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THERMOMECHANICAL BEHAVIOR OF NI-TI SHAPE MEMORY ALLOYS SUBMITTED TO HOT ROLLING

Niédson José da Silva
niedsonjs@yahoo.com.br
Cezar Henrique Gonzalez
Universidade Federal de Pernambuco, Av. Acadêmico Hélio Ramos s/n, Cidade Universitária - CEP 50740-530 - Recife-PE, Brasil.
gonzalez@ufpe.br

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

Diniz Ramos de Lima Junior
Universidade Federal de Pernambuco, Av. Acadêmico Hélio Ramos s/n, Cidade Universitária - CEP 50740-530 - Recife-PE, Brasil.
diniz.ufpe@yahoo.com.br

Leonardo M. C. Silva
Universidade Federal de Pernambuco, Av. Acadêmico Hélio Ramos s/n, Cidade Universitária - CEP 50740-530 - Recife-PE, Brasil.
leonardokgb01@gmail.com

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

Abstract. *Shape Memory Alloys (SMA) are materials who might be applied in various industrial sectors. For this fact, there is an increase of interest on improving the thermomechanical properties of these alloys. Mechanical forming of the rolling can lead to improvements in the mechanical properties of these SMA. These improvements are related to the SMA grain size reduction due to internal shear which it undergoes in passing through rolling mill, being subjected to compression and shear stresses. In this work, it was carried out the manufacture of Ni-Ti SMA and their mechanical forming in a hot rolling mill. As a result it was verified the improvement of mechanical properties and modification of transformation temperatures, essential characteristics in real applications. This analysis is very important for usage of SMA in applications that requires high mechanical strength combined with functional properties due to shape memory phenomena.*

Keywords: *Shape memory alloys, Hot-rolled, Transformation temperatures, Thermomechanical behavior, Mechanical forming.*

1. INTRODUCTION

Shape Memory Alloys (SMA) are active materials that present the capacity to recover apparently plastic deformation, induced at low temperature, through a subsequent heating above a critical temperature. This phenomenon is designated by Shape Memory Effect (SME) and it is intimately associated to martensitic phase transformation and reversible crystallographically (Otsuka & Wayman, 1998). SMA are considered electric actuators when SME is activated through the application of pulses of electric current (Joule effect). Otherwise, if SME is activated directly through the controlled variation of the temperature, they are considered as thermal actuators. When those alloys are submitted to processes of mechanical strain, it demonstrates a tendency to the increase of its strength due to the mechanical hardening. This modification of mechanical properties also induces a change in the transformation temperatures, essential characteristic for use of those materials in technological applications.

The hot-rolled is a kind of strain quite used in metallic alloys seeking the obtaining of plates or sheets. In that mechanical processing the materials are submitted to compression efforts and superficial shearing, who provides the reduction of thickness and, theoretically, reduction of grain size in the alloy.

Thus, the aims of this work are to obtain shape memory effect Ni-Ti alloys with different compositions, to submit those materials to the process of mechanical strain and to verify their effects in the mechanical and thermal properties of alloys.

2. EXPERIMENTAL PROCEDURE

This work was developed in the context of a partnership between UFPE and UFCG, through the Smart Materials Laboratory (LMI) and Multidisciplinary Laboratory of Materials and Active Structures (LaMMEA), respectively. The alloys of Ni-Ti system were selected and manufactured in LaMMEA through the process Plasma Skull Push Pull (PSPP), in which the material is melted on a fine layer of own material (DISCOVERY, 2005). This melting, was previously studied for De Araújo et al. (2008), it was accomplished in a machine that uses the thermal plasma as resource of energy transmission to melt most of the metals (except metals with high grade of zinc), besides titanium, nickel, chrome and cobalt. To assure satisfactory results in the melting of alloys, the gas argon is used inside the fusion chamber, which has as main characteristic to be inert. Fig. 1 displays the sequence of the plasma melting process. Initially, the materials are placed on crucible of melting machine Fig. 1a. The rotational electrode of tungsten originating the plasma torch in argon atmosphere, showed in 1b, that it proceed the melt of pure elements, forming the button shown in Fig. 1c. When the metal is melted it occur the automatic injection in a metallic mold, as can be observed in Fig. 1d and 1e, in which it is obtained the final product of melt, Fig. 1f.

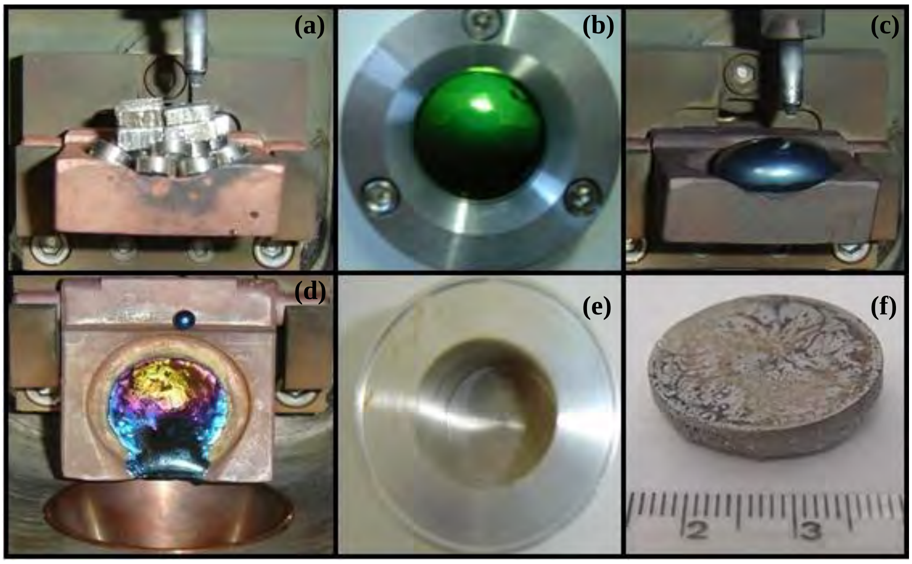

Figure 1 - Sequence of PSPP process for melting and injection in mold. (a) Alloy elements. (b) Plasma torch. (c) Button of SMA. (d) Fine layer of SMA on the crucible, after the injection. (e) Metallic mold. (f) Ingot of SMA.

In this research it were manufactured three alloys of Ni-Ti system, which according to the compositions were called Equiatomic Ni-Ti, Ni-rich Ni-Ti and Ti-rich Ni-Ti. The compositions of the selected alloys it defined in weight percent were 55Ni-Ti, 55.3Ni-Ti and 54.7Ni-Ti, respectively. To assure the chemical homogenization of the products, were accomplished a homogenization thermal treatment for 2 hours in a muffle furnace oven at 900ºC, following by quenching in water at room temperature. The ingots were cut off in a precision machine with an abrasive diamond disk, seeking the obtaining of plate used as samples for rolled process. The sheets were cut off with thickness of 1.5 mm, which after hot-rolled process, reaching thickness of the order of 0.5 mm. The hot-rolled was accomplished in a rolling mill with successive reductions of 0.05 mm and permanence time in the muffle furnace for plates heating for 60 seconds among the passes.

For each material sample, before and after the process of mechanical strain for hot-rolled, Vickers microhardness tests and Differential Scanning Calorimetry (DSC) were accomplished. Results of microhardness tests were collected over 10 indentations with distance of 1 mm between them with a penetration load of 300gf for a period of 15 s, while in DSC heating and cooling cycles were accomplished with rate of 10ºC/min between -20ºC and 120ºC.

3. RESULTS AND DISCUSSION

The materials were melted in the plasma melting furnace, in which the final product was gotten in a form of prismatic bars. The bars were cut, who resulted in plates for the accomplishment of the mechanical conformation, as shown in Fig. 2.

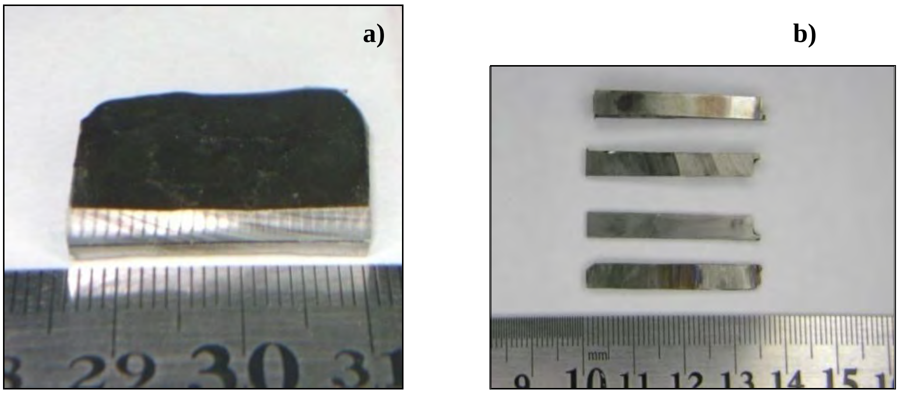

Figure 2 – Obtained final product of the plasma melting (a) and cut plates (b).

DSC procedure were accomplished on cut plates for the three manufactured alloys; defined before as equiatomic Ni-Ti, Ni-rich Ni-Ti and Ti-rich Ni-Ti. Fig. 3 shows the result of the calorimetric behavior for exothermic and endothermic steps during thermal cycling on the NiTi alloys.

The presence of the thermoelastic martensitic transformation can be observed for all studied alloys, as they demonstrate peaks in the curves, during the heating as much as cooling. In a general way, the phase transformation in the Ni-Ti system happens in two stages, in accordance with the literature (Otsuka & Wayman, 1998). During cooling the austenite (B2) change to R phase at R_s temperature, responsible for the first peak in the heat flow, and later the R phase, with rhombohedral structure, change to a monoclinic martensitic structure (B19'). During the heating the inverse transformation happens (martensite→ austenite), which is characterized by the inflection in the calorimetric heating curve. The transformation as described previously can be visualized in Fig. 3c, where the transformation stages can be clearly visualized. For the other manufactured alloys, the DSC tests didn't detect the intermediate phase in an evident way during cooling, which is probably in junction to martensitic peak during the transformation. Thus, the onset point of the exothermic peak in the high temperature side corresponds to the start temperature (M_s) of the transformation to the low temperature phase and the onset point in the low temperature side of the endothermic peak corresponds to the start temperature of the reverse transformation to the high temperature phase (A_s).

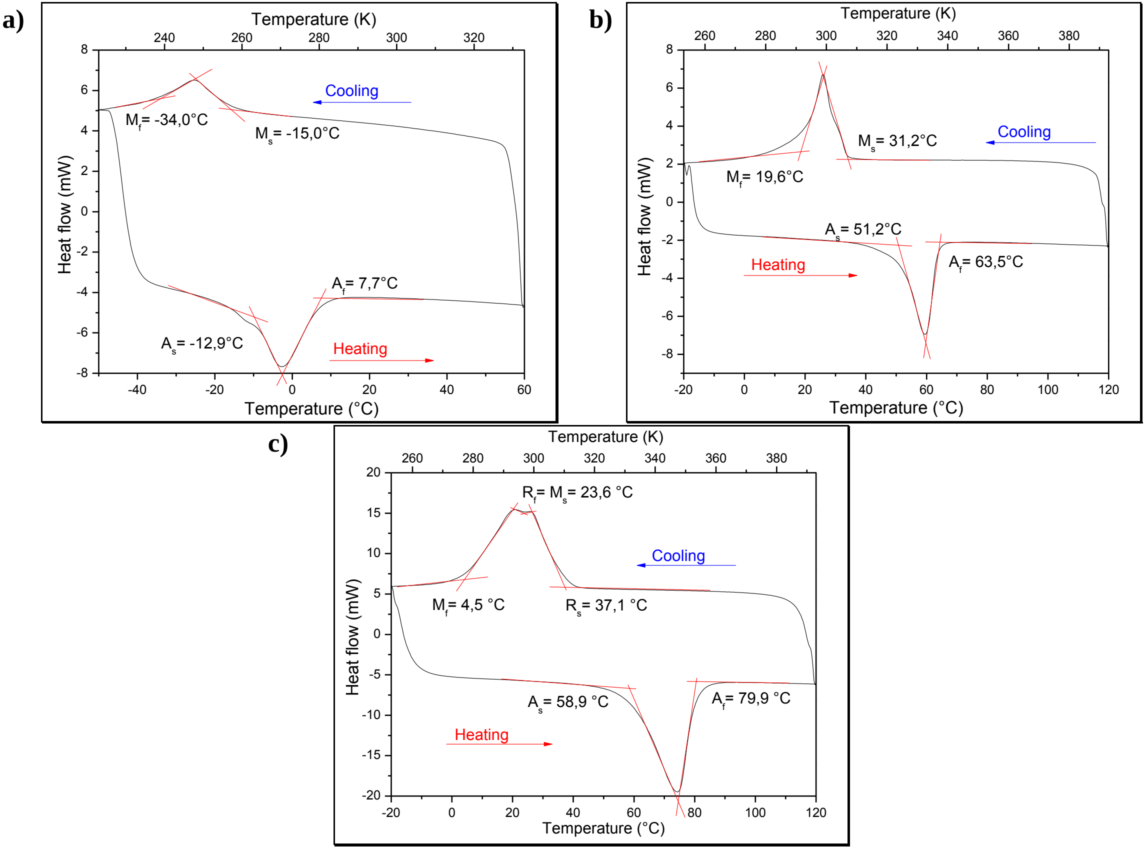

Figure 3 – Calorimetry curves of Ni-rich Ni-Ti (a), equiatomic (b) and Ti-rich Ni-Ti alloys (c).

The Vickers microhardness tests were developed for samples after the homogenization heat treatment. It is verified through Tab. 1 that the Ni-rich alloy presents lower microhardness than other alloys, what is due to the largest percentage of this element in the alloy. Moreover, the microhardness tests were made in room temperature and the only sample tested in the austenite phase was the Ni-rich, once reverse martensitic transformation temperatures (A_s and A_f) are below of room temperature. The austenite phase presents larger mechanical resistance than the one of the thermoelastic martensite, current in studied SMA. This fact indicates why the equiatomic alloy presents superiors results of microhardness than Ni-rich alloy. Probably, the equiatomic alloy already had a small percentage of the austenite phase in its structure, while for the Ti-rich alloy the whole test was accomplished in the martensitic phase. Equiatomic alloy presents values slightly higher than if was totally in martensitic phase. Although it is in the martensitic state at room temperature, the Ti-rich alloy presents superior grade superficial resistance to the alloys with smaller tenor of Ti in their composition.

Table 1 – Data of microhardness tests for homogenized alloys.

Homogenized alloys	**Microhardness (HV)**
Ni-rich	269.4 ± 6.5
Equiatomic	287.7 ± 14.2
Ti-rich	325.3 ± 21.9

After microhardness tests on homogenized plates, it was carry a hot-rolled on samples, who were after submitted to DSC test and microhardness for comparative effect. Fig. 4 displays the obtained curves of the DSC tests for the rolled samples.

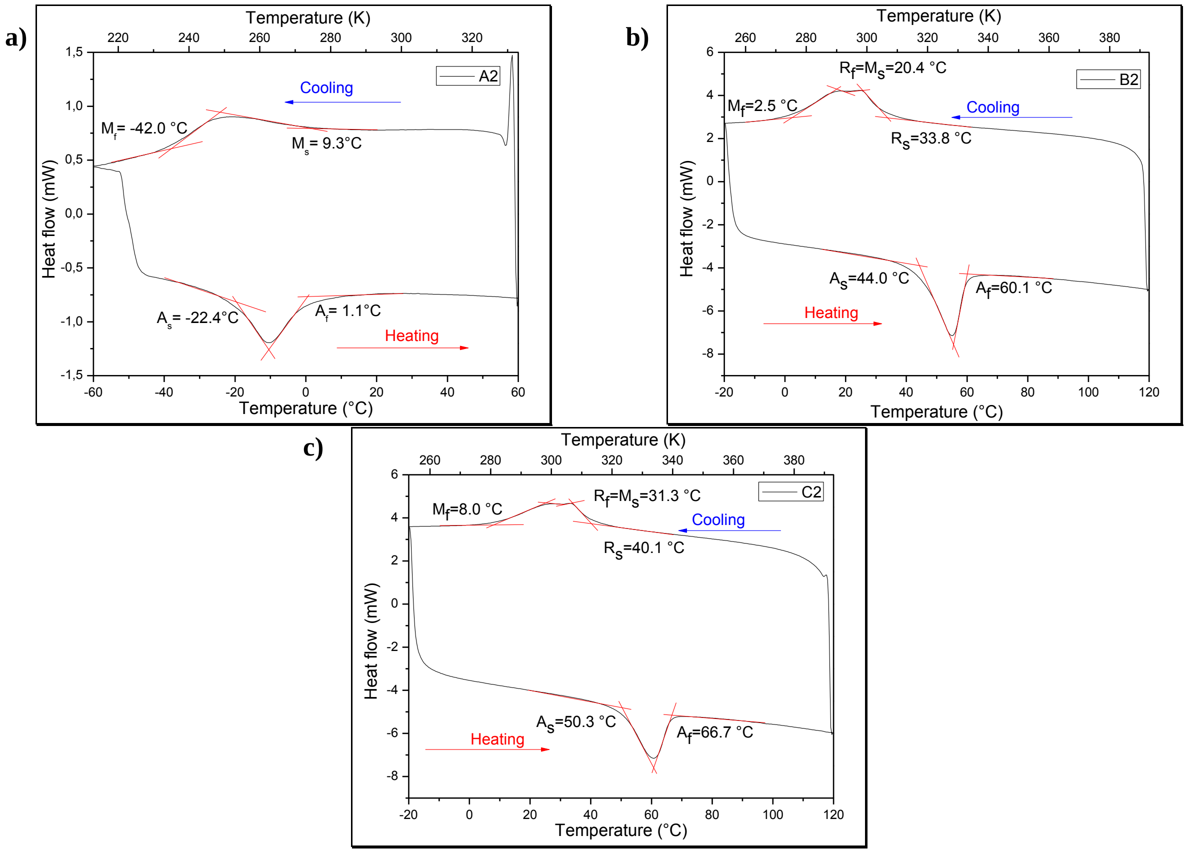

Figure 4 – Exothermic and endothermic behavior during thermal cycles for Ni-rich (a), equiatomic (b) and Ti-rich (c) hot-rolled.

Although Ni-Ti alloys of different compositions have been submitted to mechanical strength due to rolled process, all samples presented the shape memory characteristic transformations. Depending on the intensity of the deformations imposed, the material exhibit alteration on the shape memory phenomena, this result can explained by the inhibition of the phase transformation due to excessive propagation of internal defects in the material. After hot-rolled, the equiatomic alloy (Fig. 4b) began to show a direct martensitic transformation in two steps, it is probably due to a retard of martensitic transformation for the introduction of crystalline defects during hot-rolled, who caused the disaggregation among the transitions of the martensitic and R phase.

When making a comparison among the DSC curves in Fig. 3 and Fig. 4, it is verified that, in general, the transformation temperatures presented tendency to decreasing. This fact indicates that the martensitic transformation demands more energy to be completed, in other words, the defects imposed by the mechanical strain process resulted in scenery of larger difficulty to accomplish the phase transformation in the alloys. It is also notable that the thermal hysteresis has increased, which is characterized by the difference between the transformation temperatures in the heating ($Ht_{heating} = A_s\text{-}A_f$) and in the cooling ($Ht_{cooling} = M_s\text{-}M_f$), probably due to energy dissipation under the form of internal friction during transformation. This effect happens due to process of defects propagation and the presence of R phase, in the case of Equiatomic alloy.

Tab. 2 synthesize the results for samples that were submitted to the hot-rolled process, in this case is noticed a increase of microhardness in all alloys due to mechanical hardening imposed by plastic strains, although the mechanical forming process has been made at high temperature. In that aspect, it is noticed that the plastic deformations imposed to the materials has directly influence in the mechanical properties and transformation temperatures.

Table 2 – Results of microhardness tests on rolled samples.

Rolled samples	**Microhardness (HV)**
Ni-rich	370.5 ± 13.1
Equiatomic	373.4 ± 21.0
Ti-rich	396.2 ± 23.9

4. CONCLUSIONS

The manufacture of shape memory alloys through the plasma melting process was accomplished with success. It was noticed that transformation temperatures found for the samples presented quite coherent results for different compositions, being higher for alloy with larger percent of titanium and lower for those with larger percent of nickel. The mechanical forming of hot rolled demonstrated that the introduction of defects promoted increase on the samples mechanical strength , probably due to the reduction of grains size caused by the compression and shearing stress for the plastic strain. At the same time, critical temperatures of phase transformation of the three alloys decreased, from where is concluded a more energy requirement to complete the transformations integrally. In that way, it was verified that, for real applications using shape memory Ni-Ti alloys, in situations where the material is submitted to mechanical strain, it has to be consider the fact that their transformation temperatures will be decreased, as well as it can induce an increase on thermal hysteresis due to inhibition of shape memory effect. Microhardness behavior, which can indicate better mechanical properties and shape memory phenomena modification, will be analyzed in works that are in course.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

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