

APPLICATION OF DIFFERENT HEAT FLUX INTENSITIES FOR THE THERMAL PROPERTIES SIMULTANEOUS ESTIMATION OF METALLIC MATERIALS

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Abstract. *An accurate knowledge of thermophysical properties like thermal conductivity and volumetric heat capacity is very important, for example, to optimize the engineering design and the development of new materials for many applications. Nowadays, due to globalization more and more new methods are required to quickly, reliably, and accurately determine these properties. Another important aspect is the economic issue; because the lower the cost to determine the properties, ensuring reliability, the greater the chance to compete in the national and international markets. Since this is an important topic, many researchers have been developing new methods. These methods were designed in order to find feasible solutions to accomplish the measurement of these properties. Hence, this paper presents a method for the simultaneous estimation of these properties in samples of ASTM B265 Grade 2 Titanium and AISI 316 Stainless Steel. In this method, the thermal conductivity and volumetric heat capacity are determined simultaneously using the same experiment. A symmetrical assembly is used in order to minimize errors of heat flux measurements. The sample is placed between the resistive heater and the insulator. To eliminate the heat losses resulting from the convection phenomenon, the assembly was totally isolated by polystyrene plates. Furthermore, the sample has much smaller thickness than its other dimensions and the experiments are done very fast, ensuring a one-dimensional thermal model. This model uses a constant heat flux on the upper surface of the sample and insulation condition on the bottom surface. The temperature is measured on the bottom surface using type K thermocouple. In this work, different intensities of heat flux were used in the same experiment as an attempt to achieve the best condition to simultaneously estimate the properties in accordance to the analyses of the sensitivity coefficients. To determine these properties, the sequential optimization technique BFGS (Broydon-Fletcher-Goldfarb-Shanno) is used to minimize an error function defined as the square difference between the experimental and numerical temperatures. The numerical temperature is obtained by using the solution of the heat diffusion equation for the one-dimensional model. The solution was obtained numerically using the finite difference method through an implicit formulation. In order to determine the best region and experimental configuration for estimating these properties, analyses of the sensitivity coefficients are performed in set with the error function. The estimated properties are in good agreement with literature.*

Keywords: *thermal conductivity, volumetric heat capacity, simultaneous estimation, heat conduction, optimization.*

1. INTRODUCTION

Nowadays, due to globalization, more and more new techniques are required to quickly, reliably and accurately determine the thermophysical properties of materials. Another important aspect is the economic issue, because the lower the cost to determine the properties, ensuring reliability, the greater the chance to compete in the national and international markets. The technique proposed in this paper can be used, for example, to correctly choose, under the point of view thermal properties, the materials to be used in the manufacture of a heat exchanger. This choice is made by taking into account the values of thermophysical properties, which should be ideal to yield a saving that is directly linked to energy and environmental issues, widely discussed in the current global circumstances.

Another example can be a machining process which great part of the heat generated by friction between the workpiece and the cutting tool must be transferred to the tool holder, as the tool wear is directly linked to temperature increase. Thus, the right tool for the process can be chosen through the knowledge of its thermal conductivity, since this property determines the range of the working temperature of the material. From these needs, researchers have developed many techniques which are being improved continuously (Carvalho *et al.*, 2006 and Brito *et al.*, 2009). These techniques can estimate the properties simultaneously and non-simultaneously.

There are three frequently used methods among these techniques like: the Guarded Hot Plate, Hot Wire Technique, and the Flash Method. The Guarded Hot Plate Method (ASTM C177, 1997) which is widely used to determine the thermal conductivity, λ , of insulating materials, is considered by many researchers as Wulf *et al.* (2005) and Lima *et al.* (2008), among others, the most accurate and reliable. In this method, the homogeneous and isotropic sample, in shape flat plate is placed between a hot and a cold plate in such a way that the heat flux through the central area of the sample is unidirectional. Under steady state conditions, the thermal conductivity is calculated by measuring the heat flux and the mean gradient of temperature on the sample. The Hot Wire Technique presented by Blackwell (1954) became

widely used to determine the thermal conductivity. This technique is basically performed by inserting a cylindrical probe which contains a resistance wire and a thermocouple in the middle of a sample. This method can also be used to obtain the thermal diffusivity, α , requiring for this, the application of another thermocouple on the sample. A restriction to this method concerns about metallic materials due to high contact resistance between the probe and the sample, since it is very difficult to eliminate the air gaps present in the assembly. Several researchers have improved this technique in order to determine the properties of other materials (Nahor *et al.*, 2003 and Adjali and Laurent 2007). The former optimized the position of the hot wire to find food conductivity, and the latter proposed a change in methodology to determine the conductivity of a water-agar gel mixture by varying the temperature. The Flash Method developed by Parker *et al.* (1961) is used to determine the thermal diffusivity. This technique consists of applying a radiant heat pulse of great intensity and short time on a surface of a sample. It is then possible to obtain the thermal diffusivity based on the time required for the temperature on the other side to reach the maximum value. A limitation to determine the thermal conductivity in this technique is the need to know the amount of energy absorbed on the front face of the sample. Since this is a widely researched topic, new methods have been developed to eliminate the limitations of the above techniques (Shibata *et al.*, 2002, Santos *et al.*, 2005 and Coquard and Panel 2008).

Taktak *et al.* (1993) determined λ and volumetric heat capacity, ρc_p , simultaneously for a carbon fiber and epoxy compound. The assembly consisted of a square-shaped sample with prescribed heat flux condition on the top surface, and prescribed temperature on the opposite surface. The temperatures were monitored on both sides. This study aimed to demonstrate the ideal conditions to perform the experiment in order to achieve reliable and accurate results. Seeking to find the best study area to obtain the properties, an analysis of the sensitivity coefficient and the determinant was carried out. The criteria chosen for this analysis were: position of the thermocouple in relation to the heater, the time experiment, and the heating. Thus, they concluded that to obtain more accurate results, it is feasible to collect the temperature as close as possible to the heat flux and to heat up the sample to be investigated in the shortest possible time.

Dowding *et al.* (1995) used a sequential technique in transient experiments to determine λ and ρc_p simultaneously for a carbon-carbon compound. The symmetrical assembly consisted of a heater placed between two samples isolated by a non-conductor ceramic plate. This work was developed for the one-dimensional thermal model to study the influence of the position of the thermocouples on the sample by analyzing the sensitivity coefficients. The properties were estimated by varying the temperature from room temperature to 623 °C using a controlled atmosphere furnace.

Blackwell *et al.* (2000) proposed the determination of λ in the transient state. To achieve this goal, the sensitivity coefficients were analyzed to guide the design of an experiment to estimate the thermal conductivity for the steel AISI 304. The conductivity was determined by an experimental setup, where the heat conduction was considered axial on the walls of a hollow cylinder.

Borges *et al.* (2006) presented a method to obtain simultaneously and independently α and λ for conductive and non-conductive materials. One advantage of this technique refers to the fact of obtaining the properties simultaneously, but independently, since two objective functions were applied: one in a frequency domain and another in the time domain. The frequency domain function was obtained by calculating the phase of the response function of a dynamic system, and the time domain function was based on known temperatures. A disadvantage of this study is the small number of points to estimate α and how it is estimated first, since this may influence the results of λ .

Jannot *et al.* (2006) developed a Transient Hot Plate Method to determine simultaneously the thermal effusivity, b , and the thermal conductivity of metallic materials such as aluminum, titanium and steel. The proposed device uses a simple heating element inserted between a plane face sample of the material to be characterized and a sample of an insulation material. The heating element and the sample have the same area so that the heat transfer may be considered as unidirectional as long as the convective heat losses are negligible. Temperature sensors were used in order to estimate the properties by minimizing a quadratic error function between the experimental and numerical temperatures. Sensitivity studies were realized to determine the best region to analyze the properties as well as the ideal thickness of the sample. One disadvantage of this study is the large thickness of the samples, which increases the cost.

Ghrib *et al.* (2007) developed a method based on the Mirage Effect, which is possible to estimate simultaneously α and λ of metallic materials like aluminum, steel, titanium, among others. The method is based on the comparison of the amplitude variation and of the phase of the experimental thermal sign with the square root of the modular frequency. The properties were estimated when the experimental and theoretical temperature curves were coincident. The values of the estimated properties were in good agreement with the literature values. The disadvantaged of this method is the high cost of the experimental apparatus.

Borges *et al.* (2008) proposed a method to estimate the thermal conductivity and diffusivity of conductor and non-conductor materials of the small dimensions. This work is very similar to Borges *et al.* (2006), although fluximeters were not used to measure the applied heat flux. Inverse techniques based on Green's Function were used to estimate the applied heat flux. The experiment was designed by using a heater on part of the top surface, and considering isolated the others parts, ensuring a three-dimensional thermal model. Good results were found for the estimated properties of a AISI 304 Stainless Steel sample.

In the present work a method is proposed to determine simultaneously the volumetric heat capacity and the thermal conductivity for metallic materials using the same experiment. This method is based on a one-dimensional constant heat

conduction model. The applied heat flux had different intensities for each part of the experiment, in order to achieve the ideals conditions to estimate the properties. The properties are estimated by minimizing the quadratic error function based on the difference between the experimental and numerical temperatures. To minimize this function, the sequential optimization technique BFGS is used. The temperature is obtained by the numerical solution of the heat diffusion equation for the thermal model by using the finite difference method with implicit formulation. Furthermore, analyses of the sensitivity coefficients allied to the error function are performed to find the best setting and region to obtain the properties.

Therefore, the objective of this work is to develop a new methodology, seeking to eliminate the impracticable found in other studies to determine simultaneously the volumetric heat capacity and the thermal conductivity for metallic materials.

2. THEORETICAL ASPECTS

2.1. Thermal Model

Figure 1 shows the proposed one-dimensional thermal model, that consists of a sample located between a resistive heater and an insulator. To ensure the unidirectional heat flux, the sample has much smaller thickness than its others dimensions. In addition, all the surfaces, except the heated ($x = 0$), were isolated.

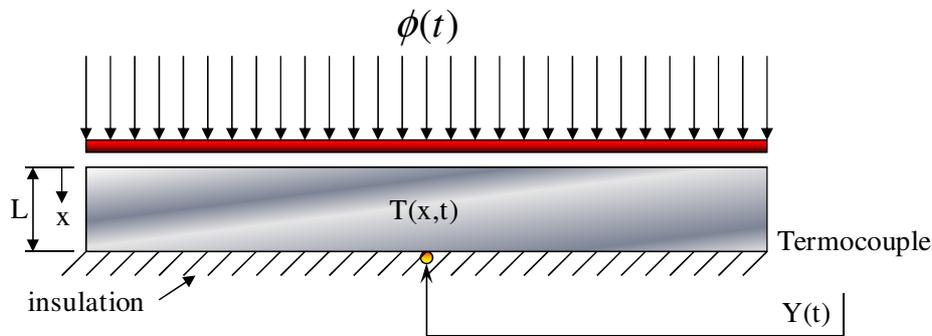


Figure 1. One-dimensional thermal model.

The heat diffusion equation for the problem presented in Figure 1 can be written as:

$$\frac{\partial^2 T(x,t)}{\partial x^2} = \frac{\rho c_p}{\lambda} \frac{\partial T(x,t)}{\partial t} \quad (1)$$

subject to the boundary conditions:

$$-\lambda \frac{\partial T(x,t)}{\partial x} = \phi(t) \text{ at } x=0 \quad (2)$$

$$\frac{\partial T(x,t)}{\partial x} = 0 \text{ at } x=L \quad (3)$$

and the initial condition:

$$T(x,t) = T_0 \text{ at } t=0 \quad (4)$$

where x is the Cartesian coordinate, t the time, ϕ the prescribed heat flux, T_0 the initial temperature of the sample and L the thickness.

The numerical temperature is obtained through the solution of the one-dimensional diffusion equation using the finite difference method with an implicit formulation.

2.2. Analyses of the best region to determine the properties ρc_p and λ

Studies of the sensitivity coefficient for each sample are performed in this work in order to determine the ideal region to estimate the properties and the best configuration of the experimental setup. This study provides information such as: the correct positioning of the thermocouples, the experimental time, and the time interval of the applied heat flux incidence. The higher the coefficients value, the better the chance of obtaining the properties reliably.

The sensitivity coefficient is defined by the first partial derivative of the temperature in relation to the parameter to be analyzed (ρc_p or λ), being written as follows:

$$X_{ij} = P_i \frac{\partial T_j}{\partial P_i} \quad (5)$$

where T is the numerical temperature, P the parameter to be analyzed (ρc_p or λ), i the index of parameter, and j the index of points. As in this work, only two properties will be analyzed, $i = 1$ for ρc_p and $i = 2$ for λ .

Besides this, analyses of the error function were done in order to guarantee that in the analyzed region there is enough information to estimate the properties simultaneously. One can verify this information if a minimum value of the function error is found when there are changes of the properties values. This error function is represented by Eq. (6) in the next section.

2.3. Volumetric heat capacity and thermal conductivity simultaneous estimation

To estimate the two properties it is necessary to use an error function based on the square difference between the experimental and numerical temperatures. This equation can be written as:

$$F = \sum_{j=1}^m (Y_j - T_j)^2 \quad (6)$$

where, m is the total number of points, and Y the experimental temperature.

Thus, it is known that the optimal value for ρc_p and λ , in other words, the value that minimizes the error function, is the value of the property to be estimated. To obtain this value you can use optimization techniques, such as the BFGS (Broydon-Fletcher-Goldfarb-Shanno) sequential optimization technique used in this work, and presented in Vanderplaats (2005). This technique is a particularity of Variable Metric Methods. The advantages of this method are the fast convergence and the ease to work with many design variables. Because it is a first order method, it is necessary to know the gradient of the error function. This gradient is calculated numerically by using the computer package Design Optimization Tools - DOT (Vanderplaats, 2005).

3. EXPERIMENTAL PROCEDURE

The experimental apparatus used to determine the properties of AISI 316 Stainless Steel and ASTM B265 Grade 2 Titanium is shown in Fig. 2. The stainless steel plate has the dimensions of 49.9 x 49.9 x 9.9 mm and the titanium plate 49.9 x 49.9 x 9.1 mm. The resistive kapton heater has a resistance of 15 Ω and the dimensions of 50.0 x 50.0 x 0.2 mm. The resistive kapton heater was used because it is very thin, allowing faster overall warming. This heater was connected to a digital power supply Instrutemp ST – 305D-II to provide the necessary heat flux. In this work, different intensities of heat flux were used in the same experiment as an attempt to achieve the best condition to estimate the properties simultaneously in accordance to the analyses of the sensitivity coefficients. To achieve this heat flux condition, the digital power supply has a configuration that allow to work at parallel or series connection. Then, we used the series condition to provide the highest heat flux for the first period of the experiment, and the parallel condition to supply the lowest heat flux for the second part. A symmetrical assembly was used to minimize the errors in the measured of the heat flux to be generated on the sample surface. In addition, the applied current and voltage values were measured by the calibrated multimeters Instrutherm MD-380 and Minipa ET-2042C. The contact between the resistive heater and the sample is not perfect; therefore the silver thermal compound Arctic Silver 5 was used to eliminate the air interstices present in the assembly. The great advantage of this compound refers to its high thermal conductivity. In addition, weights were used on top of the isolated set samples-heater to improve the contact between the components. To ensure a unidirectional flux and minimize the effect of convection caused by the air circulating in the environment, the set samples-heater was isolated with polystyrene plates. Temperatures were measured using thermocouples type K (30AWG) welded by capacitor discharge and calibrated using a bath temperature calibrator Marconi MA 184 with a resolution of ± 0.01 °C. The type K thermocouple was used to measure the temperature of the plate in contact with the polystyrene. This thermocouple was connected to a data acquisition Agilent 34980A controlled by a microcomputer. In order to obtain better results, all experiments were performed in controlled room temperature.

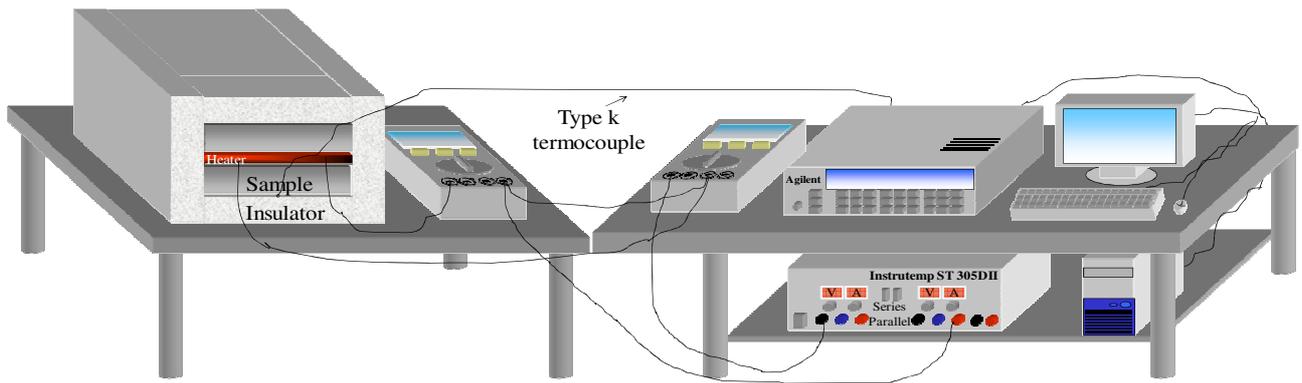


Figure 2. Sketch of experimental apparatus used to determine the properties.

4. RESULTS ANALYSES

4.1. AISI 316 Stainless Steel

Forty experiments were performed to simultaneously estimate the volumetric heat capacity and the thermal conductivity of AISI 316 Stainless Steel. Each experiment lasted 150 s, but the heat flux was imposed from 0 to 130 s. In the first part, that consists in the interval of 0 to 30 s, the applied heat flux was approximately 2640 W/m². For the second part, the time between 30 to 130 s, the imposed heat flux was around 660 W/m². The time interval used to monitor the temperature was 0.1 s.

The sensitivity analysis was performed to determine the best region to estimate the properties. This analysis was performed by using the values of λ and ρc_p obtained from Incropera *et al.* (2007). Analyses of the error function were done allied to sensitivity analysis in order to guarantee that there was enough influence to determine these properties in the selected region. Figure 3 shows the sensitivity coefficients at $x = L$ for ρc_p and λ , and Fig. 4 presents the values of the error function.

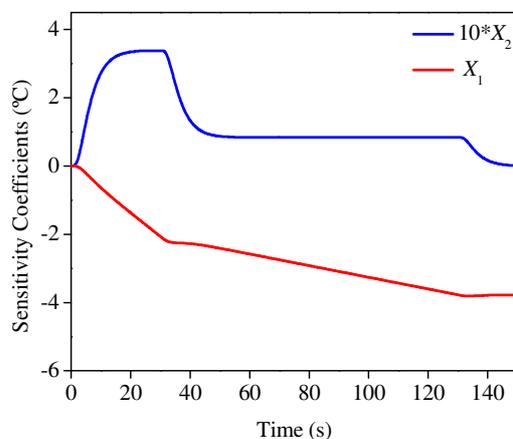


Figure 3. Sensitivity Coefficients for Stainless Steel.

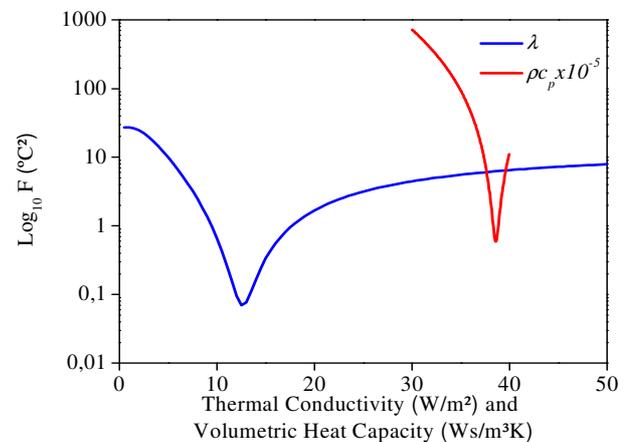


Figure 4. Error Function Values (F) for Stainless Steel.

X_1 represents the sensitivity coefficient for ρc_p and X_2 represents the sensitivity coefficient for λ , both on the isolated surface. The latter is multiplied by a factor in order to improve the visualization of the curve. By analyzing Figure 3, one can see that X_1 increases during the first 20 s, and after this, it keeps constant up to the change of the heat flux, and X_2 increases at the same proportion that the temperature increases. Because of this behavior, the highest heat flux was applied in the first period of time, resulting in a high sensitivity for λ ; and the lowest heat flux was applied on the second part in order to increase the sensitivity for ρc_p and keeps the sensitivity for λ . This procedure was done, because it is necessary to control the magnitude relation between X_2 and X_1 , in order to guarantee that the estimation will occur for the two properties. So, Figure 4 shows that there is enough influence to determine the properties simultaneously at the region analyzed, because a minimum value was found for each property. Another objective of sensitivity analysis is to determine the number of points in the curve which should be used to estimate the properties. These points to be considered should not have derivative equal to zero. The sets of points that do not fit this description should be disregarded in the estimation of properties. In this work, the points chosen to estimate the properties corresponds the points where there is applied heat flux, in other words, the interval between 0 to 130 s.

In order to check if these conditions resulted in good experiments, another analyze was done. This analyze was based in Dowding *et al.* (1995) that said: when the sum of the sensitivity coefficient of ρc_p and λ , considering the boundary conditions of prescribed heat flux on the top surface and insulation on the bottom surface, plus the temperature gradient is equal zero ($X_1 + X_2 + Y - Y_0 = 0$), the best condition and design for the experiment was achieved. Then, Fig. 5 shows the results of this analyze.

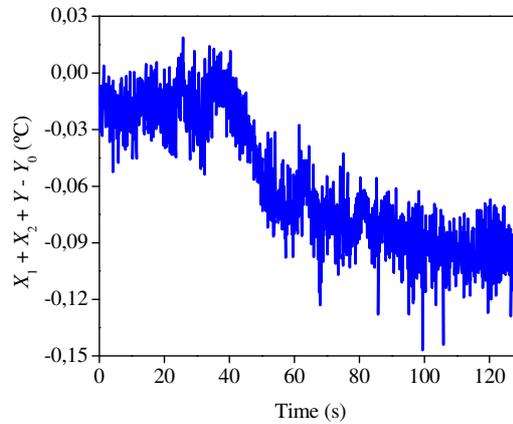


Figure 5. Analyze for the best condition and design for Stainless Steel.

One can see that the result for this analyze is very good, because the highest difference was around of 0.15 °C. Thus, this proves the well done experiment.

Figure 6 presents the distribution for experimental and numerical temperatures for the plate, at $x = L$ and the imposed heat flux at $x = L$. The numerical temperature is achieved by employing the properties values ρc_p and λ estimated for one of the accomplished experiments. These temperatures present good concordance that one can be proved by analysis of the temperature residuals. The temperature residuals are shown in Figure 7, in other words, it is the percentage difference between the experimental and the numerical temperatures. These residuals are calculated by doing the difference between the experimental and numerical temperatures, and these differences are divided by the numerical temperature. One observes the good agreement of the results for the AISI 316 Stainless Steel. For the thermocouple located on the opposite surface, a difference of up to 0.5% was sensed. These deviations may be due to contact resistance between the resistive heater and the sample, and the difficulty of isolating the experiment.

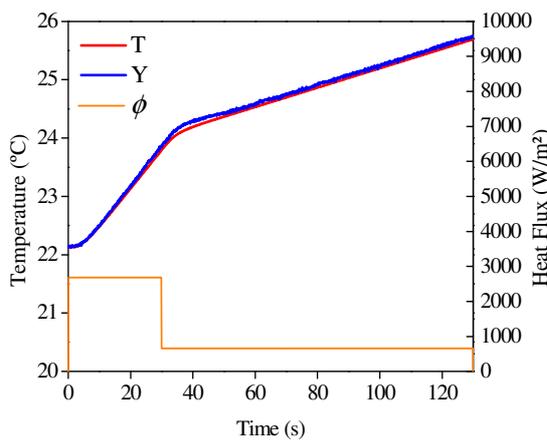


Figure 6. Numerical (T) and Experimental (Y) Temperatures with Heat Flux (ϕ) for Stainless Steel.

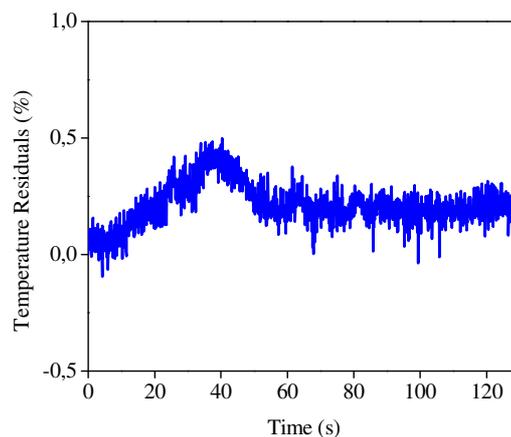


Figure 7. Temperature residuals for Stainless Steel.

Table 1 presents the mean value, the standard deviation and the error (the percentage difference between the mean and the literature value) for ρc_p and λ of AISI 316 Stainless Steel.

Table 1. Results obtained for the AISI 316 Stainless Steel.

Property	Mean	Incropera <i>et al.</i> (2007)	S. D.	Error (%)
$\rho c_p \times 10^{-6}$ (Ws/m ³ K)	3.93	3.86	± 0.04	1.78
λ (W/mK)	13.52	13.40	± 0.20	0.89

The estimated values of ρc_p and λ , when compared with the literature values, are in good agreement. However, the error found in estimating the thermal conductivity is consistent when compared with the values found in the literature.

4.2. ASTM B265 Grade 2 Titanium

This part presents an analysis of the results obtained for the determination of ρc_p and λ of an ASTM B265 Grade 2 Titanium sample. Forty experiments were carried out, and 1500 points were collected in each one, but the heat flux was applied during the 120 s. The increment of time used to get the temperatures was the same used for the AISI 316 Stainless Steel (0.1 s). The applied heat flux was about 2680 W/m² for the first part (0 to 20 s), and 675 W/m² for the second part (20 to 120 s).

The sensitivity analyses, showed in Fig. 8, were performed in set with the error function analyses, presented in Fig. 9, as described in the study of stainless steel. The values for the properties were extracted from GMTTitanium (2010).

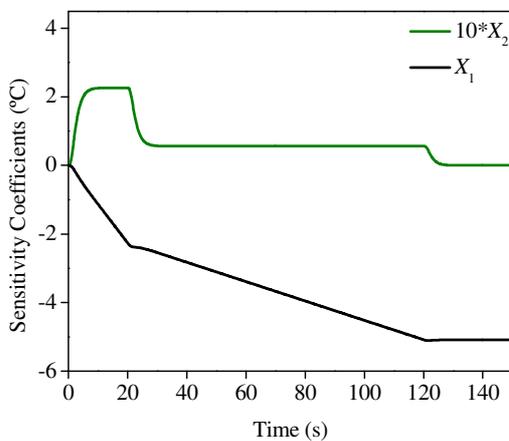


Figure 8. Sensitivity Coefficients for Titanium

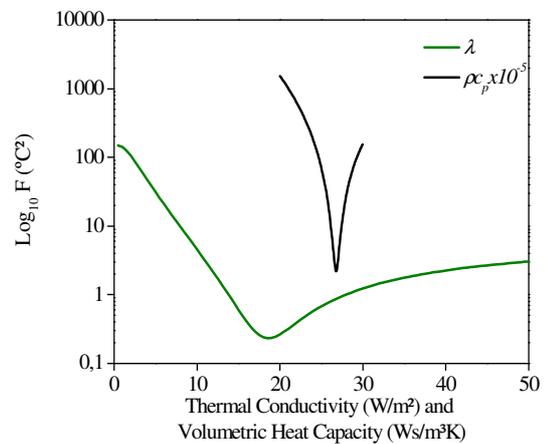


Figure 9. Error Function Values (F) for Titanium.

The determination of the titanium properties was accomplished the same way and considering the same conditions as for the stainless steel. Figure 10 showed the result for analyze of the well- design experiment. Figure 11 presents the applied heat flux and the comparison between experimental and numerical temperatures; the latter was calculated by using the properties values estimated in one experiment.

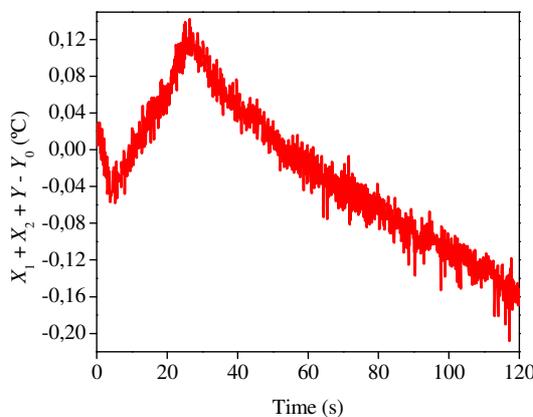


Figure 10. Analyze for the best condition and design for Stainless Steel.

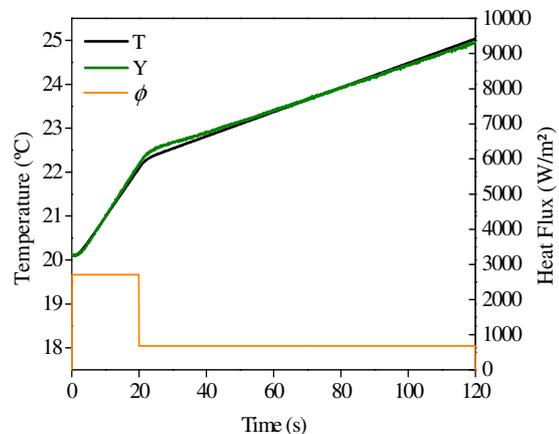


Figure 11. Numerical (T) and Experimental (Y) Temperatures with Heat Flux (ϕ) for Titanium.

These curves are in good agreement and this fact can be checked by analyzing the Fig. 12, which presents the temperature residuals. The reason for this difference, around 0.7 %, may be due to the difficulty found to isolate the experiment completely and the contact resistance between the resistive heater and the sample.

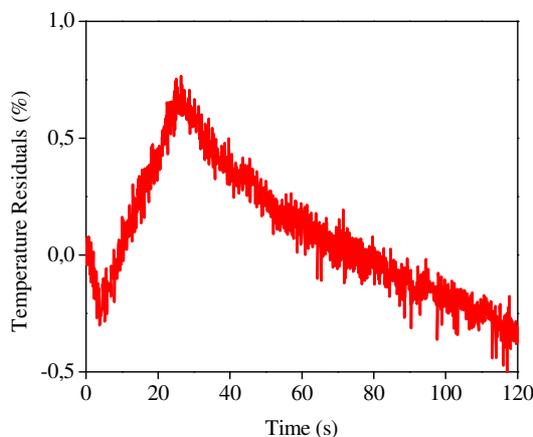


Figure12. Temperature residuals for Titanium.

Table 2 presents the mean value, the standard deviation, and the comparison with the reference value obtained from literature, for the ρc_p and λ of ASTM B265 Grade 2 Titanium.

Table 2. Results obtained for the ASTM B265 Grade 2 Titanium.

Property	Mean	GMTitanium (2010)	S. D.	Error (%)
$\rho c_p \times 10^{-6}$ (Ws/m ³ K)	2.71	2.66	± 0.05	1.88
λ (W/mK)	17.88	18.06	± 0.27	1.00

Similar to Stainless Steel, the results presented good agreement with the literature value.

5. CONCLUSION

This paper presents a technique to simultaneous estimate the volumetric heat capacity and the thermal conductivity of metallic materials applying different intensities of heat flux. Two materials were analyzed: AISI 316 Stainless Steel and ASTM B265 Grade 2 Titanium. Good results for both materials were found. This affirmation can be proved due to the small difference between the literature and estimated values, and the low standard deviation. For future work, some improvements are proposed: to do analyses considering the influence of the initial temperature variation in order to estimate these properties; and to use a three-dimensional thermal model to allow the placement of thermocouples in various points of the sample, so as to find regions which present higher sensitivity to determine these properties.

6. ACKNOWLEDGEMENTS

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