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STUDY OF THE EFFECT OF HEAT TREATMENT ON A NITI ALLOY

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Abstract. *The ability of applying and controlling materials that emit electromechanical response has enabled the development of intelligent structures to control vibrations, stiffness, shape and other applications. As shape memory alloys fit into this class of materials, with the ability to act thermomechanically, through force generation, variation of stiffness and damping, shape control, along with a variation of temperature. Knowing and understanding how to modify materials are of great importance to the industry, then it is essential to study how variations in the properties of materials, a principle, from the application of various thermal treatments at temperatures of 400, 450 and 500 ° C, with the times of 10, 20 and 30 minutes at each temperature. Such treatments applied in alloys with shape memory effect, can modify their thermomechanical properties, such as energy transformation temperatures, activation energy, among others, these variations can make or impossible an application for these alloys in the most diverse areas. In order to determine such activation temperatures, Electrical Resistance x Temperature (RET) tests, prove to be effective and within reach, allowing a calibration of temperatures by means of the tangent method.*

Keywords: *shape memorys alloy, heat treatment, processing temperatures.*

1. INTRODUCTION

Form memory alloys (SMA) are metallic materials based mainly on Nickel-Titanium, copper or iron, capable of assuming a previously defined shape when subjected to temperature variation. In the study of alloys with memory effect as actuator material, it is extremely necessary to analyze the mechanical properties with the temperature variation for the characterization of the activation potential of these materials, this activation potential occurs for several reasons among them linear response of the wire to the temperature variation, to be in the totally martensitic state at the temperature of use, to a complete activation with a low temperature variation and also for its initial austenite to be close to the temperature of use. In this article, the activation temperatures of these alloys will be determined with nine heat treatments, with a variation of three temperatures and three treatment times, where the final martensitic and austenitic transformation temperatures are defined as necessary to complete the martensitic and austenitic transformations, respectively, where from one phase to another, virtually all physical properties such as electrical resistivity, damping characteristics, elastic modulus, optical reflectivity change. The influence of time and temperature will also be observed over the phase change temperatures. These temperatures were acquired through Electrical Resistance x Temperature (RET) tests in a well developed by the UFERSA research group, with controlled temperature and heating and cooling rates. In the tests the temperatures were defined by applying the method of meeting the tangents indicated in ASTM F2004-05.

2. EXPERIMENTAL PROCEDURE

The experimental procedure involves subjecting samples of NiTi wires to different heat treatments and tests for measuring change temperatures. Table 1 shows the designation of the samples obtained, together with the applied thermal treatment conditions (Temperature x Time).

Table 1. Sample designation.

Temperature (°C)	10 minutes	20 minutes	30 minutes
400	A.1	A.2	A.3
450	B.1	B.2	B.3
500	C.1	C.2	C.3

In this work the samples used were Nickel-Titanium wires, with a diameter of 0.3 mm and a length of 100 mm, such samples were acquired through the German company Memory Metalle. Tempering heat treatments were performed using a heating rate control oven in order to maintain the parameters for all the wires.

RET (Electrical Resistance vs. Temperature) tests were performed, this one in turn was carried out with the temperature varying from -13 to 115 ° C, in a tub developed by UFERSA research group, with a heating / cooling rate of 1, 5 ° C per minute, where the values of temperature and resistance were measured by a Keysight 34972A acquisition system with an acquisition rate of 6 points per minute, thus enabling the identification of phase transformation temperatures.

To determine the phase transition temperatures, the tangent method was applied to the peaks following the ASTM F2082-06 standard.

The assembly of the system was as shown in Figure 1, so that the wires were subjected to a stabilized electric current from the voltage regulator source, and the electrical resistance was acquired by the Keysight 34972ª acquisition system directly connected to a computer, where the data was saved in excel spreadsheet.

For the data acquisition, the system was assembled to perform the test by the 4-point method, where two external points feed the sample with the desired current and the internal points collect the voltage values, thus allowing the different values of resistance.

Figure 1. System assembly.

3. RESULTS AND DISCUSSION

Through the tests of electrical resistance as a function of temperature, the graphs ΔR X T were plotted for all the samples separating them by thermal treatment temperature. In Figures 2, 3 and 4 the results for the thermal treatments of 400 ºC, 450 ºC and 500 ºC respectively, with the variation of the time of thermal treatment.

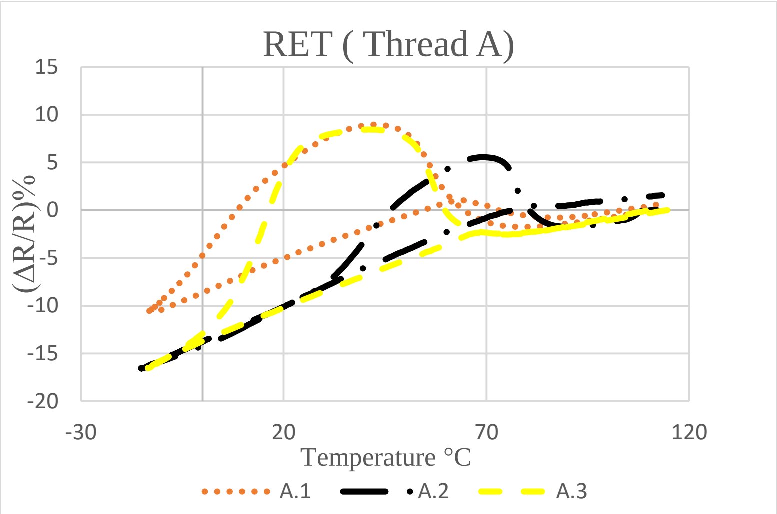

Figure 2. Thread A.

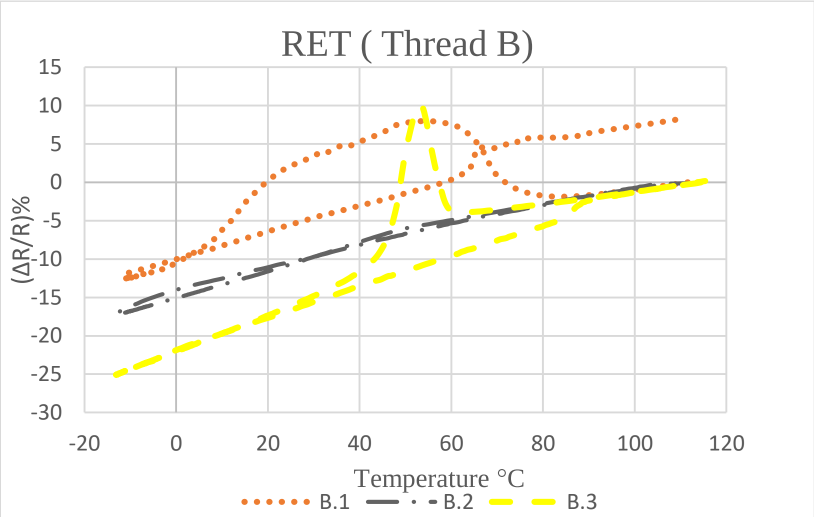

Figure 3. Thread B.

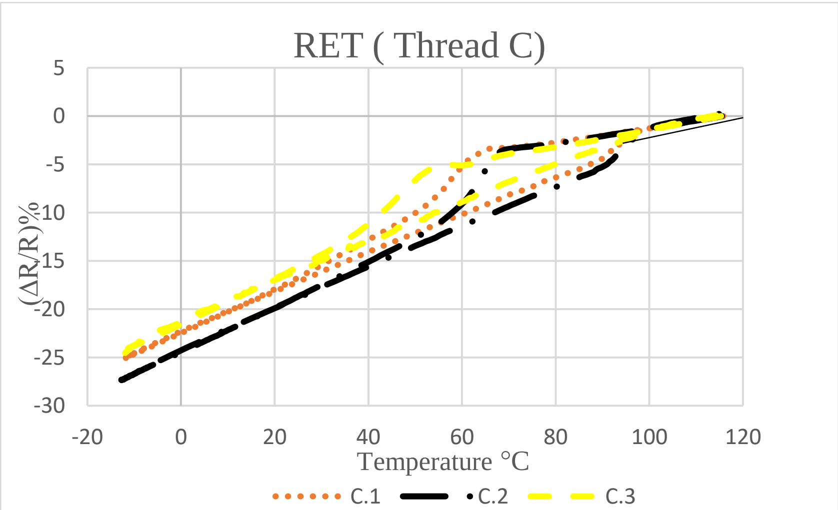

Figure 4. Thread C.

Through the Graphs 1, 2 and 3 and applying the tangent method, it was possible to determine the phase transformation temperatures for each thermal treatment applied. Table 2 summarizes these results.

From the results, it can be observed that for the thermal treatment carried out at 400 ° C, at all times the alloy presented rhombohedral phase transformation temperatures. It was also observed that at the temperature of 450 ° C, only one sample had this stage (treatment at 10 ml.). As observed by Nurveren (2007), this was already an expected result since in thermal treatments with higher temperatures, the formation of the R phase decreases due to the reduction of internal stresses.

Table 2. Phase transformation temperatures.

	Variable	M start	M end	A start	A end	H start	H end
Thread A.1	T(°C)	14,68	-5,67	61	82,5	69,5	47
Thread A.2	T(°C)	48,8	31,2	77	87	82	72,5
Thread A.3	T(°C)	22,68	8,4	66,5	79,2	63,5	52,2
Thread B.1	T(°C)	20,8	8,4	61,61	66,2	76,2	59,8
Thread B.2	T(°C)	58,2	50	40	64,5	-	-
Thread B.3	T(°C)	59,3	45,1	85,2	88,2	-	-
Thread C.1	T(°C)	54	35,5	94	98	-	-
Thread C.2	T(°C)	67,5	59,9	90	99	-	-
Thread C.3	T(°C)	62	53	87	97	-	-

Through the data summarized in Table 2, the graphs were plotted with Martensitic (Initial and Final), Austenitic (Initial and Final) and Romboedric (Initial and Final) transition temperatures. In Figures 5, 6 and 7, these graphs are shown.

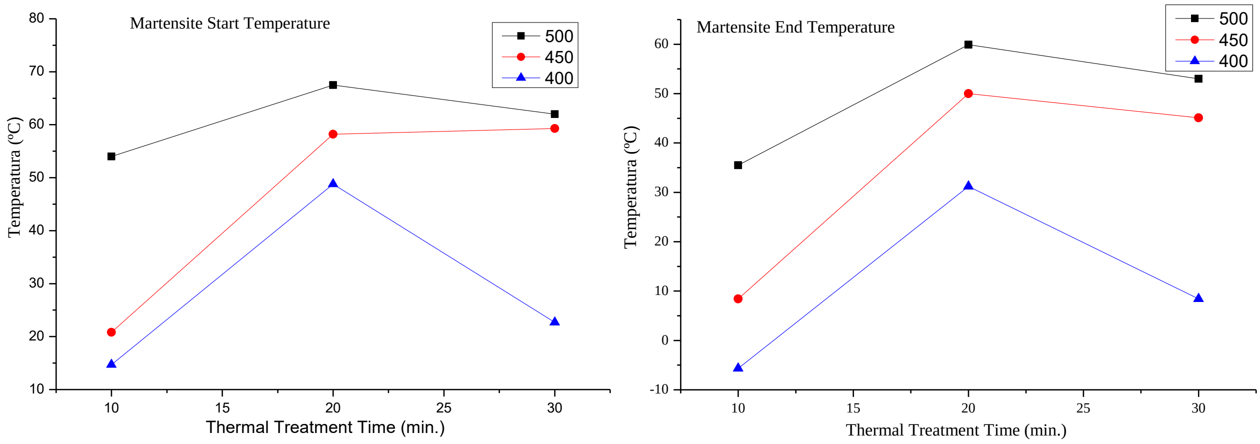

Figure 5. Martensite.

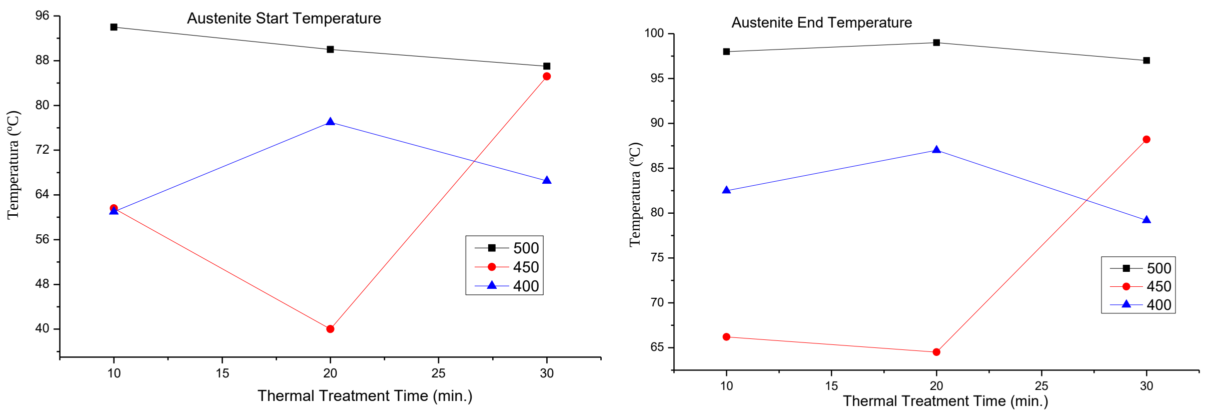

Figure 6. Austenite.

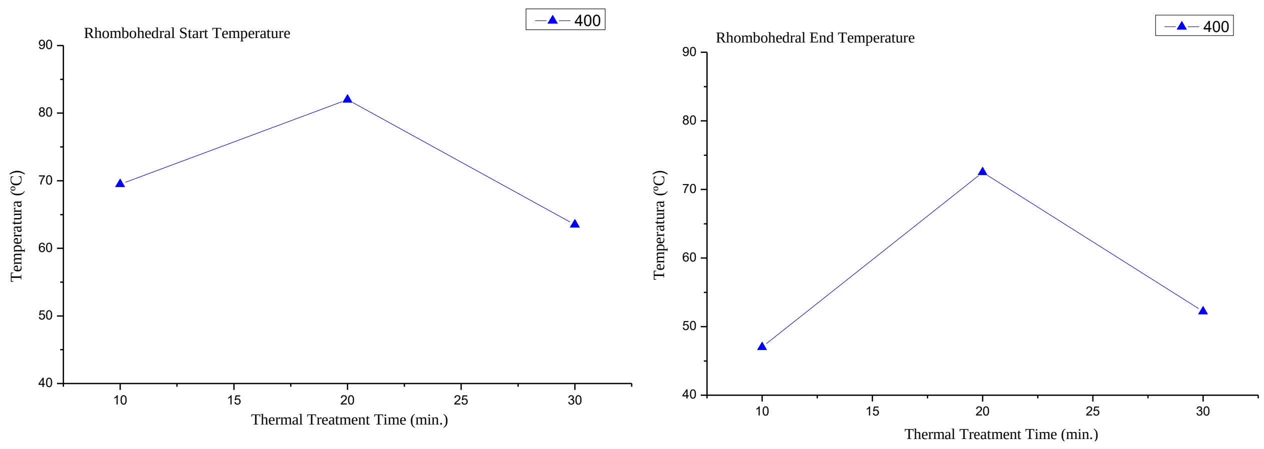

Figure 7. Rhombohedral.

Considering that one of the characteristics sought for a good actuator is the ability to change phases quickly, since the faster this phase change occurs, the faster the actuation will be, the wires that have this intermediary rhombohedral phase, may not be attractive for use as actuators, since they would go through an intermediary phase to have their complete activation. In addition, we would have a non-linear actuator, with intermediate levels of activation. It should be noted that these characteristics are directly related to the response time of the wire as actuator, so for systems that require a high speed of actuation, it is suggested that these wires would not be a good option.

By analyzing the thermally treated wires by the RET test, it was observed that there was variation of the phase transformation temperatures with the increase of the heat treatment temperature and with time, a fact already expected as reported by Wiggers (2011). It was observed from these results that not all the samples presented Final Martensite phase transformation temperature above the ambient temperature (30 °C), the samples that have the rhombohedral phase have the final martensite temperature below the ambient temperature. Figure 6 shows that the treated wires at 500 °C have higher values of austenitic transformation temperature, as well as the higher activation temperature value (final Austenite) and that the wires treated at 450 °C presented the lowest values.

In an attempt to pick up a treatment to be applied in the alloy H (NiTi equiatomic) in order to obtain wires of this alloy with improved thermal properties for use as actuators have one of these characteristics is the alloy be substantially martensitic at temperatures environment and austenitic temperature above and around the application temperature of this alloy as actuator, thus, considering the application temperature as the ambient temperature (30 °C), it is observed that for the heat treatment at 450 °C for 20 minutes (B.2) the alloy showed an initial Austenite value of 40 °C, the closest of which is the ambient temperature. For all applied treatments.

Through the phase transformation temperatures, the temperature variations (ΔT) were calculated for each phase in order to discuss which treatment generates samples with lower ΔT, since this influences the response time of the wire as actuator. In Figures 8 the graphs with the values of Δt calculated for austenite and martensite are shown, so the most indicated wires are those treated at 450 ° C, where the analysis of the treatment time shows that in the graphs of ΔT , wires A and C, those treated for 30 minutes have the lowest processing temperatures, the treatment of 20 minutes having the lowest activation temperature, but its temperature of 40 ° C is very close to the ambient temperature and may promote a transformation of phase before expected, being the wire B.1 the most indicated by the analysis of the activation temperature.

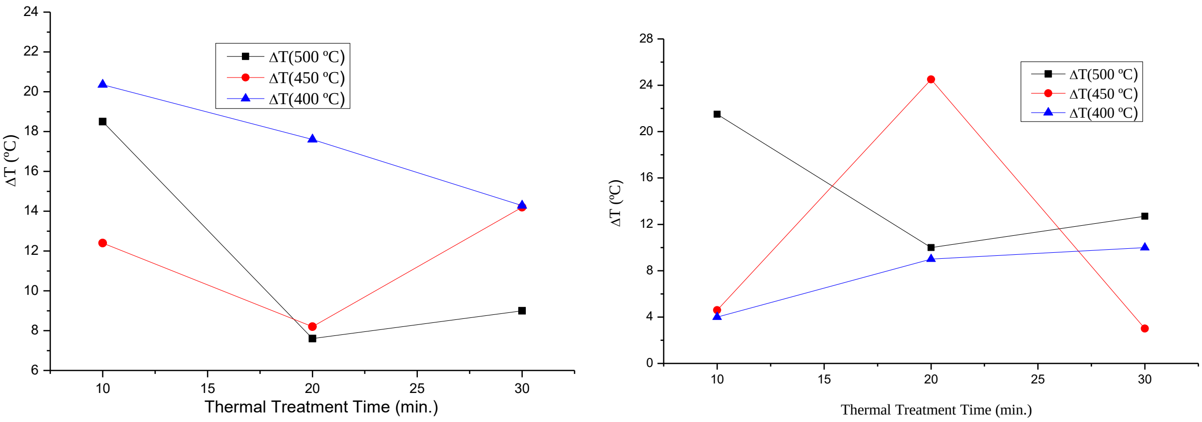

Figure 8. Temperature change for complete transformation.

4. CONCLUSION

The results show the appearance of the rhombohedral phase in the treated wires with the lowest temperature in both tests, besides the displacements of the activation temperatures during the transformation to the Austenitic phase following the increase of the duration of treatment.

An analysis of the two parameters shown, we have initially discarding the samples with the rhombohedral phase present, because they have a longer response time, we see that the wire B.3 stands out because it has a lower ΔT for the austenitic transformation, a value of ΔT is considerable for the martensitic transformation, even with intermediate values of activation temperatures, this thermal treatment being the most suitable for the wires that will be used as actuators.

The different thermal treatments applied were efficient in relation to the variation of the phase transformation temperatures, considering the need to control the activation temperature. A sequence of this study consists of the thermomechanical characterization with the objective of associating the mechanical properties to the different applied thermal treatments and, therefore, make the choice of which thermal treatment will be the most adequate to reach the greater potential of activation of the analyzed wires.

5. REFERENCES

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