Study of superelastic fatigue in Ni-Ti alloy sensors/actuators with shape memory

Estudo da fadiga superelástica em sensores/atuadores de liga Ni-Ti com memória de forma

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ABSTRACT

The fatigue behavior of Ni-Ti springs with shape memory effect, applied in the activation of flow valves was studied. Employing an unconventional method, the structural and phenomenological aspects of the shape memory effect were analyzed. The Ni-Ti alloy was characterized by: Differential Scanning Calorimetry, Optical Microscopy, Scanning Electron Microscopy and Electron Dispersion Spectroscopy. Subsequently, for the analysis of structural and functional fatigue in shape memory, sensors/actuators in the form of helical springs were submitted to the superelastic fatigue test. The test routine created was performed interspersing slow and fast cycles, controlling temperature, stress and deflection variables. Thus, it was possible to investigate the evolution of critical temperatures, thermoelastic deformations and thermal hysteresis, in specific stages of thermomechanical cycling, to identify a possible degradation of structural and functional properties. It was possible to verify the effectiveness of the proposed methodology in characterizing the Ni-Ti sensor/actuator under the studied conditions.

Keywords: functional fatigue, structural fatigue, Ni-Ti alloys, shape memory effect.

1 INTRODUCTION

Shape Memory Alloys (SMA) make up a select group of metallic materials whose shape recovery abilities are attributed to thermoelastic martensitic transformations. Belonging to this group, Ni-Ti base alloys are the most used in technological applications, as they combine the properties associated with SMA, with good mechanical properties,
high ability to recover shape, greater pseudoplastic hysteresis, resistance to fatigue, corrosion and biocompatibility [5,8].

Several technical-scientific researches about developments and applications of these Ni-Ti alloys were carried out. However some issues are still discussed in the scientific community, especially when these materials are subjected to cyclic thermomechanical requests. According to De Araújo et al.[1] there are no specific technical standards for fatigue testing of these materials, subjected to thermal cycling under mechanical loading. There are large differences in fatigue life found in the literature [2], making the results discrepant and incomprehensible. Thus, the degree of reliability of sensor/actuator applications for Ni-Ti alloys can be increased by studying the complex phenomenon of fatigue that occurs in these materials. In this context, the main objective of this work was to contribute to the understanding of the phenomenon of structural and phenomenological fatigue of the shape memory effect in Ni-Ti actuators/sensors with a helical spring profile, intended for the actuation of flow valves.

In this study, in order to better understand complex phenomena in intelligent sensors/actuators, a fatigue test methodology was proposed, based on the performance of slow and fast cycles that allowed the collection of data during thermomechanical cycling. Initially, the material was characterized in terms of its thermoelastic and microstructural properties, through differential scanning calorimetry (DSC), optical microscopy (OM) and scanning electron microscopy (SEM), and EDS (Eletron Dispersion Spectroscopy). These characterizations contributed to analyze the thermoelastic properties likely to influence the fatigue life of these materials.

The proposed fatigue test method was developed to simultaneously evaluate the structural and functional fatigue (phenomenological of the shape memory effect), making it possible to estimate the lifetime of the intelligent sensor/actuator, through the analysis of parameters such as critical temperatures of transformation, thermal hysteresis and thermoelastic deformations, analyzing the degradation of the shape memory effect. The main objective was to investigate the reliability of Ni-Ti alloys for application in flow valves and to contribute to the development of methodologies for fatigue tests in sensors/actuators with shape memory.
2 EXPERIMENT PROCEDURES

2.1 MATERIAL

The Ni-Ti alloy wire acquired from the company Memory Metalle GmbH was selected for making the helical sensors/actuators. The manufacturer's specification is BSW (Body Cold Worked), referring to use at body temperature. The alloy was cold-worked and has a nickel-rich composition of 50.2 to 50.4 at%Ni and a diameter of 0.89 mm.

The procedure used to manufacture helical springs, with traction action, was carried out from the wire formed into a template, and is described in the literature [6-10-11-12]. The actuators were made with 6 helical coils, 4 of which were active, with an external diameter of 6.0 mm and a useful length of 6.0 mm. The thermal treatment used was homogenization for 24 hours at a temperature of 500 °C, followed by quenching in water at 25 °C.

2.2 DIFFERENTIAL SCANNING CALORIMETRY TESTS

The critical temperatures of the martensitic transformations were determined through the differential scanning calorimetry test, carried out in a DSC (Differential Scanning Calorimetry) from Mettler Toledo, with a temperature range of -60 to 100 °C, with a rate of 10 °C/min.

2.3 OPTICAL MICROSCOPY

Samples of heat-treated Ni-Ti BSW wire were prepared for optical microscopy at the Laboratory of the Universidad Autonoma de Nuevo Leon – Facultad de Ingenieria Mecanica y Eletrica (UANL-FIME) and were analyzed with a Nikon Epiphot 300 optical microscope. Of the work, the cross section of the wire was analyzed with a magnification of 100X.

2.4 ELECTRON SCANNING MICROSCOPY AND EDS

After performing the optical microscopy, the Scanning Electron Microscopy (SEM) was performed at the Laboratory of UANL-FIME, using a scanning electronic
microscope Nova NanoSEM 200 from FEITM, the same segments of Ni-Ti wires were analyzed, with magnification of 8000X. X-ray energy dispersion spectroscopy (EDS) was also performed, obtaining qualitative information about the chemical elements present in the samples, and in addition to allowing the identification of the presence of precipitates in the alloy.

2.5 FATIGUE TESTS

The fatigue tests carried out on the Ni-Ti helical springs reproduced the sensor/actuator operating conditions, favorable to mechanical and functional fatigue, when used in actuating a 3/4" normally closed flow valve [10-13]. In that study, a deflection of 10.0 mm was considered as the elongation that generates enough force to open the valve. The sensor/actuator activation temperature of 85°C, considering approximately \((A_F + 10)\)°C the temperature at which the actuator contracts, returning to its initial length, due to the shape memory effect, resulting in valve opening. These parameters were established based on the characterization of the selected Ni-Ti wire. The fatigue tests were divided into two stages: fast cycling and slow cycling.

2.5.1 Rapid Cycling

The objective of this step was to submit the sensor/actuator to a high number of thermomechanical cycles, simulating the conditions that facilitate failure due to mechanical and functional fatigue. For this, the fast cycles were performed in a fatigue testing machine developed at UFPE Intelligent Materials Laboratory - LIM-UFPE, subjecting the sensor/actuator to traction and compression cycles with a frequency of 100 cycles per minute, with a deflection of 10 mm, at a constant temperature of 85°C [6-13-14].

Figure 1 shows the main parts of the fatigue testing machine, with a total of 14 components that are associated to allow the test to be carried out [14]. The fatigue machine works coupled to a programmable thermal bath with volume filled with silicone oil, whose thermal control of the test is performed with the aid of thermocouples. This machine has operating dynamics based on a connecting rod-crank mechanism, and aims to
perform alternating stress cycles, capable of inducing martensite by stress (superelasticity)[13].

Figure 1- Details of the fatigue test machine [6]

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>External guide fixator</td>
</tr>
<tr>
<td>2</td>
<td>Transverse base</td>
</tr>
<tr>
<td>3</td>
<td>Connecting rod</td>
</tr>
<tr>
<td>4</td>
<td>Sliding bushing</td>
</tr>
<tr>
<td>5</td>
<td>Inner guide plate</td>
</tr>
<tr>
<td>6</td>
<td>Internal guide plate</td>
</tr>
<tr>
<td>7</td>
<td>Fixed blade support</td>
</tr>
<tr>
<td>8</td>
<td>Movable blade support</td>
</tr>
<tr>
<td>9</td>
<td>Crank</td>
</tr>
<tr>
<td>10</td>
<td>Mounting bracket</td>
</tr>
<tr>
<td>11</td>
<td>Piston</td>
</tr>
<tr>
<td>12</td>
<td>Fixing axle</td>
</tr>
<tr>
<td>13</td>
<td>Sliding axle</td>
</tr>
<tr>
<td>14</td>
<td>DC 12Va24V motor</td>
</tr>
</tbody>
</table>

Source: prepared by the authors

2.5.2 Slow Cycling

The objective of this step was to analyze changes in the thermoelastic properties of the actuator, resulting from rapid cycling. Data were mapped on the evolution of critical transformation temperatures (AS, AF, MS and MF), thermal hysteresis and thermoelastic deformations as a function of the number of cycles performed. Figure 2 shows the schematic of the system used to carry out the Slow Cycling. The constant load thermomechanical cycling device is composed of a programmable thermal bath and a structure...
that allows the application of an axial traction load on the sensor/actuator. This structure is formed by a support to which a pulley is attached, which works in association with a kanthal wire that is the link between the mass, placed to generate the shear stress, and the transmission rod, which is in direct contact with the actuator/sensor submerged in the silicone oil.

Figure 2 - Scheme of the device for thermomechanical cycling under constant traction load

In this system, an LVDT (Linear Variation Displacement Transducer) displacement sensor and thermocouples are connected to a computer through a National Instruments data acquisition system. This equipment is managed by an algorithm, developed in Labview, which allows obtaining data on temperature variation, thermal hysteresis, displacement and number of cycles executed in the spring during the tests.

To evaluate the thermoelastic properties, the temperature range was from 15 to 85°C, in which phase transformations occur. The test frequency was 1 cycle every 2
hours. The masses used varied between 270 and 1040g, as shown in Table 3. This mass is related to the axial force applied to the spring, which produces a shearing tension in the wire.

Table 3 - Relationship between shear stress in the wire, axial force and the mass subjected to the spring.

<table>
<thead>
<tr>
<th>Mass (g)</th>
<th>Axial Force(N)</th>
<th>Shear Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>270</td>
<td>2.7</td>
<td>70</td>
</tr>
<tr>
<td>520</td>
<td>5.1</td>
<td>135</td>
</tr>
<tr>
<td>700</td>
<td>6.9</td>
<td>200</td>
</tr>
<tr>
<td>1040</td>
<td>10.2</td>
<td>270</td>
</tr>
</tbody>
</table>

Source: prepared by the authors

To exemplify the information obtained in the slow cycle, figure 3 shows the curve obtained in this cycle when the sensor/actuator was subjected to a stress of 200MPa.

Figure 3 - Temperature X Displacement curve obtained in slow cycling at 200MPa.

2.5.3 Organization of Fast and Slow Cycles

The proposed methodology, in which slow cycling was performed alternately with fast cycling, was created from the observation of previous studies [6,10,15]. The test was divided into 4 stages. Figure 4 shows the flowchart of the organization of these steps.
In the flowchart it is possible to identify a pattern, which is repeated in all stages, composed of 2 slow cycles and “N” fast cycles, where “N” is $3951$ equivalent to $600$ and $1.2 \times 10^3 ; 10 \times 10^3 ; 50 \times 10^3 ;$ and $200 \times 10^3$, for steps 1, 2, 3 and 4, respectively. This pattern is accomplished continuously, until reaching the number of rapid cycles determined for each step. Stage 1 ends when $10 \times 10^3$ fast cycles are completed, starting stage 2 which ends with $50 \times 10^3$ fast cycles, then stage 3 starts, and ends when reaching $200 \times 10^3$ fast cycles, stage 4 ends the fatigue test with a total of $1200 \times 10^3$ fast cycles, at which point the sensor/actuator is assumed to have reached infinite life.

3 RESULTS AND DISCUSSIONS
3.1 DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Figure 5 shows the calorimetric curve of the sample with treatment at $500^\circ$C for 24 hours, with transformation in only one step ($A \longrightarrow M$), without the presence of R phase, thermal hysteresis ($H_t$) around $15^\circ$C and transformation enthalpies of $25$ and $26^\circ$C.
Figure 5 – DSC curve of the sample treated at 500°C after 24h.

3.2 OPTICAL MICROSCOPY

Figure 6 shows the micrograph get by optical microscopy (OM) of the cross section of the heat-treated Ni-Ti BSW wire, with a magnification of 100X. The main morphological observation is the presence of martensitic and austenitic phases, the first being identified by structures in the form of needles without preferential orientation, and the second called retained austenite. Both phases are present at room temperature. This observation is in line with the literature, which describes that in a transformation induced by temperature, twinned martensite appears, which is the result of the combination of twenty-four variants, with different orientations from crystallographic platelets [16,17].

The presence of retained austenite suggests the possibility that the martensitic transformation is not complete, with two thermoelastic phases coexisting. The energy required to complete the transformation and the velocity of thermal cooling may have influenced the emergence of needles that need to reach a temperature even lower than the 25°C used in the analysis.
3.3 ELECTRON MICROSCOPY AND EDS

Figure 7 presents the SEM and EDS results on the same optical microscopy sample. In the micrograph it is possible to visualize the microstructure with a magnification of 8000x, showing the presence of martensite platelets and a precipitate. The chemical composition of the precipitate was analyzed by the spectrum generated in the EDS.

A possible explanation for the emergence of these titanium-rich precipitates, with 96.39 at% Ti, could be due to the annealing of the Ni-Ti alloy. This thermal treatment can induce the formation of phases such as TiNi$_3$, Ti$_3$Ni$_4$, Ti$_2$Ni$_3$ that consumes the matrix phase in Ni, generating changes in the mechanical behavior of the material [21]. The
presence of these precipitates can influence the transformation temperatures and consequent fatigue life of the material. Studies performed by Nishida et al. [18] and by Miyazaki et al. [19] showed that the formation of precipitates in these alloys is influenced by diffusional decomposition processes that involve changes in the chemical composition of the material and can influence the behavior of transformation temperatures.

3.4 FATIGUE TEST

For the analysis of functional fatigue of the sensor/actuator, the evolution of critical transformation temperatures, thermal hysteresis and thermoelastic deformations with thermomechanical cycling were analyzed.

3.4.1 Critical Transformation Temperatures

Figures 8a, 8b, 8c and 8d show the behavior of the critical transformation temperatures as a function of the number of cycles, for loads of 70, 135, 200 and 270 MPa, respectively. Regardless of the load used, these temperatures have a linear evolution and, in most cases, small variations.
Figure 8 - Evolution of transformation temperatures of Ni-Ti actuators in fatigue tests –

a) 70MPa  b) 135MPa  c) 200MPa  d) 270MPa.

Source: prepared by the authors

Figure 9 shows the critical temperatures as a function of the applied stress, in the 1st and last thermomechanical cycle (1.2*10^6 cycles). Comparing figures 9.a and 9.b, it can be seen that after carrying out 1.2*10^6 cycles, the transformation temperatures show a continuous behavior due to the thermomechanical training, which significantly influenced the evolution of these temperatures in a preferred direction.

Until 200MPa, there is a tendency for Mₛ, Mₐ and A₝ temperatures to increase. This tendency can be attributed to thermomechanical training and also the application of higher training stress [22,23]. This behavior is due to the process of reconfiguration of the stress fields generated by the dislocations present in the wire. Another factor that may contribute is the presence of precipitates, as identified in the micrograph by SEM (figure 7).
These precipitates facilitate martensitic transformation, as they act as preferential regions for nucleation reactions; thus the transformation requires less external energy (cooling), resulting in an increase in the $M_s$ temperature [20]. The results of the $A_s$ temperatures indicate that the stress of greater intensity present ease in the reorientation of the martensite needles of the actuator, allowing the element to need less temperature input to start the inverse transformation [10]. The decrease in $A_s$ occurs simultaneously with the increase in $M_s$. So, it demonstrates the effectiveness of thermomechanical training. These results imply that sensor/actuator operation requires less energy to perform the phase transformation, less thermal hysteresis, and faster and more efficient activation responses.

For the 270MPa load, the $A_s$ starts to behave in the opposite way, indicating that some phenomenon interfered in the efficiency of the actuator operation. The phenomenon of martensite stabilization has similar particularities, generally associated with increases in inverse transformation temperatures ($A_s$ and $A_f$)[28]. It is important to observe that when thermomechanical cycles are repeated in SMA, dislocations accumulate and the formation of loop-shaped dislocations occurs, which favor the formation of martensite nucleation points [30]. If these points increase in quantity, a portion of retained martensite can be obtained, which will only disappear after a new heat treatment [25]. The presence of this martensite decreases the EMF, and may even eliminate it, causing functional fatigue [29]. Therefore, for a stress of 270MPa, changes in $A_f$ and $A_s$ may be an indication
of functional fatigue. However, for the other loads in the fatigue tests performed, the actuators/sensors showed infinite life from a structural and functional point of view.

3.4.2 Thermal Hysteresis

Figure 11 shows the thermal hysteresis ($H_t$) data over the $1.2 \times 10^6$ cycles performed.

The result of the DSC for this alloy indicated a value of $15^\circ C$ of thermal hysteresis, however, when tension are added, it is observed that since the first cycle there was a significant increase in the respective values, however this increase gradually decreased with the increase of the load, for $270MPa$ the values are below $30^\circ C$. The application of loads increases thermal hysteresis, as shown in table 4.

Table 4 - Results of Thermal Hysteresis in the Fatigue Test at 70, 135, 200 and 270MPa in the 1st and last cycle.

<table>
<thead>
<tr>
<th>Nº of cycles</th>
<th>70MPa</th>
<th>135MPa</th>
<th>200MPa</th>
<th>270MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>37.9</td>
<td>37.6</td>
<td>30.1</td>
<td>29.1</td>
</tr>
<tr>
<td>$1.2\times10^6$</td>
<td>27.3</td>
<td>28.6</td>
<td>22.3</td>
<td>28.1</td>
</tr>
</tbody>
</table>

Source: prepared by the authors
Figure 11b shows that the thermal hysteresis decreases, with cycling for all loads facilitating the EMF, since the driving forces required for the transformations decrease [26]. For stress of 70 and 135MPa, this reduction was more expressive, around 10°C. The stress of 200MPa was the one that presented the lowest hysteresis values, behavior predicted when analyzing the relation between the applied stress and the phase transformation temperatures.

For the stress of 270MPa, the thermal hysteresis was little affected, showing a slight reduction of about 1°C. For this stress, after $1.2 \times 10^6$ cycles performed, the sensor/actuator had the lowest performance capacity, obtaining the highest thermal hysteresis among the selected loads. This thermoelastic property for the 270MPa load was degraded during cycling, due to martensite stabilization processes. In this way, the sensor/actuator presented functional fatigue for this load.

4 CONCLUSIONS

The results of the evolution of critical temperatures, thermoelastic deformation and thermal hysteresis, obtained in fatigue tests on Ni-Ti spring with shape memory, provide evidence that the proposed test methodology is capable of mapping the general thermomechanical behavior related to fatigue structural and functional of the sensor/actuator.

The results of the DSC, MO, SEM and EDS tests provided an analysis of the phenomena that may be involved in the fatigue process in this material. The evolution of critical temperatures allowed identifying favorable points for the actuator operation, such as for the stress of 200MPa, in which the approximation of the $A_S$ and $M_S$ peaks was identified, which is related to the reduction of thermal hysteresis and faster sensor/actuator responses. For the stress of 270MPa, in which an increase in $A_S$ and $A_F$ was verified, a phenomenon that is associated with an increase in thermal hysteresis.

These factors are related to the stabilization of the martensite that promotes degradation of the EFM. Loss of these functional properties are a strong indicator of the presence of functional fatigue in the sensor/actuator.
Thus, the results obtained in this work converged to the excellent resistance to fatigue of the Ni-Ti BSW alloy in the form of a spring, subjected to thermomechanical cycles under the conditions suggested for analysis.
REFERÊNCIAS


