HEAT TRANSFER STUDY IN A RIG OF THERMAL CONDUCTIVITY MEASUREMENT BASED ON ASTM E1225 STANDARD

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Abstract. This paper presents a methodology for the identification of the working range of a thermal conductivity rig for solid materials based on the ASTM E1225 standard. The Guarded-Comparative-Longitudinal Heat Flux Technique is applied but without the prescribed heat guard around the testing stack. A numerical approach is proposed due to its capability of exploring unknown values more easily and at lower costs. The proposed methodology consists on the comparison of pair of results for samples of same thermal conductivities but submitted to different insulation conditions for the stack assembling of the rig. Results show that the bench is suitable for essays of samples with thermal conductivities of 10 W/(mK) and beyond.

Keywords: Guarded-comparative-longitudinal heat flux technique, thermal conductivity, ASTM E1225 standard, CFX

1. INTRODUCTION

The aim of this work is to verify the working range of an experimental bench for the measurement of the thermal conductivity of solid materials. This study continues previous assembling and developments of the Guarded-comparative-longitudinal heat flux technique performed by Müller (2007), based on the ASTM - American Society of Testing Materials - standard E1225. This measurement apparatus was design and built without the heated guard around the sampling section, prescribed by the standard. That guard aims to reduce the experimental errors by avoiding heat flux to escape throughout the surfaces of the rig. Although the measurement device is out of recommendation, it can still deliver results at a determined range, to be discovered in the present work.

That technique is a simplified version of the guarded hot box technique, but with bigger uncertainties and less assembly and operation cost. Two possible methodological paths could be followed: by experimental trials, using different reference samples with its thermal properties defined by a well know laboratory; or by numerical simulation, playing a similar role of the former proposal but with some operational advantages. The numerical simulation methodology was finally chosen because of its enormous research potential. It becomes free to explore several values of materials and thermal conductivity values and the computational cost to run the simulations is lower than the one to provide reference samples and to perform the range of real experiments on the rig.

The desired output of this work is to identify the range of thermal conductivity that can be measured by the apparatus within a specific uncertainty.

2. THERMAL CONDUCTIVITY MEASUREMENT

The thermal conductivity k in W/(mK), is the ability to transfer heat, which is defined by Eq. (1), where q is the heat transfer rate in the x direction, in W; A is the area normal to direction of heat flux, in m^2 ; dT/dx is the temperature gradient in the x direction, in K/m, and k is the thermal conductivity, in W/(mK). The thermal conductivity is a property of a conducting medium and, like the viscosity, is primarily a function of temperature (Welty, 2001).

$$q_x = -kA \frac{dT}{dx} \tag{1}$$

There are several ways to measure the thermal conductivity k of a solid material. The most common methods for determining k are: guarded hot box; laser flash; hot wire; and guarded-comparative-longitudinal heat flux technique.

The Guarded hot plate consists basically of a hot plate and a cold plate. The hot plate is a central or metering heater plate surrounded by an outer or guard heater plate with a small air gap in between. Opposite to both the heater and the specimen is placed a metal plate known as cold plate, which maintains a temperature below that of the hot plate by circulating a fluid. The function of the metering heater is to dissipate a controlled amount of heat flux in order to maintain a desired temperature gradient across the central measuring area of the specimen. The guard heater maintains

that temperature close to the metering area to reduce lateral heat flux from the metering section of heater plate and specimen (Xamán, 2008).

In the flash method the front side of the sample surface is subjected to a very short burst of radiant thermal energy. The short measurement time in this method minimizes the measurement error due to the reduction of heat losses from the sample to the surroundings. It provides both excellent accuracy and reproducibility. The resulting temperature rise on the rear side of the sample surface is measured, and thermal diffusivity can be determined by comparing the measured temperature rise with the appropriate mathematical model (Parker, 1967).

The hot wire technique is relatively simple and fast as it is based on the transient measurement of the temperature rise of a uniformly heated wire. Moreover, the measurement could be made on samples with any shapes and relatively small sizes. Applying a constant electric current through the wire, a constant amount of heat per unit time and unit length is released by the wire and propagates throughout the material (Santos, 2007).

2.1 The standard test method for measuring the thermal conductivity of solids by means of the Guarded-Comparative-Longitudinal Heat Flux Technique

This standard describes a steady state technique for the determination of the thermal conductivity of homogeneous-opaque solids. This test method is for materials with effective conductivities in the approximate range 0.2 < k < 200.0 W/(mK) over the approximate temperature range of 90 to 1300 K. It can be used outside these limits with decreased accuracy.

A test specimen, whose thermal conductivity is unknown, is inserted under load between two other reference materials, with the same cross section, and known thermal properties. This assembling will be called hereon by test stack. A temperature difference is imposed along this test stack and, at equilibrium conditions (steady state), the thermal conductivity of the sample is derived in terms of the temperature differences in the respective specimens and the thermal conductivity of the reference materials. Figure 1 below is a schematic draw of the equipment, where the meter bars are made of reference materials, with known thermal conductivities.

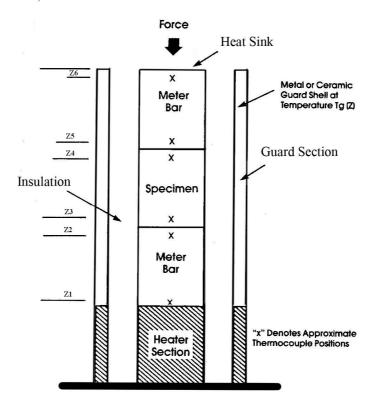


Figure 1- Schematic of a comparative-guarded-longitudinal heat flux system showing possible locations of temperature sensors along the stack

The standard strongly recommends the use of a guard that involves both the reference materials and the sample. The guard has the task of mitigate heat losses through the side of the equipment and, by doing so, imposing a one dimensional thermal gradient through the longitudinal axe, similar to the gradient in the stack.

With the values of temperature obtained by measurements, one can now calculate the heat flux rate per unit area q'' for each of the two reference materials, through the equations below:

$$q_{\text{upper}}'' = k_{\text{RM1}} \frac{T_6 - T_5}{Z_6 - Z_5} \tag{2}$$

$$q_{\text{lower}}'' = k_{\text{RM2}} \frac{T_2 - T_1}{Z_2 - Z_1} \tag{3}$$

The thermal conductivity of each one of the reference materials is calculated using the average temperatures. Although these two values q'' upper and q'' lower should agree with each other to within about $\pm 10\%$ when the heat exchange with the isolation is small. A value of thermal conductivity for the specimen at temperature (T3 + T4) / 2 is given by the equation below, which assumes no heat loss to the isolation.

$$k_{\text{sample}} = \frac{\left(q_{\text{lower}}^{"} + q_{\text{upper}}^{"}\right) \times \left(Z_4 - Z_3\right)}{2 \times (T_4 - T_3)} \tag{4}$$

This procedure of calculus requires only two thermocouples for each one of the reference materials in the stack. A third temperature sensor can be used to test the uniformity of the other two sensors as well as the data acquisition system.

2.2 Bench description

In the actual bench, assembled at UFRGS (Ceramic Materials Laboratory – LACER) the heater positions were inverted among then. The reference material close to the heat source, hereon called material I, was placed in the bottom of the stack and made of stainless steel, as it is an appropriate material for high temperature duties. Its thermal conductivity varies according to the temperature, defined by the equation (ASTM E1225)

$$k = 1.22T^{(0.432)} [K/(mK)]$$
 (5)

The second reference bar (material II), now placed at the top of the stack is made of aluminum, displaying a constant k of 138W/(mK). The lateral insulation is made of mineral wool, with an approximate thermal conductivity of 0.1 W/(mK).

The sample, the stainless steel and aluminum bodies have all the same cross section of 70 x 70mm and their heights are respectively 17.0, 37.5 and 82.5mm. The insulation cross section is 210 x 210mm.

Type K thermocouples, of 1.5mm diameter are used to measure the temperatures, which hot junctions are exactly in the center position of the column test. The minimum number of thermocouples by region is two and they are placed 2.5mm away from its bottom and top surfaces. An actual picture of this assembling is shown in Fig. 2.

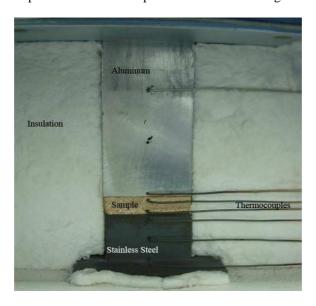


Figure 2- Thermocouples assembling on the actual rig

3. METHODOLOGY

The numerical approach was adopted here to find the working range of the thermal conductivity apparatus. The methodology is based on the generation of simulated data for a reference ideal situation where the insulation material around the stack is so efficient that it replaces the thermal guard. Data calculated for that situation are compared to those simulated with thermal losses to the insulated environment that surrounds the stack. That last configuration tries to represent the actual bench.

Temperatures at the positions where the thermocouples are placed are used in the equations provided by the standard to calculate the heat flux through the stainless steel meter bar (Eq. (2)) and in the aluminum meter bar Eq. (3). These results lead to the calculus of the estimated sample conductivity (Eq. (4)). This routine is performed for every value of sample thermal conductivities.

3.1 Implementation

The geometry and the mesh setup were performed by the pre-processing software ICEM (http://www.ansys.com). The simulation was done with CFX, which is composed by three modules or codes: pre-processing (CFX-pre), for the boundary conditions input, the solver itself, and the post-processing, which is held to analyze the outputs.

An initial tetrahedral mesh was first chosen for the simulations as it was of simple application. Prisms have been added to the bottom region, and mesh was refined near the sample body, ending up with approximately 250,000 elements and 45,000 nods.

The mesh was exported to CFX-pre. The boundary conditions defined to the adiabatic reference case were: prescribed temperature of 932° C on the heat flux input (bottom); general interface on the surfaces between each solid materials; a $800 \text{ W/(m}^2\text{K})$ convective heat transfer coefficient on the surface facing the top of the stack and a adiabatic condition for all side walls of the sample and the reference materials.

After 100 iterations of the solver it was already possible to obverse that the convergence had reached E-5 and was not going bellow this value.

In the post-processing, as the boundary condition of the side walls were fixed as adiabatic, one could look forward to find a flat line of constant temperature. Different to what was expected it was found a non-linear behavior of that temperature. Besides that, the heat flux input was not of the same magnitude of the output, indicating a non conservative energy balance.

A change from tetrahedral to hexahedral elements was applied. This mesh demands a greater implementation effort, however the number of elements is much smaller, its computational time decreases and the criteria of convergence drops drastically. Next figure displays these differences.

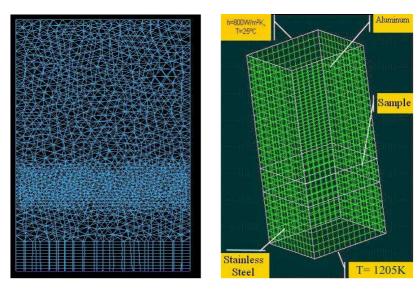


Figure 2- Geometry with tetrahedral elements (left) and with hexahedral elements (right).

The convergence achieved with the new mesh was around E-10, half of the value achieved by the original one. Mesh size dropped down from to 250,000 elements and 45,000 nods (tetrahedral elements) to 6,000 elements and 7,000 nods (hexahedral mesh). Heat flux for each surface of the solids was calculated and there were no more unbalance values for the hole set of materials (stainless steel, aluminum and the sample).

Figure 3 shows the expected flat temperature distribution when adiabatic side walls boundary condition is imposed.

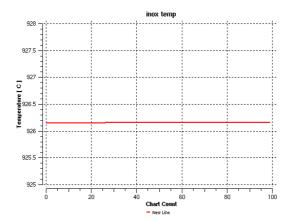


Figure 3- Temperature profile along the longitudinal direction of the stainless steel meter bar for the adiabatic condition along the boundaries

In order to reproduce the actual behavior of heat losses from the stack to the surrounding environment, the simulated volume included both the stack itself and the insulation material, living the boundaries of the problem to be coincident to the outside surface of the rig. Therefore, rather than imposing a prescribed amount of heat flux through the stack, this geometry allowed for verifying how much heat was lost to the outside and gave same insights by observing the temperature field of that insulation region, as can be seen in Fig. 4.

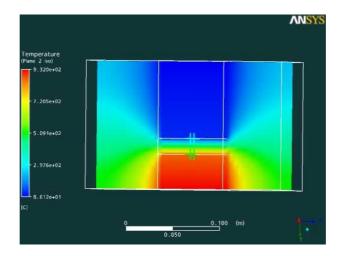


Figure 4- Temperature field calculated to both the stack and the insulation region

3.2 Case study

The following case was created in order to check the proposed methodology of simulation. The sample thermal conductivity k was taken as equal to 1 W/(mK), whereas the stainless steel meter bar conductivity was calculated by Eq. (5) and the aluminum meter bar conductivity was taken as equal to 138 W/(mK). Back to the stainless steel meter bar, its bottom temperature was prescribed as 932°C and the convective heat coefficient at the top of the aluminum bar was of 800 W/(m²K). There where no thermal resistances among the interfaces of different solids or materials. Finally, a fully adiabatic condition was prescribed for the external surfaces of the rig. The stopping criteria for the solver was set to a hundred iterations or a convergence of E-10, the grid was assemble upon hexahedral elements, with about eighty thousand nods and seventy thousand elements.

The post-processing analysis allowed for the association of the temperatures to the locations where the thermocouples were placed in the actual rig. Table 2 displays the temperature values for the positions of Fig (2)

Tab. 2 Temperatures values (K) calculated for the thermocouple positions showed in Fig. 2 ($k_{sample} = 1 \text{ W/(mK)}$).

T1	T2	Т3	T4	Т5	T6
1205,15	1141,6	1027,694	497,804	386,636	359,321

The stainless steel conductivity was calculated by Eq. (5) and its average value was 25.82 W/(mK). Along with these results and the dimensions of the materials it was possible to calculate the heat flux of the reference material I and II by Eq. (2) and (3), which values were $46,128.8 \text{ W/m}^2$ and $47,035.4 \text{ W/m}^2$, respectively. These results allowed for the calculation of the sample conductivity by the Eq. (4), whose result was 1.0425 W/(mK).

In order to compare that former result to the one expected for the rig with the heat guard, a next simulation was performed assuming that there were no heat losses from the stack to the insulation material. Besides that change, all other conditions and assumptions were kept unchanged. The thermal conductivity for that situation was found as $1.0119 \, \text{W/(mK)}$ By taking that last result as a reference, the error or deviation from the former result was of 3.032%.

4. RESULTS

A set of simulations were performed following the methodology described in this paper. The first step is to choose values of thermal conductivity for a generic sample material, within a range where there are doubts about the accuracy of the actual rig working without the hot guard. In the present paper, the sample could assume the values of 0.5; 0.62; 1.0 and 10.0 W/(mK).

The second step is to execute a double simulation for each one of these values. Each pair of simulations are called as a) the reference condition where the stack is fully insulated towards the lateral walls, also called the adiabatic condition; and \mathbf{b}) the rig condition, close to the actual one, where a thermal conductivity of 0.1 W/(mK) is associated to the insulation material that recovers the stack.

Table 3 displays the results obtained for the simulations performed for the 4 pairs of sample materials, showing fluxes along the main axis of heat transfer and, as major information, the thermal conductivities that are calculated after the application of the proposed methodology.

Table 3- Simulation results for the four sample materials assuming the existence of a reference condition (adiabatic) and a rig condition (close to the real bench)

Sample conductivity k_{sample} [W/(mK)]	Stack condition	q" Stainless Steel	q" Aluminum	T_3 - T_4	k_{rig} $k_{reference}$	$\left(\frac{k_{\mathit{rig}} - k_{\mathit{reference}}}{k_{\mathit{reference}}}\right)$
		$[W/(m^2K)]$	$[W/(m^2K)]$	[K]	[W/(mK)]	(rejerence)
0.5	Rig	26175.7	27135.0	1089.79	0.5549	0.1097
	Reference	24322.4	24321.5	1100.10	0.5000	
0.62	Rig	31280.8	32220.1	1072.97	0.6748	0.0884
	Reference	29532.1	29532.0	1082.90	0.6200	
1	Rig	46128.8	47035.4	1032.74	1.0425	0.0303
	Reference	44683.2	44681.9	1023.86	1.0119	
10	Rig	179113.6	179359.9	577.93	9.9974	0.0064
	Reference	179459.3	179282.9	576.20	10.0621	

It can be noticed that the bigger the thermal conductivity of the sample, the lower the relative difference between the expected value of the conductivities.

The thermal conductivity $k_{reference}$ displayed in the column "Stack condition" in Tab. (3) is the same as the one of the column "Sample conductivity k_{sample} ", which is a prescribed value and an input datum of the simulation. The former conductivity is calculated by Eq. (2) and (3) for the adiabatic condition, what validates the calculation procedure proposed in by the present methodology.

The smaller sample thermal conductivities values are quite close to the thermal conductivity of the insulation material around the stack. It means that heat coming from the bottom of the stack will be transferred to the top through a three dimensional path, as there will be a close concurrency among the sample and the insulation material.

The difference in heat transfer fluxes are more evident along the stainless steel side bar, where temperature levels are higher and there is a great amount of heat loss through the side walls. This heat is recovered through the aluminum side walls as the external surfaces of the apparatus are fully adiabatic.

Higher values for the thermal conductivities of the sample lead heat conduction to increase, with fewer losses to the surrounding insulation. Heat losses became less important for sample thermal conductivities above 10.0 W/(mK).

Figures 5 (left) and (right) show a significant variation on the temperature profile for the materials of the stack, as their thermal conductivity was quite different.

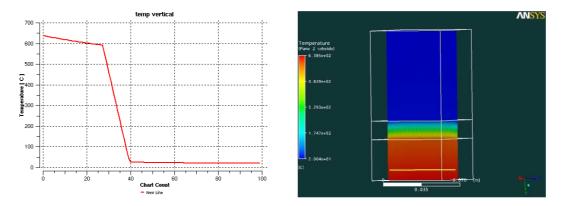


Figure 5- Temperature profile along the *z* axis without lateral heat losses (left) and temperature field for the whole rig (right).

4. CONCLUSION

The main objective of the present work was to identify a range of working values for thermal conductivities of sample materials to be tested on an existing experimental rig. The rig was built following the ASTM E1225standard which proposes a Guarded-Comparative-Longitudinal Heat Flux Technique for the measurement of thermal conductivities of solid materials. The actual situation of this assembling was that the recommended heat guard was not available and a new range of operation should be defined.

A numerical approach was proposed in order to investigate the new operational range of the rig as it could explore unknown values more easily and at lower costs. The proposed methodology consists on the comparison of pair of results for samples of same thermal conductivities but submitted to different insulation conditions for the stack assembling of the rig.

Results show that the bench is suitable for essays of samples with thermal conductivities of 10 W/(mK) and beyond. Heat losses from and for the stack are the reason for the uncertainties of the measured value of the sample conductivity.

3. ACKNOWLEDGEMENTS

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