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INCREASING THE RAPIDITY OF THERMAL CONDUCTIVITY MEASUREMENT BY THE ABSOLUTE METHOD

The results of computer investigations of the rapidity of thermal conductivity measurement by the absolute method are presented. Measurement errors caused by deviations from a linear distribution of temperature in the observable sample under the steady-state conditions have been analyzed. It has been established that in order to reach the acceptable values of these errors, measurement procedure must take a good deal of time, namely 10-15 hours to measure the temperature dependence of one sample. Methods for increasing the rapidity of reaching the steady state by the system have been developed. It has been established that alternating current passed through the sample under study permits to increase measurement rapidity by a factor of 3-5 due to accelerated heating of the sample central part by the Joule heat. Further increase of measurement rapidity can be achieved with a forced heating of the sample hot side by a reference heater. A combination of these two methods allows increasing measurement rapidity up to 8-10 times.

Key words: thermal conductivity, measurement error, rapidity, absolute method.

Introduction

General characterization of the problem. The efficiency of thermoelectric materials is the main efficiency factor of thermoelectric power converters. The success in technology and material research of thermoelectric materials is primarily dependent on clear correlation between the thermoelectric properties of materials and the technological features of their production. Finding this correlation is first and foremost dependent on the accuracy of measurement of the electric conductivity, thermopower and thermal conductivity of materials in given temperature range, as long as the results of impact on the substance can prove to be less than measurement error.

The most complicated process is thermal conductivity measurement over a wide temperature range. According to the analysis of the literature, the most consistent results can be obtained when thermal conductivity is measured by the absolute method [1-6]. Moreover, this method allows simultaneous measurement of thermopower and thermal conductivity, hence the thermoelectric figure of merit of material.

The use of the absolute method entails a problem of measurement rapidity. The necessity of reaching the steady-state conditions makes the measurements long-term, namely it takes 10-15 hours to measure the temperature dependence of one sample in the temperature range of 30 – 500 °C. In so doing, deviations from the steady state of experiment are an important factor that can affect the accuracy of thermal conductivity measurement. Deviations from the linearity of temperature distribution in the sample under study that occur in the process of reaching the steady state can serve the sources of errors in the determination of temperature difference on the sample ΔT, hence sample thermal conductivity

$$\kappa = \frac{Q}{\Delta T}\frac{l}{S}, \tag{1}$$

where Q is thermal power passed through the sample, l is the sample length, S is the sample cross-sectional area.

The purpose of this work is to develop methods for increasing measurement rapidity with the assured accuracy level.

Physical and mathematical models

A sample under study of length l and diameter d is fixed with one side to thermostat, as illustrated in Fig. 1. Thermostat temperature is T_0. Thermal power Q_0 from the sample heater is supplied to the other side of the sample whereby, upon reaching the steady state, temperature $T_г$ must be set on the sample hot side

$$T_г = T_0 + \frac{Q_0}{\kappa}\frac{l}{S}.$$

The lateral surface of the sample is adiabatically isolated.

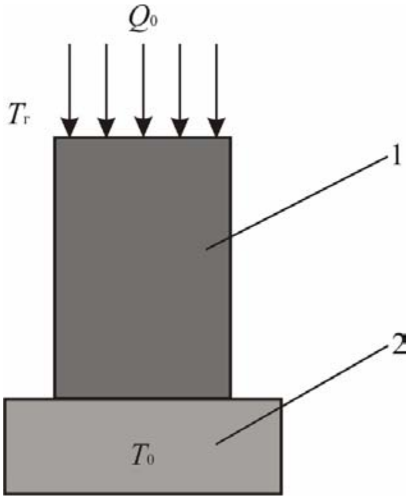

Fig. 1. Schematic of thermal conductivity measurement by the absolute method.
1 – sample under study, 2 – thermostat.

Temperature distribution in the sample upon actuation of the heater will change with time and depend on the sample properties, namely thermal conductivity κ, specific heat C and its geometric dimensions. To find this distribution at any point of the time, it is necessary to solve the unsteady-state thermal conductivity equation

$$\rho C\frac{\partial T}{\partial t} + \nabla\left(-\kappa\nabla T\right) = 0 \tag{2}$$

with the following boundary conditions:

– the sample cold side is thermostated at temperature T_0:

$$T = T_0,$$

– time-invariant thermal flux Q_0:is fed to the sample hot side

$$\boldsymbol{n}\cdot\boldsymbol{q}=\frac{Q_0}{S},$$

– the lateral surface of the sample is adiabatically isolated:

$$\boldsymbol{n}\cdot\boldsymbol{q}=0.$$

Results of investigation of measurement rapidity

To estimate the time necessary for the conduct of measurements whereby the steady-state experimental conditions are assured, it is necessary to study the effect of deviations from steadiness on measurement precision.

Time dependences of temperature distributions in the sample under study in the process of reaching the steady state by the system were obtained. Calculations were performed for a sample of *Bi-Te* based thermoelectric material of dimensions typical for thermal conductivity measurement by the absolute method – length 12 mm and diameter 8 mm. Sample thermal conductivity was 1.4 W/(m·K), heat capacity was 154 J/(kg·K). Thermostat temperature was 300 K. The heat capacity of sample heater was disregarded. If necessary, these results can be reproduced for other temperature ranges and sample dimensions.

Fig. 2 shows temperature distributions along the sample for different times of reaching the steady state by the system. It is seen that these distributions are nonlinear.

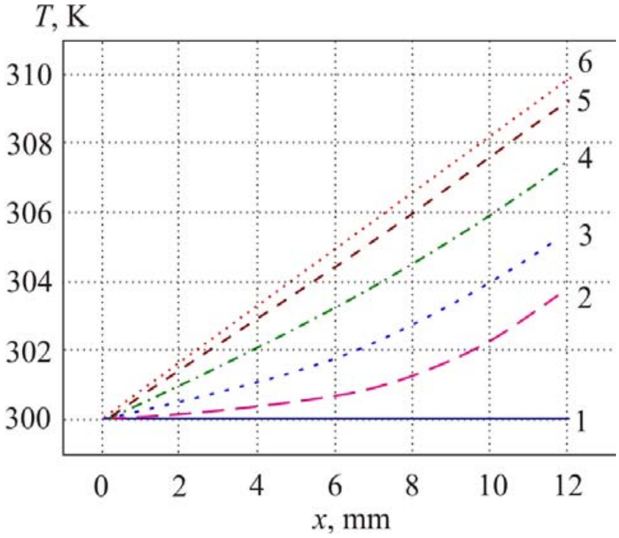

Fig. 2. Variation with time of temperature distributions along the sample
1 – 0 s, 2 – 15 s, 3 – 30 s, 4 – 60 s, 5 – 120 s, 6 – 240 s.

Deviations from the linear distribution are shown in Figs. 3 and 4. Percentagewise, the greatest deviations from the linearity will be close to thermostat. Hence, reaching the steady state by the sample hot side (sample heater) cannot serve as a guideline for the beginning of thermal conductivity measurement. This is seen in more detail in Figs. 5 and 6, showing a change in the sample hot side temperature with time and a deviation from the linear distribution of temperature along the sample at the time moment when a deviation from the expected temperature value on the sample hot side is 1% (for this case t = 224 s).

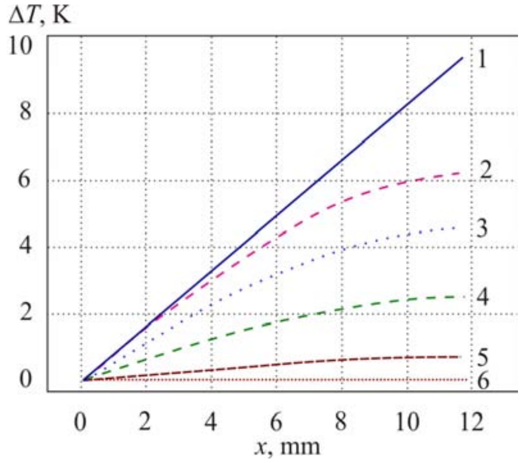

Fig. 3. Deviations from the linearity of temperature distribution along the sample upon reaching the steady state 1 – 0 s, 2 – 15 s, 3 – 30 s, 4 – 60 s,5 – 120 s, 6 – 240 s.

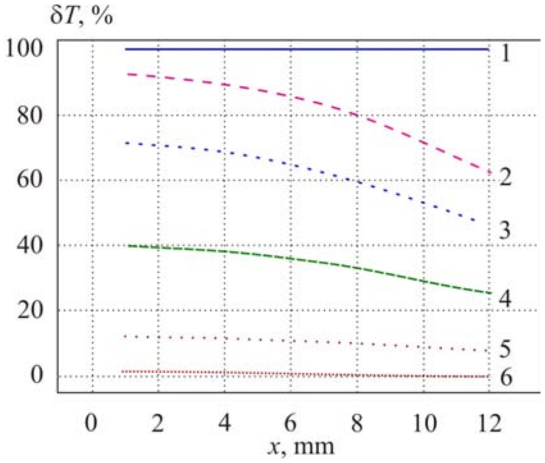

Fig. 4. Relative deviations from the linearity of temperature distribution along the sample upon reaching the steady state 1 – 0 s, 2 – 15 s, 3 – 30 s, 4 – 60 s,5 – 120 s, 6 – 240 s.

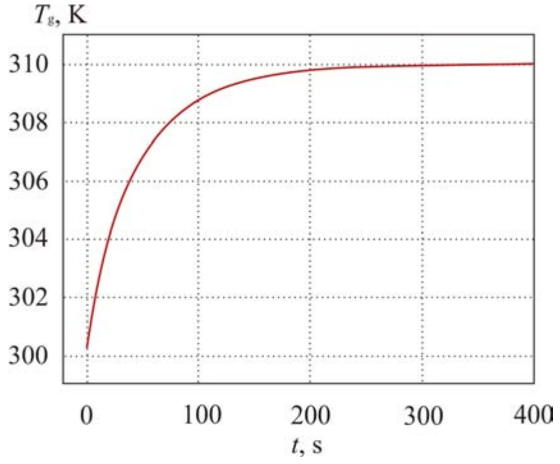

Fig. 5. Time history of the sample hot side temperature

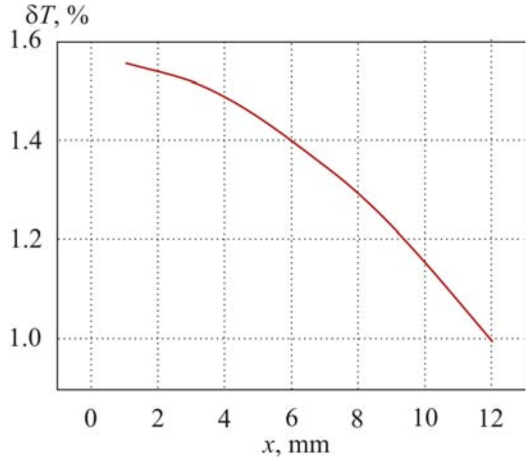

Fig. 6. Deviation from the linearity of temperature distribution along the sample (at the point of time when deviation from the steady state on the sample hot side is 1%)

Dependence of the error in thermal conductivity measurement on deviations of the sample hot side temperature from given value is shown in Fig. 7 (when measuring thermocouples are spaced 5 mm on the lateral surface of the sample).

Fig. 8 shows time dependences of the error in thermal conductivity determination, as well as the rapidity of change in the hot side temperature of the sample. A dependence of the error in thermal conductivity measurement on the rapidity of change in the hot side sample temperature is shown in Fig. 9. This dependence can serve the basis for determination of the moment of reaching the steady state by the system. Thus, for instance, to reach an error in thermal conductivity measurement less than 0.5% due to deviations from the steady state, one should wait for the time upon actuation of the heater, when the hot side temperature of the sample will change slower than 0.05 K/min.

The necessity of assuring the steady-state experimentation conditions brings up an important point of measurement rapidity. Fig. 10 shows a dependence of time t_0, during which maximum deviation from given linear temperature distribution will reduce to 1%, on the sample length.

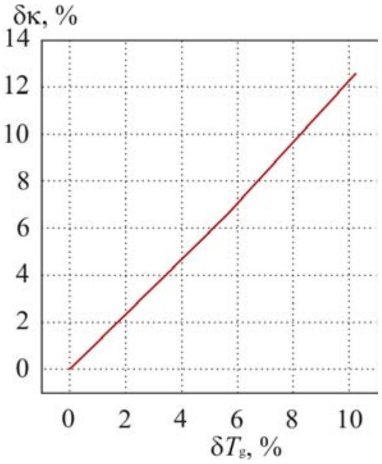

Fig. 7. Dependence of the error in determination of thermal conductivity on the accuracy of temperature stabilization on the sample hot side

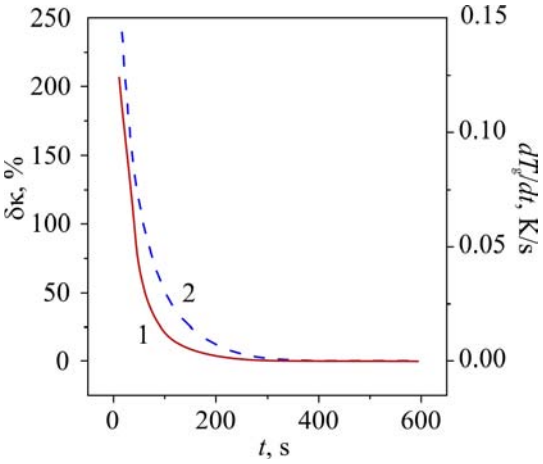

Fig. 8. Time dependences of the error in determination of thermal conductivity (1) and the rapidity of change in the hot side sample temperature (2) in the process of reaching the steady state

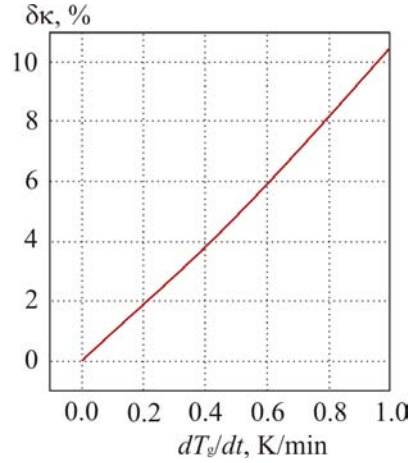

Fig. 9. Dependence of the error in determination of thermal conductivity on the rapidity of change in the hot side temperature upon reaching the steady state

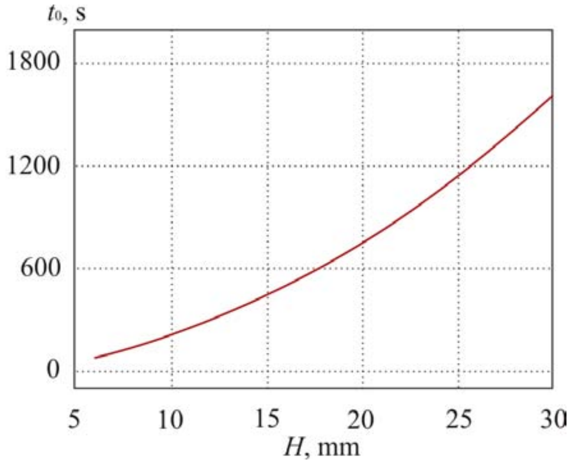

Fig. 10. Dependence of the time of reaching the steady state on the sample length

One of the variants of increasing measurement rapidity can be temporary passage of alternating current through the sample. This will accelerate heating of the central sample part due to release of the Joule heat in its bulk. Temperature distributions in the sample were studied for the case when during the initial period upon actuation of the heater an alternating current of given value is passed through the sample for some time. Thus, for instance, if during the first 30 s current 15 A is passed through the sample, then already at time instant t_0 = 96 s maximum deviation from the linear distribution will be less than 1%. These results testify that temperature distribution along the sample can reach the steady state 3 times faster compared to the case when no current is passed through the sample.

Measurement rapidity can be further increased when, alongside with alternating current passed through the sample, forced heating of the hot side of the sample is used at the initial moment upon actuation of the heater. Fig. 11 shows one of the partial cases of using this method of rapidity increase. Temperature distributions in the sample for this case are shown in Fig. 12. The time it takes for maximum deviation from the linear distribution to be within 1% is 38 s, suggesting the possibility of rapidity increase by a factor of 8-10.

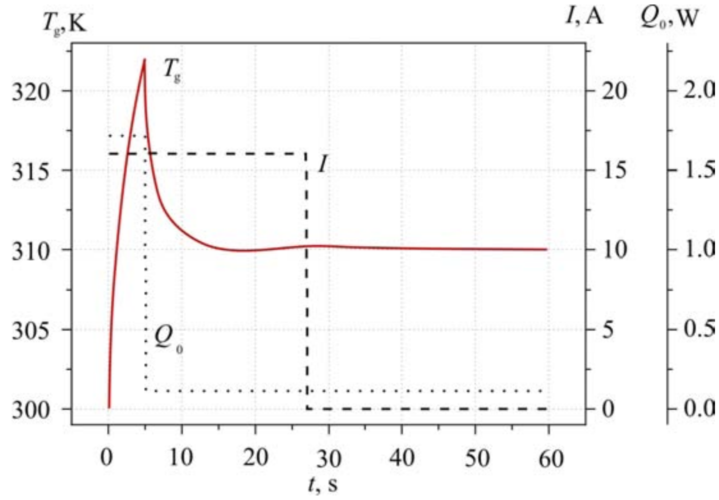

Fig. 11. Time dependences of the sample hot side temperature, current through the sample and heater power for the case of alternating current flow through the sample and forced heating of its hot side.

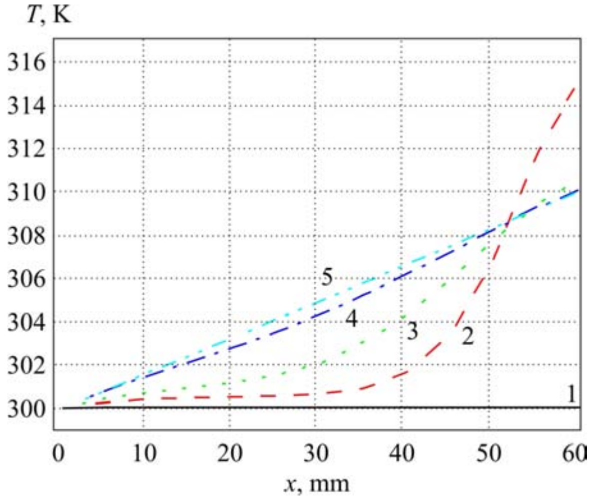

Fig. 12. Variation with time of temperature distributions along the sample for the case of alternating current flow through the sample and forced heating of its hot side. 1 – 0 s, 2 – 6 s, 3 – 12 s, 4 – 24 s, 5 – 48 s.

The above investigations serve the basis for the development of increased rapidity measuring installation which permits to expand the possibilities of using the absolute method of measurement, especially when determining the properties of large-size samples.

Conclusions

1. It has been established that a deviation of the sample hot side temperature from the given value cannot serve a criterion of reaching the steady state by the system, as long as inside the sample in this case there will be a deviation from linearity.
2. Dependences of the errors in determination of sample thermal conductivity on time in the process of reaching the steady state, as well as on deviation from the expected value of the sample hot side temperature have been obtained. It has been established that the error in determination of thermal conductivity exceeds by 20% a relative deviation from the expected value of the sample hot side temperature.
3. A method for increasing the rapidity of reaching the steady state by the system has been developed. It has been established that temporary passage of alternating current through the sample under study allows increasing measurement rapidity by a factor of 3 **-** 5, and in combination with forced heating of the sample hot side – by a factor of 10.

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