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INFLUENCE OF ANNEALING TREATMENTS ON THE THERMOMECHANICAL BEHAVIOR OF SMA SUPERELASTIC MINI COIL SPRINGS

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Abstract. *Shape Memory Alloys (SMA) are smart materials capable of generating force and/or displacements in mechanical systems. This remarkable behavior occurs due to a reversible phase-transformation that can be either thermally activated (Shape Memory Effect), or mechanically activated (Superelasticity). Making use of varied geometrical configurations it is possible to optimize the application of these SMA as thermomechanical actuators. For example, helical coils manufactured from SMA wires are capable of recovering strain levels about 60 times greater than single wires under tensile stress. This characteristic, allied to the fact that most engineering applications require the use of helical coils actuators, makes very important the proper and more accurate design of these devices. Previous studies indicate that heat treatments for relief of residual stresses, derived from i.e. manufacturing processes, allow the manipulation of properties like stiffness and energy dissipation capacity in function of strain and temperature. In this context, the purpose of this work is to present a mapping of thermomechanical properties of NiTi superelastic mini coil springs commercially available. Heat treatments at different temperatures are carried out. After each heat treatment of annealing, specimens are tested using a dynamic mechanical analyzer (DMA) in a uniaxial tensile mode with simultaneous heating to evaluate the changes in thermomechanical properties.*

Keywords: *Shape Memory Alloys; Mini coil springs; Superelasticity; Heat treatment.*

1. INTRODUCTION

Shape Memory Alloys (SMA) are advanced engineering materials capable of shape recovery either through heating and cooling – shape memory effect (SME) – or through mechanical loading and unloading – superelasticity (SE). Through a reversible phase-transformation two crystallographic solid phases appear in the SMA: martensite, at lower temperature and with lower stiffness (elasticity modulus); and austenite, at higher temperature and with higher stiffness. In order to study the mechanical behavior of SMA, besides stress and strain, the temperature variable must be included, since it determines the state of the alloy and, therefore, the macro response of the material (SME or SE). When it is deformed at the martensitic state, the SME takes place and even after mechanical unloading large strain levels are kept, being the original shape restored after a heating above the A_f temperature (when the SMA becomes totally austenitic). When deformed at the austenitic phase, the SE is observed and mechanical unloading only is necessary to promote the total shape recovery.

These materials present high actuation energy density, being able to perform large amounts of mechanical work. On the other hand they present low actuation frequency (Lagoudas, 2008) due to the thermal nature of the activation mechanism. Thus, the velocity with which the material responds depends deeply on the heat transfer properties of the system i.e. the SMA mass and its environment heat exchanges. As a result, SMA have been mostly used as actuators in small-scale systems where a faster response is achieved, little space is available and low force levels are required.

Coil springs are mechanical elements present in many engineering applications where large deflections and minor strength levels are needed. While classical coil springs are designed to bear elastic shear deformation up to 1%, SMA coil springs can achieve 6%-8% of shear deformation and still recover its original shape (An *et al*, 2012). Besides, SMA manufactured in a three-dimensional shape, as helical springs, are capable of recovering strain levels about 60 times greater than in one-dimensional elements such as SMA wires under tensile stress. Some examples of applications of SMA mini coil springs include systems where thermal activation is possible, mainly by resistive heating; and also in dynamic systems, to control the dynamic response through a change in stiffness of the SMA that follows a change in temperature.

While in the austenitic phase, presenting superelasticity, the spring's mechanical behavior is non-linearly strain dependent and at the end of one force-elongation cycle a closed loop is observed. This characterizes a hysteretic behavior and the area inside a superelastic loop is equivalent to the amount of mechanical energy dissipated per cycle. Hence, besides actuating as an elastic element restoring the original position of the system, it works as a damping element, dissipating mechanical energy. Furthermore, by increasing the temperature the SMA's stiffness increases and this difference may be enough to, for instance, avoid resonance in dynamic systems, or even eventually or periodically generate forces/displacements in quasi static applications.

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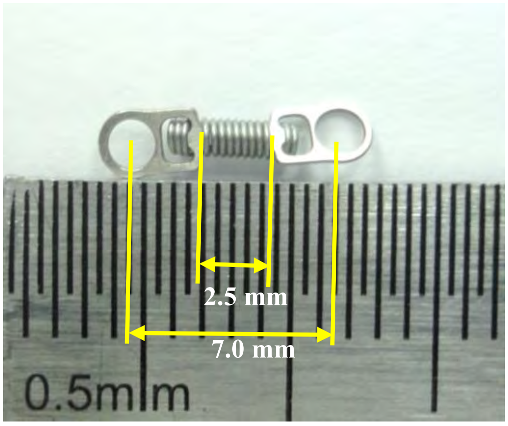
Spring's stiffness is tightly related to material's elastic modulus, which in turn is related to material itself and manufacturing processes, amongst other variables. It is known that high-density of crystalline defects, large residual stresses and distortion can be generated in cold-working manufacturing processes. More specifically, SMA properties like transformation temperatures of NiTi SMA can be significantly changed by cold or hot-working (Paula *et al*, 2004), since SMA phase transformation occurs through displacive movement of atoms and the internal stress state directly affects their mobility.

Helical springs are usually made from mechanically shaped wires and the imperfections generated in the microstructure, affecting greatly martensitic transformation behavior, can be reduced or released by annealing treatment (Lin, 2007). Previous studies indicate that stress relief annealing treatments allow the manipulation of properties like stiffness, energy dissipation capacity, and shear modulus in helical springs (Grassi *et al*, 2012). This treatment consist in heating the material at relatively moderate temperatures, under the temperature of recrystallization of the alloy. During this process atoms move to more stable positions in the crystal lattice, vacancies and interstitial defects are eliminated and some dislocations are annihilated (Kopeliovich, 2012).

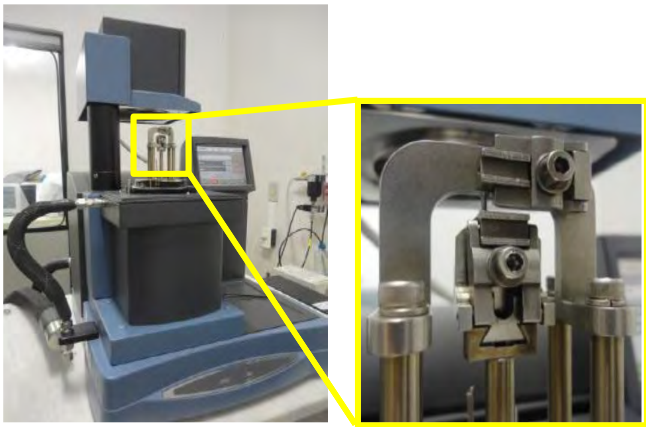
In this context, the purpose of this work is to present a mapping of thermomechanical properties of NiTi superelastic mini coil spring commercially available. Heat treatments at different temperatures are carried out. After each heat treatment of annealing, specimen is tested using a dynamic mechanical analyzer (DMA) in a uniaxial tensile mode with simultaneous heating to evaluate the changes in thermomechanical properties.

2. MATERIALS AND METHODS

The spring used in this experimental study was a NiTi superelastic mini coil spring supplied by Dental Morelli with 7 mm in length, denominated M7. This length is indicated by the manufacturer and actually correspond to the distance between the spring's eyebolts, installed at each end of the mini spring. The spring has 9 active coils (n), 1.25 mm of external diameter (D) and is manufactured from a 0.22 mm NiTi superelastic wire (d). In Figure (1a) it is possible to observe the configuration of this NiTi mini coil spring and its deformable length, of 2.5 mm. Figure (1b) shows the spring mounted on the DMA equipment's clamp.



(a)



(b)

Figure 1. NiTi mini coil spring. (a) M7 mini coil spring with 2.5 mm of deformable length. (b) M7 mounted on uniaxial tensile clamp of DMA Q800, from TA Instruments.

Using the dynamic mechanical analyzer (DMA), model Q800 from TA Instruments, in a uniaxial tensile mode the NiTi mini spring went through the following sequence of experimental tests:

1. *Training*: since it was detected the *as received* mini spring was not mechanically stabilized, 250 superelastic cycles were performed until a total deformation of 500% at 500%/min loading and unloading rate;

2. *Thermomechanical characterization*: after training each NiTi mini spring was submitted to uniaxial tensile tests at five temperatures: 30°C, 35°C, 40°C, 45°C, and 50°C. The strain levels used were the same as in training process, but at strain rate of 100%/min. These tests allowed the evaluation of stiffness and energy dissipation capacity properties as function of temperature;

3. *Annealing heat treatment*: the NiTi springs were furnace annealed for 20 min followed by air quench. The used annealing temperatures were: 200°C, 300°C, 400°C and 500°C. After each heat treatment the NiTi springs passed through steps 1 and 2 again;

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4. *DSC test*: along with the mini spring M7 one other sample was annealed at each temperature and tested by differential scanning calorimeter (model Q20 from TA Instruments) between -50°C and 150°C in order to observe the effect of heat treatment over SMA phase transformation temperatures. In order to avoid influence from cutting process, DSC samples were cut using a diamond disk mounted on a metallographic cutter under low rotation speed.

From the obtained results, stiffness (K, N/m) and energy dissipation capacity (E, mJ) values were calculated using the methodology shown in Fig. (2). Shear stress (G, GPa) at 30 °C was also calculated using Eq. (1) for classical coil springs. These properties values were discussed as function of test temperature and annealing temperature.

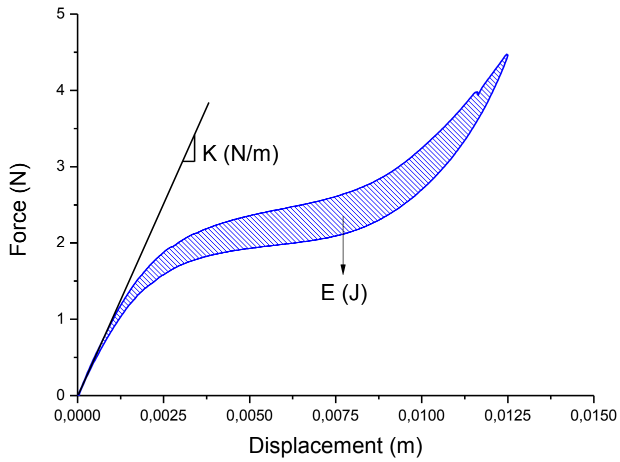

Figure 2. Scheme of a superelastic curve of the NiTi SMA mini spring showing the methodology to calculate stiffness of austenitic phase (K) and energy dissipation capacity (E).

$$F = \frac{Gd^4}{8D^3n} \quad \rightarrow \quad G = \frac{8KD^3n}{d^4} \tag{1}$$

Where F is the force imposed on the spring in Newtons; G is the shear modulus of the material in Pascal; K is the spring's stiffness in N/m; D is the spring diameter in meters; d is the wire diameter in meters; and n is the number of active coils.

3. RESULTS AND DISCUSSION

Figure (3) show a set of force vs. strain superelastic curves demonstrating how the annealing temperature affects qualitatively the thermomechanical behavior of the NiTi superelastic mini spring. Figure (3a) show this behavior in function of test temperature for the as received NiTi superelastic mini spring, where it can be seen a proportional increase in force with increasing temperature. In Figures (3b) to (3f) the thermomechanical behavior as a function of annealing temperature is observed. As expected, an increase in annealing temperature caused a decrease in the force to achieve the same strain level. However, the annealing at 200 °C and 300 °C caused practically no change in superelastic behavior, regardless the test temperature, indicating the existence of a minimum temperature for the stress relief heat treatment. Since these mini springs and NiTi SMA are widely used in biomedical field, this result assures, for example, that a sterilization temperature below 300 °C will not affect thermomechanical response of the actuator. On the other hand, annealing at 400 °C and 500 °C caused a significant decrease in force levels and an increase of superelastic loop area, indicating a decrease in stiffness and increase in energy dissipation capacity.

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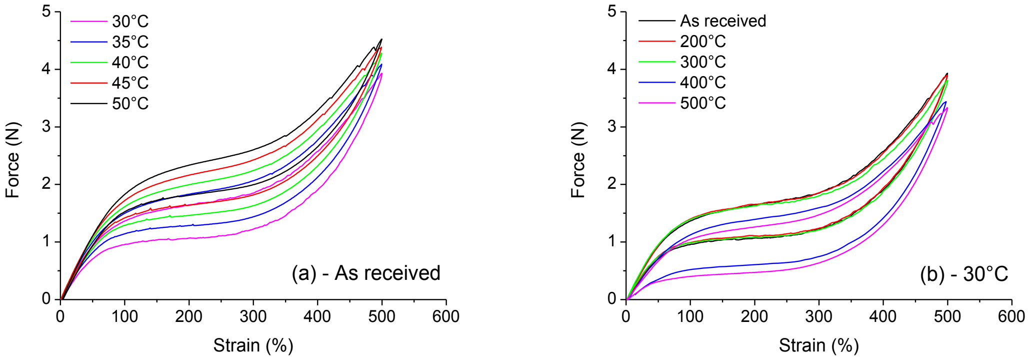

Figure 3. Superelastic behavior of the M7 mini spring sample in function of temperature and annealing temperature (continues).

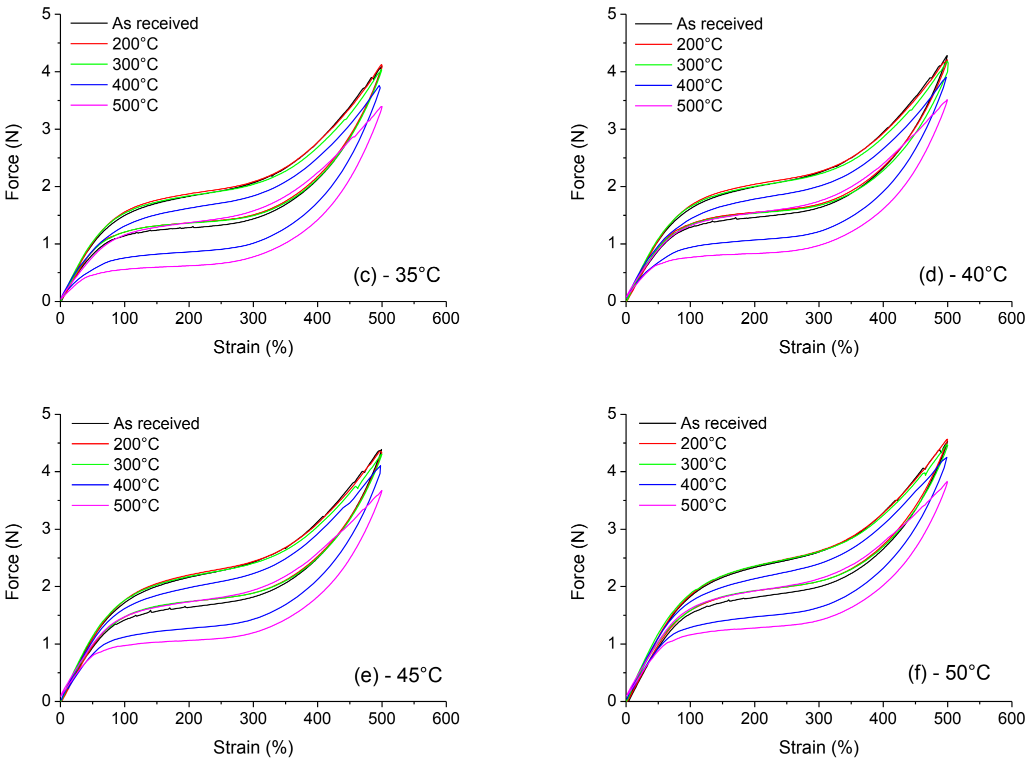

Figure 3. Superelastic behavior of the M7 NiTi mini spring in function of temperature and annealing temperature.

Figure (4) show the stiffness K (N/m) of austenitic phase calculated from curves in Fig. (3) from 0 % to 30 % strain levels, guaranteeing an homogeneous phase at all tested temperatures.

Absolute values of stiffness in Fig. (4a) indicate that in this phase only annealing treatments at 400 °C and 500 °C were capable of decreasing the stiffness. For the other studied annealing temperatures, stiffness values found were relatively close. For all heat treatments it was observed an increase in stiffness with increase of test temperature. This result is equivalent to the Clausius-Clepeyron relationship between stress and temperature (Otsuka & Wayman, 1998), since even K is calculated from force values and not stress values, these two are related by a constant (shear area).

Figure (4b) show the percentage variation in function of test temperature, related to the first obtained value of K (at 30 °C). The annealed NiTi mini springs at 400°C and 500 °C had a higher maximum variation (at 50°C), of about 18% and 37%, respectively. For the other annealing temperatures a lower level of variation was observed, between 6.5% and 13% (at 50°C). Figure (4c) was plotted to allow a better visualization of the effect of annealing temperature on stiffness. K values are here compared to the results of the NiTi mini springs in the a*s received* state. As discussed above, little variation of K is observed until annealing at 300 °C, indicating the existence of a minimum temperature (between 300°C and 400 °C) at which the stress relief heat treatment is effective.

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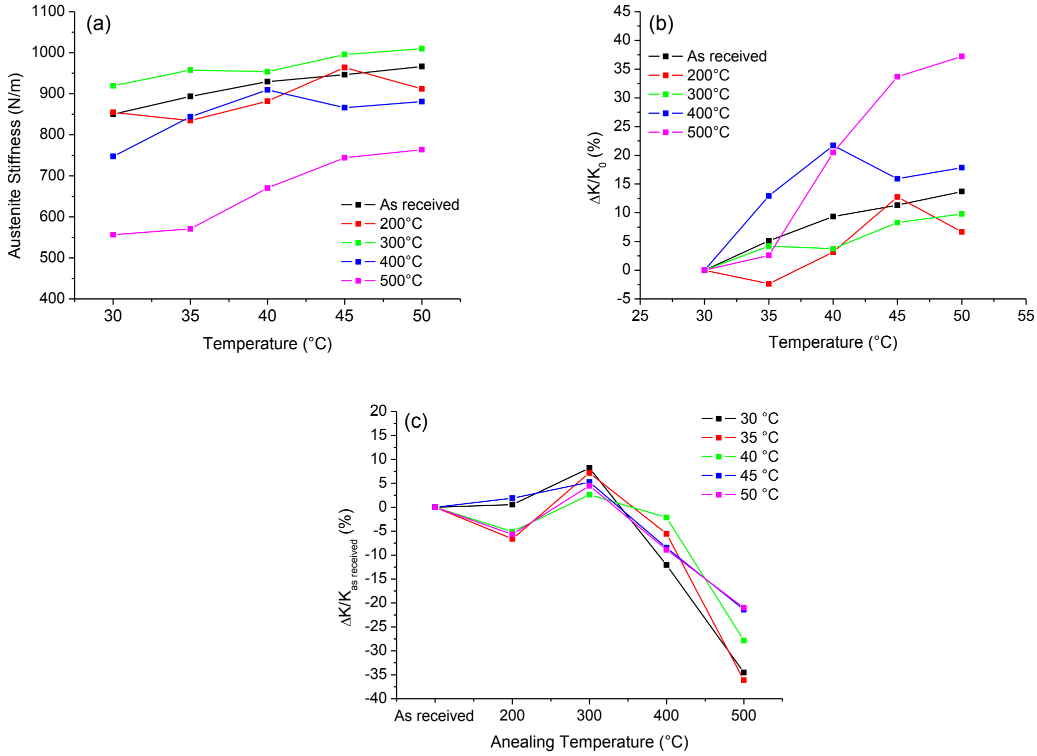

Figure 4. Stiffness behavior of M7 NiTi mini spring in function of test temperature and annealing temperature. (a) Absolute values. (b) Percentage variation related to the first value (at 30°C). (c) Percentage variation related to the stiffness value of the *as received* sample.

Figure (5) contains data plots of energy dissipation capacity calculated as the area inside the superelastic loop from Force (N) vs. Displacement (m) curves. As expected, energy dissipation behaves as opposed to stiffness and a practically linear decrease of E was observed when test temperature increases. In Figure (5a) it is noticed two levels of E: a lower level with the NiTi mini spring at the as received state and annealed at 200 °C and 300 °C; and a higher level, with results from NiTi mini spring annealed at 400 °C and 500 °C. Figure (5b) shows that the maximum variation of E was about -19 % with an increase of 20°C in test temperature for the annealed sample at 500°C. Figure (5c) shows a relatively homogeneous variation of E with increasing annealing temperature for the different test temperatures. Again, two levels of E can be easily detected, in accordance with absolute values in Fig. (5a).

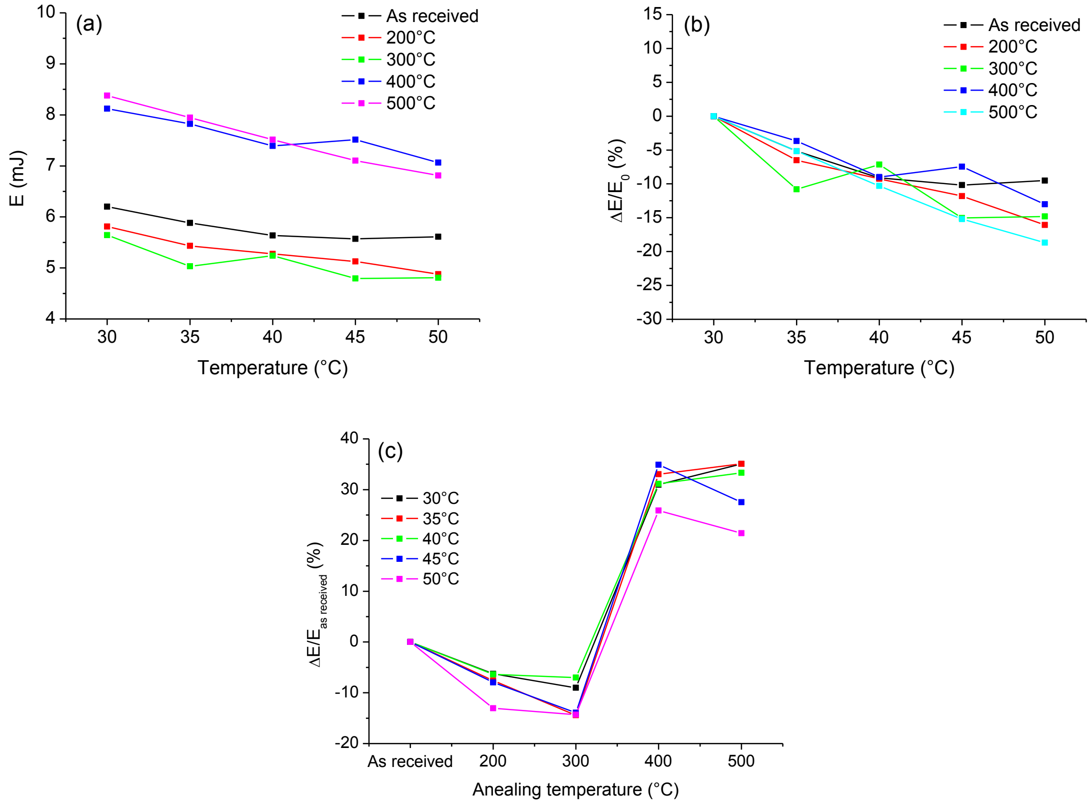

Figure 5. Energy dissipation capacity of M7 NiTi mini spring in function of test temperature and annealing temperature. (a) Absolute values. (b) Percentage variation related to the first value (at 30°C). (c) Percentage variation related to the E value of the *as received* sample.

From the obtained stiffness values and NiTi mini spring dimensions, the shear modulus (G, GPa) was calculated with Eq. (1). The results are shown in Tab. (1), along with the percentage deviation from the mean of a reference range (25 ~ 40 GPa) pointed by An et al (2012). Figure (6) illustrates the variation of G with annealing treatment. Is it seen that only the 500 °C annealing was effective to bring the G value to the reference mean value (32.5 GPa).

Table 1. Variation of shear modulus (G, GPa) of M7 NiTi mini spring sample for the different annealing temperatures.

Annealing temperature	**As received**	**200 °C**	**300 °C**	**400 °C**	**500 °C**
G (GPa, at 30 °C)	51.0	51.3	55.2	44.9	33.4
$\Delta G/G_{mean}$ (%)*	57	58	70	38	3

* Mean value pointed by literature (32.5 GPa).

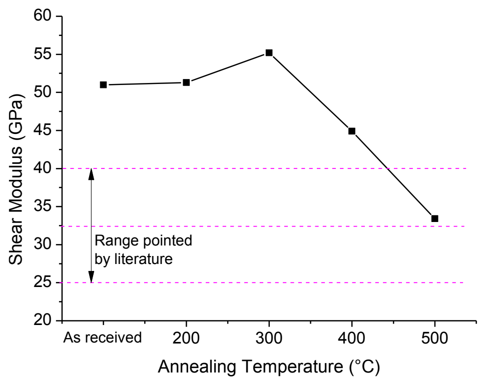

Figure 6. Shear modulus values (at 30°C) in function of annealing temperature.

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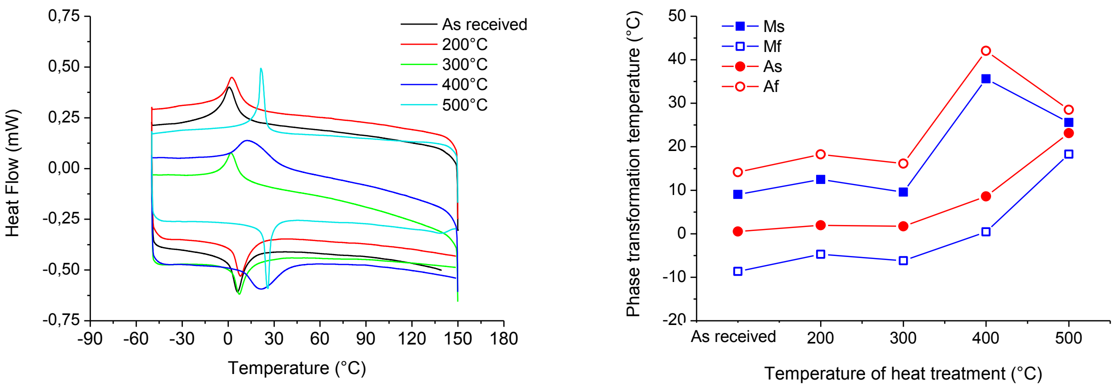
The phase transformation temperatures obtained from DSC results are shown in Table (2) in function of annealing temperature. The DSC curves and values from Tab. (2) are plotted in Fig. (7). Only after annealing at 300°C, the phase transformation temperatures changed, becoming higher. Thermal hysteresis also remained practically constant (difference between A_s and M_s or A_f and M_f). With annealing at 400°C, hysteresis achieved its highest level, decreasing with annealing at 500°C.

Table 2. Phase transformation temperatures obtained from DSC for different annealing temperatures.

	Phase Transformation Temperature			
Annealing temperature	**M_s**	**M_f**	**A_s**	**A_f**
As received	9.03	-8.66	0.51	14.15
200 °C	12.48	-4.71	1.95	18.24
300 °C	9.57	-6.2	1.72	16.14
400 °C	35.62	0.42	8.57	42.07
500 °C	25.58	18.31	23.12	28.49



(a) (b)

Figure 7. Thermal behavior of the NiTi SMA mini springs. (a) DSC curves for the spring sample treated at different annealing temperatures. (b) Phase transformation temperature (M_s, M_f, A_s and A_f) obtained from DSC curves.

4. CONCLUSIONS

This study allowed knowing more deeply the effects of annealing temperature over superelastic behavior of NiTi mini springs. Contrary to what was first assumed, the proportional increase in annealing temperature (from 200°C to 500°C) did not provoke a proportional variation in the thermomechanical behavior. More specifically, it was detected the existence of a minimum value of annealing temperature at which the stress relief is effective. Other important conclusions obtained in this study are summarized as follows:

- A proportional increase in force to achieve the same strain level was observed with increasing temperature, in accordance to a Clausius-Clepeyron law for shape memory alloys;
- Although it was not proportional, an increase in annealing temperature caused a decrease in the force to achieve the same strain level;
- Stiffness values for the austenitic phase were affected by heat treatment, decreasing mainly after 300°C. In this phase, only annealing treatments at 400 °C and 500 °C were capable of decreasing the stiffness;
- Energy dissipation capacity (E) behaves as opposed to stiffness and a practically linear decrease of E was observed when test temperature increases. Also, two levels of E were detected: up to 300°C annealing and other for 400°C and 500°C annealing;
- For the shear modulus, it was verified that only the 500 °C annealing was effective to bring the G value to the reference range value pointed by literature;
- For the phase transformation temperatures, only from 400°C annealing a change was detected. All temperatures became higher from this annealing temperature.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

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