THERMAL CONDUCTIVITY OF CALCIUM SILICATE BOARDS AT HIGH TEMPERATURES: AN EXPERIMENTAL APPROACH

CONDUTIVIDADE TÉRMICA DE PLACAS DE SILICATO DE CÁLCIO A TEMPERATURAS ELEVADAS: UMA ABORDAGEM EXPERIMENTAL

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ABSTRACT

Thermal conductivity analysis of fire insulation materials is of great importance for determining the critical temperature of structures. The magnitude of this thermal property has a significant influence on the analysis of temperature distribution and heat flow which depends essentially on the thermal properties of the protection material. Knowing accurate information about the effects of high temperatures on thermal conductivity is an important prerequisite for a performance based design of fire safety in buildings. Therefore, an investigation of two different calcium silicate boards has been performed to demonstrate how the thermal conductivity is affected when exposed to high temperatures. A set of experimental tests is presented. They were conducted in different techniques such as: the transient plane source (TPS) and the guarded hot plate (GHP).

RESUMO

Para desenvolver um projeto de engenharia de segurança contra incêndio é imprescindível conhecer os efeitos que as temperaturas elevadas originam nas propriedades térmicas dos materiais de proteção ao fogo. Esta informação é essencial para a aplicação dos métodos simplificados de cálculo. Assim, apresenta-se uma abordagem experimental para determinar a condutividade térmica de duas placas de silicato de cálcio distintas utilizadas como material de proteção passiva contra incêndio. São apresentadas duas metodologias e os seus resultados para a determinação das propriedades à temperatura ambiente e temperaturas elevadas: (i) regime estacionário (Guarded Hot Plate); (ii) regime transiente com o Transient Plane Source (HotDisk).

1. INTRODUCTION

Recently, several types of calcium silicate-based building material products have been developed for high-temperature insulation and fire resistive material applications. These fire protection materials are widely used to prevent the propagation of flames and to keep the lowest possible temperature in a fire situation, where the temperature can reach values between 700 and 1000 ºC. Such materials normally exhibit a high degree of thermal stability and when exposed to temperatures on the order of 1000 ºC some phenomena occur, such as the mass loss (10-15 %) and the shrinkage generally less than 2%, (Chi T. Do, 2007).

Numerous characteristics of calcium silicate are responsible for the high efficient thermal performance. A series of endothermic reactions occurs at different temperature intervals, among them, a dehydration process, which happens in the temperature range between 100 and 250 ºC. During this thermophysical phenomenon, most of the energy released by the fire is destined to evaporate the water molecules (≈5%) that are contained in the crystalline structure, resulting in an increasing of thermal resistance of this material, (Kolaitis & Founti, 2013; Silva, 2016).

However, there is a huge difficulty in fully obtaining of the benefits which this method is able to provide, owing to the lack of information about the thermal conductivity variation of protective materials when they are exposed to high temperatures, (Wang, Burgess, Wald, & Gillie, 2012).

Knowledge of the thermal transport properties of building materials is greatly important considering the ideal air conditioning inside houses over the range of cold and hot climates. Furthermore, there is the main influence on the temperature development of compartments fire. Beyond that, the insulation material is the only material among still and the fire and consequently it is the principal protection of still. It is well known that when this metal is exposed to high temperatures, the mechanical properties decrease substantially (≈ 40%), this fact evinces the necessity of suitable use of thermal conductivity,

The simplified method contained in Eurocode 3, part 1.2 (CEN 2005b) is widely used to design security building structure, by determining how the temperature develops in a structure surrounded by a protection material. The model consists of a simplified resolution of the differential equation of heat conduction and currently, the method considers that the thermal properties of the protective materials remain constant regardless the temperature ranges and as a result it does not represent a real fire situation, (Mesquita, Piloto, Vaz, Vila Real, & Ramos, 2005; Wang et al., 2012). According to (CEN, 2005), the increases of temperature of the protect steel are given by the equation (1).

$$\Delta T_s = \frac{\frac{K_p A_p}{V(T_g - T_s)}}{c_s \rho_s d_p \left(1 + \frac{\phi}{3}\right)} \Delta t - \left(e^{\frac{\phi}{10}} - 1\right) \Delta T_g \quad ; \qquad \Delta T_s \geq 0 \geq \Delta T_g \tag{1}$$

where, ΔT_s represents the temperature evolution, based on normalized fire curves, K is the thermal conductivity, A is the area, T_g and T_s represents the instantaneous temperature of the gases and steel, respectively, and $\phi = c_p \rho_p d_p / c_s \rho_s \left(\frac{V}{A_p}\right)$.

Along the years, several European laboratories and researchers have developed experimental tests using different techniques, such as guarded hot plate, hot-wire and the transient plane source in order to characterize the thermal conductivity of calcium silicate boards as a temperature function. According to Salmon, due to the divergences and uncertain results obtained, the values found could not be certified,(Salmon, 2001). Recently, Dale develop a research to verify the thermal conductivity variation as a temperature function, considering the specific mass and porosity of the material. The tests were conducted by the transient plane source technique (TPS). The author also implemented the Russell´s theoretical model and previously results from other surveys for comparison purposes,(Chi T. Do, 2007).

From this perspective, the elaboration of this paper consists in characterizing the thermal conductivity as a function of temperature, by comparing two experimental techniques.

2. THE TRANSIENT PLANE SOURCE THEORY

Thermal techniques are typically classified under steady state and transient methods. The hot-disk is an equipment based on Transient Plane Source theory, which was first developed by Gustafsson. This technique is able to measure thermal properties as a function of temperature, which has been the research focus of several researchers, (Gustafsson, 1991). Gustafsson applied this technique to determine thermal properties of cecorite 130P, a ceramic material based on cordierite. Almanza characterized several low density polyethylene foams through TPS and Rapid K, (Almanza, Rodríguez-Pérez, & De Saja, 2004), that there are also reports on construction materials approached by (Log & Gustafsson, 1995). The materials covered in this work were: extrude polystyrene, PMMA, cecorite 130P, stainless steel and aluminium.

Among the various advantages that this device presents comparing with other methods, it is worth highlighting: the time required to perform the measurements is shorter, the values of thermal conductivity and thermal diffusivity are reported simultaneously in the results. Furthermore, this technique encompasses a high measurement range that can be applicable to measure the thermal conductivity of materials ranging from 0,02-400 [W/m^{2}K], (He, 2005; Solórzano et al., 2008) . The Fig 1 illustrates a schematic diagram of the components engaged in the hot-disk assembly.

This equipment uses a sensor that takes the measurements and at the same time produces the necessary energy to increase the sample's temperature. The sensor has a standard electronic system engraved in a thin layer of metal, and the surfaces are covered by an insulating material such as Kapton or Mica, (He, 2005; Patrick T Summers, 2015).

At the beginning, an electric current flow through the sensor which is large enough to raise the sample's temperature around 1-2K. As a result, the sensor resistance is modified

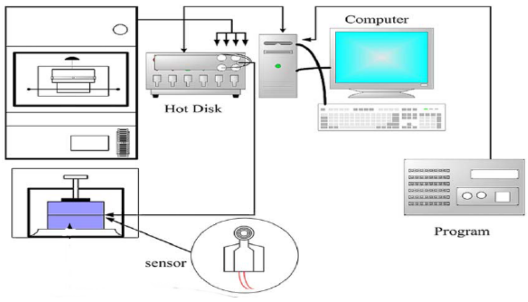

Fig 1 - Basic layout of the apparatus, (Al-Ajlan, 2006).

and then, a corresponding voltage drops on the sensor. During the recordings of these parameters, for a certain period of time, which are counted from the beginning of the process it is possible to obtain accurate information about the heat flow, (Log & Gustafsson, 1995). This method was recognized and standardized by ISO-22007-2, which recommends through the

Table 1 the initial experimental parameters for different types of materials.

Thus, it is convenient to express the variation of the resistance measured by the sensor as a function of the temperature increase, as shown in equation (2).

$$R(t) = R_0\big(1 + ¥\Delta T_{(\tau)}\big) \qquad (2)$$

where R_0 is the initial resistance of nickel, ¥ is the nickel temperature coefficient and $\Delta T_{(\tau)}$ is the temperature variation measured by the sensor expressed in terms of an only variable τ, defined as:

$$\tau = (t/\theta)^{\frac{1}{2}} \; with \; \theta = a^2/\alpha \qquad (3)$$

Table 1 - Recommendation for initial experimental parameters, (ISO, 2008).

	Metal alloy	Dense ceramic	Steel	Ceramic	Polymer	Insulation material
Thermal conductivity [W/mK]	170	40	14	1,5	0,19	0,028
Thermal diffusivity [mm^2/s]	69	11	3,7	0,96	0,11	0,75
Temperature increase [K]	0,3	0,5	1	0,8	1,3	2,5
Probe radius [mm]	15	6,4	6,4	6,4	6,4	15
Specimen thickness [mm]	30	10	10	10	15	30
Specimen diameter [mm]	90	40	40	40	40	90
Measurement time [s]	5	10	10	40	160	160
Power output [W]	4	3	2	0,5	0,25	0,1

$$\Delta T(y,z,\tau) == \left(4\pi^{3/2}\, aK\right)^{-1} \int_0^{\tau} \frac{d\sigma}{\sigma^2} * \int_A dy`dz` * Q\left(y`, z`, t`\right) \exp\left\{-\frac{\left(\left(y-y`\right)^2 + \left(z-z`\right)^2\right)}{4\sigma^2 a^2}\right\} \tag{4}$$

$$D(\tau) = \left(m(m+1)\right)^{-2}. \int_0^{\tau} \sigma^2 \left\{\sum_{l=1}^{m} l \sum_{k=1}^{m} k \exp\left(-\frac{(l^2+k^2)}{4m^2\sigma^2}\right) L_0\left(\frac{lk}{2m^2\sigma^2}\right)\right\} d\sigma \tag{5}$$

where t is the measurement time from the start of the transient heating, θ is the characteristic time, which depends on the parameters of the sensor and also the sample, a is the hot-disk radius and finally α is the thermal diffusivity of the sample.

The theoretical expression capable of accurately determining the values of thermal properties is obtained through the resolution of the thermal conduction problem specifically for the TPS element. According to Gustafsson, in most cases it is possible to express the raise of temperature in terms of τ , rearranging the terms regarding this variable the temperature increment at the can be calculated by (4) (Gustafsson, 1991).

The temperature variation can also be related with the power released by the sensor. However, there is a mathematical expression for each geometry assumed by the sensor, square or circular. The equation (6) can be used for this purpose for the disk shape sensor. The expression for the square sensor is available in (Gustafsson, 1991).

$$\Delta T(\tau) = P_0 \left(\pi^{\frac{3}{2}} ak\right)^{-1} D(\tau) \tag{6}$$

where P_0 is the total output power and $D(\tau)$ is a geometric function given by (5), in which, L_0 is the modified Bessel function available in (He, 2005).

The probing depth measures how far into the specimen, in the direction of heat flow, the heat wave has travelled during the total time calculation. Therefore, the test specimen thickness must be larger or equal to the probing depth. In addition, the probing depth is always close or slightly less than the sensor diameter, see (7), (Log & Gustafsson, 1995).

$$\Delta_{prob} = 2\sqrt{\alpha t_{max}}\,, \qquad 1{,}1a < \Delta_{prob} < 2a \tag{7}$$

One of the main advantages of transient techniques over the steady state technique is that the effects of the contact resistance can be removed after the software calculation in the analysis of the experimental data. This enables the achievement of a very accurate data collection of thermal properties for a wide range of materials.

Thermal conductivity measurements were produced in pairs of insulation materials using a hotdisk thermal analyser. Regarding the hotdisk, the cut of the samples was manufactured in a square form with each size measuring 75 mm due to the limited dimensions of the furnace holder. The experiments were conducted in the equipment hotdisk 2500S and a furnace in which allows the tests realization according to the international standard ISO 22007-2. Fig. 2 represents the main equipment employed to perform the analyses.

Fig. 2 - Main devices employed. (a) hotdisk equipment e (b) furnace.

It is important to emphasize that this method requires a perfect contact between the sensor and the surface of the two specimens in order to ensure that there is no external interference in the measurement, so it is necessary that both samples be of the same material with similar dimensions, (Almanza et al., 2004). For the tests conducted under room temperature the sensor encased in Kapton was selected, whereas, for measurements obtained through the furnace the Mika sensor was employed because it is more appropriate for high temperatures.

Although the calcium silicate may have a non-homogeneous microscopic structure, in this paper, it was assumed that the physical properties are independent of the direction considered, thus, the measurements were obtained through isotropic materials configuration. For each test performed, the calcium silicate samples were used in pairs with the sensor positioned in the centre. The tests were conducted towards 12 sets of samples, in which individual readings were collected for each input parameter and desired temperature. Fig. 3 and Fig. 4 illustrates the experimental arrangement and the software inputs to perform the measurements.

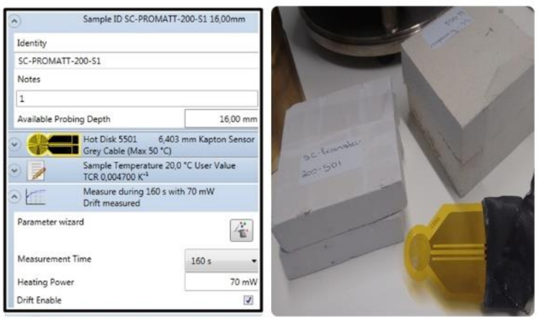

Fig. 3 - Configuration for room temperature.

Fig. 4 - Configuration for high temperatures.

3. THE GUARDED HOT PLATE

Although techniques based on the transient regime have grown substantially in recent years, in Europe, the guarded hot plate remains the most widely used equipment to determine the thermal conductivity of insulations materials. In principle, the operation of this device consists of establishing a steady temperature gradient over a known thickness of the specimen by controlling the heat flow from the hot plate to the cold plate. Normally the steady techniques require a long time to achieve the stationary state and it is regarded the most disadvantage comparing with the transient techniques, (Al-Ajlan, 2006; Hammerschmidt, 2002).

In this system the temperature at each point is independent of time, in addition, the heat flow is transferred by diffusion in only one direction. Thus, for the longitudinal heat flow without loss of radial energy, nor generation of energy inside the solid, the Fourier's law can be applied to determine the thermal conductivity, see (8).

$$K = \frac{Ql}{A(T_2 - T_{1)}} \tag{8}$$

where, T_1 and T_2 represent the temperatures of the cold and hot sides of the sample, respectively, Q is the output energy of the equipment, A and l denote the area and the thickness of the sample and finally K is the thermal conductivity.

This technique is widely applied to characterize the fire-resistant materials, in which the value of thermal conductivity must be within the range of 0,01- 6 [W/mk], in addition, the samples may be exposed to extreme temperatures conditions, ranging from the melting point of the nitrogen (~78 [K]) to the melting point of the steel (~1810) depending on the type and restrictions of the equipment. A simple representation of the apparatus is provided by Fig. 5.

This machine consists of various components such as: the solid sample (A) with the cross-section area is positioned inside an appropriate holder and placed between the upper electrical hot plate (B) and the lower thermostated cold plate (C).

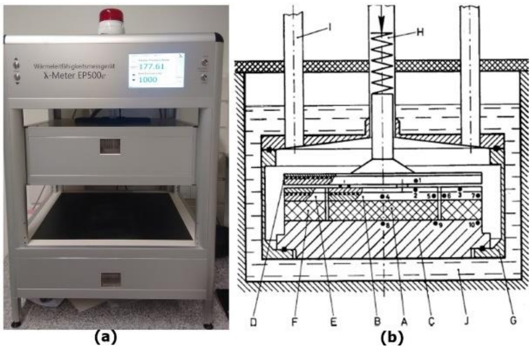

Fig. 5 - The guarded hot plate, (a) the equipment in use, (b) the scheme of the basic components.

The hot plate dissipates a constant electric power $P = UI$, this input power flows to the cold plate through the sample as homogeneously as possible. Thus, the known heat flow leads to a variation of the sample temperature in which allows the measurement of the thermal conductivity. Two guard heaters, the guard plate (D) and the guard ring (E) that surround the hot plate are responsible to establish a unidirectional and uniform heat flow. A push rod (H) can be adjusted from outside to ensure that the stack remains tightly packed. The working temperature is set by several thermostat (J) which control the temperature measuring points of the apparatus.

To measure the thermal conductivity of insulation materials by using the guarded hot plate technique it is recommended to use a sample area of 500 $[mm^2]$, however, there are 3 types of support to apply in case of using smaller dimensions: version A (200x200[mm]), version B (250x250[mm]) and version C (150x150[mm]). Once this support is being used, it is important to guarantee that the thickness of the holder be slightly thicker than the specimen. For this paper the holder applied was type C. Moreover, for materials with insulation characteristics it is not recommended the use of compensation panels with temperatures sensors, the sample surface must be flat and smooth to ensure the suitable contact between the hot plate and the sample. Additionally, the maximum pressure for insulation material is 1000 $[Pa]$. Basically, the operation of this machine involves the following 4 steps to execute the tests as described in Fig. 6.

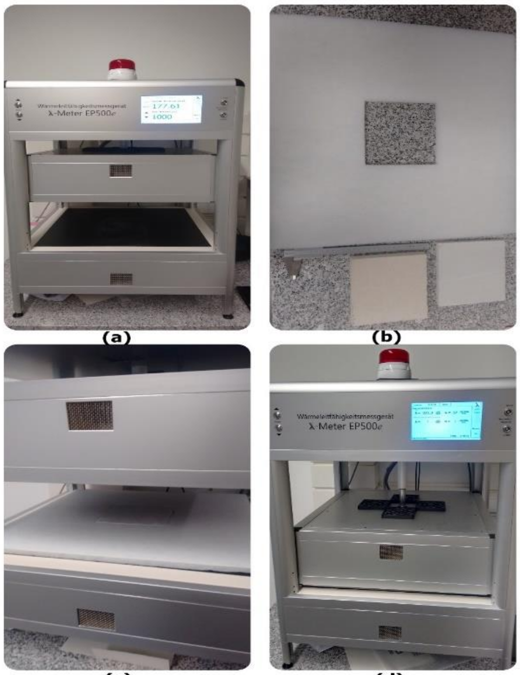

Fig. 6 - Basis steps to perform the tests. (a) calibration of the machine, (b) and (c) preparation and insertion of the holder and sample inside the equipment, (d) application of the properly pressure to start the test.

Initially, the first task to be made is the calibration of the equipment in the zero coordinate, this step must be done without the holder and the sample as shown in (a). Then, it is necessary to take measurements of the mass, area and thickness of the sample (b), because these values are required in the software interface. The stage (c) consist of the adjustment of the holder in the properly position, it is worth mentioning that, before going forward and initialize the test it is crucial make the verification whether the holder's thickness is slightly thicker than the specimen in order to obtain the fitting pressure, otherwise, there are several thickness holders available which can be used as wedge to achieve the acceptable region.

5 - RESULTS AND DISCUSSION

Two types of calcium silicate boards, Promatec_H based on fire protective construction boards with cement binder and Promatec_200 light with promaxon binder and resistant to moisture, were tested under controlled thermal conditions in order to verify the behaviour of thermal conductivity of insulation materials at high temperatures.

The samples were provided by their manufacturer in a size of 2500 mm x 1200 mm by 15 mm for Promatec_200 and 3000 mm x 1250 mm by 20 mm for Promatec_H. The testing samples were cut and prepared from the original dimensions in function of the test. The Table 2 provides some physical information about these materials.

The

Table 3 and Table 4 summarizes the values of measured thermal properties of calcium silicate boards provided by hotdisk under ambient conditions. According to these results, the hotdisk error data indicates a standard deviation $< 0{,}1$ for all thermal properties as shown in the tables below. The oscillation of these results for each specimen are well represented in Fig. 7.

Regarding the guarded hot plate, the range of temperature was not so significant owning to the limitations of the equipment. By using this technique, thermal conductivity measurements were performed for a set of 8 specimens of calcium silicate boards, the samples were prepared nominally with 150 mm by 150 mm due to the size of the holder. Then, the specimens were submitted in a range of temperature of 10 ºC, 23 ºC and 50 ºC to investigate the behaviour of the thermal conductivity. As it was expected, the results provided in Fig 8 shows an evidence that, as the temperature rise the value of thermal conductivity increases.

Table 2 - Sample properties provided by the manufacturer.

Material	Moisture Content (%)	ρ (kg/m^3)	K (W/mK)
Pramatec H	5-10	870	0,175
Promatec 200	1-2	835	0,189

Table 3 - Thermal properties under room temperature of PROMAT-H provided by hotdisk.

Sample ID	K (W/mK)	α (mm^2/s)	Cp (kJ/kgK)
SC-P-H-S1	0,229	0,416	0,632
SC-P-H-S2	0,213	0,426	0,574
SC-P-H-S3	0,232	0,360	0,743
SC-P-H-S4	0,218	0,393	0,637
SC-P-H-S5	0,227	0,368	0,710
SC-P-H-S6	0,233	0,370	0,725
SC-P-H-S7	0,222	0,419	0,608
SC-P-H-S8	0,232	0,474	0,562
SC-P-H-S9	0,239	0,395	0,696
SC-P-H-S10	0,254	0,394	0,741
SC-P-H-S11	0,261	0,412	0,728
SC-P-H-S12	0,249	0,389	0,736
Average	0,234	0,401	0,674
Standard dev.	0,014	0,030	0,065

Table 4 - Thermal properties under room temperature of PROMAT-200 provided by hotdisk.

Sample ID	K (W/mK)	α (mm^2/s)	Cp (kJ/kgK)
SC-P-200-S1	0,266	0,349	0,878
SC-P-200-S2	0,275	0,539	0,586
SC-P-200-S3	0,251	0,336	0,858
SC-P-200-S4	0,248	0,320	0,888
SC-P-200-S5	0,248	0,318	0,897
SC-P-200-S6	0,261	0,344	0,870
SC-P-200-S7	0,263	0,345	0,874
SC-P-200-S8	0,273	0,347	0,904
SC-P-200-S9	0,255	0,346	0,849
SC-P-200-S10	0,262	0,399	0,754
SC-P-200-S11	0,260	0,347	0,863
SC-P-200-S12	0,259	0,517	0,576
Average	0,260	0,376	0,816
Standard dev.	0,008	0,071	0,111

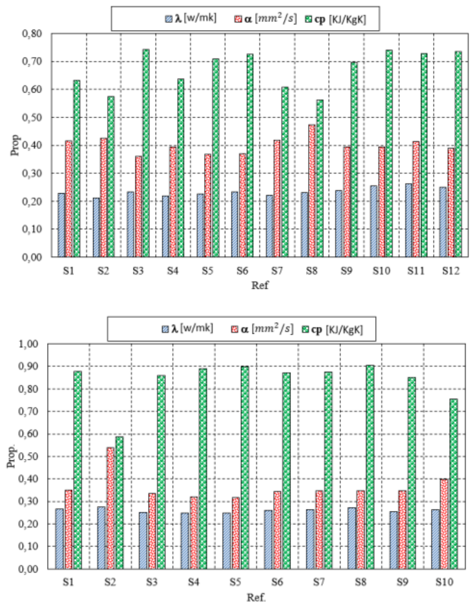

Fig. 7 - Thermal properties of calcium silicate promat H (top) and Promatec 200 (bot.) under ambient conditions.

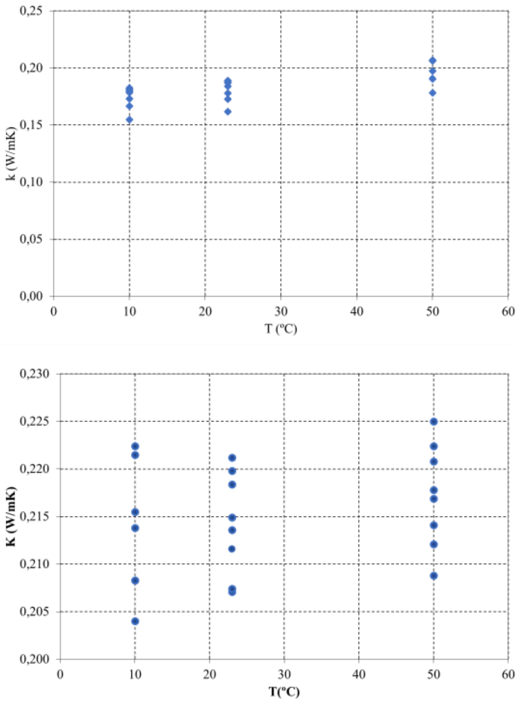

Fig 8 - Average measured thermal conductivity for calcium silicate promatec-H (top) and promatec-200 (bot.).

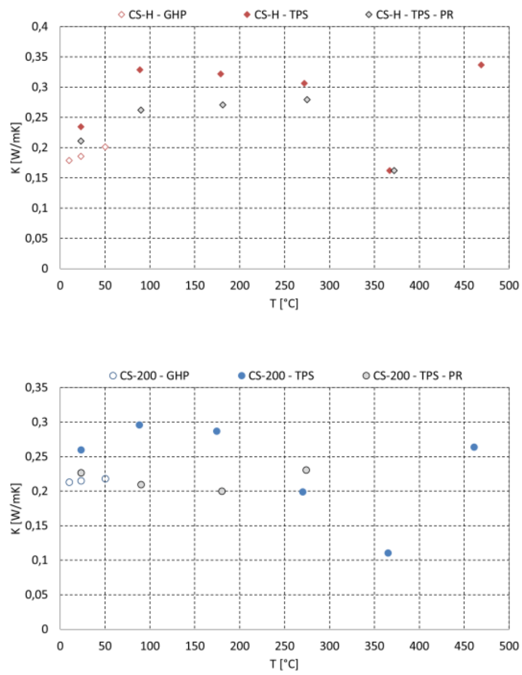

Fig 9 - Average measured thermal conductivity for calcium silicate promatec-H (top) and promatec-200 (bot.).

The hotdisk and a furnace were used simultaneously to run a cycle of tests at the following temperatures: room temperature (23ºC), 100 ºC, 200 ºC, 300 ºC, 400 ºC and 500 ºC. At each measurement temperature four readings were collected. According to the international standard, the input parameters for insulation materials must be low in order to prevent damages to the sensor due to overheating. Therefore, the sensor was calibrated to release 0,07 [W] for a measurements time period of 160 [s]. To avoid the sensor and furnace damage all high temperature tests were done using a nitrogen environment.

The following figure presents these results and their comparison with the measured values at ambient temperature, from the TPS and GHP methods. Also a second cycle for each material was done after being submitted to elevated temperature, and presented in the graphs with the index "PR".

From the figures it can be verified the thermal conductivity variation with temperature and a significate decrease between 350 and 400 [ºc], for both materials, certainly due to chemical reactions in this temperature range.

This behaviour must be reanalysed performing more experimental tests in an oxidative atmosphere.

5. CONCLUSIONS

Transient plane source and guarded hot plate technique were used in the present work to verify the behaviour of thermal conductivity of two insulation materials.

The results of the two methods, guarded hot plate and transient plane source were compared with the aim to analyse the reliability and to determine which one provides better results to measure insulation and highly porous materials.

The GHP technique presents greater viability in the efficiency of the measurement, since the sensors used in the TPS have high cost and limited lifespan, this fact bounds the amount of tests.

The thermal conductivity of calcium silicate obtained by both technique under ambient conditions is very similar. The thermal conductivity of calcium silicate boards was observed to be a function of temperature and it increases with increasing temperatures The elevated temperature results show that the thermal conductivity vary with temperature in a non-linear way.

The influence of porosity on thermal conductivity was also analysed as a result of the dehydration. Whilst the evaporation process takes place, there is a significant increase in the quantity of pores in the material. Thus, according to the results it is possible to assume that, the larger the pore quantity the smaller is the effective thermal conductivity of the materials.

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