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EFFECT OF SOLUBILIZATION ON THE MECHANICAL PROPERTIES OF NITI TUBES

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Abstract. *NiTi alloy presents particular properties: they can recover large deformations by temperature variation (memory effect) or by discharge (pseudoelastic effect). Due to these properties, this alloy presents itself promising at structures control. This paper examines the influence of martensite solubilizations on mechanical properties of NiTi alloy (55%w.p. Ni and 45%w.p. Ti). To obtain the properties of the alloy was used Vickers microhardness test, Differential Scanning Calorimetry (DSC), X-Ray Diffraction and Tensile Test according to ASTM F-2516. First, the results of the DSC as received NiTi tubes confirmed the presence of the martensitic phase at room temperature. Then the specimens were heat treated at 500°C for 60 minutes and water cooled at room temperature. After treatment, the martensitic* *transformation temperatures decreased and only austenitic phase was present at room temperature. The heat treatment reduced 22.6% the value of microhardness. On tensile tests it was observed that the specimen treated obtained 1% of residual deformation.*

Keywords: *NiTi, Shape memory alloy, Pseudoelastic, Solubilized martensite, Phase transformation*

1 INTRODUCTION

Every year, earthquakes take the lives of thousands of people, and cause property damage worth billions. Designing structures to withstand seismic forces is of primary concern for civil engineers. Conventional seismic design approaches rely on the ability of structures to dissipate the input earthquake energy through inelastic deformations in specially designed regions of the structural system while avoiding an overall loss of structural stability and integrity (Ozbulut et al., 2011).

To overcome this deficiency, there is some various studies about it. Nickel–titanium (NiTi) shape memory alloys possess the ability to recover strains on the order of 8% or more due to a highly reversible thermoelastic martensitic transformation. Depending on the testing temperature, NiTi demonstrates two different strain recovery responses: pseudoelasticity and shape memory. Pseudoelasticity (also termed superelasticity) refers to the spontaneous recovery of strains in initially austenitic NiTi upon removal of the applied stress, and is attributed to the reversible stress-induced austenite-to-martensite phase transformation. Shape memory describes the recovery of strains in initially austenitic or martensitic NiTi after unloading and subsequent heating above a critical temperature (Gall et al., 2008).

The austenitic transition temperature of the NiTi alloy can be adjusted to the desired value by changing the time and temperature of the heat treatment in pseudoelastic NiTi alloys. Their results showed that the heat treatment temperature should be adjusted between 400-500°C and the heat treatment time should be less than 60 minutes (Yeung et al., 2001).

This paper examines the martensite solubilization heat treatment influences on mechanical properties of NiTi alloy (55%Ni and 45%Ti). The objective of this work is to report the behavior of phase transformation, pseudoelastic deformation and microhardness of two types of NiTi tubes. The first kind is the NiTi Tube As Received (whitout heat treatment) and the second is the NiTi tube Heat Treated at 500°C for 60 minutes followed by immediate cooling in water at room temperature in order to remove any martensite phase and leaving only austenite phase at room temperature. The purpose of this study is to analyze and compare the hysteretic behavior, the phase transformation, identify the material's phase at room temperature, and measure the microhardness of NiTi pipes after heat treatment that solubilized the martensite phase. This paper aims confirm if this heat treatment brings better behavior as a Hysteretic Damper applied in the control of vibrations in structures by the way that NiTi Heat Treated could gain larger hysteric area at Tension Test according ASTM F-2516.

2 EXPERIMENTAL PROCEDURES

2.1 Material and specimen preparation

The Nickel-Titanium (NiTi) tubes were acquired from Memry – SAES Group Company. The technical specifications are described at table 1.

Table 1. Technical specifications of NiTi tubes

Technical Specifications	Values
Outside Diameter	10 mm
Inside Diameter	9 mm
Length	~800 mm
Composition: Nickel (Ni)	54 to 55% wt
Composition: Titanium (Ti)	44 to 45% wt
Composition: Oxide (O)	< 2% wt

By Scanning Electron Microscopy and Energy Dispersive Spectroscopy the chemical composition of alloy was confirmed according Table 1.

2.2 Heat treatment - Martensitic phase solubilization

The heat treatment was carried out to solubilize the martensitic phase. So, the sample was treated at 500°C for 60 minutes of isothermal holding and then water cooled at room temperature.

This heat treatment was based on the result obtained by Yeung et al., (2001), Villamarin (2013) and Wang et al., (2011).

2.3 Specimen's Preparation - NiTi as received and NiTi heat treated

The NiTi tube samples were cut using Cold Cutting Machine of the Struers®, Secotom- 15 model, with Diamond Cut-off Wheel Mod20 Struers®. All samples were of sizes close to 3mm in length satisfying the geometric condition of the crucible for the DSC and XRD analyzes. The cooling's cut was followed by ultrasound bath with distilled water per 15 minutes by ultrasound equipment of the brand Quimis®.

2.4 Differential scanning calorimetry – DSC

Differential Scanning Calorimetry (DSC) was used to determine the phase transformation temperatures of the NiTi tube specimens.

After de preparation process described at last subtitle (2.3) the samples whit mass about 11mg at 23mg were analyzed in the DSC machine in according with ASTM F2004 (2016). The equipment used to perform the test is the PerkinElmer brand, model 8500. Adopting as test conditions:

a) Atmosphere with nitrogen flow;

b) Heating rate of 20°C / min;

c) Aluminum crucible;

d) Tested between -70°C and 100°C.

2.5 X-ray diffraction – XRD

To determine the phases presents in the NiTi Tubes samples the XRD analyzes were carried out with the following parameters: sweep of $2\theta = 20°$ to 150°, with a pitch of 0.05° per reading and speed of 1° per minute.

For the assays, an equipment of the brand Rigaku, model Ultima IV was used. The diffractograms were analyzed in Crystallographica Search Software Match, which uses ICDD crystallographic data (The International Center for Diffaction Data). The phases present in the material were revealed by identifying the crystalline plans.

2.6 Microhardness test

The Vickers microhardness tests were carried out according ASTM E384 (2016). The measurements were performed with a load of 5 N (HV 0.5) under the surface of the samples for 10 seconds. In each sample 5 indentations were obtained, so that the mean and standard deviation for these results were calculated.

For the tests, an equipment of the brand Emco-test, model DuraScan was used.

2.7 Tensile test

The Tensile Tests were carried out according ASTM E8 (2016), in specific section 6.9 (Specimens for Pipe and Tube). Snug-fiting metal plugs were inserted far enough into the ends of such tubular specimens to permit the testing machine jaws to grip the specimens properly. The length of each plug was 50mm. The plugs were not extending into that part of the specimen on which the elongation is measured. Deformation is measured over a length of four times the diameter according following ASTM E8. The Figure 1 represents the project of each specimens for tensile test in accordance the ASTM E8 (2016).

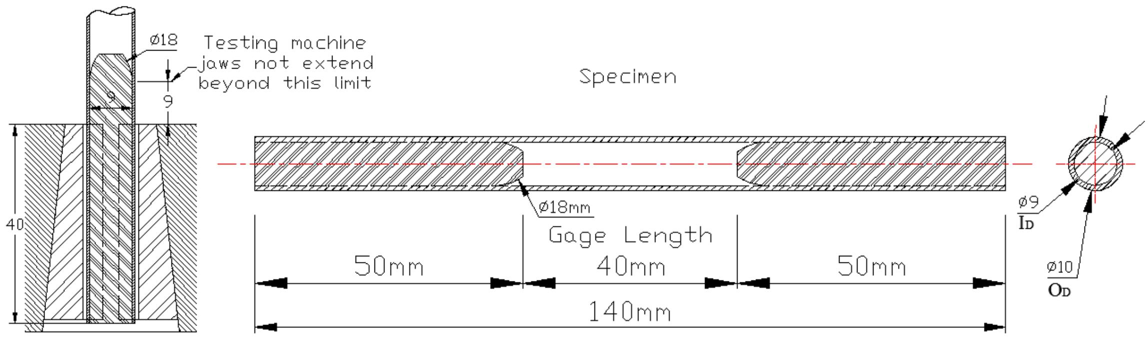

Figure 1. NiTi tube specimens

The tensile test was performed on the universal tensile machine of the manufacturer MTS 809 Landmark Servohydraulic Test System, in the air, at room temperature 22°C with tolerance about ±2°C. The crosshead speed of tensile test was run at 0.05 mm / min and the grip pressure was 7 MPa and a 100 kN load cell was used.

All Tensile Test was following all the recommendation and procedures of ASTM F2516 (2014), Standard Test Method for Tension Testing of Nickel-Titanium Superelastic Materials.

The graphic at the Figure 2 illustrates all related terms extracted of the Tensile Test characterizing the behavior of NiTi Tube. Figure 2 also indicates which points in the graph should be used for calculating the results.

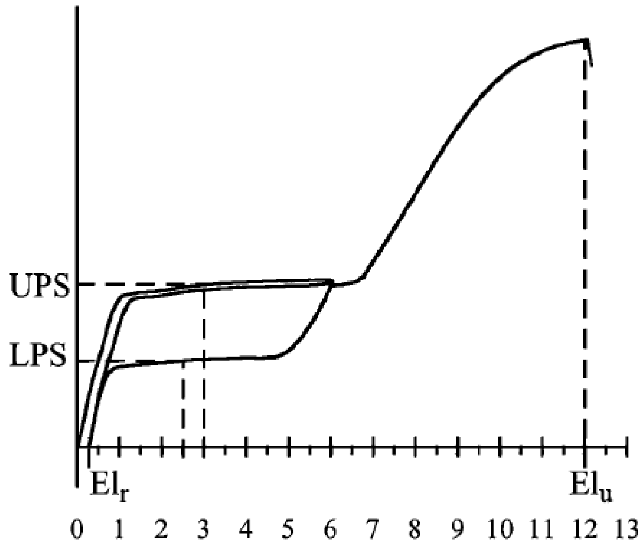

Figure 2. Terms illustrated on Typical Stress-Strain Diagram of Superelastic Nitinol (NiTi) ASTM F2516 (2014). UPS: where occurs the transformation phase from B2 (asutenite) to B19' (martensite). LPS, where occurs the transformation phase from B19' to B2.

3 EXPERIMENTAL PROCEDURES

3.1 Differential scanning calorimetry – DSC

The DSC curves were obtained in the range of -70 °C to 100 °C for both samples, NiTi Tube As Received (NiTi AR) and NiTi Tube Heat Treated (NiTi HT).

In Figure 3, the presence of a phase transformation peak during heating (red curve) and cooling (blue curve) was observed in NiTi AR, such condition the direct transformation of martensite into austenite occurs, between -8.13ºC and 35.25ºC. In the cooling curve the transformation of austenite into martensite occurs between 32.18ºC and -12.37ºC. So, analyzing these temperatures of phase transformation was concluded that at room temperature the as received sample presents austenitic and martensitic phases.

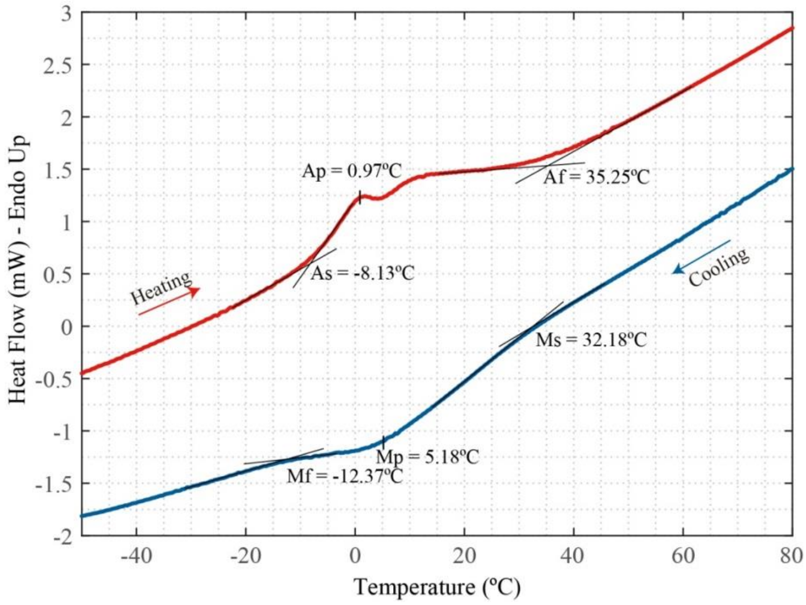

Figure 3. DSC graphic result at As Received (AR) conditional.

The dsc of NiTi HT present too phase transformation peak during heating and cooling the direct transformation of martensite into austenite occurs, between -19.72ºC and -0.97ºC, confirming that at room temperature the alloy is completely austenitic and therefore pseudoelastic. In the cooling curve the transformation of austenite into martensite occurs between -29.89ºC and -43.19ºC. The figure 4 illustrates that the alloy is fully austenitic at room temperature, confirming the effectiveness of the applied solubilization treatment.

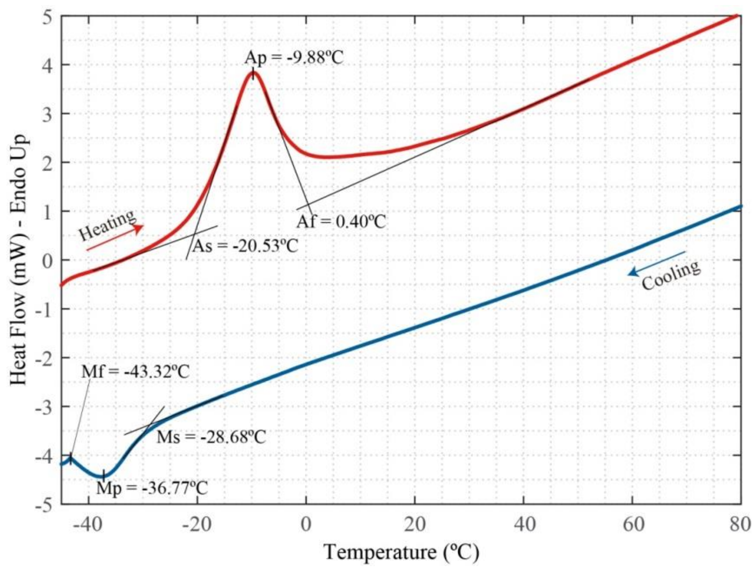

Figure 4. DSC graphic result at Heat Treated (HT) conditional.

3.2 X-ray diffraction – XRD

Figure 5 shows the XRD results for the sample heat treated (HT). In general, the results show that the specimens had austenite phase (B2), that is, they exhibited pseudoelastic behavior.

In the diffractograms it can be observed that all the alloys presented four peaks of greater intensity, around 2θ = 42°, 62°, 78° and 93°.

By comparison with ICDD Sheets at Crystallographica Search Software Match, it can be concluded that the peak 2θ = 42°, is indicative of the NiTi alloy (B2 phase=austenite) and the monoclinic system, martensitic phase of the alloy at room temperature appear at NiTi AR

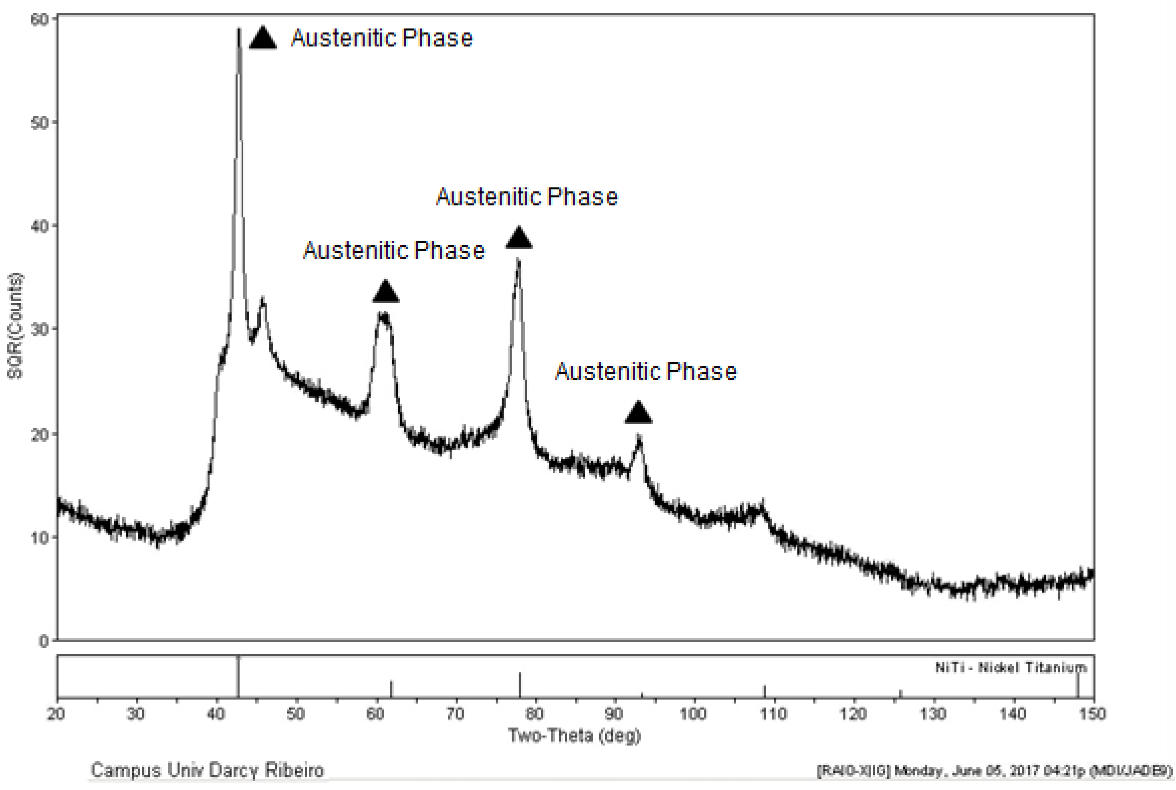

Figure 5. XRD result at HT conditional

3.3 Microhardness – HV0,5

The results of Vickers microhardness are showed in Table 2.

Table 2. Vickers microhardness (HV0,5) results

Vickers Hardness Test (HV 0,5)		
Values	NiTi (AR)	NiTi (HT)
Average value	328.6	254.2
Medium	327	254
Standard deviation	7.8	7.2

It can be observed that the microhardness values are affected by the heat treatment. After the heat treatment of 500°C, the hardness decreased by 22.64% when compared to the AR sample.

3.4 Tensile Test Results

At Figure 6 until 8 are the tensile graphics of studied specimen.

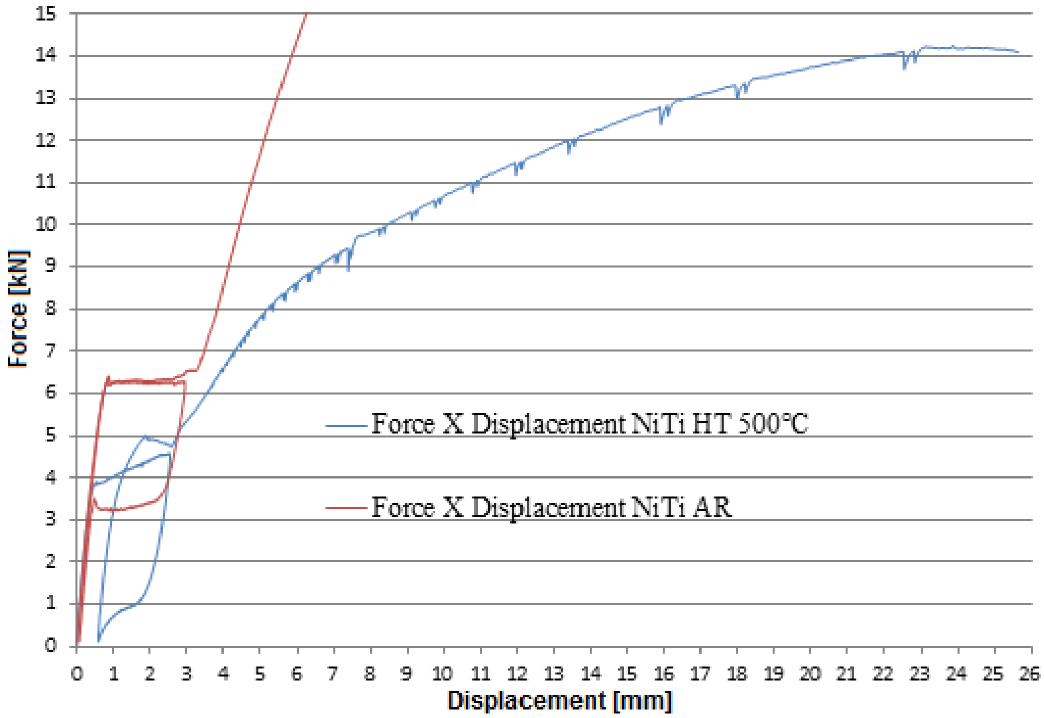

Figure 6. Force [kN] versus Displacement [mm] result of NiTi Tube AR and HT

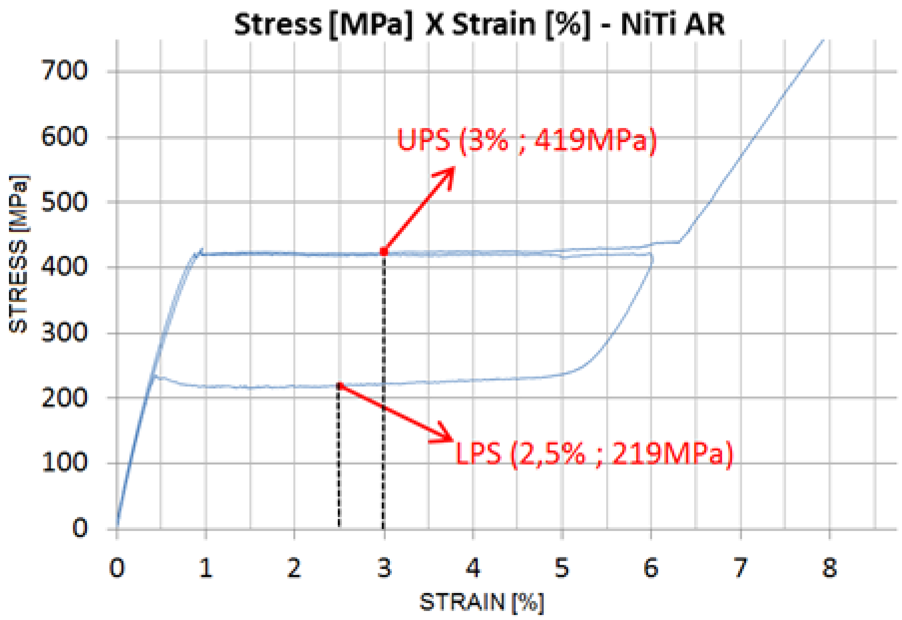

Figure 7. Stress-Strain result of NiTi Tube As Received – Pseudoelastic behavior

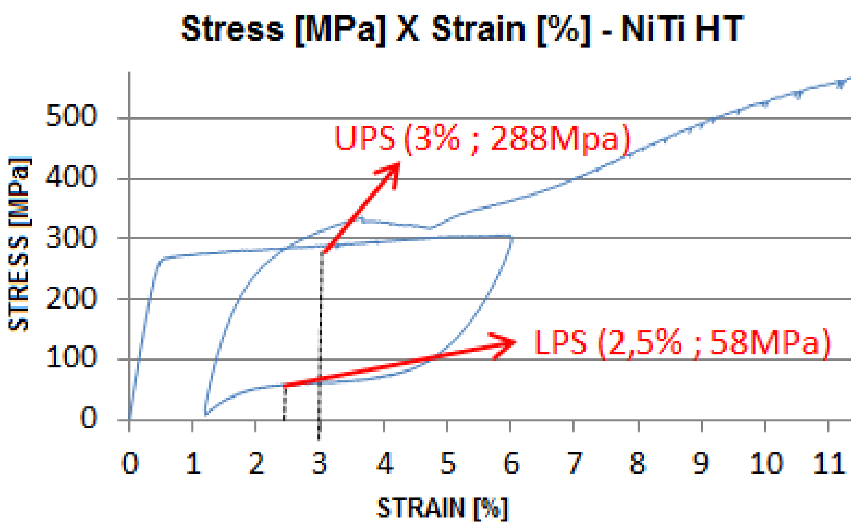

Figure 8. Stress-Strain result of NiTi Tube Heat Treated

The summary of all mechanical properties found at this research are illustrated at table 3.

Table 3. Summary of the mechanical properties

Summary of the mechanical properties found		
Properties	NiTi (AR)	NiTi (HT)
Vicker Hardness (HV)	328	254
Present Phase at Room Temperature	Austenite+Martensite	Austenite
UPS [Mpa]	419	288
LPS [Mpa]	219	58
Elr [%]	0.01	1.18
Elu [%]	25	61.56
Yield Strength [Mpa]	420	290
Tensile Strength (Ultimate strength) [Mpa]	1162	944

Analyzing the tensile tests results was observed that the heat treatment decrease the mechanical resistance of the NiTi tube, that is coherent with the decreasing at microhardness.

4 CONCLUSION

Analyzing the results is possible to conclude that the heat treatment applied on NiTi Tube As Received revealing just austenite phase at room temperature. Also the heat treatment decreased 22.6% of alloy's microhardness. In view of Tensile Test, NiTi Heat Treated increased the hysteretic area and in spite of the pseudoelastic behavior was observed in both conditions the NiTi HT reached 1% of residual deformation in comparison of specimens non-treated (AR). But in other hands the heat treatment decrease the Yield Strength by 31% and Tensile Strength about 18%, reducing 31% of UPS and 73% of LPS of NiTi HT.

ACKNOWLEDGEMENTS

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