Application of the ASTM D5470 standard test method for thermal conductivity measurements of high thermal conductive materials

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ABSTRACT

Purpose: The purpose of the present study was to demonstrate the procedure for determining the thermal conductivity of a solid material with relatively high thermal conductivity, using an original self-designed apparatus.

Design/methodology/approach: The thermal conductivity measurements have been performed according to the ASTM D5470 standard. The thermal conductivity was calculated from the recorded temperature values in steady-state heat transfer conditions and determined heat flux.

Findings: It has been found from the obtained experimental results that the applied standard test method, which was initially introduced for thermal conductivity measurements of thermal interface materials (TIMs), is also suitable for materials with high thermal conductivity, giving reliable results.

Research limitations/implications: The ASTM D5470 standard test method for measurement of thermal conductivity usually gives poor results for high conductive materials having thermal conductivity above 100 W/mK, due to problems with measuring heat flux and temperature drop across the investigated sample with reasonably high accuracy.

Practical implications: The results obtained for the tested material show that the presented standard test method can also be used for materials with high thermal conductivity, which is of importance either for the industrial or laboratory applications.

Originality/value: The thermal conductivity measurements have been carried out using an original self-designed apparatus, which was developed for testing broad range of engineering materials with high accuracy.

Keywords: Thermal conductivity, Thermal resistance, Steady-state heat transfer, The ASTM D5470 standard

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PROPERTIES

1. Introduction

One of the methods of measuring thermal conductivity that is commonly used in laboratory practice is the one presented in the ASTM D5470 standard [1]. This method is intended for measuring contact thermal resistance and effective thermal conductivity of solid materials and greases (e.g. thermal interface materials – TIMs [2]) with thermal conductivity values in the range of 0.1-10 W/mK. Measurements of materials outside this range is very difficult because in one case there are problems with proper insulation of the system and on the other case recorded temperature gradients are burden with relatively high uncertainty. The essence of the method is to register the temperature drop in the layer of the material being tested (with an unknown thermal conductivity), placed between two blocks (cuboid or cylindrical bodies) made of a material with a known and usually very high thermal conductivity. Because of the role they play in the measurement system, these blocks are called HFM (Heat Flow Meter) blocks. One of these blocks is heated and the other is cooled; it is carried out in such a way that the temperature distribution in the blocks and also in the tested sample is one-dimensional and fixed in time. In this system, measuring the temperature distribution along the length of the blocks allows one to determine the temperature gradient, and thus the density of heat flux flowing through the blocks and the sample under test. Along the length of both HFM blocks there are temperature sensors located. The values recorded during the measurement are temperatures along the length of the blocks and the thickness of the test sample placed between the HFM blocks. Based on the values of these quantities, as well as the known thermal conductivity of the HFM blocks, the following are determined: heat flux density flowing through the tested sample, temperature drop along the thickness of the sample, equivalent heat flow resistance as well as the effective heat conductivity of the sample material and thermal contact resistance (also called adhesive thermal resistance for samples in the form of pastes or liquids) on the border between the tested sample and HFM blocks.

The purpose of the article is to present the procedure for measuring thermal conductivity in accordance with ASTM D5470 standard, which is carried out on a test stand designed at the Department of Thermal Engineering of the Silesian University of Technology in Gliwice. The article presents the results of the tests obtained for samples made of CuZn40Pb2 (CW617N) brass alloy. The value of the thermal conductivity of this type of brass is in the range of 100-130 W/mK, which is outside the range for which the method described is usually used.

2. Computation methodology of a thermal resistance

In the classical heat transfer theory, it is assumed that for isotropic bodies, the heat flux density vector is proportional to the temperature gradient, according to Fourier's law:

$$\dot{q} = -\lambda \nabla T \tag{1}$$

where λ is the thermal conductivity expressed in W/(mK) and T is the body temperature in K.

The proposed method of measuring thermal resistance is based on the analytical solution of the heat conduction equation for a one-dimensional problem of heat flow in a flat plate. The determined heat flow in a flat plate made of a material with a given, constant heat conductivity λ and thickness δ, infinite or thermally insulated in the other two directions, is described by a differential equation, which for such a one-dimensional task takes the form:

$$\frac{d^2 T}{dx^2} = 0 \tag{2}$$

along with the boundary conditions:

$$\begin{aligned} T(x=0) &= T_1 \\ T(x=\delta) &= T_2 \end{aligned} \tag{3}$$

where temperatures T_1 and T_2 are the values of the temperature field on the edges of the plate. The boundary problem (2, 3) has an analytical solution in the form of:

$$T(x) = \frac{T_2 - T_1}{\delta} x + T_1 \tag{4}$$

The temperature distribution in a flat plate is a linear function, where the quotient $\frac{T_2 - T_1}{\delta}$ is the temperature gradient in the plate, which in this case has a constant value. Knowing the temperature distribution, it is possible to determine, using the Fourier equation (1), the value of the density of heat flux conducted through the plate:

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$$\dot{q} = \frac{\lambda}{\delta}(T_1 - T_2) \tag{5}$$

where the quotient $\dfrac{\delta}{\lambda}$ is the heat conduction resistance in the plate. In the case of a system composed of many plates, in which the heat flow is realized in a perpendicular direction to the plane of the plates, exactly the same heat flux flows through each layer, while the temperature drops resulting from the individual heat conduction resistances add up (this system is called a series connected system).

The density of heat flux flowing through an array of plates – assuming that the temperatures T_1 and T_2, respectively, are known on the outermost surfaces of the panels – can be determined from:

$$\dot{q} = \frac{T_1 - T_2}{\dfrac{\lambda_1}{\delta_1} + \dfrac{\lambda_2}{\delta_2} + \cdots + \dfrac{\lambda_n}{\delta_n}} = \frac{T_1 - T_2}{\sum\limits_{i=1}^{n} \dfrac{\lambda_i}{\delta_i}} \tag{6}$$

When one-dimensional heat flow is considered through three contacting solids, as shown in Figure 1, two of which are HFM blocks with known properties, and the third body between the HFM blocks is the material being tested with an unknown thermal conductivity, and temperatures on the edges of HFM blocks are known values, then equation (6) takes the form:

$$q = \frac{T_1 - T_2}{\dfrac{\lambda_1}{\delta_1} + R_{cont,1} + \dfrac{\lambda}{\delta} + R_{cont,2} + \dfrac{\lambda_2}{\delta_2}} = \frac{T_1 - T_2}{\dfrac{\lambda_1}{\delta_1} + \dfrac{\lambda}{\delta} + \dfrac{\lambda_2}{\delta_2} + 2R_{cont}} \tag{7}$$

where temperatures T_1 and T_2 are the temperatures of the external surfaces of the HFM blocks, λ_1 is the thermal conductivity of the material of the hot HFM block, δ_1 is its length in the direction of heat flow, λ_2 is the thermal conductivity of the cold material of the HFM block, δ_2 is its length in direction of the heat flow, while $R_{cont,1}$ is the contact resistance of the heat flow between the hot HFM block and the tested sample expressed in $(m^2K)/W$, and $R_{cont,2}$ is the contact resistance of the heat flow between the cold HFM block and the tested sample, expressed in $(m^2K)/W$. Usually, both contact resistances between the tested sample and HFM blocks are combined in one resistance and assume that they are equal, because HFM blocks are made of the same material. The existence of additional resistance on the contact surface of two solids is due to its imperfection associated with the surface roughness of these bodies.

For the construction of the measuring system, it was assumed that the temperature in HFM blocks is linearly dependent on the coordinate parallel to the direction of the heat flow (equations (2-4)). With a relatively small variation in temperature along HFM blocks, the variability of the thermal conductivity with temperature can be neglected. Therefore, temperature readings can be approximated with reasonable accuracy by the linear functions:

$$T_1(x) = a_1 x + b_1$$
$$T_2(x) = a_2 x + b_2 \tag{8}$$

where functions $T_1(x)$ and $T_2(x)$ are temperature distributions in the hot and cold HFM block, respectively (Figure 1). The directional coefficients a_1 and a_2 of the obtained linear functions are equal to the temperature gradient (equation (1) and (4)) in the hot and cold HFM block:

$$\nabla T_1 = a_1$$
$$\nabla T_2 = a_2 \tag{9}$$

Knowing the value of the temperature gradient and the values of the thermal conductivity λ_1 and λ_2 of HFM blocks, using the Fourier equation (1), it is possible determine the heat flux density flowing through each of these blocks:

$$\dot{q}_1 = -\lambda_1 \nabla T_1 = -\lambda_1 a_1$$
$$\dot{q}_2 = -\lambda_2 \nabla T_2 = -\lambda_2 a_2 \tag{10}$$

In fact, even with very good insulation of the system, there are heat losses that will be higher for a hot HFM block than for a cold one. In addition, the temperature values measured with thermocouples have a certain error. Hence, both heat fluxes are not equal to each other; according to the literature, they should not differ by more than 5% of the measured heat flux. Finally, the value of the heat flux flowing through the tested sample is determined as the arithmetic mean of the values obtained for HFM blocks:

$$\dot{q} = \frac{\dot{q}_1 + \dot{q}_2}{2} = -\frac{\lambda_1 a_1 + \lambda_2 a_2}{2} \tag{11}$$

Next, the temperature drop observed at the junction of two solids, i.e. between two surfaces of the test sample, is

determined, e.g. by extrapolating the distribution of temperature values determined for HFM blocks:

$$\Delta T = T_1(\delta_1) - T_2(\delta_1 + \delta) = (a_1 - a_2)\delta_1 - a_2\delta + b_1 - b_2 \quad (12)$$

where δ_1 is the length of the hot HFM block, δ_2 is the length of the cold HFM block, δ is the thickness of the sample being tested, while a_1, b_1, a_2 and b_2 are coefficients of linear functions approximating the temperature distributions in the HFM blocks (equations (8)). Knowing the heat flux and the temperature drop in the test sample, the thermal resistance through the sample is determined from the formula:

$$R_{sample} = \frac{\Delta T}{\dot{q}} = -2\frac{(a_1 - a_2)\delta_1 - a_2\delta + b_1 - b_2}{\lambda_1 a_1 + \lambda_2 a_2} \quad (13)$$

In the above equation, there are only directly measured quantities, such as HFM block lengths (δ_1 and δ_2) and the thickness of the test sample (δ), and values determined on the basis of the temperature distribution measured along the length of the HFM blocks.
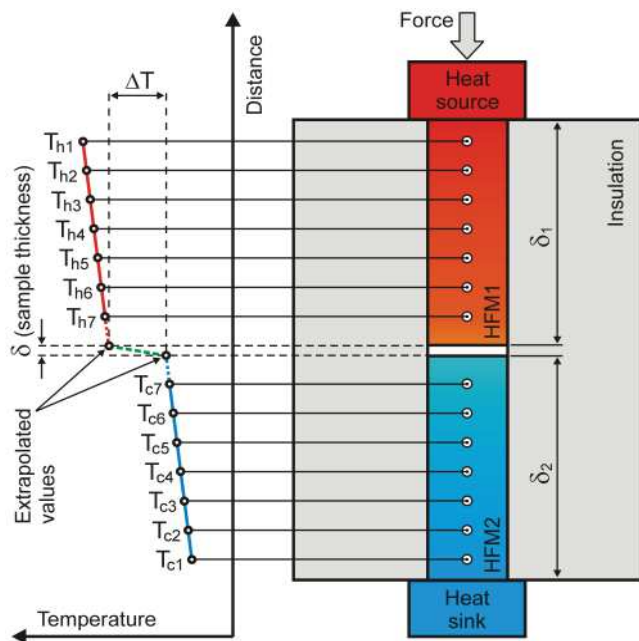

Fig. 1. Schematic view of the measurement system and methodology

In a general case, the thermal resistance through the test sample determined from the equation (13) contains a term related to the resistance arising at the interface of the tested sample and HFM blocks and a term related to the heat conduction in the test sample:

$$R_{sample}(\delta) = 2R_{cont} + \frac{\delta}{\lambda} \quad (14)$$

where δ is the sample thickness, λ is the thermal conductivity of the tested sample, while R_{cont} is the thermal resistance arising at the contact of the tested sample with HFM blocks. Basically, value of the contact resistance results mainly from imperfect contact adhesion of the tested sample to the surface of HFM blocks. It can be assumed that this value is constant for a given configuration, the tested material and the material of HFM blocks with which it is in contact. According to the equation (14), the observed thermal resistance through the tested sample – determined on the basis of measurements using the relationship (13) – can be treated as a linear function of the sample thickness. Therefore, when measuring the same material for samples of different thicknesses, approximating the results obtained with a linear function, it is possible to determine both the contact resistance as well as the thermal conductivity of the tested sample.

3. Materials

A commercially available CuZn40Pb2 (CW617N) brass alloy was chosen as a tested material due to its relatively high thermal conductivity. Three square sheet plate samples (40x40 mm^2) of different thickness (0.5; 1.0; 2.0 mm) were prepared for the investigations. The basic thermal properties, i.e. heat capacity and thermal conductivity were determined using Thermal Analysis Instrument STA 409 PG and Laser Flash Apparatus (LFA) Netzsch LFA 457 (NETZSCH-Geratebau GmbH, Germany), respectively. The selected properties of material tested are listed in Table 1.

Table 1.
Selected properties of tested material

Density, g/cm^3	Heat capacity, J/gK	Thermal conductivity, W/mK
8.44	0.377 (for 50°C)	119.0 (for 50°C)

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4. Measurement procedure and apparatus

For measurements of thermal conductivity, an own construction test stand (Fig. 2) was used, which was designed in accordance with the general guidelines presented in ASTM D5470-06 [1] and literature [3,4]. The main element of the apparatus is a measuring system consisting of two HFM blocks (Fig. 3), in the form of rectangular solids with a square base with a side of 40 mm and a height of 120 mm. In accordance with the construction requirements, a pure aluminium (a material with a high thermal conductivity) was used to manufacture these blocks. Seven blind holes were drilled in these blocks (from the lateral surface to the axis of the block) and thermocouples were placed in them (as in Fig. 3).
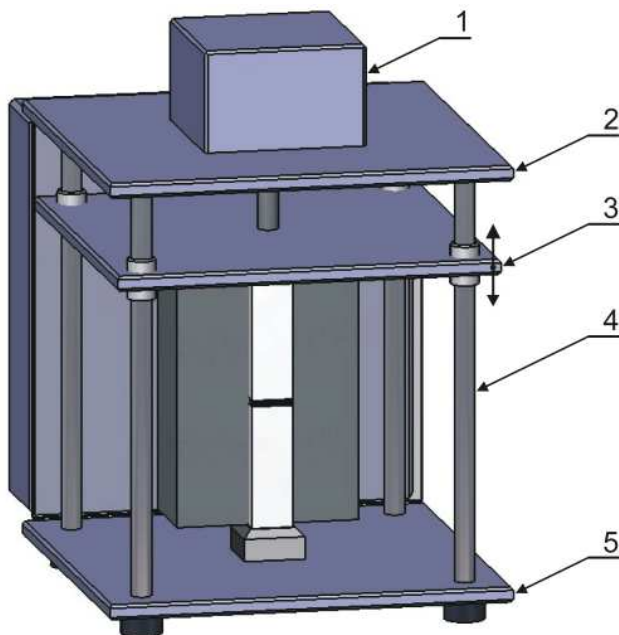
Fig. 2. Model of the test stand; 1 – drive system, 2 – upper mounting plate, 3 – moveable mounting plate, 4 – guide columns, 5 – lower mounting plate

A heating element was attached to the upper HFM block, and a cooling element was attached to the lower HFM block, and thus forces a temperature difference value and, as a result, the heat flow through the test sample. Both blocks was thermally insulated with a polymer foam material (in Fig. 1 and Fig. 2 only the fragment of insulation is shown, so that the HFM blocks are visible) with very good insulating properties (difference of about four orders of magnitude in the value of the thermal conductivity), which guarantees one-dimensional heat flow

through such a system. This allows one to easily determine the heat flux flowing through the sample. Additionally, front surface of the blocks was covered with some black matt paint with an emissivity of about 0.93, so that it was possible to register thermographic images on them to show the temperature distribution over the length of these blocks.
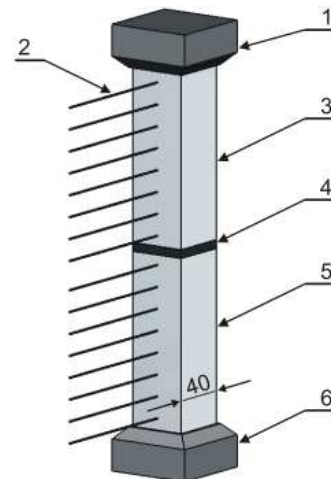

Fig. 3. Model of measuring system of the test stand; 1 – heat source, 2 – thermocouples, 3 – heated HFM block, 4 – tested sample, 5 – cooled HFM block, 6 – heat sink

The dimensions of the test samples (40 x 40 mm^2) were chosen to cover the entire base surface of the HFM blocks. Prior to the measurement, some thermal contact material was applied to both surfaces of the sample to obtain the required heat flow efficiency between the HFM blocks and the sample.

5. Results

The results presented below were obtained during the measurement in which the set values of heating temperature were 60°C and 70°C (i.e. the value recorded on the upper surface of the hot HFM block), while the value of the cooling temperature was 20°C for both cases (i.e. the value recorded on the bottom surface of the cold HFM block). Figure 4 shows an example thermal image recorded at the front surface of the HFM blocks with a temperature profile along the measurement line (white vertical straight line). Figure 5 shows the temperature values obtained during the measurement in a steady state using thermocouples located inside the HFM blocks (in this figure the zero point on the x axis is located in the middle of the thickness of the test sample).
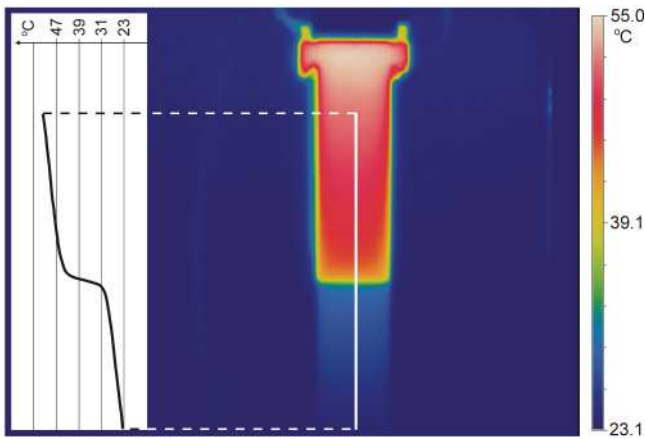
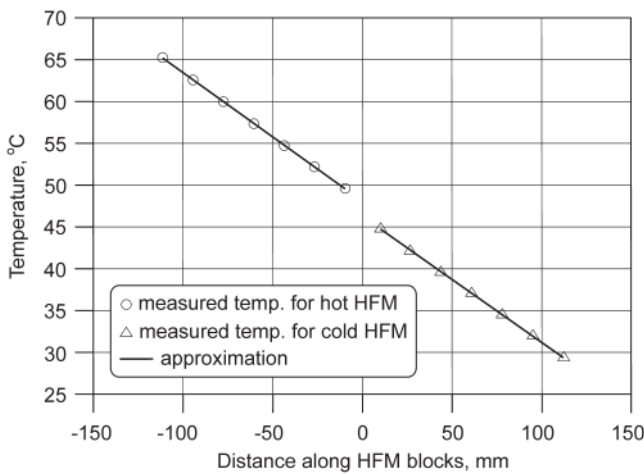Fig. 4. Exemplary thermal image captured on the surface of HFM blocks [5]

Fig. 5. Exemplary temperature values recorded inside the HFM blocks for 2.0 mm sample

Fig. 6. Obtained values of thermal contact resistance for different thickness of tested sample

Table 2.
Experimental results

No.	Sample thickness, mm	Thermal resistance, mm^{2}K/W	Thermal conductivity, W/mK
1	0.5	40.22 (for 60°C)*	
2	1.0	44.99 (for 60°C)*	
3	2.0	53.34 (for 60°C)*	
4	0.5	39.83 (for 70°C)*	114.94
5	1.0	44.43 (for 70°C)*	
6	2.0	52.95 (for 70°C)*	

* heating temperature of hot HFM block

Figure 6 shows the determined values of the thermal resistance depending on the thickness of the test sample, which were approximated by a straight line with the equation given in the legend of this figure. According to the procedure for determining the equivalent thermal resistance described earlier, the inverse of the directional coefficient of the straight line (a = 8.70 mmK/W) is the thermal conductivity of the tested material. Table 2 summarizes the test results, i.e. individual values of the thermal resistance (for a given sample thickness) and the resulting thermal conductivity. The value of thermal conductivity obtained in the presented experiment, equal to 114.94 W/mK, is in very good agreement with value found in the literature: 114.67 W/mK [6].
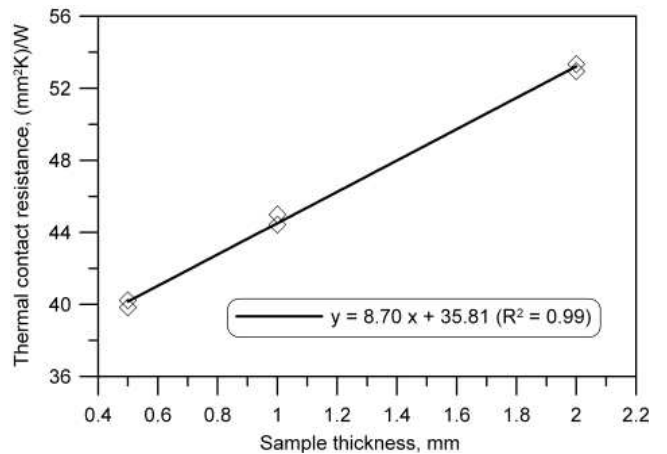
6. Conclusions

In the present work the accuracy of the thermal conductivity measurements using an original self-designed apparatus has been studied. The procedure based on the ASTM D5470 standard test method is typically used in practice to measure thermal conductivities of materials in the range from 0.1 up to 100 W/mK. However the extreme values are difficult to measure accurately because in case of weak conductors there is a problem with precise measurement of the heat flux, while in case of good conductors there is a problem with accurate measurement of temperature drop across the investigated sample. The paper presents the application of the method to test the selected material having relatively high thermal conductivity (CuZn40Pb2 brass alloy). The value of the thermal conductivity (114.94 W/mK) obtained with the developed test stand is similar to the value (119.0 W/mK)

obtained from standard LFA technique, which proved the high accuracy of this apparatus. The experimental results demonstrate the suitability of the considered standard test method also for testing the materials with relatively high thermal conductivity.

Acknowledgements

References

[1] ASTM D5470-06, Standard Test Method for Thermal Transmission Properties of Thin Thermally Conductive Solid Electrical Insulation Materials, ASTM International, 2006, DOI: 10.1520/D5470-06.

[2] C. Liu, Y. He, C. Chen, M. Chen, W. Yu, Synergistic enhancement of heat transfer between irregular shaped particles and boron nitride flakes at low loading, Results in Physics 15 (2019) 102599, DOI: 10.1016/j.rinp.2019.102599.

[3] J.R. Culham, P. Teertstra, I. Savija, M.M. Yovanovich, Design, assembly and commissioning of a test apparatus for characterizing thermal interface materials, Proceedings of the 8th Intersociety Conference on Thermal and Thermomechanical Phenomena in Electronic Systems "ITherm2002", San Diego, 2002, 128-135, DOI: 10.1109/ITHERM.2002.1012448.

[4] R. Kempers, P. Kolodner, A. Lyons, A.J. Robinson, A high-precision apparatus for the characterization of thermal interface materials, Review of Scientific Instruments 80/9 (2009) 095111, DOI: 10.1063/1.3193715.

[5] Z. Buliński, S. Pawlak, R. Białecki, W. Adamczyk, T. Krysiński, Determination of the thermal conductivity of solids based on the ASTM D 5470 standard, Steel, Metals & New Technologies 3-4 (2019) 57-62 (in Polish).

[6] E. Cuce, P.M. Cuce, T. Guclu, A. Besir, E. Gokce, U. Serencam, A novel method based on thermal conductivity for material identification in scrap industry: An experimental validation, Measurement 127 (2018) 379-389, DOI: 10.1016/j.measurement.2018.06.014.