23rd ABCM International Congress of Mechanical Engineering
December 6-11, 2015, Rio de Janeiro, RJ, Brazil

CHARACTERIZATION OF MECHANICAL PROPERTIES OF AN ALLOY WITH SHAPE MEMORY - NITINOL

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Abstract. *Alloys with shape memory are materials that undergo a deformation after, have the ability to return to its size and shape when subjected to an appropriate heat treatment. Nitinol is a nickel-titanium alloy (NiTi) that has this feature. In robotics, researchers seek to continually develop orthotics using actuators with features and capabilities that enable playback of the functions of the various motor systems of the human body. These systems have intended to support or detain a body segment, prevent or correct deformities and assist or recover some motor function disability, aiming at improving the quality of life of these individuals. This work aims to characterize the mechanical properties of a wire of a shape memory alloy Nitinol (NiTi) to propose the development of a mechatronic system for an orthosis prototype to simulate the function of the muscles of a human limb to perform the movements of a joint. Microhardness tests were performed in the alloy to determine its strength and metallographic tests to analyze its microstructure. In addiction, we developed a device capable of performing extension and flexion tests using electrical current to set the maximum percentage of plastic deformation supported by alloy to return to its original state.*

Keywords: *Memory shape, Nitinol, Mechanical properties*

1. INTRODUCTION

The Media usually offers information and data about personal injuries caused by different external factors. Traffic accidents, work-related injuries and physical violence (amongst others) are the main causes of debilitating injuries.

Although the use of orthotics is currently increasing, its high cost makes it impossible for all disabled patients to have access to its implementation. There are several technologies that can be used regarding the conception and construction of orthotics. Among these we must stress microelectronics, robotics, and the use of alloys that enable movement through phase transformations that occur in low temperature environments. These alloys are known as “shape memory alloys” [Kauffman, 1996]. Research on the last-mentioned technique is still at a very early stage regarding its use on the construction of orthotics. However, its implementation in other areas (including medicine), is more advanced. A highlight of the use of Nitinol wires is found in orthodontics [Silva, 2012; Vearick, 2006].

Nitinol is an equiatomic alloy of nickel and titanium (NiTi) which has excellent properties such as, being highly resistant to corrosion, biocompatibility, good ductility and high ability to absorb mechanical vibrations. Moreover, its low modulus of elasticity and its high resilience turn these alloys into good performers when great variety of movement is needed. On the other hand, when compared to other alloys with great ductility, these thermo-active alloys (as they are commercially known) offer a greater range of movements in cooling situations [Hirano, 2009].

Therefore, this article aims to highlight the Nitinol alloy (nickel-titanium) featuring its electrical and mechanical properties in order to understand its behavior regarding its implementation on the construction of orthotics.

2. METHODOLOGY AND RESULTS

This document is an experimental study which shows the feasibility of using the shape memory alloy Nitinol on the construction of an active knee orthotics.

A passive-knee orthotics was used for the mechanical design. The Nitinol alloy was added to this orthotics aiming to act as the knee's extensor and flexor muscles.

Several tests were carried out in order to characterize the highest percentage of plastic deformation the alloy can bear before it returns to its original shape [Paiva, 2004; Tai, 2011]. Some of the tests were: micro hardness tests to determine its mechanical resistance; metallographic tests to analyze its microstructure; and the development of a device able to bear flexion and extension tests by using electric current.

2.1 Micro hardness test

The samples for the tests were prepared at the Metallurgical Engineering Lab at PUC-MG University. The following procedures were needed for the samples: segments of Nitinol wires (0.6mm diameter and 10cm length) were embedded at cold temperatures, sanded and polished at aluminum paste. Samples were divided into two different groups: wires on their original state (from the factory) belonged to group A; in group B we listed those wires which have undergone a heating treatment.

The wires were put on an industrial oven at 580ºC for 10 minutes. At this temperature the alloy is at its austenite phase. Right after that, the samples were water cooled in order to cause martensitic transformations, which enables the memory shape characteristics on samples from group B. Regarding this group, all samples were on transversal and longitudinal positions in order to monitor possible variations on its hardness.

Once the samples for the tests were ready, the analysis was carried out with the Micro durometer. According to the tests' results, and compared with the hardened alloys, we can observe that the average hardness of the transversal segments of the annealed alloy (after the heating treatment) is 381HV and 310HV. The standard deviation is for both +/- 10 gf/mm^2. These figures are not surprising when dealing with annealed materials which also have their own altered mechanical properties (caused during the production process) that increase their hardness. On Table 1 we can see the results from the Vickers Hardness tests on the NiTi alloy on both states, hardened and annealed alloys.

Table 1: Data from the Vickers hardness test

	Section	Tests	Strength test	Load (grams)	Timing (seconds)	Hardness (gf/mm2)		Average (gf/mm2)		Standard deviation (gf/mm2)	
						HRC	**HV**	**HRC**	**HV**	**HRC**	**HV**
Group A – Hardened alloy	Transversal	1	980.7mN	100	10	39	391	**38.90**	**381.00**	**1.00**	**10.00**
		2				37.9	371				
		3				38.9	381				
		4	1.961 N	200		37.5	367	**37.50**	**367.00**	**0.00**	**0.00**
Group B- Annealed alloy	Longitudinal	1	980.7mN	100	10	26.8	278	**27.40**	**282.00**	**0.60**	**40**
		2				27.4	282				
		3				28	286				
		4	1.961 N	200		26.9	278	**26.60**	**278.00**	**0.00**	**00**
	Transversal	5	980.7mN	100		31.4	313	**31.00**	**310.00**	**0.69**	**50**
		6				30.2	304				
		7				31.4	313				
		8	1.961 N	200		30.1	303	**30.10**	**303.00**	**0.00**	**00**

(a) (b)

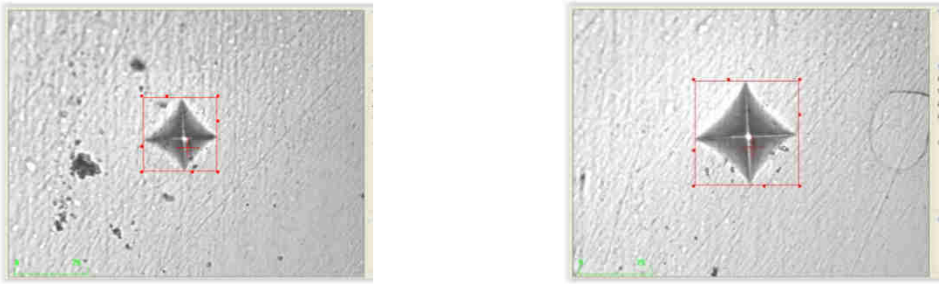


Figure 1. a) Transveral microhardness 500x (annealed alloy) 304HV 0.1/10; b) Transversal microhardness 500x (annealed alloy) 303HV 0.2/10;

2.2 Metallographic tests

The samples used on this test were prepared at the Metallographic Lab of the CETEC SENAI/FIEMG (Technological Centre of Minas Gerais). The samples followed the cold mounting process and the Nitinol alloy was presented on its annealed state after having undergone the sanding and polishing treatments. Once all treatments were finished the samples were ready for longitudinal and transversal assessment.

The following steps were followed in the process:

- There were two different types of alloys: some alloy segments on their original shape and some annealed segments (which previously remained for 10 minutes in a rectilinear position at 580 ºC and were quickly cooled). They were all cut and put into resin (cooled resin to avoid possible alternations on the atmosphere's microstructure);
- Once the samples were ready they were all sanded (using sandpaper with grits between 80 and 120) and later put on a grinding and polishing machine with diamond suspension.
- After the polishing treatment we proceed to soak the segments in a mixture of hydrofluoric (HF), nitric acid, (HN03) and acetic acid (C2H4O2) –in a proportion of 1:5:5 respectively. Each segment was immersed in the solution during 6 seconds.

Once the samples had undergone the treatments and helped with the optical microscope we proceed to analyze their microstructures. Both sections (transversal and longitudinal) were analyzed.

In Figure 2 we can observe a micrographic sample on its longitudinal section. Once sample has undergone heat treatment.

(a) (b)

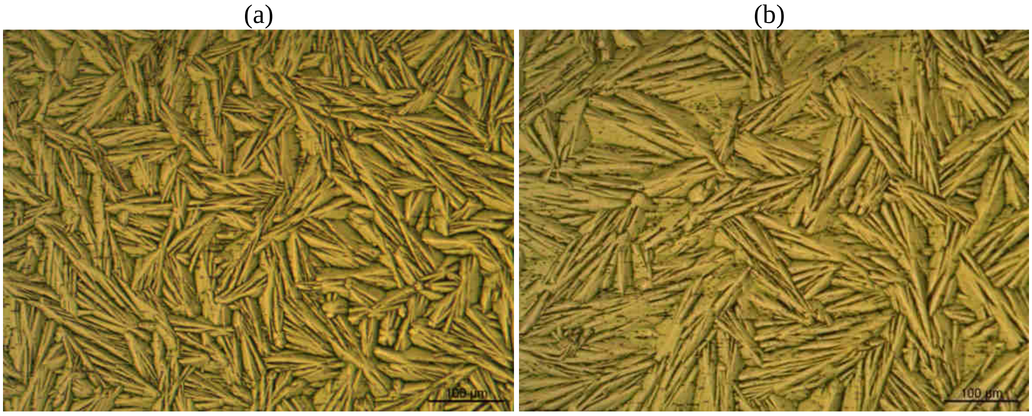


Figure 2. (a) Nitinol alloy on its original state. Soaked in a chemic solution of 1 ml HF, 5ml HNO3 and 5ml of $C_2H_4O_2$ – 1000x. (b) Austenitized Nitinol at 580ºC and cooled in water. Solution of 1 ml HF, 5ml HNO_3 and 5ml of $C_2H_4O_2$ – 50x – 282 HV.

Figure number 3 shows a micrograph of the transversal section with and without heat treatment.

(a) (b)

Figure 3. (a) NiTi alloy on its original shape. Soaked in a solution of 1 ml HF, 5ml HNO_3 and 5ml $C_2H_4O_2$ – 1000x – 381 HV and (b) Austenitized NiTi alloy at 580ºc and water cooled. Soaked in a solution of 1 ml HF, 5ml HNO3 and 5ml $C_2H_4O_2$ – 1000x – 310 HV.

Along the micrographs of the analyzed samples it can be observed a predominance of martensitic areas (in the shape of needles). That confirms that during the samples preparation (sanding and polishing) the microstructure of the materials was affected. That means there were martensitic areas created by deformation. It is an appellant consequence in this type of procedures. The morphological assessment of the martensite phase is unclear since the technique used was the mechanical polishing. The most appropriate technique is the electrolytic polishment. Unfortunately, we did not have access to the required tools and the tests were not carried out with the appropriate parameters.

2.3 Testing device for memory shape alloys

At this stage of the study the objective was developing a testing device for different types of Nitinol in order to assess the memory shape alloy properties. The testing device was used to gather data about the strength and displacement occurred when the alloy recovers its original shape when acting against an elastic element with a variable rigidity. The data resulting from the tests and based on the mechanical work carried out can be used to design the device that will simulate the knee's extensor and flexor movements. Therefore, it is essential that the thermo-dynamic behavior is tested with different formats.

On figure 4 we can observe the workbench developed to test the Nitinol. Different elements were required in this workbench: a potentiometer, an aluminum lever arm, a temperature sensor, two relay, two transistors and a standard elastic spring.

(a) (b)

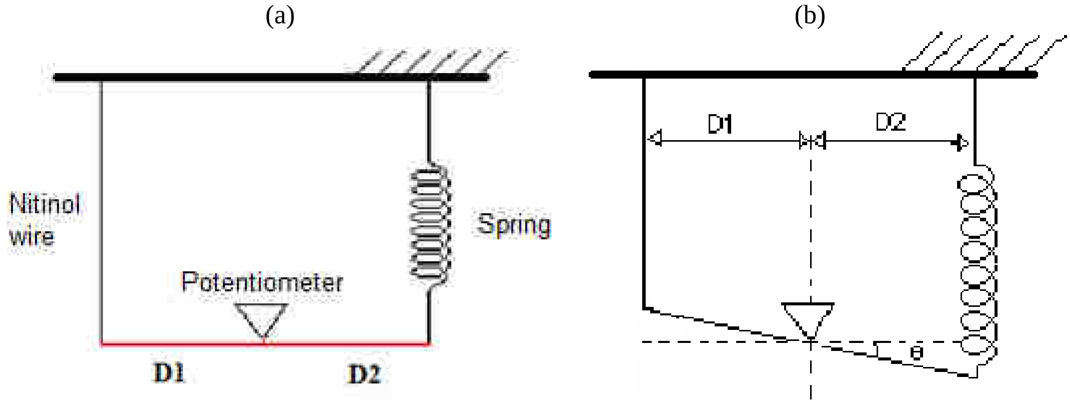


Figure 4. Workbench design

Along the work hardening the lever arm deforms the spring, which allows us to measure the strength and the timing for each temperature according to the electrical charge. The Nitinol wire is connected to an insulating plug to the structure in one of the lever's arm sides which leaves the structure de-energized when applying different electric currents through the wire.

In the Figure 4-b, the basis for the device's functioning comes from the Archimedes' Law of the Lever. The edges at the lever system are mobile so the spring always remains stretch with 0 deformations. When the wire shrinks, due to the shape memory properties, the lever arm D1 inclines so the other arm D2 also inclines and stretches the spring. The angle the lever arm creates on the horizontal axis is θ. F1 represents the force Nitinol is applying. F2 is the spring's force. In practice θ is small since the displacement caused by the shape memory is small when compared to the length of the lever arm.

Regarding the static equilibrium we know that no force is being exercised on the bar. Therefore, the result of the forces acting on it are null as well as the sum of the moments at all points of the device. Since angle θ is small, *sin (θ) = tan (θ) and cos (θ)*, then:

$$F1 = \frac{K\,\theta\,D2^2}{D1} \tag{1}$$

In order to recover its original shape, Nitinol undergoes a heating treatment. The heating follows the Joule effect; that means an electric current goes through the wire. The current applied in the process was between 1 and 3A and tensions varied from 12 to 20V. For the data gathering we used a computer and some circuits connected to an Arduino one. We developed a program with the software LabVIEW in order to codify the data and work on the machine (Figure 5-b). With the mentioned program we accessed the angle set by the potentiometer which allows the calculation of the force Nitinol is exercising (using the equation above-mentioned). The screen also offers, in real time, the force/time chart when activating the relays as well as showing the alloy's temperature data and allows for adjustments to the springs' elastic constants and the workbench. Figure 5 shows the resulting workbench:

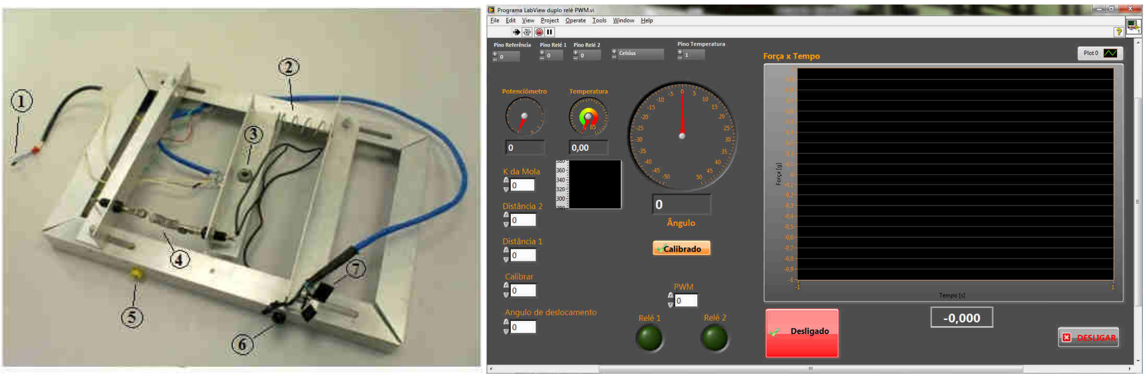


Figure 5. a) Nitinol Workbench - Spring. b) Software developed at LabVIEW.

Captions:

1. Temperature sensor;
2. Standard spring;
3. Potentiometer;
4. Nitinol wire;
5. Input voltage to the alloy;
6. Common output voltage between the plugs;
7. Data cable.

Once the device is ready tests can be carried out in different ways. For example, using several wires at the same time or controlling the heating on each of them. We could also test the wire with a different shape as for example in a spring format.

The first step was to get three standard springs and their elastic constants: k1 = 211.73 N/m; k2 = 213.26 N/m; and k3 = 661.35 N/m. In order to increase the deformation area and therefore, guaranteeing a stronger force, the Nitinol wires were shaped as springs. The different shapes for the wires were: NiTi1 = 10 mm; NiTi2 = 20 mm; and NiTi3 = 20 mm. The tests were carried out with the three different springs with a 100% deformation in all Nitinol wires. The electric current used was 2A. Table 2 shows the data obtained for each wire:

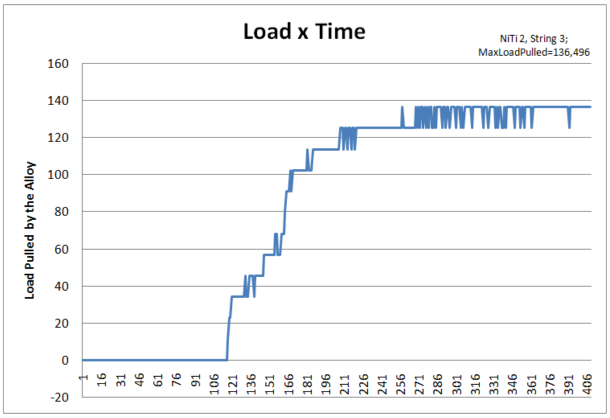
Table 2. Peak deformation forces on the alloys.

NiTi - 1	Applied force to get the highest deformation [N]	The equivalent of N in grams (gf)
Spring 1	0.406	41.35

Spring 2	0.286	29.13
Spring 3	1.281	130.62
NiTi - 2		
Spring 1	0.438	44.62
Spring 2	0.572	58.27
Spring 3	1.339	136.5
NiTi - 3		
Spring 1	0.864	88.03
Spring 2	0.929	94.68
Spring 3	1.227	125.12

Observing the results we note that the highest force applied by the Nitinol wire was 1.339. The time needed to recover its original shape was of 3 seconds. Figure 6 shown the force versus time curve resulting from the test.

Figure 6. Force versus time curve.



3. CONCLUSIONS

According to the results we can affirm that the highest force a wire of 0.6mm could apply in order to obtain a 100% deformation (in a spring shape of 20mm length) is 1.339 N. The needed time to recover its original shape was rather surprising since the wire was too fine. Once the current was activated it immediately shrank. It can be clearly observed that the wires behavior is very similar to a human's muscle behavior.

As expected from the micrographic tests, we also observed several martensitic areas on the alloy in its original shape (hardened) as well as after having undergone a heat treatment. However, a big percentage of these areas were caused by the polishing process (polishing with diamond suspension). It affected both groups of wires which made it difficult to achieve a more concrete analysis of the images.

Thanks to the hardness test we were able to measure the NiTi hardness both hardened and annealed. Using the table on the appendix we could link its hardness to its tensile strength. As we had expected, the hardness was higher on the treated wire. The average hardness was 381HV 0.1/10 on the hardened wire and 310HV 0.1/10 on the treated wire. These tests enable us to assess and confirm that the alloy was able to bear tractive forces which are the ones required on a knee's flexor and extensor movements.

The workbench developed to test the alloy (NiTi) met the expectations (find out the highest strength the alloy can apply). However we must also mention perfect precision was not achieved due to friction and inexactness on the potentiometer and the impossibility of finding a spring with high elastic constants and of an appropriate dimension to be tested in the workbench.

Nevertheless, the obtained results are good enough to know the force the wire can apply. We obtained a force of 1.310 N on a wire in spring shape of 20mm length. Due to geometry issues, the first tests carried out with the acrylic structure simulating the knee's movements did not provide the expected results. High friction was produced during the flexor movement where the steel wires where held (which means its impossibility to be used on the project).

Once the structure was ready and printed with a 3D printer, the results were as expected and the flexor and extensor movements of the knee were possible. The alloy was designed to bare a force of 1.344 N on the lever arm which goes to the leg. However, this force is still not enough for the developed system. Although the results of the wire at the tests were satisfactory, this alloy was not the most adequate for the project. Compared to other NiTi alloys we have also studied, the one used in the project was not strong enough to offer the flexor and extensor movements; and it also experiences several changes at different temperatures.

4. ACKNOWLEDGEMENTS

The authors wish to acknowledge the Department of Mechanical Engineering of PUC-Minas, the Metallography Lab of CETEC SENAI/FIEMG, CAPES and FAPEMIG for the financial support.

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