ELECTRICAL RESISTANCE MEASUREMENTS IN CFRP COMPOSITES WITH NiTi SHAPE MEMORY ALLOY WIRES

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Abstract. *The possibility of uniting two or more different materials to obtain structures capable to feel and adapt to environment alterations and operational conditions, has been leading to the development of active composites with functional properties that makes possible the control of form, vibration, rigidity and/or structural integrity monitoring. These characteristics are very well accepted in modern technological applications. In this context, active composites were manufactured using pre-impregnated (Pre-Pregs) of epoxy with carbon fibers (CFRP) and NiTi shape memory alloy (SMA) thin wires embedded in different conditions, besides copper electrodes for electrical resistance monitoring of the specimens. The NiTi SMA wires, the carbon fibers and the obtained CFRP-NiTi composites were characterized by electro-thermomechanical tests of electrical resistance as a function of temperature and deformation, thus verifying the efficiency of embedded electrodes in monitoring the structure when submitted to three points bending cycles.*

***Keywords**: Active Composites, NiTi Alloys, Electrical Resistance, Smart materials and structures.*

1. INTRODUCTION

Smart composites are structures capable to detect changes in the operational and/or environmental conditions and promote adaptations through activation of actuators, seeking to maintain a satisfactory behavior previously established (Tebaldi *et al.,* 2006). This process is led through the integration of three basic elements: (i) sensor that register internal and external informations, (ii) actuators that accomplish work or apply forces and (iii) central control systems that make decisions and send orders. Thus, these systems are structures that contains inherent potentialities of acting, detecting and controlling, through the combination of several active materials (Michaud, 2003), looking for properties as precision, effectiveness, functionality, durability and adaptability.

Among the active materials, the Shape Memory Alloy (SMA) stand out for your thermomecanical properties of force generation and/or deformation as a function of temperature besides the capacity to feel and to act. In the development of active structures, SMA are usually used in the form of thin wires, which are considered as linear actuators that allow direct integration in composites reinforced with fiber in polymeric matrix, without losing the structural integrity of the material (Jang and Kishi, 2005). The one-way or the two-way shape memory phenomena can generate intense recovery forces through the temperature change when SMA thin wires are integrated into composite matrix (Otsuka and Wayman, 1998).

The Carbon Fiber Reinforced Polymers (CFRP) present very appropriate properties for developing smart composites. Beyond presenting a high mechanical resistance associated with a low specific mass, these composites have the capacity to conduct electrical current, making possible its structural monitoring (Chung, 2001). Structural health monitoring refers to the monitoring of the structure integrity for the purpose of hazard mitigation. This monitoring concerns mainly the non-destructive sensing of the damage in the structure. In this context, the electrical resistance method is particularly effective for detecting small and subtle defects in CFRP composite materials (Chung, 2001). One of the forms that have been employed for this end is the inclusion of electrodes in the CFRP through punctures, what can damage the structure originating points of stress concentration (Baere *et al.,* 2007). An alternative proposal to attenuate this problem consists on embedding copper electrodes as a composite constituent.

The present work proposes to manufacture CFRP active composites with embedded NiTi SMA wires in different conditions (as-received, annealed and trained after annealed) and copper electrodes embedded perpendicularly of the carbon fibers direction in order to monitoring the composite structure properties when submitted to three points bending tests at different temperatures.

2. EXPERIMENTAL PROCEDURE

2.1. Preparation of Ni-Ti SMA wires

Before fabrication the active CFRP-SMA composite beam specimens, the NiTi SMA wires were stabilized by training using a thermal cycling under constant load procedure. Firstly, the as-received NiTi wire was annealed at 400ºC for 900s. Two meters of the heat-treated NiTi wire under a dead weight corresponding to a tensile stress of 200MPa was submitted to 1000 heating and cooling cycles (contraction and expansion) using electrical resistive heating (De Araujo *et al.,* 2008).

2.2. Design of the CFRP-NiTi active composites

The CFRP-NiTi composites developed in this study consists of four layers of unidirectional carbon fiber pre-pregs tape manufactured by Hexcel Composites (UK) containing five NiTi SMA wires evenly distributed along the neutral plane of the designed beam. The Hex Ply 8552 pre-preg consist of an epoxy matrix of high resistance and carbon fibers. The NiTi wires can be activated by resistive heating while copper electrodes distributed perpendicular to the fibers direction (carbon and NiTi) are used to monitor electrical resistance changes in the structure, as shown in the Figure 1.

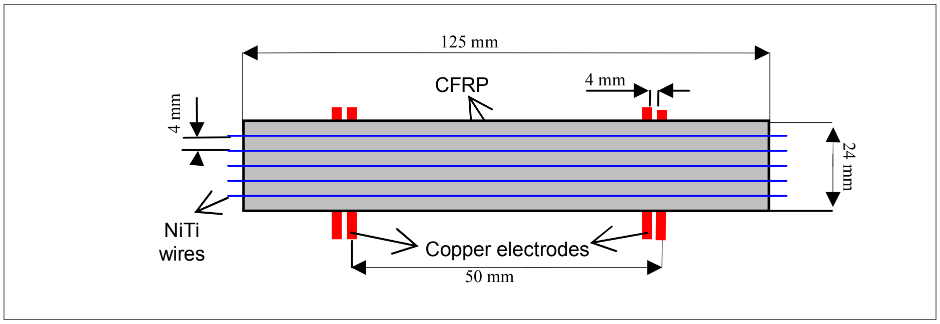

Figure 1. Schematic drawing of the CFRP-NiTi active composite

2.3. Electro-themomechanical characterization of the Ni-Ti SMA wire, carbon fibers and CFRP

For the characterization of NiTi SMA, carbon fibers and CFRP-NiTi composites, electrical resistance change ($\Delta R/R$) was measured using the four-probe method while cyclic temperature or cyclic stress was applied. A DC programmable power supply was used for input electrical current into the CFRP specimens through the NiTi wires. The copper electrodes were connected to a data logger (Agilent Technologies) interfaced to a PC.

The thermal characterization of the NiTi wires and CFRP-NiTi composites was carried out with the immersion in a silicone oil bath apparatus previously designed for this task (Reis *et al.*, 2006). The temperature (T) was varied from 120ºC to -10ºC. For the characterization of carbon fibers, single fibers obtained by dissolving away the epoxy polymer from the CFRP prepreg were subjected to similar electrothermal testing applied for the NiTi wires and CFRP samples.

After pure thermal characterization, cyclic three point bending tests were performed in an INSTRON 5582 universal testing machine. For these tests it was used a displacement rate of 2mm/min, coming to a maximum displacement of 3mm and coming back for an intermediate position of 1mm from the zero point, oscillating between 1mm and 3mm. Twenty cycles were realized, as indicated by the schema of Figure 2.

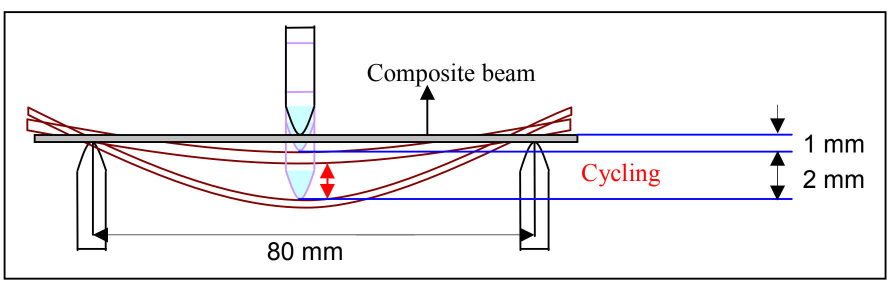

Figure 2. Schematic drawing of three points bending cycles in CFRP-NiTi active composites

3. RESULTS AND DISCUSSIONS

3.1 Preparation of the CFRP-NiTi active composites

Figure 3 shows the four CFRP-NiTi beams manufactured with the NiTi wires in different states: one without the NiTi wires and the other three ones with embedded wires in three different conditions (as-received, annealed and trained after annealed). The obtained specimens with dimensions of about 125mm x 24mm x 0,78mm are showed in Figure 3(a). Figure 3(b) shows the curves for the temperature and applied pressure as a function of time during the molding process of the CFRP-NiTi composites. These results indicate a good control of the cure process under constant load.

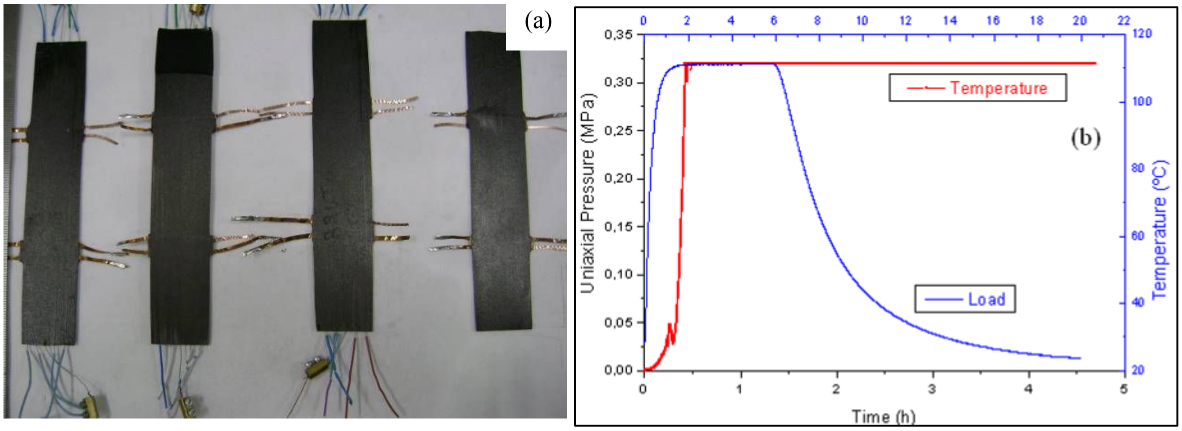

Figure 3. CFRP-NiTi active composites (a) and cure cycle (b)

3.2 Electrothermal characterization of the NiTi wires and CFRP composites

Figure 4 shows the ΔR/R – T curves for the NiTi wires in the as-received (a), annealed (b) and trained after annealed (c) states. The inexistence of phase transformation in the as-received NiTi wire is clearly observed in Figure 4(a), that presents a classic linear behavior of electrical resistance in function of temperature during cooling and heating. Figures 4(b and c) reveal the presence of the reversible martensitic transformation for the annealed and trained NiTi wires with phase transformation temperatures determined by the tangent method. The annealed and trained wires transforms in two-step during cooling, from austenite to the R-phase and then to martensite, as is well known from literature (Otsuka and Wayman, 1998). The reverse transformation by heating occurs in a single step. Table 1 summarizes the phase transformation temperatures for the annealed and trained wires.

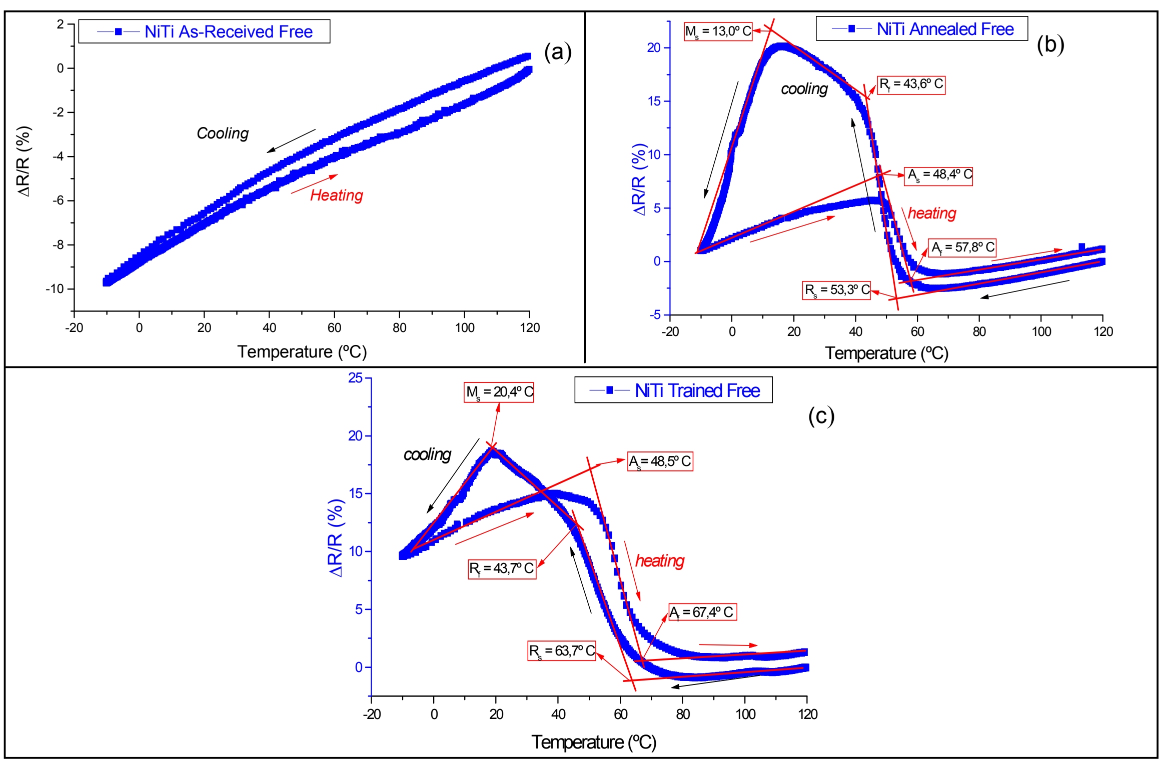

Figure 4. Electrical resistance change in function of the temperature for the NiTi wires: as-received (a), annealed (b) and trained (c)

Table 1. Transformation temperatures of the NiTi SMA wires

NiTi Wire	***R_S (ºC)***	***R_f (ºC)***	***M_S (ºC)***	***A_S (ºC)***	***A_f (ºC)***
Annealed	53,3	43,6	13,0	48,4	57,8
Trained	63,7	43,7	20,4	48,5	67,4

Figure 5 shows the behavior of the electrical resistance for CFRP and pure carbon fibers (without epoxy resin). Was observed that for CFRP without the NiTi wires (Figure 5a), an inverse linear variation of about 7 % with hysteresis exists in electrical resistance. Similar behavior was observed for the pure carbon fibers (Figure 5b) where electrical resistance present a variation of about 5,6% after the thermal cycle. Asanuma *et al.* (2006) has identified similar results in high performance CFRP/metal active laminates.

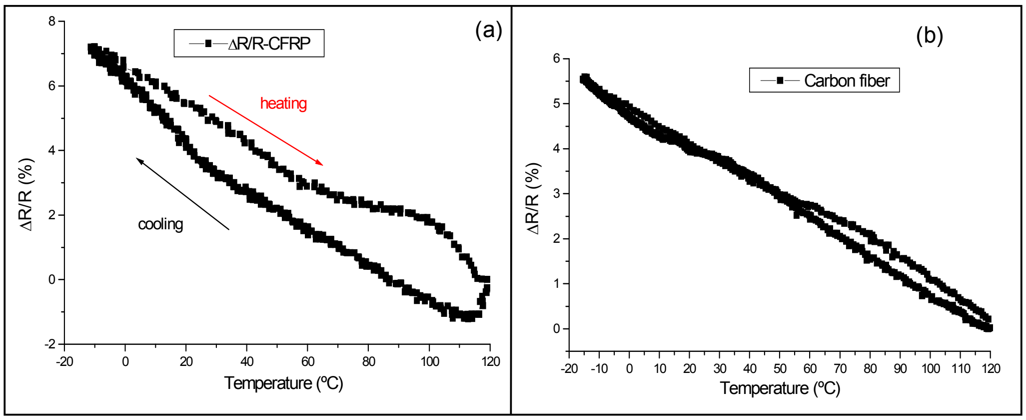

Figure 5. Electrical resistance change in function of temperature for the CFRP (a) and pure carbon fibers (b)

Figure 6 show the behavior of the electrical resistance in function of temperature for the CFRP-NiTi active composites. It was verified that for the composites with annealed NiTi wires (Figure 6b) and trained (Figure 6c), the electrical resistance variation with the temperature accompanies the behavior of the NiTi wires, presenting approximately the same critical phase transformation temperatures of them.

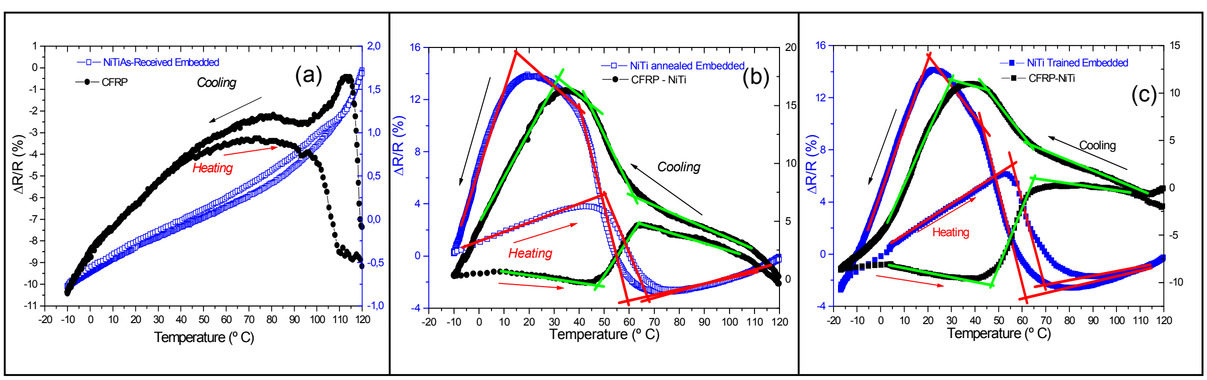

Figure 6. Electrical resistance change in function of temperature for the CFRP composites with NiTi wires in the following conditions: as-received (a) annealed (b) and trained (c)

Tables 2 and 3 summarize the values of the phase transformation temperatures for annealed and trained wires, free and embedded in CFRP, respectively. It was observed a displacement of the peaks of transformation during cooling. This increase in transformation temperatures is probably related to the presence of residual internal stresses from the manufacturing process. It is well established that when a SMA is submitted to a thermal cycle under an external load, the transformation temperatures increases (dislocate in right direction), in agreement with a Clausius-Clayperon law (Otsuka and Wayman, 1998).

Table 2. Transformation temperatures of the annealed NiTi wires embedded in CFRP and free

Annealed NiTi wires	***R_s (ºC)***	***R_f (ºC)***	***M_s (ºC)***	***As (ºC)***	***A_f (ºC)***
Embedded	56,7	41,0	14,6	49,0	66,7
Free	53,3	43,6	13,0	48,4	57,8

Table 3. Transformation temperatures of the trained NiTi wires embedded in CFRP and free

NiTi wires trained	***R_s (ºC)***	***R_f (ºC)***	***M_s (ºC)***	***A_s (ºC)***	***A_f (ºC)***
Embedded	62,8	43,3	21,3	53,6	75,7
Free	63,7	43,7	20,4	48,5	67,4

3.3 Electro-themomechanical characterization of the CFRP-NiTi composites

During these tests the electrical resistance variation (ΔR/R) of the inferior and superior layers of the CFRP-NiTi smart composites as a function of the imposed displacement by the test machine was monitored (Figure 2). Figures 7, 8 and 9 shows the behavior of ΔR/R in the composites with the as-received (Figure 7), annealed (Figure 8) and trained (Figure 9) NiTi wires, when submitted to three-point bending cycles in three different temperatures. The CFRP-NiTi specimens were tested in three different states: at 30ºC (R-phase), 60ºC (R-phase + austenite) and 90ºC (austenite). It was verified in all cases that there is a qualitative relationship between ΔR/R and imposed displacement of the CFRP-NiTi composites along the cycling.

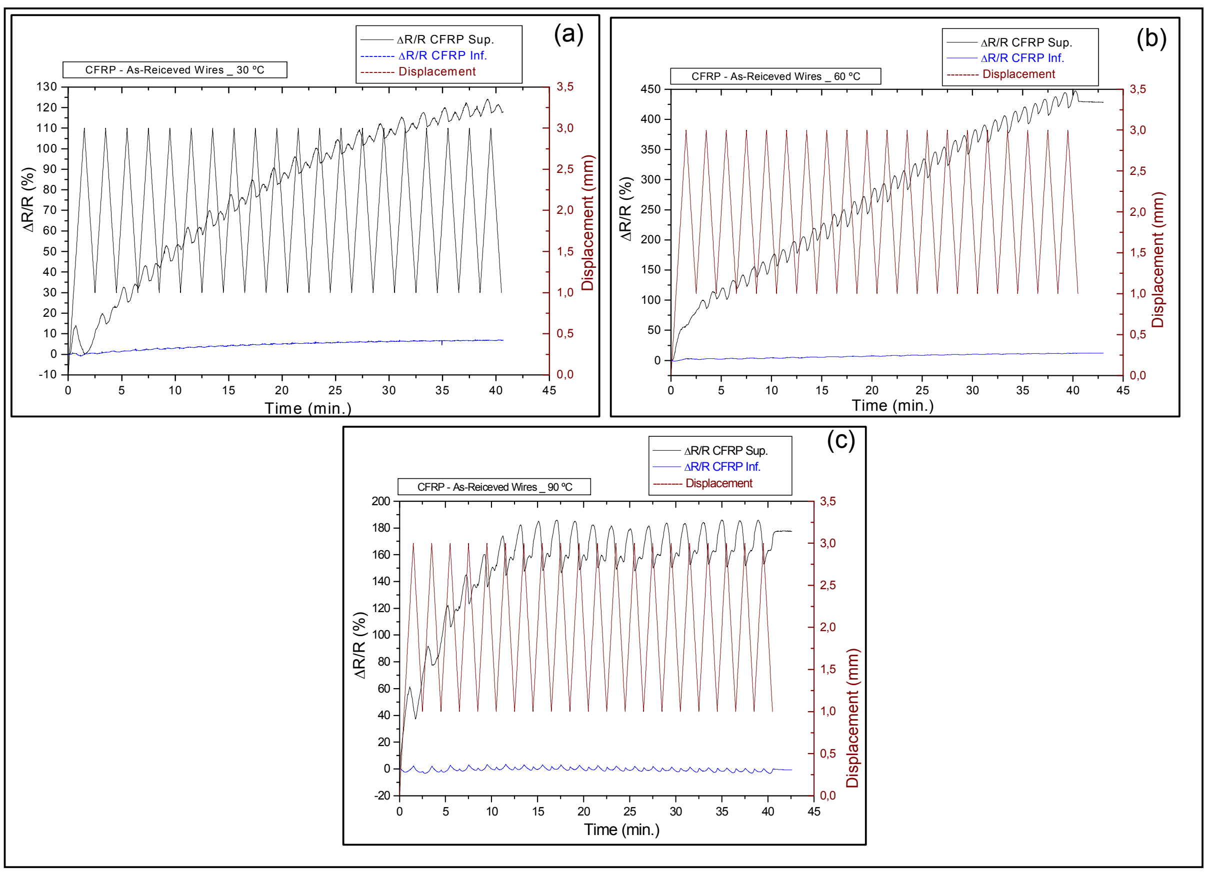

Figure 7. Behavior of ΔR/R for the superior and inferior layers and displacement in function of the time for the composites with as-received NiTi wires: 30 ºC (a), 60 ºC (b) and 90 ºC (c)

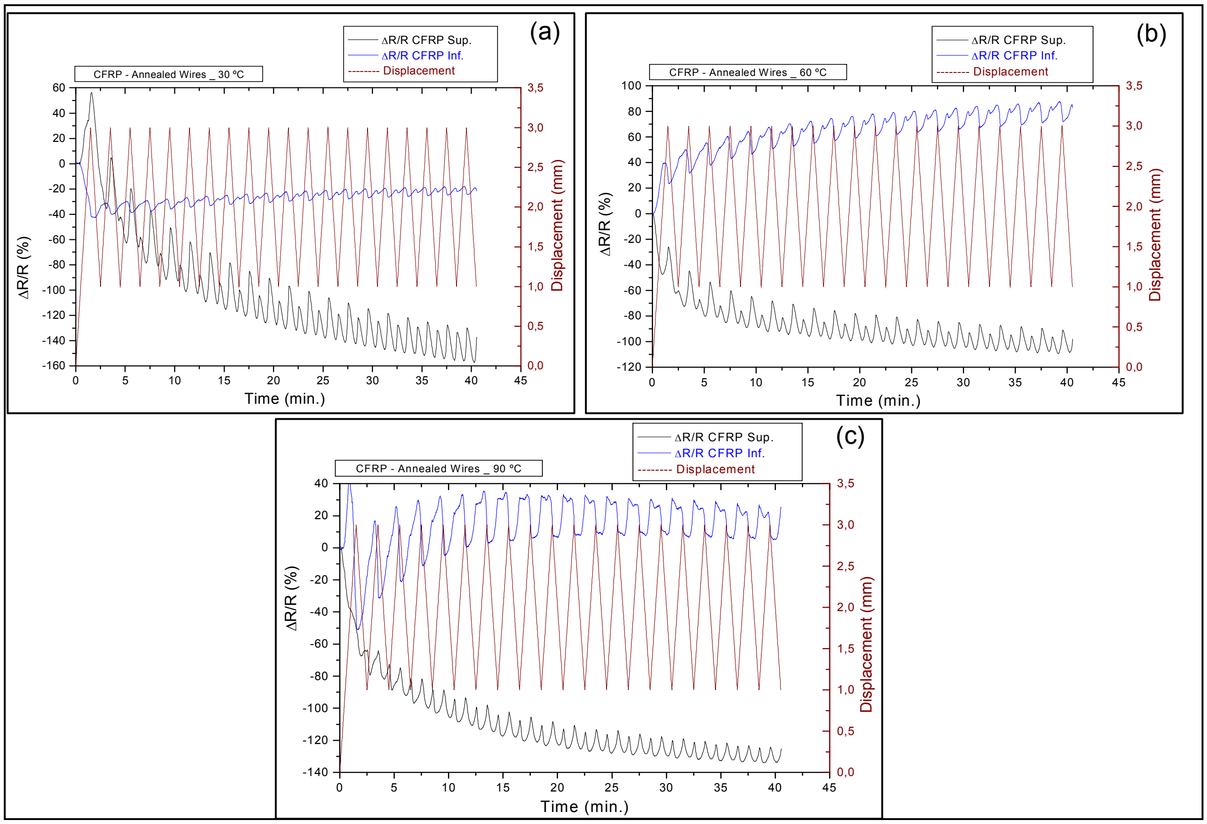

Figure 8. Behavior of ΔR/R for the superior and inferior layers and displacement in function of the time for the composites with annealed NiTi wires: 30 ºC (a), 60 ºC (b) and 90 ºC (c)

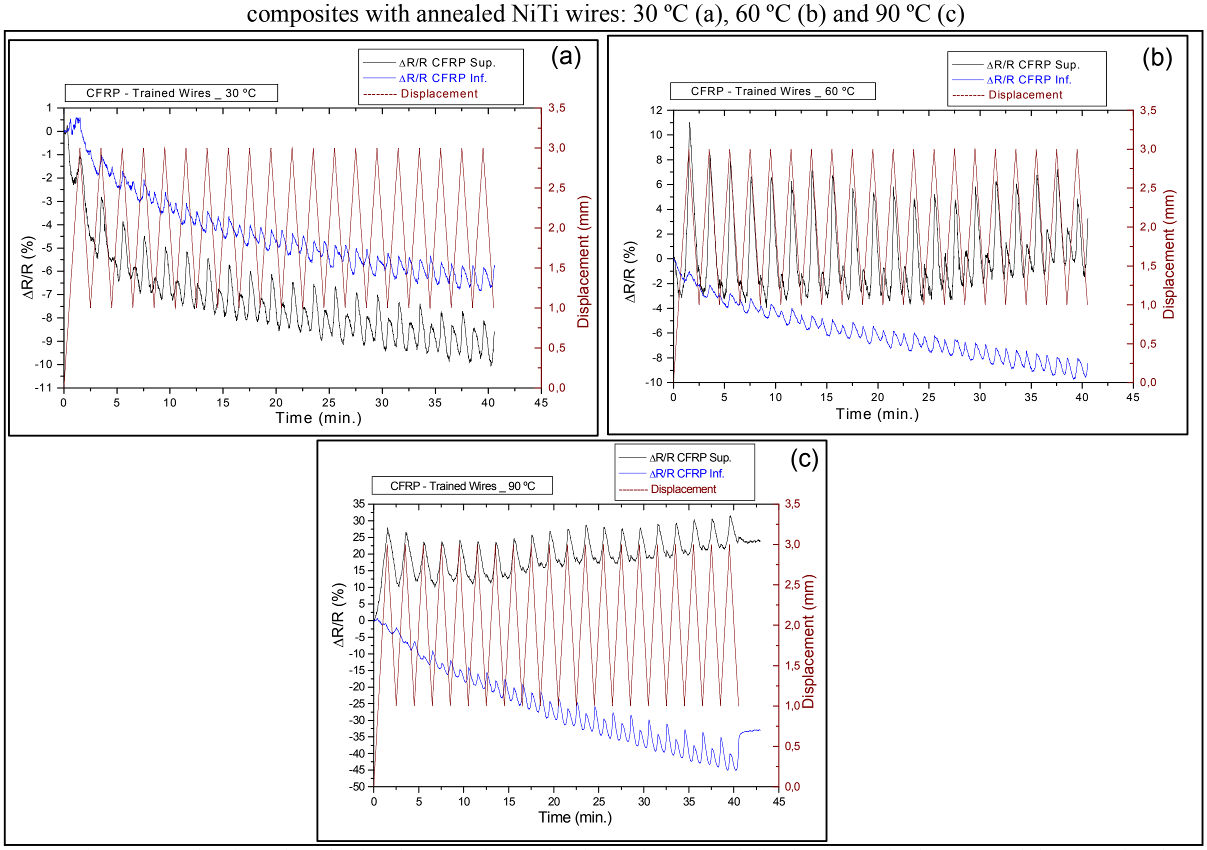

Figure 9. Behavior of ΔR/R for the superior and inferior layers and displacement in function of the time for the composites with trained NiTi wires: 30 ºC (a), 60 ºC (b) and 90 ºC (c)

Figures 7, 8 and 9 shows that the electrical resistance didn't reset to zero at the end of each cycle occurring an irreversible ΔR/R (positive or negative), that increase after each cycle. Similar results were obtained by Wang and Chung (1996 and 1997), Chung (2001) and Todoroki and Ueda (2006) during uniaxial tensile tests. As explained by these authors, the irreversible decrease in ΔR/R after the first cycle (even when the strain is completely reversible) is due to the irreversible decrease in the neatness degree of the fiber arrangement, due to rupture or other changes in the fiber networking, as separation or crack that break the contact between carbon fibers and reducing the number of contacts between carbon fibers of different layers.

The value of ΔR/R for the superior layer of the CFRP-NiTi composite with as-received wires (Figure 7) has varied positively for the three tested temperatures. As in Todoroki and Ueda (2006), this positive variation indicates a good electrical contact between the copper electrodes and the CFRP. For the tests carried out at 30 and 60ºC this ΔR/R variation increased during the cycles, while for the test at 90ºC, a stabilization starting from the seventh flexure cycle happened. The increase of the temperature, mainly for the tests at 90ºC, may have improved the level of direct contact between the carbon fibers and the copper electrodes due to changes in the properties of the epoxy resin.

The behavior of ΔR/R (Figures 7) repeats with the flexure level, of form that presents two picks, maximum or minimum (depending on the layer), during loading and two during the unloading. For the superior layer the value of ΔR/R is minimum when the loading is zero and from the beginning of the loading a direct variation with the level of flexure is observed, where ΔR/R increases with the increase of the flexure until next to the half of the loading, and later starts to have an inverse behavior again (it diminishes with the increase of the loading) reaching a minimum value of ΔR/R in the maximum displacement, smaller than the of the begin of the loading. In the unloading was observed an opposite behavior. In a study about the behavior of ΔR/R in carbon fibers reinforced composites when subjected to traction efforts, Wang and Chung (1996 and 1997) also observed similar results, evidencing that ΔR/R initially decreases and later increases with the loading, forming two picks. In accordance with these authors this behavior occurs due to variation in the degree of alignment of fiber carbon. Other authors as Chung (2001) and Todoroki and Ueda (2006), also obtained similar results for fatigue tests in CFRP. It was also verified that the behavior of the superior layer is exactly the opposite of the inferior layer. That is due to the fact that they suffer different efforts (compression or traction).

Contrarily to the case of CFRP with as-received NiTi wires, the Figure 8 reveals that the system with annealed NiTi wires presents a negative accumulation of ΔR/R for the superior layer and positive for the inferior layer, for all the test temperatures. In this case, the values of ΔR/R in the inferior layer start to present bigger values, also evidencing a clear qualitative relation with the displacement. These results demonstrate that the state of the NiTi wire also affects the behavior of the electric resistance. It was also verified that it had an increase in the values of ΔR/R with the increase of the test temperature for the superior and inferior layers. Again, the increase of the testing temperature can have provoked small variations in the properties of the resin, improving the electric contact between carbon fiber and the copper electrodes, what reflects in a better ΔR/R reply in the layers. The superior layer ΔR/R varied negatively in the three test temperatures. The inferior layer passed lightly of a negative variation in the test at 30ºC for a positive variation at 60ºC and 90ºC.

Observing the Figure 9 we can notice that the sample with trained wires had variations of ΔR/R of quite inferior intensities from the observed for the other cases (Figures 7 and 8). These variations were negative in the inferior layer for the three test temperatures, presenting an increase of the value with the increase of the temperature. The superior layer presented negative variations of ΔR/R for the test performed at 30ºC (Figure 9a) and positive for 60 (Figure 9b) and 90ºC (Figure 9c). It was also observed the variation of ΔR/R increases with the increase of the test temperatures. These results indicate the contraction effect tendency stronger in the trained NiTi wires, since the temperatures of 60 ºC and 90ºC are enough to activate the transformation of the threads, originating more important intern efforts than those presented by the treated wires.

For the samples with treated NiTi wires and in the three test temperatures (Figures 8 and 9), an inverse behavior is observed compared to the sample with as-received wires, with a variation of ΔR/R on the superior layer that initially decreases with the loading, reaching a minimum pick that inverts, reaching a maximum pick of ΔR/R when the loading is maximum. During the unloading, ΔR/R decreases, reaching a minimum value and later it increases producing again another pick. It was also observed for the inferior layer of the system with treated wires that the variation to ΔR/R has an inverse behavior compared to the superior, similarly to what was seen in the system with as-received wires. For the system with trained wires, the variation of ΔR/R of the inferior layer presents identical behavior to the superior layer for the three temperatures.

Figures 10, 11 and 12 shows the evolution behavior of corresponding compression loads to the cycles of flexure in the three temperatures (30, 60 and 90ºC) for the three different composites samples, when submitted the flexure with central arrow between 1mm and 3mm.

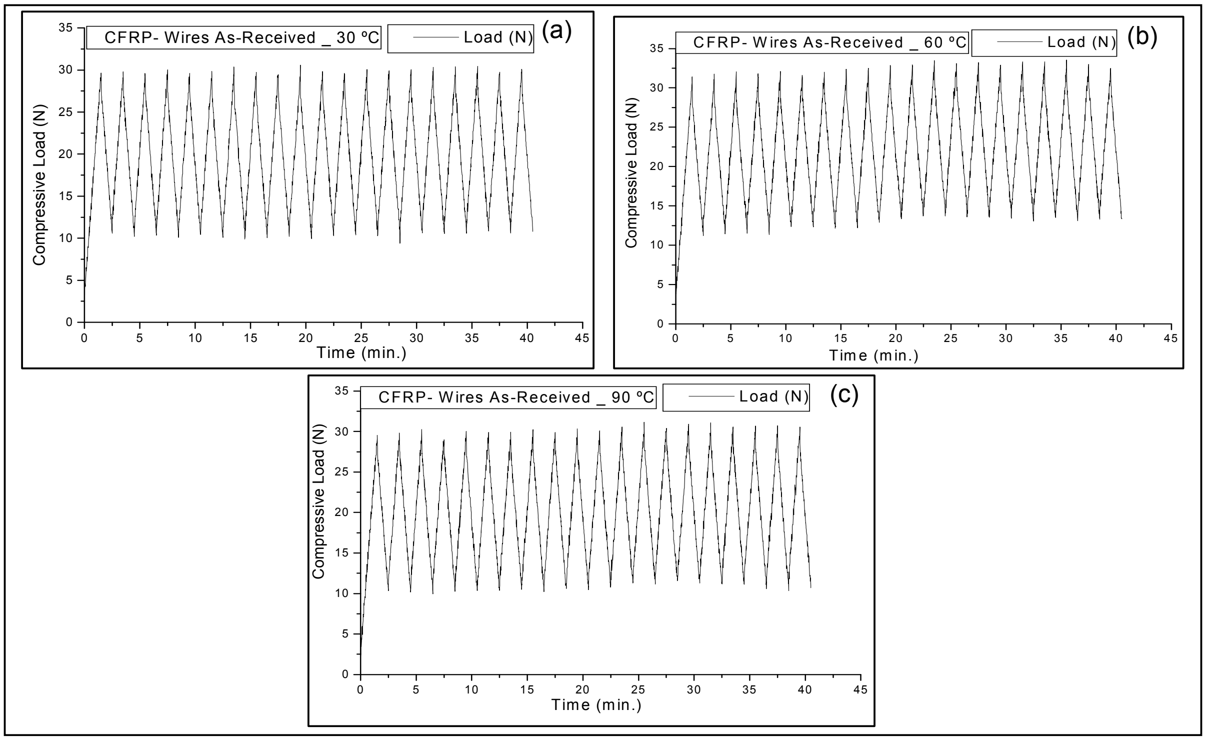

Figure 10. Evolution of the mechanical loading during the cycling of CFRP with as-received NiTi wires: 30ºC (a), 60ºC (b) and 90ºC (c)

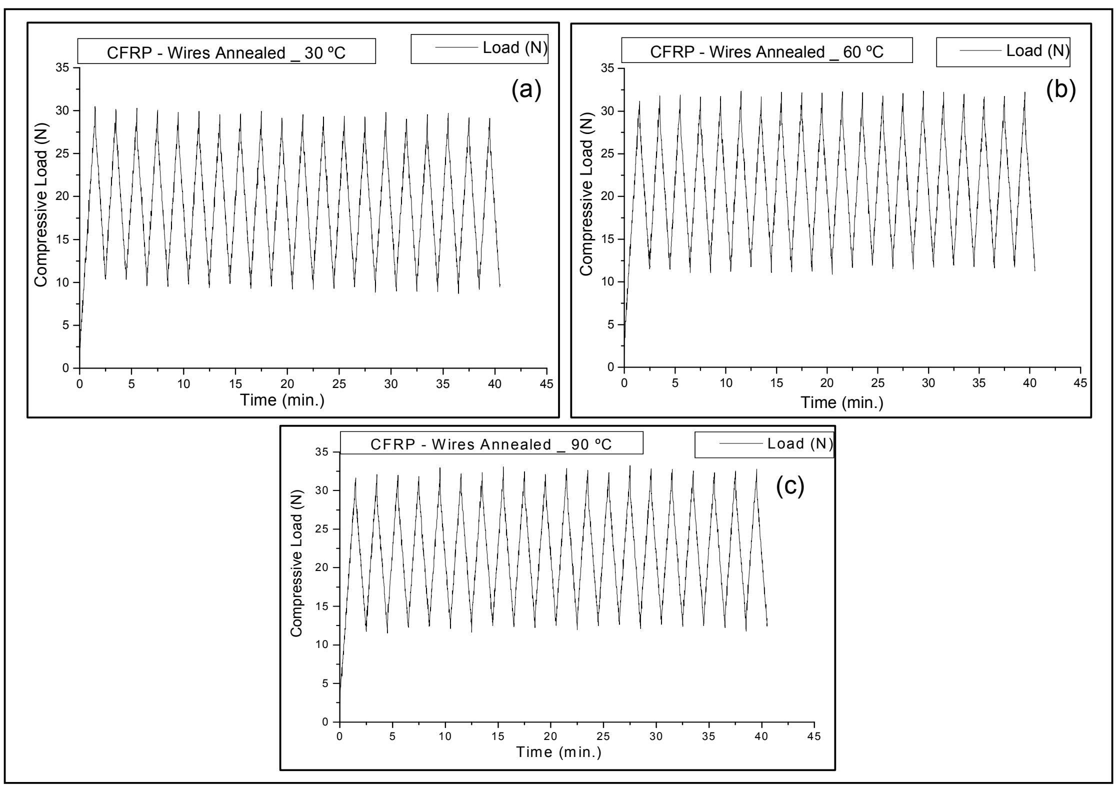

Figure 11. Evolution of the mechanical loading during the cycling of CFRP with annealed NiTi wires: 30ºC (a), 60ºC (b) and 90ºC (c)

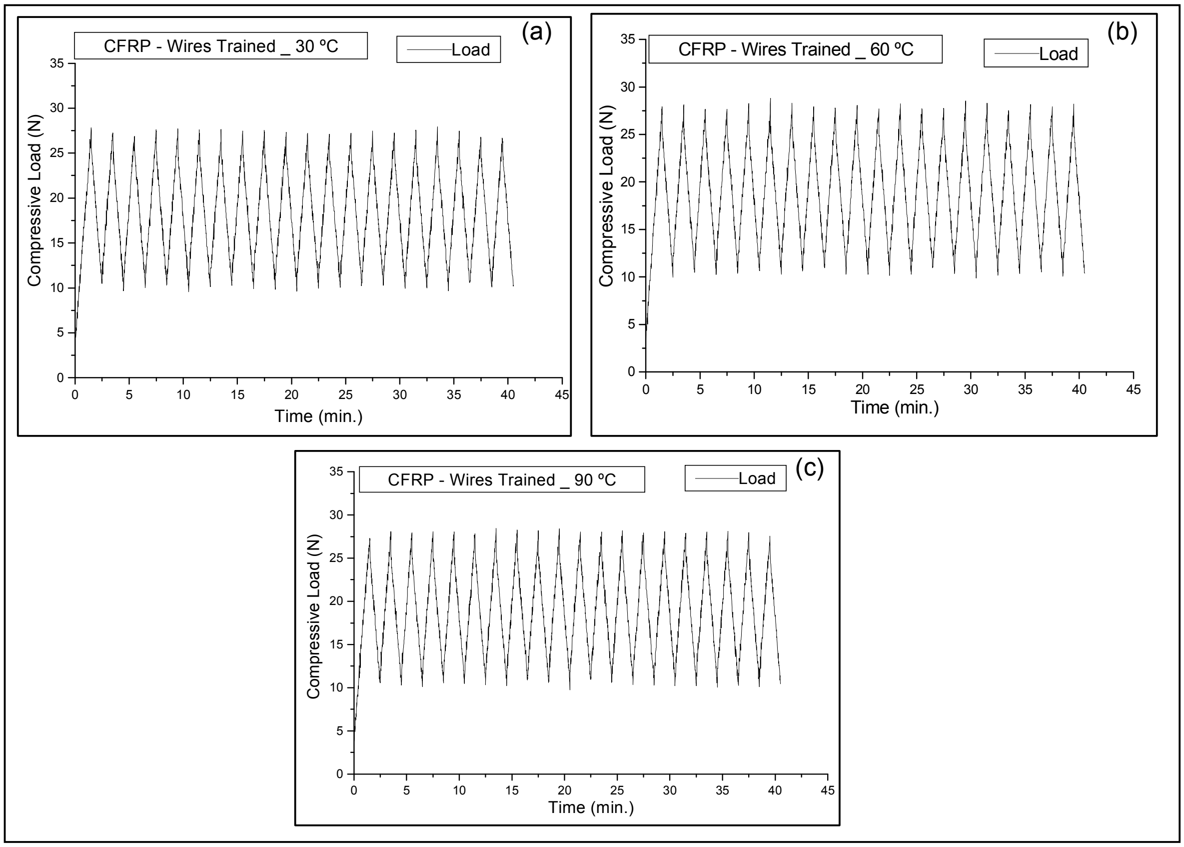

Figure 12. Evolution of the mechanical loading during the cycling of CFRP with trained NiTi wires: 30ºC (a), 60ºC (b) and 90ºC (c).

Was verified a maximum compressive load approximately constant with the temperature variation for a same sample. This fact can be justified because the test temperature didn't reach of the composites samples glass temperature (T_g ~ 165ºC). It was verified for different samples, that the sample with wires annealed presented the largest values of compressive load, for the three rehearsed temperatures and that the system with trained wires presented the smallest values. The values of the compressive load minimum and maximum for the three composites samples in the three test temperatures are willing in the Tables 4 and 5.

Table 4. Minimum values of the compressive load in the CFRP-NiTi composites

NiTi wire	30ºC	60ºC	90ºC
As-received	9,41 N	10,99 N	10,06 N
Annealed	9,65 N	11,49N	11,86 N
Trained	9,84 N	10,00 N	10,41 N

Table 5. Maximum values of the compressive load in the CFRP-NiTi composites

NiTi wire	30ºC	60ºC	90ºC
As-received	30,32 N	30,29 N	30,03 N
Annealed	31,44 N	31,6 N	32,03 N
Trained	27,80 N	27,97 N	28,05 N

4. CONCLUSIONS

The electro-thermomechanical characterization of active composites beams made of CFRP with embedded NiTi SMA wires in different states was experimentally investigated. It was observed that for the composites tested using three bending cycles at different temperatures there were verified both positive and negative changes in electrical resistance. In fact it is possible due to the contact level between the copper electrodes and the carbon fibers.

The results obtained for each composite are reproductive in cycling, as well as with the temperature change (for a same sample).

The copper electrodes allowed the monitoring of the electrical behavior of the CFRP-NiTi active composites when submitted to electro-thermomechanical tests, defining qualitative relationships among the variation of electrical resistance ($\Delta R/R$) and the load level and deflection in a three-point bending mode, so constituting a manner of monitoring the stability of these systems.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE