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Characterization Techniques of a Shape Memory Nickel Titanium Alloy †

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Abstract: This paper presents a characterization processes study of metallic alloys, more specifically the shape memory alloys (SMA) composed by Nickel and Titanium (NiTinol). Two different wire suppliers were studied, starting with metallographic analysis until observe the contours of the grain wires. Differential scanning calorimetry (DSC) test was also performed to obtain phase transformation temperatures of the NiTinol alloys. Finally, after several tensile tests, some results were obtained for stresses, strains, elasticity modules and maximum rupture deformation.

Keywords: shape memory alloy; NiTinol; characterization processes

1. Introduction

Although the first shape memory alloy (SMA) has been discovered in 1932 by Arne Olander [1], the widely known nickel and titanium equiatomic alloy under the name NiTinol was discovered only in 1960 by Buheler and Wiley at the Naval Ordnance Laboratory (responsible for the suffix "nol" in the alloy name) [1–3].

NiTinol has proven to be the most advantageous of the known shape memory alloys, receiving much attention from the scientific community (as well as industrial and product designers) in recent decades. Nowadays this alloy receives a great investment from the automotive, aerospace and even civil engineering industries [1,2,4].

One of the great advantages of NiTinol is the possibility of adjusting its properties according to its chemical composition, and heat and/or mechanical treatments. The material can change between the austenitic phase and the martensitic phase without a diffusion process, thus allowing a return to the initial geometry, even after considerable deformation, but when subjected to a certain temperature. This effect is called *Shape Memory Effect* [1,3,4]. While the ability to recoverable large mechanical stresses is called *Superelasticity*, being these two features the major differential of NiTinol from others alloys [3–5].

There is also the possibility of a double shape memory effect. To achieve this, it is necessary to "work the alloy" known as *Training*, which occurs by introducing residual stresses to the material through applying certain and repetitive mechanical requests. This allows it to take on a cold form shape and a different hot form shape. However, excessive SMA training results in tension degradation, torsion or bending [1].

Transformation temperatures in commercial NiTinol alloys are usually in the range of −100 °C to +100 °C, with hysteresis ranging from 30 K to 50 K, with a temperature error of ±5 °C, which corresponds to a difference of ±0.05% in alloy composition [3]. The global smart materials market

moved around USD 19.6 billions in 2010, with an expected annual growth rate of 12.8% by 2016, with worldwide patents exceeding 20,000 registers [1].

2. Experimental Procedure

2.1. Description of Materialst

In the present work, two NiTinol alloys purchased from different laboratories were analyzed: the first from supplier 1—European supplier, with a diameter of 0.5 mm, a finishing austenitic temperature A_f of −10 ± 5 °C and, according to the supplier, 56.08% of Ni in its chemical composition. The second material analyzed came from the supplier 2—North American supplier, having a diameter of 2 mm and an A_f temperature of 20 ± 5 °C.

To determine the metallurgical, physical and chemical properties of the material were performed metallographic analysis, tensile tests and differential scanning calorimetry.

2.2. Metallographic Preparation

To encapsulate the sample, Clarofest 572 clear acrylic resin was used.

The embedded samples were classified in three different ways: "A" and "B" are from supplier 1, where the first one did not suffer plastic deformation while the second one did; "C" is from supplier 2 and has not undergone plastic deformation.

After sandpapering, the samples were mechanically polished with amorphous colloidal silica. The chemical attack used to reveal the grain boundary of the material was by applying a solution containing nitric acid with hydrofluoric acid (5:1 ratio) over the NiTinol alloy for a period of 2 to 3 min at room temperature.

2.3. Preparation for Differential Calorimetry Scan

The ASTM [6] sets standards for DSC testing for residual stress free NiTinol alloys having a nominal Nickel weight of 54.5% to 57%.

Slough [7] has shown that varying the heating and cooling rates does not show much difference in the transition regions and, that the higher the rate, the higher the phase transformation peaks, although lower rates do not compromise the results. Aiming to achieve the best representation, a rate of 20 °C/min was used in the tests performed in the present work.

An 87.01 mg portion of the sample from supplier 1 was treated at 850 °C for 15 min at an atmospheric pressure of –70 kPa and cooled in water. The sample from supplier 2 had a mass value of 40.85 mg and was treated at the same temperature for a time of 20 min at a pressure of −20 kPa and cooled in air.

Even under vacuum, both samples were found to have an oxidized surface. As large oxide layers can result in changes in DSC, polishing of these oxides was required. Thus, chemical polishing was performed with the same solution used for the chemical etching process during metallographic preparation, i.e., nitric acid and hydrofluoric acid in a 5:1 ratio.

The samples were placed in centrifuge tubes with the addition of just over 1.5 mL of the solution over a period of 1:30 h and shaken eventually throughout the polishing. Considerable removal of the oxide was noted and the solution was greenish in both cases. The mass of sample from supplier 1 decreased by 9.65% and that from supplier 2 decreased by 10.01%.

The DSC test was performed covering a temperature range between −70 °C and 100 °C, which is a region where it is safe to perform the aluminum crucible test and which coincides with the range in which phase changes are usually present in commercial alloys [3].

The DSC samples from suppliers 1 and 2 respectively contained 38.33 mg and 36.76 mg. The temperature change history for a fixed temperature range of 20 °C/min was as follows: The material started at room temperature of 25 °C and was heated to 100 °C, followed by cooling to −100 °C, again undergoing heating to 100 °C, then cooled to 20 °C so that it could be removed from the equipment.

2.4. Tensile Test

The test piece consists of a nickel-titanium wire with a diameter of 0.5 mm and a length of 50 mm. For both tests the load displacement application rate was 0.5 mm/min. Since the rupture test is performed until the specimen fails, no extra parameters are required, however for the cyclic test a displacement rate of 0.5 N/s has been set, with a preload of 10 N so as to leave the specimen stretched and an amount of 20 cycles.

3. Results

3.1. Metallography

The results obtained through metallographic analysis are only able to determine the grain boundary, and it is not possible to determine the phases present in the microstructure. Figure 1 shows the comparison of the results of samples "A" and "B". It is possible to observe that, although one of the samples suffered plastic deformation and the other did not, the grains showed no noticeable differences in their geometries.

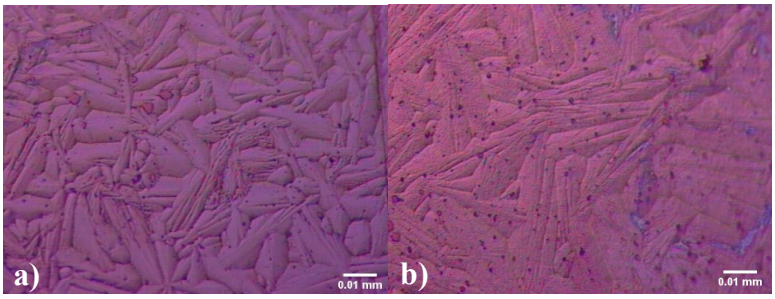

Figure 1. (**a**) Grain Outlines of Sample "A"; (**b**) Grain contours of sample "B". 50× magnification.

The sample from supplier 2, did not show much difference in geometry and grain size compared to samples from the other supplier, through the qualitative analysis performed.

3.2. DSC

The DSC results for supplier sample 1, shown in the diagram in Figure 2, demonstrated that the supplier-indicated transition temperature was found. The final austenitic transformation temperature was −11.8 °C within the range of −15 °C to −5 °C indicated by the manufacturer.

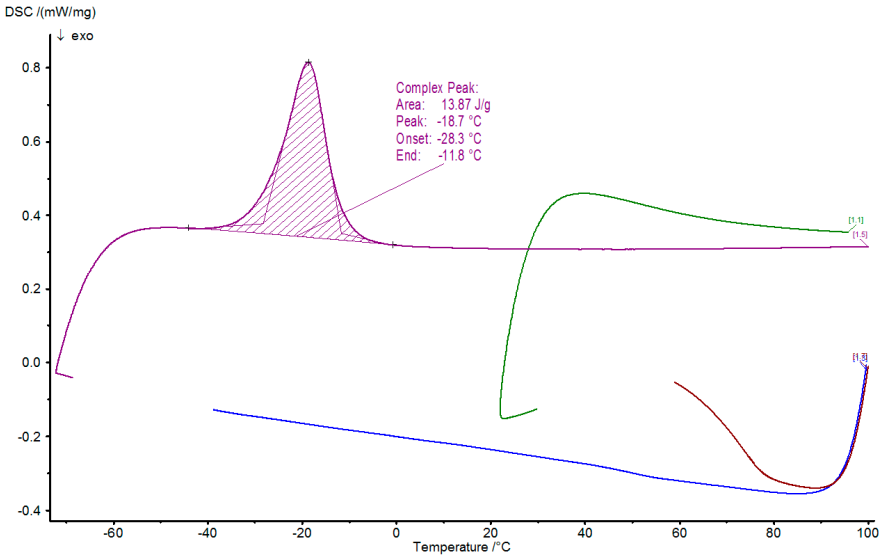

Figure 2. DSC result for the sample supplier 1.

The DSC result for supplier sample 2, was not conclusive.

3.3. Tensile Failure and Cyclic Tensile Tests

The data of the stress-strain diagram obtained is described in Table 1. For the cyclic test, the data is described in Table 2.

Table 1. Properties obtained by the traction-testing test.

Properties	Value
Breaking stress	1088.6 MPa
Maximum break deformation	31.17%
Elasticity Module—Austenite	39.03 MPa
Elasticity Module—Austenite—Martensite	10.04 MPa

Table 2. Properties obtained by the cyclical test.

Alloy Characteristic Properties	Stress (MPa)
Initial Stress—Direct Transformation	464.4
Final Stress—Direct Transformation	536.8
Initial Stress—Inverse Transformation	261.8
Final Stress—Inverse Transformation	190.8

4. Conclusions

- Mechanical characterization through tensile tests was achieved, since the characteristic stress data and elastic modulus were obtained;
- Thermal characterization presented partially complete results, due to the equipment limitations for temperatures below −50 °C, obtaining only the transition temperatures A_f and A_s;
- Since the results were obtained through the DSC it is concluded that metallographic techniques and measurements as well as the treatment of the material according to the norm can be standardized for future experiments;
- The grains did not show considerable differences in their geometry when subjected to plastic deformation;
- Supplier 1 characteristic alloy stresses were successfully obtained;

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