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THERMAL DIFFUSIVITY IDENTIFICATION OF NICKEL-TITANIUM SMA USING A PERIODIC TEMPERATURE FIELD

José Ricardo Ferreira Oliveira
Federal University of Uberlandia, Faculty of Mechanical Engineering, Experiment and Modelling Heat Transfer Laboratory. João Naves de Ávila Avenue, 2121, Zip Code 38900-402, Uberlandia City, Minas Gerais State, Brazil.
jose.ricardo@ufu.br

Luiz Roberto Rocha de Lucena
Federal University of Campina Grande, Department of Mechanical Engineering, Experimental Laboratory of Heat Transfer and Fluid Mechanics. Aprigio Veloso Street, 882, Zip Code 58429-140, Campina Grande City, Paraiba State, Brazil.
luiz.roberto@ufcg.edu.br

Rômulo Pierre Batista dos Reis
Federal University of Campina Grande, Department of Mechanical Engineering, Multidisciplinary Laboratory of Materials and Active Structures. Aprigio Veloso Street, 882, Zip Code 58429-140, Campina Grande City, Paraiba State, Brazil.
romulopierre@ufersa.edu.br

Carlos José de Araújo
Federal University of Campina Grande, Department of Mechanical Engineering, Multidisciplinary Laboratory of Materials and Active Structures. Aprigio Veloso Street, 882, Zip Code 58429-140, Campina Grande City, Paraiba State, Brazil.
carlos.araujo@ufcg.edu.br

Celso Rosendo Bezerra Filho
Federal University of Campina Grande, Department of Mechanical Engineering, Experimental Laboratory of Heat Transfer and Fluid Mechanics. Aprigio Veloso Street, 882, Zip Code 58429-140, Campina Grande City, Paraiba State, Brazil.
celso.rosendo@ufcg.edu.br

Abstract: *Shape Memory Alloy (SMA) is a kind of metalic materials that changes his mechanical properties when induced by temperature (shape memory effect) or strain (superelastic effect). These characteristics make SMA´s have applications in several projects in areas such as control and automation, automotive, aeroespace and biomedical. In these applications, the phenomenea linked to heat transfer process play a fundamental role. Thermal diffusivity is a very important thermophysical property in the analysis of thermal energy diffusion problems. This work had the purpose of determining this property in a Nickel-Titanium SMA using a periodic heat field. For this, an experimental device was build whose principle of operation based on the Angstrom´s Method, which makes use of a periodic heat flux in the sample, generating in this a periodic heat flow. Thermocouples were installed in the sample to capture the temperature signals generated by the periodic heat flow. Amplitude and phase of these signals were obtained by graphical analysis software. The thermocouple closest to the heat source was adopted as reference, whereas the ratio of amplitudes and the phase between the signals recorded by the others thermocouples in relation to that thermocouple were calculated. These results were used in mathematical models to identify the thermal diffusivity, whose value found, when compared with the values available in the literature, obtained a good agreement, considering the range of uncertainty adopted.*

Keywords: *Thermal Diffusivity, Thermophysical Properties, Shape Memory Alloys, Angstrom´s Method, Experimental Device.*

1. INTRODUCTION

A consequence of the rapid and perennial occurrence of technological advancement is the development of a class of new materials known as intelligent materials, capable of exerting functions of sensors and actuators [1]. Among these materials are the shape memory alloys (SMA), which are metallic materials with the ability to recover their original shape at certain characteristic temperatures (shape memory effect), even under high applied loads and large inelastic deformations, or undergo great stress without plastic deformation (superelasticity) [2]. These peculiarities make SMAs a unique kind of material chosen for several projects, in areas such as control and automation, automotive, aerospace and biomedical [3]. In these projects, the processes of heat transfer play a fundamental role, which motivates the growing number of researches and development of new techniques to identify thermophysical properties of SMA [4]. In this context, the techniques called periodic techniques are of great importance in measurements at low temperatures [5]. A

usual periodic technique is that proposed in 1861 by swedish physicist Anders Jonas Angstrom (1814-1874), which used a periodic heat flow over the test sample, causing a periodic temperature field in this sample.

An extremely important thermophysical property is thermal diffusivity. It reflects the ratio of the energy that a medium can carry by the diffusion process and the energy it can store, that is, thermal diffusivity shows how quickly heat can propagate in a given material.

The objective of this work is thermal diffusivity experimental identification of Nickel-Titanium SMA and compare the results obtained with the values available in the literature.

SIMBOLOGY

Symbol	*Description*	*Unit*	*Symbol*	*Description*	*Unit*
A	*Amplitude model*	-	T_m	*Average temperature*	*ºC*
A_f	*End of austenitic transformation*	*ºC*	x_n	*Thermocouple position in the sample*	*m*
A_s	*Start of austenitic transformation*	*ºC*	α	*Thermal diffusivity*	*m²/s*
L	*Sample length*	*m*	ε	*Phase angles reference*	*rad*
R_f	*End of phase R transformation*	*ºC*	Ψ	*Phase angle model*	*rad*
R_s	*Start of phase R transformation*	*ºC*	μ	*Uncertainty of thermal diffusivity*	*m²/s*
t	*Time*	*s*	σ	*Deviation from the literature*	-
T	*Temperature*	*ºC*	ω	*Thermal frequency*	*rad/s*

2. MATHEMATICAL MODEL

In order to arrive at the mathematical model that represents the physical system of this work it is admitted that the experiments are carried out with the use of thermal insulation in contact with the lateral area of the sample, thus increasing the radial thermal resistance, ensuring that the heat flow occurs in the axial direction. In this case, the Biot number would assume values much smaller than 1 [6] for the experimental conditions in question, implying, therefore, that the temperature in each section of the sample is uniform during the transport of energy, so that the conduction heat transfer along the sample is one dimensional. It is further assumed that the thermal diffusivity variation with temperature is negligible and that the medium is isotropic with constant properties. Thus, considering a sample of length L, the mathematical model, the initial condition and the conditions of the problem are given respectively by:

$$\frac{\partial^2 T}{\partial x^2} = \frac{1}{\alpha}\frac{\partial T}{\partial t} \tag{1}$$

$$T(x,0) = 0\,; \quad 0 \le x \le L \tag{2}$$

$$T(L,t) = A(L)\sin(\omega t + \varepsilon) \tag{3}$$

$$T(0,t) = 0 \tag{4}$$

A periodic heat flux with a given frequency was imposed on the upper part ($x = L$) which causes a periodic temperature field in the sample. The lower end was maintained at a constant temperature by being in contact with a fluid at which the temperature was controlled through a thermoregulator bath. The solution of this model was proposed by [5], and can be seen in Eq. (5):

$$T(x,t) = A(x)\,sin(\omega t + \varepsilon + \Psi) + 2\pi\alpha \sum_{n=1}^{\infty} \frac{n(-1)^n[\alpha n^2\pi^2\,sin(\varepsilon) - \omega L^2\,cos(\varepsilon)]}{\alpha^2 n^4\pi^4 + \omega^2 L^4} sin\left(\frac{n\pi x}{L}\right) e^{-\left(\frac{\alpha n^2\pi^2 t}{L^2}\right)} \tag{5}$$

Alternatively, periodic permanent temperature field solution was obtained when this regimen was reached [5]. For this, it is assumed that the transient variations of the temperature field cease when time increases (the transient term of Eq. (5) disappears when $t \to \infty$) such that in long times the condition of periodic regime is reached permanent. This solution is given by Eq. (6), whose deduction is in [4].

$$T(x,t) = A(x)\,sin(\omega t + \varepsilon + \Psi) \tag{6}$$

$$A = \left|\frac{sinh[x\beta(1+i)]}{sinh[L\beta(1+i)]}\right| = \left[\frac{cosh(2\beta x) - cos(2\beta x)}{cosh(2\beta L) - cos\,2\beta L}\right]^{1/2} \quad (7)$$

$$\Psi = arg\left\{\frac{sinh[x\beta(1+i)]}{sinh[L\beta(1+i)]}\right\} \quad (8)$$

$$\beta = \sqrt{\frac{\omega}{2\alpha}}$$

3. METHODOLOGY

3.1 Experimental device

Experimental device was designed and constructed to thermal diffusivity identification using a periodic temperature field. The device shown in Fig. 1 is comprised of the data acquisition, heating, cooling, vacuum, sample and test device systems.

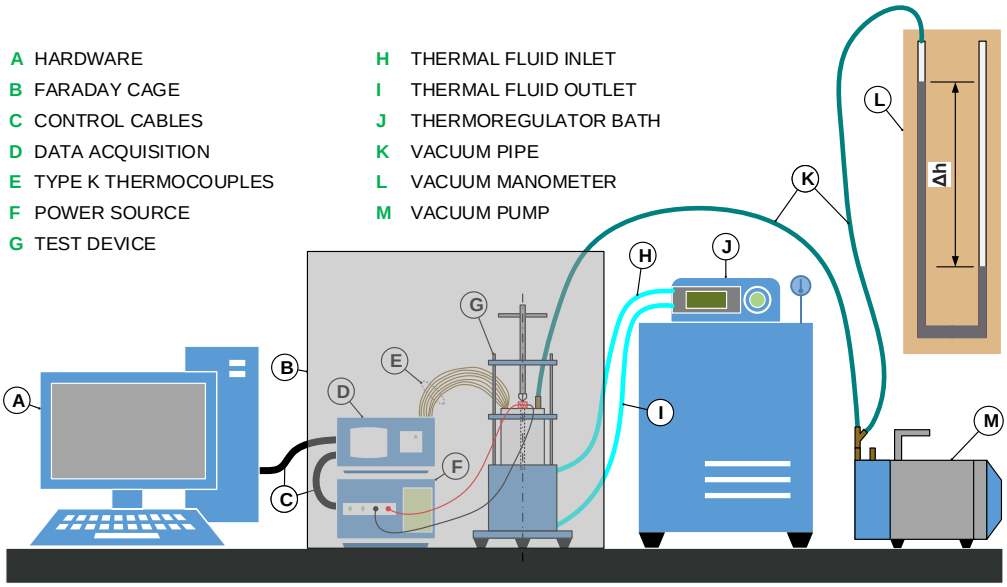

Figure 1. Experimental device

3.2 Sample Manufacturing

Figure (2) shows Nickel-Titanium sample manufacturing process. Mass quantities of nickel and titanium, both of high purity, were selected to form two melt fillers, each with 15g. Through the plasma skull push-pull process [7], performed on the Discovery All Metal machine, these loads were melted and turned into knobs, which were cast and injected into a Power Cast 1700 machine, which uses the induction melt process with centrifugal injection. The material being injected into a solid ceramic coating mold, the cavity of which is the desired cylindrical geometry. Due to the size limitation that can be used in the Power Cast 1700, the sample had a final dimension of 12,7 x 10^{-3} m in diameter and 24,5 x 10^{-3} m in height, disregarding the branch inherent in the casting process. Injected product was removed from the mold and placed in a metallographic cutter in order to separate the sample from the branch. Sample and the branch were subjected to two heat treatments: in the first, they were submitted to a temperature of 850 °C for 1 hour, in a vacuum furnace, and then tempered in water; in the second treatment, were subjected to a temperature of 550 °C for 2 hours, and then tempered in water. After the heat treatments, the lower and upper faces of the NiTi sample were sanded and the upper face was also polished, in order to reduce the Thermal Resistance of Contact between this face and the electric resistance (utilized for heating of that sample). DSC (Differential Scanning Calorimetry) is a thermal characterization process, was used to identify the phase transformation temperatures of that sample. This process results in the curve shows in Fig. 3.

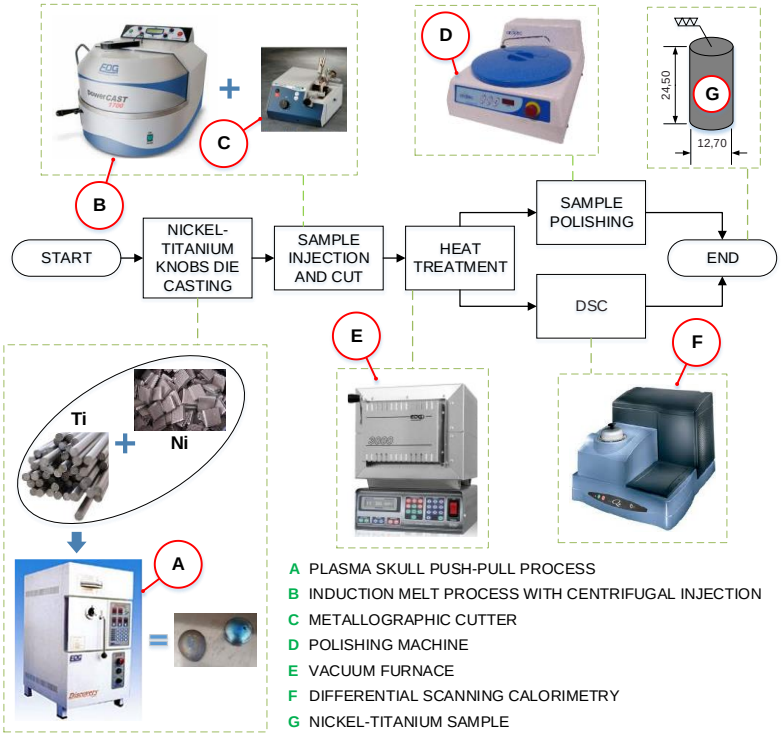

Figure 2. Ni-Ti sample manufacturing process

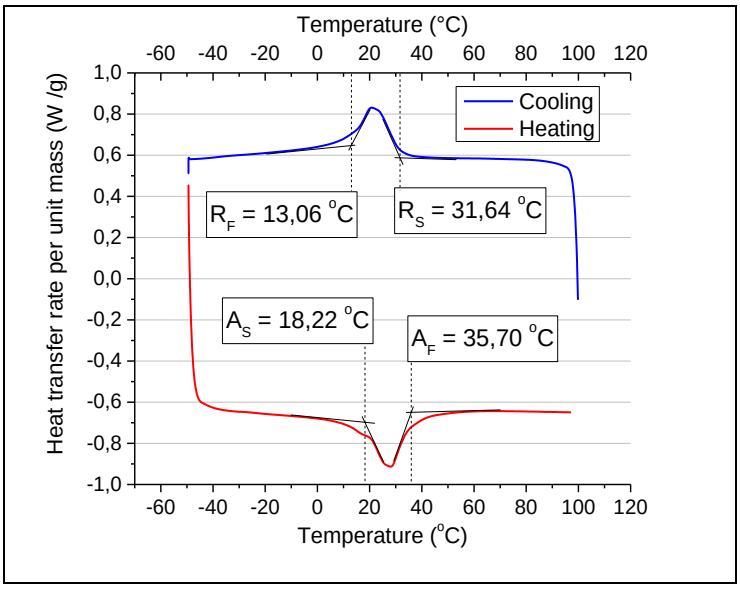

Figure 3. DSC curve of NiTi sample

According to Fig. 3, the sample presented two distinct phases in the temperature range comprised by the DSC. The austenitic phase, which may occur during heating, showed the beginning and end temperatures of phase transformation 18,22 °C and 35,70 °C, respectively, and the R phase, a martensitic variant, which may run during cooling and had the phase transformation start and end temperatures 31,64 °C and 13.06 °C, respectively. The achievement of such temperatures aims to identify the temperature ranges where the sample consists of only one phase, making possible the comparison of thermal diffusivity values.

3.3 Thermocouple installation and calibration

Fourteen type K thermocouples, 0,1 x 10^{-3} m in diameter, were installed along the sample, according to Fig. 4.

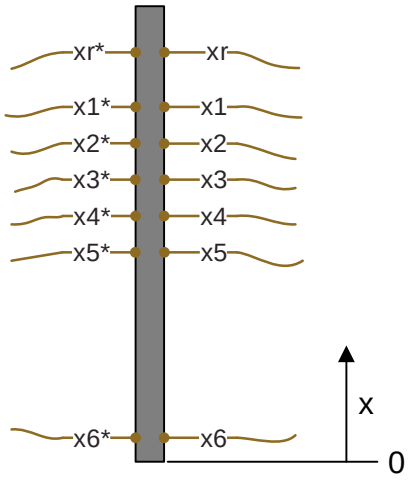

Figure 4. Thermocouples identification and arrangement in the sample

Thermocouples locations were defined based on sensitivity coefficient studies. The technique used to fix the thermocouples was capacitive discharge welding. In order to minimize the errors of the temperature values to be measured, calibration procedures of the installed thermocouples are performed before the experiments for thermal diffusivity identification. These procedures, which were carried out in a metallic container, initially with melting ice and then with boiling water according to Fig. 5, consist of the lifting of thermocouples calibration curves, which were used to correct the temperature measured [in the thermal diffusivity identification experiments] in real temperature values.

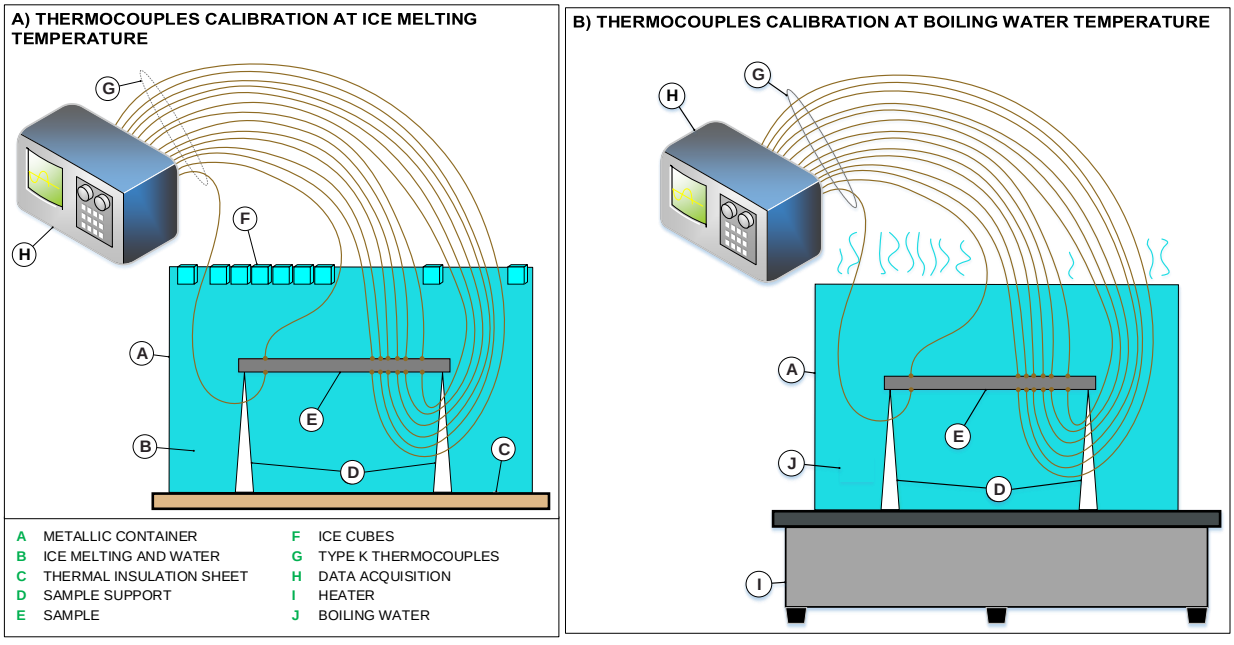

Figure 5. Thermocouples calibration

4. RESULTS AND DISCUSSIONS

From the six experiments that were performed, in 3 of them, the temperatures of the sample were below A_s and in the other 3 above A_f, aiming to thermal diffusivity identification of the R (martensitic variant) and austenitic phases, respectively, from the values of A or Ψ of the thermal signals captured by thermocouples. The values of ω: 21,9 x 10^{-3}, 30,7 x 10^{-3} and 51,1 x 10^{-3} rad/s were adopted. Figure 6 shows the temperature profile due to the periodic heat flux, for ω = 30.7 x 10^{-3} rad/s and temperatures of the Permanent Periodic Regime above A_f. The values of A and Ψ were identified from the moment at which the transient perturbation connected to the initial condition is dissipated. Such instant was termed as the initial instant (t = 0 s) of the periodic permanent regime. Figures 7 and 8 show the values of A versus ω for the R and austenitic phases, respectively. Figures 9 and 10 show values of Ψ versus ω for the R and austenitic phases, respectively.

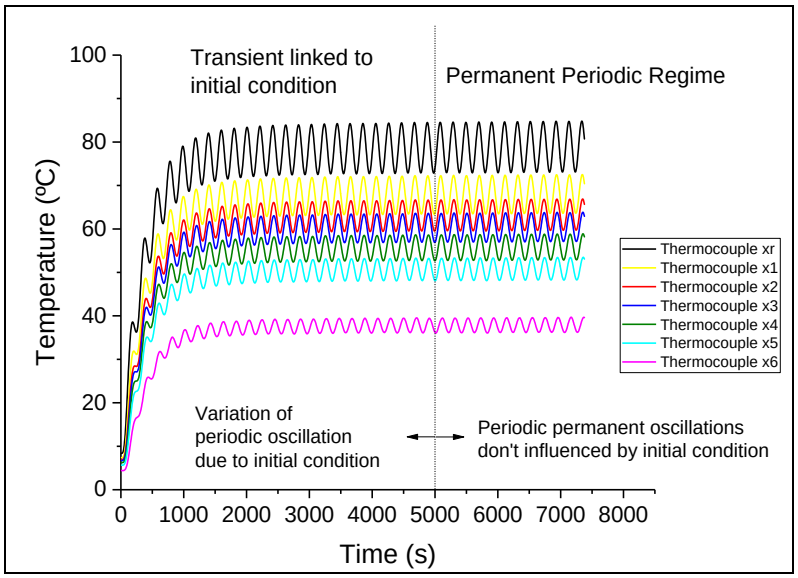

Figure 6. Sample temperature profile due to a periodic heat flow

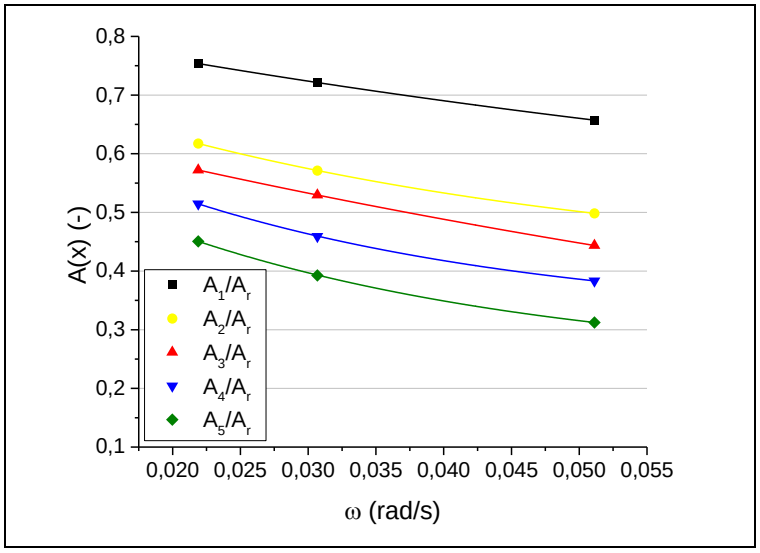

Figure 7. A(x) *versus* ω; R phase

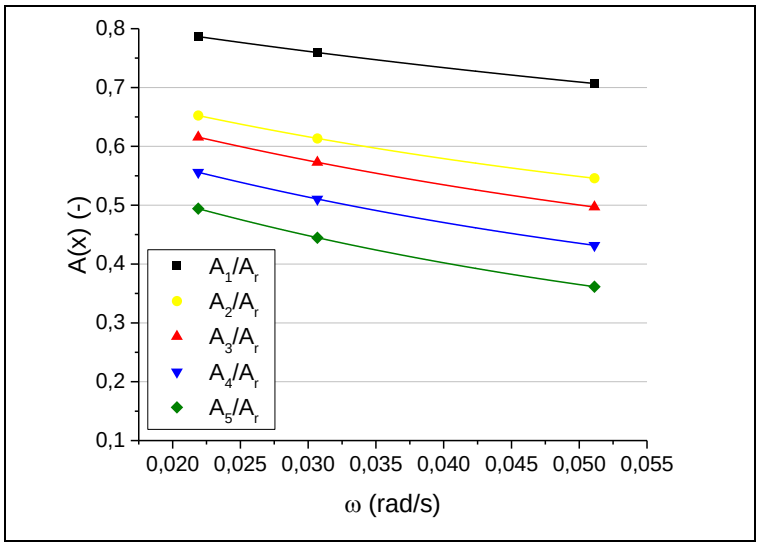

Figure 8. A(x) *versus* ω; Austenitic phase

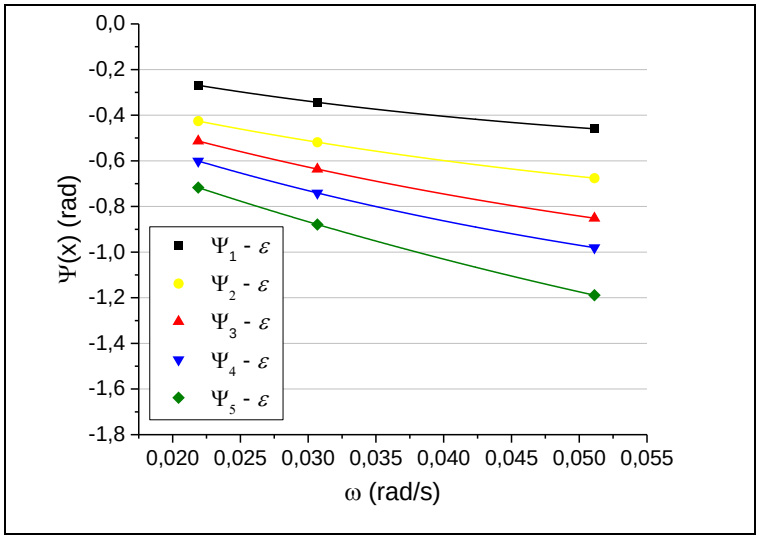

Figure 9. Ψ(x) *versus* ω; R phase

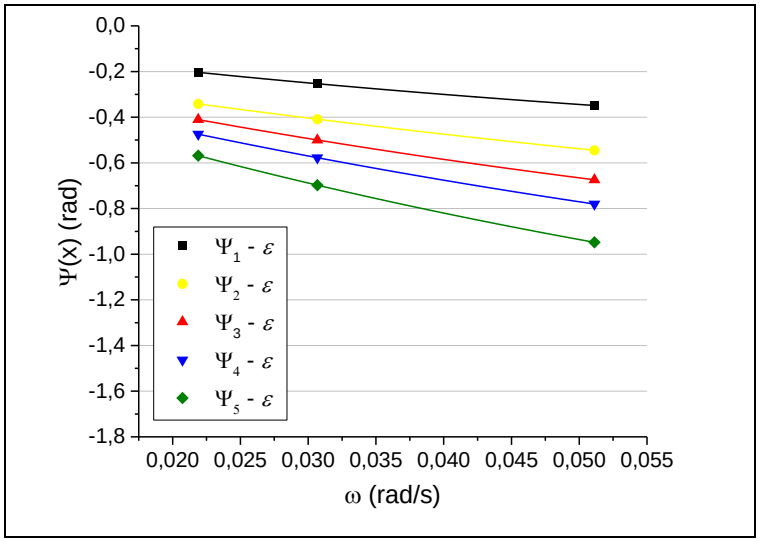

Figure 10. Ψ(x) *versus* ω; Austenitic phase

The temperature curves shown in Fig. 6 prove what was shown by [5] for the resulting temperature field in a sample subjected to a periodic heat flux: as time increases, the transient perturbation is dissipated, and thermal field becomes a field with permanent periodic oscillations. The "Permanent Periodic Regime" is then constituted, and for the case shown in Fig. 6, such condition is reached after 5000 s from the beginning of the experiment.

The graphs obtained for $A(x)$ versus ω, Fig. 7 and 8, show that the amplitude of the model decreases exponentially with the increase in thermal frequency, whereas the curves obtained for $\Psi(x)$ versus ω, Fig. 10, show that the lag between the temperature signals increases exponentially with increasing thermal frequency. Such behaviors were presented by [5] and obtained experimentally by [8].

The values of A and Ψ of each experiment, determined from the relation of the temperature profiles captured by thermocouples x_1 to x_5 in relation to those captured by thermocouple x_r, were inserted in their respective models, Eq. (7) and (8). Thus, for each experiment, 5 thermal diffusivity values through Eq. (7) and 5 values were identified through Eq. (8). From these values, according to [9], the mean value of α and the standard uncertainty μ (α) were obtained. In the graphs of Figs. 11 and 12, thermal diffusivity average identified for the R and austenitic phases, respectively, as a function of ω, are presented as well as the standard uncertainties described in the upper part of each bar.

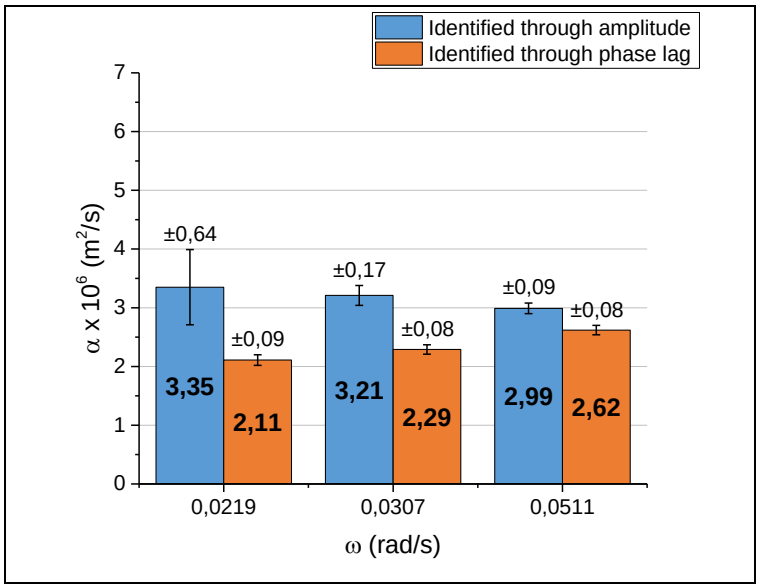

Figure 11. NiTi SMA thermal diffusivity (R phase)

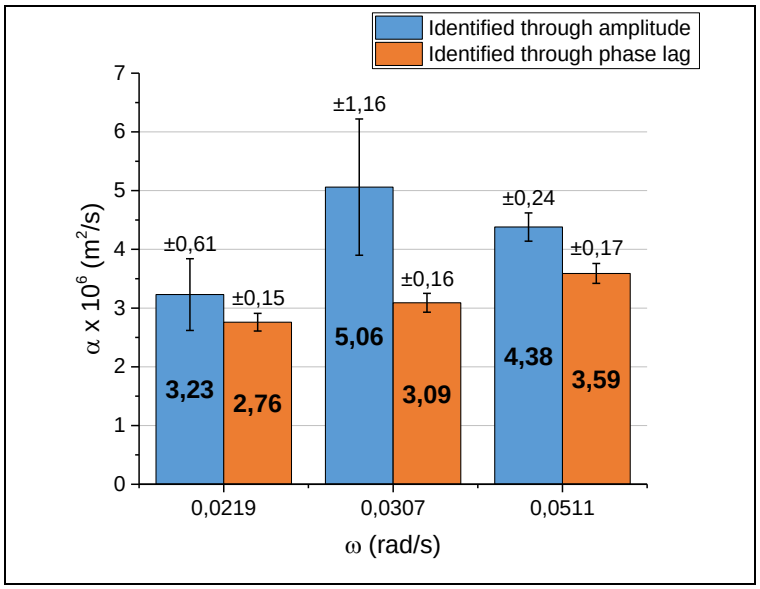

Figure 12. NiTi SMA thermal diffusivity (austenitic phase)

Figure 11 and Figure 12 shows thermal diffusivity mean values of R and austenitic phases. These are identified by both the amplitude and the phase lag, which are closer to the values available in [10]. With respect to the values identified from the amplitude model, for both the R and the austenitic phases, the values closest to those of [10] were obtained at a thermal frequency of 51,1 x 10^{-3} rad/s. In the same way as those obtained from the phase lag model. Table 1 shows the comparison between such values.

Table 1. NiTi SMA thermal diffusivity values comparison

NiTi SMA	$[\alpha_A \pm \mu(\alpha)]$ x10^6 (m^2/s)	$[\alpha_\Psi \pm \mu(\alpha)]$ x10^6 (m^2/s)	[10]	σ_A (%)	σ_Ψ (%)
R phase	2,99 ± 0,09	2,62 ± 0,08	3,11	-3,85	-15,76
Austenitic phase	4,38 ± 0,24	3,59 ± 0,17	4,72	-7,20	-23,94

It can be seen from Tab. 1 that thermal diffusivity values obtained in the present work, both in the R phase and in the austenitic phase, with smaller percentage deviation in relation to the values of [10], were identified from the amplitude model, Eq. (7). These values were obtained on the thermal frequency of 51,1 x 10^{-3} rad/s, as above mentioned.

Playing the trend observed for [11] and [12], average thermal diffusivity of the austenitic phase NiTi alloy is about 40% higher than the R phase (martensite variant) according to the present results.

Thermal diffusivity variation with the temperature increase can be considered negligible, since the values of maximum temperature variation in each experiment are small. Also, it can be stated that thermal diffusivity varies according to the internal structure of the material, since there is a considerable difference between the values identified of this thermal property for the two phases presented by the NiTi SMA, which have the same chemical composition, but different internal structures.

5. CONCLUSIONS

When thermal diffusivity identified values of the R and austenitic phases were compared to each other, it was found, just as [11; 12], that austenitic phase thermal diffusivity is about 40% greater than that of the phase with martensitic structure. This, according to [12], is due to the fact that the mean free path between the electrons and the phonons (vibrational energy of the crystalline lattice) is much larger in the austenitic phase. That is, the electrical resistivity of austenite is much smaller than that of the martensitic structure. It can also be concluded that thermal diffusivity mean values identified in this research for the NiTi SMA in the R and austenitic phases are within an acceptable range of uncertainty when compared to those available in [10].

6. ACKNOWLEDGEMENTS

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