal conductivity when using pressure contacts for current and heat supply to the measured sample. The measurement errors for the case of point contacts are determined. It is established that the errors in the measurement of thermal conductivity and electric conductivity can reach 45%.*

*The results of research on the methods of reducing these errors by creation of metal contact coatings on the sample end surfaces are presented. It is established that if nickel and copper layers are available on the sample end surfaces, even with the worst arrangement of one point contact, the error in the measurement of electric conductivity lies within 1.5-1.8%.*

*The errors in the measurement of thermal conductivity are determined. It is established that for Ni-Cu-Ni contact structure with a tungsten anti-diffusion plate these errors with the worst arrangement of point contacts will be reduced to 7.4% in case of one contact point on both sample end surfaces and to 1% – in case of three contact points.*

**Key words**: measurement error, electrical conductivity, thermal conductivity.

## Introduction

*General characterization of the problem*. Absolute method is one of the most reliable methods of measuring the temperature dependences of thermoelectric material properties [1, 2]. The main sources of errors when using this method and the possibilities of their minimization are detailed in [3-6]. The achieved values of errors are as follows: thermal conductivity – up to 2.4%, thermoEMF – up to 0.8%, electric conductivity – up to 0.7%, thermoelectric figure of merit – up to 4.7% [7].

In so doing, one of important measurement problems is to assure a reliable electric and thermal contact between the sample under study and the structural components of measuring installation. This is due to the fact that bad contacts can result in considerable deviations from the uniformity of temperature and electric potential distributions in the sample, and, respectively, in measurement errors. This is particularly important at high-temperatures measurements, when the use of solders becomes impossible, and pressure contacts can be even of point type.

Therefore, the *purpose of this work* is to determine the errors in the measurement of electric conductivity and thermal conductivity that arise when using pressure contacts due to distortions of temperature and electric potential uniformity distributions, and to develop methods for minimization of these errors.

## 1. Physical, mathematical and computer models

In the case of using pressure contacts, the electric potential and temperature in the near-contact areas are distorted. For the reduction of this effect, measuring probes and thermocouples are arranged possibly far from the ends on the sample lateral surface. This allows eliminating the errors in the determination of sample temperature when measuring sample thermoEMF. However, in the determination of electric conductivity and thermal conductivity, deviations from the uniformity of heat and current distribution in the sample will cause errors in that case as well.

### 1.1. Measurement of electric conductivity

A sample of thermoelectric material shaped as a cylinder of length l and diameter d (Fig. 1) was considered. Openings were made and cylinder probes were installed on the lateral sample surface. It was considered that electric current to the sample is supplied through point contacts on its end surfaces, i.e. sample contacts to the heater and the thermostat, which in the absolute method are used to measure thermal conductivity and simultaneously serve as current leads when measuring electric conductivity. Arrangement of current supply points on the sample end surface can be arbitrary. Sample electric conductivity is found by the formula

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

where $U$ is voltage drop between measuring probes, S is cross-section area of the sample.

Current density nonuniformity due to non-ideality of contacts will lead to the errors in the measurement of electric conductivity calculated by formula (1).

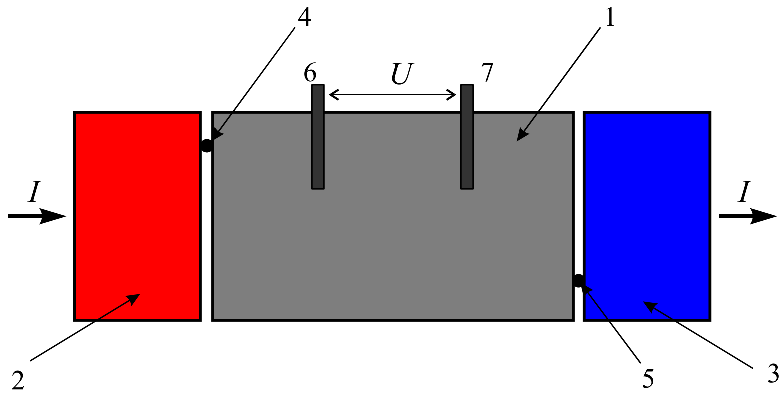


*Fig. 1. Physical model for studying the influence of contacts on the accuracy of measurement of thermoelectric material electric conductivity*
*1 – sample under study, 2 – heater, 3 – thermostat, 4, 5 – point contacts for current delivery to the sample, 6, 7 – measuring probes.*

To determine the effect of contact arrangement, it is necessary to calculate the electric potential distribution in the sample based on the equation

$$-\nabla\left(\sigma\nabla\varphi\right) = 0\,. \tag{2}$$

with the following boundary conditions:

1) for sample end surface free from contact to current leads, and the sample lateral surface

$$\mathbf{n}\left(\sigma\nabla\varphi\right) = 0\,; \tag{3}$$

2) for points of current supply to the sample

$$\mathbf{n}(\sigma\nabla\varphi) = I . \tag{4}$$

The error in the determination of electric conductivity $\delta\sigma$ is found by the formula

$$\delta\sigma = \left|1 - \frac{U/I}{U_{id}/I_{id}}\right| \cdot 100\% , \tag{5}$$

where $U$, $I$ is potential difference between the probes and current through the sample according to the model; $U_{id}$, $I_{id}$ is potential difference between the probes and current through the sample in the ideal case when current density is identical along the entire sample.

**1.2. Measurement of thermal conductivity**

When measuring thermal conductivity, the analysis of the sutuation is somewhat complicated by the presence of heat transfer due to radiation and gas thermal conductivity in the area without a direct contact between the sample and the heater and thermostat (Fig. 2).

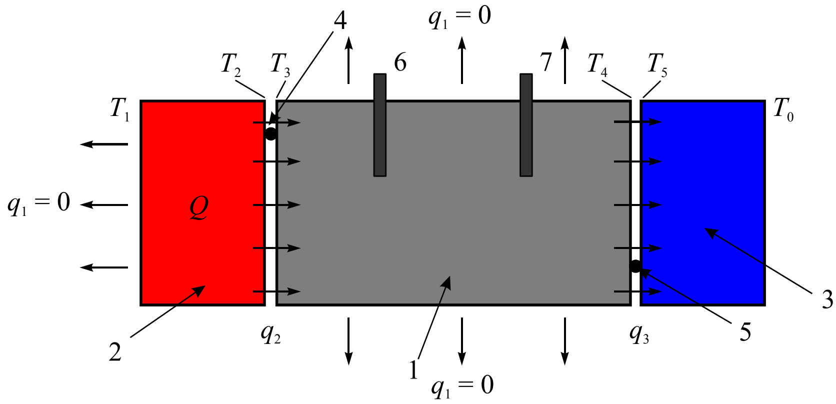


*Fig. 2. Physical model for studying the influence of contacts on the accuracy of measurement of thermoelectric material thermal conductivity. 1 – sample under study, 2 – reference heater, 3 – thermostat, 4, 5 – areas of direct contact between the sample and the heater and thermostat, 6, 7 – measuring probes.*

To find the distribution of temperature, it is necessary to solve the thermal conductivity equation

$$\nabla(-\kappa \cdot \nabla T) = Q , \tag{6}$$

where $\kappa$ is thermal conductivity, $Q$ is internal source of heat (equal to $Q_h$ for the heater volume and zero for sample volume).

The boundary conditions for such a problem:

1) thermal insulation of the lateral surface of sample and reference heater

$$q_1 = 0 ; \tag{7}$$

2) thermostat fixed temperature

$$T = T_0 ; \tag{8}$$

3) heat transfer in the gap between the heater and the sample through gas layer (xenon) and due to radiation

$$q_2 = -\varepsilon\sigma_B\left(T_2^4 - T_3^4\right) - \kappa_{Xe}\nabla T ; \tag{9}$$

4) heat transfer in the gap between the sample and the thermostat through gas layer (xenon) and due to radiation

$$q_3 = -\varepsilon\sigma_B\left(T_4^4 - T_5^4\right) - \kappa_{Xe}\nabla T ; \tag{10}$$

5) heat transfer from the heater to the sample and from the sample to the thermostat in the area of their direct contact

$$T_2 = T_3,\ \kappa_{heater}\nabla T = \kappa_{sample}\nabla T,$$
$$T_4 = T_5,\ \kappa_{sample}\nabla T = \kappa_{thermostat}\nabla T. \tag{11}$$

For the calculations, computer simulation via Comsol Multiphysics package was used.

## 2. Results of computer simulation of the errors in the measurement of electric conductivity

The following variants of places of point contact between the sample and current leads 1-1, 1-2, 1-3, 2-2, 2-3, 3-3, 4-4, 4-2, 4-5 (Fig. 3) were considered for the case of $l$ = 9 mm, $d$ = 6 mm, the distance between the probes $a$ = 5 mm.

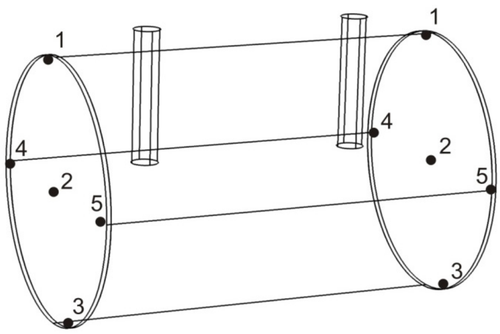


*Fig. 3. Arrangement of current supply point contacts on the sample end surfaces.*

According to computer simulation, the errors in the measurement of electric conductivity in this case can reach 45%. For current density leveling in the sample, hence, for the reduction of these errors, a thin metal layer, for instance, nickel, can be applied on the end surfaces.

Dependences of a relative error in the measurement of electric conductivity on the thickness of nickel coating $h$ for said current flow directions are shown in Figs. 4, 5.

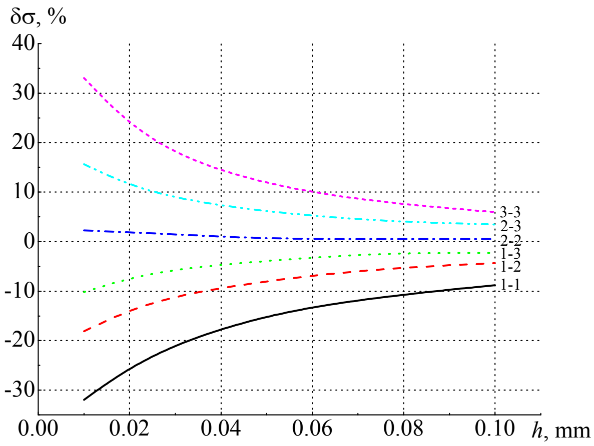


*Fig. 4. Dependences of a relative error in the measurement of electric conductivity on the thickness of nickel coating for different current flow directions (1-1, 1-2, 1-3, 2-2, 2-3, 3-3).*

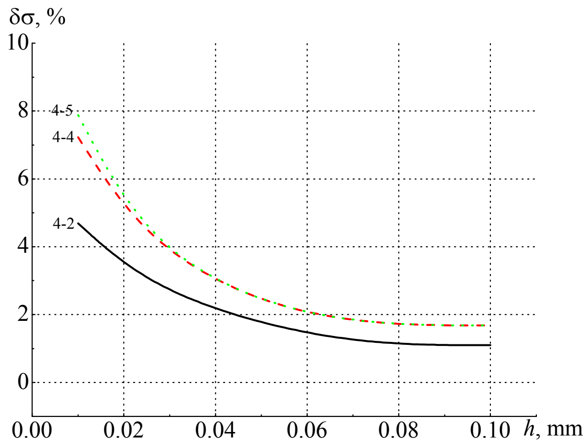


*Fig. 5. Dependences of a relative error in the measurement of electric conductivity on the thickness of nickel coating for different current flow directions (4-4, 4-2, 4-5).*

For better equalization of electric potential on the sample end surface, an additional copper layer can be applied. Dependences of the error in the measurement of electric conductivity on the thickness of copper coating for different current flow directions are shown in Fig. 6.

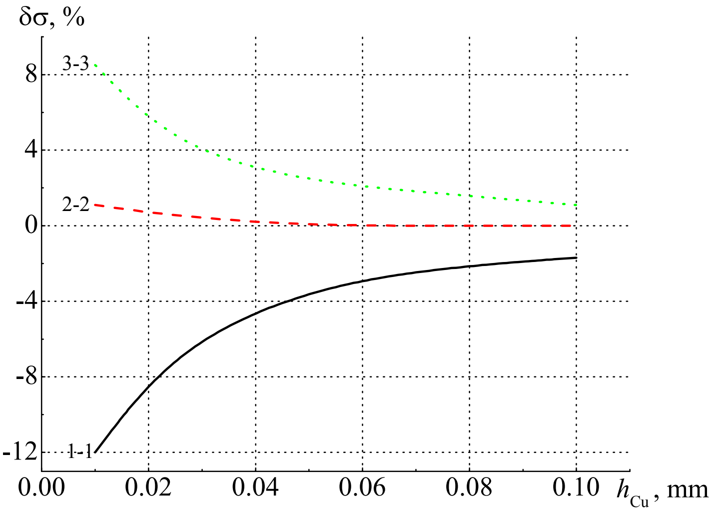


*Fig. 6. Dependences of the error in the measurement of electric conductivity on the thickness of copper coating for different current flow directions (nickel thickness – 10 μm).*

As is seen, even with the worst arrangement of one point contact, the error in the measurement of electric conductivity lies within 1.5-1.8% (in the presence on the sample end surfaces of 10 μm nickel layer and 100 μm copper layer).

## 3. Results of computer simulation of the errors in the measurement of thermal conductivity

For the considered case of nickel and copper contact coating Fig. 7 shows the dependences of the errors in the measurement of thermal conductivity on the thickness of heat-levelling copper layer. It is considered that a direct contact between the sample and the heat-exchange surfaces takes place in the area of diameter $d_{cont}$ (for the case of 0.1 μm shown in the figure) and can be arbitrarily arranged on the end surfaces.

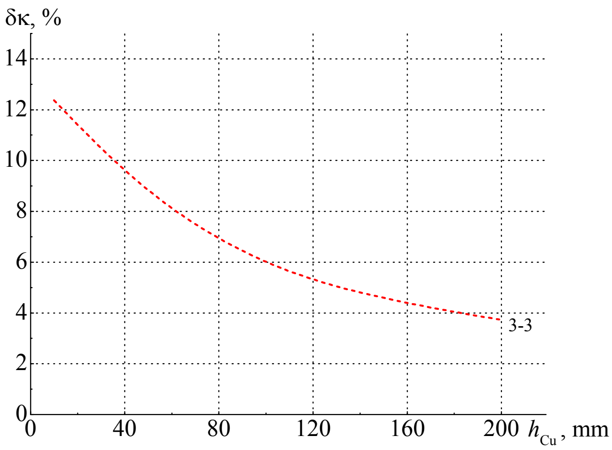


*Fig. 7. Dependences of the error in the measurement of thermal conductivity on the thickness of copper coating*

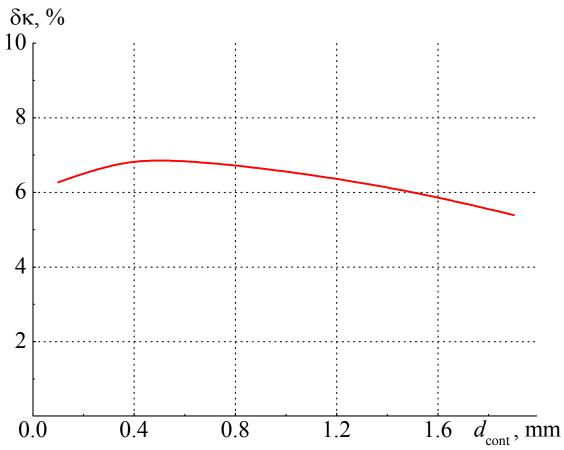


*Fig. 8. Dependences of the error in the measurement of thermal conductivity on the diameter of direct*

*(nickel thickness – 10 μm)* *contact area between the sample and the heat-exchange surfaces.*

Dependence of the error on the contact diameter is shown in Fig. 8. In the case at hand the thickness of nickel coating is 10 μm, of copper coating – 100 μm.

Because of heat transfer due to radiation in the gap between the sample and the heat-exchange surfaces, the value of measurement error will be a function of temperature. This dependence for different variants of point contact arrangement and the thickness of copper contact coating is shown in Fig. 9. As is evident, with a rise in temperature, the errors will be reduced.

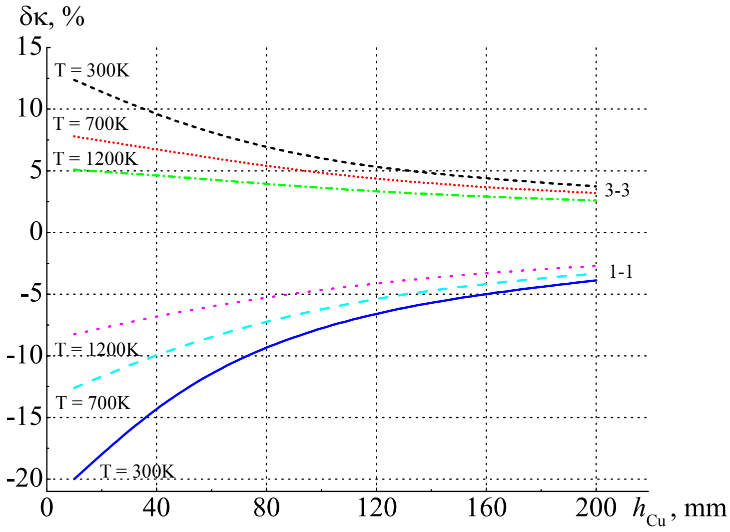


*Fig. 9. Dependences of the error in the measurement of thermal conductivity on the thickness of copper coating for different variants of point contact arrangement between the sample and the heat-exchange surfaces and different temperatures.*

Another important issue when assuring good contacts is their resistance to elevated temperatures. Experimental studies have shown that at temperatures up to 900°C there can take place a diffusion and adhesion of the heater and the thermostat surfaces to the sample metal coating. This will make it impossible to dismount the sample after the measurements. To prevent this, thin anti-diffusion plates can be placed on both sample sides.

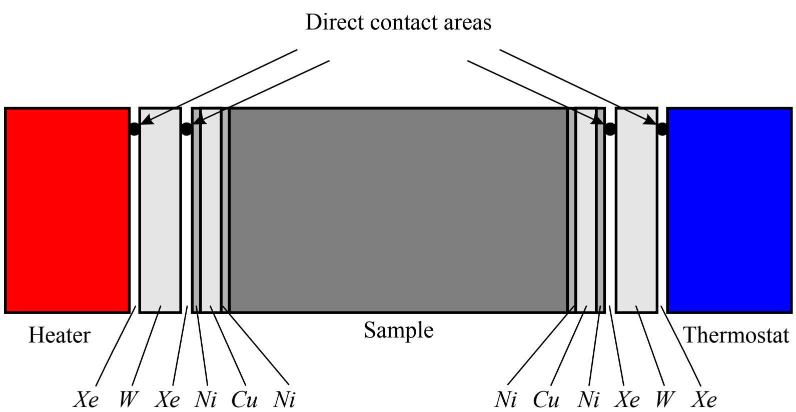


*Fig. 10. Schematic of contact structure.*

So, the case of a contact structure shown in Fig. 10 was also considered. It has the form of nickel coating (10 μm), copper coating (10 μm) and again nickel coating (10 μm) applied on the sample end surfaces. A sample with such a coating is forced against the heater and the thermostat through tungsten plates (200 µm thick), so as to assure a direct contact between the sample and the plate and between the plate and the heat exchanger at symmetrical points. Simulation results for different arrangement of such contacts along the sample radius are shown in Fig. 11.

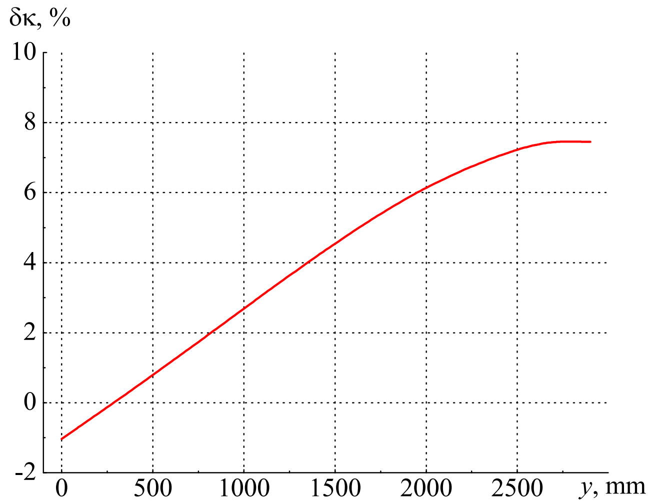


*Fig. 11. Dependence of the errors in the measurement of thermal conductivity at place of arrangement of point contacts on the sample end surfaces.*

Thus, with the worst arrangement of contacts (one per end surface) the error in the measurement of thermal conductivity will make about 7.4%.

In practice one should expect at least three points of contact between the sample end surfaces and the heater and thermostat. Computer studies of this case were performed that showed considerable reduction of the errors, when with the worst arrangement of three contact points on the sample surface the errors in the measurement of thermal conductivity will not exceed 1%. The errors in the measurement of electric conductivity in this case will be reduced to ~ 0.5%.

## Conclusions

The errors in the measurement of electric conductivity and thermal conductivity for the case of point electric and thermal contacts between the sample and current and heat leads are determined. It is established that these errors with the worst arrangement of contact points will reach about 45%.

The methods of reducing these errors by creating on the sample end surfaces of metal contact coatings based on nickel and copper are developed. It is established that for standard sizes of sample under study, in the presence on the sample end surfaces of 10 µm nickel layers and 100 µm copper layers, even with the worst arrangement of one point contact the error in the measurement of electric conductivity will make up to 1.8%, of thermal conductivity – up to 7.8%. In the presence of three contact points, these errors will be reduced to 0.5 and 1%, respectively.

The case of using tungsten anti-diffusion plate to avoid sample contact to the surfaces of current and heat leads at high temperatures is considered. It is established that such a plate will scarcely affect measurement errors.

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