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# Mechanical behavior of cycled shape memory alloy

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## Abstract

Shape memory alloys (SMA) are often used in many industrial, medical and automotive fields due to the ability to recover the initial shape caused by external loads. This aspect is due to the variations of the microstructure that may depend on thermal and/or mechanical causes. So far, many models have been developed in order to describe the mechanical behaviour of SMA but most of them do not take into account the real microstructure.

In this work a conventional equiatomic NiTi SMA has been investigated in both terms of microstructure evaluation and mechanical cycling behaviour up to degradation the memory ability, which has obtained at around 100 cycles. The cycling behaviour has been investigated and a model has been proposed to predict the mechanical behaviour and the ability to recover the initial microstructure.

The damaged alloy has been investigated in terms of fracture surface analysis obtained by conventional tensile tests after 1, 50 and 100 cycles.

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*Keywords:* Fracture; Cycling; Shape memory alloy; NiTi.

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## 1. Introduction

Shape memory alloys are a broad category of materials that includes metallic alloys and some types of polymers. They may regain their original shape even after experiencing significant deformations caused by external mechanical loads (Volpe et al. 2014).

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Removing the external loads may have two effects (Furgiuele and Maletta 2010):

1. Without any more external activity, the first shape is received immediately
2. After heating the material above its critical temperature, the original shape is restored.

In the first case the material is characterized by a critical temperature lower than that of the environment and the material is a shape memory characterized by a pseudoelastic effect (Kuribayashi et al. 2006 and Daymond et al. 2007).

In the second situation, the substance is classified as a shape memory alloy that requires an external energy source (thermal energy) to restore its original shape (Iacoviello et al 2018).

This is due to the ability of the alloy to alter its lattice at relatively low temperatures, changing from a stable low-temperature lattice known as austenite to a new lattice known as martensite (Vantadori et al. 2018 and Di Cocco and Natali 2018).

Since the transition temperature is much lower than the recrystallization temperature, it is possible to change the microstructure without altering the boundaries. As a result, the number of crystals remains constant and the lattice change does not indicate migration of atoms between crystals (Berto et al. 2021 and Gollerthan et al. 2009).

In recent years, numerous investigations have been conducted on various elements of shape memory alloys. In some investigations on the behavior of copper base SMA, for instance (Volpe et al. 2014), the grains are clearly visible using metallographic LOM, allowing the behavior of the boundaries during lattice variation to be demonstrated. Other investigations on the nanohardness (Muller et al. 2012), impact (Cui et al. 2022) or fatigue behavior (Maletta et al. 2011 and 2012) of NiTi SMA demonstrated the influence of microstructure change on mechanical behavior. More recent research (Sgambitterra et al. 2016) emphasizes the interaction of temperature, although there are no more studies related to the measurement of the microstructure change caused by mechanical effect related to the mechanical behavior of SMAs.

In the last years, Di Cocco et al. 2018 proposed a simple model cable of calculating the effective microstructure evolution induced by mechanical loads in cycling tests and later in Berto et al. 2021 a relationship between effective microstructure and mechanical behaviour has been proposed.

In this work tensile fracture micromechanisms have been analysed in order to evaluate the influence of the cycling on the tensile fracture behaviour of an equiatomic SMA characterised by a pseudoelastic behaviour.

## 2. Materials and methods

Low-cycling structural alteration was evaluated using an equiatomic NiTi alloy with a pseudoelastic mechanical behavior (PE). In the equilibrium state diagram of the examined alloy, there is a crossing of the solution limits of two separate phases, which is unique for this alloy. Chemical composition has a significant impact on mechanical behavior since weekly variations in Ni or Ti levels alter the stability of phases and can alter the memory characteristics of an alloy.

The critical temperature of stable austenite was found to be lower than the room temperature thanks to the thermomechanical method used on the material under investigation. As a result, the investigated alloy is characterized by a PE behavior; it is able to recover its initial shape when load is null also even at high values of deformation.

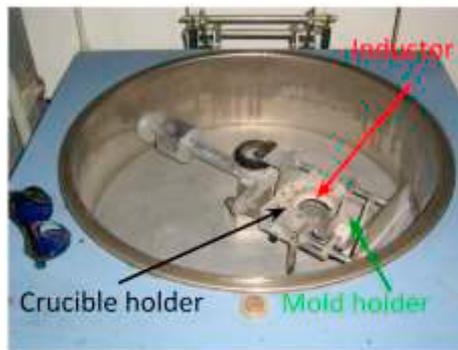


Fig. 1. The rotating oven used for specimen production.

The vacuum furnace used in the laboratory to produce the equiatomic NiTi alloy characterized by a pseudoelastic behavior, is shown in Fig. 1. The specimens are produced by spark-erosion machining the shape of which is shown in Fig. 2. The dimensions of the specimens are necessary for the use of a patented tool able to perform load/unload cycles on the specimens and stopping the programmed deformation test by allowing to perform X-Ray diffraction to be performed on the calibrated length under loading conditions.

The specimen deformation and the applied load were measured by means of a Linear Variable Differential Transformer (LVDT) and two load cells (10 kN each).

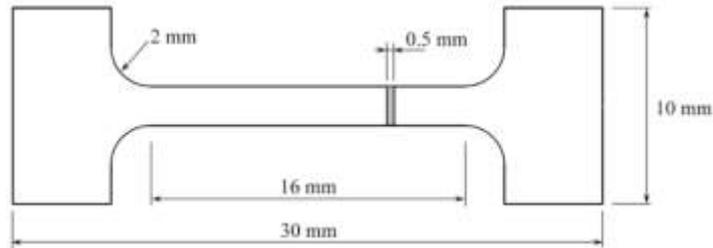


Fig. 2. Cycling specimen dimensions.

At room temperature, incremental isothermal tensile tests were performed with increasing specimen elongation after 1, 10, 50, and 100 cycles. In particular, the loading frame that housed the specimen was withdrawn from the testing apparatus for each loading step, at set deformation values, and examined using a Philips diffractometer in order to assess the XRD spectra. A vertical Bragg-Brentano powder goniometer and the Philips X-PERT diffractometer were used to make the XRD measurements.

A step-scan mode with a step width of  $0.02^\circ$  and a counting duration of 2 s per step was employed in the  $2\theta$  range between  $30^\circ$  and  $90^\circ$ . CuK Monochromatic radiation (40 kV – 40 mA) was used. Using PowderCell software, theoretical diffractogram calculations and structure model development were carried out.

A finite element simulation was also used to relate the gross engineering strain to the effective engineering strain, as shown in the next section.

In order to evaluate the changes in fracture micromechanisms caused by cycles, specimens subjected to 1 cycle and 100 cycles were employed in a conventional tensile test up to failure.

### 3. Results

The traditional three stages of shape memory alloys show a first stage where the behavior is the linear elastic range characterized by the Young's modulus of austenite, a second stage where the slope sharply decreases and the microstructure changes from austenite to martensite, and a third stage where the behavior is still linear elastic with the Young modulus of martensite — this is how the NiTi alloy behaves under tensile loads.

The unloading stages are nearly identical, however due to hysteresis phenomena, the starting points of the stress and strain microstructure evolution change.

The mechanical differences between loading and unloading curves are depicted in Fig. 3.

The evolution of the microstructure is shown in Fig 4, where the diffraction spectra taken at initial test (zero deformation), at maximum deformation, and at complete recovery of the shape are shown. It is possible to highlight that the peaks present at zero deformation in the initial conditions and after one cycle are characterised by the same diffraction angle, instead at  $\epsilon=10\%$  only a peak is present at higher angle values.

It means that at  $\epsilon=0\%$  the diffraction is due to the presence of a cubic austenite both in the initial conditions and after 1 cycle in recovered shape, and at  $\epsilon=10\%$  the diffraction spectrum is due to the presence of martensite phase (complete transformation of austenite in martensite).

The spectrum of martensitic phases is correlated to a monoclinic phase.

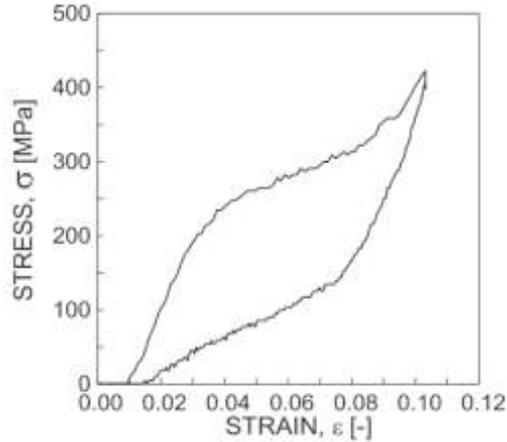


Fig. 3. Stress-Strain behavior of NiTi in the cycle 1.

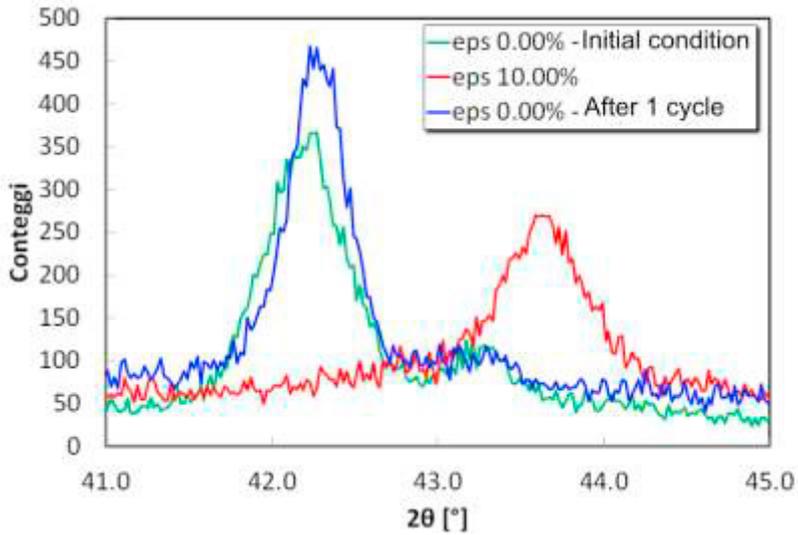


Fig. 4. Diffraction spectra at different strain.

Fig. 5 shows the effect of the hysteresis taking into account the diffraction of the  $\epsilon=5\%$  specimen achieved under loading and unloading conditions. It is worth noting that the difference of the stress level is not the only differences of the hysteresis, but as is shown in the diffraction spectra, the differences are also in terms of microstructure. Under loading conditions, the main phases present in the microstructure is the austenite (red peaks), while in unloading condition it is the martensite (green peaks).

According to the model proposed by Di Cocco et al. 2018 and Berto et al. 2021, in order to calculate the mechanical behaviour of the NiTi, Young modulus can be expressed by the relation (1)

$$E = \frac{A}{1 + K_A \frac{\partial A}{\partial \epsilon}} E_A + \frac{M}{1 + K_M \frac{\partial A}{\partial \epsilon}} E_M \tag{1}$$

where the amount of the austenite A and the martensite M can be calculated using the relations (2)

$$A = \frac{D e^{-C\varepsilon}}{1 + D e^{-C\varepsilon}} \tag{2}$$

$$M = 1 - A = \frac{1}{1 + D e^{-C\varepsilon}}$$

Considering the values of parameters C and D (taken from Berto et al. 2021) the comparison between the actual and the calculated cycling behaviour at cycle 50 is shown in Fig. 6.

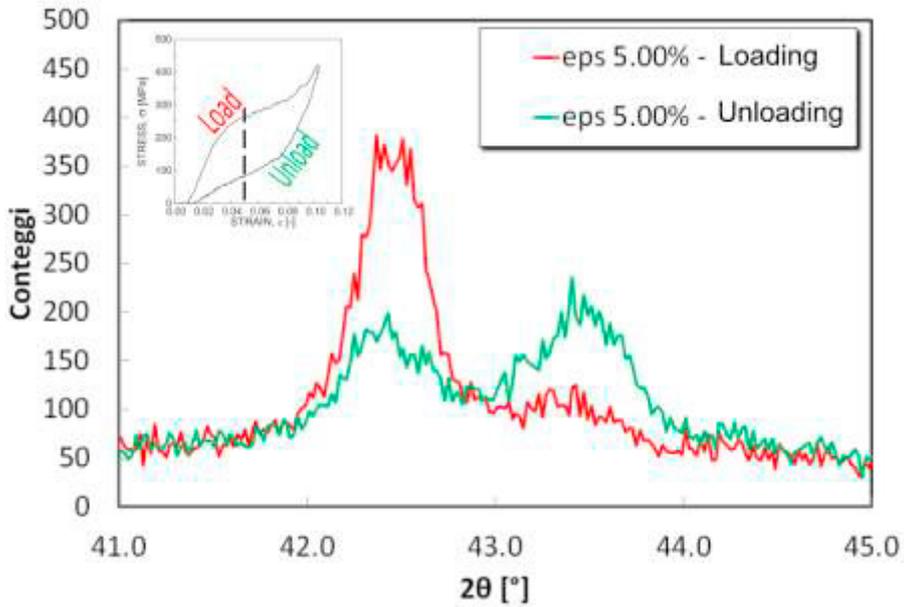


Fig. 5. Diffraction spectrum and microstructure at  $\varepsilon=5\%$  under loading and unloading conditions.

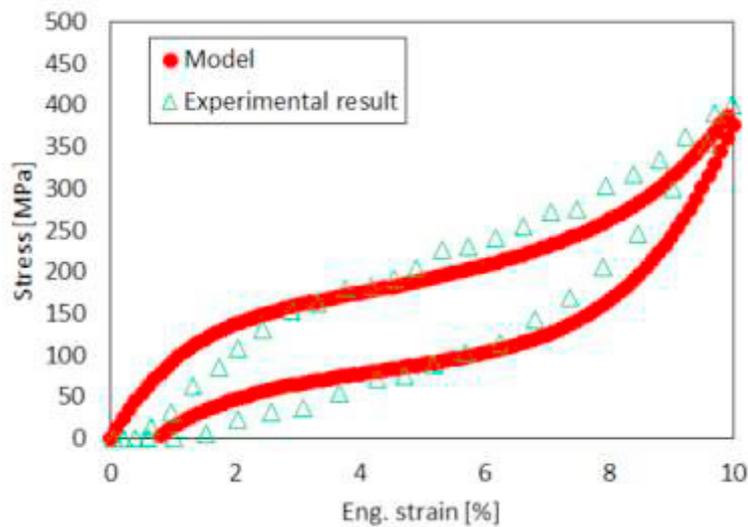


Fig. 6. Comparison of measured and calculated behavior of NiTi at cycle 50.

As can be seen, there is some residual martensite that cannot change into austenite, preventing a full recovery of the original shape. This is the result of cyclic deterioration of the alloy.

Performing a conventional tensile test on specimens up to failure after 1 cycle and after 100 cycles reveals various fracture micromechanisms on the fracture surfaces.

As shown in Fig. 7 the fracture surface of the specimen at 1 cycle is characterized by a conventional ductile cup-cone fracture, as opposed to the fracture surface of the specimen at 100 cycles, where the main fracture micromechanisms are characterized by a minor level of necking due to the unchanged martensite.

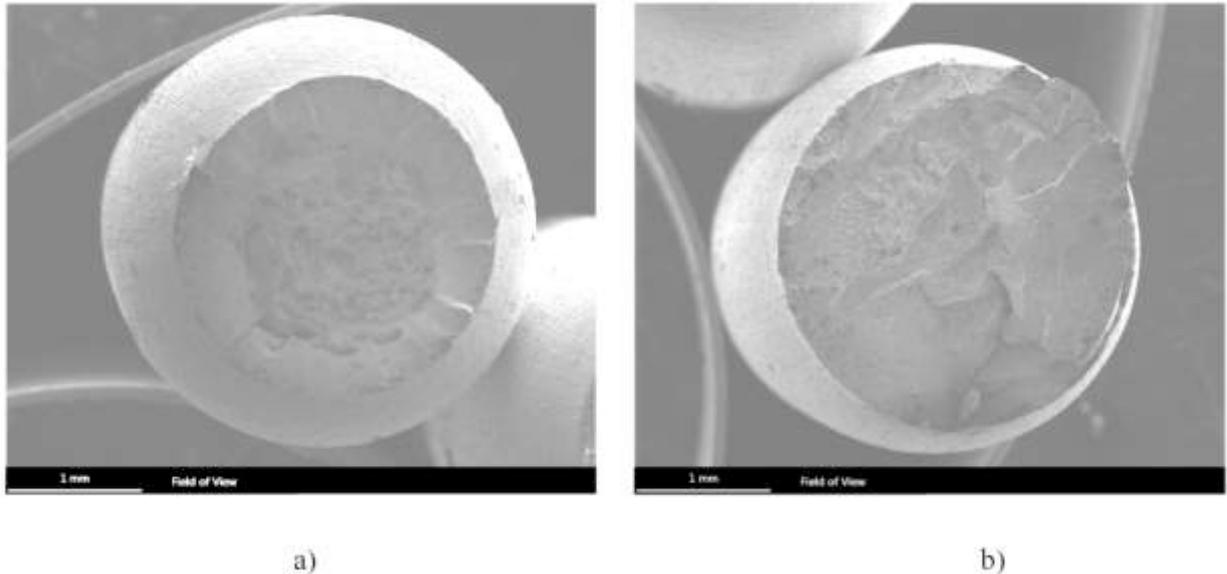


Fig. 7. Tensile fracture surface observations: a) after 1 cycle, b) after 100 cycles.

### Conclusions

This research examines the cyclic evolution of mechanical behavior considering the actual evolution of austenite and martensite using an equiatomic NiTi shape memory alloy. The following is a summary of the findings:

1. A hysteresis phenomenon is present not only in terms of stress-strain curve, but also in terms of microstructure evolution
2. The Berto et al. 2021 model is able to predict the cyclic mechanical behavior of Shape memory alloys, which are distinguished by a transition from austenite to martensite and vice versa without the presence of any other intermediate phases
3. The alloy subjected to high cycling exhibits residual martensite, probably due to its cyclic deterioration
4. The fracture surfaces of the cycled specimens revealed the presence of a minor necking due to untransformable martensite.

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