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The effect of heat and mechanical treatments on the NiTi SMA tensile behaviour

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Abstract

Equiatomic NiTi alloys exhibit shape memory or superelastic behaviour. This remarkable property allows them to "remember" their original shape. The key lies in a reversible change in their crystal structure (phase transition) triggered by stress or temperature, without any permanent rearrangement of the crystals (recrystallisation). Unlike traditional metals, the grains can deform but maintain their overall arrangement. Initially, in shape memory alloys with pseudoelastic behaviour, the stable phase is the austenite, characterised by a cubic lattice. Under tension, austenite behaves elastically at first. However, a unique phenomenon occurs upon reaching a critical stress level: instead of yielding or plastically deforming, austenite transforms into a new phase called martensite with a monoclinic lattice.

This study examined the tensile behavior of an equiatomic NiTi shape memory alloy characterised by a pseudoelastic effect after various thermomechanical treatments. The resulting stress-strain curves were employed to assess the influence of these treatments on parameters within two constitutive models from the literature: Di Cocco and Natali's microstructure evolution model and Bellini et al.'s model.

Keywords: NiTi, SMA, Treatments

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Nomenclature

EA	engineering elastic modulus of austenite
EM	engineering elastic modulus of martensite
KA	stiffness parameters of the mechanical model of austenite phase
KM	stiffness parameters of the mechanical model of martensite phase
C	cyclic parameter of the structural model
D	hysteretic parameter of the microstructural model

1. Introduction

Shape memory alloys (SMAs) are metallic materials that have the ability to recover their original shape after undergoing apparently permanent deformations (Silva Lobo et al (2015)). This peculiar characteristic, known as the shape memory effect, is due to a reversible solid-solid phase transformation between a martensitic structure at low temperature and an austenitic one at high temperature (Suravarna et al. (2024)).

Among SMAs, Nickel-Titanium (NiTi) based alloys, commonly known as Nitinol, have attracted considerable interest in the scientific and industrial fields thanks to their excellent mechanical properties, such as high resistance to corrosion, biocompatibility, and superelasticity (Amadi et al (2024)).

Superelasticity, or pseudoelasticity, manifests itself as the ability of the material to withstand considerable deformations in a given temperature range and to recover them completely upon release of the applied load. This behaviour, linked to stress-induced martensitic transformation, opens the way to numerous innovative applications in various sectors, including (El-Feky et al. (2023), Costanza et al. (2024)):

- Biomedical engineering: cardiovascular stents, orthodontic devices, minimally invasive surgical instruments.
- Aerospace: wing morphing actuators, solar panel deployment systems.
- Robotics: soft robot actuators, microgrippers.
- Civil engineering: anti-seismic devices, reinforcement of structures.

The microstructure of NiTi alloys plays a crucial role in determining the mechanical properties and superelastic behaviour. The austenitic phase, stable at high temperatures, has a face-centred cubic (CFC) crystalline structure, characterised by an atom at each cube's vertex and an atom at the centre of each face, as stated by Otsuka and Ren (2005). This structure gives the material high symmetry and ductility. Stable at low temperature, the martensitic phase can take on various crystallographic variants, including the monoclinic martensite B19', characterised by a unit cell with unequal angles and sides, as stated by Miyazaki and Otsuka (1989). The transformation from austenite to martensite involves a significant change in the arrangement of atoms in the crystal lattice and a reduction in symmetry, allowing the material to accommodate significant deformations.

Microstructural defects, such as crystalline grains, grain boundaries, precipitates and dislocations, significantly influence the nucleation and propagation mechanisms of martensite and, therefore, the characteristics of superelastic behaviour as shown in Hornbogen (2004). For example, the size of austenitic grains can influence the transformation temperature and crack propagation of martensite as investigated by Khalil-Allafi et al. (2009).

Numerical modelling plays a fundamental role in understanding and predicting the mechanical behaviour of superelastic NiTi alloys. Several approaches have been proposed in the literature, including:

- Phenomenological models: describe the macroscopic behaviour of the material through constitutive relations that link stress, deformation, and temperature. An example is the Graesser-Cozzarelli model (Graesser and Corazzelli (1991)), which considers the hysteresis in loading-unloading cycles.

- Micromechanical models: they are based on the description of phase transformation mechanisms at the microscopic level, considering the evolution of the material's microstructure. Among these, "phase field" models introduced by Levitas et al. (2002) simulate the nucleation and growth of martensites.
- Multiscale models: integrate phenomenological and micromechanical models to describe the material behaviour on different length scales accurately.

The choice of the most appropriate model depends on the specific application and the level of detail required. For example, for the design of medical devices, phenomenological models may be sufficient to predict the global mechanical response. In contrast, micromechanical or multiscale models are more suitable for studying fatigue or fracture.

Despite significant progress in modelling superelastic NiTi alloys, some challenges remain to address. In particular, accurate modelling of hysteresis, strain rate effect, and the influence of microstructural defects represents an active field of research.

The microstructural transitions in a NiTi alloy during a multistage loading-unloading cycle can be studied by using the X-ray diffraction, in order to assess the microstructural transformations under mechanical uniaxial deformation, as described by Di Cocco et al. (2013).

Some approaches to solving inverse elasticity problems are based on regression algorithms to estimate materials and/or loading parameters by fitting the experimentally evaluated displacement field to representative analytical solutions, as done by Sgambitterra and Niccoli (2021) and Merlin et al. (2015).

A different model to predict the material behaviour and ability to recover its initial microstructure after cycling has been proposed by Bellini et al. (2022), where the investigated alloy exhibited some residual martensite after cycling, which may be due to cyclic damage. The model was verified with an equiatomic NiTi alloy characterised by a pseudoelastic behaviour. Di Cocco and Natali (2018) found that the model was able to accurately predict the microstructure quantities at different imposed strains both in loading and unloading conditions.

An integrated model to predict the microstructure evolution and mechanical properties of a NiTi SMA has been proposed by Bellini et al. (2021). The model takes into account the hysteresis and the effect of cycling on both microstructure and mechanical behaviour.

The crack growth rate is strongly influenced by the R ratio, which is the ratio of the minimum to maximum stress intensity factor. This is mainly due to the presence of brittle inhomogeneities at the grain boundaries (Di Cocco et al. (2014)).

This article aims to analyse the application of a structural-mechanical model to predict the tensile behaviour of an equiatomic NiTi shape memory alloy. The main modelling approaches will also be discussed, highlighting the advantages and limitations of each method.

2. Material and methods

This study utilised wire samples of equiatomic NiTi shape memory alloy with a diameter of 0.8 mm and a nominal calibrated length of 70 mm. The sample wires were deformed to an imposed strain of 10% at room temperature (20°C) to induce a complete martensitic transformation. They were then heated to 300 °C.

All samples were subjected to tensile testing until failure at a low strain rate (10^{-5} s^{-1}). The resulting stress-strain curves were used to validate a mechanical behaviour model proposed by the authors in Di Cocco and Natali (2018) and Bellini et al. (2021). This model accounts for the microstructure competition between austenite and martensite phases in the alloy.

2.1. Microstructural model and Mechanical model

The microstructural model employed in this study was proposed by Di Cocco and Natali (2018), and it assumes that the austenite-martensite and martensite-austenite transformations are driven by the minimisation of a thermodynamic potential, E. The following relationships between the austenite and martensite fractions at imposed deformations ε can be derived:

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

The parameters C and D are dependent on the state of the alloy and govern the microstructural behaviour at different numbers of cycles (parameter C) and the hysteresis during loading-unloading (parameter D).

The mechanical behaviour model, proposed by Bellini et al. (2021), assumes that during the austenite-martensite transformation, the mechanical energy is not only used for elastic deformation but also for the phase transformation itself. Consequently, the total elastic modulus depends on the deformation ε and is calculated as the sum of the contributions from the austenite and martensite phases, including the amount of austenite transforming into martensite:

$$E=EA\cdot\frac{A}{1+KA\frac{dA}{d\varepsilon}}+EM\cdot\frac{M}{1+KM\frac{dM}{d\varepsilon}} \tag{2}$$

where EA and EM are the elastic moduli of the austenitic and martensitic phases, respectively, and KA and KM are parameters that correlate the energy used for the phase transformation with the elastic mechanical energy.

3. Results and discussion

The tensile curves demonstrate good repeatability under identical conditions. A representative curve is shown in Fig. 1, where four different stages can be observed:

- Elastic Stage (Austenite): Initial linear elastic behaviour corresponding to the austenitic phase.
- Transformation Stage: A sharp decrease in slope indicating the stress-induced austenite-to-martensite transformation.
- Elastic Stage (Martensite): Another linear elastic region representing the martensitic phase.
- Plastic Stage: Final stage characterised by plastic deformation leading to specimen failure.

Strain gauges were not employed for local strain measurements; therefore, all data are presented in engineering terms. Consequently, EA and EM represent the engineering elastic moduli of the wire, referred to as the austenite and martensite phases (see Table 1).

Table 1. Experiment and model parameters.

Parameters	Experiment	Model
EA	13200 [MPa]	13200 [MPa]
EM	9200 [MPa]	9200 [MPa]
KA	---	0.4
KM	---	0
C	---	80
D	---	800

Using the structural model (1) and the mechanical model (2), the parameters obtained by minimising the error between the model and experimental tensile curve are shown in Table 1, where KA=0.0 and KM=0. This means that during the tensile test, a part of the energy supplied to the austenitic phase is used for the transformation from austenite to martensite, while the energy supplied to the martensite is totally used for the mechanical response.

As shown in Fig. 1, the curve obtained by applying the module fits the experimental curve well. In particular, the initial part of the austenitic stage and the martensitic stage are similar, while there are larger errors in the second stage. Focusing on the second stage, where the transformation of austenite into martensite occurs, there is an underestimation of the real stress at the beginning of the plateau and an overestimation in the final part. This discrepancy can be attributed to the minimisation algorithm that does not take into account the quadratic error but only the absolute error.

From the coupled structural and mechanical model, it is possible to have an estimate of the actual amount of austenite and martensite at each point of the second stage.

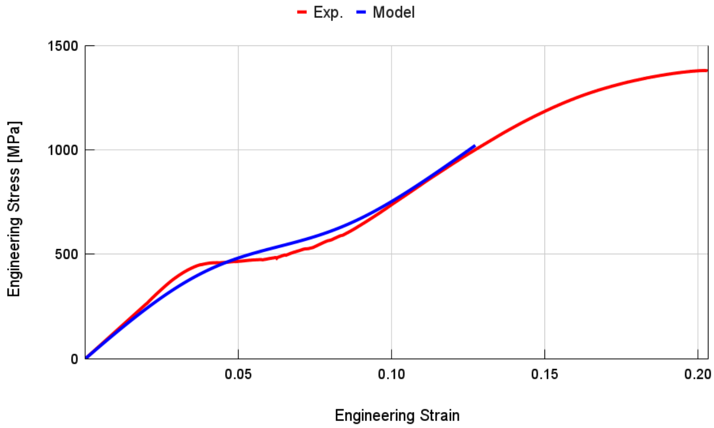

Fig. 1. Stress-strain curves: model compared to the experimental results.

Fig. 2 shows the trend of the austenite decrease in the first three stages, and it can be noted that the austenite decrease does not follow a linear trend. This is also confirmed in literature (Di Cocco and Natali (2018)) and in particular, in case of load, there is a very accentuated austenite decrease in the first part of the second stage. This austenite decrease causes a lowering of the stress-strain curve since part of the energy supplied to the specimen is used precisely for the phase transition.

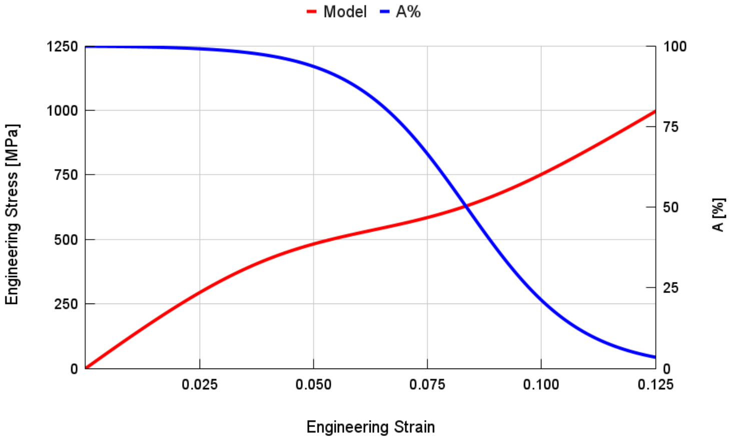

Fig. 2. Prediction of Austenite percentage during the tensile tests.

It follows that in the middle of the second stage, the amount of austenite is not 50%, as has also been demonstrated in several papers, such as in Di Cocco et al. (2013).

The fracture surfaces obtained from the tensile tests show the typical plastic behaviour of these NiTi alloys (Fig. 3) with the formation of a "cup-cone" type fracture and the presence of shear deformation on the external part and microdimples in the internal part. The fracture occurred when the alloy presented only the martensitic phase with a monoclinic microstructure, as reported by Otsuka et al. (2005). For this reason, the behaviour of the martensite in shape memory NiTi alloys is ductile, while in traditional martensitic steels, the fracture tends to be of the brittle type because the martensite is characterised by a tetragonal lattice.

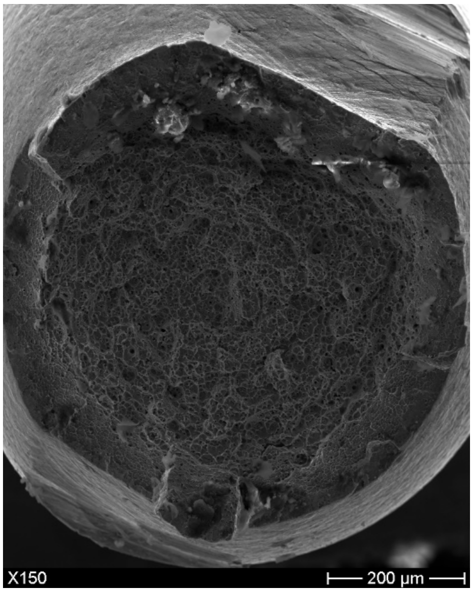

Fig. 3. Tensile fracture surface.

4. Conclusions

According to the experimental results, the following conclusions can be summarised:

- The Austenite-Martensite transitions are not linear.
- The Stress – Strain behaviour of SMA depends on the Austenite and Martensite volume fractions, which rely on the quantities of Austenite and Martensite are transforming.
- The parameters relating to the description of the microstructure variation, C and D, allow to describe the microstructure evolution during the tensile tests.
- The parameter KA is not null, and the KM is null; it means that during the Austenite-Martensite transformation the energy supplied to the martensite is fully involved in the mechanical behaviour, while part of the energy supplied to the austenite is used to the microstructural evolution.

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