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ANALYSIS OF THE EFFECT OF HEAT TREATMENTS IN THE MANUFACTURING PROCESS OF SHAPE MEMORY ALLOY SPRINGS

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Abstract. *Shape memory alloys (SMAs) possess the capability to generate large displacements and forces when imposed to thermomechanical loadings, this outcome is due to a phase transformation process during the loadings (martensite to austenite and the other way around). Because this effect SMAs has been used in numerous applications. In those functions, several use SMA springs as vibration absorbers or as actuators. The spring manufacture process can lead to changes on the thermomechanical properties of the material, making it critical to understand how the heat treatments that create the springs affect its operation. Depending on the properties the SMA can have mainly two different behaviors: Shape memory effect, when is needed to heat the material to recover the deformation; or Pseudoelastic, when the deformation caused by the mechanical loading recovers as soon as the loading ceases. This study aims to experimentally establish how the heat treatments influence on the properties of SMA. Through a set of different heat treatments, the characterization will be carried out by quasi-static and DSC tests, using groups of the wire as received, wire after the heat treatment and the springs itself. From the DSC tests, will be detected the phase transformation temperatures and evaluated which phase is stable at room temperature. From the quasi-static tests, will be measured the transformation tensions and observed the behavior at room temperature of the SMA. Finally, the results will be analyzed and determined the optimal heat treatments for each desired behavior.*

Keywords: *Shape Memory Alloy, Spring, Heat Treatment*

1. INTRODUCTION

Shape memory alloys (SMAs) are materials capable of undergoing a solid-to-solid reversible phase transformation due to mechanical and/or thermal loadings (Lester, *et al*., 2015; Savi, *et al*., 2015). This property has been known since the 1930s, but the interest only came when researchers at the US Naval Ordinance Laboratory (NOL) detected the effect in NiTi alloys (Savi, et al., 2015), which are able to large strains recovery, up to 10%(Hora, *et al*., 2014; Lester, *et al*., 2015; Miller and Lagoudas, 2001). One of the several applications of NiTi alloys is in the form of actuators, they can be used in the form of wire, despite de limited stroke it can produce high recovery forces. In a trade-off between stroke and force, the wires can be shaped as springs (Follador, et al., 2012; Frost, *et al.*, 2016). Nitinol springs are made by constraining the wire in the spring shape and put through a heat treatment.

The thermomechanical response of SMAs suffers significant change due to material processing (Miller and Lagoudas, 2001; Heidari, *et al.* 2016). So, to better understand how your spring actuator is going to behave, is crucial to comprehend the effects of the spring manufacturing process has on the behaviour of the alloy.

2. EXPERIMENTAL PROCEDURE

For the experiments were used a commercial nitinol wire with the diameter of 0.9mm. The company provided a detailed chemical composition, as shown in Tab. 1. From the table was calculated the alloy to be Ni 50.90 at. %-Ti. To verify the data, a specimen of the wire was solution treated at 1223 K for 3.6 ks then was made a DSC analysis to verify the M_s temperature and compare with the results found by Frenzel *et al* (2010), as seen on Fig. 1.

From this analysis was determined that the alloy is Ni 50.88 at. %-Ti, proving that the composition presented by the manufacture is fairly accurate.

Table 1. Wire chemical composition

Ni	Cr	Cu	H	Fe	Nb	C	Co	N	O	Ti
55.97	0.0030	0.0007	<0.0050	0.019	0.0001	0.0261	0.0001	0.0015	0.0258	Bal.

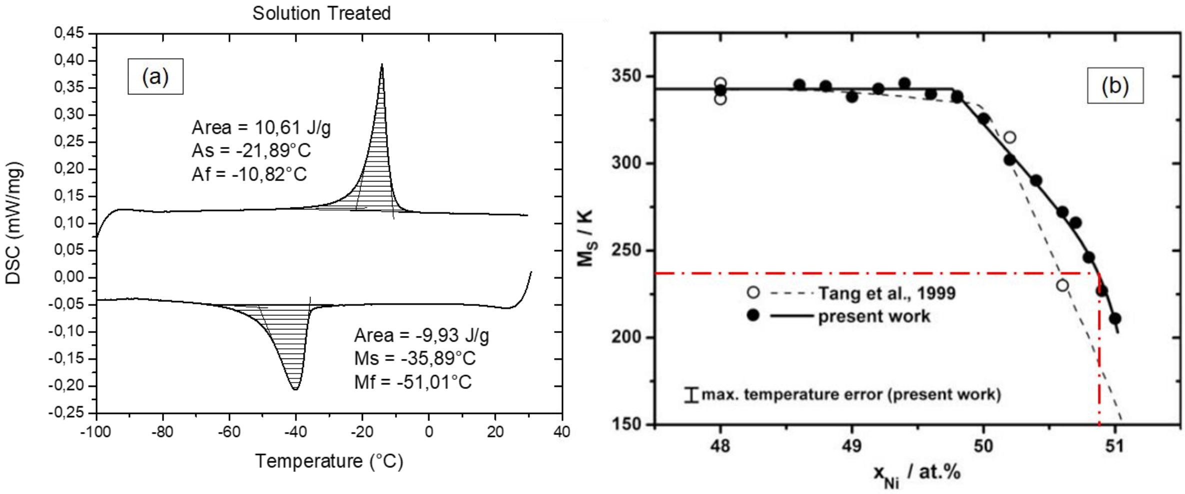


Figure 1. (a) DSC results on the solution treated specimen and (b) comparison with the results found by Frenzel *et al.* (2010), in red is the specimen of this study.

To study the effects of the stress applied to the wire during the manufacturing process of nitinol springs, a series of heat treatment was made on springs with two different spring index and a stress-free wire. The spring indexes are 6.6 and 13.2 (rolled on M6 and M12 bolts).

The effects of the heat treatment are going to be analyzed by performing the treatments with different temperatures and two cooling methods, according to Tab. 2, keeping the specimen for 1.8k seconds on the indicated temperature, followed by the according cooling method.

Table 2. Heat Treatments

Temperature	465°C	Cooling Method	Water
	540°C		Air

For each heat treatment will be made four springs and two stress-free wires, Fig. 2. With the two as received wires, there will be a total of 16 springs and 10 wires. From the 26 specimens will be removed 2 samples each for a DSC analysis, in which will be determined the transition temperatures and the possibility of the Phase-R appearance, using a DSC 200 F3 Netzsch (Fig. 3a), doing cycles at a rate of 10K/min. Th

Figure 2. Springs and wires from the 465°C water-cooled heat treatment.

Later, the specimens will be put through mechanical loading cycles with controlled temperature, using a INSTRON 5982 with a thermal chamber (Fig. 3b). There will be done 10 cycles, then the specimen will be heated above the A_f temperature. Next they will be put through a couple more cycles with the temperature fixed at 23°C (room temperature), this last cycles will be used to analyze the specimen behavior. On the springs will be used eight active coils.

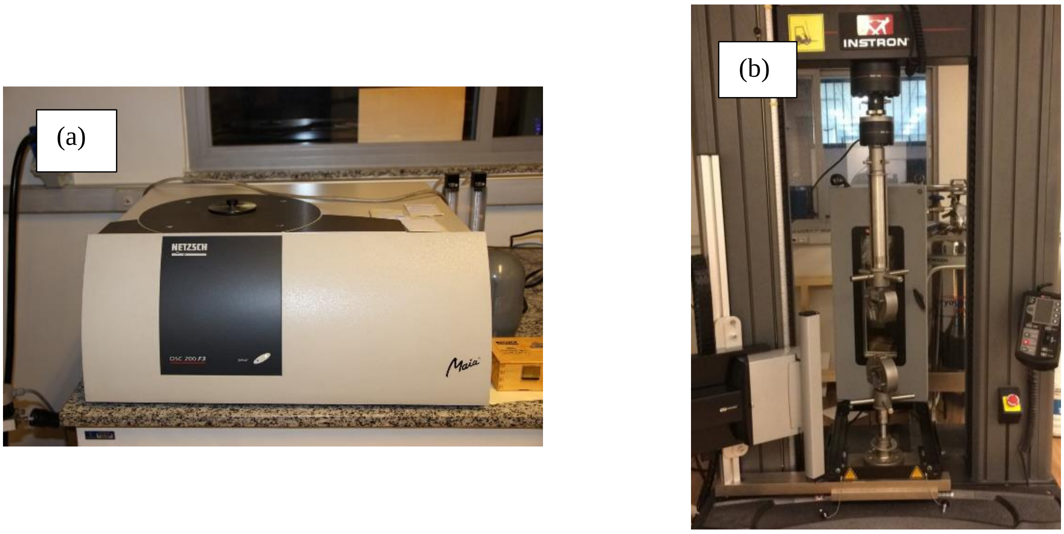


Figure 3. (a) NETZSCH DSC 200 F3. (b) INSTRON 5982

3. RESULTS AND DISCUSSION

From the DSC results will be determined the martensitic start and finish temperatures (M_s and M_f), which are the temperatures that begins and ends the transformation from austenite to martensite during the cooling process, A_s and A_f are the temperatures on the heating process, showing when starts and finish the transformation from austenite to martensite (Laplanche, et al., 2017). It is also possible to appear an intermediary R-phase, that are determined by another peak on the DSC results and gives the R_s and R_f temperatures, start and finish respectively Comparing the results with the as received wire, the influence of the heat treatments on the transition temperatures can be determined. Figure 4 shows the DSC results of the wire, as received and the different annealing methods. When compared the results of the as received wire, Fig. 4a with the solution treated, Fig. 4a it shows a reduced latent heat due to the thermomechanical process made by the manufacture

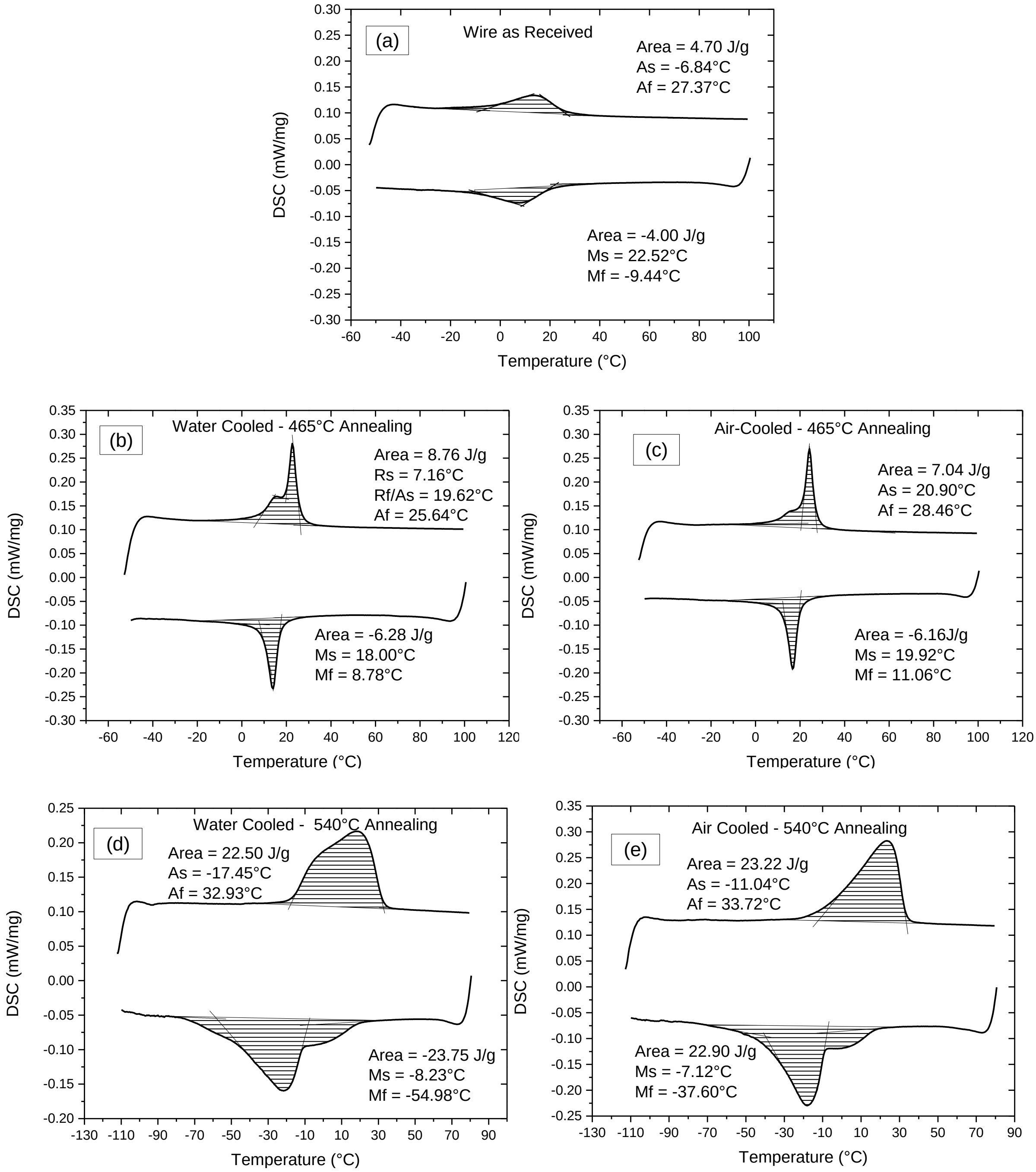
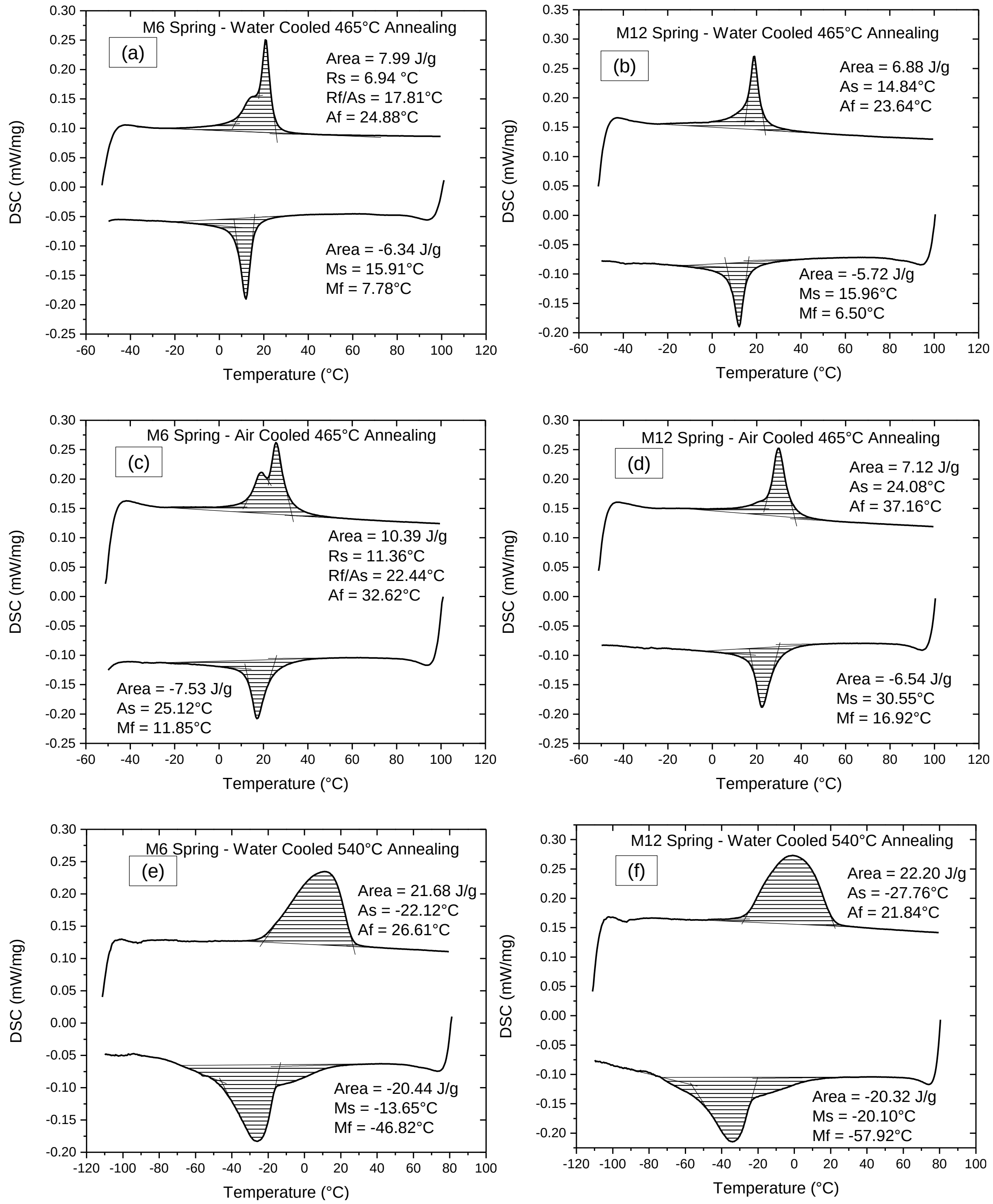


Figure 4. DSC results of the wires: (a) as received, (b) 465°C water cooled annealing, (c) 465°C air cooled annealing, (d) 540°C water cooled annealing, (e) 540°C air cooled annealing

Comparing the results, is seen that when is increased the annealing temperatures the latent heat also increases. This occurs because the as received wire has an internal stress that inhibit the transformation of fraction of martensite into austenite (Lagoudas and MIller, 2001). Also the annealing process precipitates Ti_3Ni_4 which causes the martensitic transformation to the R-phase to occur, causing the second transformation peak seen on the DSC results, and this additional transformation helps to further increase the transformation heat. The two peaks are close in some heat treatments, such as the water cooled – 540°C annealing, this is probably due to the diminished annealing time, only 1.8ks. It can also be observed that with an increased cooling time the peaks associated with the R-phase tends to decrease and in some cases disappear, this is presumably caused because a higher cooling time allows the precipitate to grown and is already established that for the R-phase transformation to happen is needed a high density and fine precipitate.

At first, looking at all of the DSC results on the springs, Fig. 5, there is few differences comparing to the wires, only an increase on the transformation temperatures for a slower cooling method and the appearance of the R-phase transformation on the M6 specimen on the reverse transformation in lower temperature annealing. When compared with the wire results the water cooled specimen for the lower temperature annealing kept the same transformation temperatures, for the higher temperature annealing all the results shown a decrease on the temperatures. Is also important to notice that, especially for the air cooled higher temperature annealing there is a widened on the forward transformation temperatures, M_f shifts to lower temperatures and M_s to higher. Causing the transformation to occur in a greater range of temperature.



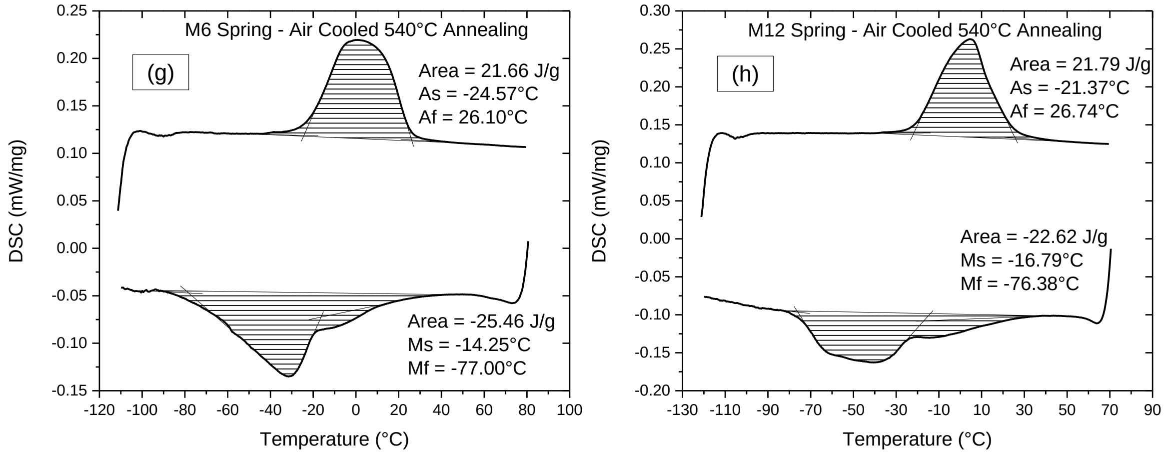
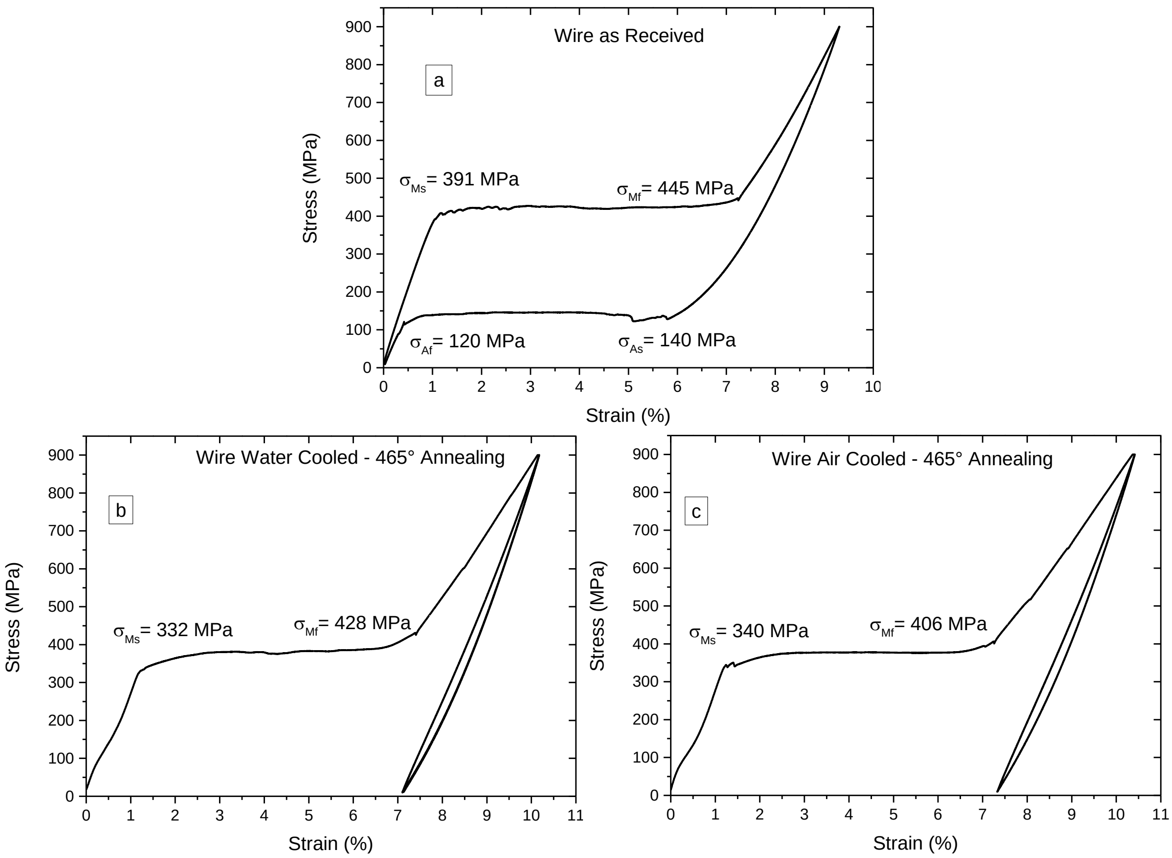


Figure 5. DSC results of the springs: (a) M6 spring, water cooled, 465°C annealing; (b) M12 spring, water cooled, 465°C annealing; (c) M6 spring, air cooled, 465°C annealing; (d) M12 spring, air cooled, 465°C annealing; (e) M6 spring, water cooled, 540°C annealing; (f) M12 spring, water cooled, 540°C annealing; (g) M6 spring, air cooled, 540°C annealing; (h) M12 spring, air cooled, 540°C annealing

Although these differences are not so large, reaching 10°C in some cases, they are significant because the transformation temperatures A_f and M_s are close to room temperature, which can change the macroscopic effect. This will be seen analysing the tensile tests.

From the Tensile cycle tests will be seen the change in the behaviour caused by the annealing. On the wires, the transition stresses will be evaluated, and the springs will show the energy removed from the system by measuring the hysteresis area and verifying the change in the constant. All the tests will be executed at room temperature (23°C). Because the two different spring index



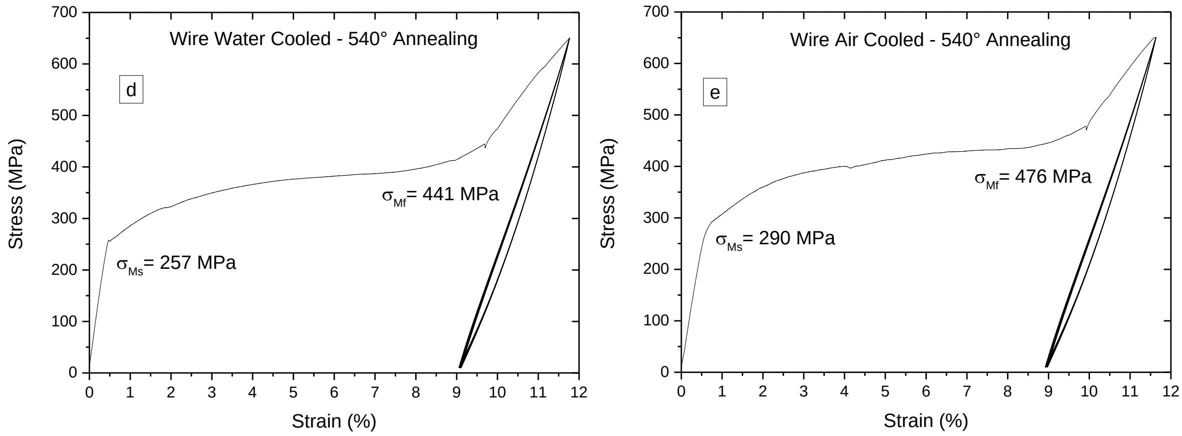


Figure 6 – Last cycles on the wires: (a) as received; (b) 465°C water cooled annealing; (c) 465°C air cooled annealing; (d) 540°C water cooled annealing; (e) 540°C air cooled annealing

Figure 6a shows the last cycle on the tensile test on the wire as received, with the transition stress shown, it has a pseudoelastic effect, as expected when looking at the DSC result (Fig.4a), despite the test temperature is slight bellow A_f still is above M_s.

The results of the wires after the heat treatment are shown in Fig. 6b through 6e, all specimen had shape memory effect, this is due to the fact that even though the test temperature are above M_s, they are below the austenitic transformation peak. Also the annealing process generates inner stresses that induces the transformation stresses to drop, with exception being the higher temperature heat treatment that drops even more σ_{Ms}, but increases significantly the transformation hardening, causing σ_{Mf} to increase. Other thing to notice is that annealing increases the strain on the wires, the wire as received reaches 9% strain with 900 MPa, the annealed specimen reached 10% or higher strain, the higher annealing affected the wire in a manner that it reaches 11% strain in only 650 MPa, causing the maximum stress to drop about 28%

On the spring tensile tests, to reach similar stress levels, will be used different extensions for the M6 and M12. The M6 springs will be extended 30mm and the M12 springs 50mm, both reaching around 250 MPa as maximum shear stress (450MPa *von Mises* stress). The maximum extension for each spring is shown on Fig.7 along with the springs without any load.

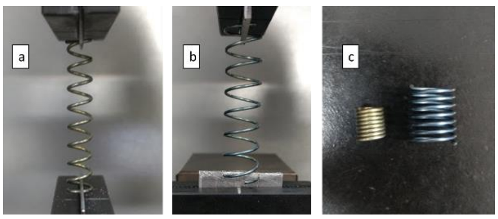
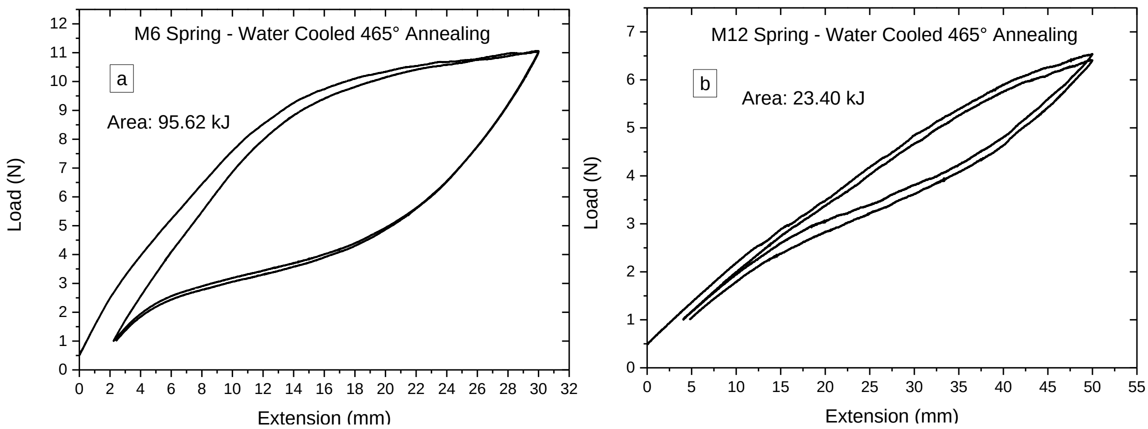


Figure 7. Maximum extension on (a) M6 springs, (b) M12 springs and (c) springs without load



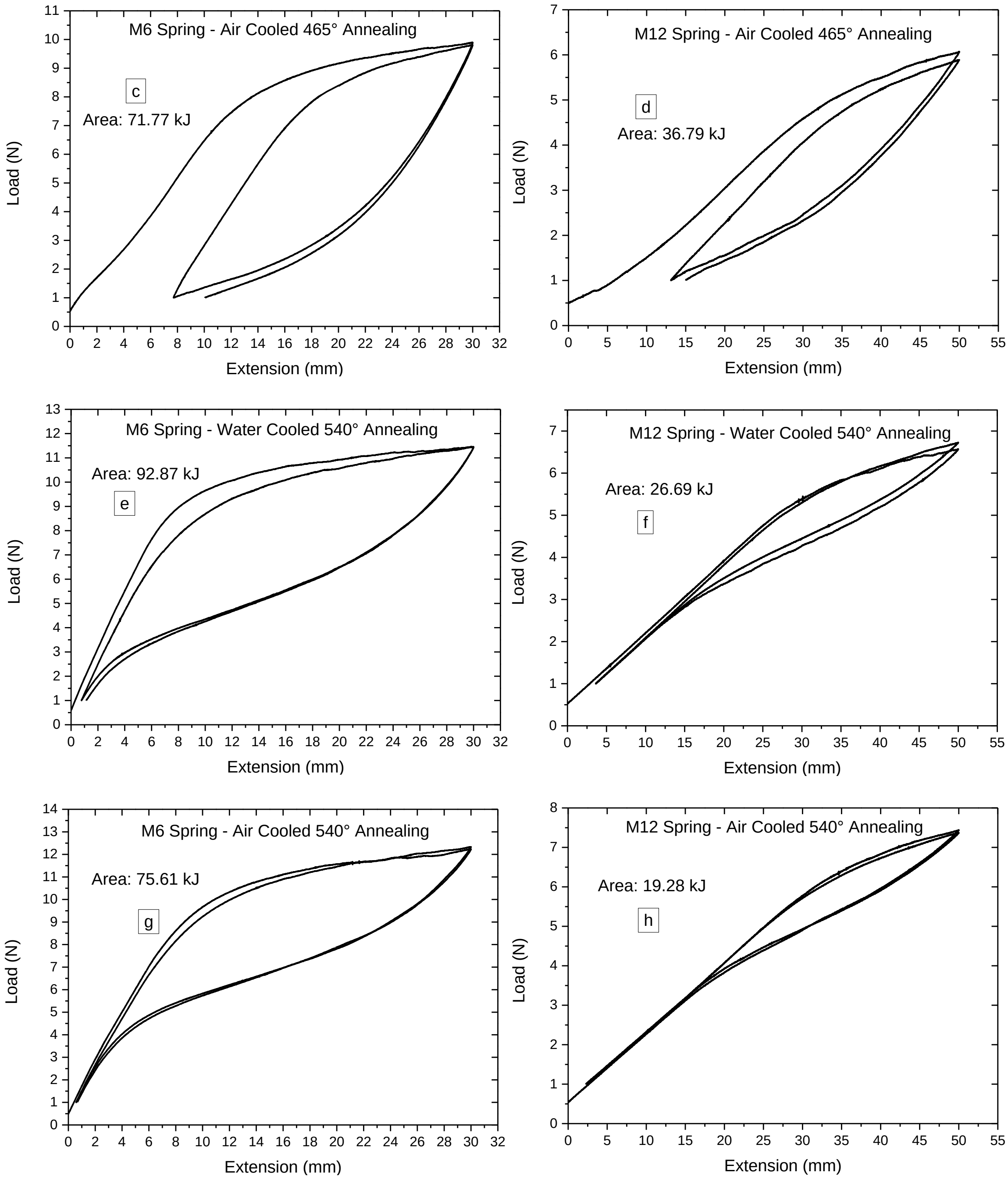


Figure 8. Cycle test of the springs: (a) M6 spring, water cooled, 465°C annealing, (b) M12 spring, water cooled, 465°C annealing, (c) M6 spring, air cooled, 465°C annealing, (d) M12 spring, air cooled, 465°C annealing, (e) M6 spring, water cooled, 540°C annealing, (f) M12 spring, water cooled, 540°C annealing, (g) M6 spring, air cooled, 540°C annealing, (h) M12 spring, air cooled, 540°C annealing

Figure 8 shows the results of the tensile tests on the springs, because the test were made at room temperature, looking at the DSC results on Fig. 5, all of the specimen were above A_f, so as expected they showed a pseudoelastic effect, with exception the two springs annealed at 465°C and water cooled. This two springs presented a mixed effect, presenting a hysteresis loop characteristic of the pseudoelastic effect and also retained an amount of strain. It can be also seen that heat treatments have little effect on the stress to begin the transformation, having a bigger effect of the reverse transformation stress and the spring stiffness.

Table 3 shows the spring stiffness, it shows that there’s a significant increase with the higher annealing temperature. About 35% on the M12 spring and 85% on the M6 spring. Also the water cooled method gives a slightly higher stiffness when compared with the air cooled, except for the two 465°C air cooled annealed springs, but their behaviour is because there is a percentage of martensite in the start of the tests.

Table 3. Springs initial stiffness

	M6		**M12**	
	Water Cooled	**Air Cooled**	**Water Cooled**	**Air Cooled**
465°C	622.61N/m	673.53 N/m	130.85 N/m	165.63 N/m
540°C	1155.49 N/m	1024.08 N/m	177.43 N/m	170.07 N/m

Another important aspect of SMA springs is the size of the hysteresis loop. Figure 8 shows that using a faster cooling method gives a bigger hysteresis loop, about 30%. The M12 air cooled 465°annealed spring gave the highest loop, but that is because on the M12 springs the 3N weren't enough to begin the transformation and this spring transforms during the whole loop.

4. CONCLUSIONS

On the reason to study the effects of the heat treatment on the manufacturing of SMA springs a set experiments were made. The heat treatments shown a few differences comparing the wires with the springs. A few degrees less to the transition temperatures in most the cases. Despite this changes are small, since the temperatures A_f and M_s are so close to room temperature, they affected the macroscopic behavior of the alloy. The same heat treatments that made shape memory effect wires, made pseudoelastic springs, with a exception.

Analysing the SMA springs, that are the main intent in this study, it was able to see that it can be obtained several different springs, with a few adjustments on the heat treatment. Was able to get over 50% gain on the spring stiffness by just changing the annealing temperature.

Also is possible to change the hysteresis loop size, although the difference is not so considerable as the spring stiffness. The control the variation of the hysteresis loop is done by adjusting the cooling speed. With a higher speed giving the greater loop.

5. ACKNOWLEDGEMENTS

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