Mechanical features analysis of the Nitinol alloy under microgravity conditions

Fabio Díaz Palacios[1]*, Maritza Irahola Zalles*[2]*, Jhon Ordoñez Ingali*[3]*, Diego Rojas Arancibia*[4] *and Gabriel Rojas Silva*[5]

1 Research Professor, Mechatronics Engineering,
2 Research Engineer, Biomedical Engineering,
3 Research Engineer, Mechatronics Engineering,
4 Research Engineer, Mechatronics Engineering,
5 Research Engineer, Mechatronics Engineering.

Abstract— **This article is intended to analyze changes in mechanical qualities of the alloy known as Nitinol (Nickel and Titanium) by means of two experiments realized under microgravity conditions compared to those of earth gravity. The first experiment is to analyze the angular velocity during the reconfiguration process of the material. On the other hand, the second experiment is to analyze the elasticity coefficient and rupture force capacity of the same material. The results were obtained at the Drop Tower, property of the ZARM (Center of Applied Space Technology and Microgravity).**

Important terms – Nitinol, ASD-R, PDA-R, Microgravity,

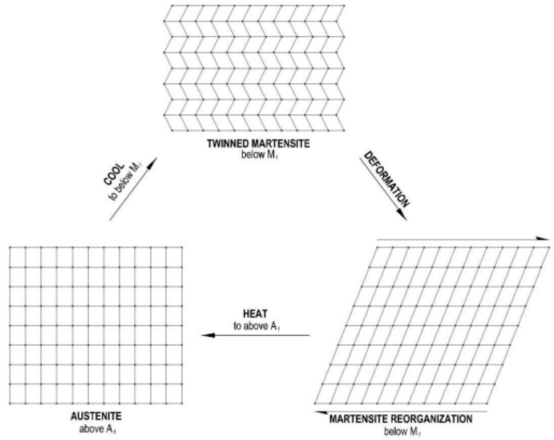
I. INTRODUCTION

In order to establish a proper context for this experiment, it is necessary to start by giving certain previous definitions:

- Nitinol. This material was created at the US Naval Ordnance Laboratory. It is a metal alloy of Nickel and Titanium with three important mechanical features: it is biocompatible, it is superelastic and it is considered as an intelligent material due to its "shape-memory".[1]
- Thermal treatment. Nitinol's "shape-memory" feature is attributed to the change of a reversible phase of an austenite microstructure into a martensite microstructure, as shown in Figure-1.[2]

Figure-1

Schematic representation of the "shape-memory"



(Source. Nitinol, the alloy with a memory: its physical metallurgy, properties, and applications, Jackson, Wagner, Wasilewski,. 2006).

The easiest and cheapest way to manipulate the transformation properties of the "shape-memory" alloys is through a "thermal treatment", where its effect over the material depends directly on exposure time and temperature, as shown in the Time-Temperature-Transformation (TTT) diagram that illustrates the Af temperature that may be reached according to exposure time and temperature during thermal treatment, Figure-2.

This work was sponsored in part by PFM S.R.L., company which provided economic and material support for the experiment. The promotion was due to Universidad Católica Boliviana "San Pablo". It is also worth to mention and be thankful to UNOOSA and ZARM organizations that made significant collaboration along the development of the whole research.

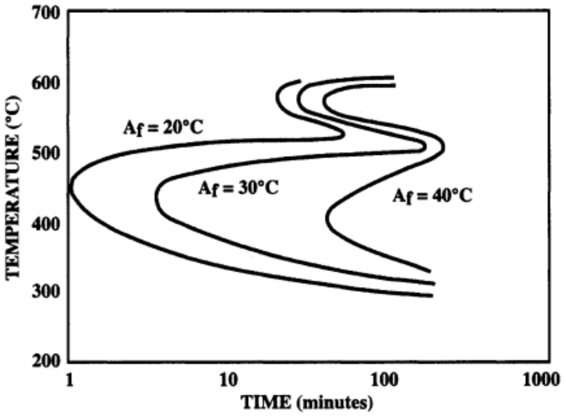
---

[1] Nitinol, the alloy with a memory: its physical metallurgy, properties, and applications

[2] Nitinol, the alloy with a memory: its physical metallurgy, properties, and applications

Figure-2

TTT Diagram for Nitinol



(Source. Nitinol, the alloy with a memory: its physical metallurgy, properties, and applications, Jackson, Wagner, Wasilewski,. 2006).

Since the Nitinol was provided by PFM S.R.L.[3] whose medical devices (PDA-R[4] and ASD-R[5]) are focused on the treatment of congenital cardiopathies and they already have a process that has been validated and approved by certificating institutions such as DEKRA[6] (CE marking), it is pretended to analyze the Nitinol under the same temperature conditions of the thermal treatment of these devices. It is known that a 500ºC thermal treatment for superelastic material is adequate and accepted, while the exposure time varies according to the Af reconfiguration temperature (austenite finish temperature) to be obtained.

II. EXPERIMENT 1

Therefore, for the following experiment, known as "Angular reconfiguration analysis of Nitinol", the Nitinol was subjected to several types of thermal treatment by testing different exposure times as well as different wire diameters, considering the aforementioned 500ºC temperature as a fixed parameter.

The main objective is to obtain an Af temperature higher enough than room temperature of the experiment (approximately between 15[ºC] and 20[ºC]) in order to avoid the wire to reconfigure before desired and in an uncontrolled way. The exposure time during the tests, ranged between 20[min] and 60[min] while the wire diameter ranged between 0.12[mm] and 1.05[mm]. The selected result for such effect is shown in the following Table-1:

Table-1

Material features and selected TT for Angular Reconfiguration Experiment

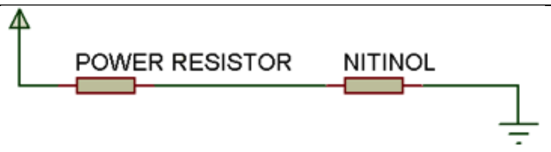
Nitinol		Thermal Treatment	
Diameter [mm]	Length [mm]	Temperature [°C]	Time [min]
0,8	150	500	40

(Source. Home-made, 2015).

Since the experiment is mainly focused on an aerospace environment, the experiment did not only looked for the motor activation of the Nitinol wire in an angled movement but also, it was intended to find the way of generating motor thermal energy by means of a method capable of being used in general void space conditions. Therefore, it was chosen the use of electricity in order to deliver the thermal energy to this material, that is, the Nitinol wire was used as a common electrical resistance inside an electronic circuit (Figure-3) where the retained energy is dissipated through heat.

Figure-3

Electrical diagram for the Angular Reconfiguration Experiment



(Source. Home-made, 2016)

Therefore, the measurements generated by the experiment, whose electrical features are illustrated in Figure-2, are shown in the following Table-2:

Table-2

Electrical features of the Angular Reconfiguration Experiment

Electrical component	Electrical features		
	Resistance [Ω]	Voltage [V]	Current [mA]
Power Resistance (Protection)	2,5	2,5	870,0
Analysis Resistance (Nitinol)	25,0	21,5	

(Source. Home-Made, 2015)

---

[3] PFM S.R.L. is a Company focused on design, production, sale and commercialization of Class III medical devices

[4] PDA-R: Patent Ductus Arteriosus

[5] ASD-R: Atrial Septum Defect

[6] DEKRA: Global provider of management services

However, when dealing with heating by electricity in order to recover the Nitinol's shape, a difficulty appears. It is that the temperature measurements cannot be made with some kind of routine temperature sensor. This makes the control over temperature a complicated issue. That is why the transformation level for this experiment is determined based on the obtained displacement and not on the temperature or the electrical features to be analyzed. Thus, the whole electrical system and the control are considered as a black box that only shows that the motor activation of the Nitinol wire is viable. However, it is possible to say that the estimated energy amount dissipated by the Nitinol wire is given by the following Equation-1 of the Joule effect:

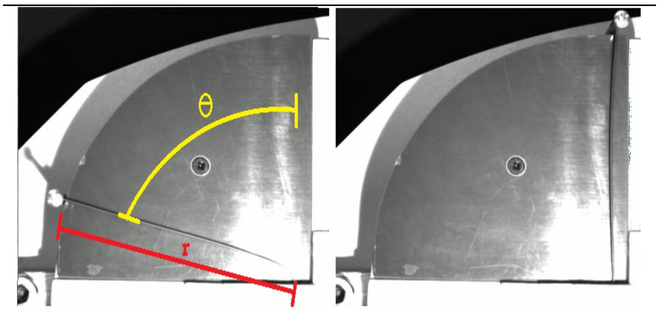
$$E = V{*}It \qquad (1)$$

As a result, there is a mainly heating energy dissipated by the Nitinol considering data in Table-2 and an exposure time of approximately 4[s]: E = 215.0 [W/s] = 0.04 [CHU/s] (Celsius heat unit per second).

That being said, and looking to determine the angular reconfiguration velocity features of the Nitinol wire, the experiment deals basically with the analysis of the electro-activated angular movement of the Nitinol wire on a platform that, given the aforementioned environmental thermal and Thermal Treatment conditions, the Nitinol wire can first be had in an angled shape and then in a straight position, as shown in Figure-4:

Figure-4

Experiment 1 pre and post electrical stimulation



(Source. Speed Camera photo inside the "Catapult Type" First Launch Capsule, 2015).

Thus, through the determination of this factor under Microgravity conditions and when comparing data with earth conditions, the following hypothesis was established: Hypothesis-H1: "Under microgravity conditions, the Nitinol wire has a higher angular velocity as for its reconfiguration capacity compared to the angular velocity that was obtained under earth gravity conditions".

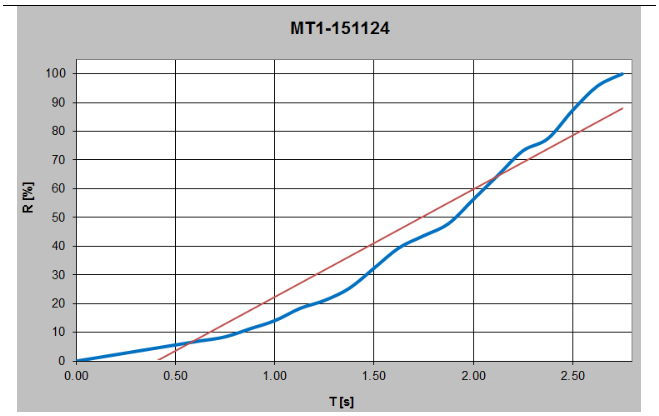
For this purpose, two simultaneous tests were done with the same electrical and Thermal Treatment characteristics applied to the Nitinol wire. Even though these tests may be redundant, it is highly important to have a large amount of data to be analyzed because despite having similar wires with the same experimental features, the result of each test is different. This is mainly due to the difficulty of applying exactly the same thermal treatment to wires, as well as providing the same pre-experiment environmental thermal features to them. Therefore, the highest amount of tests is necessary in order to be able to compare results in a more statistically credible way.

When having two equal tests, two results were obtained in earth conditions and two results in microgravity conditions for each launch. When having four launches, eight earth results and eight Microgravity results were obtained, as shown in the following Figure-5.

Figure-5

Reconfiguration vs. Experiment 1 Time graph

(Launch MT1-151124)



(Source. Home-made, 2016)

1. Angular velocity. First and with a simple visual inspection thanks to the high speed camera (500fps) located in the Launch Capsule and focusing on this experiment, the initial angular deformation was determined (Figure-4), then the angular velocity can be properly calculated, obtained from the relation of the covered angle with regard to the time used for this movement.

Based on angular Cinematic, it is established that the angular velocity turns out to be the expression of a covered angle with regard to time, where the wire's length is also useful to determine the tangential velocity of the wire at the point farthest from the movement axis. Therefore, two equations are established (Equation-2 y Equation-3):

$$W = \Theta / t \qquad (2)$$

$$V = Wr \qquad (3)$$

Where:

Θ=Covered angle

W= Angular velocity

V= Tangential velocity

t= Reconfiguration time

The fist results were obtained under earth gravity conditions so then they can be compared with Microgravity results, described in the following Table-6:

Table-6

Reconfiguration velocities results

Test code	Earth gravity	Microgravity	Reconfiguration time [s]	W [rad/s]	V [mm/s]
GT1-151124	X		3,16	0,41	29,87
			3,75	0,33	24,09
GT2-151125	X		3,01	0,29	21,18
			3,88	0,27	19,66
GT3-151126	X		4,15	0,27	19,89
			3,25	0,24	17,95
GT4-151127	X		3,60	0,28	20,71
			4,00	0,28	20,56
MT1-151124		X		0,31	22,28
			3,61	0,24	17,48
MT2-151125		X	2,25	0,43	31,62
			2,52	0,42	30,24
MT3-151126		X	2,75	0,47	34,60
			2,68	0,50	36,33
MT4-151127		X	2,18	0,30	21,97
			3,89	0,23	17,10

(Source. Home-made, 2016).

At the same time, it can be demonstrated that both, the angular and tangential velocities, are somehow higher under Microgravity conditions than those under earth gravity conditions. That is, under Microgravity conditions, the angular velocity is approximately 20% higher than that under earth gravity conditions.

However, in order to understand this change, there are two main factors affecting the Nitinol's reconfiguration velocity as well as its percentage and uniformity. The first factor to be considered is the transfer velocity of electric power. Since the transfer velocity of electric power for both Microgravity and earth gravity conditions is possible through air, it may be concluded that the energy travels at the same velocity for both cases and therefore it is not the cause for the variation in the results.

The second factor to be considered is the heat transfer by convection, that is, when a gas or a fluid contacts a solid surface at a different temperature, the resulting thermal energy exchange process is called convective heat transfer.

Thus, there are two types of processes: free or natural convection and forced convection. During free convection, there is a constant heat exchange since the solid material is resting on the gas or fluid, while during forced convection an external driving force moves a fluid over the solid at a different temperature.

In this experiment, under earth gravity conditions, the solid (Nitinol wire) is on a gas element (air) with a movement flow and therefore it is a forced convection. But, under Microgravity conditions, the landscape changes since the solid moves over the same element but with no flow whatsoever. Therefore, and from this point of view, it can be concluded that the main difference between results in Experiment 1 is related to the convective heat transfer.

Despite the amount of heat transferred by the analyzed solid (Nitinol wire) is the same under earth gravity conditions as well as under Microgravity conditions since they are both in the same transfer mean and in both cases the solid is not structurally modified, it may be concluded that the main difference is that under Microgravity conditions, the air temperature changes in time because with no air flow, the air surrounding the solid warms up and therefore the amount of transferred heat with regard to exposure time diminishes which results in a greater heat retention in the solid. It may be concluded that when increasing the retained heat, the reconfiguration velocity should tend to be higher as well, as shown in Table-6.

Finally and as can be seen in Table-6, the sample size is 8 wires per study type (2 wires per launch) and therefore a prudent statistical analysis is useful in order to answer to Hypothesis-H1. To begin, we prepare a results table (Table-7) for the determination of statistical t by means of Equation-4:

Table-7

Results table for statistical t – Experiment 1

Test code	Angular velocity W	Average W	Quasi-variance
GT1-151124	0,41	0,30	0,0026
	0,33		
GT2-151125	0,29		
	0,27		
GT3-151126	0,27		
	0,24		
GT4-151127	0,28		
	0,28		
MT1-151124	0,31	0,36	0,0110
	0,24		
MT2-151125	0,43		
	0,42		
MT3-151126	0,47		
	0,50		
MT4-151127	0,30		
	0,23		

(Source. Home-made, 2016)

$$t = \frac{\underline{X}-\underline{Y}}{\sqrt{\frac{(n-1)\hat{S}_1^2+(m-1)\hat{S}_2^2}{n+m-2}}\sqrt{\frac{1}{n}+\frac{1}{m}}} \qquad (4)$$

Where:

$\underline{X}, \underline{Y}$ = Samples average or mean
$\hat{S}_1^2, \hat{S}_2^2$ = Samples quasi-variances
n, m = Size of the 2 samples
t = Statistical student t obtained

In this respect, the t_{obtained} reaches this calculated value:

$$t = \frac{0{,}30 - 0{,}36}{\sqrt{\frac{(8-1)0{,}0026 + (8-1)0{,}0110}{8+8-2}}\sqrt{\frac{1}{8}+\frac{1}{8}}}$$

$$t = 0{,}441$$

Finally, the critical T student is found with the T student distribution table under the following parameters:

$$95\%\ \text{confidence} = \frac{a}{2} = \frac{0.05}{2} = 0.025$$
$$\text{Degrees of freedom} = n + m - 2 = 8 + 8 - 2 = 14$$

Therefore, the critical T student according to its corresponding distribution tables is:

$$t_{\text{critical}} = 2{,}145$$

In this respect, it can be observed that the $t_{\text{obtained}} << t_{\text{critical}}$ and therefore part of Hypothesis-H1 is accepted when corroborating that statistically speaking the Nitinol wire has better angular velocity characteristics under Microgravity conditions compared to the angular velocity under earth gravity conditions.

III. EXPERIMENT 2

Considering definitions made in Experiment 1 and thermal features of Thermal Treatment, this experiment must begin by stating that the verification of the wire was chosen with the largest diameter used within the range of medical devices produced by PFM S.R.L. as described before. Therefore, the same thermal treatment was used and applied to the device itself. That is, a 0.17 [mm] diameter wire was selected, corresponding to the largest ASD-R device (Center 30) within the range of devices produced by PFM S.R.L., whose thermal treatment is divided into three different stages according to exposure time: first one for 20 [sec], second one for 20[sec] as well and finally a third one for 2[min], all of them under a 500[°C] temperature. Therefore, the selected material is shown in the following Table-8:

Table-8
Material characteristics and selected TT for Elasticity and Rupture Experiment

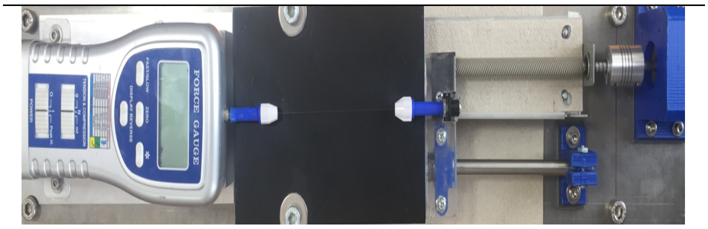
Nitinol		Thermal treatment		Samples amount
Diameter [mm]	Length [mm]	Temperature [°C]	Time [s]	
0,17	150	500	20, 20, 120	8

(Source. Home-made, 2015).

In search of determining the elasticity and rupture characteristics of the Nitinol wire, the experiment is basically the determination of elastic coefficient and rupture force of Nitinol wire under Micrograviry conditions and then the comparison to data under earth conditions. In the first place, we pose Hypothesis-H2: "The Nitinol wire under Microgravity conditions has elongation and rupture characteristics similar to those obtained under earth gravity conditions".

The traction test is divided mainly into two analysis stages: Elongation and Rupture Force. However, it should be mentioned that there is a preparation for the test in both cases, consisting in holding a piece of 150[mm] Nitinol wire (test tube) between both precision crocodile clamps and setting a 70[mm] distance between them (Xi). One clamp is fixed to the force measurement instrument (Dynamometer. Internal code: 1047-DN2) while the other clamp is fixed to a linear movement mechanism that is controlled by a step motor (1/4 step limited to 3,3[A] current), as shown in Figure-6:

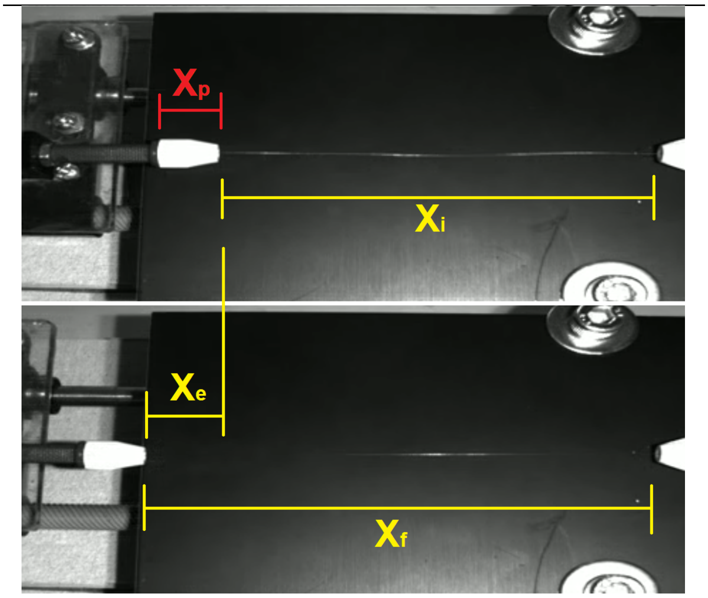
Figure-6
Preparation of Experiment 2



(Source. Photograph of Experiment 2, 2015).

2. Elongation. First and with a simple visual inspection thanks to the high speed camera (500fps) located in the Launch Capsule and focusing this experiment, the maximum elongation distance before rupture (Xf) was determined, so the elongation distance can be properly calculated (Xe) as shown in Figure-4, and it must be say that due to dimensional precision terms, the aforementioned distance of one of the precision crocodile clamps was chosen as a pattern measure (Xp = 13,5[mm]).

Figure-7
Initial and final photos of Experiment 2 inside the Launch Capsule (Launch MT1-151124)



(Source. Photograph of the experiment by Speed Camera inside the First Launch Capsule “Catapult Type”, 2015).

Based on Hooke’s elasticity law, when talking of a longitudinal stretching, it is established that the unitary stretching or elongation coefficient of the Nitinol wire itself treated as an elastic material, can be calculated through Equation-5, where δ is the stretching or elastic deformation (Xe = Xf - Xi) and L is the original length (Xi) of the wire:

$$\varepsilon = \delta/L \qquad (6)$$

The fist results were obtained under earth gravity conditions so then they can be compared with Microgravity results, described in the following Table-9:

Table-9
Hooke’s elongation coefficient results

Test code	Earth gravity	Microgravity	δ [mm]	L [mm]	Elongation coefficient
GT1-151124	X		17,91	91,24	0,20
GT2-151125	X		17,26	93,84	0,18
GT3-151126	X		15,99	95,98	0,17
GT4-151127	X		16,93	95,64	0,18
MT1-151124		X	15,75	96,08	0,16
MT2-151125		X	17,43	91,80	0,19
MT3-151126		X	12,19	91,45	0,13
MT4-151127		X	15,47	90,45	0,17

(Source. Home-made, 2016).

That is, under Microgravity conditions, the elongation coefficient is approximately 10% lower than that under earth gravity conditions.

However, as can be seen in Table-3, the sample size is 4 wires per study type (1 wire per launch) and therefore a prudent statistical analysis is useful in order to answer to Hypothesis-H2. To begin, a results table was prepared (Table-10) for the determination of statistical t by means of Equation-4:

Table-10
Results table for statistical t – Experiment 2

Test code	Elongation coefficient ε	Average ε	Quasi-variance
GT1-151124	0,20	0,18	0,00016
GT2-151125	0,18		
GT3-151126	0,17		
GT4-151127	0,18		
MT1-151124	0,16	0,16	0,00055
MT2-151125	0,19		
MT3-151126	0,13		
MT4-151127	0,17		

(Source. Home-made, 2016)

In this respect, the t_{obtained} reaches this calculated value:

$$t = \frac{0{,}18 - 0{,}16}{\sqrt{\frac{(4-1)0{,}00015 + (4-1)0{,}00055}{4+4-2}}\sqrt{\frac{1}{4}+\frac{1}{4}}}$$

$$t = 1{,}512$$

Finally, the critical T student is found with the T student distribution table under the following parameters:

$$95\%\ \text{confidence} = \frac{a}{2} = \frac{0.05}{2} = 0.025$$

$$\text{Degrees of freedom} = n + m - 2 = 4 + 4 - 2 = 6$$

Therefore, the critical T student according to its corresponding distribution tables is:

$$t_{\text{critical}} = 2{,}4469$$

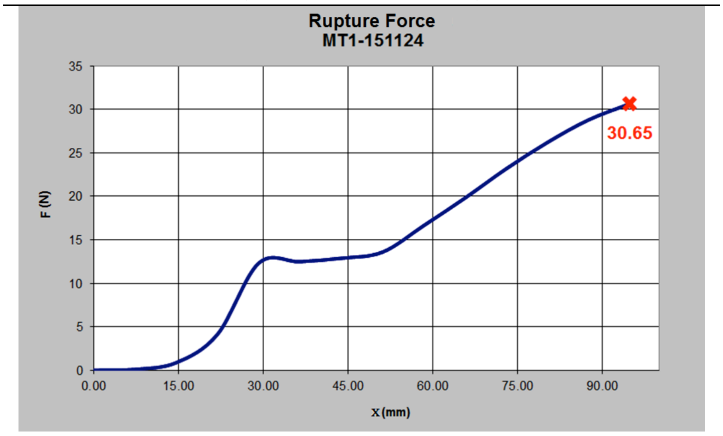
In this respect, it can be observed that the $t_{obtained} < t_{critical}$ and therefore part of Hypothesis-H2 is accepted when corroborating that statistically speaking the Nitinol wire has the same elongation characteristics under Microgravity conditions compared to those under earth gravity conditions.

3. Rupture force. This is the second part, the analysis of data stored by the Dynamometer in a type SD card through a design and programming interface for data storage at a 1 data per 0.2[sec] rate. It can be observed, in a graphical way, the rupture force of Nitinol wire during launch under Microgravity conditions as it is shown in Figure-8:

Figure-8

Force vs Distance Graph from Experiment 2

(Launch MT1-151124)



(Source. Home-made, 2016).

Results obtained during aforementioned tests are described in the following Table-11:

Table-11

Rupture Force results table

Test code	Earth gravity	Microgravity	Rupture force [N]	Average [N]	Quasi-variance
GT1-151124	X		27,48	29,24	2,86
GT2-151125	X		28,14		
GT3-151126	X		30,33		
GT4-151127	X		31,00		
MT1-151124		X	30,65	28,57	2,65
MT2-151125		X	28,37		
MT3-151126		X	26,68		
MT4-151127		X	28,58		

(Source. Home-made, 2016).

That is, under Microgravity conditions, the elongation coefficient is 2% lower than the one obtained under Earth Gravity conditions.

At the same time, a statistical comparison analysis between samples was done, where since the statistical parameters remain the same that in the elongation analysis, we maintain a critical student t of 2.4469, where the obtained t is calculated through Equation-4:

$$t = \frac{29{,}24 - 28{,}57}{\sqrt{\frac{(4-1)2{,}86 + (4-1)2{,}65}{4+4-2}}\sqrt{\frac{1}{4}+\frac{1}{4}}}$$

$$t = 0{,}571$$

In this respect, it may be observed that the $t_{obtained} << t_{critical}$ and therefore the remaining part of Hypothesis-H2 is accepted when corroborating that statistically speaking the Nitinol wire has the same rupture force characteristics under Microgravity conditions compared to those under earth gravity conditions. Therefore, the whole Hypothesis-H2 is accepted.

IV. CONCLUSIONS

Through every tests performed, it was established that, regarding Experiment 1 (Angular reconfiguration analysis of Nitinol) it was not only probed that the use of this material is viable under space conditions when being electro-stimulated by simple electricity, but also this material has a higher energy performance under Microgravity conditions compared to Earth Gravity conditions, due mainly to its heat retention quality by means of forced convection in absence of gravity, which generates a response capacity in terms of angular velocity of approximately 70% more in Microgravity.

As for Experiment 2 (Elasticity and rupture analysis of Nitinol), it could be observed that for both elongation and rupture force characteristics, the Nitinol has a mechanical performance slightly higher in earth gravity conditions in contrast to Microgravity conditions (10% in Elongation Coefficient and 2% in Rupture Force). The reason for this slight difference may be the result of several analyzed factors, being the most important, the room temperature. For earth gravity tests it was approximately 13[°C] +/- 2[°C] since there was a monitoring and environmental control system in order to avoid the tests linked to Nitinol reconfiguration (Experiment 1) to be disturbed by room temperature. However, due to the launch characteristics, the preparation of the Capsule took around 2 or 3 hours from which we can deduce that its internal temperature gradually increased, leading to reduce the elastic coefficient and rupture force capacity of Nitinol. This was mentioned before in the theoretical framework, the Nitinol is directly related to the room temperature where it interacts.

Therefore, not only Hypothesis-1 and Hypothesis-2 were positively answered but also, they make room for research in the use of this material for applications in robotics and aerospace technology as an ideal mechanical actuator in small dimensions, since the physical qualities of the material are not lost in Microgravity conditions (elongation capacity and rupture force). Also, its shape-memory characteristic is considerably increased.

REFERENCES

[1] S. Domingo, S. Puértolas , F. Sánchez, L. Gracia-Villa y J.A. Puértolas. (2002). Aplicación de la Superelasticidad, del Niti a los Stents: Consideraciones Termomecánicas. VIII Congreso Nacional de Propiedades Mecánicas de Sólidos, Gandia

[2] Curtis M. Jackson, Herbert Julius Wagner, Roman Jerzy Wasilewski. (2006). Nitinol, the alloy with a memory: its physical metallurgy, properties, and applications. United States. National Aeronautics and Space Administration

[3] Fenómenos de transporte – Transferecia de calor. 2016, Sitio web: http://www.unet.edu.ve/~fenomeno/F_DE_T-165.htm

[4] Solid Works – Coeficiente de transferencia de calor por convección. 2016, Sitio web: http://help.solidworks.com/2011/spanish/SolidWorks/cworks/LegacyHelp/Simulation/AnalysisBackground/ThermalAnalysis/Convection_Topics/Convection_Heat_Coefficient.htm