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# ON THE FUNCTIONAL FATIGUE OF NITINOL SHAPE MEMORY WIRES IN SUPERELASTIC REGIME: AN EXPERIMENTAL INVESTIGATION

**Henrique Martinni Ramos de Oliveira**
**Allysson Daniel de Oliveira Ramos**
**Estephanie Nobre Dantas Grassi**
**Carlos José de Araújo**
**Wanderley Ferreira de Amorim Júnior**
Federal University of Campina Grande, Rua Aprígio Veloso, 882 , Campina Grande - PB, CEP: 58429-140, Brazil

***Abstract:*** *The use of NiTi shape memory alloys (SMA) working in superelastic regime cover various fields of applications such as medicine, dentistry and civil engineering, making it necessary a more specific knowledge of the behavior of these special materials when subjected to cyclic mechanical stresses. Therefore, experimental studies concerning fatigue behavior of these functional materials is of fundamental importance to support design of applications. The tests performed in this work have focused on evaluating the fatigue behavior of superelastic NiTi SMA (NiTiNOL) wires subjected to tensile loading controlled by mechanical stress, determining the evolution of some functional properties. The functional parameters evaluated were dissipated energy, transformation stresses, accumulated strains and superelastic strains, determined according to the intensity of mechanical tensile stress, which was varied between 500 MPa and 800MPa, at a frequency of 1 Hz. It was verified that the performance characteristics of the Nitinol wires depends directly of the tensile stress and number of cycles. For the first cycle, the dissipated energy ($E_D$), accumulated strain ($\varepsilon_p$) and superelastic strain ($\varepsilon_{SE}$), increases with increasing the peak stress. In general, increasing the number of cycles leads to more accumulation of $\varepsilon_p$ causing a reduction of $E_D$ and $\varepsilon_{SE}$ in a low cycle fatigue behavior.*

***Keywords:*** *Shape memory alloys, Nitinol wires, Superelasticity, Functional fatigue.*

## 1. INTRODUCTION

The shape memory alloys (SMA) are known as active or smart materials that have the ability to recover large strains (up to 10%), returning to its original shape by heating or after mechanical unloading. When retrieving a pseudo residual strain by heating, the phenomenon is called Shape Memory Effect (SME). In this case, a deformation imposed on the material in a low temperature crystallographic phase (martensite) and stiffness is fully recovered by a simple heating to a higher temperature and a greater stiffness (austenite) returning to its initial state. When the recovery of the imposed deformation occurs at a constant high temperature just by removing the applied force initially in the austenite phase, the phenomenon is called superelasticity (SE) (Otsuka and Wayman, 1998; Lagoudas, 2008). This SE phenomenon is also characterized by power dissipation in each cycle due to the irreversibility of the stress vs. strain behavior which causes hysteresis (Otsuka and Wayman, 1998).

The superelastic behavior of SMA occurs above a phase transformation temperature called autenitic finish temperature ($A_f$), where the entire crystallographic structure of the material is in the austenite phase. Applications of the SE phenomenon are present in several mechanical elements like superelastic screws (Travassos, 2010), Belleville washers (Pereira et al., 2011) and orthodontic devices like micro coil springs and arch wires, where the response of the releasing force during mechanical unloading occurs more consistently and smoothly than in other classic materials (Dos Reis, 2007). Besides applications in static regime, the SE is desirable in dynamic regimes due to dissipation of energy in each hysteretic stress vs. strain loop. In this context, numerous devices using superelastic SMA as vibration and impact absorbers have been developed, as reported recently by Aquino (2011).

In various application fields of SMA the fatigue behavior is a major factor for design and implementation of devices such as sensors and actuators based on the SE phenomenon. Especially in applications where work regime is dynamic, the study of fatigue life is imperative to ensure not only the structural functions of the material, but also the functionality of the active element under working conditions.

Functional fatigue in SMA can be understood as the number of cycles which the material exhibits degradation of its function, affecting the overall functionality of the system. An example would be a decrease in the recoverable strain associated with superelastic effect ($\varepsilon_{SE}$). In superelasticity, it is known that this type of fatigue is considered as the number of cycles below which it can be ensured that the functionality of the superelastic SMA is not compromised. It can be determined by the variation of $\varepsilon_{SE}$ and dissipated energy ($E_D$). The variation of $\varepsilon_{SE}$ and $E_D$ is observed as the mechanical hysteresis in the stress vs. strain behavior decreases as the number of cycles increases, stabilizing with the cycle evolution. Furthermore, the increase in accumulated strain ($\varepsilon_p$) and the variation in the phase transformation stresses are also characterized as an effect of functional fatigue in SMA, compromising its operation.

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The objective of this work is to make a contribution in order to better understand the behavior of NiTi superelastic wires when subjected to dynamic uniaxial tensile loads, in regard of the functional fatigue. The NiTi SMA wires were obtained from orthodontic arch wires with 0.50 mm in diameter, supplied by Dental Morelli (Brazil). Fatigue tests were performed using the electrodynamic Electropuls E10000 testing machine, from Instron, at maximum stress values of 500, 600, 700 and 800 MPa and oscillation frequency of 1 Hz. The first and last superelastic cycles, following a $2^n$ function, were compared to obtain values of functional fatigue life.

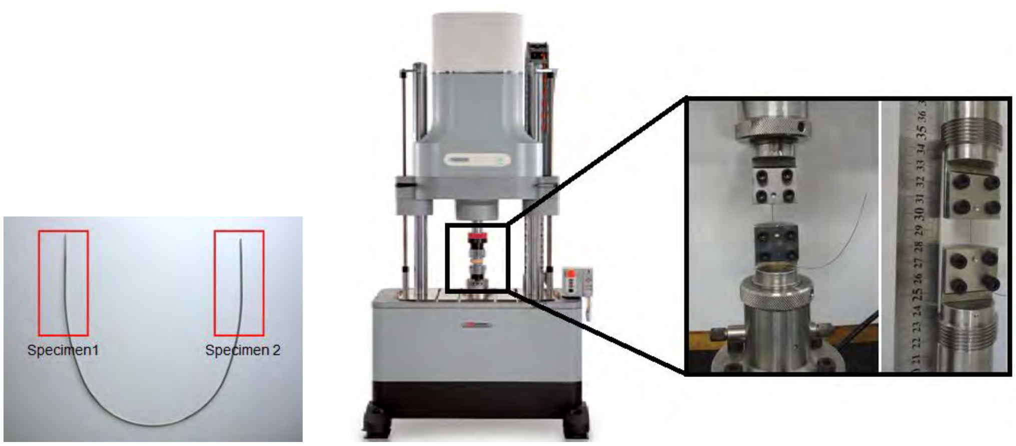
## 2. MATERIALS AND METHODS

For this study, NiTi superelastic SMA orthodontic wires with 0.50 mm in diameter, supplied by Dental Morelli Brazilian Company, were used.

To determine the mechanical parameters for dynamic tests, a tensile monotonic test was performed in a NiTi wire. From this first experiment it was determined the levels of mechanical stress to be used for the evaluation of functional fatigue. Four stresses levels were chosen: 500, 600, 700 and 800 MPa. Tests were carried out under stress control and frequency of 1 Hz. This range covers the three situations in which the SMA can be found: the austenitic crystallography phase present at higher temperature; the martensitic crystallography phase induced by stress at higher temperatures; and an intermediate state in which a mixture of these two phases is present, during stress-induced phase transformation. In addition, for each level of mechanical stress, four tests were performed to enable a minimum statistical analysis.

The cycling frequency was fixed at 1 Hz for each stress value. This frequency value was selected to allow the exchange of heat generated in the NiTi SMA wire due to forward and reverse phase transformations during cyclung, avoiding self-heating and subsequent rise of material's stiffness due to the accumulation of phase transformation latent heat (De Oliveira et al, 2012).

For the dynamic tests, it is showed in Fig. (1) the NiTi SMA wire specimens (Fig. 1a) that employed to cyclic axial loads in the electrodynamic testing machine Instron E10000 (Fig. 1b). From these dynamic tests it was possible to observe the evolution of functional fatigue parameters of the NiTi SMA wires.




(a) (b) (c)

**Figure 1**. NiTi SMA superelastic wire and testing machine. (a) NiTi superelastic orthodontic wire with 0.50 mm in diameter. (b) Electrodynamic testing machine (Instron E10000). (c) Detail of the NiTi arch wire assembled for testing.

Stress-strain curves for $2^n$ cycles (2nd cycle, 4th, 8th, 16th, 32nd, 64th, etc.) were plotted to allow the analysis of the functional fatigue of superelasticity phenomenon. The parameters studied to evaluate this type of fatigue were: the change in dissipated energy per unit volume (J/m$^3$), calculated using Eq. (1) which corresponds to the area of the stress vs. strain loop (Kang et al 2012); the accumulated strain ($\varepsilon_p$), resulting from the accumulation of internal stresses caused by the introduction of defects during mechanical cycling; and the superelastic strain ($\varepsilon_{SE}$), from the first to the last superelastic loop for each loading condition.

$$E_D = \oint \sigma d\varepsilon \tag{1}$$

Figure (2) shows a schematic stress vs. strain loop from which the functional parameters are evaluated as a function of number of cycles.

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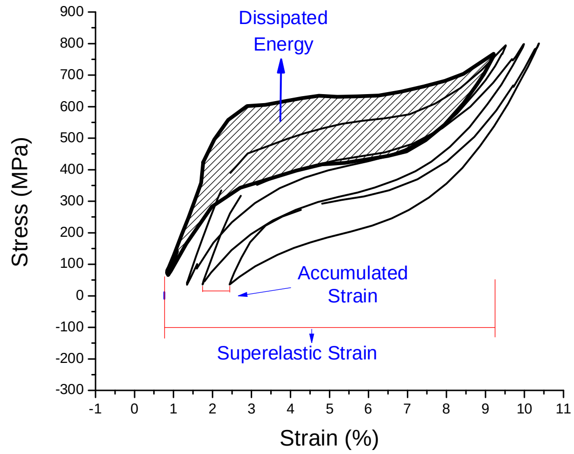


**Figure 2.** Schematic representation of the superelastic behavior for *n* fatigue cycles until failure.

In order to determine the variation of phase transformation stresses ($\sigma_{Ms}$, $\sigma_{Mf}$, $\sigma_{As}$ and $\sigma_{Af}$), the tangent method was used in the stress vs. strain loops. In this method these critical stresses are evaluated by drawing tangents to the start and final regions of stress vs. strain curves, as described in Fig. (3). The evolution of the stresses in each superelastic cycle for the various loading conditions were recorded and plotted for further analysis.

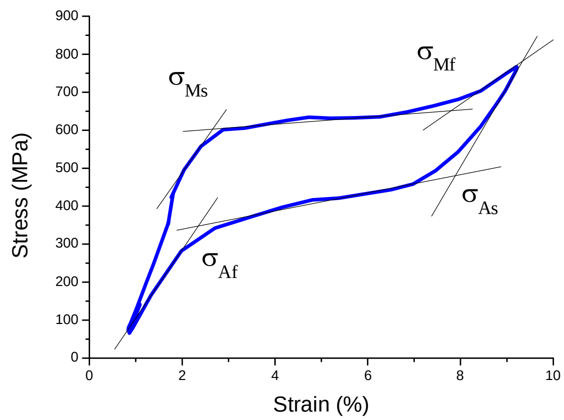


**Figure3**. Schema showing the tangent method used to define the phase transformation stresses in a stress vs. strain loop.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Pre-Fatigue Tests

#### Monotonic Tensile Tests

Figure (4) show the result of the quasi-static tensile test performed on the NiTi SMA wire. The resulting stress vs. strain curve can be divided in three regions:

• Region I [0, 500 MPa]: 100% austenitic microstructure with linear elastic strain (up to 2%). Releasing the applied load in this region the material will return to the point of zero strain in a linear stress vs. strain path;

• Region II [from 500 MPa to 600 MPa]: austenite starts to transform into martensite by the stress induced mechanical field. During the phase transformation, the material deforms approximately 6% with almost no increase in stress. By releasing the loading in this region, the material returns to the zero strain through a non-linear path. It is the phenomenon called superelasticity;

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• Region III [600 to ~ 1200 MPa]: 100% martensitic microstructure induced by the applied stress, after the complete phase transformation of the material. After 600 MPa stress-induced martensite has linear elastic strain up to about 1200 MPa. From this point the strain becomes plastic, until rupture.

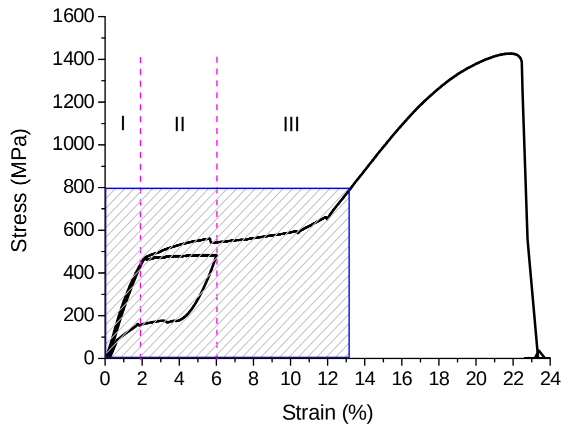


**Figure 4**. Stress vs. strain behavior for the monotonic tensile test in NiTi SMA wire used to study the functional fatigue.

From the monotonic experiment, four levels of mechanical stress were selected, up to the region of elastic strain of stress induced martensite (hatched area in Fig (4)) for the fatigue tests: 500 and 600 MPa for the case of partial phase transformation, and 700 and 800 MPa for the case of complete phase transformation.

### 3.2 Functional Fatigue

To examine the functional fatigue of the NiTi SMA wire, it was used only the results from specimens broken with the maximum number of cycles for each test condition. The data of the number of cycles to failure are summarized in Table (1).

**Tabela 1**. Number of cycles to failure the NiTi SMA wire for each fatigue test condition.

| **Frequency / Peak Stress** | **800 MPa** | **700 MPa** | **600 MPa** | **500 MPa** |
|---|---|---|---|---|
| ***1 Hz*** | *T1* - 9538<br>*T2* - 5496<br>*T3* - 6226<br>*T4* - 6036<br>***Mean – 6824***<br>***SD – 1589.65*** | *T1* - 8083<br>*T2* - 7634<br>*T3* - 7035<br>*T4* - 5607<br>***Mean – 7089***<br>***SD – 933.31*** | *T1* - 6442<br>*T2* - 8907<br>*T3* - 9271<br>*T4* - 5932<br>***Mean – 7638***<br>***SD – 1467.81*** | *T1* - 21592<br>*T2* - 11329<br>*T3* - 13313<br>*T4* - 13777<br>***Mean – 15002***<br>***SD – 3913.83*** |

Figure (5) show the evolution of the stress vs. strain behavior under superelastic loop until failure for each peak stress defined in Tab. (1). The superelastic loops are presented following the $2^n$ function for better visualization of the results, specially the first and last cycle before failure. These curves allow the evaluation of qualitative evolution of the superelastic loop as it accumulates residual strains with cycles.

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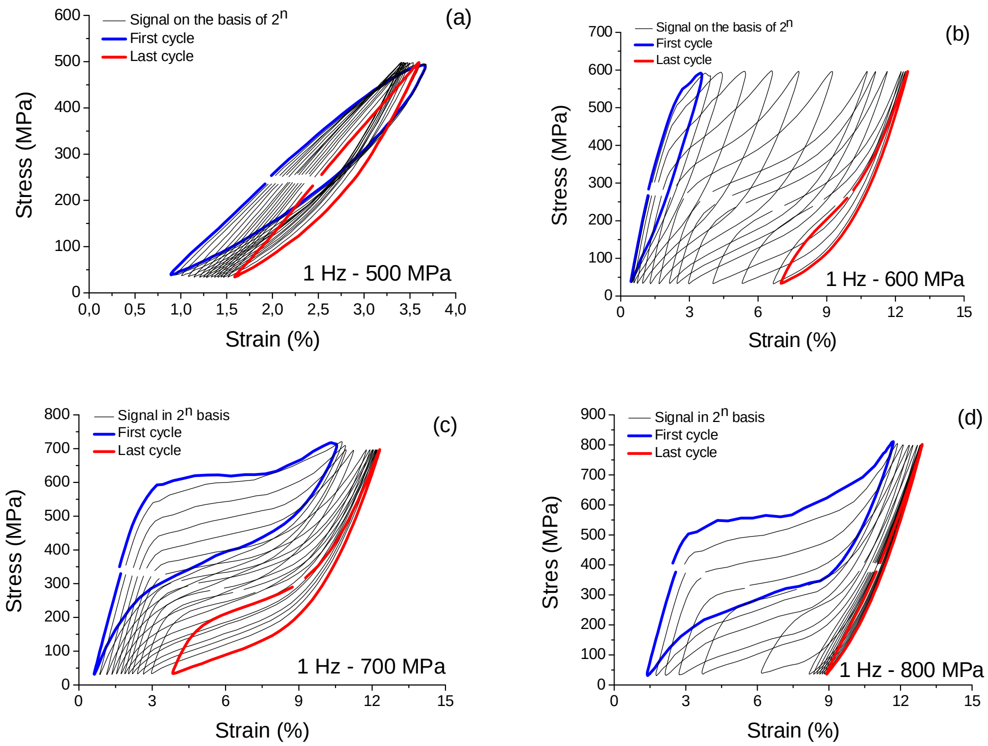


**Figure 5.** Evolution of the superelastic behavior of the studied NiTi SMA wire at 1 Hz under 500, 600, 700 and 800 MPa.

Figure (6) allows observing the influence of superelastic stress-controlled amplitude on the evolution of the dissipated energy for the studied test conditions.

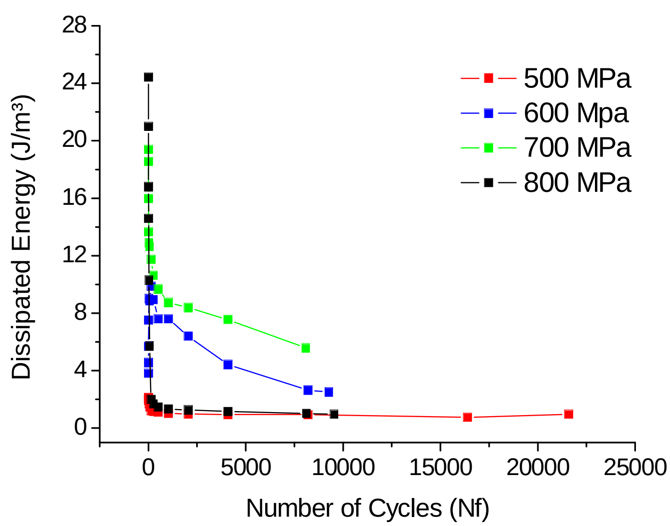


**Figure 6**. Dissipated energy by the NiTi SMA wire at 1Hz under 500, 600, 700 and 800 MPa.

It can be verified, for all test conditions, that up to approximately 300 cycles occurs an evolution of dissipated energy ($E_D$) that tend to stabilization after this initial phase at about 1.0 J/m$^3$ for moderate values of peak stresses (500

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and 600 MPa). For higher values of peak stresses (700 and 800 MPa), stabilization of $E_D$ is not clear, varying quasi linearly between 9.0 and 3.0 J/m$^3$.

For 500 MPa it was noticed a slight reduction on the amount of $E_D$, from 2.0 to 1.0 J/m$^3$. However, in general, the ability to dissipate energy was reduced because this is a region where the phase transformation has not yet occurred, or occurred in a punctual region of the NiTi SMA wire.

For 600 MPa it was verified an increase of $E_D$ for the initial cycles until stabilization. It was observed from the dynamic tests that this level of mechanical stress is insufficient to initiate the stress induced phase transformation (austenite to martensite). The hypothesis is that increasing number of cycles and consequent accumulation of plastic strain creates an internal stresses field which helps phase transformation leading the NiTi SMA wire attains the superelastic regime and increasing the capacity of energy dissipation per cycle, until stabilization.

For 700 and 800 MPa there was observed a large decrease of $E_D$ in the first few cycles before stabilization. At these stress levels the complete phase transformation occurs since the first cycle and therefore higher levels of power dissipation are achieved. The rapid decrease in the capacity to dissipate energy in the first few cycles is due to the phenomenon of the mechanical hysteresis stabilization. During this stabilization occurs the introduction of permanent defects in the microstructure, creating a state of residual internal stress, thereby facilitating the formation of martensite variants when the SMA is loaded (Lagoudas, 2008). Mechanically, this means that with cycling, will be required smaller values of energy to occur the phase transformation until a saturation/stabilization value is reached.

The amount of $E_D$ depends directly on the applied stress level, so the ability to dissipate energy is expected to increase proportionally as the applied mechanical stress field grows. The functionality of the studied superelastic SMA clearly degraded with the cyclic strain for the stress levels which provided, in the first cycles, a complete phase transformation.

Figure (7) shows the accumulated strain ($\varepsilon_p$) for the NiTi SMA wire until the rupture. The accumulation of $\varepsilon_p$ occurs because the cyclic tests are operated on stress control mode. This accumulation of permanent strain during consecutive cycles is responsible for the phenomenon of stabilization of the superelastic behavior and explains the behavior of the dissipated energy shown in Fig. (6).

It is known that the stabilized value of residual strain, as well as the evolution from the first cycle, is directly dependent on the applied stress. It means that the higher peak stress, the greater accumulated strain until stabilization, except for 700 MPa, that showed a lower cumulative strain compared to 600 MPa.

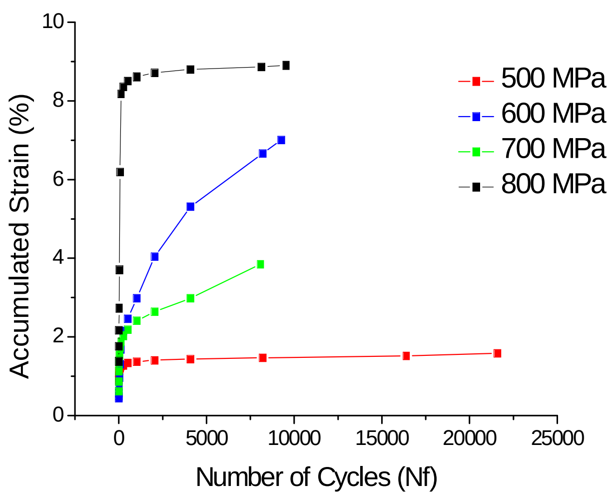


**Figure 7.** Accumulated permanent strain in the NiTi SMA wire during cycling under 500, 600, 700 and 800 MPa at 1Hz.

Figure (8) shows the evolution of the reversible superelastic strain ($\varepsilon_{SE}$) in the NiTi SMA wires subjected to cyclic tensile tests. The reduction of $\varepsilon_{SE}$ is related to a decrease in the superelastic loop, as shown in Fig. (2), caused by the introduction of defects in the internal structure of the NiTi SMA wires due to the several mechanical cycles realized.

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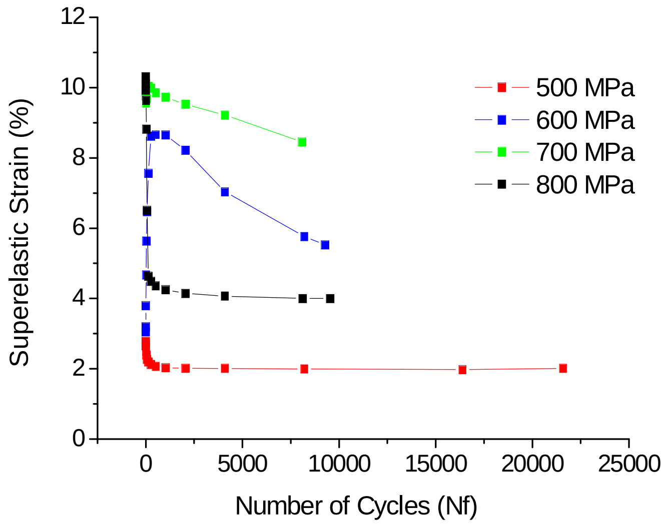


**Figure 8.** Reversible superelastic strain in the NiTi SMA wire for 500, 600, 700 and 800 MPa at 1Hz.

For 500 MPa, practically no evolution of this functional parameter was observed, with few changes in $\varepsilon_{SE}$ (3 % to 2 %). As previously mentioned, for this stress level phase transformation does not occur or occurs very punctually into the microstructure of the NiTi SMA wire.

For 600 MPa, an increase in $\varepsilon_{SE}$ was observed during the first cycles until stabilization (between 4 and 6 %). Similarly to the behavior verified for $E_D$, at this level of mechanical stress the phase transformation is induced, transforming the austenitic phase, high stiffness, for the martensitic phase, low stiffness and better condition to achieve greater strains. After this initial stage, a decrease followed by tendency of stabilization of the recoverable strain is observed with increasing number of cycles.

For higher stress levels (700 and 800 MPa) were noted more recoverable strains during initial cycles. However, such as cycling controlled stress levels introduces defects represented by an accumulation of more severe plastic strain, which further increases internal stresses, which, together with higher external stresses, always leads to a degradation of larger superelastic strain recovered at each cycle.

Figure (9) shows the variations in transformation stresses ($\sigma_T$) of NiTi SMA wires for cyclic tensile stresses of 700 and 800 MPa. It is noteworthy that only from these mechanical stress levels is that the NiTi SMA wires showed complete phase transformation, unlike the stress of 500 and 600 MPa, which showed only partial phase transformation (Figure 4). These changes in $\sigma_T$ also correspond to a degradation of the functionality of the NiTi SMA wires, come to contribute to a shorter life-span wire.

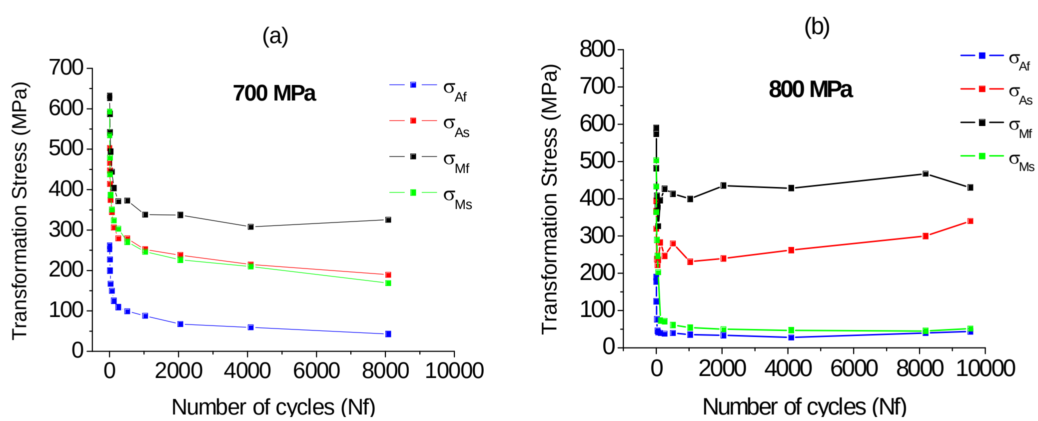


**Figure 9.** Evolution of the critical stresses during phase transformation of NiTi SMA wires. (a) For superelastic stress amplitude of 700 MPa. (b) For superelastic stress amplitude of 800 MPa.

In dynamic loading conditions, the thermomechanical behavior of SMA has specific characteristics. Overall, there was a significant reduction of critical stresses for 700 and 800 MPa. The stress to induce start formation of martensite, $\sigma_{Ms}$, for example, is reduced from 500 MPa to only 50 MPa in the case of peak stress of 800 MPa. All decrease of critical stresses shown in Fig. (9a) and (9b) can be attributed to the support provided by the phase transformation internal stress field created by defects associated with the accumulation of plastic strain in the austenitic phase.

In order to investigate the fatigue life of the superelastic NiTi SMA wire, a Wohler curve was plotted, as shown in Fig. (10), with number of cycles to failure as a function of applied stress. Each point in the Wohler curve, with its standard deviation values, represents the average of number of cycles up to failure for each loading condition (Table 1). The fatigue life in all tests was of the order of a few thousand cycles. This behavior is expected in the case of stress induced phase transformation in SMA and this type of fatigue, according to Lagoudas (2008), is known as "transformation-induced low cycle fatigue".

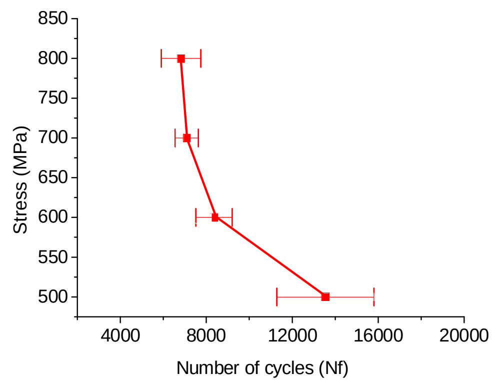


**Figure 10**. Wöhler curve for each stress level applied in the studied NiTi SMA wire at 1 Hz.

Figure (10) shows, on a first observation, that the NiTi SMA wire has a fatigue life of about 14,000 cycles for the lowest stress level (500 MPa). It is clear the direct influence of peak tensile stress so that the higher the stress, lower the fatigue life of the NiTi SMA wire (number of cycles up to failure).

## 4. CONCLUSIONS

In this work, the degradation of the functional properties of circular cross-section NiTi superelastic wires, subjected to different types of mechanical cyclic loading under stress control, more specifically for four peak tensile stresses (500, 600, 700 and 800 MPa) and a test frequency fixed at 1 Hz.

From the obtained results it was concluded that the fatigue life of the NiTi SMA wire under cyclic superelasticity is influenced by some parameters of dynamic loading. It was observed that both, the dissipated energy (ED) and the accumulated strain ($\varepsilon$p), reach a stabilization after approximately 300 cycles. At first it is observed a decrease in (ED) and increase in $\varepsilon$p and then a stabilization of each. The ED stabilized value, as well as $\varepsilon$p of the first cycle showed directly proportional to the maximum mechanical stress applied. It is also observed a significant reduction on the critical stress to induce phase transformation at 700 and 800 MPa. The Wohler curve proved that the fatigue life decreases with the increasing maximum mechanical stress applied in the NiTi SMA wires, degrading faster as the applied mechanical stress increase.

## 5. ACKNOWLEDGEMENTS

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