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DEVELOPMENT AND THERMOMECHANICAL CHARACTERIZATION OF SHAPE MEMORY ALLOY CONICAL ACTUATORS

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Abstract. *Shape Memory Alloys (SMA) are smart materials receiving a special interest by being able to recover pseudo plastic strains of high intensity (~5%) by means of a simple heating. Therefore, this study aims to evaluate the overall manufacturing and thermomechanical behavior of bifunctional SMA Belleville actuators with homemade technology for force generation in bolted joints through the Shape Memory Effect (SME) and Superelasticity (SE). For this purpose, it were selected Ni-Ti and Ni-Ti-Nb SMA that present superelastic behavior at room temperature (~25°C). After obtaining the conical Belleville actuators with different height/thickness (h/t) ratios for each chosen SMA composition, thermomechanical characterization was carried out using the technique of Differential Scanning Calorimetry (DSC) and mechanical cycling tests at different temperatures and strain levels using of two universal testing machines, one of them equipped with a heating chamber. Based on the force-deflection behaviors obtained, one can notice a wide range of recoverable strain at temperatures near or above room temperature. Furthermore, it was found through experimental results compared with literature that the mechanical characteristics of this type of actuator is directly associated with the h/t ratio. The results demonstrated the potential of this conical SMA actuator to the development of practical applications.*

Keywords: *Shape Memory Alloys, Belleville actuators, Shape Memory Effect, Superelasticity.*

1. INTRODUCTION

In the current stage of scientific and technological development in the area of smart materials, researches worldwide advances have allowed their effective use as thermomechanical sensors and actuators. Within this group of functional materials, Shape Memory Alloys (SMA) present main phenomena associated with their special thermomechanical behaviors, as shape memory effect (SME) and superelasticity (SE) (Lagoudas, 2008). These SMA are able to recover the original geometry (or develop substantial forces by restricting their recovery) when deformed pseudo plastically at low temperature and then heated (SME case). When at high temperature and submitted to mechanical load and unloading (stress), SMA recovers the imposed strain (SE case).

In the year 2000, the organizational units SINTEF Petroleum Research, Saga Petroleum, Norsk Hydro, Statoil and Chevron, along with researchers from the Norwegian University of Science and Technology and the company Memry Europe, engaged in a design concept of intelligent bolted joint. For this design, it were employed smooth cylindrical SMA washers for force generation in bolted joints, widely used in platforms, pipelines and processing plants (SINTEF, 2000).

These SMA actuators may be able to recover a pre-load level reduced due to a possible relaxation of a bolted joint by exposure to high temperatures (between 50 and 150 °C) and mechanical vibrations. For that, this joint must be equipped with a system containing an SMA washer actuator pre-deformed under compression, a heating system and a monitoring system. Thus, the monitoring system would be able to detect a critical predetermined load level and coupling force and control the heating system in order to activate the expansion of the actuator by SME and thereby regain force coupling. If the bolted joint with bolts work naturally heated, the dilatational load compensation, obtained by SME activation, can occur naturally by increasing the joint's tightness, since the phenomenon is contrary and greater than the thermal expansion effect. In this case, the joint's relaxation monitoring and heating SMA actuator systems are not required.

In addition to the force generated by SME restriction, another potential on the use of such SMA actuators would be the stiffness increase of the bolted joint after continuing increasing the temperature above the final austenite temperature A_f (martensite to austenite transformation) (Simões, 2012). This activated state may provide the actuator

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larger strains under loads almost constant, i.e., no reduction in preload due to the phenomenon of SE, which would not be possible in a common joint. Thus, the actuator may present a bifunctional behavior with force generation associated with higher stiffness, depending only on the applied temperature.

With this new recovery load system applied to bolted joints, the use of a SMA conical actuator assures tightness minimizing the use of external torque. This, in turn, allows a reduction or even an elimination of the shear stresses in the bolt. From this, these joints become more reliable, so as to reduce the occurrence of failures.

Thus, the overall objective of this experimental study was to evaluate the thermomechanical behavior of bifunctional SMA Belleville actuators, manufactured with national technology, to generate force in bolted joints used in the Oil and Gas sector, through SME and SE.

2. THEORETICAL BASIS

2.1. Shape Memory Alloys: Shape Memory Effect (SME) and Superelasticity (SE)

Shape Memory Alloys (SMA) are materials that have the special feature of recovering high permanent strains levels when subjected to heating. These materials can easily be deformed when are below the critical temperature for the formation of a martensite structure (M_f), in which these materials behave in a ductile manner. When the SMA is subjected to heating, above the critical temperature for the formation of an austenite phase (A_s), it recovers the pseudo plastic strain, start returning to its original shape.

The thermomechanical behavior associated with thermoelastic transformation of SMA mainly involves the following phenomena: Shape Memory Effect (SME) and Superelasticity (SE).

The SME is the ability to recover an apparent plastic strain through a suitable temperature field. This phenomenon is associated with a thermoelastic martensitic phase transformation (Otsuka and Wayman, 1998). When a SMA is maintained at a temperature above A_f and subjected to an increasing mechanical loading, one can notice there is a significant strain increasing, shortly after the elastic region of strain, with a substantially constant stress level. This is due to the fact that at that temperature the martensite can be stress-induced.

Figure 1 illustrates a SMA behavior that can be tested at two different temperatures. At a temperature greater than A_f, there is the superelastic effect, where after the formation of a high strain level and the load removed, the material returns to its initial state showing a stress hysteresis. The same material tested at a temperature below M_f presents SME, because after unloading there is a residual plastic strain which can be recovered by heating above A_f.

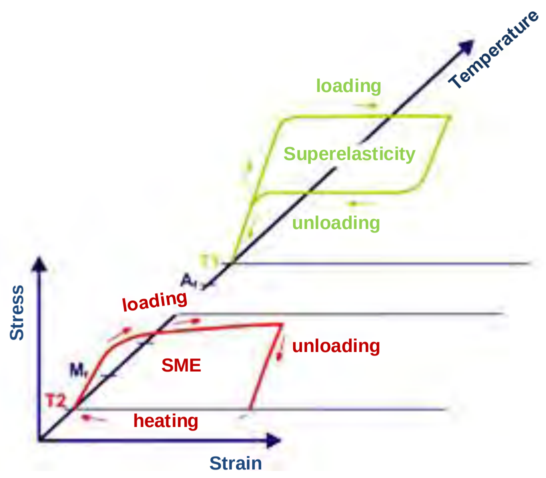

Figure 1. Thermomechanical stress-strain behavior of a SMA at different temperatures.

The strain level, under mechanical loading, which can be completely recovered after the subsequent unloading may reach values up to 10%, depending on the SMA. This value is very significant if compared to the value of the elastic strain levels of steel, which does not exceed 0.2% (Semião, 2010).

2.2. Design of Belleville Washers

Belleville washers, patented in France by J. F. Belleville in 1867, are conical washers which have a non-linear relationship between applied load and deflection, as pointed out in Fig. (2). This mechanical behavior makes them very useful in some applications. These mechanical elements are extremely compact and can endure large compressive

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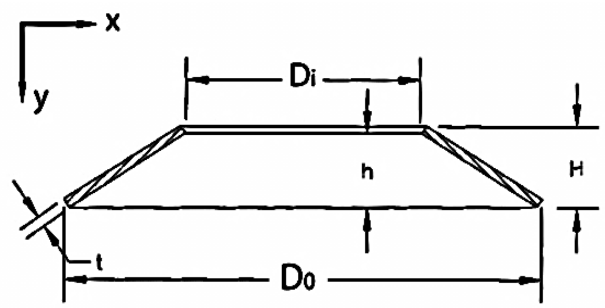
stresses with very limited deflections. Therefore, these washers are used when high loads are required as well as tight spaces are available (Norton, 2006).

According to Shigley *et al*. (2006) the non-linear relationship of the force (F) required to promote a deflection (y) in these elements can be calculated from Eq. (1). Figure 2(a) show the main geometrical parameters used for the design of Belleville washers.

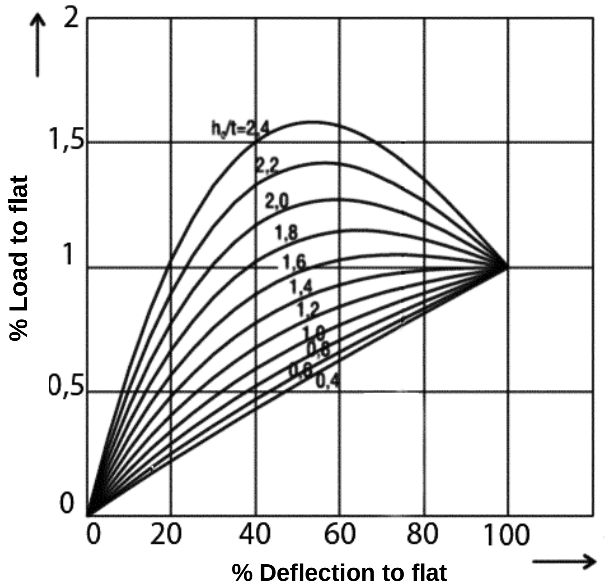
$$F = \frac{4.E.y}{K_1.D_0^2\left(1-v^2\right)}.\left[(h-y)\left(h-\frac{y}{2}\right).t+t^3\right] \tag{1}$$

where: D_i is the internal diameter, D_o is the outer diameter, where D_o is equal to $2D_i$, t is the thickness of the washer, R_d is the ratio between the diameters (D_o/D_i), v is Poisson's ratio, E is the material's elasticity modulus and K_1 is a constant parameter, depending on the element's geometry.

The curves shown in Fig. 2(b) are originated from Eq. (1) and represent the load-deflection relationship for different *h/t* ratios of the washer.



(a)



(b)

Figure 2. Mechanical behavior of Belleville Washers. (a) Main geometric parameters for designing. (b) Load-deflection behavior for different *h/t* ratios (Schnorr, 2003).

In this regard, one can merge the Belleville washer geometry features with the nonlinear thermomechanical behavior of a SMA to originate a very promising concept. It is expected generation of forces sufficient to provide preloads in bolted joints, reducing the probability of failure in case of different sorts of loading.

3. MATERIALS AND METHODOLOGY

3.1 SMA Selection

The main criterion used for the SMA selection was to obtain a final activation temperature (A_f) near to room temperature (~ 25 °C). Thus, one has chosen a Ni-Ti SMA with 55.3Ni-44.7Ti (% wt) and another with addition of Nb, which chemical composition was 48Ni-38Ti-14Nb (% wt). According to Zhao *et al*., (2006), both of them must exhibit superelastic behavior at room temperature.

These SMA actuators must be capable of generating forces when deformed at low temperatures and heated through the SME, and alternatively may present SE when mechanically loaded at temperatures higher than the room temperature in this case.

3.2 SMA Bellevile Washer Manufacturing

To manufacture the SMA washers, the metals which constitute the alloy (Ni, Ti and Nb) were commercially purchased and then melted into a machine that uses a plasma melting process followed by injection molding, called

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Plasma Skull Push Pull (PSPP) (De Araújo *et al.*, 2009). The melting process sequence is shown in Fig. (3), in a simplified manner.

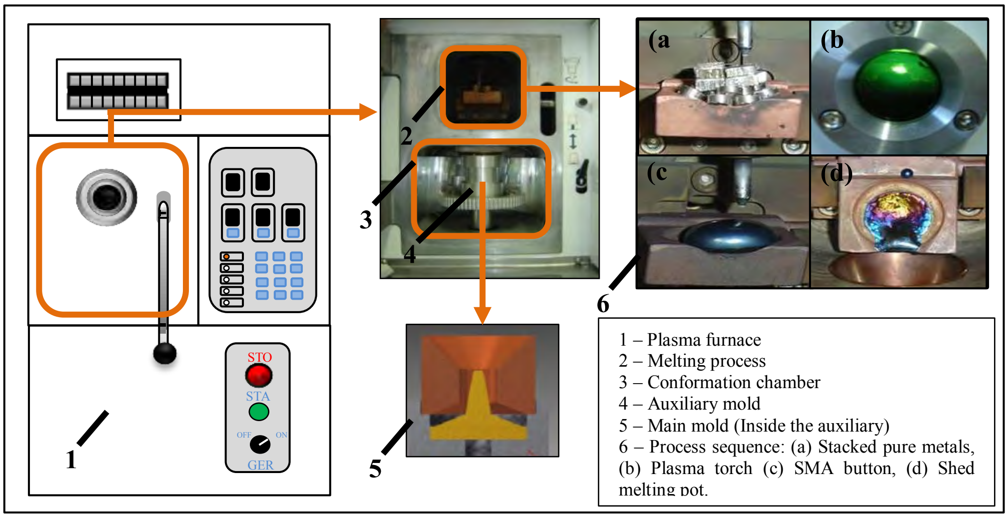

Figure 3. Scheme of the manufacturing process used to obtain the SMA Belleville washers.

The melting was carried out by stacking the pure metals in a copper melting pot and under a tungsten electrode (item 5a). In this process, the metal is molten in a thin layer on material itself, in an argon environment, and then injected into a metal mold (item 4) leading to obtaining a SMA cylindrical bar (not shown), with diameter of 22 mm and height of 30 mm.

After obtaining the SMA cylinders one could manufacture SMA washers by using a conventional machining process (turning), according to the dimensions established in Tab. (1). The *h/t* ratios were chosen so that one could make a comparison of mechanical properties of the manufactured actuators and the literature. Some of the manufactured actuators can be seen in Fig. (4).

Table 1. Geometric parameters of Ni-Ti SMA Belleville washers manufactured in this work.

Alloy (% weight)	**Washer's code**	**Outer Diameter D_o (mm)**	**Inner Diameter D_i (mm)**	**Height *h* (mm)**	**Thickness *t* (mm)**	***h/t***
55.3Ni-44.7Ti	ATNi - 1	21	10	3,2	2,1	1,5
	ATNi - 2	21	10	3,5	1,9	1,8
48Ni-38Ti-14Nb	ATNb - 3	21	10	3,2	2,1	1,5
	ATNb - 4	21	10	3,5	1,9	1,8
	ATNb - 5	21	10	3,8	1,7	2,2

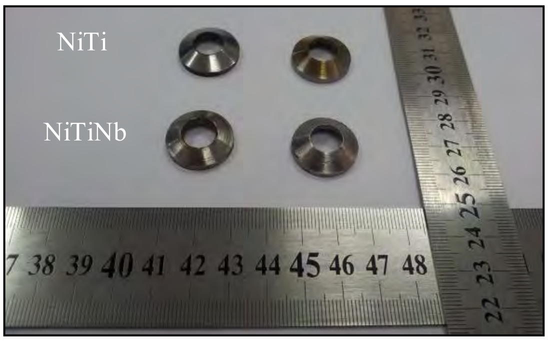

Figure 4. SMA Belleville washers.

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After the manufacturing and finishing processes, each washer was submitted to a heat treatment at a temperature of 850 °C during 30 minutes, with subsequent water quench at room temperature (~ 25 °C).

3.3 Thermal Characterization

The thermal characterization of the SMA washers was carried out by Differential Scanning Calorimetry (DSC), using the calorimeter Model Q20 from TA Instruments. In order to determine the transformation temperatures of the SMA washers and also evaluate the heat treatment's effects, thermal cycles were performed for all the specimens. The tests were carried out at the temperature range from 120 °C to -70 °C, with a heating and cooling rate of 5 °C/min.

3.4 Thermomechanical Characterization: Stiffness x Temperature

Before the test of Stiffness as a Function of Temperature (SFT), the SMA washers were subjected to a superelastic stabilization. This stabilization was carried out using a dynamic testing machine (Electropuls E10000, INSTRON). This procedure consists in carrying out 50 loading-unloading cycles at a frequency of 0.1 Hz at room temperature (~ 27 °C). It is noteworthy that at this temperature the structure is fully austenitic (Simões, 2012). The tests were set as follows: pre-load of 300N, compression upper limit of 14% (% height), compression lower limit of 4% (% height) and loading-unloading rate of 0.1 mm/min.

For tests of SFT in each washer, it was used a universal testing machine (INSTRON 5582 equipped with a heating chamber control). The set for this test can be seen in Fig. (5).

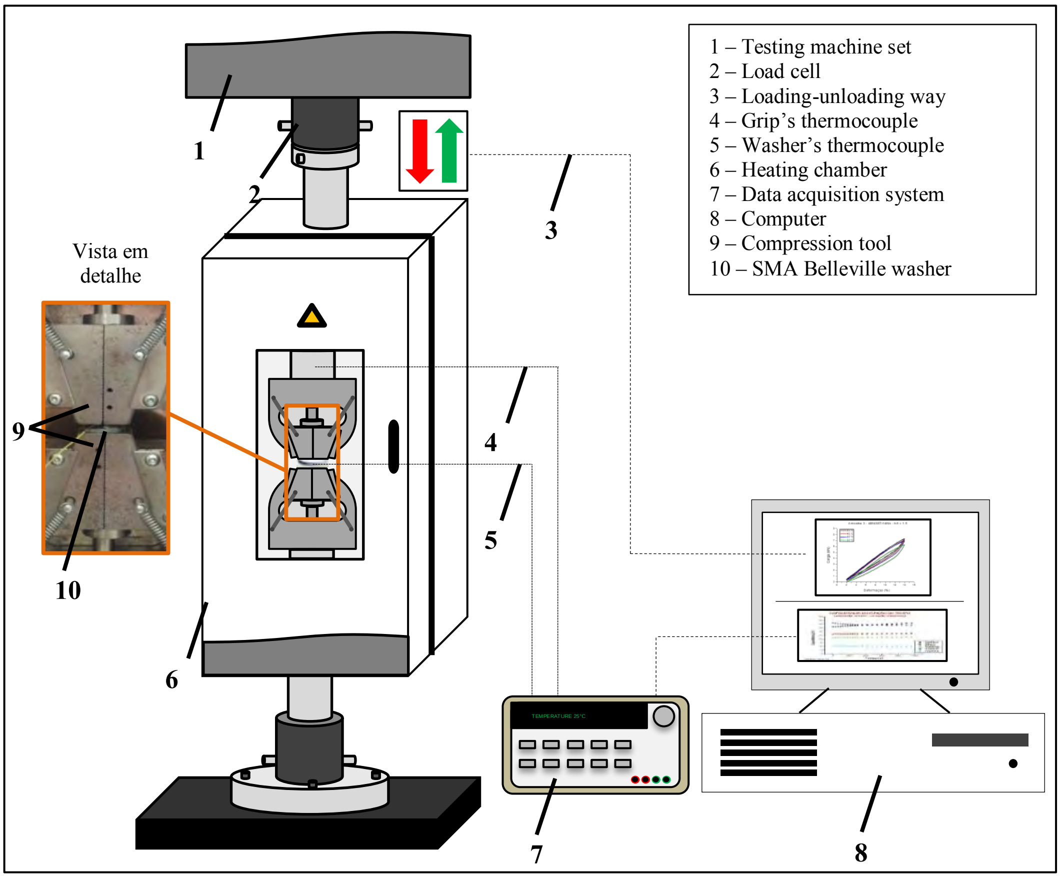

Figure 5. Experimental set up for SFT tests in the SMA washers.

These tests were carried out as follows: pre-load of 300N, compression upper limit of 14% (% height), compression lower limit of 2% (% height), loading-unloading rate 5 %/min and test temperatures of 25°C, 35°C, 45°C e 55°C.

3.5 Superelastic Behavior

To evaluate the superelastic behavior of the manufactured SMA Belleville washers one could repeat the same test procedures of Fig. (5). The main difference is that now the specimens were subjected to higher compressive deformation levels, in a range from 20% to 40% (% height) at a rate of 5 %/min. These tests were carried out at 25 °C, 35 °C and 45 °C. Afterwards, one could compare the loading behavior between the manufactured washers and classic Belleville washers whose behavior is obtained from Eq. (1).

4. RESULTS AND DISCUSSIONS

4.1 Thermal Characterization: DSC

All of the manufactured washers were subjected to DSC thermal analysis before and after heat treatment. After the test, it has been generated a graph of heat flow versus temperature. By the method of tangents applied to the transformation peaks, defined by the international standards ASTM F2004, F2005 (ASTM 2005) and F2082 (ASTM 2006), there were obtained the transformation temperatures of the SMA washers. The results are summarized in Tab. (2).

Alloy (% weight)	**Specimen/Status***		**M_f (°C)**	**M_s (°C)**	**A_s (°C)**	**A_f (°C)**
55.3Ni-44.7Ti	WNi 1	UNTR	-44.8	-2.6	-9.3	**34.5**
		HTR	-56.3	-9.0	-24.7	**26.3**
	WNi 2	UNTR	-38.4	15.2	-8.0	**46.0**
		HTR	-54.0	1.8	-15.6	**18.9**
48Ni-38Ti-14Nb	WNb 3	UNTR	--	--	--	--
		HTR	-56.4	-30.9	1.5	**22.1**
	WNb 4	UNTR	--	--	--	--
		HTR	-59.4	-44.2	-5.4	**13.1**
	WNb 5	UNTR	--	--	--	--
		HTR	-48.8	-29.1	3.6	**28.1**

*UNTR: untreated; *HTR: heat treated.

Comparing the temperature data obtained for the two SMA, it has been noticed that the heat treatments provided a shift in the phase transformation temperatures.

For WNi specimens, in general, there was a decrease in the transformation temperatures. This behavior indicates that probably the manufacturing process causes the appearance of an internal stress field which is relieved after heat treatment. As internal stresses tend to increase the transformation temperatures (De Araujo *et al.*, 2000), these were reduced by the homogenization caused by the heat treatment.

In the case of WNb specimens, before heat treatment no transformation (DSC peaks) is detected in the temperature range scanned. It is also remarkable that after heat treatment the transformation clearly appears and the Ni-Ti-Nb SMA tend to exhibit superelastic characteristics at room temperature, as indicated by the A_f values.

4.2 Thermomechanical Characterization: Stiffness x Temperature

To evaluate the increasing of stiffness as a function of temperature from curves of force versus deflection, there was defined a constant k, called stiffness.

Thus, the washer's stiffness was determined by the slope of the force-deflection curve during loading, as illustrated in Fig. 6(a). Figure 6(b) shows the results of compression tests carried out on a Ni-Ti-Nb SMA washer with h/t ratio of 1.8, as a function of temperature.

Qualitatively, it is observed from Fig. (6) that stiffness increases with temperature. The stiffness values calculated using the defined method are shown in Fig. (7).

By observing the values of stiffness as a function of temperature for Ni-Ti and Ni-Ti-Nb washers in Fig. (7), there is an approximately linear relationship between temperature and stiffness, as shown by the respective regression coefficient (R^2) for each curve fitting. This enforces the application of these washers in bolted joint where the tightening can increase with the temperature increase, avoiding bolt loosening.

Comparing Ni-Ti specimen with those obtained by Simões (2012), the stiffness values are increasing in a range of h/t ratios between 1.0 and 1.5, as shown in Fig. 7(a). The same can be said for Ni-Ti-Nb specimens in Fig. 7(b).

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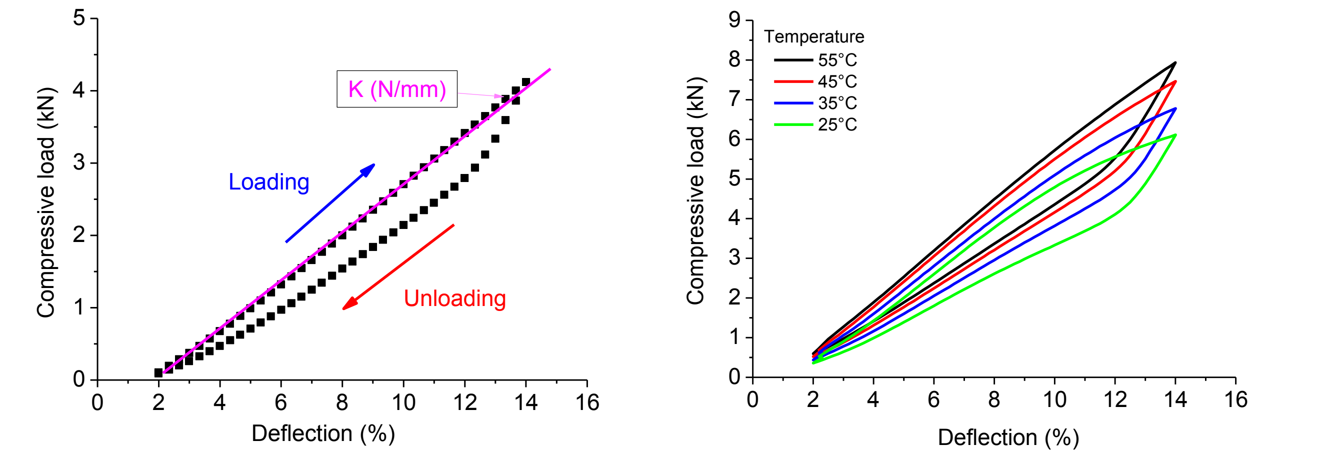
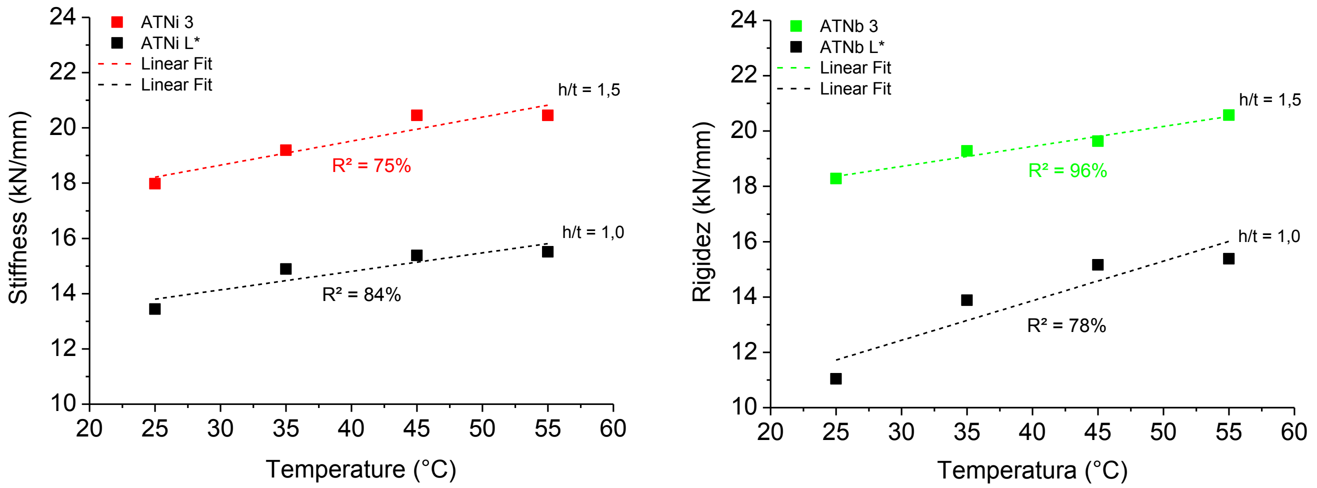


(a) (b)

Figure 6. Load – deflection of the SMA washers. (a) Calculation of a washer's stiffness during loading (Simões, 2012). (b) Force-deflection behavior of a Ni-Ti-Nb washer at different test temperatures: 25 ° C, 35 °, 45 ° and 55 ° C.



(a) (b)

Figure 7. Stiffness as a function of temperature for the manufactured SMA washers with different *h/t* ratios. (a) Ni-Ti. (b) Ni-Ti-Nb. L* values found in the literature, obtained by Simões (2012).

4.3 Superelastic Behavior

The characteristics of SE curves of the SMA washers and the results of loading-unloading tests for a 38Ti-14Nb-48Ni (wt%) Belleville washer with *h/t* of 2.2 can be seen in Fig. 8(a).

Comparing the aspect of the superelastic curves obtained for the specimen in Fig. 8(a) with those presented by Speicher (2010), with an *h/t* ratio of 2.0 and composition of 50.8Ni-49.2Ti (atomic %), shown in Fig. 8(b), it is clear that there is a qualitative similarity, especially for higher deformation levels.

Both, curves obtained in this work and those obtained by Speicher (2010), show a load peak followed by load drop forming a duck-face-shaped curve, as can be seen in Fig. (8). This is due to the fact that the *h/t* ratios are fairly similar in these two studies and, as shown in Fig. 2(b), this parameter defines the characteristic shape of the force-deflection curves. Qualitatively, from checking Fig. 8(a) for various deformation levels, the curves have the same shape, clearly demonstrating that the characteristic curve is independent of the imposed deformation, but rather the *h/t* ratio as shown in Fig. 2(b). The main difference between the curves obtained by Speicher (2010) and the ones obtained in this work is the residual deformation present at the end of unloading. This fact can be directly associated with internal defects (voids) in our material caused by the manufacturing process.

Besides varying maximum deformations in the previous experiment, another experiment was conducted to a Ni-Ti washer with *h/t* ratio of 1.5 (WNi 1), this time varying the test temperatures. For this specimen one could use a set maximum deformation of 40% (% height) and there has been obtained the load behavior at temperatures of 25, 35 and 45 °C. The results of this test are shown in Fig. (9). Based on the analysis of these results, it appears that with increasing temperatures, the compressive load increases significantly. It is also noticeable that the greater the test temperature is, the lower is the residual deformation hysteresis displayed for each specimen.

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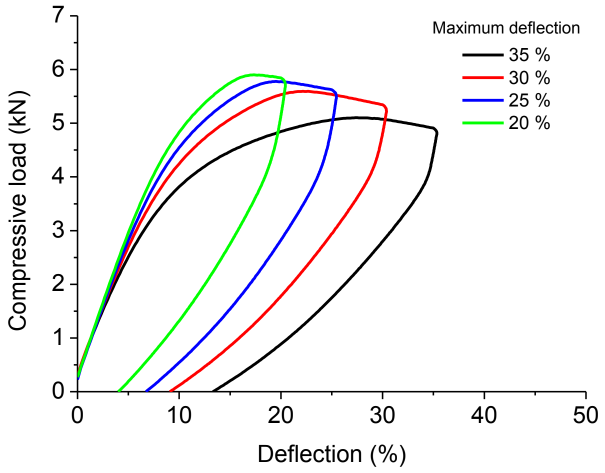
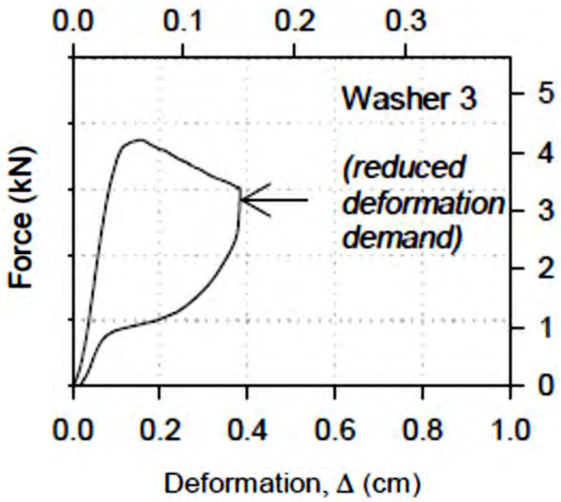


(a)



(b)

Figure 8. Superelastic behavior of Ni-Ti SMA washers. (a) Load as a function of various deflection levels of a Ni-Ti-Nb SMA Belleville washer with *h/t* = 2.2. (b) Behavior of a Ni-Ti Belleville washer with *h/t* = 2.0 subjected to a deformation of about 50%, studied by Speicher (2010).

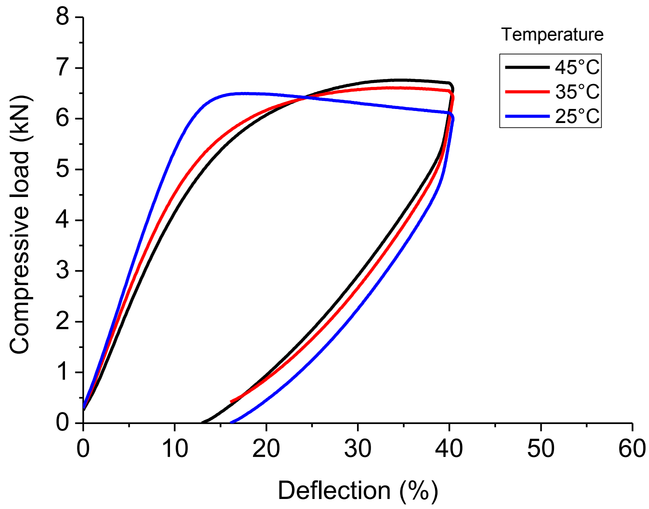


Figure 9. Superelastic behavior for the SMA washer WNb 3 with *h/t* ratio of 1.5, deformed up to 40%.

Although this washer presents a different *h/t* ratio from that used by Speicher (2010), which was about 2.0, the force-deflection curve at a temperature of 25 °C (in blue) was similar to that obtained by the second. This demonstrates that there is a transition value of *h/t* from which the SMA washer acquires a behavior similar to that shown in Fig. (9) and there are deformation and temperature levels to which this behavior starts.

4.4 Theoretical Analysis

The mechanical superelastic behavior of the SMA washers was evaluated at a room temperature (25 °C) by applying the Eq. (1), replacing the corresponding parameters for each Belleville washer. The elasticity modulus ranged from 30 to 40 GPa, as measured using a dynamic mechanical analyzer (Simoes, 2012). The result of this theoretical-experimental comparison for a Ni-Ti-Nb specimen with *h/t* of 1.5 is shown in Fig. (10).

Through the analysis of the behavior observed in the theoretical and experimental curves one can see that the theoretical curve given by Eq. (1) to classic Belleville washers follows the experimental curve until the deformation reaches approximately 11%, from which the theoretical load continues increasing and experimental tends to settle. As it is noticed in the experimental curve, the specimen elastic deformation in the austenite phase is from about 10 to 11%, from which there is formation of stress-induced martensite, and hence there is load stabilization with a slight drop tendency for the SMA specimen.

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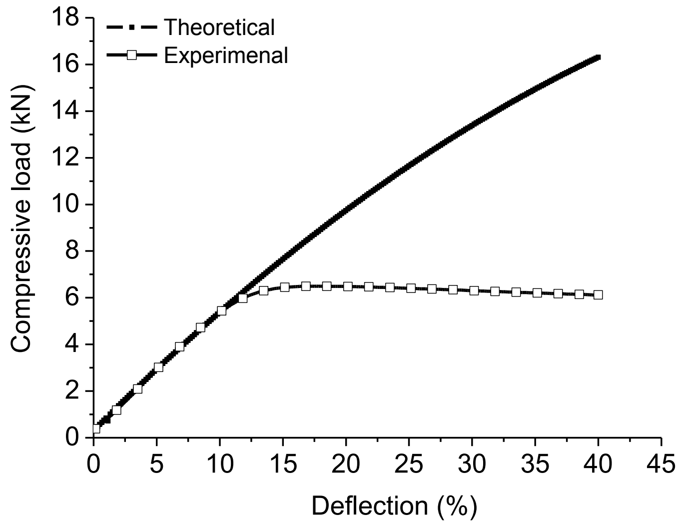

Figure 10. Theoretical and experimental comparison of force-deflection behavior at room temperature (~ 25 °C) during loading of a Ni-Ti-Nb SMA washer with *h/t* of 1.5.

5. CONCLUSIONS

Based on the results presented in this work, one could attest the relevance of application of SMA for the development of preload elements, such as Belleville washers, due to the fact that these materials present the phenomena SME and SE at relatively low temperatures.

In terms of manufacturing process, it was possible to use the PSPP technique to obtain SMA billets and then combine it with conventional machining to obtain the designed conical washer specimens, providing a reasonable surface finish thereof.

From the thermal characterization via DSC, it was possible to verify the influence of heat treatment on transformation temperatures of the studied SMA.

During the analysis of the SMA washers' stiffness as a function of temperature, it was proved that the stiffness increases with temperature. It was also found that, for deformation of over 12% (% height) there is a linear relationship between the temperature and stiffness for all the *h/t* ratios studied.

Bearing in mind the aspect of force-deflection curve of SMA Belleville washers, it was established that the curves obtained in this work show a similarity with some others found in literature, for similar *h/t* values. This fact proves that the behavior of these actuators is directly related to that ratio and thus one can select a *h/t* ratio according to the desired kind of application.

The theoretical-experimental analysis of force-deflection curves during mechanical loading of the washers showed that the theoretical behavior given by the characteristic equation for classic Belleville washers, it is valid to a value of deformation of up to 10%, from which there is a marked divergence between this behavior and the experimental. This certainly is associated with the formation of stress-induced martensite of SMA, which causes a load plateaus with a decreasing tendency.

Finally, it was possible to discern the great feasibility of manufacturing commercial SMA Belleville washers developed in this work, exploring the phenomena characteristic phenomena of SMA, both for use in high tech specific applications as well as replacing Belleville washers of traditional equipment.

6. ACKNOWLEDGEMENTS

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