DEVELOPMENT OF A SINGLE-SIDED GUARDED HOT PLATE APPARATUS FOR THERMAL CONDUCTIVITY MEASUREMENTS

by

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This work presents the development of an experimental set-up for measurements of thermal conductivity of solid materials, such as ceramics, polymers, rubbers, glasses, biological materials, etc. whose thermal conductivity lies in the approximate range between 0.1 and 2 W/mK. The set-up was designed on the principle of the single-sided guarded hot plate method. In order to find the optimal design for generation of traceable 1-D heat flux through an investigated 300 mm × 300 mm specimen, a numerical heat transfer finite element method analysis was performed. The principal components of the measuring apparatus, such as hot plate with thermopile, cold plate, and auxiliary hot plate, were constructed according to the obtained results of simulations. Software for the control of experiment and data acquisition was developed using the LabVIEW programming environment.

Key words: *thermal conductivity, ceramics, polymers, rubbers, glasses, biological materials, guarded hot plate method*

Introduction

The guarded hot plate (GHP) method is the standard technique for measuring thermal conductivity of solid materials in the range from about 0.01 to 15 W/mK [1]. Insulation materials, such as fiberglass, polystyrenes, polyurethanes, foams, *etc.*, as well as mid-range heat conductors, such as ceramics, glasses, polymers, woods, *etc.*, may be successfully tested by the GHP method and many high quality national laboratories in the world developed their own apparatuses for using this method and provide direct traceability to the unit of thermal conductivity to industry.

Two basic variants of the GHP method are distinguished in literature: the double-sided (2S-GHP), and the single-sided (1S-GHP). The first one implies the use of two identical specimens for investigation, while the latter uses only one specimen. In the Department of Thermal Engineering and Energy of the Vinca Institute of Nuclear Sciences, Belgrade, two apparatuses for thermal conductivity measurements were developed in the past. One was based on the 2S-GHP method and dedicated for the characterization of 500 mm × 500 mm specimens of thermal insulation materials (thermal conductivity below 0.1 W/mK), [2, 3]. The other was designed according to method described in British Standard BS 874: 1973 [4, 5]

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and used for thin, cylindrical specimens with 75 mm in diameter of materials with thermal conductivity between 0.15 and 2 W/mK. The temperature range of both was from room temperature to approximately boiling point of water.

The new experimental apparatus had to be applicable for materials with higher thermal conductivity, and the measurement procedure should lead to more reliable results and better accuracy. After corresponding literature review, the choice was the 1S-GHP method which uses specimens with dimensions 300 mm × 300 mm and is appropriate for materials from the quoted group.

This paper presents a description of modelling applied, and realization of an apparatus based on the 1S-GHP principle. Particular attention was paid to the design of its most important parts, the hot and cold plates, and the main and guard heaters. 3-D modelling of the whole apparatus was also applied and it gave better understanding of parasitic heat flows, as well as an overall system behavior in different experimental conditions. Beside the apparatus, relating control and data acquisition software for performing the measurements was also developed, and it is briefly described at the end of the paper.

Method

The guarded hot plate technique in the single-sided mode of operation was introduced by Jakob in 1949 [6], who used it for thermal conductivity measurements of disk-shaped small-sized solid samples. In order to minimize influence of environmental conditions, the entire assembly was placed in a Dewar vessel. Afterwards, several authors developed and applied this type of apparatus. To avoid edge heat losses, an improved 1S-GHP apparatus was designed by Hemminger and Jugel [7]. Their apparatus was immersed in a liquid medium, whose temperature was maintained few degrees lower than the mean specimen temperature. Detailed analysis of uncertainty assessment of that apparatus was performed by Hammerschmidt [8]. Similarly, Zarr [9] presented an uncertainty assessment procedure for a NIST 1016 mm guarded hot plate apparatus [10] and provided an extensive example of uncertainty computation for single-sided mode of operation for a low-density fibrous-glass blanket thermal insulation. Another one-sided guarded hot plate apparatus was built by Filla [11] for testing various 1-8 mm thick ceramic samples and in a wide temperature range, between 400-1400 K. The most recently, Dubois and Lebeau [12] presented an 1S-GHP apparatus for the examination of large specimens, up to 40 cm in thickness, made of crop-based materials and non-altered straw bales.

The principle of the 1S-GHP method is based on the condition of stationary 1-D heat conduction through the specimen material in the form of thin disk or plate. The stationary heat flux is generated at the lower specimen base, while the other specimen surface is kept at a constant temperature. This process is described by the simplified Fourier law in one dimension:

$$Q = \lambda A \frac{\Delta T}{d} \tag{1}$$

where Q is the heat flow rate through the specimen, λ – the thermal conductivity of the specimen, A – the cross-sectional area of the specimen perpendicular to the heat flux, d – the thickness of the specimen, and ΔT – the temperature difference between the lower and upper specimen surface.

In practical realization, the 1S-GHP method implies the use of following main elements: hot plate, cold plate, and auxiliary hot plate, as presented in fig 1. The hot plate generates the uniform heat flux, the cold plate maintains the constant temperature, while the auxiliary hot plate produces additional flux which compensates the heat flow from the hot plate in direction opposite to the specimen. As the heat from the hot plate is generated and controlled by a built-in electrical heater, the relating heat flow rate is equal to electrical power generated by the heater. If one assumes that the generated heat flows only through the specimen, thermal conductivity can be obtained from eq. (1) by direct measurements of the electrical power, temperature difference, and parameters from the applied experimental set-up.

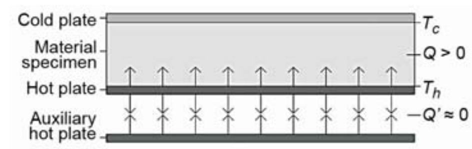

Figure 1. Principle of the 1S-GHP method

Although the method is based on the simple principle, realization of one-dimensional heat flux is not straightforward. In that sense, a 3-D finite element method (FEM) model was developed in order to have better understanding of the heat transfer through the system and its main elements. Also, the results of simulations can be helpful during measurements for the reduction of experimental time.

Modeling

The 3-D modelling started with definition of apparatus structure, geometry and dimensions of its main parts. The choice of the specimen surface was 300 mm × 300 mm, the maximum specimen thickness was set to 100 mm and the temperature difference across the specimen to about 10 °C, which is the average value in the range proposed in [13]. In order to have lower power of the main and guard heaters which produce lower fluxes, and at the same time to allow testing of thinner specimens where higher fluxes appear, the maximum allowable heat flux through the specimen was chosen to be 1000 W/m^2. According to the principle of the 1S-GHP method, the cross-section of the experimental assembly is shown in fig. 2.

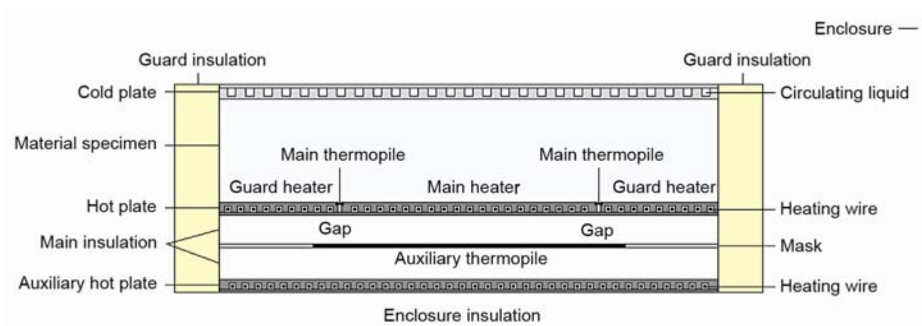

Figure 2. Cross-section of the proposed experimental assembly (dimensions are not proportional)

After defining the parameters and basic structure of the apparatus model, in the next step its main elements with corresponding materials for their construction were designed. In order to provide a perpendicular heat flow through the specimen, the model of the hot plate consists of two separate heaters, the main and the guard, where the purpose of the guard heater is to compensate lateral heat conduction from the main heater. The main and guard heater are separated with 1 mm air gap, each one residing on a duralumin alloy supporting metallic plate. On its lower side the plate has a grooved pattern for holding the NiCr20% heating wire. The pattern and relating dimensions are given in fig. 3. The auxiliary hot plate of the

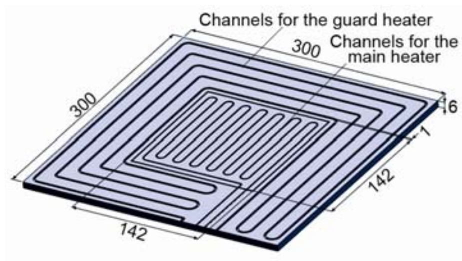

Figure 3. Model of the hot plate lower side

same material as the main hot plate was designed to accommodate single heater over its whole lower side.

The heat flow between the two hot plates is monitored by an auxiliary thermopile, modeled as a plastic disk, 172 cm in diameter and 5 mm thick. It is placed between two layers of the main insulation, consisting of a 1 mm thick sheet of silicon rubber. The purpose of this thermopile is to indicate existence of heat exchange between the main and the guard heater, which should be close to zero in desired operating conditions. For ensuring the best possible uniformity of heat flux between the plates, a 5 mm thick mask is placed around the auxiliary thermopile. The mask was modeled of amorphous glass with thermal conductivity similar to that of the auxiliary thermopile plastic disk, estimated to about 0.9 W/mK.

A copper cold plate with a channel for circulation of cooling liquid (fig. 5) maintains constant temperature of the upper specimen surface. For purpose of whole system modelling, the cold plate was treated as a homogeneous metallic block at a fixed temperature. High thermal conductivity of copper allowed such an approximation.

The guard thermal insulation (Styrodur, 3 cm thick) surrounds the lateral sides of the assembly, while another insulation material (polystyrene beads) fills the complete enclosure volume, as shown in fig. 2.

The values of all related thermal and electrical properties of materials used for the modelling were taken from literature [14-16].

All heating wires were defined as different heat sources, while the entire cold plate was modeled as a heat sink. After meshing the defined geometry, stationary state heat conduction equation was applied to every mesh element assuming the following boundary conditions: constant temperature at the upper specimen surface (temperature of the cold plate), equal heat fluxes and temperatures at all inner boundaries (thermal resistances at all contact surfaces were neglected), and zero heat fluxes at outer boundaries.

In the stationary state, the heat flow rate, Q, is equal to:

$$Q = Q_{\mathrm{mh}} - Q_{\mathrm{p}} \tag{2}$$

where Q_{mh} is the electrical power of the main heater, and Q_{p} – the total parasite heat flow rate. The latter quantity represents the sum of all undesirable heat fluxes, such as those existing through the gap and between the hot and auxiliary hot plate, as well as fluxes appearing due to edge effects of the specimen. The 3-D heat transfer simulations were performed for different values of specimen thermal conductivity and thickness, *i. e.* for 0.1, 0.5, 1, and 2 W/mK and for 10, 50, and 100 mm, respectively. In each simulation, the cold plate temperature was set to 25 °C. The power of the main heater, Q_{mh}, was varied in order to provide a temperature difference across the specimen of about 10 °C, while the powers of the guard and auxiliary heater, Q_{gh} and Q_{ah}, were altered for a maximum possible reduction of the parasitic heat flux, q_{p}. Then, the heat flux though the specimen, q_{s}, was directly calculated from the simulations results.

The results of performed simulations with the values of q_{s} and q_{p} among the others are shown in tab. 1. For all cases, except for that of d = 10 mm and λ = 2 W/mK, the obtained heat flux through the specimen was less or equal than the maximum allowable one. The simulated values of the powers from tab. 1 were used for a subsequent realization of all three heaters.

Table 1. Simulation results for the complete 3-D model of the experimental assembly for different values of specimen thermal conductivity, *λ*, and thickness, *d*

d [mm]	λ [Wm^{-1}K^{-1}]	Q_{mh} [W]	Q_{gh} [W]	Q_{ah} [W]	q_s [Wm^{-2}]	q_D [Wm^{-2}]	ΔT [°C]
10	0.1	2.02	7.05	1.07	10.17	–0.01	10.02
	0.5	10.1	33.23	1.18	500.87	–0.02	10.02
	1	20.2	64.45	1.25	1001.8	0.00	10.02
	2	40.4	122.5	1.22	2003.6	0.00	10.02
50	0.1	0.4	1.54	0.99	19.85	–0.01	9.93
	0.5	2.02	6.95	1.02	100.14	0.04	10.02
	1	4.04	13.61	1.06	200.32	0.04	10.02
	2	8.08	26.76	1.12	400.68	0.04	10.02
100	0.1	0.2	0.84	0.97	9.92	0.00	9.92
	0.5	1.01	3.57	0.98	50.12	–0.03	10.03
	1	2.02	6.93	1	100.15	0.03	10.02
	2	4.03	13.56	1.05	199.83	0.03	10.01

In practice, the simulations are used for initial settings of the heaters power, while further adjustments may be performed upon reaching each stationary state. It is interesting to note that the power of the auxiliary hot plate, Q_{ah}, is about 1 W in all cases. This leads to conclusion that undesirable heat flux between the two hot plates varies negligibly with the changes of Q_{ah}, which makes straightforward the setting of this power during the experiment. Also, an estimated value of the parasite heat flux is used for necessary corrections in later data reduction procedure.

Furthermore, based on simulations results, it was possible to examine the uniformity of temperature distribution on the surface of the heaters. In particular, temperature homogeneity of the main heater was the most important factor for the accuracy and precision of the apparatus. In this work, temperature difference across the main heater was about 0.1 °C, which was obtained with standard deviation of 0.03 °C.

Realization

As already mentioned, the aim of this work was to build an experimental set-up for testing solid materials with thermal conductivity values from 0.1 to 2 W/mK, in the temperature range 10-50 °C. All the elements of the experimental set-up were made according to models described in the previous section. The plates of the both hot plates were cut off a duralumin block and each one was firstly polished on the both sides, then carved with the corresponding grooved patterns and filled with the heating wire, as shown in fig. 4 for the main hot plate. Finally, the upper side of the main hot plate was covered with a 2 mm thick plate, also made of duralumin alloy.

The heating wires, made by THERMOCOAX®, had an electrical resistance per length of 14 Ω/m for the main heater and 6 Ω/m for the guard heater, which gave a total resistance of embedded heaters 25 Ω and 11 Ω, respectively. The total resistance of the embedded auxiliary heater was 70 Ω. The main thermopile was made of K type thermocouple wires,

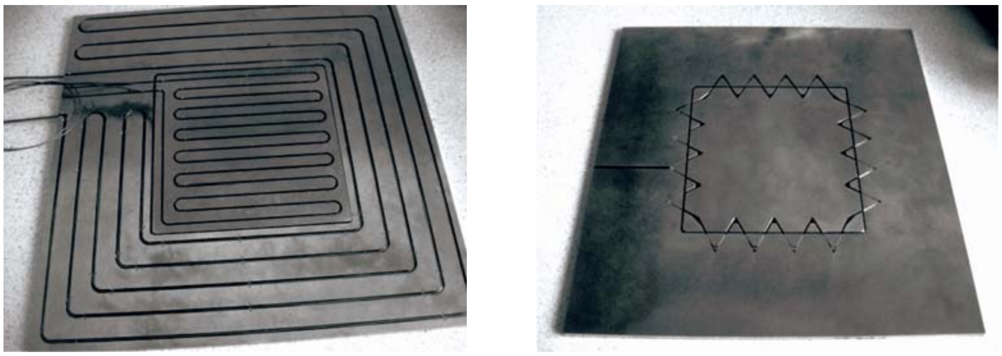

Figure 4. Photos of the main hot plate bottom (left) and upper side (right)

0.2 mm in diameter, whose 32 hot junctions in total were welded inside the corresponding upper side groove of the hot plate.

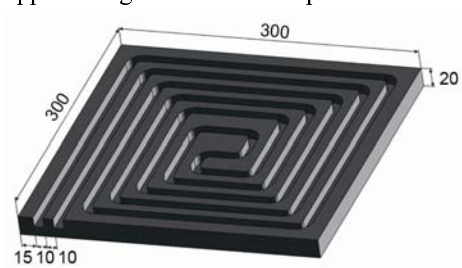

Figure 5. Draft of the cold plate upper side

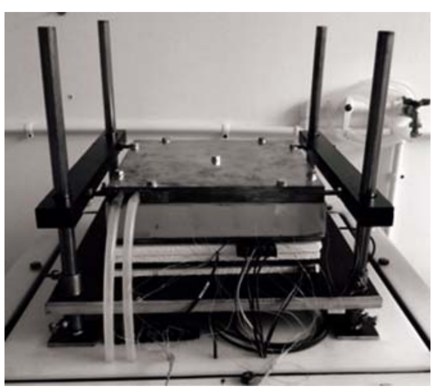

Figure 6. Photo of the assembled apparatus without the auxiliary insulation and the chamber

The dimensions of the main heater were the same as used in the simulations (fig. 3). The gap between the main and guard heater was fixed by using an epoxy glue placed in four symmetrical points on the supporting bottom duralumin plate.

The cold plate was cut from a relatively pure copper block. A double spiral channel was grooved on the upper side of the plate for the cooling liquid circulation, as presented in fig. 5. The same side of the plate was then covered by another 5 mm thick copper plate, which was fixed on by the epoxy glue. In order to minimize thermal contact resistances between surfaces, as well as the influence of different surface emissivities on the heat transfer through the system, thin layers of colloidal graphite were deposited on all relating surfaces of the hot and cold plates.

The auxiliary thermopile used in this work was an ultra-sensitive heat flux sensor made by HUKSEFLUX®, while the relating mask was cut accordingly of a window glass plate. Thermal conductivities of the sensor supporting material and mask were determined previously by the disc type apparatus.

The central part of the assembly was mounted on a 2 cm thick support of Bakelite with special stainless steel cylindrical anchors for carrying the cold plate and enabling its vertical movement (fig. 6). The lateral side of the construction was thermally insulated by auxiliary insulation layers made of Styrodur and the complete structure was surrounded by polystyrene beads and enclosed within a plastic chamber.

A schematic representation of the complete experimental set-up is given in fig. 7. A digital 3-channel 150 W DC source [17] realized in the Laboratory for Thermal Engineering and Energy, Vinca Institute for Nuclear Sciences supplies electrical power to all three heaters. The power of the main heater is obtained by measuring potential drop across the heater, and across series 0.1 Ω standard resistor, model LEEDS & NORTHRUP 4360. All the measurements are performed with a multi-channel digital multimeter model KEITHLEY 2700, and both the power source and the multimeter are connected via RS232 interface to a PC.

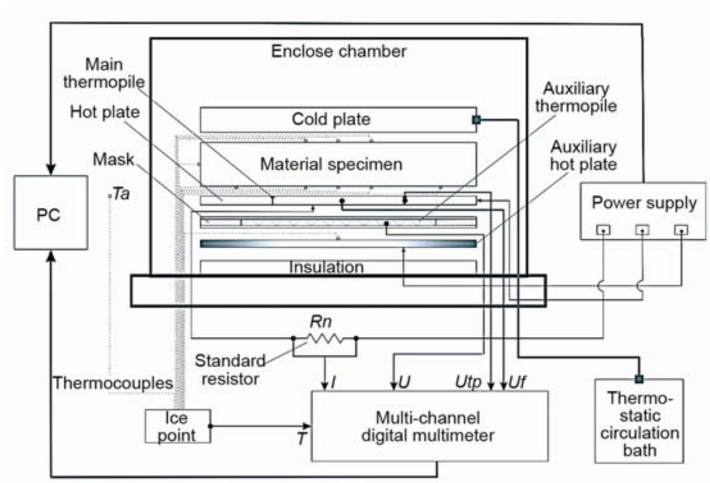

Figure 7. Schematic representation of the apparatus

Beside the voltage drop and current of the main heater, the multimeter measures the voltage responses of the main and auxiliary thermopile, as well as those of all temperature sensors. Temperature measurements are performed by K type thermocouples 0.1 mm in diameter, with cold junctions kept at the ice-point temperature. As it can be seen in fig. 7, the thermocouple hot junctions are placed at several locations, mainly on the specimen upper and lower surfaces. In order to provide a good thermal contact between thermocouple hot junctions and specimen, thin glycerin layers are spread at all relating surfaces.

For the regulation of the cold plate constant temperature, a thermostatic circulation bath, model JULABO F34-ED, is used, with ethanol as a circulation liquid. Its stability in the temperature range 10-50 °C is about 0.03 °C. The ice-point temperature is realized according to [18] by a mixture of crushed ice and distilled water kept in a Dewar flask, model KGW ISOTHERM 29BE.

Control of the used instruments, measurement process and data acquisition are performed by software [19] for this purpose developed in the LabVIEW programming environment. The program enables settings and adjustments of many parameters, such as the access point of devices, signals names, channel selection, sampling time, supply voltages, *etc.* Also, it makes possible a real time graphical and numerical presentation of all measuring signals, which is important for monitoring the system stability and achievement of a stationary state heat transfer. The left-hand side of fig. 8 shows a program tab with all measured temperature values, where the lower graph shows an ambient temperature signal. The right-hand side of the same figure illustrates another tab with average temperature values of the specimen upper and lower surface, mean specimen temperature, heat flux through the specimen and between the two hot plates, as well as preliminary values of the specimen thermal conductivity. The final results are obtained after subsequent detail analysis of all measured signals and data reduction procedure, which is performed by using ordinary computational software.

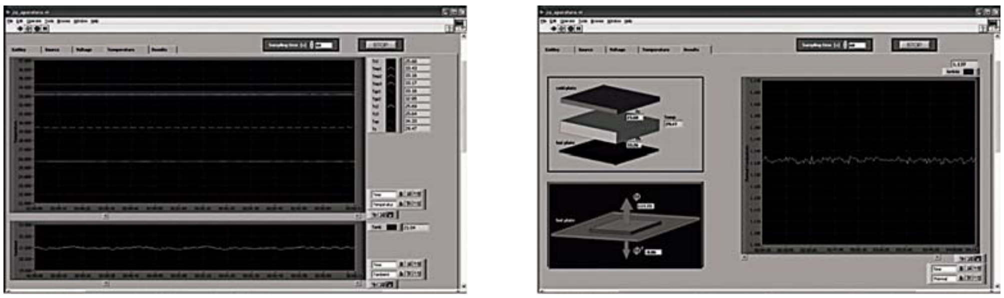

Figure 8. Example of program tabs with measured temperature signals (left) and preliminary results (right)

Conclusion and further work

The paper presents modelling and realization of a single sided guarded hot plate apparatus for testing solid materials on thermal conductivity in the range 0.1-2 W/mK and from 10 to 50 °C. The developed 3-D FEM model gives insight into the temperature and heat flux distribution inside and outside the specimen, which provides a better understanding of the apparatus behavior. The model also offers an estimation of the both separate and total parasitic heat fluxes, which is important for the accuracy and uncertainty of final results.

Validation of the apparatus and measurement and data reduction procedure were performed by using a standard reference material and related results will be presented in a separate paper. The complete experimental set-up will be tested by inter-comparison measurements in the framework of the European project "14RPT05 Eura-Thermal".

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