24th COBEM - 2017

24th ABCM International Congress of Mechanical Engineering
December 3-8, 2017, Curitiba, PR, Brazil

COBEM-2017-0330
STUDY OF THE DYNAMIC PROPERTIES OF A SMA MECHANICALLY SHAPED BY HOT ROLLING AND ECAE

Pedro Sabino dos Santos Barbosa
Niédson José da Silva
Federal Institute of Pernambuco, Caruaru, PE, Brazil
pedrosabyno@hotmail.com
niedson.silva@caruaru.ifpe.edu.br

Cezar Henrique Gonzalez
Carlos Augusto do Nascimento Oliveira
Universidade Federal de Pernambuco, Av. Acadêmico Hélio Ramos s/n, Cidade Universitária - CEP 50740-530 - Recife-PE, Brasil.
cesarchg@yahoo.com
cano.oliveira@gmail.com

Carlos José de Araújo
Universidade Federal de Campina Grande, Rua Aprígio Veloso, 882, Bairro Universitário - CEP 58429-900 – Campina Grande-PB.
carlos@dem.ufcg.edu.br

Abstract. *Shape memory alloys are materials often called intelligent because they have the ability to recover apparently plastic deformations in response to adequate external thermal stimuli. These alloys have great potential application as thermo-mechanical actuators in several sectors of the industry, which justifies the study and improvement of their properties in order to explore current needs in several areas of engineering. In this work, a shape memory alloy of the NiTi family was subjected to mechanical shaping by hot rolling and ECAE (Equal Channel Angular Extrusion) to improve the mechanical properties of these alloys. The samples were characterized by Differential scanning calorimetry (DSC) and dynamic-mechanical analysis (DMA) before and after the mechanical conformation processes. As a result, considerable changes were observed in the behavior of the calorimetric curves and damping of the samples, making it possible to compare the tests and the conditions of preparation of the material.*

Keywords: *NiTi Alloy, DSC, DMA, Mechanical Conformation, ECAE.*

1. INTRODUCTION

Shape Memory Alloys (SMA) are metallic materials that can respond to temperature changes leading to significant macroscopic deformation. This work was developed using plasma fusion technology, in order to obtain products with composition closer to the nominal and to add more detailed information about it in the literature. The shape recovery characteristic makes SMA well suited for practical technological applications. Some of the compositions studied in the literature have low mechanical resistance, while others show high brittleness when submitted to mechanical demands. Therefore, it is necessary to improve the mechanical properties of these alloys, aiming at practical applications with greater strength and reliability. The conformation processes selected for the study were the Hot Rolling and the Equal Channel Angular Extrusion (ECAE), because it allows the comparison between a well consolidated process in the literature and another in development.

In this work, a NiTi family alloy having the shape memory effect was fabricated by the plasma melting process and mechanically shaped by hot rolling and equal channel angular extrusion techniques. It was found that the transformation temperatures as well as the damping capacity of the alloy were changed for different processing techniques.

2. EXPERIMENTAL PROCEDURE

This project was developed through a technical cooperation between IFPE and UFPE in the context of shape memory alloys.

The manufacturing of the Ni-Ti alloys was carried out on the Discovery All plasma melting equipment from EDG Equipamentos, which uses thermal plasma as the energy transmission medium for melting the materials and argon as an inert gas to avoid contamination by oxidation. Figure 1 shows the furnace scheme (1a-d) and the typical Plasma Skull Push Pull (1e-f) process for a SMA of the Ni-Ti system. The elements used in manufacturing were commercially pure nickel (> 99.9%), supplied by Votorantim Metals, and biomedical titanium ASTM F67-00 (grade 2), supplied by TiBrasil. Initially the pure elements are placed in a copper crucible in decreasing order of melting point, as shown in Figure 1e (1). A rotating tungsten electrode gives rise to a plasma torch in an argon atmosphere, as shown in Figure 1e (2), causing the pure elements to merge and forming the SMA knob shown in Figure 1e (3). Once the metal is completely molten, the injection, Figure 1e (4), is automatically performed in a cylindrical metal shell to provide the product in the form of the mold inside the shell (Figure 1f). To ensure good homogeneity, prior to injection to obtain the final product, the Ni-Ti SMA button was melted 5 times.

In order to obtain samples in a format suitable for carrying out the tests and characterizations, an adaptation was made in the die (Figure 1d) of the machine and the preparation of a bi-split mold to obtain the shape of prismatic bars.

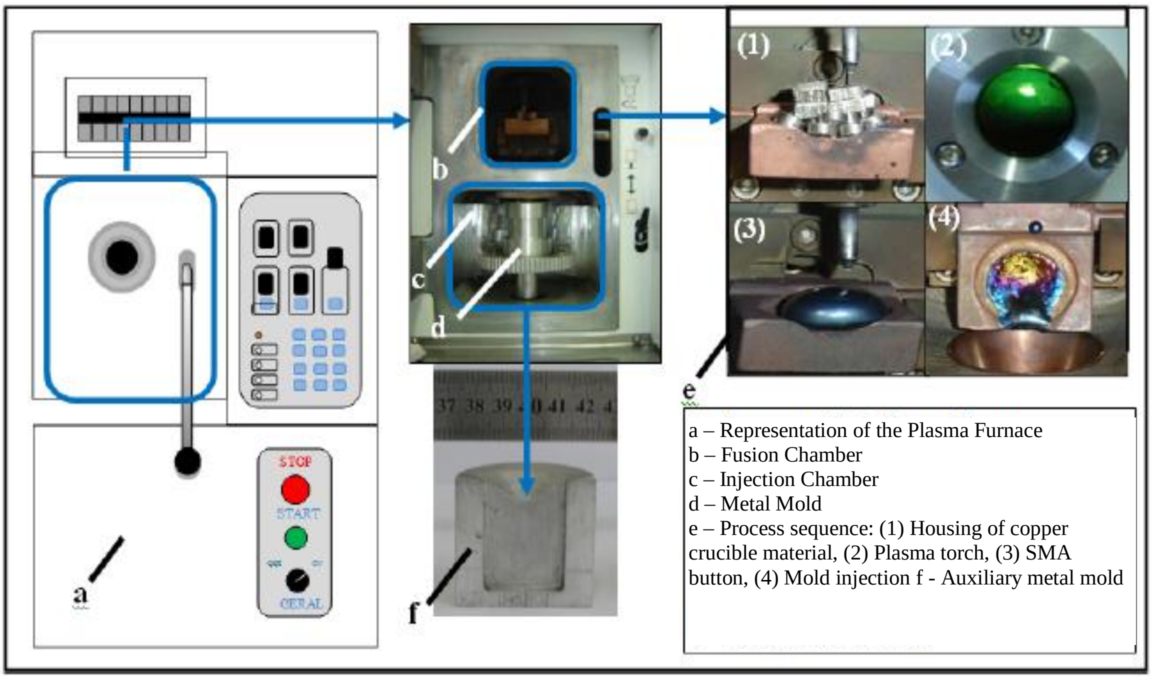

Figure 1 - Plasma furnace scheme (a-d) and PSPP process sequence for injection and mold injection (e-f). Source: Adapted from Pereira et al. (2013)

2.1 Preparation of the samples

The products obtained by the PSPP melt were heat treated at 900 °C for 2 hours to ensure the homogeneity of the ingots manufactured. The sample was sectioned in a diamond blade precision cutter, aiming at the preparation of plate samples for the rolling process and square prisms for angular extrusion.

Hot rolling was performed using muffle furnaces, located in the DEMEC / UFPE Foundry and Heat Treatments Laboratory, and a small electric laminator. The reduction occurred by means of successive passes through the mill, reaching 0.05 mm of plastic deformation per pass in the direction of the thickness of the sample. The reduction of the sample thickness was 66.7% with respect to the sliced slices, with a temperature of 900 °C in the oven. For the ECAE processing, the samples were cut in square prismatic format of 5x5 mm^2. The extrusion operation was performed by compressing a punch supported on the SMA billet at a rate of 0.5 mm / min, as can be seen in Figure 2.

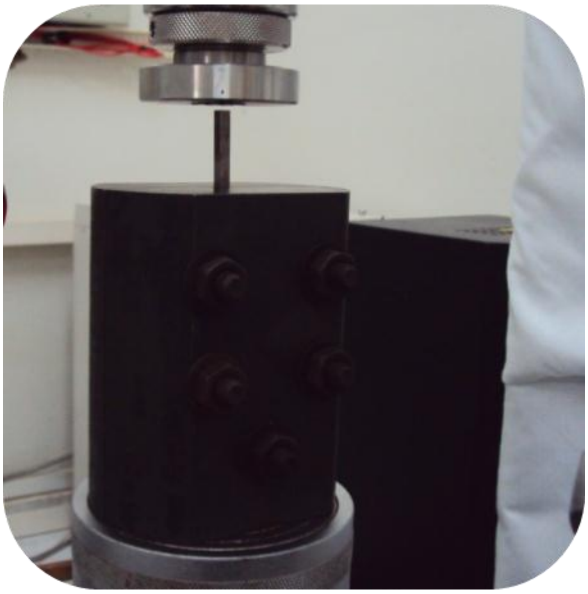

Figure 2 - Structure used for ECAE conformation. Matrix with punch prepared for extrusion

2.2 Characterization

One of the most used techniques for determining phase transformation temperatures is the Differential Scanning Calorimetry (DSC), which measures the temperature and heat flow associated with transitions occurring in the materials. The samples were analyzed in a temperature range of -20°C to 120°C, with a heating / cooling rate of 10°C / min, according to what is widely used in the literature (Aghamiri et al, 2013; Alexandrou et al, 2006; Karaca et al, 2013; Khaleghi et al, 2013). To avoid oxidation of the material, the DSC heating chamber was filled with nitrogen gas.

For dynamic analysis, DMA (Dynamic Mechanical Analyzes) tests were performed on an equipment that is installed on an anti-vibration bed to avoid external disturbances in the data collection. The experiment was conducted in single cantilever mode (Figure 3), following standard parameters: frequency of 1 Hz, heating rate of 2ºC / min and amplitude of oscillation of 5 µm (Van Humbeeck, J., 2003). The SMA Ni-Ti slides used have a useful length and width of approximately 17 and 5 mm, respectively. Prior to all assays, the samples were cooled to temperatures around -30 ° C for data collection of the reverse martensitic transformation on heating. As in the DSC trials, the transformation temperatures obtained through the dynamic test curves were determined using the tangent crossing method.

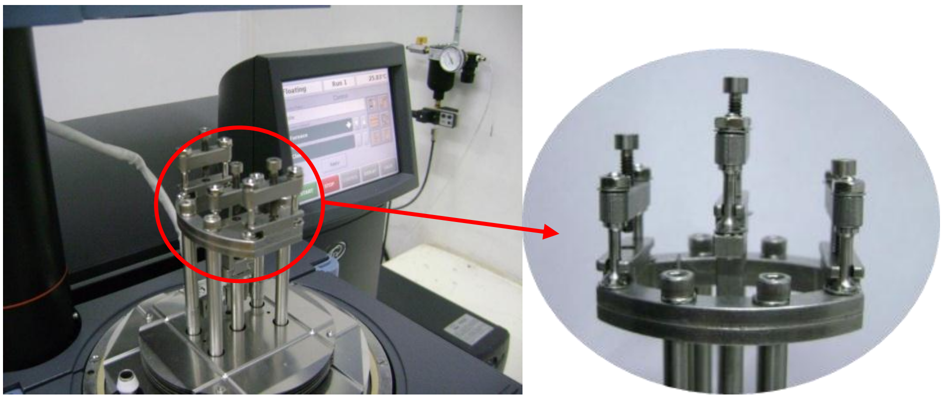

Figure 3 – *Cantilever* type support for samples.

3. RESULTS AND DISCUSSION

Table 1 presents the nomenclature of the analyzed samples.

Table 1 - Nomenclature of Ni-Ti alloy samples.

Sample	Homogenized	Laminated	Extruded
Rich in Ti	C1	C2	C4

3.1 DSC Tests

As shown in Figure 4 the DSC curves for NiTi alloy samples can be seen.

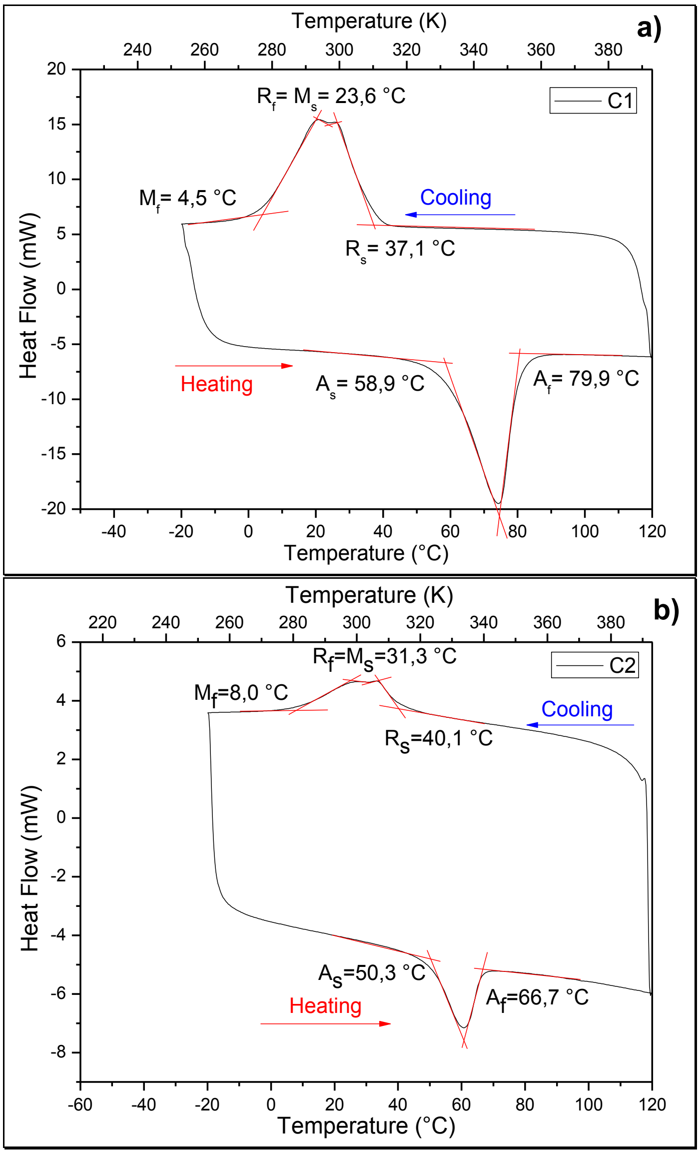

Figure 4a – Calorimetry curve for the studied Ni-Ti samples C1 (a) and C2 (b).

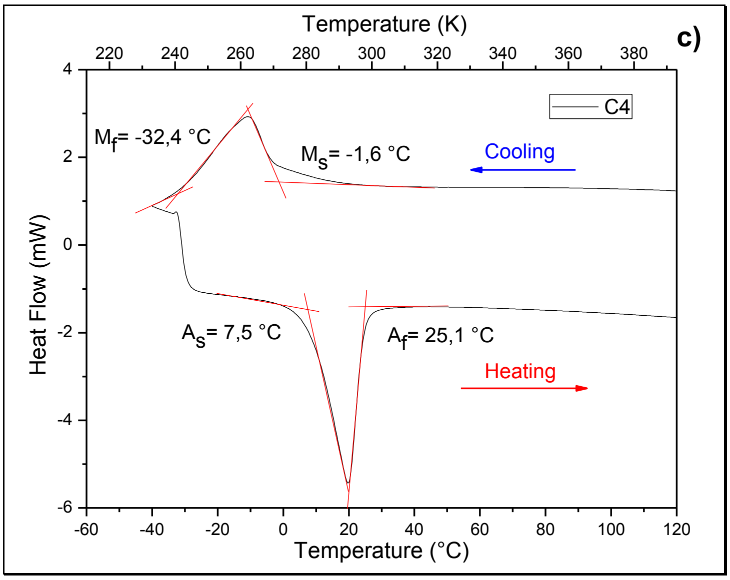

Figure 4b – Calorimetry curve for the studied Ni-Ti sample C4 (c).

Figure 4 shows the DSC curves for the analyzed samples, which show characteristic behavior for binary Ni-Ti alloys with shape memory effect potential. Although the sample C1 was subjected to plastic deformation due to shear, the homogenization treatment, followed by quenching in water, was sufficient to generate the reversible thermoelastic martensitic type phase transformation, as observed in Figure 4a. In this way, reverse martensitic transformation during heating and direct martensitic transformation can be observed during cooling. Similar behavior was found in mechanically shaped samples by lamination (Figure 4b) and by ECAE (Figure 4c).

It was verified that the mechanically formed samples showed reduction of the transformation temperatures, being evidenced in sample C2, which shows more severe deformation due to the passage through the matrix channels.

Table 2 summarizes the transformation temperatures, enthalpies and thermal hysteresis obtained for the samples studied. The thermal hysteresis of the transformation was determined by the temperature difference A_f – M_S, as proposed by Kockar et al. (2007).

Table 2 – Transformation temperatures of samples.

Sample	Transformation temperatures							
	A_s (ºC)	A_f (ºC)	ΔH (J/g) Heating.	R_s (ºC)	$R_f = M_s$ (ºC)	M_f(ºC)	ΔH (J/g) Cooling.	H_t (ºC)
C1	58,9	79,9	18,9	37,1	23,6	4,5	19,4	56,3
C2	50,3	66,7	20,0	40,1	31,3	8,0	19,8	35,4
C4	7,5	25,1	20,7	-	-1,6	-32,4	21,9	26,7

The presence of the R phase requires more intense cooling to complete the direct phase transformation, increasing the temperature range in which the transformation takes place. Sample C1 presented 19.4 J/g to complete the transformation in the cooling. By checking the sample C2, it was verified that there was a reduction of the transformation temperatures, when compared to the ones obtained in the homogenized samples. This is due to the fact that defects introduced through lamination make it difficult to transform. Due to the state of internal stresses, the amount of energy of the transformations found from the curves is greater than that verified in the homogenized sample, which indicates a higher enthalpy change for the transformation to be integrated.

The results obtained from the extruded sample showed that the degree of deformation of the material was quite severe when passing through the matrix channels, considering that the transformation temperatures of the alloys were much lower when compared to the samples that went through the lamination forming the hot. Due to this reduction in processing temperatures, the temperature range for the DSC was extended to -60 °C. It can be seen that, due to the internal stress state of the samples, the enthalpy of the sample increased during the phase transformation, which was increased to 21.9 J/g during the cooling of sample C4, while the thermal hysteresis was reduced according to the degree of severity of the mechanical conformation.

3.2 DMA Tests

According to the literature, Ni-Ti based alloys have a high damping capacity during phase transformation and in the martensitic phase, whereas their austenitic phase shows a much lower energy dissipation (Lu et al., 2003).

Figure 5 shows the damping curves, during heating, provided by the dynamic tests. It was found that, as in the DSC tests, also the reduction of the transformation temperatures occurred. In addition, it can be noticed the decrease in the energy absorption capacity of the shaped samples, especially in the sample that was submitted to the ECAE.

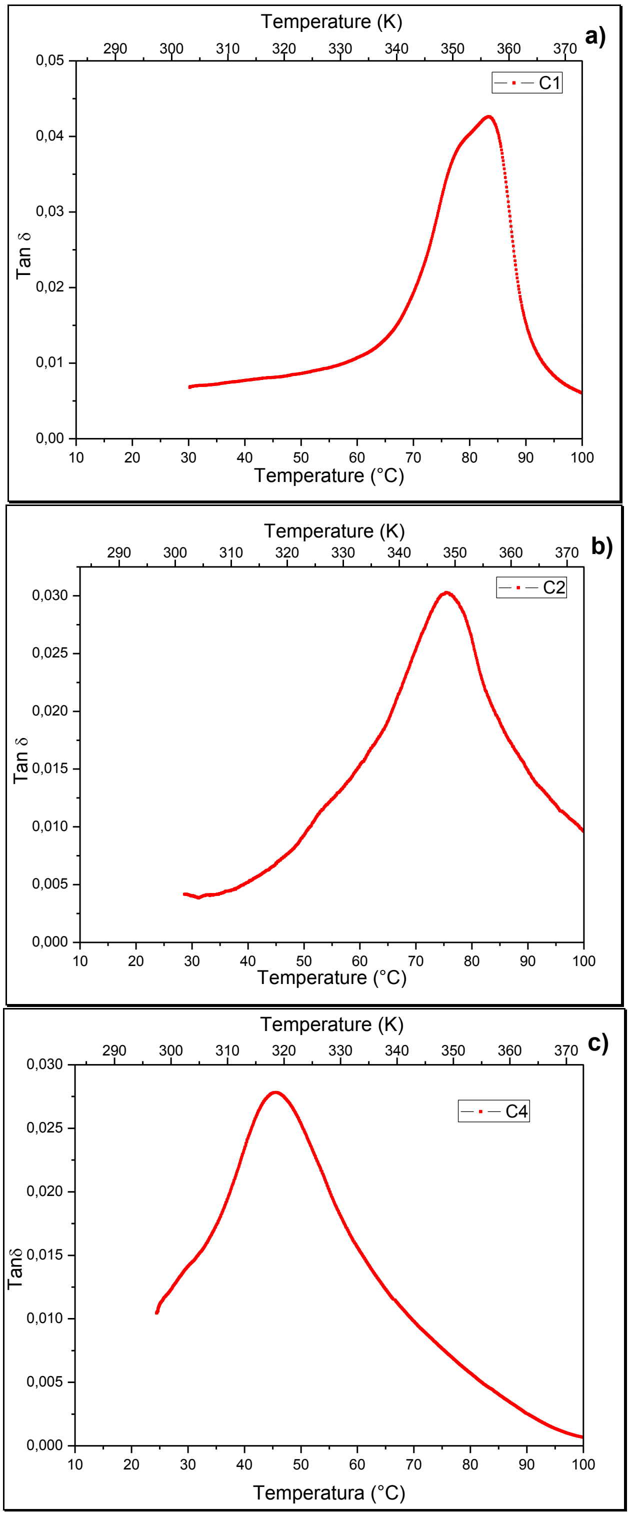

Figure 5– Temperature versus damping capacity for C1 (a), and C2 (b), and C4 (c) samples.

Through the curves of the tangent variation of the phase angle, Tanδ, with the temperature, it can be observed that the studied samples present the characteristic peaks of the thermoelastic reverse martensitic type transformation, which translate a considerable increase in the damping capacity of the temperature, an increase in the energy absorption of the material. This damping capacity is a dimensionless property and quantitatively translates the energy that the material can dissipate. In the SMA, this effect is closely related to the movement of the martensite / austenite interfaces during the transformation. The increase in damping can also be correlated to the defects of dislocations and their mobility in the material. Physically, at the temperatures in which the peaks occur, there is less vibration caused by impacts and agents external to the material.

By observing the results obtained, starting from the ambient temperature up to 100 ºC, the phase transformation peak can be clearly perceived, where the low temperature phase (martensite) has a higher damping capacity than austenite, which is due to the energy absorption caused by the martensite platelets. The reduction of the damping capacity comes from the increased rigidity of the material. This is due to the fact that there is less energy dissipation due to the structure corresponding to high temperatures in the alloy (austenite), as reported by Silva (2009). As verified in the DSC test, the C4 sample had lower transformation temperatures, which is due to the severe mechanical conformation caused by ECAE. In addition, the Tanδ peak indicates that this sample presents a lower degree of energy absorption than the others.

4. CONCLUSIONS

In this work a study of the effect of the mechanical conformation on a Ni-Ti family alloy with shape memory effect was made by the Plasma Skull Push Pull method. From the results presented, the following conclusions can be established:

- The manufacture of Ni-Ti family alloys by the technique called Plasma Skull Push Pull, as well as their preparation for the conformations were carried out successfully;
- The processing temperatures of the laminated and extruded samples were reduced relative to the homogenized. The extruded sample showed a more significant alteration, indicating a more severe plastic deformation during ECAE;
- The sample C4 showed a slightly more marked enthalpy change for the martensitic transformation, which can be attributed to the difficulty of internal mobility imposed by the plastic deformation;
- In the DMA tests, inflexions were verified in the curve of the loss factor Tanδ, corresponding to the damping capacity as a function of temperature, from which it can be observed that, due to the plastic deformations, the energy absorption is reduced in the samples. This difference in damping capacity directly influences practical applications that require mechanical demands;
- It is also possible to verify in the dynamic test the reduction of the transformation temperatures provoked by the mechanical conformations, being the most sensitive one that went through the ECAE process.

Through the obtained results, it was demonstrated that the mechanical conformation ECAE influences more severely in the thermomechanical properties of the NiTi SMA. This can be verified by introducing defects in the material revealed by the more pronounced reduction of the transformation temperatures and the damping capacity of the samples studied. This reduction in the damping capacity is associated to the difficulty of internal mobility in this sample.

5. ACKNOWLEDGEMENTS

This work was supported by IFPE for the concession of a scholarship to the first author through the PROPESQ.

6. REFERENCES

Aghamiri, S. M. S., Ahmadabadin, M. N., Shahmir, H., Naghdi, F., Raygan, S., 2013. Study of thermomechanical treatment on mechanical-induced phase transformation of NiTi and TiNiCu wires. Journal of the mechanical behavior of biomedical materials, Vol. 21, pp. 32-36.

Alexandrou, G. B., Chrissafis, K., Vasiliadis, L. P., Pavlidou, E., Polychroniadis, E. K., 2006.SEM Observations and Differential Scanning Calorimetric Studies of New and Sterilized Nickel-Titanium Rotary Endodontic Instruments. Basic Research-Technology, Vol. 32, pp. 675-679.

Lu, X. L., Cai, W., Zhao, L. C., 2003. Damping behavior of a Ti44Ni47Nb9 shape memory alloy. Journal of Materials Science Letters, Vol. 22, pp. 1243-1245.

Karaca, H. E., Kaya, I., Tobe, H., Basaran, B., Nagasako, M., Kainuma, R., Chumlyakov, S., 2013. Shape memory behavior of high strength Ni54Ti46 alloys. Materials Science & Engineering, Vol. 580, pp. 66-70.

Khaleghi, F., Allafi, J. K., Chianeh, V. A., Noori, S., 2013. Effect of short-time annealing treatment on the superelastic behavior of cold drawn Ni-rich NiTi shape memory wires. Journal of Alloys and Compounds, Vol. 554, pp. 32-38.

Kockar, B., Karaman, I., Kulkarni, A., Chumlyakov, Y., Kireeva, I. V., 2007. Effect of severe ausforming via equal channel angular extrusion on the shape memory response of a NiTi alloy. Journal of Nuclear Materials, Vol. 361, pp. 298-305.

Silva, N. J., 2009. Study of the thermomechanical behavior of a Ni-Ti alloy with shape memory using dynamic-mechanical analysis (DMA). Master's Dissertation, Federal University of Campina Grande, Campina Grande-PB, Brazil, pp. 113.

Van Humbeeck, J., 2003. Damping capacity of thermoelastic martensite in shape memory alloys. Journal of Alloys and Compounds, Vol. 355, pp. 58-64.

7. RESPONSIBILITY NOTICE

The authors are the only responsible for the printed material included in this paper.