SERBIAN JOURNAL OF ELECTRICAL ENGINEERING
Vol. 6, No. 3, December 2009, 479 - 488

UDK: 536.251

Correction on the Influence of Thermal Contact Resistance in Thermal Conductivity Measurements Using the Guarded Hot Plate Method*

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Abstract: This work considers the influence of finite thermal contact resistances which exist in thermal conductivity measurements of homogeneous and poor thermal conductive materials using the guarded hot plate method. As an example of correction method proposed in this work, different experimental results obtained from a standard reference material sample (with the conductivity of about 1 W/mK) have been presented.

Keywords: Thermal conductivity, Guarded hot plate method, Poor thermal conductive materials.

1 Introduction

The guarded hot plate method is a standard method for the measurements of thermal conductivity of solids in the range from 0.1 to 10 W/mK. It is used for materials such as ceramics, polymers, materials of biological origin, and other. The method can be applied in a wide working temperature range, from 80 K up to 1200 K. The main principle of the method is one-dimensional steady-state heat conduction through a sample under test. According to Fourier's law of heat conduction, thermal conductivity is a function of heat flux and temperature gradient through the material.

Advantages of the method are the simplicity of mathematical model and the direct measurement of physical quantities – length, electrical power and temperature, resulting a relatively small expanded measurement uncertainty (below 4%). On the other side, major disadvantage is achieving the thermal equilibrium in the system (stationary heat conduction through the sample), as well as a need for repeating measurements in order to perform additional corrections due to non-ideality of boundary conditions, which makes experiments time-consuming.

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*Award for the best paper presented in Section *New Materials*, at Conference ETRAN 2009, June 15-19, Vrnjačka Banja, Serbia.

Another problem related to this method is a potentially great influence of finite thermal contact resistances between the sample and other elements of the measurement system on measurement results. This problem is particularly significant if air is present on the contact surfaces are rough and filled with air. In this case, the effect of the finite contact resistances on the final measurement result can be significantly reduced by applying external mechanical pressure or by inserting a thin layer of some fluid or solid with high thermal conductivity value.

In this paper, a technique for correcting the influence of finite thermal contact resistances on the measurement result, applicable for case of homogenous solid materials with smoothed surfaces is presented. In order to investigate the influence of thermal contact resistance between the samples and neighboring elements of the measurement system a series of two experiments on a pair of standard reference material (ILFORD glass) has been performed. In the first experiment contact surfaces were not covered with a contact medium (only the air was present) and in the other the air was substituted with the glycerin. Results are presented and compared to available reference data.

2 Description of the Apparatus

The apparatus used in this work consists of the central metering part, guard, isolation chamber, power supply and temperature controlling system, and acquisition system, as shown in Fig. 1.

The central part of the apparatus comprises the main heater (heat source), two samples made of the same material and size, placed above and below the main heater, and two coolers (heat sinks) above and below the samples and heater between them. The main heater and the coolers are made of brass, whose thermal conductivity is many times greater than expected thermal conductivity of the samples.

The temperature of the coolers is maintained by a circulating fluid from the thermostat 1 (Fig. 1). The central part is in the middle of the isolating chamber and fixed in a particular frame with a bolt and a calibrated coil.

The guard as an element of the central part is shaped as a ring composed of two C-shaped halves. Each half consists of a heater surrounded by a thermal insulation. When assembled, the guard surrounds the lateral surfaces of the main heater and samples, reducing heat losses from these surfaces to achieve the one-dimensional heat transfer through the samples.

The temperature of the isolation chamber that surrounds central part of the apparatus and the guard is maintained using a thermostat with circulating fluid (thermostat 2 in Fig. 1) in order to reduce the effect of the convective heat exchange inside the chamber. Convective heat transfer can be entirely eliminated if a vacuum-pump is used to extract the air from the interior of the isolating chamber.

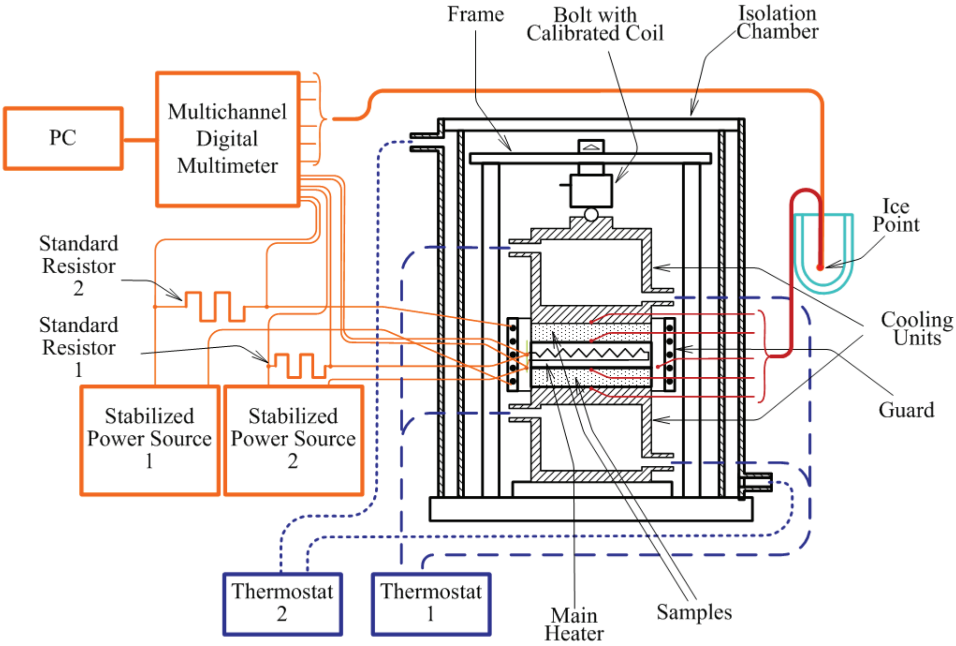


Fig. 1 – *Schematic description of the guarded hot plate apparatus.*

The power supply system consists of two electrical circuits, each implying a stabilized DC power source and two standard resistors used to measure the current through the main heater. Heat generated by the main heater is measured.

Acquisition system is consisted of a multi-channel digital multimeter and several temperature sensors – K type thermocouples. Their hot junctions are placed in the vicinities of contact surfaces of coolers, heater and guard, and cold junctions are kept at fixed ice point temperature. The multimeter is connected to a personal computer and controlled by a specially written LabView program.

More details on this apparatus are given in [1].

3 Analysis of the Influence of Thermal Contact Resistance

Steady-state heat conduction through the samples is achieved in equilibrium state when the heat flux from the heater to coolers becomes constant. In such conditions, assuming the one-dimensional heat transfer through the samples, the thermal conductivity of sample material, λ, is calculated as

$$\lambda = \frac{Ph}{2A\Delta T}, \quad (1)$$

where:

P - power released as heat by the main heater.

A - cross section area of one sample, multiplied by 2 for two-sample apparatus.

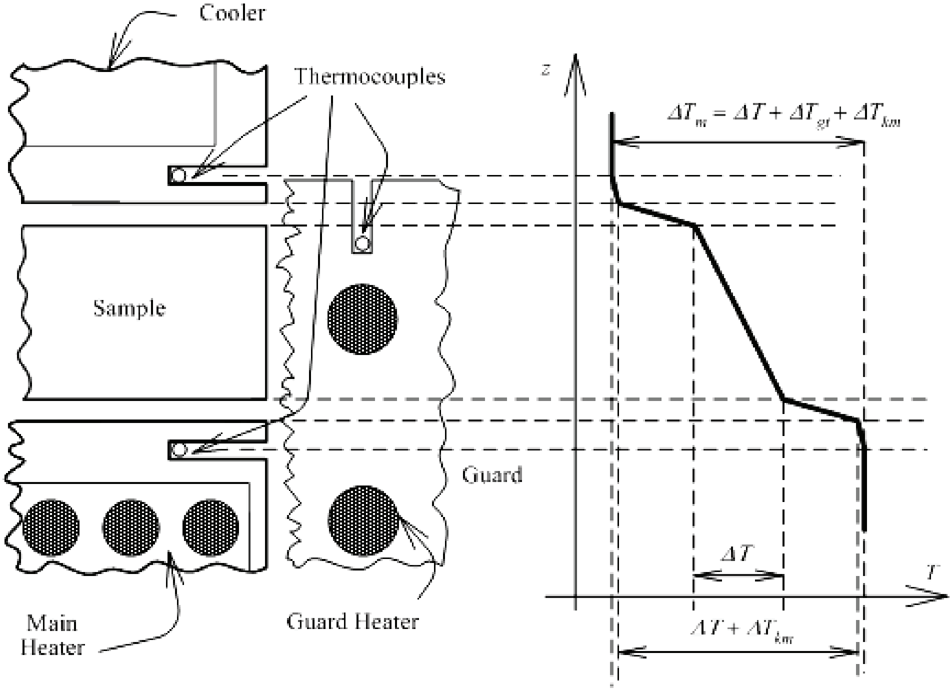
h - sample thickness.

ΔT - average temperature drop between hot and cold surface on both samples.

Analysis and data reduction depends on the characteristics of observed material, as well as of different parameters of the apparatus. Even though this method is based on a simple mathematical model, uncertainties of some parameters can significantly affect measurement results. For example, A and h can change due to variant atmospheric, mechanical or thermal conditions, while temperature measurement using thermocouples in different points in the system usually gives a great contribution to the combined uncertainty of the final measurement result.

The most significant problem is a consequence of heat loss from the lateral surfaces of the heater and the samples due to temperature unbalance (i.e. difference between the temperature in the guard and the mean temperature through the sample), thoroughly described in [1]. In brief, if the guard temperature is smaller than the mean sample temperature, a part of the heat generated by the main heater transfers from the sample to the guard, causing ΔT in (1) to be smaller, and vice versa. This effect can be corrected experimentally for every pair of samples on every operating temperature: with the main heater off and the guard heater on, several guard heater power values produce different guard temperature and steady-state ΔT values. Temperature unbalance, calculated from the measured values of the main heater, the coolers, and the guard temperatures, combined with the corresponding temperature drop ΔT, are used to construct the correction curve (line). When both main and guard heater are on, the temperature unbalance and ΔT are measured. ΔT values is then corrected using the correction line by interpolating the unbalance value to zero.

Temperature measurement can give a relatively great contribution to the combined uncertainty of calculated thermal conductivity value. In this work, thermocouples have been used as temperature sensors despite of their known disadvantages [2]. Thermocouples are very suitable for this cause mostly because of their small dimensions that merely affect temperature distribution in the central part of the apparatus. The ideal locations to place the temperature sensors are on the surfaces where the samples and the heating/cooling bodies are in contact. However, the final dimensions of the thermocouple junctions prevent good thermal contact on these surfaces. Instead, the thermocouple junctions are usually placed in narrow holes specially made in heaters and coolers bodies, approximately 1 mm below their contact surfaces, as shown in Fig. 2a.



a) b)

Fig. 2 – a) *Locations where temperatures of the main heater, upper sample and upper cooler are measured;* b) *Axial temperature profile through the measurement section.*

As a consequence, finite thermal conductivities of heater and cooler bodies produce temperature difference between the locations of thermocouples and corresponding surfaces, where also exist temperature gradients due to the surface roughness (Fig. 2b).

Under conditions of steady-state, one-dimensional (axial) heat conduction through the system, the measured temperature drop, ΔT_{m}, is equal to the sum of the temperature drop between sample surfaces, ΔT, the total temperature drop through heater and cooler, ΔT_{gt}, and the total temperature drop on both sample contact surfaces, ΔT_{km}:

$$\Delta T_{\mathrm{m}} = \Delta T + \Delta T_{\mathrm{gt}} + \Delta T_{\mathrm{km}} . \tag{2}$$

The relation between ΔT and thermal conductivity of the sample is given in (1), and it can be assumed that the same law could be applied for other components of measured temperature drop ΔT_{m}:

$$\lambda_{\mathrm{gt}} = \frac{Ph_{\mathrm{gt}}}{2A\Delta T_{\mathrm{gt}}}, \quad \lambda_{\mathrm{km}} = \frac{Ph_{\mathrm{km}}}{2A\Delta T_{\mathrm{gt}}} , \tag{3}$$

where h_{gt} and λ_{gt} are the total distance between the thermocouples and surfaces nearest to them and thermal conductivity of heater/cooler body material, respectively, and h_{km} and λ_{km} are the total "effective thickness" of the contact layers and thermal conductivity of the contact medium, respectively.

Total effective contact layer thickness, h_{km}, cannot be measured directly; a method to determine this parameter could be to perform a series of repeated experiments using the same sample and main heating power, but different contact media with different thermal conductivity values. So, for example, if in the first experiment the contact surfaces of the samples are coated (or sprayed) by a medium whose thermal conductivity value is λ_{km1}, and at some main heating power P we measure temperature drop ΔT_{km1}. Substituting these to (1) gives some thermal conductivity value λ_{m1}. In the second experiment the same samples, are coated by another contact medium with thermal conductivity λ_{km2}, and at the same heating power P we measure temperature drop ΔT_{km2}, and use these values in (1) to calculate value λ_{m2}. Temperature drop difference under changed contact medium condition from these two experiments is

$$\Delta T_{\mathrm{m1}} - \Delta T_{\mathrm{m2}} = \frac{Ph_{\mathrm{km}}}{2A}\left(\frac{1}{\lambda_{\mathrm{km1}}} - \frac{1}{\lambda_{\mathrm{km2}}}\right) , \tag{4}$$

and can be used to determine h_{km}. The correction of the influence of thermal contact medium to the total temperature drop can be performed by extrapolating ΔT_{km1}, ΔT_{km2}, ΔT_{km3}, etc. to the value $\Delta T_{\mathrm{m}\infty}$, that would be measured in an ideal case where thermal conductivity of the contact medium is infinite, $\lambda_{\mathrm{km}\infty} \to \infty$. Equation (4) could also be written for the measurement results from the first experiment (P, ΔT_{km1}, λ_{m1}) and the ideal experiment (P, $\Delta T_{\mathrm{km}\infty}$, $\lambda_{\mathrm{m}\infty}$):

$$\Delta T_{\mathrm{m1}} - \Delta T_{\mathrm{m}\infty} = \frac{Ph_{\mathrm{km}}}{2A}\frac{1}{\lambda_{\mathrm{km1}}} . \tag{5}$$

From Eqs. (4) and (5) the amount for measured temperature drop corrected to the influence of finite thermal contact resistances is:

$$\Delta T_{\mathrm{m}\infty} = \Delta T_{\mathrm{m1}} - (\Delta T_{\mathrm{m1}} - \Delta T_{\mathrm{m2}})\frac{\lambda_{\mathrm{km2}}}{\lambda_{\mathrm{km2}} - \lambda_{\mathrm{km1}}} . \tag{6}$$

Multiplying (6) with $2A/(hP)$ gives a following equation for thermal conductivity values corrected to error due to finite thermal contacts resistance on sample surfaces:

$$\frac{1}{\lambda}=\frac{1}{\lambda_{\mathrm{m1}}}-\left(\frac{1}{\lambda_{\mathrm{m1}}}-\frac{1}{\lambda_{\mathrm{m2}}}\right)\frac{1}{1-\frac{\lambda_{\mathrm{km1}}}{\lambda_{\mathrm{km2}}}}. \tag{7}$$

The last equation consists of only thermal conductivity values calculated from two experiments, λ_{m1} and λ_{m2} and known thermal conductivities of contact media, λ_{km1} and λ_{km2}, respectively. However, it must be noticed that it is derived under several assumptions that should always be verified when performing measurements and in later reduction of acquired data.

The assumption of the steady-state condition means that every single measurement lasts several hours. Moreover, it is very difficult to control precisely main heater power during the measurement: Once set, the central heater power must be fixed until steady state condition in the system is reached, and in (3) – (7) it is assumed the same value for P in every measurement.

When ΔT_{m} measurements are not performed with the same main heater power, the values ΔT_{m1}, ΔT_{m2}, and $\Delta T_{\mathrm{m\infty}}$, should be scaled to ΔT_{m01}, ΔT_{m02}, and $\Delta T_{\mathrm{m0\infty}}$, that correspond to a common central heater power P_0. In that sense, (6) can be written in the following form:

$$\Delta T_{\mathrm{m0\infty}}=\Delta T_{\mathrm{m01}}-(\Delta T_{\mathrm{m01}}-\Delta T_{\mathrm{m02}})\lambda_{\mathrm{km2}}/(\lambda_{\mathrm{km2}}-\lambda_{\mathrm{km1}}). \tag{8}$$

Multiplying (8) with $2A/(hP_0)$ also gives (7) in the same form.

Another important assumption is that the thermal conductivity value is calculated at a certain average sample temperature value, so (7) is valid only if λ_{m1} and λ_{m2} are obtained from results acquired at the same working temperature T_{sr}. This condition is also difficult to satisfy during the measurement, but if we measure thermal conductivity for one experimental setup on multiple working temperatures it is possible to interpolate λ_{m1} and λ_{m2} to the same working temperature values and successfully apply correction given in (7). Generally, thermal conductivity dependence on mean sample temperature is linear, so the linear interpolation of λ_{m1} and λ_{m2} can be applied to give satisfactory results. However, the linear dependence should be verified from results from no less than three working temperatures for each experimental setup.

4 Experimental Results

As an example of applying a correction to the influence of finite thermal contact resistances, a series of two experiments has been performed. A pair of samples used in these measurements is made of glass (labeled by the manufacturer as Z2), the standard reference material for thermal conductivity in the range from 0.1 to 10 W/mK [3]. For the first experiment contact surfaces were dry (filled with the air), and for the second, they were coated with a thin layer of glycerin. Both experiments were performed at three mean temperatures.

The results of initial data reduction – the values of thermal conductivity of glass, before applying the correction to the contact resistance influence, are given in **Table 1**. For each λ_{m1} (with the air as a contact medium) and λ_{m2} (with the glycerin as a contact medium) the correction to the temperature unbalance has been made as described in [1]. Column λ_{ref} in **Table 1** contains reference thermal conductivity values of the material in [4].

Table 1

Uncorrected values of thermal conductivity glass in cases of the air (λ_{m1}) *and the glycerin* (λ_{m1}) *as a contact medium on samples surfaces at three working temperatures each.*

T_{sr} [°C]	λ_{ref} [$Wm^{-1}K^{-1}$]	λ_{m1} [$Wm^{-1}K^{-1}$]	λ_{m2} [$Wm^{-1}K^{-1}$]
36.5	1.017	0.826	-
55.7	1.036	0.847	-
85.0	1.066	0.868	-
30.4	1.011	-	0.987
55.0	1.036	-	1.006
79.8	1.060	-	1.032

Maximum difference between λ_{m1} in **Table 1** and the values form the line obtained using the least squares method is 0.003 $W^{-1}K^{-1}$ (0.3 %), while for λ_{m2} values the greatest deviation from their optimal line is 0.002 $W^{-1}K^{-1}$ (0.2 %), giving enough reason to conclude that the linear law approximation for relation of λ_{m1} and λ_{m2} to working temperature T_{sr} is valid. According to proved linear dependence, values needed to calculate corrections according to (7) can be calculated by the linear interpolation of λ_{m1} and λ_{m2} values given in **Table 1**, i.e. the pairs of λ_{m1} and λ_{m2} values for each mean temperature T_{sr} are provided. Having values for thermal conductivity of the air and the glycerin from [4], equation (7) can be applied for all six values T_{sr} in **Table 1**. Calculation results are given in **Table 2**.

Results from two experiments without and with the correction to thermal contact resistance are given in Fig. 3, as well as the literature values for the sample material. It is obvious in Fig. 1 that the results of applied correction are remarkably close to the literature values in the entire working temperature range.

Table 2

Corrected values for thermal conductivity of glass (λ).

T_{sr} [°C]	λ_{ref} [$Wm^{-1}K^{-1}$]	λ_{m1} [$Wm^{-1}K^{-1}$]	λ_{m2} [$Wm^{-1}K^{-1}$]	λ [$Wm^{-1}K^{-1}$]
30.44	1.011	0.822	0.987	1.005
36.54	1.017	0.826	0.992	1.010
55.01	1.036	0.843	1.006	1.027
55.71	1.036	0.847	1.009	1.028
79.78	1.060	0.864	1.032	1.054
84.98	1.066	0.868	1.035	1.049

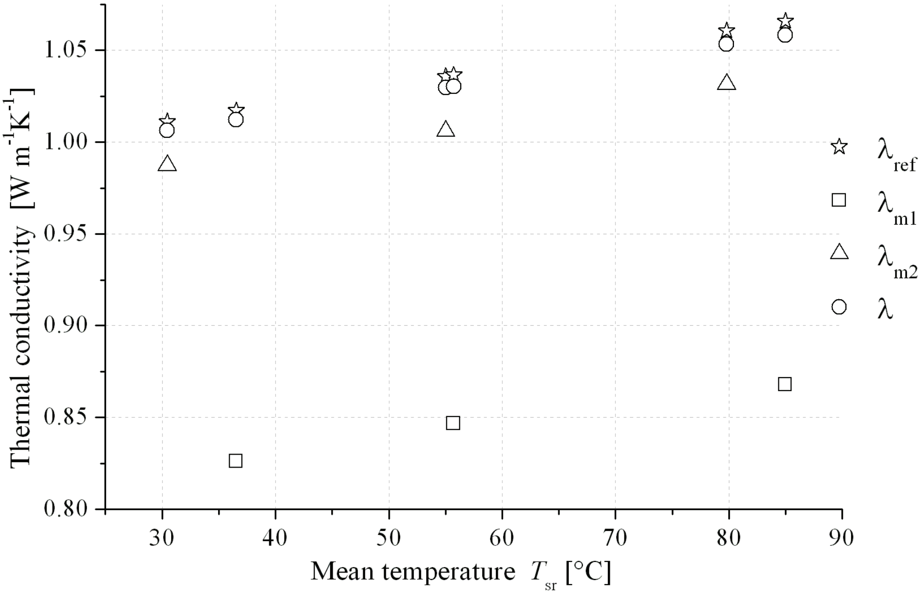


Fig. 3 – *Results of measurements and applied correction.*

4 Conclusion

Two experiments for determining thermal conductivity value of glass Z2 using the guarded hot plate method have been performed in order to examine the influence of finite thermal contact resistances to the measurement results.

According to values from two experiments, it has been noticed that there is a significant dependence of used medium to coat samples’ surfaces to reduce thermal contact resistance between samples and neighboring heating/cooling elements of the apparatus. This effect causes a systematic deviation from reference values, from more than 18%, if the air was used as contact medium, to less than 3% in the case of the glycerin. The absolute deviation of experimental values compared to literature values is approximately same at all working temperatures and using different main heater power values.

On the other hand, with the correction described in this paper, corrected results from both experiments differ from the literature values for less than 1% at all working temperatures.

The data reduction shown in this paper can be used to correct the results from a single measurement performed on the samples of another homogenous and non-porous materials (coated with glycerin layer over appropriate surfaces), without need to repeat the experiment with a different contact medium.

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