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SIMULTANEOUS MEASUREMENT OF THERMOPHYSICAL PROPERTIES OF SHAPE MEMORY ALLOYS USING A PERIODIC TEMPERATURE FIELD

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Abstract. *The characterization of materials has been crucially important for engineering due to the fact that new materials present more specific behaviors and properties. A group of advanced materials that has been outstanding on its applicability in engineering are the Shape Memory Alloys (SMA) which can be used as thermomechanical sensors and actuators. Among the relevant thermophysical properties are the thermal conductivity, thermal diffusivity and specific heat, which have great influence on the kinetics of heat transfer of the materials. For the SMA, the determination of these properties is very important, since functional behaviors, as the Shape Memory Effect (SME) and superelasticity (SE), are deeply related to temperature changes. Using a periodic temperature field is possible to determine experimentally these thermophysical properties. To carry out the measurements, the temperature field along a SMA cylindrical specimen is monitored by micro thermocouples distributed throughout the sample's body tested. Thus, one could obtain different amplitude and phase's values for each thermocouple. These parameters obtained experimentally are applied to a mathematical model which allowed the determination of thermal diffusivity. Thermal conductivity was obtained with the average temperature field and the specific heat was given from the definition of thermal diffusivity.*

Keywords: *Shape Memory Alloys, Thermophysical properties, Shape Memory Effect, Periodic Temperature Field.*

1. INTRODUCTION

Due to intense technological evolution that has occurred in recent years there has been increased the necessity of determining the thermophysical properties of materials with more accuracy and reliability, encouraging the development of new techniques. These thermophysical properties of materials can be simply defined as properties that vary as a function of the temperature, without changing their chemical identity.

Given this reality, there is a necessity of being studying techniques that improve the way to determine these properties in order to know how their effects may affect the lifespan of a project and its cost-benefit. Some of the most important thermophysical properties are the following: thermal conductivity, thermal diffusivity and specific heat.

The thermal conductivity (k) is the physical property of a material which indicates its ability to conduct heat. This property, which is classified as a transport property, basically provides an indication of the rate at which energy is transferred by diffusion and it depends on the physical structure of matter (Incropera *et al.,* 2008). On the other hand, the specific heat (c_p) can be defined as the amount of heat per unit mass required to raise the material's temperature of 1ºC (Bezerra Filho, 1998). Finally, the thermal diffusivity (α) is the relationship between the amount of energy that the environment can transfer (shown by thermal conductivity) and the amount of energy it can store. Precise knowledge of these properties is very important on heat conduction problems (Bezerra Filho, 1998).

Considering the importance of thermophysical properties in terms of mechanical design that involve heat transfer, it is known that there are currently a number of new metallic materials for which there are very few data cataloged of such properties, as is the case of Shape Memory Alloys (SMA).

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These SMA are metallic materials that have the ability to recover their original shape by heating above a critical temperature after being pseudo plastically deformed. This phenomenon is directly associated with a reversible phase transformation which occurs in the solid state by either varying the temperature or the mechanical load applied to the material. This structural transformation, directly related to the material's temperature, causes significant changes in many physical and mechanical properties of SMA (Otsuka and Wayman, 1998).

Thus, many potential applications for SMA involve heat transfer processes such as, for example, the application of these materials as sensor and/or actuators. Then, heat transfer processes are very important for developing SMA applications, which leads that the thermophysical properties of the material must be well characterized for the simulation of their thermal behavior when used in a thermomechanical device.

In this regard, this paper presents the design and construction of an experimental setup to obtain thermophysical properties of SMA, using the method of periodic heat flow that simultaneously allows the determination of thermal conductivity, thermal diffusivity and specific heat.

2. MATERIALS AND METHODOLOGY

2.1 Mathematical Modeling

The method chosen in this work to measure the thermophysical properties is known as periodic heat flow method, which was originally used for Angstrom *et al*., 1971. This method consists in setting a heat source whose temperature varies periodically as a function of time on one end of a cylindrical sample. The thermophysical properties are obtained from measurements of temperature fields recorded by thermocouples installed at different positions of the specimen length. Then, through a process of parameters identification one can obtain amplitudes, phases and average temperatures of the periodic signals provided by the thermocouples. These parameters are applied in a mathematical model to obtain the thermal conductivity and thermal diffusivity. The specific heat is then obtained from its definition using these two properties previously determined (Bezerra Filho, 1998).

In the method of periodic heat flow, the Biot number for the experimental conditions must be less than 0,1 (only metallic materials will be tested), which implies that the temperature in each cross section of the specimen is uniform and the process of heat conduction can be considered unidimensional.

Considering that the thermal properties are constant, the equation that governs the heat conduction problem is given as in Eq. (1).

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{\alpha}\frac{\partial T}{\partial t} \qquad 0 < x < L \quad e \quad t > 0 \tag{1}$$

where T is temperature (K), x is the spatial coordinate (m), L is the cylinder's length (m) and t is the time (s). The initial condition is given by Eq. (2).

$$T(x,0) = T_\infty \qquad 0 \le x \le L \tag{2}$$

where T_∞ is the cold source temperature (K).

On top of the sample (see detail of Figure 2), it was imposed a periodic heat flow with a specific frequency. This periodic flow is equal to the power dissipated by the heater, which can be seen in Eq. (6). Thus, one can have as a boundary condition at $x = 0$ the Eq. (3).

$$k\frac{\partial T(0,t)}{\partial x} = q_0 + q_0\cos(\omega t) \tag{3}$$

where q_0 is the heat flow (W/m^2), and $\omega = 2\pi f$ (f is the signal frequency in Hz).

At the lower end of the cylindrical specimen (see detail of Fig. 2), there is a heat transfer between the specimen and the Peltier cell whose temperature is constant. With these considerations, it is possible to write the following boundary condition at $x = L$, as follows in Eq. (4).

$$T(L,t) = T_\infty \tag{4}$$

The thermal modeling is then established by the set of Eq. (1), (2), (3) and (4) (Bezerra Filho, 1998).

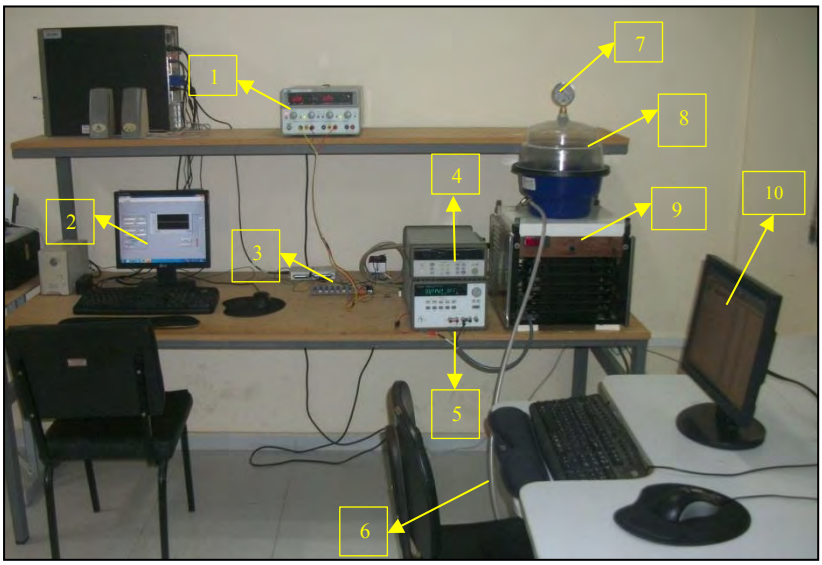
The temperature field solution of this model is the sum of three thermal fields: permanent (T_p), transient (T_t) and sinusoidal (T_s). As the time increases, the transient perturbation linked to the initial condition tends to zero as shown by Carslaw and Jaeger (1959) and the temperature field is the sum of two thermal fields: permanent and sinusoidal. The

ISSN 2176-5480

sum of these two fields is the periodic regime established. The solution to this problem is presented in more detail by Bezerra Filho (1998).

2.2 Experimental set up and procedures

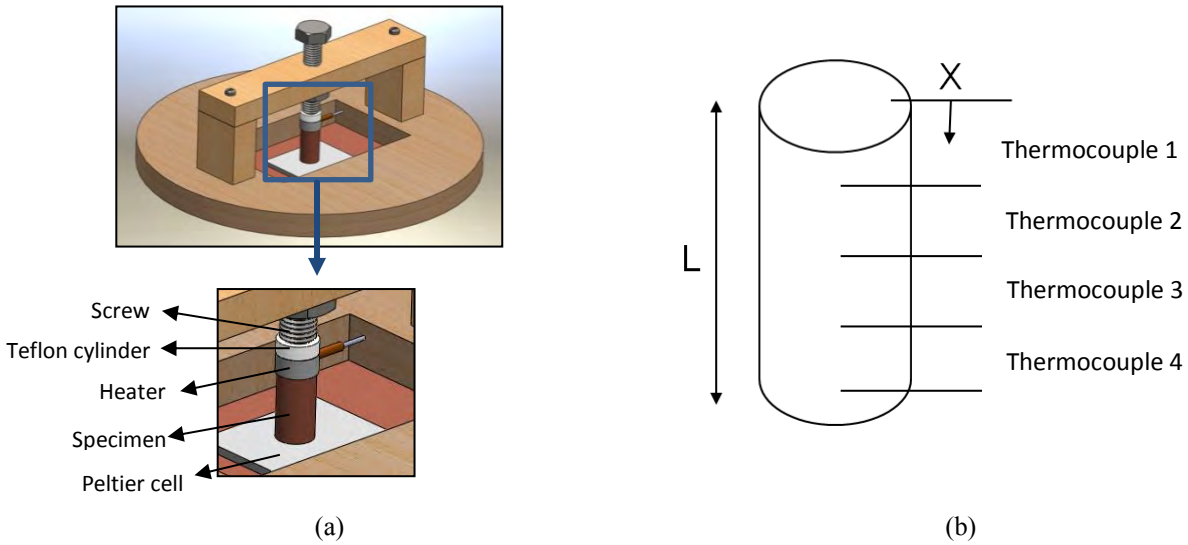
In order to undergo the metallic specimen to a periodic heat flow of known frequency and thereby identify the thermophysical properties, an experimental set up was developed by Reis (2010). At first this equipment was adapted for this work, due to the fact that its original function was to measure the transformation temperatures of SMA. The final apparatus can be seen in Figure 1. In this figure one can see all the components of the experimental set up. This test bench can be divided into four main systems: heating, data acquisition, cooling, vacuum, and specimen.



1- Peltier cell's power supply;
2- Peltier cell's program control;
3- Control circuits;
4- Data acquisition system;
5- Heater's power supply;
6- Vacuum hose;
7- Vacuum meter;
8- Container;
9- Equipment SMRT-Plus;
10- Heater's program control.

Figure 1. Experimental apparatus adapted to measure the thermophysical properties of metallic materials.

The container, Item 8 in Fig. 1, has the function of storing the cylindrical specimen during the experiment. To provide a good thermal contact between the specimen, the heater and the Peltier cell, the assembly showed in Fig. 2a has been employed. The Teflon cylinder is used as a thermal insulator. The distribution of the K thermocouples on the surface of the cylindrical specimen can be seen in Fig. 2b.



(a) (b)

Figure 2. Details of the cylindrical specimen. (a) Specimen Fastening. (b) Distribution of thermocouples.

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The heating system consists of an electrical resistance of 6.3 Ohms, indicated as heater in the detail of Fig. 2, and a power supply (Item 5 in Fig. 1). The periodic heat flow with known frequency is produced by a stabilized power supply controlled by a computer program (item 10 in Fig. 1). This program commands a rectified voltage to the heating system whose value is given by Eq. (5).

$$V = V_m \left|\cos(w_e t)\right| \tag{5}$$

where V_m is the amplitude of voltage (volts), w_e is the electrical frequency (rad/s) and t is the time (s). This voltage gives a regular electrical power Pe (W) in the heater given by Eq. (6).

$$P = P_m(1+\cos(wt)) \tag{6}$$

where P_m is the average power (W) and w is the thermal frequency (rad/s), which is twice the electrical frequency.

The periodic heat flow with known frequency is produced with the power supply shown in Fig. (1), controlled by a computer program made in Microsoft Office Excel using a sequence of commands (macros). In this program, the desired number of cycles for each experiment can be established by the user.

The apparatus presented in Fig. (1) uses a cooler based on Peltier effect (Peltier cell), indicated in the detail of Fig. (2), as cooling system of the specimen. For this application, the Peltier cell temperature was kept constant at 0 °C (273K), so that its control was carried out by a program developed in LabView 8.2 by Reis (2010). The temperature of the Peltier cell is kept constant with variations of ± 0.015 ° C during the experiment.

The data acquisition system used a computer program to display the collected data (Item 10 in Fig. 1) and a data acquisition system (item 4 in Fig. 1) Agilent, model 34970A equipped with a multiplexer module of 20 channels accurately reading up to 6 ½ digits. For the vacuum system of the container one could use a vacuum pump (not shown) coupled to the hose shown in the item 6 in Fig. (1).

2.3 Specimens manufacturing

To carry out the tests there were prepared five specimens, two of them are known materials that have their thermophysical properties provided by the manufacturer (electrolytic copper, stainless steel and yellow brass), and three samples of SMA. The cylindrical specimens had the dimensions showed in Tab. (1). There was also a process of polishing the specimens face in order to reduce the thermal contact resistance. The very careful preparation of specimens is necessary to obtain results that can be compared with those provided by the manufacturer.

Table 1. Dimension of samples.

Diameter (mm)	12,70 ± 0,02
Length (mm)	25,00 ± 0,02

The SMA specimens were fabricated by a plasma melting process followed by injection into a metallic mold (Plasma Skull Push Pull - PSPP), proposed by De Araújo *et al.*, 2009. The nominal chemical composition of the fabricated SMA cylindrical specimens and transformation temperatures can be seen in Tab. (2). All these specimens also were undergone to cutting and polishing processes to improve the surface finish. After the PSPP process, the obtained product was heat-treated at a temperature of 1123 K (850 °C) during 900 s (15 minutes) with subsequent water quenching. This heat treatment is intended to ensure uniformity of material properties.

Table 2. Chemical composition and transformation temperatures of SMA specimens.

SMA specimen code	**Composition (% Weight)**	**Transformation temperatures (^{o}C) (De Araújo *et al.*, 2009)**
Specimen 1	55Ni-45Ti (Otsuka *et al.*, 2005)	M_s = 29.2, M_f = 18.6, A_f = 52.6
Specimen 2	82,5Cu-13,5Al-4Ni (Otsuka & Wayman, 1998)	M_s = 83.0, M_f = 69.1, A_s = 80.1 , A_f = 90.9
Specimen 3	48Ni-38Ti-14Nb (Zhao *et al.*, 2006)	M_s = -23.1, M_f = 48.0, A_s = -7.2 , A_f = 23.6

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2.4 Test procedures

For these thermal experiments previously mentioned, at first there were welded four thermocouples (K type with diameter of 100 μm) at the specimen surface, one at the Peltier cell and another at the heater. Thus, each sample is set as shown in Fig. (2).

Before the test, it was used a thermal grease on the specimen's edges to reduce the contact resistance between specimen, heater and Peltier cell. After the assembly, the container is closed and vacuum is performed to prevent the heat transfer by convection. This vacuum was of the order of 540 mmHg. Following, the Peltier cell is turned on via Labview and after some time maintains its temperature constant at 0 ° C (273K). The data acquisition system is also switched on and then the program that controls the heater. In this program, the heater's voltage was regulated to 10 volts, the wave period to 312. In this way, one could obtain an output power of approximately 19 W. Once turned on the heater, the temperature status takes at about 45 minutes to reach the steady-state and then the test is stopped.

To identificate the thermophysical properties, the temperature data are treated using identification process of the average temperatures, amplitudes and phase of the thermal response signal. This process consists in to fit the obtained curves by the tests to the model's equation (Eq. 7).

$$T = T_m + A \times \cos(\omega t - \psi) \tag{7}$$

where T_m is the signal average temperature, A is the signal amplitude and ψ is the signal phase.

3. RESULTS AND DISCUSSIONS

3.1 Temperature profiles

At first the tests were carried out on specimens made of commercial materials (copper and brass), and then with the others specimens made of SMA. The results for the temperature profiles of the commercial brass can be seen in Fig. (3).

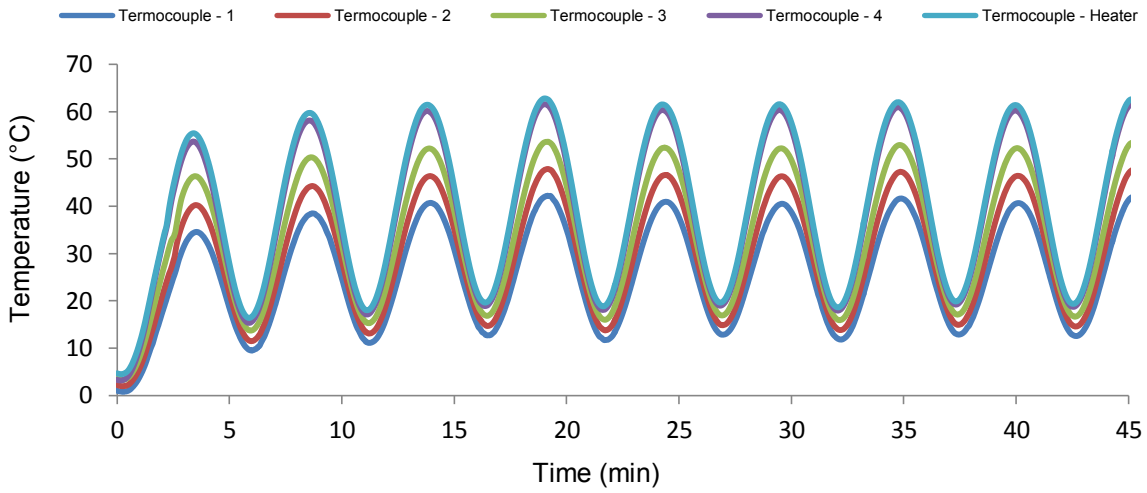

Figure 3. Temperature profiles in different positions of a brass specimen submitted to a periodic heat flow.

3.2 Calculation of thermophysical properties

From the temperature profiles obtained for all specimens one could perform the identification process of average temperatures, amplitudes and phases of the thermal signals. The average temperature obtained for a brass sample is shown in Fig. (4), depending on the position of each thermocouple.

This identification process was performed by adjusting the curves through the program Origin 8.0 and with these data one could use the software Wolfram Mathematica 7 to solve the mathematical model proposed by Bezerra Filho (1998) to determine the thermophysical properties.

To calculate the thermal conductivity (k), the average temperatures of the signal were used to solve the Eq. (3). For the thermal diffusivity (α) there were used the equations which are based on the amplitude and the phase of the thermal signal, respectively. Then, the specific heat (c_p) is calculated by its definition in Eq. (8).

$$c_p = \frac{k}{\rho \alpha} \tag{8}$$

ISSN 2176-5480

where ρ is the specific mass of the material in Kg/m³.

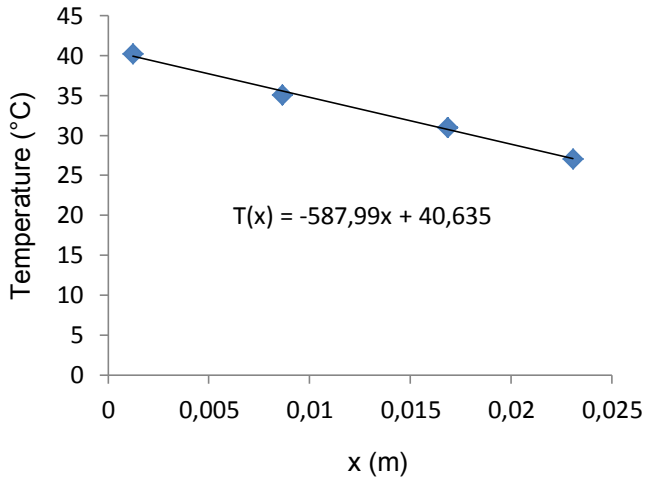

Figure 7. Average temperatures as a function of each thermocouple position for a brass specimen.

For the tested specimens, the results obtained are shown in Tab. (3) for materials commercially purchased and in Tab. (4) for the manufactured SMA.

Table 3. Thermophysical properties of specimens commercially purchased.

Yellow Brass	***Experimental***		***Manufacturer***	***Error (%)***	
	Amplitude	Phase		Amplitude	Phase
Thermal diffusivity (m^2/s)	33.6×10^{-6}	31.8×10^{-6}	35.3×10^{-6}	4.8	6.2
Specific heat (J/kg.K)	447.9	473.2	401.0	11.7	18.0
Thermal conductivity (W/m)	127.5		120.0	6.3	
Stainless Steel (AISI 304)	***Experimental***		***Manufacturer***	***Error (%)***	
	Amplitude	Phase		Amplitude	Phase
Thermal diffusivity (m^2/s)	3.77×10^{-6}	3.25×10^{-6}	3.76×10^{-6}	0.30	13.80
Specific heat (J/kg.K)	479.0	556.0	502.4	4.7	10.7
Thermal conductivity (W/m)	14.3		15.1	5.4	

As pointed out in Tab. (3), the thermal conductivity results obtained for yellow brass and stainless steel had a difference of 6.3% and 5.4%, respectively, compared to the properties provided by the manufacturer. When considering thermal diffusivity, calculated from amplitude values, such difference becomes 4.8%, for brass, and 0.3% for stainless steel. The diffusivity values obtained from the phase signal showed a greater error than those from amplitude values. This was attributed to a greater sensitivity of the mathematical model to those values. Overall, the results are within an acceptable margin of error for such properties when comparing the experimental results with those provided by the manufacturer.

Regarding to the results for the SMA specimens shown in Tab. (4), also considering the transformation temperatures summarized in Tab. (2), one can say they were obtained for the austenite phase for specimen 3 and during phase transformation (martensite to austenite) for specimens 1 and 2, since the temperature range of the experiment was from 20 °C (293K) to 130 °C (403K). Comparing the experimental results for the thermal conductivity of SMA with those cataloged by Faulkner *et al.*, 2000 and Otsuka & Wayman (1998), it has been noticed that the NiTi (specimens 1 and 3) and CuAlNi (specimen 2) present a difference of 4.3 %, 5.0 % and 6.6 %, respectively. These differences might be associated with the process used to manufacture the specimens, since slight variations on the SMA chemical composition may lead to considerable variations on the SMA properties (Otsuka & Wayman, 1998).

ISSN 2176-5480

In general, there are few published studies concerning measurements of these thermophysical properties in SMA. For example, Schlosser *et al*., 2007 and Favier *et al*., 2007 assumed c_p and k to be constant in studies carried out with polycrystaline NiTi SMA. However, Faulkner *et al*., 2000 have shown substantial differences in c_p and k for austenite and martensite. The differences seem to be greater for the thermal conductivity. By working with a polycrystalline NiTi SMA wire, Faulkner *et al*., 2000 obtained a ratio of two between the thermal conductivity of austenitic (k_A) and martensite phases (k_M) , i.e. k_A = 28 W/m.K and k_M = 14 W/m.K. Specific heat values given in the literature for NiTi and CuAlNi SMA are also scattered, but in our study we decided to take a mean specific heat for the two SMA and two phases calculated from the data summarized by Otsuka & Wayman, 1998: c_p=450 J/kg.K.

The obtained results in this work showed a certain differences when estimating the thermal diffusivity either using the amplitudes values or the phase signal. It can also be noticed that the value depends on the thermocouple used as reference. These differences can be explained by the fact that the thermocouples that are close to the heater suffer influence of a temperature field that is not one-dimensional due to its construction. As it moves away from the heater, there is a tendency of approaching to the estimated results. Hence, the calculation of the diffusivity was done with an average of thermocouples that showed the diffusivity values close to each other.

For the thermal diffusivity, and consequently for specific heat, of the NiTi the errors were not included in Tab. (4) because the literature values (c_p=450 J/kg.K) are not reliable for these SMA. Considering that the values of c_p for conventional materials presented in Tab. (3) and for CuAlNi SMA in Tab. (4) were in accord with literature, we consider that our results for thermal diffusivity and specific heat values of NiTi SMA are correct.

Table 4. Thermophysical properties of manufactured SMA specimens.

Specimen 1 - 55Ni-45Ti	***Experimental***		***Literature*** [3]	***Error (%)***	
	Amplitude	Phase		Amplitude	Phase
Thermal diffusivity (m^2/s)	17.1×10^{-6}	21.0×10^{-6}	10.8×10^{-6}	---	---
Specific heat (J/kg.K)	199.2	162.5	450	---	---
Thermal conductivity (W/m.K)	21.9		21.0	4.3	
Specimen 2 - 82.5Cu-13.5Al-4Ni	***Experimental***		***Literature*** [4]	***Error (%)***	
	Amplitude	Phase		Amplitude	Phase
Thermal diffusivity (m^2/s)	16.4×10^{-6}	18.6×10^{-6}	16.5×10^{-6}	0.6	12.7
Specific heat (J/kg.K)	448.3	394.7	450	0.4	12.3
Thermal conductivity (W/m.K)	52.6		49.3	6.6	
Specimen 3 - 48Ni-38Ti-14Nb	***Experimental***		***Literature*** [4]	***Error (%)***	
	Amplitude	Phase		Amplitude	Phase
Thermal diffusivity (m^2/s)	17.4×10^{-6}	20.5×10^{-6}	6.6×10^{-6}	---	---
Specific heat (J/kg.K)	164.8	139.6	450	---	---
Thermal conductivity (W/m.K)	18.9		18.0	5.0	

(3) Average value of k from Faulkner et al., 2000; (4) Average values of k and c_p from Otsuka & Wayman, 1998.

In a general way, the results obtained for the thermophysical properties of the tested materials exhibited a good convergence, regarding mainly the results for commercial materials, as stainless steel and brass, validating the employed technique for the measurements with SMA. Some perceived discrepancies might be related to:

- Thermal contact resistance between the specimen and heater and between the specimen and Peltier cell;
- Thermal contact resistance between thermocouples and specimens;
- Heat transfer by radiation;
- Curve fitting performed to obtain the parameters;
- Sensitivity of the mathematical model to the parameters, particularly the equation that uses the signal phase;
- Existence of a two-dimensional temperature field near the heater.

ISSN 2176-5480

4. CONCLUSION

The technique of indirect measurement of thermophysical properties through a periodic heat flow signal was proposed in this work to determine thermal diffusivity, thermal conductivity and specific heat of conventional metallic materials and shape memory alloys. In general, accurate results were obtained when comparing with values provided by manufacturers and literature.

Through this method one can identify the thermal diffusivity either using amplitude or thermal signal phase values. The large amount of results that were calculated for the thermal diffusivity, varying only the reference thermocouple, allows one to have information about the measurement error. The discrepancies found in the calculation of diffusivity are related to the sensitivity of the model to the data that enter into it, mainly in the equation that uses the phase signal.

In relation to the thermal conductivity, the small differences observed occur mainly due to the heat loss related to contact resistance between the heater and specimen and the specimen and Peltier cell. Errors in the specific heat are associated with the accuracy of the other two properties.

Overall, it was possible to design, assembly and validate a experimental test bench for indirect measurement of thermophysical properties of SMA, for which there are not many literature data. Based on the results obtained for the tested conventional metals, it is believed that the results obtained for the thermophysical properties of SMA are very close to the real ones, considering the found errors.

5. ACKNOWLEDGEMENTS

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