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MEASUREMENT OF THERMOELECTRIC PROPERTIES OF MATERIALS AT HIGH TEMPERATURES

Results of research on the errors arising in the measurement of thermoelectric properties of materials by the absolute method in the temperature range of 30 – 900 °C are presented. It is established that the main measurement error is due to radiation from the surface of samples. It is also established that at temperatures of 600 – 900°C the use of gradient radiation screens becomes inefficient, namely the error of thermal conductivity measurement increases to 25 – 30 %. The efficiency of reducing the error of such measurements through use of powder thermally insulating materials is studied. It is established that their application combined with gradient thermal screens allows reducing the value of errors to 1.5 – 5.5 %. An experimental device that employs these methods of errors reduction is described.

Key words: measurements, absolute method, thermoelectric parameters, errors.

Introduction

General characterization of the problem. Creation of new thermoelectric materials, efficient at elevated temperatures, is one of important tasks in thermoelectricity [1-3]. Its solution requires methods and equipment to measure thermoelectric properties of materials with possibly high accuracy. In [4-6] it is shown that the acceptable measurement accuracy is efficiently provided by the absolute method using special gradient radiation screens. When measuring the figure of merit of material by this method, the error at temperatures up to 500 °C is not more than 4.7 %. At higher temperatures the use of radiation screens becomes inefficient and measurement errors are drastically increased.

The purpose of this work is research on physical factors causing the increase in errors at temperatures up to 900 °C, finding the ways for reduction of their impact on the accuracy of measurement, development of precise methods of measuring thermoelectric properties of materials and creation on their basis of corresponding measuring equipment.

Physical, mathematical and computer models

The model comprises a cylinder sample of length l and diameter d, a reference heater, a thermostat and a screen with a heater (Fig. 1).

The thermostat temperature is T_0, the reference heater and screen heater temperature is T_1; the thermal conductivity of sample material is κ_1, of the reference heater – κ_2, of the screen – κ_3, of the screen heater – κ_4; the emissivity factor of the sample is ε_1, of the reference heater – ε_2, of the screen – ε_3, of the screen heater – ε_4, of the thermostat – ε_5.

The model takes into account heat exchange due to radiation between the sample, screen, sample heaters and thermostat surfaces; heat transfer along the sample and screen; heat exchange due to radiation between the screen and thermostat.

To find temperature distribution in the measuring device, a system of thermal conductivity equations should be solved for each of its elements

$$\nabla(-\kappa_i \nabla T) = Q_i, \tag{1}$$

where Q_i is power of internal heat sources.

To solve this problem, the COMSOL Multiphysics software package was used

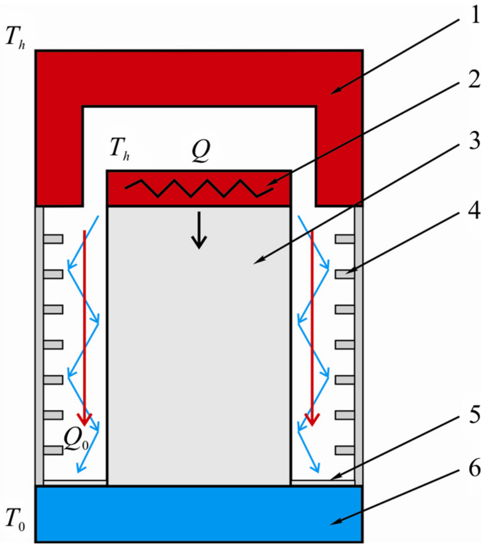


Fig. 1. Physical model of a device for measurement of thermoelectric material parameters by the absolute method: 1 – screen heater, 2 – reference heater, 3 – sample under test, 4 – screen with radiation rings, 5 – reflector, 6 – thermostat.

The boundary conditions that take into account heat exchange due to radiation between the measuring system members:

$$q = \varepsilon_i (G - \sigma T^4), \tag{2}$$

where σ is the Stephan-Boltzmann constant, G is heat flux due to radiation

$$G = G_m + F_{amb} \sigma T_{amb}^4, \tag{3}$$

G_m is heat flux from other device members, F_{amb} is viewing field factor equal to viewing field share that is not subject to other surfaces, T_{amb} is temperature at a distant point in the directions included to F_{amb}. Coefficient G_m which depends on mutual arrangement of surfaces is calculated by introducing into computer model of additional variable J assigned by equation

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

Results of investigation of the effect of radiation on thermal conductivity measurement accuracy at high temperatures

With expansion of operating temperature range, the role of radiation will increase. Fig. 2 shows the values of errors δκ at determination of heat flux through the sample versus measurement temperature for different values of absorption factors of the sample and reference heater surfaces. It is seen that even with the use of radiation rings on the screen and a reflector on the thermostat the errors reach 25 – 30 %.

The results obtained testify to the necessity of taking additional measures for the reduction of uncontrolled heat losses due to radiation from the surface of measured sample.

One such measure can be the use of powder heat insulating materials that fill the space between the sample and radiation screen. One of possible materials is perlite. Perlite thermal conductivity in the temperature range of 30 to 900 °C is given in Fig. 3.

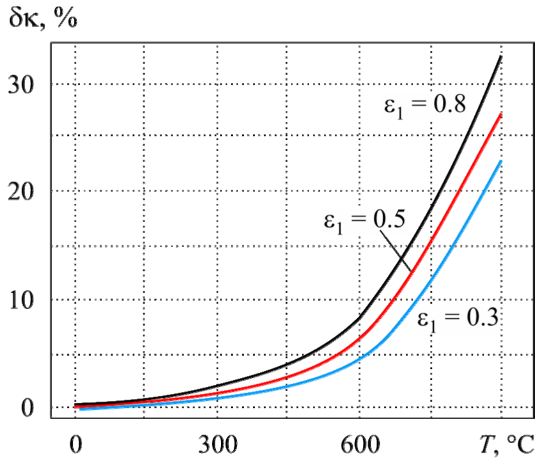


Fig. 2. Temperature dependences of errors in thermal conductivity measurement for different values of sample emissivity.

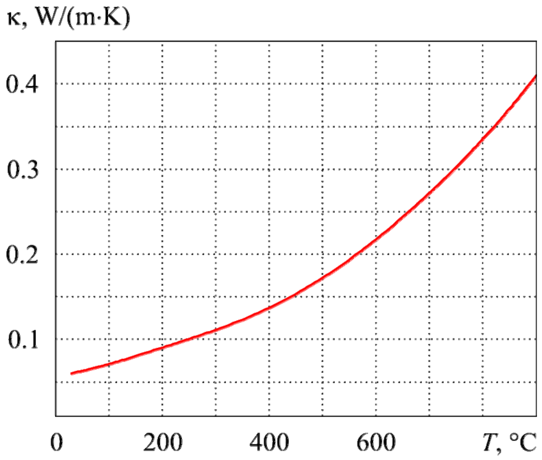
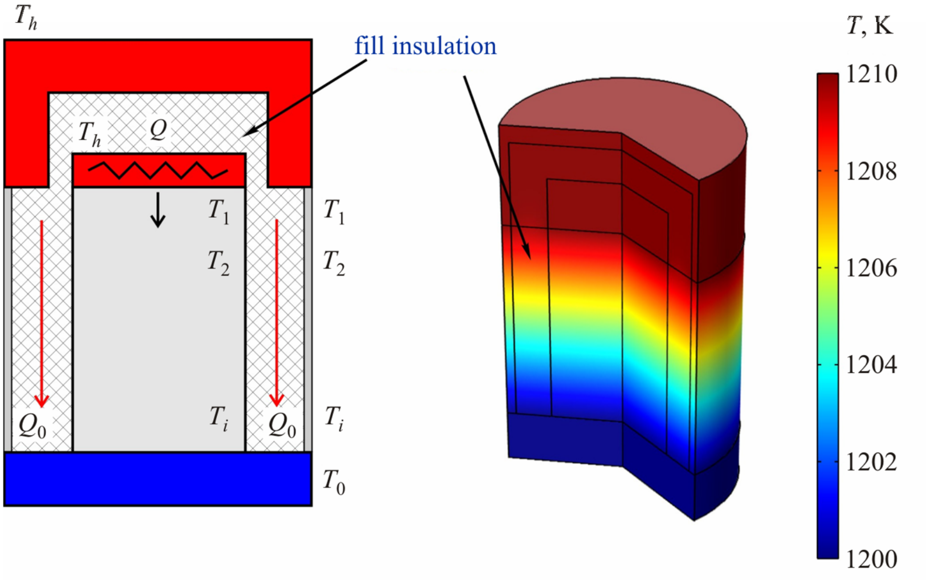


Fig. 3. Perlite thermal conductivity in the temperature range of 30 – 900 °C.

Computer simulation was used to investigate measurement errors for the model given in Fig. 4.



a) *b)*

Fig. 4. Use of perlite for the reduction of heat losses due to radiation a) physical model; b) temperature distribution obtained with the aid of COMSOL Multiphysics.

Simulation results are shown as temperature dependences of thermal conductivity measurement errors δκ for different values of sample thermal conductivity (Fig. 5).

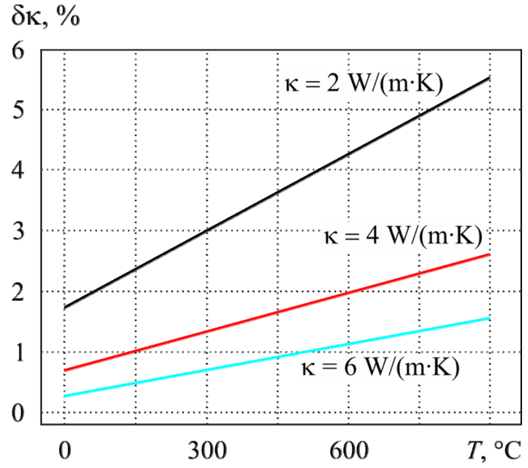


Fig. 5. Effect of fill insulation on thermal conductivity measurement errors.

As is evident, the use of thermal insulation allows reducing the measurement errors of κ to 1.5 – 5.5 %.

Description of measuring unit design

The results obtained were used to develop a measuring unit of installation for determination of thermoelectric properties of materials in the range of temperatures 30 to 900 °C (Fig. 6).

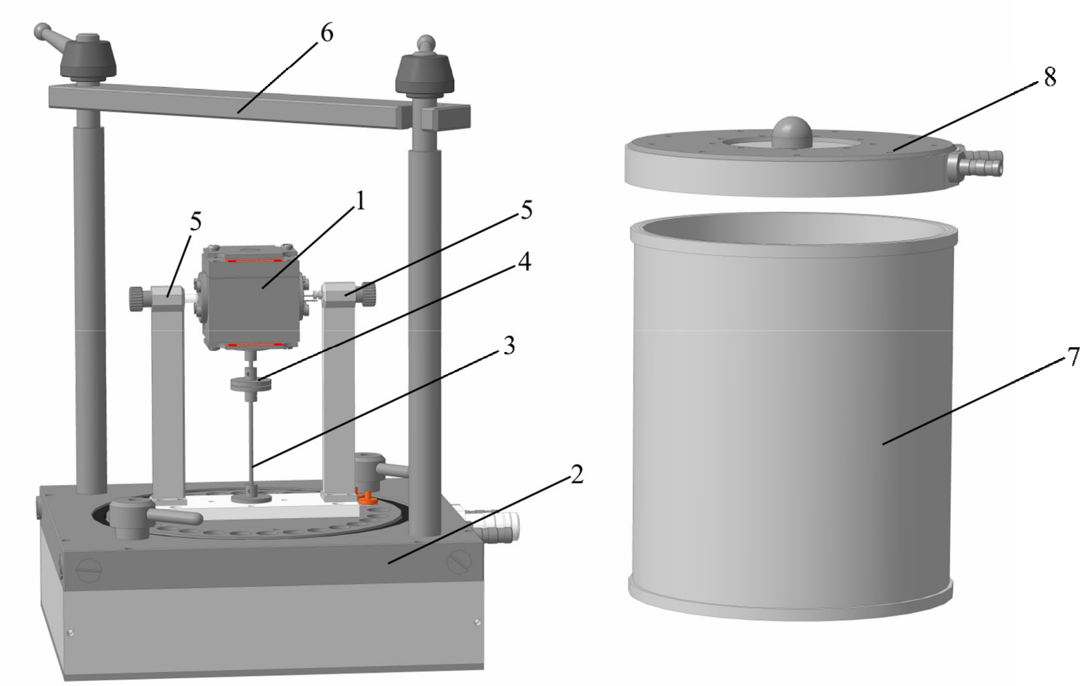


Fig. 6. Measuring unit of installation for determination of thermoelectric properties of materials.
1 – measuring device, 2 – water-cooled base of measuring unit,
3 – measuring thermostat post, 4 – thermostat locking heater, 5 – sample pressure device,
6 – bell jar pressure device, 7, 8 – lid of water-cooled bell jar.

Sample under test is placed inside the measuring device and pressed together with the heater to mounting platform (Fig. 7).

To reduce measurement errors, thermal switches are employed and powder heat insulating material is used to fill free space inside the measuring device.

To prevent the external surface of device from overheating, the measuring unit is also filled with heat insulating material.

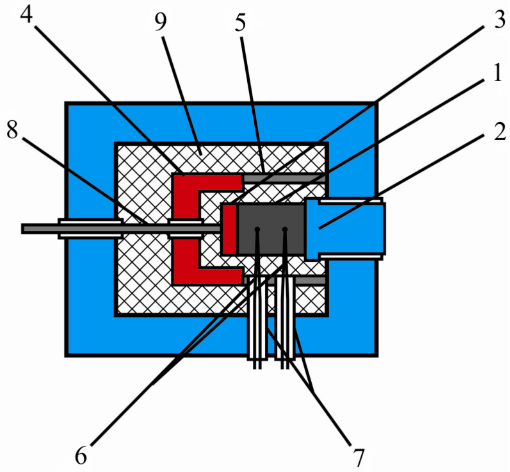


Fig. 7. Schematic of measuring device. 1 – sample under test, 2 – mounting platform, 3 – reference heater, 4 – protective heater, 5 – screen, 6 – thermocouples, 7 – sample pressure device, 8 – thermal switches, 9 – fill insulation.

Fig. 8 shows temperature distribution inside the measuring unit obtained by computer simulation. Computer studies allowed determining the necessary powers of background, reference and protective heaters and optimizing the unit design to achieve the isothermal conditions inside the measuring device where the sample is located. Moreover, the values of errors in the measurement of the other thermoelectric parameters were determined for the developed measuring unit, which are as follows: thermoEMF ~ 1.5 %, electric conductivity ~ 1.5 %, thermoelectric figure of merit ~ 10 %.

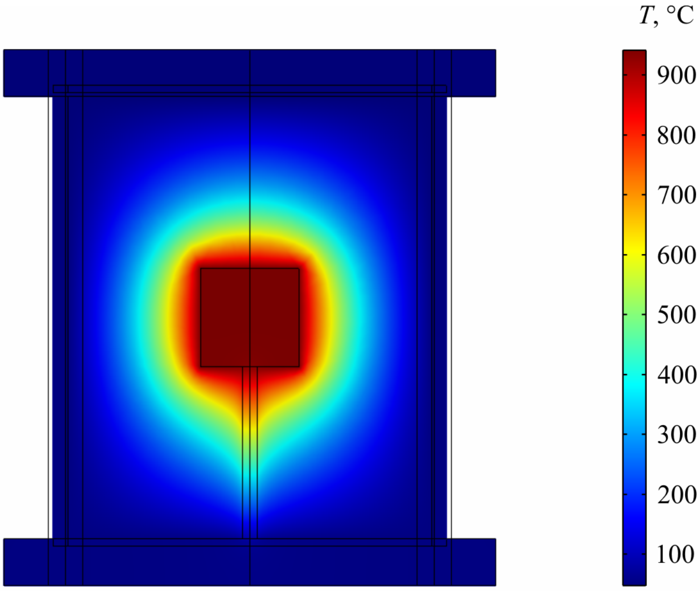


Fig. 8. Temperature distribution inside a passive thermostat.

Conclusions

1. The errors of thermal conductivity measurement by the absolute method at temperatures up to 900 °C have been studied. It has been established that the use of radiation screens alone for the minimization of heat losses from the sample surface results in the errors up to 25 – 30 %.
2. The values of errors in the measurement of thermal conductivity when using fill insulation have been calculated. For the model of measuring device in hand they make 1.5 to 5.5 %.
3. The measuring device has been developed and the values of its errors have been determined which at a temperature up to 900 °C are as follows: thermal conductivity ~ 5 %, thermoEMF ~ 1.5 %, electric conductivity ~ 1.5 %, thermoelectric figure of merit ~ 10 %.

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