Thermomechanical analysis on Ti-Ni shape memory helical springs under cyclic tensile loads

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Abstract: *Recent developments of smart actuators using the two-way shape memory effect (TWSME) for industrial applications are becoming more common in scientific research. Shape memory alloys (SMAs) present some characteristics, which make it unique material to be use in applications that require strength and shape recovery. Ti-Ni alloys are an important class of memory alloys due to the shape memory effect (SME) and superelasticity (SE), which are govern by the thermoelastic martensitic transformation (MT). This material was been used to manufacture smart actuators for mechanical industry devices and several other applications in areas as medicine, robotics, aerospace, petroleum and gas industries. In general, Ti-Ni SMAs undergo one-step (austenite → martensite) transformation, but in some cases, there exists intermediate R-phase giving rise two-step (austenite → R-phase → martensite) transformation. This investigation is interesting due to the importance of knowing the actuators response to external stimulus (heat source, electrical current and/or external stress). In this work it was investigated the mechanical behavior in helical actuators produced from Ti-Ni alloy commercial wires. Different characterization techniques were employ for analyzing the samples as: differential scanning calorimeter (DSC), scanning electron microscopy (SEM), optical microscopy (OM) and a non-commercial apparatus developed to apply an external traction stress in helical actuators during thermal cycles.*

Keywords: Shape Memory Alloys, Ti-Ni Alloys, Heat treatment, R-Phase transformation and Thermoelastic properties.

1. INTRODUCTION

Recently, many studies are being developed in the materials field with the aim to understand the transformation phenomena which involving shape memory smart materials. These materials present phase transformations that allow the research and development of actuator/sensor elements. Mechanical properties of Ti-Ni alloys are very interesting for developing smart actuators manufactured from these non-conventional materials. In many technical applications these actuators need to generate force and avoid degradation of the shape memory effect caused by the martensitic stabilization processes[1, 2]. Shape memory and mechanical properties induce adequate conditions to obtain elements capable to produce mechanical works to be use on industrial applications in areas as medicine, robotics, aerospace, petroleum and gas. Several shapes might be used to obtain a smart sensor/actuators, however helical spring shapes are more interesting because of their deflection, power generation and elastic constant[3].

This research was developed as a mechanical and metallurgical study of Ti-Ni commercial wires with a composition of Ti-50.4at%Ni. Several heat treatments were investigated in order to modify the alloys transformation temperatures. The objectives were to dislocate the critical temperatures to values close to room temperature, besides of understand the R-phase formation, something very common in Ti-Ni alloys submitted to cold work.

Despite several studies to understanding the R-phase formation in the Ti-Ni alloys, there are yet few applications that could use the narrow ranges of shape recovery presented by this phase. Several research indicate that the presence of the R-phase during martensitic transformation (two-steps, B2→R→B19´, where B2 and B19´ represent crystalline structures of the austenitic and martensitic phases, respectively) because of hardening of the matrix phase, hindering the generation of stress fields associated to dislocation reconfiguration process that facilitate the TWSME (Two Way Shape Memory Effect)[4, 5]. A thermoelastic study on shape memory springs, allows the clear understanding of both, the R-phase (R) and martensite phase (B19´) and their behavior due to external stimulus.

In this work some internal defects as dislocations and precipitates, which influencing on the behavior of the shape memory actuator will be treated; also, thermal and thermomechanical cycles that induce martensite variants reorientation, as well as, defects formation which have influence on the actuator thermoelastic strain and critical transformation temperatures modifications are investigated.

2. EXPERIMENTAL PROCEDURE

A cold-drawn Ti-Ni SMA wire with composition of Ti-50.4at%Ni and diameter of 0.89 mm was used to manufacture a helical spring actuator. The actuator was fabricated by forming a wire around a screw and then subjected to heat treatment at 400ºC and 500ºC during specific periods of time. Shape memory springs obtained had 6.0 mm outer diameter, four active coils and 6.0 mm long.

Initially, a study was developed with as-received wire. These wires were subjected to differential scanning calorimeter (DSC) to define martensitic transformation temperatures (martensite start-M_s, martensite finish-M_f, austenite start-A_s, austenite finish-A_f, rhombohedral start or R-phase start-R_s and R-phase finish-R_f). Thermal cycles in DSC were carried out in a range between -60°C and 90°C at constant rate of 10°C·min^{-1}. After this, samples were submitted to heat treatments in order to identify the critical transformation temperatures. Specimens were homogenized in a furnace at 400°C (HT1) and 500ºC (HT2) in periods of time of 1, 2, 4, 8, 12 and 24 hours, followed by quenching in water at 25ºC. For the actuators obtainment it was used the homogenization at 400ºC and 500ºC for 24 hours called (HT1) and (HT2), respectively.

The internal stress and damping capacity was conducted in a Dynamical Mechanical Analysis system. The samples were submitted to a single cantilever fixation and a heating process was developed from 20ºC to 170ºC with rate of 5ºC·min^{-1}. Other parameter used was frequency of 1 Hz and amplitude oscillation of 5µm.

The microstructure of the material was analyzed in order to identify precipitation and to visualize the martensitic variants. Optical Microscopy, Scanning Electron Microscopy and Energy Dispersive Spectroscopy were carried out to analyze Ti-Ni wires submitted to HT1 and HT2. Wires were cold-mounted by compound resin, after that, wires were roughened using sand-paper and polishing was performed with alumina of 1 and 0.5 µm. Samples were etched using HF-HNO3-CH3COOH, in the ratio of 2:5:5 for periods of 1 or 2 seconds.

Shape memory springs (SMS) were submitted to thermomechanical cycles in a loading special apparatus. This apparatus is constituted by a programmable silicon oil bath, linear variation displacement transducer (LVDT), thermocouple and a data acquisition system. SMS were subjected to 40 thermal cycles under constant shear stress of 35, 70, 105, 135, 170, 200, 235 and 270 MPa in a temperature interval between 25 and 140°C. The heating and cooling rate was estimated in 10 and 4ºC.min^{-1}, respectively[4, 6, 7].

From data acquisitions, curves of deformation versus temperature and temperature versus number of cycles were plotted. Figure 1 shows a typical graph obtained during the training process, as well as, the critical transformation temperatures under stress (A_S, A_f, M_S and M_f) using the tangent method, thermoelastic strain (ε_t = difference between the strain at low and high displacement), thermal hysteresis (H_T) and vertical displacement of hysteresis loops (X)[4, 6].

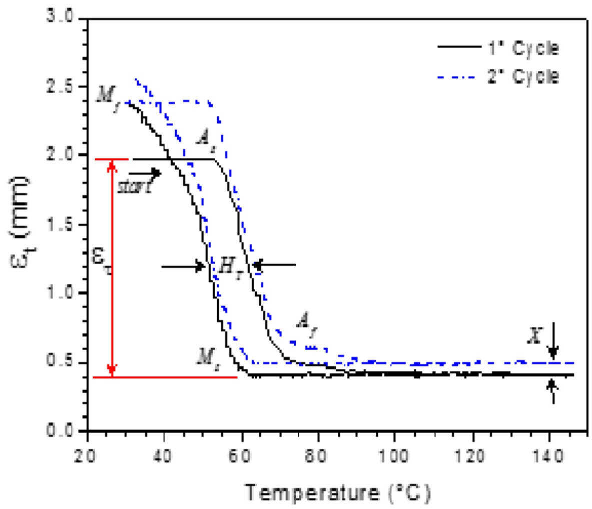

Figure 1. Characteristic parameters of the shape memory effect in a strain versus temperature curve.

3. RESULTS

3.1. Influence of heat treatment on phase transformations

This investigation was based on transformation temperatures and enthalpy changes according to the heat treatment. The material was analyzed in as-received and heat treatment conditions.

Figure 2(a) shows curves of heat flow as function of temperature for DSC analysis on Ti-Ni wire in as-received conditions. DSC results do not shows any signal of transformation peaks for direct and inverse transformation. It might be explained from the material structure, which was filled by process defects. These defects act like obstacles for phase transformation occurrence because they block the movement of martensitic variants[4, 5, 8, 9].

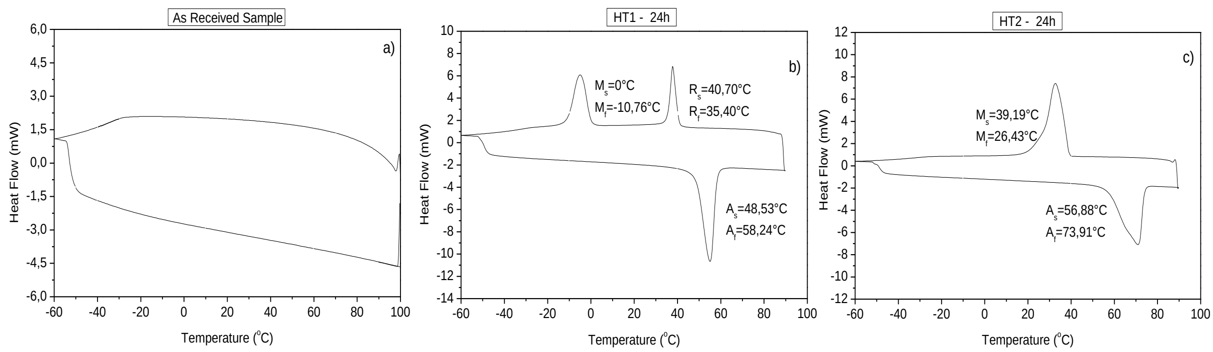

Fig. 2. DSC analysis for Ti-Ni wire for: a) as-received condition, b) HT1-24h and c) HT2-24h.

Table 1 shows DSC results for samples heat treated at 400ºC and 500ºC for 1, 2, 4, 8, 12 and 24 hours, respectively.

Table 1. Critical temperature transformation for samples heat treated at 400ºC and 500ºC.

Time (h)	HT1								
	As	Af	ΔH (J/g)	Ms	Mf	ΔH (J/g)	Rs	Rf	ΔH (J/g)
1	33,84	42,95	20,56	-22,17	-38,77	12,30	33,30	25,65	6,59
2	40,25	50,21	21,90	-14,30	-30,83	14,81	35,31	28,93	7,05
4	44,10	59,56	19,69	-12,44	-33,72	13,33	38,15	27,89	7,38
8	45,23	55,17	22,88	-7,22	-19,97	14,48	40,22	34,83	8,70
12	46,76	56,67	22,54	-4,18	-16,27	14,14	40,23	34,65	7,75
24	48,53	58,24	21,03	0,06	-10,76	12,16	40,70	35,40	7,10
Time (h)	HT2								
	As	Af	ΔH (J/g)	Ms	Mf	ΔH (J/g)	Rs	Rf	ΔH (J/g)
1	16,69	27,28	20,31	-23,89	-35,92	13,96	8,43	2,65	4,99
2	19,15	31,27	22,16	-16,80	-32,15	16,82	9,45	2,89	5,91
4	23,07	36,26	19,82	-6,59	-24,78	12,43	12,55	6,10	4,32
8	26,07	43,53	22,41	1,45	-19,50	18,42	15,00	8,40	7,79
12	36,00	64,31	24,84	33,54	2,80	26,06	x	x	x
24	56,88	73,91	24,81	39,19	26,43	25,99	x	x	x

DSC analysis showed that samples thermally treated present modifications on phase transformation and its temperatures had been dislocated. All samples heat treated at 400ºC exhibit a transformation in two steps (B2→R→B19´) for all annealing periods. According to studies two steps transformation occurs due to precipitates formed during heat treatment in rich Ni alloys[10, 11]. Temperatures were dislocated in the sense of increasing the critical transformation temperature. Annealed samples at 400ºC showed A_s variation from 33.8ºC for 1 hour to 48.5ºC for 24 hour ageing. M_s showed variation from -21.2ºC for 1 hour to 0ºC for 24 hours ageing.

Samples heat treated at 500ºC showed austenitic, R-phase and martensitic phase evolutions, where transformation temperatures tend to reduce the thermal hysteresis. In the ageing test carried out at 12 hours it is observed that R phase practically disappears, but a much larger hysteresis is also observed. The modification observed on DSC results, where temperature and transformation phases are involved, this changes have been investigated in the literature and two main causes are reported: near-equiatomic Ti-Ni SMA lightly rich in nickel can form precipitates during heat treatment at high temperatures, such as Ti_3Ni_4, Ti_2Ni_3 and $TiNi_3$[8, 10, 12]. These precipitates have influence on MT (martensitic transformation) because act as preferential sites for nucleation reaction, inhibiting and/or suppressing the appearance of R-phase. Precipitation occurs from diffusional processes which involve changes in local chemical composition, modifying transformation temperatures in such a way that increase of M_s transformation temperature[7, 13-16]. A second important factor is about deformation stage or dislocation density configurations. This factor depends mainly of the material processing method, thermomechanical heat treatments and others. The wires used in this work were obtained by a cold-drawn process that results in a material with high dislocation density. Normally, alloys with high dislocation density show two transformation peaks during cooling which corresponding R and B19´phases[4, 16, 17].

3.2. Microstructural investigation

Figure 3 shows images of microscopy for samples heat treated with HT1-24h and HT2-24h. Acicular structures similar to martensitic variants can be visualized. The images revealed morphological distinction between them. Figure 3(a) shows a high number of well-defined acicular structures with different orientation. Compared to samples heat treated at HT1, samples subjected at HT2 shows less acicular form and larger grains due to a higher heat treatment temperature. According to literature M_s transformation temperature increase with the increasing of grain size[18, 19]. DSC results shown on tables 1, summarize transformation temperatures and enthalpy values for heat treated samples.

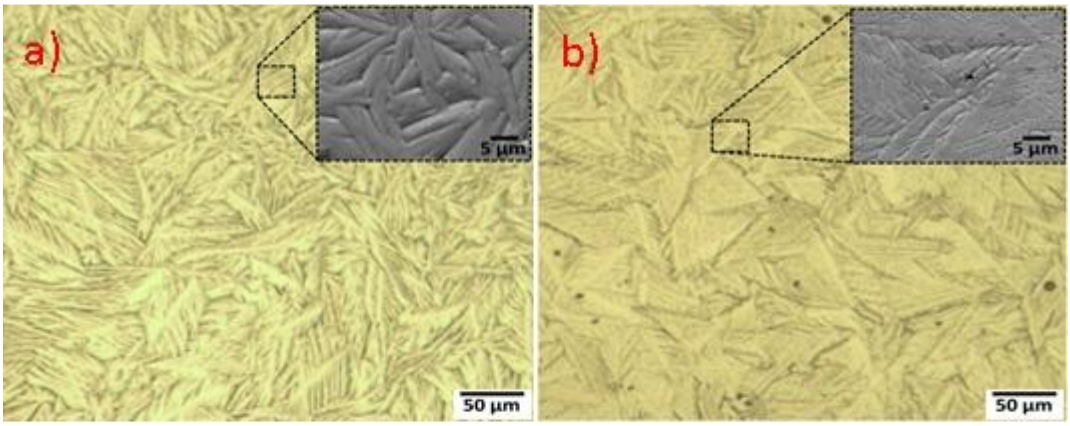

Figure 3. Microscopy images for heat treated Ti-Ni alloy, (a) HT1-24h and (b) HT2-24h.

Figure 4 shows energy dispersive spectroscopy (EDS) results for samples submitted to HT1-24h and HT2-24h. EDS was developed in a small region of the samples. Both samples show peaks of Ti and Ni elements. Results indicate in a qualitative way that the samples studied are Ni-rich. According to the manufacturer, the alloy involved in this research is a Ni-rich, with a chemical composition of Ti-50.4at%Ni.

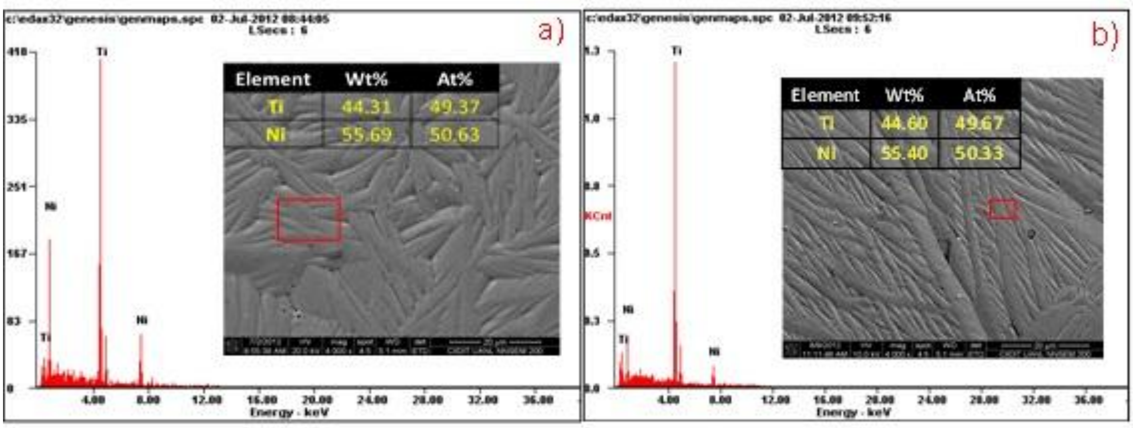

Figure 4. Energy dispersive spectroscopy (EDS) results for samples submitted to a) HT1-24h and b) HT2-24h.

3.3. Thermomechanical analysis

Thermomechanical heat treatment process were performed in thermal silicone oil bath, known in literature as a training process[20]. The apparatus used for this work, was able to developing the heating and cooling on the actuators, which consequently induced phase transformation in the samples. A constant shear stress was applied in the actuator in order to induce martensite variants in a preferential direction in relation with the applied stress. During the process the training leads formation of preferential variants who induces shape modification and transition on the transformation temperatures[21].

The obtained graphs from training process allowed the studied on several shape memory effect parameter modifications (transformation temperatures, hysteresis, thermoelastic deformation and enthalpy). Thermal hysteresis and thermoelastic effect are also important parameters on the actuators behavior. Thermoelastic parameters are capable to indicate the shape memory efficiency as a function of the linear displacement. Figure 5 shows curves of thermoelastic effect versus temperature during forty cycles for tensile stress of 105MPa for HT1 and HT2 heat treatments.

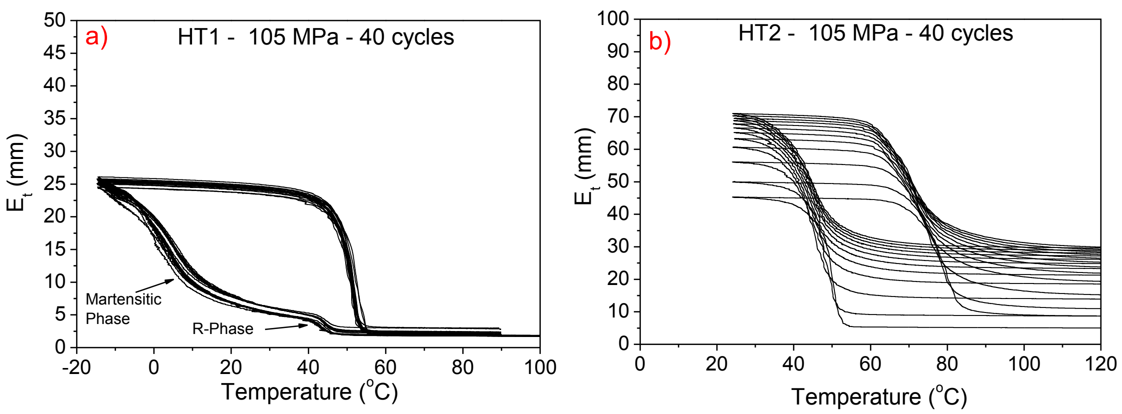

Figure 5. Thermoelastic effect versus temperature during forty cycles for tensile stress of 105 MPa, (a) HT1.

Among the most important information obtained during the training process, it was observed the strain variation due to temperature, the vertical displacement on hysteresis loops, the increasing on the thermoelastic strain (E_t) caused by stress increment, besides of the double "s" observed during cooling on samples heat treated at HT1. The vertical displacement on hysteresis loops might be attributed to transformation induced plastically (TRIP) due to accumulation of small plastic deformation[22, 23]. Other factors also may contribute, as for example: spring actuator displacement due to rotation, martensitic stabilization process and martensitic variants reorientation[6].

Figure 6 shows thermoelastic strain curves (E_t) for each applied tensile stress. Initially, thermoelastic strains show an increase according with stress increments, but their evolutions for each heat treatment during training cycles exhibit same differences.

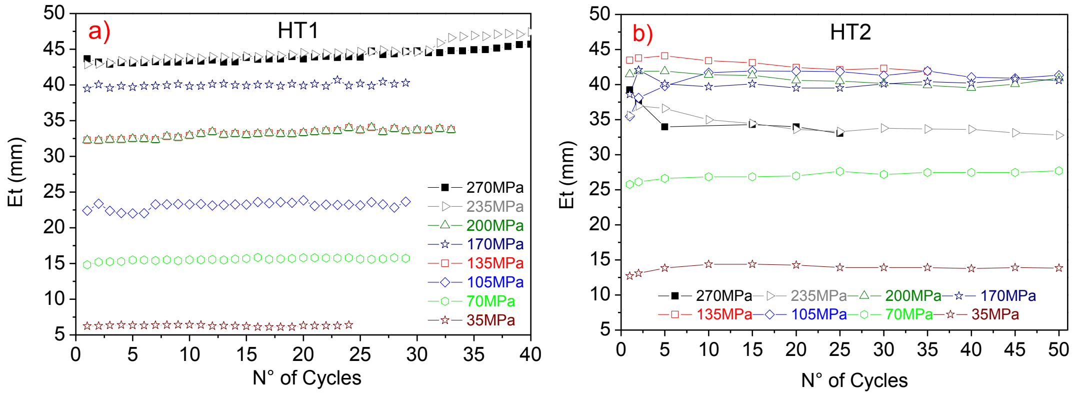

Figure 6. Thermoelastic deformation versus cycles for 35, 70, 105, 135, 170, 200, 235 and 270 MPa stress. a) HT1-24h and b) HT2-24h.

Evaluating samples submitted to HT1 it was observed that for 70 and 105 MPa a gradual increase during training cycles until practically strain stabilization for the last cycles. It was also observed for results obtained with stress of 200 MPa a sharp fall on thermoelastic strain to levels near the obtained for 135MPa. It is possible that this particular stress might be involved with R phase transformation suppression. In this case (200 MPa) it was observed during experimental procedure that the two steps transformation (B2→R→B19´) starts to be replaced by one step transformation (B2→B19´)[12].

During the training process occurs martensitic variants reorientation where internal stresses generated by dislocation fields becoming preferential according to stress direction[20]. For low and intermediate stresses, internal stress fields are formed gradually in each cycle, increasing thermoelastic strain during training. In this sense, 105 and 135 MPa stress present the best results in comparison with tests developed, whereas 170 and 200 MPa stress produce a decrease of the thermoelastic strain due to the rapidly saturation of internal stress fields. These behaviors have been observed in cooper based shape memory alloy springs[5].

Actuators heat treated with HT2 also exhibit an increase on the thermoelastic strain with stress increments; however, for low levels of stress, the actuators produced a better efficiency on the TWSME comparing to HT1 results. Stress of 105, 135, 170 and 200 MPa present almost the same thermoelastic strain near 40.0 mm (see fig. 5). The main difference from HT1 results is that for 235 and 270 MPa, the actuators exhibit a better SME. The main results suggest that due to R-phase transformation mechanism and the developing of preferential variants orientation for samples heat treated at HT1-24h a better SME efficiency[24].

The efficiency of the TWSME might be evaluated by temperature transformation evolution. Figure 7 shows the evolution of (M_S) for both heat treatments. Samples heat treated with HT1 shows for all studied situations an increase on transformation temperature when increasing cycles and stress, and a more homogeneous temperatures evolution. The actuators obtained used from samples heat treated at HT2 showed higher values of M_S and a high level of permanent deformation for 235 and 270 MPa which did not allow the continuity of the test for those conditions.

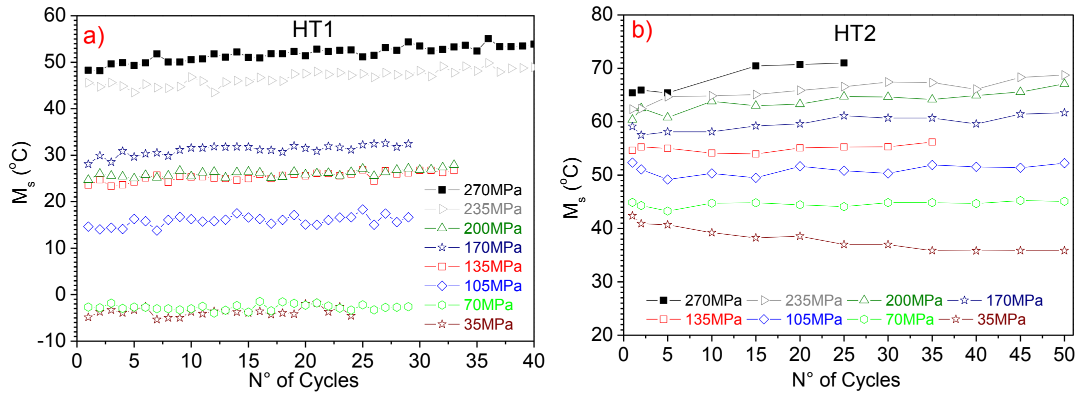

Figure 7. Ms evolution for 35, 70, 105, 135, 170, 200, 235 and 270 MPa applied stresses, a) HT1 and b) HT2.

3.4. Training Process vs. Internal Friction

It was already observed that internal friction in near equiatomic Ti-Ni alloys is related to stress barrier for martensite reorientation. Has been found that these stresses are strongly related with the annealing temperature, internal stress fields and their interaction with other lattice defects25. Heat treatment and training process seriously affects the internal friction behavior in Ti-Ni alloys26, 27. These events have an importance on the reconfiguration of Ti-Ni alloy defects, inducing modification that might change the material properties such as memory effect, damping capacity, strength, hardness, and others26,27. Martensitic transformations are associated to the shear stress direction applied during the procedure. According to studies Ni/Ti ratio has influence on internal stress degree in the parent and martensite phase26. Internal defects propagation hinders the movement of martensite-austenite interface, inducing thermoelastic transformation to require more energy to overcome internal stress between phases. The increase on transformation energy can be visualized on the Ms behavior, which increases with increasing applied stress and number of cycles (fig.7)6, 20. Figure 8 shows internal friction (tan δ) (fig.8(a)) and storage modulus (fig.8(b)) as function of temperatures during cooling step by DMA. Figure 8(a) indicates that the peak temperature for austenite to R phase and R phase to martensite transformation occur at 115ºC and 73ºC, respectively for HT1 heat treatment. Figure 8(b) indicates austenitic transformation to occur at 100ºC for HT2. Figure 8 also exhibit a high level of internal friction on samples submitted to HT1 where internal friction reaches 0,1528. Same studies developed a relation between R-phase transformation and damping capacity where the occurrences of R-phase significantly soften the storage modulus and thus increase the internal friction29-32. Samples heat treated with HT1 exhibit a higher internal friction, compared with HT2. It is possible that due to the more high temperature (500ºC) near recrystallization temperature (580ºC) the material induced more easily changes on stress field's interaction and also precipitate formation due to Ti-Ni decomposition.

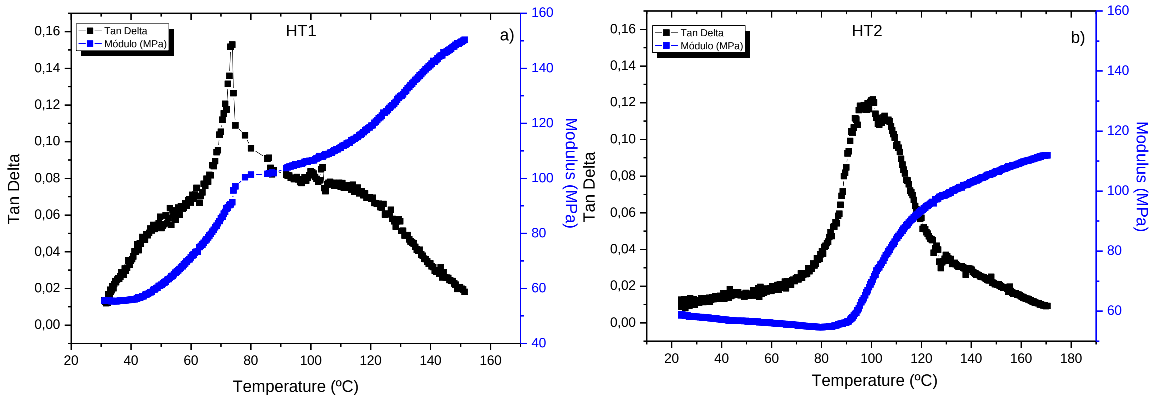

Figure 8. Internal friction tan δ and storage modulus as a function of temperature during step cooling for Ti-Ni alloy by DMA.

Figure 9 shows M_S versus stress for 1, 5, 10, 15, 20 and 25 cycles. For both heat treatments M_S is increasing when stress is being increase. For samples heat treated at HT1 (fig.9(a)) it is detect two points under the linear fit. These points refer to stress of 70 and 200 MPa, exactly the same values who exhibit a sharp fall on thermoelastic strain (200MPa - fig.7). It is possible that 70 MPa might be a stress where internal friction starts its influence on martensitic transformation, resulting in an M_S lower than those obtained for 35 MPa. Changes observed for 200 MPa might be due to interaction of stress fields on martensitic and R-phase during training process.

Figure 9(b) shows samples heat treated at HT2 where M_S for 35 MPa has been reduce when the number of cycles is increasing. For stress between 70 and 170 MPa, the M_S exhibits a very narrow variation, being this temperature parameter very close for different cycles. For stress between 200 and 270 MPa, M_S becomes very wide for increments with the number of cycles.

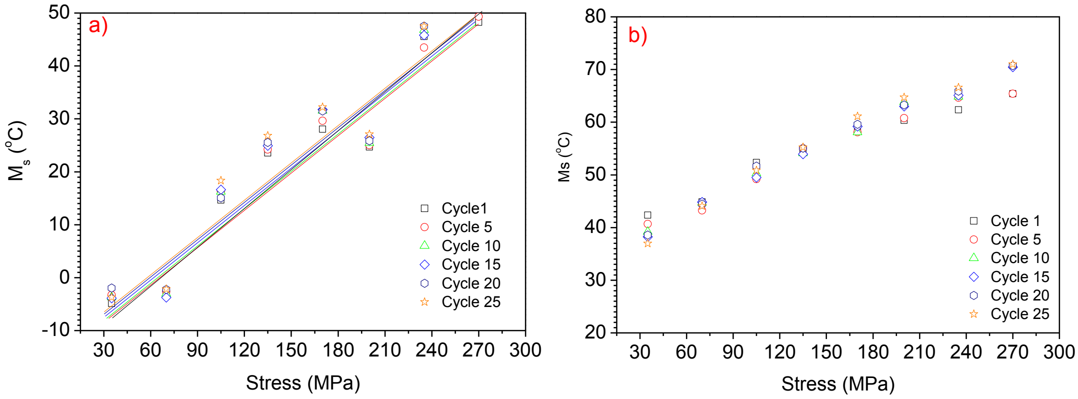

Figure 9. M_S variation due to stress and thermomechanical cycles.

4. CONCLUSIONS

In the present study can be concluded that:

Aging Ni-rich Ti-Ni alloys dislocate the transformation temperature and tend to suppress the R-phase transformation due to elevated aging periods and heat treatment temperature.

R-phase transformation seems to improve TWSME due to elevate interaction with stress fields. The interaction between martensitic and R-phase stress fields induce an increase on temperature transformation and a martensitic variants preferential orientation. HT1 heat treatment seems to conserve better the interaction between stresses fields than the HT2, and so, more efficient shape recovery.

The ageing heat treatment HT1 and HT2 has different effects upon the samples, where HT1 exhibit a two steps transformation and HT2 a single step transformation. The R-phase involved with two steps transformation interacts with internal stress fields inducing modifications on transformation temperatures, internal friction and shape memory efficiency.

5. ACKNOWLEDGMENTS

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