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High-Temperature Thermal Conductivity Measurement Apparatus Based on Guarded Hot Plate Method

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Abstract An alternative calibration procedure has been applied using apparatus built in-house, created to optimize thermal conductivity measurements. The new approach compared to those of usual measurement procedures of thermal conductivity by guarded hot plate (GHP) consists of modified design of the apparatus, modified position of the temperature sensors and new conception in the calculation method, applying the temperature at the inlet section of the specimen instead of the temperature difference across the specimen. This alternative technique is suitable for eliminating the effect of thermal contact resistance arising between a rigid specimen and the heated plate, as well as accurate determination of the specimen temperature and of the heat loss at the lateral edge of the specimen. This paper presents an overview of the specific characteristics of the newly developed "high-temperature thermal conductivity measurement apparatus" based on the GHP method, as well as how the major difficulties are handled in the case of this apparatus, as compared to the common GHP method that conforms to current international standards.

Keywords Heat transfer · High-temperature guarded hot plate · Thermal conductivity · Thermal contact resistance

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List of symbols

GH	guarded hot plate
HTTCMA	high-temperature thermal conductivity measurement apparatus
TCR, R	thermal contact resistance
λ	thermal conductivity
$dQ(z)$	heat flow in axial direction
dQp	heat loss in radial direction
A	metering area of the heater plate
$t_{RS}(z)$	temperature function in the metering zone
$t_{RG}(z)$	temperature function in the guard zone
tig	temperature of the inner surface of the gap
teg	temperature of the outer surface of the gap
Δt	temperature drop across the specimen
d	specimen thickness
t_m	mean temperature of the specimen
dt/dz	temperature derivative of the specimen
Pe_{MZH}	electrical power supplied to the heater plate
Q	heat flow at the inlet section of the specimen
Qg	heat flow loss at the guard-center gap
Qg_t	total heat flow loss across the center-guard gap
Qg_l	conductive heat flow across the gap
Qg_R	radiative heat flow across the gap
tis	temperature of the inlet section of the specimen
tos	temperature of the outlet section of the specimen
thp	temperature of the hot plate surface
tcp	temperature of the cold plate surface
q	density of heat flow rate
Ri	TCR at inlet section of the specimen
Ro	TCR at outlet section of the specimen

1 Introduction

One major difficulty with the "guarded hot plate" (GHP) method is ensuring that there are no lateral heat losses, so the density of heat flow rate through the sample is effectively one-dimensional. Another problem is the potentially significant influence on the measurement results of the non-vanishing thermal contact resistances between the sample and the elements of the measuring instrument.

This paper presents an overview of the specific characteristics of the newly developed high-temperature thermal conductivity measurement apparatus (HTTCMA), highlighting the different manner of treating the above-mentioned problems compared to a high-temperature guarded hot plate (HTGHP) that conforms to standards ISO 8302:1991(E) [1] and CEN/TS 15548-1:2011 [2].

The specific scientific objectives include experimental validation of a theoretical model of radiative heat transfer existing between the metering zone and the guard zone of the HTTCMA. The new approach consists of modified design of the apparatus, modified position of the temperature sensors and new conception in the calculation method, applying the temperature at the inlet section of the specimen instead of the temperature difference across the specimen. This alternative technique based on temperature measurement within a rigid specimen from thermal protection material is suitable for eliminating the effect of the thermal contact resistance (TCR), as well as for accurate determination of the specimen temperature and of the radial heat loss along the specimen.

The work was performed within the framework of the European Metrology Research Program titled ”Metrology for Thermal Protection Materials.” The EMRP SIB52 project provides new techniques for the next generation of national standard instruments and identifies reference materials with a thermal conductivity in the range $0.02\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$ to $1\mathrm{W \cdot m^{-1} \cdot K^{-1}}$ and at temperatures in the range from 70 °C to 650 °C.

2 Principle of the HTTCMA

In the case of the HTTCMA built in-house at MKEH, a new conception was applied. The aim is not to minimize the measurement errors, but to quantify them, and to take all of them into account as corrections, in this way minimizing the measurement uncertainty.

The principle of operation of the HTTCMA is the GHP principle, having a heated lateral guard. The design differences between a conventional GHP and HTTCMA are related to optional elements given in the standard CEN/TS 15548-1:2011 [2].

As advantage of the new approach can be mentioned the role of the thermometer’s position in accurate determination of the specimen temperature and elimination of possible high TCR arising between a rigid specimen and the heated plate. Another advantage is the accurate determination of the lateral heat losses, as well as the new calculation method of the thermal conductivity (λ), permitting the possibility of a larger temperature drop across the specimen.

The disadvantage of this new measurement procedure is that the measurement is more time consuming due to different positioning of the temperature sensors.

2.1 Characteristics of the HTTCMA

The HTTCMA is a double specimen apparatus (Fig. 1); therefore, the density of heat flow rate used in calculating the thermal conductivity of a test specimen is determined with half of the power supplied to metering area of the heater plate. The thermal conductivity range is from $0.02\ \mathrm{W \cdot K^{-1} \cdot m^{-1}}$ to $5\mathrm{W \cdot K^{-1} \cdot m^{-1}}$, and the apparatus works in the temperature range from 70 °C to 800 °C.

The apparatus consists of a central metering section in which measurements are taken and a surrounding guard section. However, the heater plate is divided into a central heater and a lateral guard heater that are separated by a center-guard gap.

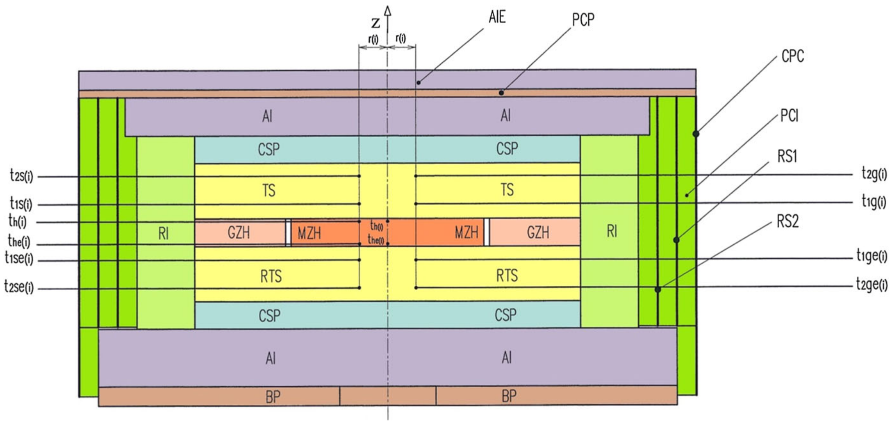


Fig. 1 Scheme of the in-house built HTTCMA

The heater plate of 320 mm diameter, composed of a central measuring zone of 200 mm diameter and a guard zone of 60 mm, is placed horizontally. The thickness of the test specimen of isolation material is limited to 20 mm by the 2 mm width guard-center gap, situated between the center metering section and lateral guard section. In standard CEN/TS 15548-1:2011 [2] is given that the thickness of the specimen shall be at least ten times the width of the gap.

The HTTCMA is composed from measuring zone heater (MZH), guard zone heater (GZH), test specimens (TS and RTS), cold surface plate (CSP), axial and radial insulations (AI, RI, PCI and AIE), protective cover plates (PCP, CPC), radiative screens (RS1, RS2) and base plate (BP) (Fig. 1).

2.2 Design Differences Between HTGHPs and the HTTCMA

2.2.1 Heater Plates

Considering the recommendations given by the standard ISO 8302:1991(E) [1] that at high operating temperatures the heater plate has to be thicker to ensure uniform temperature distribution across the plate, the thickness of the heater plate is 30 mm. The cold plate is not heated as is optional in the standard CEN/TS 15548-1:2011 [2].

The material of the heater plate needs to be mechanically stable under thermal cycling and to be opaque to thermal radiation in order to avoid direct radiation of the heating wires of the interfaces between plates and specimens. Usually, materials with high thermal conductivity have low emissivity. The standard ISO 8302:1991(E) specifies that the surfaces of the plates must have a total hemispherical emissivity above 0.8. The preferred way to control the emissivity of the plate surfaces is to coat them with a high emissivity coating. But these coatings have short service life and are not validated for long-term durability in these special circumstances inherent in a HTGHP technique using up to 800°C. Furthermore, it is highlighted by the standard [1] the possibility of chemical reaction between the coating and the test material, resulting changes in thermal resistance of the specimen.

To avoid the potential durability problems with these coatings and their potential chemical reaction with the test specimen, the heater plate of the HTTCMA was made from thermal resistant stainless steel material AISI 314 & Grade: 1.4841AS/EN 10095, with the composition of 24.72 % Cr and 19.17 % Ni. In this case, the constantly high emissivity of the heater plate of 0.8 is obtained by forming an initial thin oxidation film on the thermal resistant metal. The relative emissivity of the heater plate was determined by comparison method between a contact and a non-contact thermometer. First, the surface temperature of the heater plate was determined with a contact thermometer. Then, the emissivity given by the non-contact thermometer was adjusted until this thermometer indicates the same temperature as the contact thermometer.

The majority of elevated temperatures isolation materials, as ceramics and silicates, have high thermal contact resistance (TCR), as presented in sect. 4, resulting in less information about the temperature of the specimen if the sensors are placed in the heater plates. Therefore, there is a high risk that the two temperature gradients across the specimen and the active (heated) edge guard will differ. In order to avoid this problem and based on the standard CEN/TS 15548-1:2011 [2] giving the active edge guard as optional element, a passive edge guard was used for the HTTCMA, composed from insulation and radiative screens.

2.2.2 Position of the Temperature Sensors

Determination of the thermal conductivity by the GHP method involves the measurement of the temperature difference between the opposite faces of a test specimen when a constant heat flow passes through it.

Use of sheathed temperature sensors is recommended by the standard CEN/TS 15548-1:2011 to limit the degradation of these sensors due to oxidation. It is furthermore specified that the temperature difference across the specimen can be measured either by temperature sensors embedded in the heater plates in contact with the specimen, or by temperature sensors embedded in the specimen itself.

The common measurement procedure is measuring the temperature difference across the specimen by temperature sensors embedded in the heater plates in contact with the specimen.

In case of the HTTCMA, a number of four–four S-type sheathed thermocouples are placed in holes inside the upper specimen and the lower one, respectively, as presented in Fig. 1. Another two S-type sheathed thermocouples are placed in grooves on the upper and lower surface of the heater plate (Fig. 1). All temperature sensors are movable in the radial (z) direction. Actual temperatures in different points of the metering zone (MZ) and the guard zone (GZ) of the specimens are measured by moving manually the sensors. By applying the extrapolation method, the temperature of the surfaces can be determined without distortion.

Based on these measurements effectuated by temperature sensors placed inside the specimen, the correction function can be determined.

The change of the heat flow in axial direction ($dQ(z)$) is equal with the heat loss in radial direction (dQp).

$$dQ\left(z\right) = \lambda \cdot A \cdot \frac{d^2t\left(z\right)}{dz^2}dz \tag{1}$$

$$dQp = \frac{2\pi \cdot \lambda}{\ln \frac{R_G}{R_S}} \cdot \left[t_{RS}\left(z\right) - t_{RG}\left(z\right)\right]dz \tag{2}$$

The temperature drop across the test specimen can be determined from the following differential equation:

$$\frac{d^2t\left(z\right)}{dz^2} = \frac{2\pi}{A \cdot ln\frac{R_G}{R_S}} \cdot \left(a + b \cdot z\right) \tag{3}$$

where A is the metering area of the heater plate, R_S and R_G are the positions of the temperature sensors at the metering zone and the guard zone of the specimen, and $t_{RS}(z)$ and $t_{RG}(z)$ are linear functions determined with measurements. Based on the difference of these two functions, coefficients a and b can be calculated.

The points in the graph (Fig. 2) are temperature values measured within the specimen. Based on these measurement results, the temperature function ($t(z)$) and the

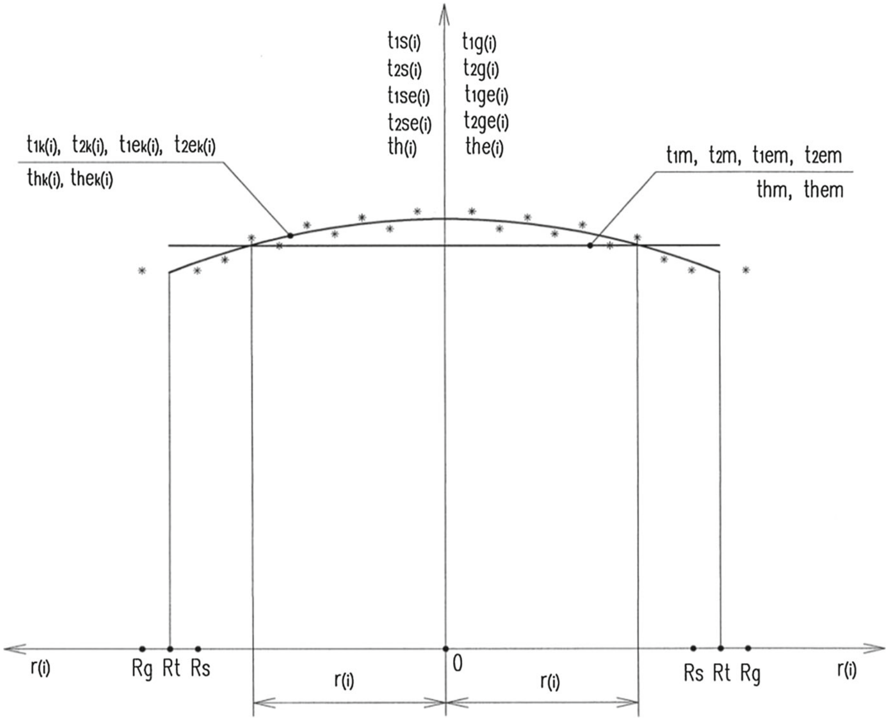


Fig. 2 Determination of temperature function within the rigid test specimen

mean value can be determined.

$$t(z) = a_0 + a_1 \cdot z + a_2 \cdot z^2 + a_3 \cdot z^3 \tag{4}$$

By integrating Eq. 3, the coefficients a_0, a_1, a_2 and a_3 can be determined.

The discrepancies in thermal conductivity measurements caused by this technique of placing the thermometers inside the specimen itself are less than 1 %.

With the HTTCMA, the imbalance error is measured directly, using two movable sensors placed in grooves at the upper and lower surface of the heater plate and applying extrapolation method (Fig. 3). In this way, the real temperature of the surfaces surrounding the gap is not perturbed.

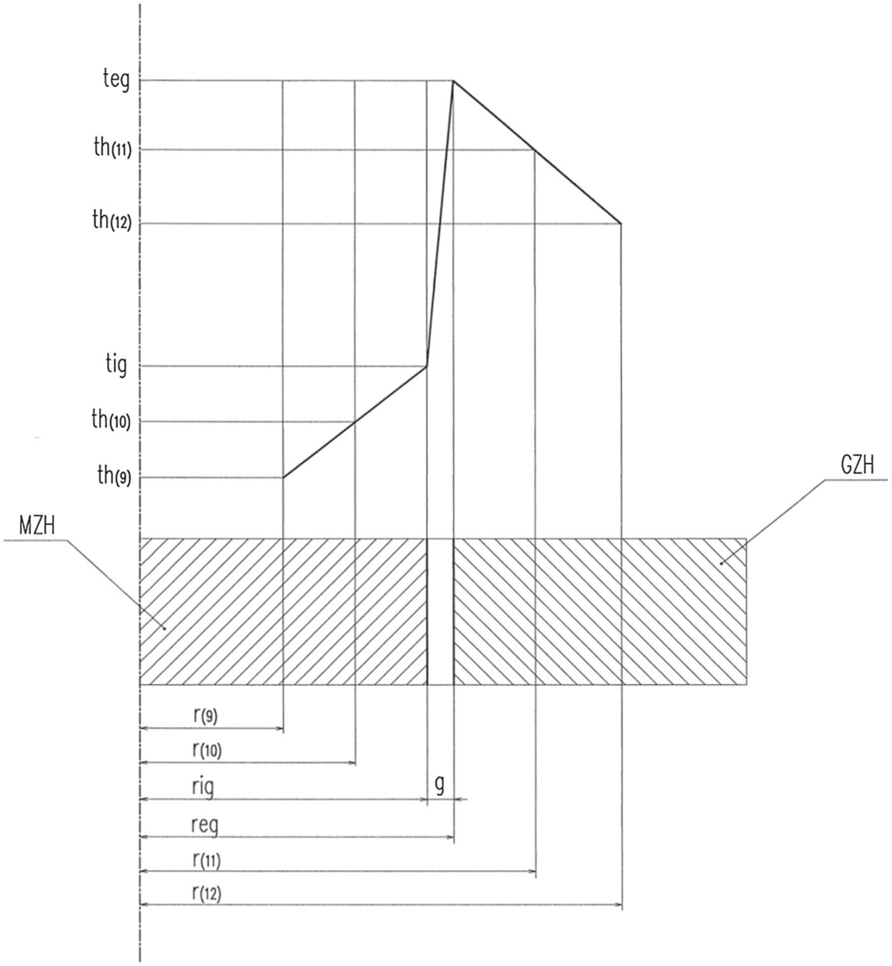


Fig. 3 Determination of guard-center gap imbalance

Figure 3 presents the gap (g) between the metering zone heater (MZH) and guard zone heater (GZH), and the extrapolated temperatures of its inner and outer surface (*tig* and *teg*). By measuring the temperatures $t_h(9)$ and $t_h(10)$ in the MZH, the temperature *tig* of the inner surface of the gap can be determined with extrapolation method. By measuring the temperatures $t_h(11)$ and $t_h(12)$ in the MZH, the temperature *teg* of the outer surface of the gap can be determined in the same manner.

It is a common practice in HTGHP to fill in the guard-center gap with opaque insulation, in order to reduce the radiant heat transfer across the gap. However, it is practically almost impossible to homogeneously fill in a guard-center gap of 2 mm in diameter. The unknown composition of a filled gap results in unknown characteristics and behavior. In some cases can be identified in the gap three types of material with unknown distribution: powder (e.g., alumina cement or calcium silicate), air and metal (due to thermopiles situated in the gap, used for measurements). An unfilled gap gives better measurement uncertainty, because the physical and thermophysical properties of the air and its behavior inside the gap are well known. For this reason, the guard-center gap in our HTTCMA is unfilled, resulting acceptable thermal resistance and lower uncertainty component.

2.2.3 Determination of the Thermal Conductivity of the Specimen

Considering the classical GHP method, at steady state the thermal conductivity, λ, of the specimens is given by

$$\lambda(t_m) = \frac{Q}{A(t_h)} \frac{d(t_m)}{t_h - t_c} \tag{5}$$

where Q is the power supplied to the central heater of heater plate, t_h is the mean temperature of the specimen hot face, t_C is the mean temperature of the specimen cold face, t_m is the mean specimen temperature, $A(t_h)$ is the metering area of the heater plate and $d(t_m)$ is the specimen thickness.

Applying the method used in case of the HTTCMA, at steady state the thermal conductivity (λ) of the specimens is given by the relation:

$$\lambda = -\frac{Q}{A \cdot \left(\frac{dt}{dz}\Big]_{z=0} + \frac{dte}{dz}\Big]_{z=0} \right)} \tag{6}$$

where A is the metering area of the heater plate, (dt/dz) and (dte/dz) are temperature derivatives of the upper and the lower specimens and Q is the electrical power (Pe_{MZH}) supplied to the heater plate, corrected to heat flow loss at the guard-center gap (Qg).

$$Q = Pe_{MZH} - Qg \tag{7}$$

Comparing Eqs. 5 and 6 can be seen that, in case of our method, the thermal conductivity λ does not depend on specimen thickness, on the mean temperature of the specimen and on the temperature of the specimen cold face, but it depends only on the derivative of temperature at the specimens hot faces ($z = 0$), determined by extrapolating the values measured inside the specimen. In this way, the thermal conductivity (λ) determination is not affected by the temperature drop Δt across the specimen.

The measurement method applied is suitable for quantifying the degree of divergence from unidirectionality of the HTTCMA. The heat loss at the lateral edge of a 50-mm-thick high-density calcium silicate (HDCaSi) specimen, in the wide temperature range from 70 °C to 800 °C is less than 5 % of the heat flow crossing the specimen.

In this case, the temperature drop across the specimen can be larger than 70 K as given in the standard CEN/TS 15548-1:2011 [2], because the temperature function inside the specimen is known. The advantage of the applied method is the higher accuracy of the thermal conductivity (λ) determination at high temperatures, due to the possibility of a larger temperature drop.

3 Experimental Validation of the Theoretical Model of Radiative Heat Transfer Between MZH and GZH of the In-House Built HTTCMA

From the basic difference between radiation and the convection and conduction energy-exchange mechanism, it is evident that the importance of radiation becomes intensified at high temperature levels [3].

A distinguishing feature of radiative transfer is that no medium needs to be present between two locations for radiant interchange to occur. The radiation exchange in an enclosure composed of diffuse-gray surfaces (in our case concentric cylinders) and in steady-state conditions can be expressed by the Stefan–Boltzmann law [4,5]:

$$Q = \sigma \cdot A_1 \cdot \frac{T_1^4 - T_2^4}{\frac{1}{\varepsilon_1} + \frac{A_1}{A_2}\left(\frac{1}{\varepsilon_2} - 1\right)} \tag{8}$$

where σ is the Stefan–Boltzmann constant, A_1 and A_2 are the areas of the covered and the cowering bodies, respectively, ε_1 and ε_2 are the emissivity of the two bodies and T_1 and T_2 are their absolute temperatures.

The total heat flow loss along the center-guard gap (Qg_t) is composed only from conductive and radiative components. Because of the small thickness of the gap, there is no convective heat flow along the gap.

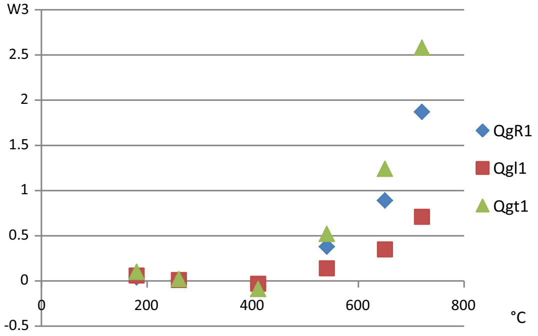
$$Qg_t = Qg_\lambda + Qg_R \tag{9}$$

The conductive heat flow across the gap can be written as:

$$Qg_\lambda = \lambda a \cdot Ag \cdot (tig - teg) / (Reg - Rig) \tag{10}$$

where λa is the thermal conductivity of the air, Ag is the area of the gap, (tig-teg) is the temperature drop across the gap and (Reg-Rig) is the wideness of the gap.

Fig. 4 Radiative contribution to center-guard imbalance



Considering the metering zone heater and the guard zone heater are from the same material, their emissivity is identical. Therefore, the radiative heat flow can be written as [5]:

$$Qg_R = \sigma \cdot Ag \cdot \varepsilon \cdot \left(Tig^4 - Teg^4\right) \tag{11}$$

where ε is the emissivity of the heater plate material and *Tig* and *Teg* are the absolute temperatures of the inner and outer surfaces of the gap.

In Fig. 4, an example is presented of radiative contribution to guard-center gap imbalance. *Qgl* is the heat transport by conduction, *QgR* is the heat transport by radiation, and *Qgt* is the total heat transport across the gap.

The experimental validation of the theoretical model shows that, in the temperature range from 150 °C to 800 °C, the radiative heat transport along the center-guard gap is less than 1 % of the electrical power applied. Taking this heat loss into consideration results in a low uncertainty component.

4 Determination of the Thermal Contact Resistance (TCR) Arising Between a Rigid Specimen and the Heated Plate

When a junction is formed by pressing two similar or dissimilar metallic materials together, only a small fraction of the nominal surface area is actually in contact because of the nonflatness and roughness of the contacting surfaces. If a heat flux is imposed across the junction, the uniform flow of heat is generally restricted to conduction through the contact spots. The limited number and size of the contact spots result in an actual contact area which is significantly smaller than the apparent contact area. This limited contact area causes a thermal contact resistance (TCR).

The TCR depends upon many geometric, thermal and mechanical parameters, of which the following are important [6]: geometry of the contacting solids (surface roughness, asperity slope and waviness), gap thickness, thermal conductivities of the contacting solids and the interstitial substance, hardness or yield pressure of the contacting asperities (which affects the plastic deformation of the highest peaks of the solid), modulus of elasticity of the contacting solids (which affects the elastic deformation of the wavy parts of the interface) and average temperature of the interface (which affects material physical properties).

In EMRP SIB52 project, rigid reference samples of high-density and low-density calcium silicates (HDCaSi and LDCaSi), used as high- quality thermal protection materials, suitable for this wide temperature range up to 800 °C, were characterized. The TCR of these silicate or ceramic-type rigid thermal protection materials dominate the TCR of the metal (Fig. 5) [7], because the TCR depends not only on the roughness and flatness of the contacting surfaces, but also on the difference in thermal conductivity of the contact spots of these surfaces.

Two different methods are available for determination of TCR (R), one based on the thickness of the air layer (d), while the other on the temperature differences (dt) of the contacting surfaces.

$$R = \frac{d}{\lambda} \tag{12}$$

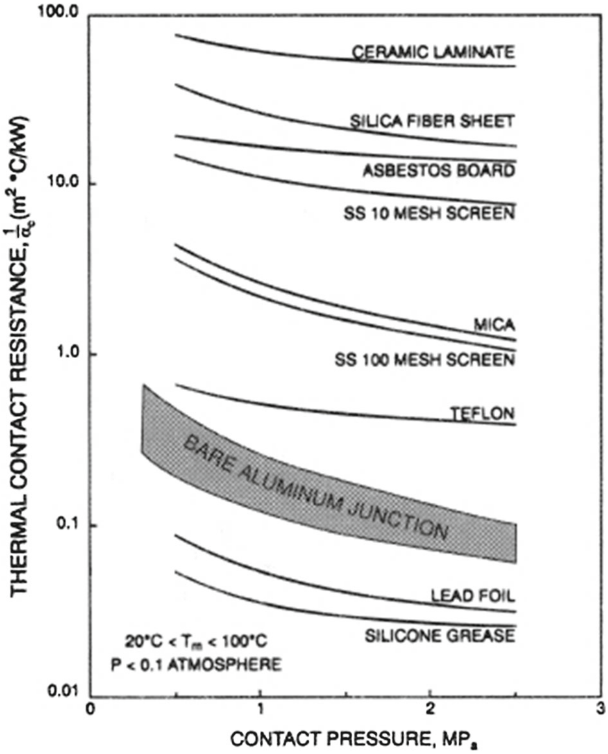


Fig. 5 Contact resistance for selected materials for thermal isolation

$$R = \frac{dt}{q} \tag{13}$$

where λ is the thermal conductivity of the air and q is density of heat flow rate which crossing this section. The thickness of the air layer is difficult to determine due to the fact that depends on many factors as contact pressure, roughness, flatness of the contacting surfaces. If it is possible to measure the temperature of both contacting surface, this method is the most easy and precise way to determine the magnitude of the TCR.

At MKEH, different experiments have been performed to evaluate the TCR, based on Eq. 13. The first experiment is presented in [8]. A setup was developed for modeling a GHP. The effect of the TCR on thermal conductivity of the specimen was experimentally evaluated, by comparing different arrangements of the temperature sensors. The effect of the emissivity was also studied.

In the second experiment, the HTTCMA was used for determination of the TCR, based on Eq. 14. In Fig. 6, HP is the hot plate, CP is the cold plate, S is the specimen, *thp* is the temperature of the HP surface, *tcp* is the temperature of the CP surface, *tis* is the temperature of the S inlet section, *tos* is the temperature of the S outlet section, q is the density of heat flow rate, *Ri* is the TCR at inlet section, and *Ro* is the TCR at outlet section.

$$t_{is} - t_{os} = t_{hp} - t_{cp} - (Ri + Ro) \cdot q \tag{14}$$

TCR given in the scientific literature [7] (Fig. 5): for metals is from 0.1 to 1 ($\mathrm{m}^2 \cdot {}^\circ\mathrm{C} \cdot \mathrm{kW}^{-1}$), for rigid isolation materials is from 10 to 100 ($\mathrm{m}^2 \cdot {}^\circ\mathrm{C} \cdot \mathrm{kW}^{-1}$). The TCR determined with the HTTCMA for the HDCaSi material is around 13 ($\mathrm{m}^2 \cdot {}^\circ\mathrm{C} \cdot \mathrm{kW}^{-1}$), which is in good agreement with the value given in the literature. The experimental results are within the measurement uncertainty [8] and show that the magnitude of TCR is significant in the whole temperature range (Fig. 7).

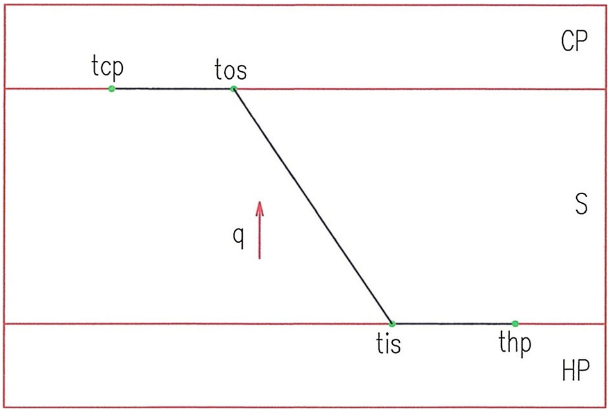


Fig. 6 Determination of the TCR with the HTTCMA

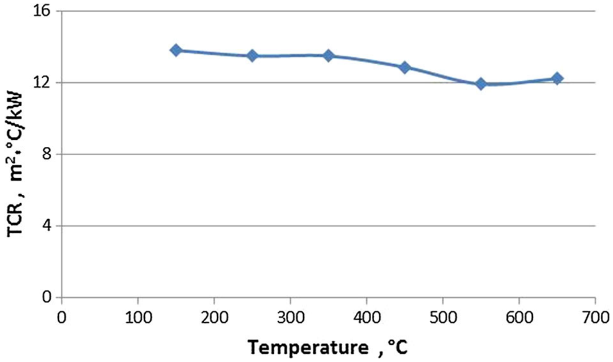


Fig. 7 Determination of the TCR with the HTTCMA

The technique for measuring high-temperature thermal conductivity, placing the thermometers inside the specimen itself, causes an error of less than 1 %. Considering the usual technique by placing the thermometers inside the heater plates and considering the high thermal contact resistance (TCR) arising between a rigid sample [3] (e.g., HDCaSi) and the heated plate of a high-temperature guarded hot plate (HTGHP), the discrepancies caused in thermal conductivity measurements can be at least 6 %.

The effect of a high TCR is not taken into consideration neither in the standards ISO 8302:1991(E) and CEN/TS 15548-1:2011, nor at the common measurement procedure of thermal conductivity of a rigid specimen by GHP.

5 Summary

An alternative calibration procedure has been developed using apparatus built in-house, to optimize thermal conductivity measurements. The new approach compared to those of usual measurement procedures of thermal conductivity by guarded hot plate (GHP) consists in modified design of the apparatus, modified position of the temperature sensors and new conception in calculating the thermal conductivity.

This paper has presented an overview of the specific characteristics of the newly developed HTTCMA, specially related to heater plates, guard-center gap, edge guards and position of the temperature sensors. Considering the significant thermal contact resistances of the thermal insulation materials characterized in EMRP SIB52 project, the paper has provided solutions how the major difficulties related to either the heat loss at lateral edge of the specimen or the effect of the thermal contact resistance between specimen and heater plate can be handled. A significant advantage of the measurement method developed is the suitability for quantifying the degree of divergence from unidirectionality of the HTTCMA, as well as accurate determination of the specimen temperature.

The knowledge from the outputs of the EMRP SIB52 project is required to improve measurement uncertainties, to investigate the anomalies between reference laboratories, and to finalize the new European measurement standard for thermal conductivity measurements of insulation at elevated temperatures by revising the actual CEN/TS 15548-1 standard.

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