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# CHARACTERIZATION OF AN ACTIVE COMPOSITE OBTAINED WITH SHAPE MEMORY ALLOYS

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**Abstract.** *The incorporation of wires with shape memory alloy (SMA) embedded into a metal or polymeric matrix obtains active structures. However, those polymeric matrices have better results in a wide range of applications for this kind of application. Nevertheless, composites obtained from shape memory alloys (SMA) and polymeric matrix, in spite of exhibiting an excellent activation potential, do not have good mechanical properties. Recent studies are attempting to show the possibility of obtaining an active composite with improved mechanical properties through the introduction of SMA into an epoxy matrix, as carbon fiber reinforced polymers (CFRP) as well glass fiber (GFRP). The aim of the present study is to evaluate the activation potential of composite (CFRP) containing Ni-Ti SMA wires embedded. In this context, the objective of this research is to validate the activation potential of CFRP composites containing Ni-Ti SMA wires embedded in this matrix. small plates of composites were made by incorporating NiTi wires into pre-impregnated CFRP sheets with carbon fibers positioned at an angle in NiTi wire. The activation potential of the CFRP / NiTi compounds was investigated using thermomechanical characterization for the application of the Dynamic-Mechanical Analysis (DMA) technique. An initial analysis of a pre-embedded (CFRP) aeronautic with different inclined positions of carbon fiber was performed varying the elastic modulus in order to determinate the more adequate matrix for introducing Ni-Ti SMA wires (elastic modulus of CFRP very close to Ni-Ti wires in martensitic state) to obtain active composite. Results shows that the developed composites have the ability of being activated and the active potential increases with the increase of the volumetric fraction of NiTi wires embedded in the CFRP matrix.*

**Keywords:** *Active composites, CFRP, Shape memory alloys, Natural frequency.*

## 1. INTRODUCTION

The natural evolution of the structural material in the last 40 years has been developed in order to find the higher and specific properties, besides offering a flexibility and functionality higher than common components. This evolution has taken important due to the enormous interest by the use of composite materials in several applications. Based on

this tendency, an exploration of the original properties offered by the advanced composites in relation with the poor properties obtained using conventional structural materials, such as steel, aluminum and Hood.

Intelligent composites are structures with the ability to detect changes in operating and environmental conditions, besides promoting an adaptation through activation of actuators, trying to maintain a satisfying behaviour previously established (Tebaldi et.al, 2006). The integration of three basic elements conducts this process: (i) sensors that register internal and external information, (ii) actuators able to develop mechanical works or application of force and (iii) control systems capable to obey and emitting orders. So, are considered structural systems that contain inherent in its structure the potential of act, detection and control through the combination of various materials (Michaud, 2003), looking properties as accuracy, efficiency, functionality, durability and adaptability.

Among the functional materials employed shape memory alloys (SMAs) are the most important because they present excellent thermomechanical properties to generation of force and/or deformation as function of temperature. Current development of active structures, the SMAs is generally used as wires which have high potential as actuators with diameters below 0.2 mm, these characteristics allows the direct integration into reinforced composites with fiber in a polymeric matrix, all this without losing the structural integrity of the material (Jang & Kishi, 2005).

Recently the adherence of SMAs into composites has been very attention to reinforce composites into a polymeric matrix as CFRP (Carbon Fiber Reinforced Polymers) due to this combination of materials can present adequate properties to develop smart composites. Besides of present a higher mechanical resistance associated with a low specific mass since CFRP has the capacity of conduce electric current allowing its structural monitoring (Chung, 2001).

Some kinds of active structures can be obtained thought the incorporation of wires based on SMAs which present the shape memory effect (SME) into a metallic or polymeric matrix. As polymeric matrices have a major applicable for these purpose because they have an elastic modulus lower than wires based on SMAs. Giving training to the obtained composites from the join of SMA + pure polymeric matrix, despite of offer an excellent activation potential not present high mechanical properties, such as an elastic modulus less than 10 GPa. A new alternative to obtain an active composite with improved mechanical properties consists in obtaining active composites by the incorporation of SMA-wires into an epoxy matrix with carbon fibers (CFRP) or glass fibers (GFRP). In this context, the objective of the present study is to validate the activation potential of CFRP composites containing Ni-Ti SMA wires embedded in this matrix. In this context, small plates of active composites were made by incorporating NiTi SMA Wires into pre-impregnated CFRP sheets with the carbon fibers positioned at an angle to NiTi ( $15^\circ / 0^\circ / 15^\circ$ ). The activation potential of the obtained CFRP / NiTi composites was investigated using thermomechanical characterization whit Dynamic-Mechanical Analysis (DMA) technique.

## 2. EXPERIMENTAL PROCEDURE

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 whit (diameter = 0,29mm) 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.

Initially a study was carried out on how the good of the constituents to be used for the preparation of the components, through the characterization of the phase transformation temperatures of the yarns, the glass transition of the epoxy resin and the mechanical properties. In order to evaluate the composition of CFRP-NiTi samples with efficient activation demonstration potential, such as CFRP good evaluated at different fiber angles, different NiTi alloys (Alloy H and Alloy M) were evaluated.

For developing this project, were made two types of specimens which were distinguished from each by the kind of Ni-Ti used (alloy H and M), denominated CFRP-NiTi H and CFRP-NiTi M respectively.

The Ni-Ti wires used were thermally treated at  $450^\circ\text{C}$  during 30 minutes with a posterior cooling in argon and chemically cleaned with  $3\%\text{HF}+15\%\text{HNO}_3+82\%\text{H}_2\text{O}$  (% in volume) during 10 minutes. According with Xeu in his methodology utilized (Xu, *et al*, 2003) to removing any oxide layer deposited on the surface of the samples after heat treatment. The Fig. 1 presented the sample configuration.

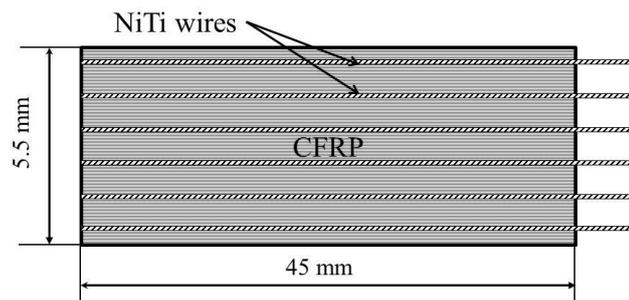


Figure 1. Schematic design of active composites CFRP-NiTi.

The NiTi wires, CFRP and the obtained CFRP-NiTi composites were characterized by Dynamical Mechanical Analyses (DMA) and Differential scanning calorimetric (DSC), verifying the behavior of the storage modulus (E) and the temperature of phase transformation when heated. The samples were tested in DMA by single cantilever mode (Tensile and flexure mode) with a rate of 2°C/min, frequency of 1 Hz and amplitude of 10 µm and in the DSC with a rate of 5°C/min.

For testing the specimens as DMA as DSC was used from TA Instruments being DMA Q800 and DSC Q20 respectively. In other hand, optical microscopy (OM) was performed using a Olympus, model BX51.

### 3. RESULTS AND DISCUSSION

Initially, before to producing composites was realized a previously characterization of the Ni-Ti wires and of the CFRP matrix employed in this study in order to know its thermo-mechanical properties. In Fig. 2, is showing the variation of heat flow in function of temperature, 2a for Ni-Ti wires (alloy H and M), while Fig. 2b-c show the variation behavior of the storage modulus in function of the temperature for samples of Ni-Ti (alloy H and M) and CFRP matrix without wires.

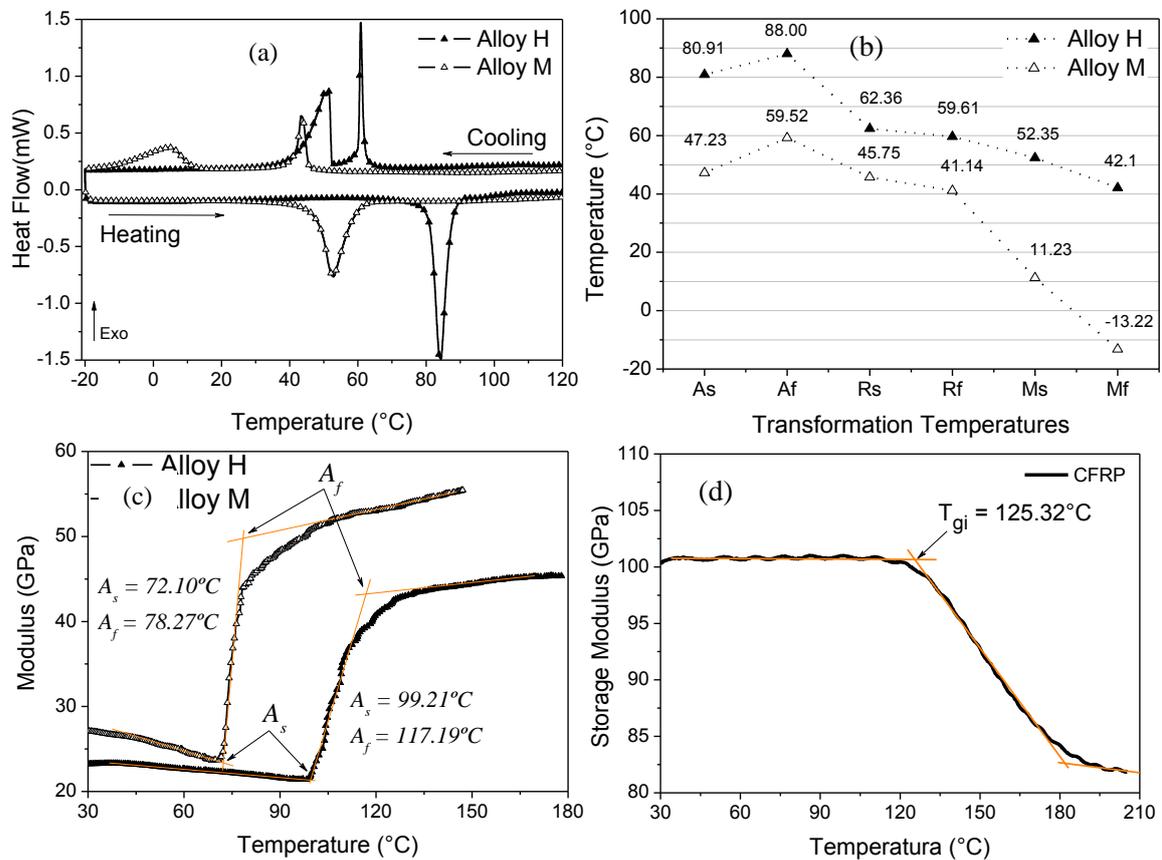


Figure 2. DSC and DMA characterization of NiTi wires

Applying a method of tangents on DSC and DMA curves, can be determined the phase transformation temperatures for both alloys. It observed a difference between the temperatures measured by DSC as DMA. This fact is associated to the heat transfer phenomenon suffered by metallic samples tested on DMA, this because the sample temperature is lower than temperature in the heating chamber of the DMA equipment (Vilar et al, 2011). Based on temperature values, it observed that Ni-Ti wire corresponding to the alloy H presents a completely martensitic phase at room temperature, while that Ni-Ti wire corresponding to alloy M show an intermediate phase known as rhombohedral phase. As composites CFRP-NiTi produced are tested close to environment temperature (30°C) until a temperature which allow the total phase transformation of the composite's wires at (110°C). Wires corresponds to alloy M do not present their phase transformation completely and therefore their total efficiency will not achieved. Maximum temperature for composites CFRP-NiTi was determined in function of the glass temperature corresponding to a polymer used as matrix

because it represents an important limit where the material suffer a high decrement of stiffness due to diminution in the viscosity of the resin and consequently a decreasing in adhesion properties causing a transfer of load directly to the reinforcing fibers. Thereby was decided making test based on the composite functionality respecting at a maximum limit of 110°C lower at the glass transition which allow a total phase transformation of the wire for austenitic state.

Based on results obtained by DMA (figure 4c) can be verified that CFRP presents a good thermal stability at 110°C with an initial glass temperature ( $T_g$ ) about 125.32°C which is higher than temperature final of activation of the Ni-Ti wires ( $A_f$ ), thereby can be fully transformed without involve the integrity of CFRP composite.

Finally, for Ni-Ti wires (figure 2c) a huge increment of storage modulus during phase transformation can be appreciable, for alloy H rising from 21 GPa to 45 GPa on fully austenitic state and 23 GPa to 52 GPa for alloy M. For both wires an increase on storage modulus about 100% was observed.

Analyzing the values of storage modulus corresponding to the Ni-Ti wires (alloys H and M) besides of composite CFRP which is fully aligned, can be noted a huge difference caused by the phase transformation, highlighting that Ni-Ti wires present lower modulus than CFRP. Results reported by Vilar et al (2011) show that the introduction of Ni-Ti wires into composites with high modulus did not produce composites with the capacity of activation, because an increasing on stiffness of the Ni-Ti wires is not enough to promote variations of modulus in the composite produced, besides that wires present a volumetric fraction relatively lowest in comparison with CFRP matrix.

Thereby, in order to obtain an active composite producing by CFRP-NiTi, was based in the methodology described before to produce a composite of CFRP with the appropriate angle to attempt a structure where the mechanical properties are as much as possible approximate at the Ni-Ti wires, inclusive before to the phase transformation.

Initially, were built composites in both ends, i.e with the carbon fibers completely aligned in relation with the NiTi wires (in this way the storage modulus of CFRP takes the maximum value), and a second configuration where the carbon fibers are at 90° respecting to the NiTi wires (in this way the storage modulus of CFRP takes the minimum value). In order to avoiding a collapse of the composite produced in the second configuration were placed two of the four layers at 90° giving a same direction as layers as NiTi wires, these samples are denominated as CFRP-NiTi-01(carbon fibers at 0°) and CFRP-NiTi-02 (carbon fibers at 90°). Figure 3 shows results of the storage modulus ( $E$ ) and damping modulus ( $Tan \delta$ ) behavior in function of the temperature for CFRP-NiTi-01.

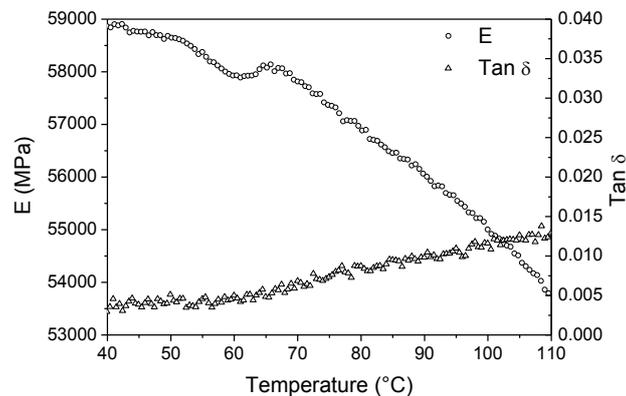


Figure 3. Dynamical-Mechanical properties of the CFRP-NiTi-01

As can be seen in figure above, the introduction of the NiTi wires cause a decrement on the storage modulus of the composite CFRP-NiTi-01, which did not happen with the CFRP pure, obviously because the modulus of the NiTi wires is lower than CFRP matrix. During the heating for activating the NiTi wires were not observed significant variations which could be associated with the activation potential of the CFRP-NiTi-01 composite obtained, being observed only a small peak corresponding to NiTi phase transformation in a range from 60°C to 70°C. The storage modulus decreased was attributed to the fact that CFRP has a storage modulus higher than NiTi wires. A similar behavior was reported by Bettini et. al., they secured that when NiTi wires are oriented on the same direction to the reinforcement fibers exist a higher adherence between them. In the other hand, this configuration leads generally to a low activation power due to the high stiffness of the host material, being necessary high volume fractions to obtain a structure with activation potential.

How can be seen in Fig. 4 are presented results corresponding to the variations of storage modulus and  $Tan \delta$  in function of the temperature, for composite CFRP-NiTi-02.

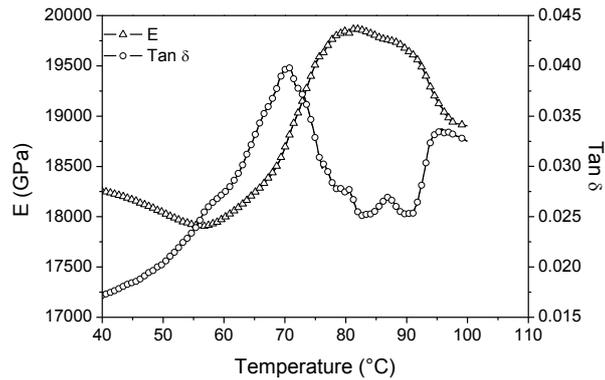


Figure 4. Dynamical-Mechanical properties of the CFRP-NiTi-02

Based on the behavior observed in figure 4, can be corroborated that with the introduction of two of the four layers of CFRP positioned at an angle of  $90^\circ$  is promoted an important reduction on storage modulus of CFRP-NiTi-02, giving a maximum value of 18.2 GPa, which is very close to the modulus's value for NiTi wires on martensitic state (23 GPa). This diminution in modulus is attributed mainly at the high invasiveness caused by the NiTi wires positioned at  $90^\circ$  in relation to the carbon fibers. In this case, during heating was observed an increase about 10% in storage modulus of CFRP-NiTi-02 around the phase transformation temperature of the NiTi wires. At the same time, was observed a great increment of damping modulus. Although, a composite with high activation potential was obtained, the results are not good because the mechanical properties were not very high as was expected for a structure reinforced with carbon fibers. Also, through optical microscopy important structural defects affect the use of these composites.

Figure 5 shows optical images corresponding to CFRP-NiTi-01. A low level of invasiveness of the wires on the composite can be seen, so that the wire becomes part of the CFRP layers (see Fig. 5a), despite all that, was observed a good interface between the NiTi wires and the CFRP matrix (see Fig. 5b).

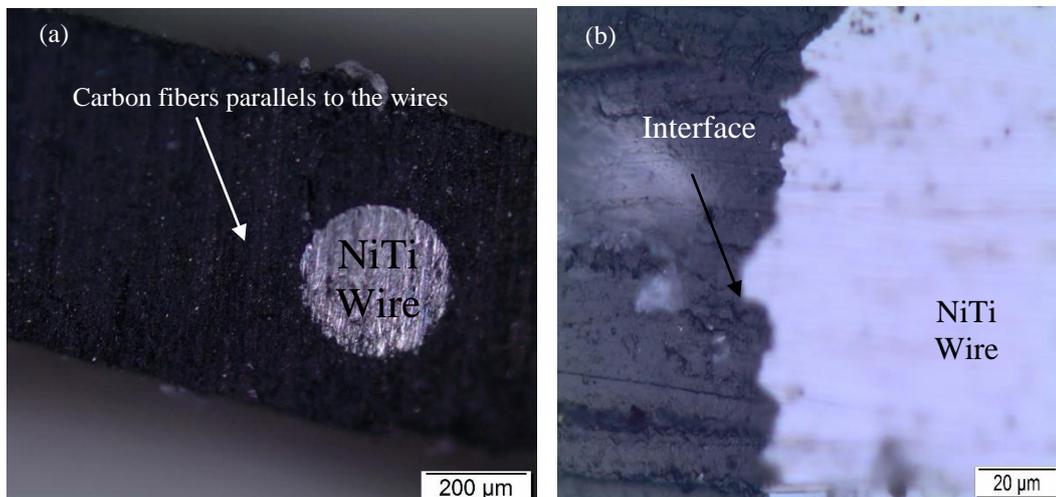


Figure 5. Optical microscopy of the CFRP-NiTi-01

For those composites where the NiTi wires were introduced into the CFRP matrix perpendicularly, it was observed a high level of invasiveness of the wires into a defective interface which presents some gaps and low adherence between the CFRP layers. It was verified clearly that NiTi wires has a weak integration with the CFRP and a high superficial area does not in direct contact with the reinforcing fibers, but with the epoxy resin a high adherence can be seen, for understanding in a better way see Figure 6.

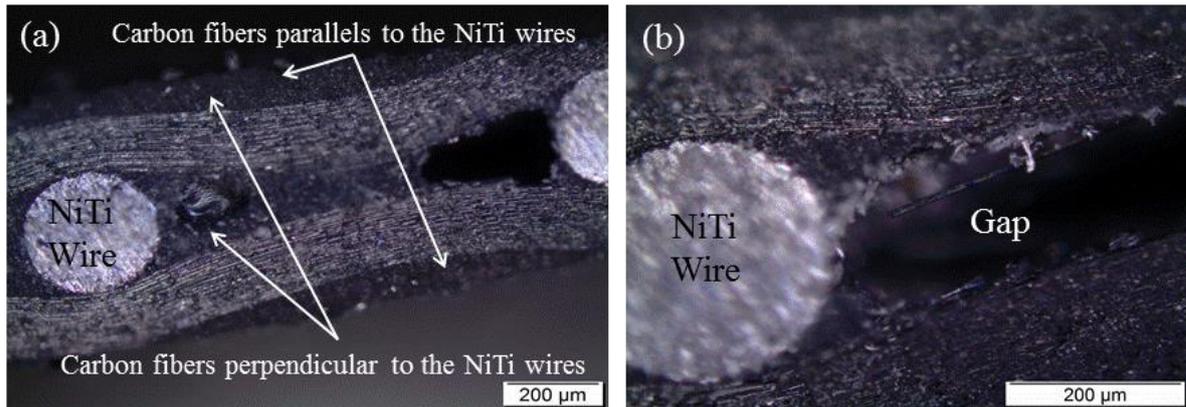


Figure 6 Optical microscopy of the CFRP-NiTi-02

Accordingly, a composite with low Young's modulus was obtained (lower than NiTi wires modulus) but a good activation potential. Therefore, defect presents explain the stiffness increasing of 10% in a range from 60 to 80°C, which was not maintained at temperatures above 80°C.

Comparing Fig. 5 and 6, can be observed that in the CFRP-NiTi-02 the NiTi wire is embedded into the polymeric matrix, while the fibers are spaced. On the other hand, CFRP-NiTi-01 presents a direct contact between carbon fibers and NiTi wires, around the circumference. According to reports published by Faiella et. al. (2011), this direct contact in both materials leads to a counteraction to the unidirectional fibers over the NiTi wires causing high load transference between the metal and polymer. So, it is necessary an optimization of the system, in order to producing composites with a low invasiveness level and, at the same time, with a storage modulus as close as possible to the NiTi wires, besides of presenting a good adherence as CFRP and NiTi wire layers. All this, lead us to develop a deeply study of the carbon fibers at different angles in relation to the NiTi wires. Figure 7 shows the storage modulus behavior of the composites with carbon fibers placed at different angles (45, 40, 35, 30, 25, 20, 15, 10, 5 and 0°).

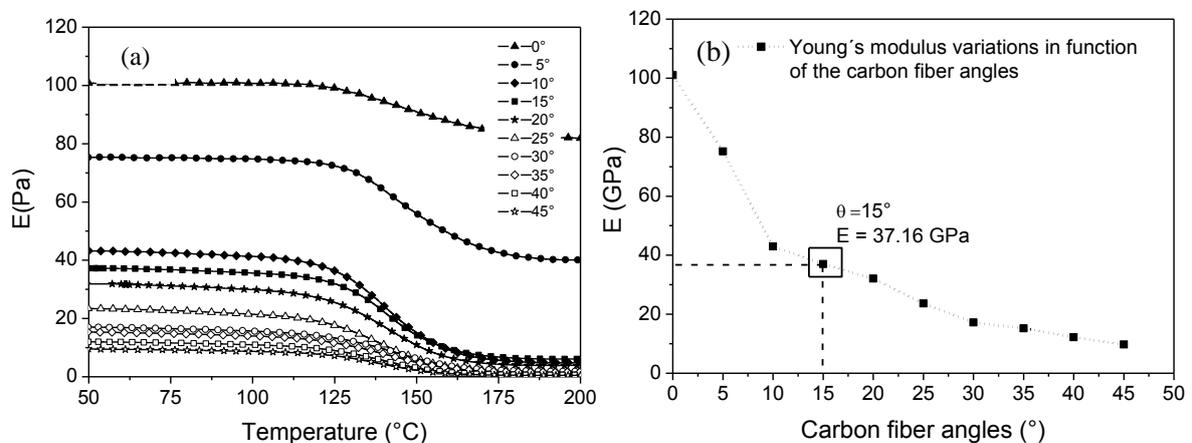


Figure 7 Storage modulus variations in function of the temperature for CFRP composites at different angle

From figure 7, can be seen that storage modulus of CFRP suffers a great decrease respecting to the increasing of angle which the carbon fibers are positioned. Therefore, it is expected that CFRP with the carbon fibers aligned at 15° represents a good option to design CFRP-NiTi composites with best properties. Initially, can be observed that storage modulus (42.5 GPa) is lower than those presented by NiTi wires in austenitic phase. In this way, can be estimated that there would not an invasiveness of NiTi wires due to they are positioned by a relatively small angle in relation to the carbon fibers. At the same time, these composite presents a storage modulus near to the NiTi wires before that before transformation phase appears, conferring to the composite CFRP-NiTi good characteristics of functionality higher than those presented in Fig. 4.

Thus, from these results were produced two types of composites configured such that NiTi wires are at 15° in relation to the carbon fiber: i) NiTi wire from the alloy M (diameter=0.29mm) and ii) NiTi wire from the alloy H (diameter=0.305mm). These new samples were called CFRP-NiTi-H and CFRP-NiTi-M. Were conected 7 composite samples with different yarn fractions for each alloy, using the carbon fibers at 15°.

During the thermomechanical characterization in DMA data of the modulus of elasticity and damping factor ( $\tan \delta$ ) were collected. Figure 8 shows the result of this test obtained for one of the samples CFRP-NiTi H with the behavior of the Elastic modulus as a function of temperature.

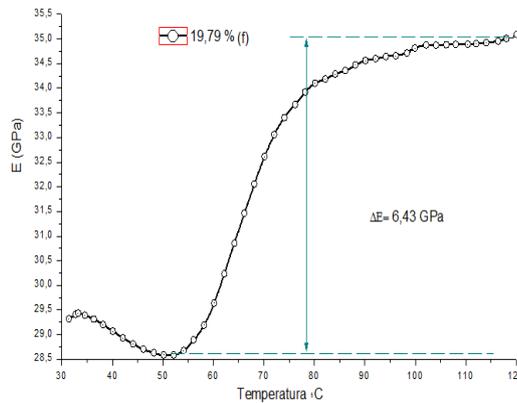


Figure 8. Behavior of the elasticity modulus in function of temperature for one of the CFRP-NiTi-H samples.

Figure 8 shows that the behavior of the mechanical properties obtained qualitatively resembles results by authors such as Ni et al (2007) and Raghavan et al (2010), with an increase of the elasticity modulus in the temperature range where a phase transformation of NiTi wires occurred.

Due to the results obtained in the tests carried out in DMA, it was verified that the NiTi activation leads to a recovery of the manufactured composites thermal stability, even for smaller volumetric fractions for a study. The thermal properties of elevated temperatures (smaller than  $T_g$ ) without compromising thermomechanical properties than the possibilities of using the structures before transformation. system.

For all cases, note the elastic modulus flow increases with the increase in the volumetric fraction of the NiTi wires up to a threshold value in which even increasing the fraction of the wires or gain begins to decrease. This phenomenon develops a discontinuity between NiTi and CFRP wires. The CFRP supports up to a certain percentage of NiTi wires without their structure being compromised. From a given value the heterogeneity is high and a charge transfer between a matrix and easier become weak compromising an integrity and thus a good of the composite. As the number of wires increases, a situation in which CFRP (lower and upper) layers are almost untouched causes a weak interface between the constituents. Figure 9 shows an optical microscopy image of a sample of CFRP-NiTi where a volumetric fraction of yarn is high. For this case it is verified that there is almost no contact between a layer of upper and lower CFRP.

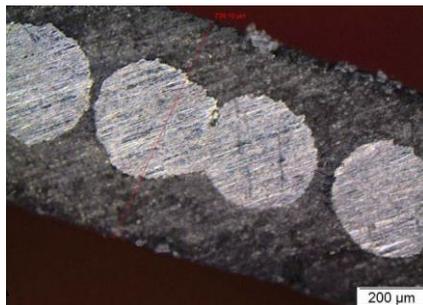


Figure 9. Optical microscopy of a sample of CFRP-NiTi with high volumetric fraction of NiTi wires.

The samples tested in the tensile mode showed higher stiffness gains as a function of the volumetric fraction, when the NiTi wires are activated, than the tests performed in the bending mode, even using approximately equal volume fractions. Figure 10 shows the percentage increase in modulus of elasticity in the samples tested in the two modes (traction and bending). The samples tested in tensile mode showed higher stiffness gain as a function of the volumetric fraction, when the NiTi wires are activated.

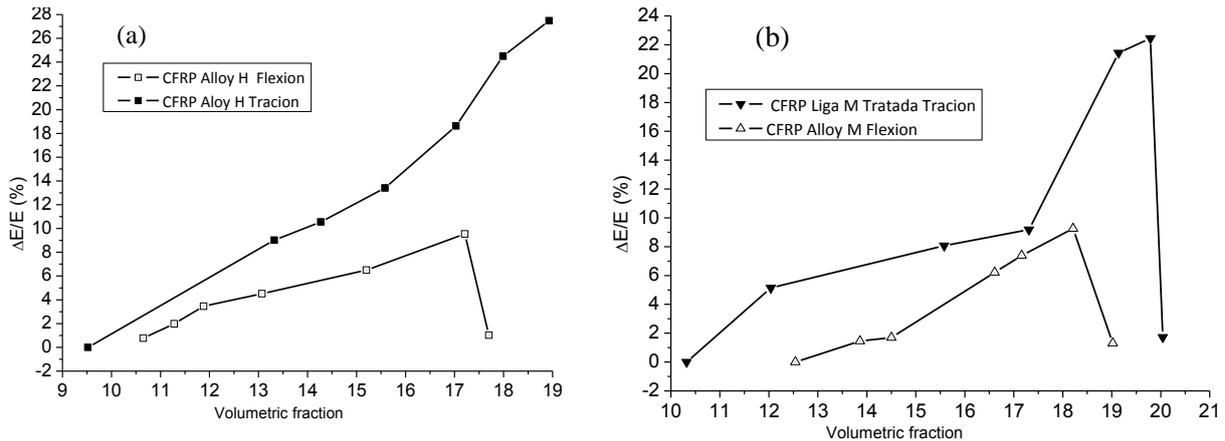


Figure 10. Percentage of modulus of elasticity. (a) Alloy H; (b) Alloy M.

The results presented in Fig. 10 indicate that there were greater percentage gains of modulus of elasticity for the tests performed in the traction mode. This phenomenon is probably due to the fact that the NiTi wires are positioned along the neutral line of the composites and during bending the neutral line of a structure remains unchanged Hibbeler (2010), that is, it does not participate in the subjection of stresses. For the traction test, the load is applied in the axial direction to the NiTi wires and they participate fully.

These results show that the composites with NiTi alloys of the H-alloy show higher modulus gain values than the composites with alloys of the alloy M. The highest values were observed for the composite with the alloy H, when tested in the uniaxial traction.

For smaller volumetric fractions of alloy NiTi yarns, higher modulus of elasticity increases than for alloy M, and this phenomena was observed for the two test modes (flexion and tensile).

The variation of its natural frequency with the activation of the wires of the CFRP-NiTi with treated M-alloy wires is shown in Fig. 11.

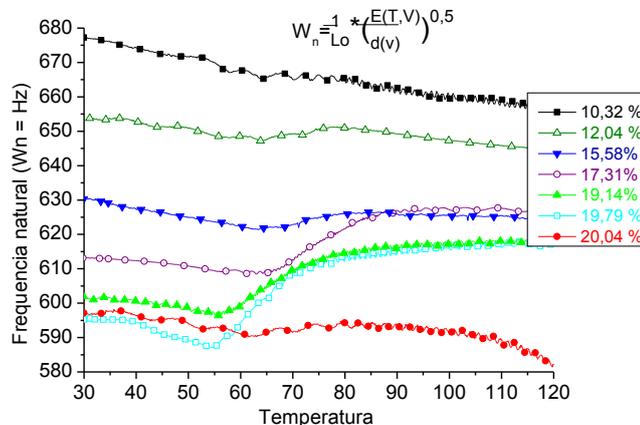


Figure 11: Variation of Natural Frequency x Temperature.

As the natural frequency of a beam is directly proportional to its rigidity, a similar behavior is observed to the elastic modulus, so it is possible to alter the natural frequency of a structure through heating and consequently variation of the elastic modulus of the actuating wires with shape memory alloys.

#### 4. CONCLUSIONS

It is observed that with the heating there are positive variations of the natural frequency, which implies a real activation potential with regard to the possibility of attenuation of mechanical vibrations by the increase of the stiffness of the structure and consequent displacement of the natural frequency of the excitation frequency.

Finally, this study showed that active composites obtained, using the CFRP as a matrix for the incorporation of NiTi actuator wires, represent an excellent choice of material for application in aeronautical projects, presenting mechanical properties and active potential well above those already observed in composites that use polymer and aluminum resins

as an inlay matrix and can be applied as temperature and expansion sensors for rigidity, damping and consequently vibration control, among other specific applications that may require the active potential that these structures can provide.

## 5. ACKNOWLEDGEMENTS

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