ECTP 2002
The Sixteenth European Conference on Thermophysical Properties
1 - 4 September 2002, Imperial College, London, UK

Thermal conductivity and specific heat measurement of low conductivity materials using heat flux meters

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Abstract. Heat flux meters, based on tangential temperature gradient, have been developed and are used to measure specific heat and thermal conductivity of low conductivity solid materials. A plane square sample is placed between two heat flux meters, while a skin heater and heat sink are, respectively, placed over and under these components. Starting from a constant temperature all the system is heated in a constant rate, moving toward a final steady-state condition. The specific heat is obtained from the energy accumulated inside the sample during the transient regime and the thermal conductivity from the final steady state condition. A special numerical code was developed in order to simulate the measurement process and the influence of each component, mainly the influence of the lateral heat transfer loss on the measured properties. Some experimental results will be presented, as well as, some discussion and some results about errors involved in the whole process.

1 Introduction

A lot of new materials has been developed and introduced for use in buildings, industry in general and other applications. Such new materials and the old ones need to have their properties determined, including thermal properties. Hot guarded plate (HGP) method has been used and standardized in several countries (ISO/DP 8302, 1991; BS 874, 1986; ASTM C-177, 1997) for thermal conductivity determination. Methods based on heat flow meter (HFM) have also been standardized and allow obtain the same property with the advantage of being faster and simpler compared to HGP. An intercomparison program using HFM methods was described in Salmon and Tye (2000), involving some UK and Eire organisations. Mathis (2000) presents a review on transient thermal conductivity methods as hot wire method, laser flash and transient plane source method.

The method discussed here was investigated previously (Güths, 1990), using thicker HFM, with representative lateral heat loss. A more complex method was implemented by Guimarães, (1993), used to measure thermal conductivity and thermal diffusivity. Following the design and construction of thinner HFM and skin heaters, the first method was reconsidered in order to measure simultaneously two properties as indicated.

2 Experimental Apparatus and Methodology

The experimental apparatus is depicted in a schematic sketch in figure 1. HFM, based on tangential temperature gradient, are used to measure specific heat and thermal conductivity of low conductivity solid materials. A plane square sample is placed between two heat flux meters, while a skin heater is placed over these components. A water-refrigerated plate is used under them to maintain a constant temperature and as a heat sink. All the apparatus is protected with thermal insulation, in order to reduce the lateral heat loss.

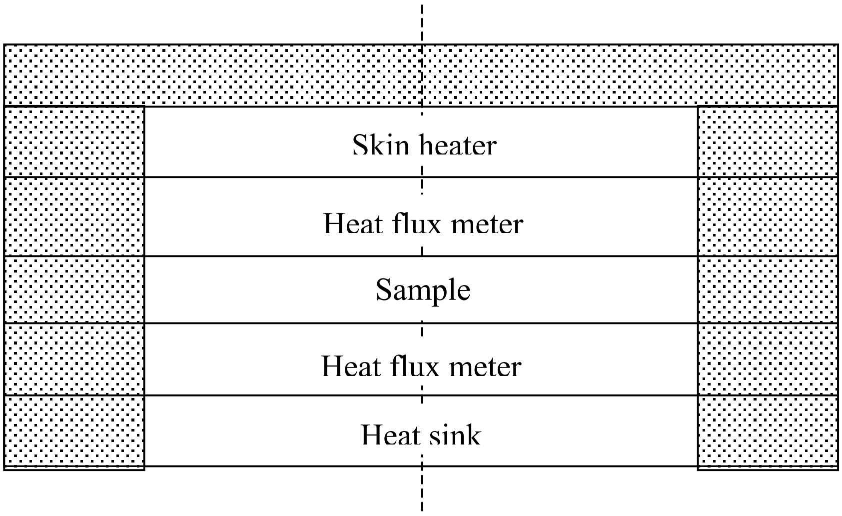

Figure 1. Schematic representation of the experimental apparatus (out of scale).

All the components have a square section of 10cm x 10cm. The HFM and the heater are, respectively 1 and 0.5 mm thick. A special cooper-constantan thermocouple is designed as part of the central area of the flux meter and this one is drawn as a thermopile constituted by a series of tangential thermocouples with a high sensitivity to transversal heat flux. The transducers are calibrated using the electrical power dissipation inside the skin heater and following a process described in Güths (1994).

The measurement process starts from a uniform initial temperature (T_i) condition imposed by the cold plate, normally around ambient temperature. The heater is turned on, producing a constant rate of heat generation during all the measurement. The system evolves from the initial constant temperature toward a final steady-state regime. During such evolution a certain quantity of thermal energy is accumulated inside the sample. This quantity is estimated as the net heat flux difference between upper (q_1) and lower (q_2) HFM signals integrated along the process. The final temperature of the sample is the average temperature, considering upper surface (T_1) and lower surface (T_2) temperatures, according to figure 1. The specific heat value is estimated using equation (1), where ρ, L and A are respectively, the sample density, area and thickness. Δt represents the time interval used to acquire temperature and heat flux signals.

$$c_p = \frac{1}{\rho L A}\left(\sum_{i=1}^{n} q_1 - \sum_{i=1}^{n} q_2\right).\Delta t / \left(\frac{(T_1 + T_2)}{2} - T_i\right)$$
(1)

The thermal conductivity is calculated using equation (2) from the final steady-state condition. The variables q_1 and q_2 furnish the averaged heat flux through the sample.

$$k = \frac{1}{2}(q_1 + q_2)\frac{L}{A.(T_1 - T_2)}$$
(2)

3 Numerical Simulations

Some difficulties are associated to this kind of measurement, as contact resistance, temperature determination of sample surfaces, HFM calibration, and so on. However, the most important problem inherent to this method is the lateral heat loss in the sample. Such heat loss is responsible for the difference observed in the heat flux transducers signals, not so important when thermal conductivity is measured, but essentially important when specific heat is determined. As the last one is calculated by signal integration from the beginning up to the end of the measurement, the difference between heat flux signals do not allow to establish clearly when the measurement must be stopped. Continuous calculations show the specific heat value increasing indefinitely even so steady-state regime is attained. In order to reduce the heat loss effect, a correction will be necessary, and such correction will be dependent on the sample thickness.

A numerical code using Fortran language was specially developed as a tool to determine the relative importance of the heat loss on the values of the measured properties. The explicit form of finite difference method was considered to solve the heat equation applied individually to all components of the apparatus. The explicit method is very time consuming, but it is easier to be implemented. The numerical code allows obtain the temperature and heat flux distribution, the lateral heat loss in the sample, and to evaluate the values of the properties determined following the same process used in the experimental method. These values are compared with those used to promote the simulation, allowing estimate the associated experimental error.

Following the simulation, figure 2 presents the upper and lower surface heat fluxes during the transient regime up to steady-state condition, for a nylon sample's 6.2 mm thick. The property values considered in the simulation are: k = 0.26 W/mK, c_p=1730 J/kgK and ρ=1140 kg/m^3. The observed difference between heat fluxes is the sample's accumulated energy, which is used to compute the specific heat value, according to equation (1).

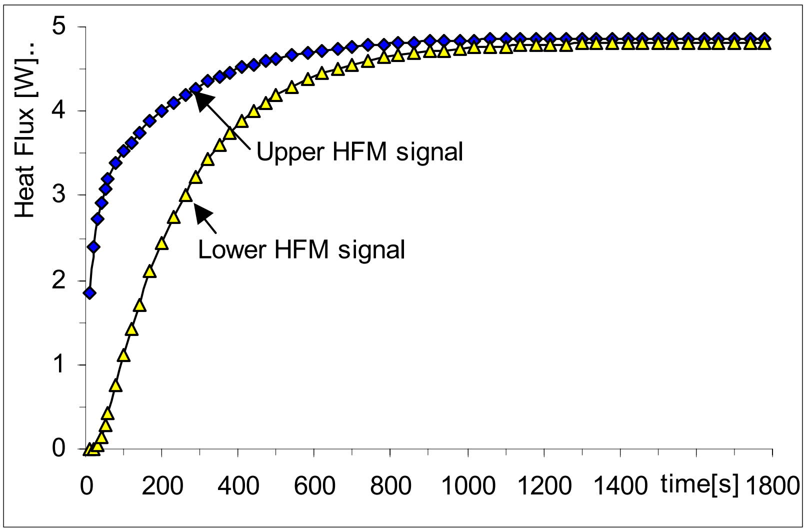

Figure 2. Upper (HFM-1) and lower surface (HFM-2) heat fluxes, for nylon sample 6.2 mm thick.

Similar behaviour is observed in figure 3, where a 15.3 mm sample is simulated. The curves are more distant than those presented in the previous figure. More energy is accumulated inside the sample, and a more important difference is observed between upper and lower surface heat fluxes, through the heating regime and when the system attains steady-state condition.

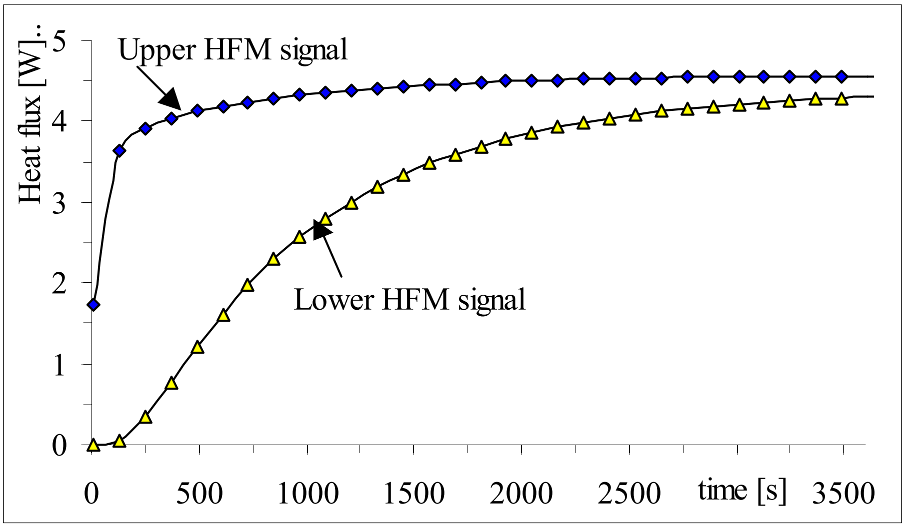

Figure 3. Upper (HFM-1) and lower surface (HFM-2) heat fluxes, for nylon sample, 15.3 mm thick.

The temperature distributions inside the sample are plotted in figure 4, for the thinner sample. The thickness (6.2 mm) was divided in 10 nodes, the temperatures ranging from 26 to 36.5 ºC. In the radial direction ¼ of the sample was taken into account, including also 10 nodes in such direction (the x origin is the sample center, and L_{sample} is a half of the sample width). The lateral insulation was also considered, including 5 nodes. The configuration presented in figure 4 is the final configuration when the steady-state condition is attained. A more uniform distribution is observed in the low temperature surface, in contact with the lower transducer (HFM-2). On the hotter sample surface a temperature reduction is observed from the center to the border, showing clearly a lateral heat loss toward the insulation. As the cooling plate covers only the sample area, the insulation material shows some nodes with higher temperature when compared with the sample temperature at the same level. Some energy is lost near the upper surface of the sample and a little fraction is recovered to the sample near the lower surface.

Specific heat results for a nylon sample (6.2 mm thick), is plotted in the figure 5, through the measurement time. The reference value is depicted as a horizontal line and such value is used in the simulation. The estimated value was obtained as defined in equation (1), following the accumulated difference between signals from upper and lower HFM. A constant increasing in the specific heat value has the heat loss as the responsible agent. Even so steady-state condition is attained, heat flux on upper sample face is more important than the equivalent on the lower face. Such differences, from lateral heat loss, are accumulated erroneously as energy inside the sample. While the measurement is carried on, the specific heat value increases continuously. Two different corrections are then applied: in the first one the upper HFM signal is limited by the maximum value obtained in the lower HFM signal (the final value). This one is taken as true value, without interference from the lateral heat loss. The second type (called proportional correction), considers a correction applied on the heat flux on the upper surface equal to the ratio of final heat flux on the lower surface to final heat flux on the upper surface. Both corrections stop the increasing tendency of c_p value, as presented in the figure 5, where the proportional correction represents to be more adequate for the sample considered.

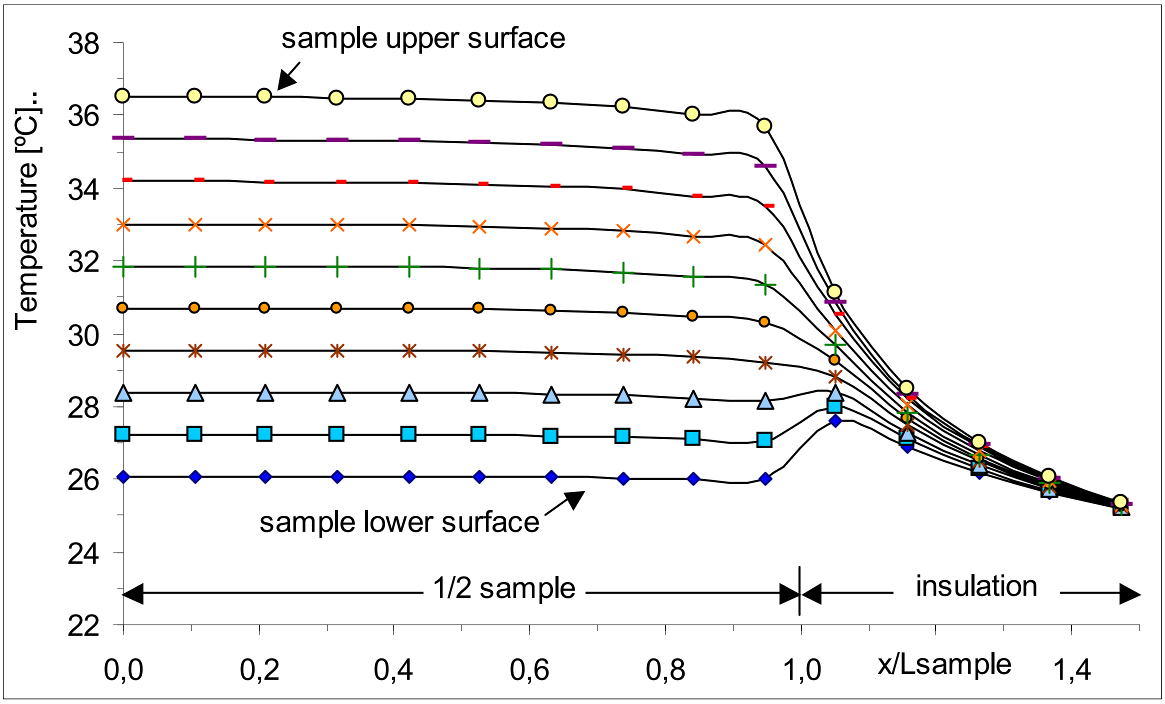

Figure 4. Temperature distributions along parallel plans from upper to lower sample surfaces (sample thickness – 6.2 mm).

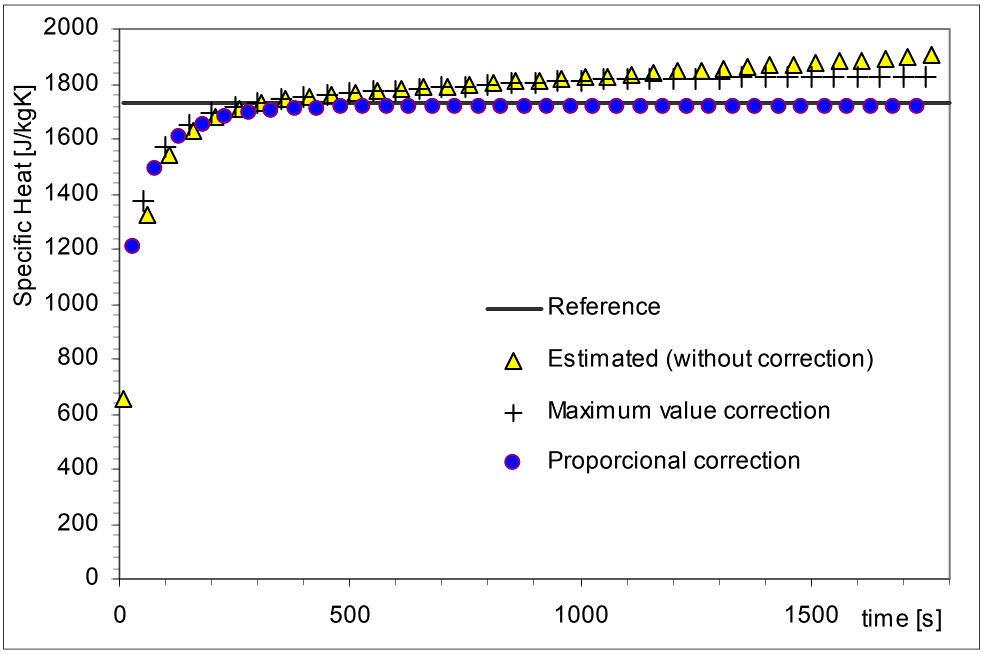

Figure 5. Specific heat estimations with different correction methods (thickness - 6.2 mm).

Similar analysis was performed for a thicker sample (15.3 mm) and the results are summarized in the Table 1 as an error related to the standard value used in the simulation. In order to reduce the influence of lateral heat loss on the measured values, another heat flux transducers were constructed, with the same dimensions as the previous ones, but using only the central area as sensitive area (50 mm x 50 mm). The remaining external area has the same characteristics, but only the central area is active and responsible for the measurement. Some results presented in the table 1 from measurement simulations, show a small error in the c_p value for the value obtained from equation (1), without any correction, and a smaller error when the correction based on the maximum value is applied. When the sensitive area in the HFM is only the central area, the proportional correction method promotes a more significant error and must be discarded.

Table 1. Simulated errors (%), in the specific heat calculation.

Sample thickness [mm]	Measurement area [mm x mm]	No correction - Equation (1)	Correction: Maximum value	Correction: Proportional
6.2	100 x 100	10.2	5.3	1.0
6.2	50 x 50	0.3	0.0	1.3
15.3	100 x 100	12.1	0.6	9.6
15.3	50 x 50	3.6	1.0	6.9

Thermal conductivity determination is also simulated and some results are presented in figure 6. The property value should be calculated at the end, when the system reaches steady-state condition. However as it could be observed in figure 6, there is a convergence toward the reference value faster than expected. It could be observed in addition, that time interval necessary to achieve the steady-state condition is dependent on the sample thickness. In this case HFM with sensitivity only in the central area were considered.

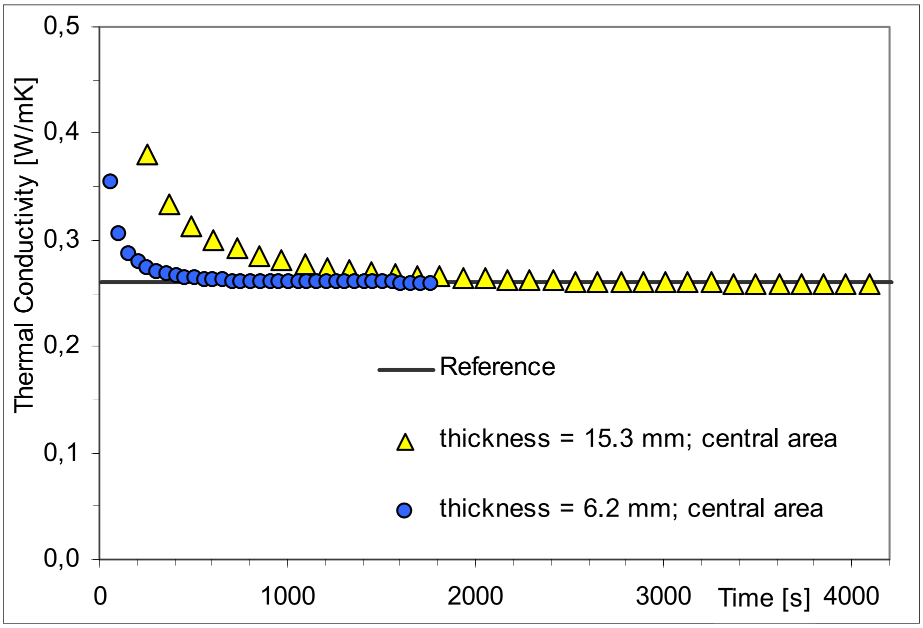

Figure 6. Thermal conductivity estimation for samples with distinct thickness (HFM sensitive only in the central area).

4 Experimental Results

Some experimental results were obtained and are presented here. The chosen sample material was nylon, with properties presented in the last topic. Before using the experimental apparatus to measure properties, a checking on the transducers behavior has been executed and the results are presented in figure 7. The results show a heat flux rate variation imposed on the transducers in order to verify the system inertia. A small difference appears when a fast variation is imposed, which is reduced when a constant power is restored.

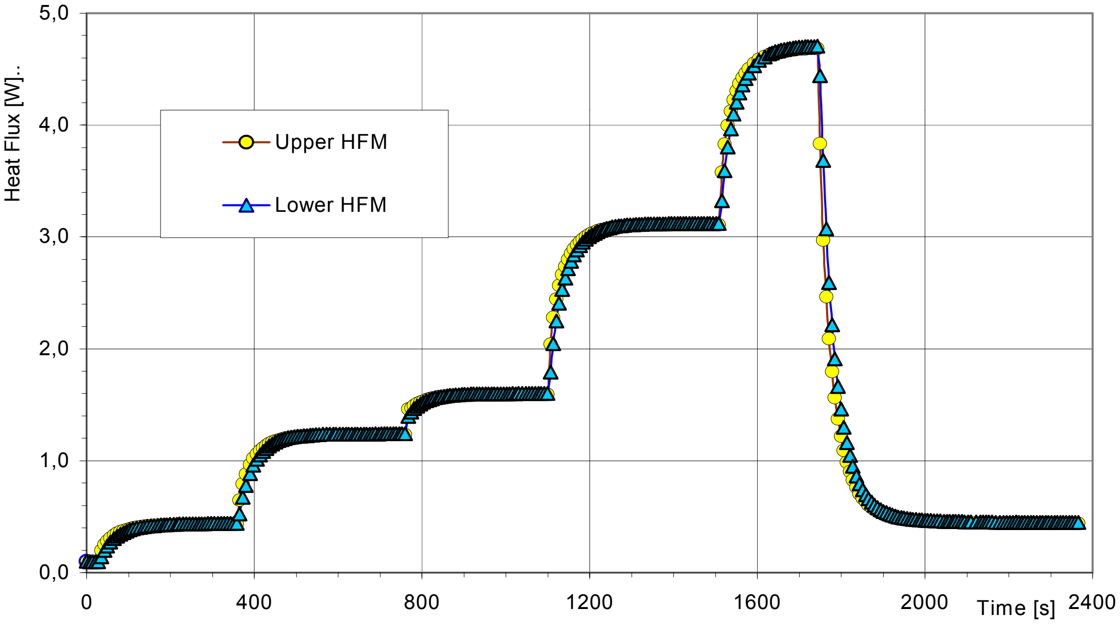

Figure 7. Transducers response checking to an imposed heat flux rate (without sample).

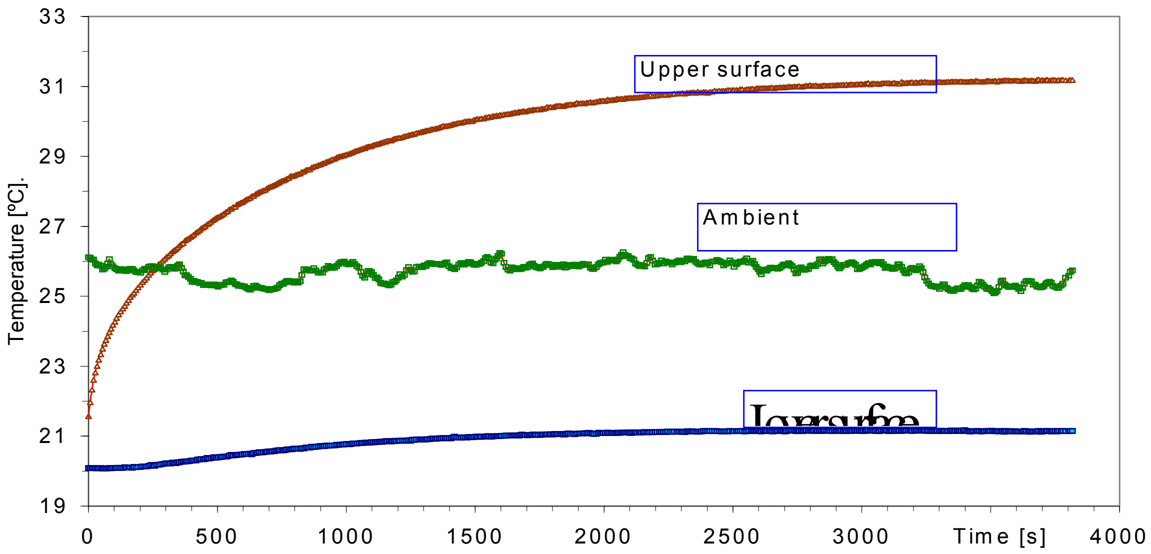

Figure 8. Temperature evolution during measurement process – ambient and sample surfaces.

As an example, figure 8 shows the temperature distributions for a sample 15.3 mm thick. Upper surface temperature experiments a strong growth, because of the proximity with the heat source. Otherwise, the temperature in the lower surface of the sample has a more attenuated progression, stabilizing near the refrigeration bath temperature, in this case bellow ambient temperature. The time necessary to stabilize is about one hour.

Table 2. Experimental results for nylon samples.

Material	density [kg/m^3]	Thermal conductivity [W/mK]	Specific heat [J/kgK]
Nylon 6.2 mm	1.15 10^3	0.22	1.73 10^3
Nylon 15.3 mm	1.14 10^3	0.27	1.74 10^3
Nylon 6.2 + 15.3 mm	-	0.28	1.73 10^3

Some experimental results for nylon samples are presented in table 2, for two specific thicknesses and a combination of these two samples. A good agreement is observed for specific heat, but some differences are present in the thermal conductivity results. These are the first results and new improvements are going to be implemented in order to obtain more confidence in the complete measurement system. Of coarse, it will be necessary to compare the results for a specific sample, whose properties are well known, as it happens in an intercomparison program.

5 Conclusions

The results have shown the possibilities of applying the presented method for specific heat and thermal conductivity determination using a plane sample. The thermal conductivity determination is straightforward in the sense that the measurement and calculations are very simple. However for the specific heat the process is more complicated and a continuous data acquisition is necessary. A correction must be applied to the measured heat transfer value in order to impose a limit on such variable. This correction constitutes a drawback, but does not discard the method that must be improved and can be used to furnish good results in a secondary level measurement.

Acknowledgement. This work was supported by CNPq – Conselho Nacional de Desenvolvimento Científico e Tecnológico - Brazil.

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