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Experimental investigation of the pseudoelastic behaviour of NiTi wires under strain- and stress-controlled cyclic tensile loadings

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Abstract:

Pseudoelastic NiTi based shape memory alloys (SMA) wires have a good potential in civil engineering applications and often used as dampers in anti-seismic or cable structures when a rate varied cyclic loadings is applied. In accordance with these applications, an experimental characterization of NiTi wires under strain- and stress-controlled cyclic loadings at various strain rates is presented. Based on these loadings, it is shown that the stress–strain response of the material evolves up to a complete stabilization with the increase of the number of cycles. This evolution is accompanied with the degeneration of pseudoelasticity and transformation ratcheting in the case of strain- and stress-controlled mode, respectively. Also, it was found that the cyclic deformation of pseudoelastic NiTi SMA is significantly rate dependent.

Keywords:

Pseudoelastic NiTi; Cyclic loading-unloading; Degeneration; Ratcheting; Strain rate dependence.

1. Introduction:

Nickel-Titanium (NiTi) Shape Memory Alloys (SMA) represent an interesting group of metals exhibiting specific characteristics which have been largely experimentally studied, including pseudoelasticity and a high damping capacity [1-3]. Pseudoelasticity refers to the material's ability, above a transition temperature, to recover strains isothermally during a mechanical load/unload cycle, usually *via* a hysteresis loop. This behaviour is associated with the nucleation of a low temperature phase (martensite) to a high temperature phase (austenite). Such transformation is known as martensitic transformation. It has been found that NiTi alloys can exhibit a high damping capacity during phase transformation or in the martensitic state, which open a new application field in engineering [4,5]. Within this framework, NiTi based SMA are dedicated to the application of damping devices in order to reduce vibration amplitudes of bridge cables subjected to potential damages [1].

However, one of the major limitations facing the industrial use of SMA is the degradation of the alloy when subjected to cyclic loadings as reported by several authors [6-8]. In fact, the change of the hysteretic stress-strain response of the alloy, under fatigue cycling loads, can be associated to several microstructural mechanisms, like the formation of stabilized martensite, detwinning, grain reorientation, slip deformation and the generation of lattice defects mainly in the form of dislocations [9,10]. Indeed, during cyclic loading, twins become larger and dislocations glide easily and shear precipitates. Some studies reported observing the twinning phenomenon only along the stress plateau, during martensite transformation. Continuing deformation beyond the stress plateau induces detwinning of the martensite, accompanied by the production of dislocations [6].

Moreover, during cycling loads there is both an increase of residual strain and a decrease of the hysteresis loop area. Consequently, the capabilities to recover the applied deformation and to dissipate energy are affected and so functional properties are gradually lost. Understanding the different physical mechanisms on relation to the cyclic behaviour seems to be essential. The stabilized states as well as the maximal dissipation capacity are required for anti-seismic structures. In fact, SMA must be mechanically loaded (cyclic loadings) prior to be tested until the stabilization of behaviour is reached. Consequently, no longer additional residual strain is expected, stress required for phase transformation remains constant (stabilized value) and the working stress range can be adapted *via* a pretension load [2].

The strain rate was assumed as a fundamental parameter, the range of interest for seismic applications being much higher than for current applications of SMA [11]. Experimental research works [12-14] have shown the dependence of the mechanical response of pseudoelastic SMA structures on strain rate. This dependence is the result of a strong thermo-mechanical coupling rather than a viscosity's effect as shown in ordinary metals [15]. NiTi wires, presented later, are used as damper devices to reduce the vibration amplitudes of civil engineering cables. These wires are subjected to a high number of mechanical working cycles in tension (no compression or bending). Therefore, it is required to understand the dependence of strain rates on the pseudoelastic behaviour under cyclic loadings up to the stabilized state. Recently, this dependence has been experimentally highlighted on trained pseudoelastic NiTi with equiatomic composition [16-18].

However, to the author's knowledge, few early published works have pointed up the effect of strain rate on the pseudoelasticity degeneration of untrained NiTi SMA [14] under strain-controlled mode. In addition, the cyclic response under stress-controlled one as well as the

interactions between pseudoelasticity degeneration and rate dependence haven't been well documented in the literature for pseudoelastic NiTi SMA.

Therefore, in this work, the rate-dependant deformation of untrained pseudoelastic NiTi wires under strain- and stress-controlled cyclic tensile loadings at room temperature (RT) is investigated. First, a description of the studied material will be given. On one hand, thermal analysis details of the differential scanning calorimetry (DSC) test will be presented in order to discern the different phases existed in the alloy and the corresponding phase changes. On the other hand, the austenitic microstructure will be analyzed. Then, a series of cyclic tensile loading-unloading tests at various strain rates will be presented, and the effect of strain rate on the cyclic behaviour of NiTi will be therefore discussed.

2. Material and experimental details:

Polycrystalline Nickel-Titanium wires (diameter of about 2.46 mm) were used in this investigation. The material was provided by Memry Corporation (Connecticut, USA) a SAES group getters company (Italy) and its chemical composition is given in Table 1. Indeed, the alloy was Ni₅₂ at.%. The nominal and atomic alloy compositions have been determined with SEM-EDX analysis (Figure 1) and results have shown a percentage of Ni and Ti close to those given by the supplier. Therefore, NiTi alloys used in this investigation were rich in terms of Ni element.

It is well-known that the interesting properties of SMA are the result of a reversible solid-solid phase martensitic transformation. The transformation from austenite to a martensite and *vice versa* is associated with the release and the absorption of latent heat. Thus, DSC analysis was used to measure the phase transformation temperatures and the latent heat due to phase change. In this work, DSC samples (34.300 mg of NiTi) were first heated to 100°C, cooled to -140°C and heated again to 100°C, at heating/cooling rates of 5°C/min.

In order to analysis the initial microstructure of NiTi, samples were mechanically ground using SiC abrasive paper and polished to a final polishing step with a diamond suspension monocrystalline (9, 3 and 1 μm). To be sure that there was no contamination appearing after polishing, ultrasonic cleaning was required. The microstructure features were investigated using a LEICA type metallographic microscope after etching with a diluted solution of hydrofluoric and nitric acids (10HF+25HNO₃+150H₂O) for 75 seconds.

Tensile tests were carried out using a Tensile Testing Machine WDW with a maximum load of 10kN. Tests were performed under cyclic loading conditions on specimens whose initial wire sample lengths (L_0) were 120 mm. An extensometer was used to provide the strain

value with a reference length of 50 mm. The test temperature was set to 25°C (RT). Due to a strong localization in the pseudoelastic NiTi, a heterogeneous strain field will exist along the axial direction of the wire and the measured strain by the extensometer should be denoted as a nominal strain. On one hand, for the strain-controlled mode, four nominal strain rates, i.e., $3.3 \times 10^{-4} \text{ s}^{-1}$, $1.3 \times 10^{-3} \text{ s}^{-1}$, $6.6 \times 10^{-3} \text{ s}^{-1}$ and $1.6 \times 10^{-2} \text{ s}^{-1}$ were prescribed in the cyclic tests. It should be noted that for the case at lower strain i.e., $3.3 \times 10^{-4} \text{ s}^{-1}$ the number of cycles was prescribed to be 60 and two maximum tensile strains were tested (3 and 7%). For the ones at higher strain rates (1.3×10^{-3} , 6.6×10^{-3} and $1.6 \times 10^{-2} \text{ s}^{-1}$), the number of cycles was prescribed to be 100 and the maximum tensile nominal strain for each load case was set as 7%. On the other hand, for the stress-controlled mode, four nominal stress rates were prescribed, i.e., 6.3 MPa. s^{-1} , 25.3 MPa. s^{-1} , 126.3 MPa. s^{-1} and 315.6 MPa. s^{-1} . 60 cycles were performed for the lower stress rate (i.e., 6.4 MPa. s^{-1}) and 100 cycles were tested for the other tests. The maximum tensile nominal stress used in these tests was set as 855 MPa.

3. Results and discussion:

3.1. Differential Scanning Calorimetry

Figure 2 shows a DSC thermograph of the NiTi alloy with the characteristic transition temperatures. The start (subscript s) and finish (subscript f) temperatures have been estimated with the tangent method as depicted in Figure 2. The material is characterized by an austenite finish temperature $A_f=15^\circ\text{C}$; therefore, it exhibits a fully austenitic structure at RT. Moreover, the graph clearly shows a two-stage direct transformation from high temperature phase (B2 cubic Austenite) to rhombohedral phase (R) to low temperature stable phase (monoclinic B19' Martensite). A two-stage inverse transformation B19'-R-B2 characterizing the heating branch of the curve is also observed. In the literature, authors have claimed that the B2-R transformation can be introduced by many different ways. Addition of third element, such as Fe and Al, is effective in suppressing the B2-M transformation to lower temperatures to reveal the B2-R transformation [19]. Moreover, R-phase transformation can be induced in binary NiTi alloys by different thermomechanical treatments, such as cold working [20], thermal cycling and post-deformation annealing treatments [21]. For Ni-rich NiTi alloys (Ni>50.5 at.% practically), ageing treatment causes the R-phase transformation [22,23]. In fact, the meta-stable Ni_4Ti_3 particles promote the nucleation of R-phase, which then becomes easier than the nucleation of monoclinic martensite [23]. Moreover, the sequential B2-R was reported previously by Waitz et al., [24] in case of nanocrystalline NiTi. It has been shown

that the R-phase, forced by the constraints of the grain boundaries, precedes the martensite and so the martensitic transformation is suppressed in the ultrafine grains.

The R-phase affects the damping capacity of NiTi alloys [2]. Moreover, the latent heats released or absorbed during the phase changes have been determined by integrating areas between the peaks and their baselines. The different values of latent heats, except those coming from the transformation $R \rightarrow B19'$ during cooling, are indicated in Figure 2. These values are in accordance with the results provided by Schlosser [25].

3.2. Metallurgical analysis

After etching with the diluted solution, distinct grain boundaries for the samples are apparent (Figure 3), and the bulk microstructure of austenitic NiTi is revealed. In order to more clarify the grain boundaries, grain boundary map was extracted (Figure 4(a)) and the average size of the different grains was measured by MIPAR software. The austenitic grains are so fine with an average diameter of about 8 μm (Figure 4(b)). Otherwise, dark particles characterizing the microstructure, were analyzed with SEM-EDX and it is shown that Titanium is the main element which forms these precipitates (Figure 5). Dark gray particles are supposed to be Ti_2Ni particles and those appearing as black spots are identified as TiC particles. All these particles are presented in Ni-rich NiTi alloys [26].

3.3. Pseudoelasticity degeneration and cyclic deformation of NiTi wires at strain-controlled mode

3.3.1 Stress-strain response

Figure 6 displays the cyclic stress-strain of the NiTi wires obtained in the cyclic loading-unloading tests at various strain rates, i.e., $3.3 \times 10^{-4} \text{ s}^{-1}$, $1.3 \times 10^{-3} \text{ s}^{-1}$, $6.6 \times 10^{-3} \text{ s}^{-1}$ and $1.6 \times 10^{-2} \text{ s}^{-1}$ and at a maximal strain of 7 %. The stress-strain curves obtained in the 1st, 2nd, 20th and 60th are given in Figure 6(a), and the ones obtained in the 1st, 2nd, 20th, 60th and 100th are given in Figures 6(b), 6(c) and 6(d).

Some common observations can be made from these experimental results. During cycling and at each strain rate, the hysteresis loop evolves, becomes smaller and reaches a stabilized state. Indeed, the energy dissipated during martensitic transformation, given by loop's area, decreases with the increase of the number of cycles. Moreover, it's seen from Figure 7(a) that the stabilization of hysteresis loops during cycling is accompanied with a decrease in the martensitic transformation yield stress. As noted, σ_{Ms} exhibits a marked decrease in the first few cycles until reaching a saturated value. It has been suggested that the presence of residual

martensite as well as the presence of a heterogeneous structure would facilitate the martensitic transformation by lowering σ_{Ms} [27]. The decrease in martensitic transformation yield stress may be also a result of grain reorientation through cyclic tensile loadings [28]. Moreover, it is readily concluded that the evolution of martensitic transformation with number of cycles **strongly depends** on the strain rate. Cycling at higher strain rates was found to induce a more rapid decline of the critical stress for martensite formation, as revealed by Strnadel et al., [29]. In particular, at a strain rate of $3.3 \times 10^{-4} \text{ s}^{-1}$, a marked reduction of the value of σ_{Ms} from 529 to 332 MPa has been observed too. Moreover, residual strain appears at the end of the second unloading and increases progressively with cycling, until a stabilized value is reached (Figure 7(b)). It is pertinent to note that the stabilization is reached rapidly when the strain amplitude is the lowest. As shown in Figures 8(a,b), the stabilized values of martensitic transformation yield stress and residual strain are reached more rapidly for 3 %. This result is in contrast with the findings reported by Ng and Sun [30]. In fact, the latter have shown that the number of cycles to stabilization is unaffected by the strain amplitude. Besides, the **apparent** elastic modulus at loading (E_{loading}), which is between those of austenite (E_A) and martensite (E_M), decreases with cycling (Figure 8(c)). According to the literature, [1,15,31], this decrease could be attributed to R-phase formation. It is worth **noting** that the reduction of values of E_{loading} , as well as σ_{Ms} , **becomes** more evident for the higher values of applied deformation.

3.3.2. Origin of residual strain

According to many researches, as function of the chemical composition of the considered alloy and also the strain amplitude, the residual strain can have two origins. On the one hand, the first origin can be ascribed to the plasticity (macro-plasticity) due to dislocation motions in the material and in the case of high levels of strain amplitude [32,33]. On the other hand, the residual strain is associated to the blocked martensite (micro-plasticity) in the parent austenitic phase at macroscopic free stress state [34,35]. This is true in the case of lower levels of strain amplitudes when the phase transformation is incomplete. However, plasticity may develop for low level strain amplitudes [8]. In order to distinguish between the two origins, a thermal flash test was proposed by Saint Sulpice et al., [36]. This test consists of heating the specimen up to 200°C for 30 seconds and following the evolution of the residual strain. Indeed, a rise of temperature leads the jammed martensite to transform back into austenite. If the residual strain decreases significantly, the physical origins of this strain are the blocked martensite [8]. Nevertheless, plasticity is an irreversible process at this temperature level. Thermal flash test was applied after a cyclic tensile test performed on the

same NiTi wires used in this work, for a strain amplitude of 7 % and at a strain rate of $1.3 \times 10^{-3} \text{ s}^{-1}$ after stabilization (i.e., after 100 cycles) and it has been observed that the reduction of residual strain after the test is low and so it is principally due to plasticity (Figure 9). This result is in accordance with observations made by Helbert et al., [2] in the case of 3% of strain amplitude and at a strain rate of $0.005\% \text{ s}^{-1}$.

3.3.3. Rate-dependant cyclic deformation

Cyclic stress-strain curves obtained in the 1st, 2nd, 20th, 60th and 100th cycles by controlling nominal strain at each strain rate as shown in Figure 10. As can be seen, there is a significant pseudoelasticity degradation, i.e., the transformation start stress and responding peak stress per cycle decrease, the residual strain accumulates and the hysteresis loop becomes narrower and narrower with the increasing number of cycles. For instance, martensitic yield stress increases with strain rate (529 MPa for a strain rate of $3.3 \times 10^{-4} \text{ s}^{-1}$ versus 631 MPa for a strain rate of $1.6 \times 10^{-2} \text{ s}^{-1}$). This increase can be assumed to the high heating due to the thermo-mechanical coupling related to the presence of the R-phase [2] and also to localization effects. As explained by Ortin and Planes [37], during forward phase change and because of the exothermic nature of martensitic transformation, the temperature of the alloy increases due to heat generation. So, the austenitic phase which is stable at high temperature becomes more stable and higher stress is required to drive the formation of martensitic during the direct transformation. In addition, during the reverse transformation, the temperature decreases making martensite more stable. Therefore, stress must be decreased further for the transformation from martensite back into austenite to continue. As a result, the slope of stress plateau (i.e., transformation hardening modulus) in the loading part increases monotonically with the strain rate. Thus, rate dependence seems to result from strong thermo-mechanical coupling, pseudoelasticity degeneration and temperature dependence [38]. Stress fluctuations (i.e. the irregularities observed before the flat plateau and before the reverse transformation) observed in Figure 10 are associated with the localization effect. Many studies have been conducted dealing with localization phenomenon in NiTi alloys and it has been shown that the nucleation of martensitic transformation fronts along the wire is eased when strain rate rises [30,39,40]. Moreover, it is worth to note that there is a clear independence of the residual strain *versus* strain rates for the high levels of strain rates (6.6×10^{-3} and $1.6 \times 10^{-2} \text{ s}^{-1}$), contrary to the case of lower strain rates (3.3×10^{-4} and $1.3 \times 10^{-3} \text{ s}^{-1}$) (Figures 10(a,b)).

It can be observed that the saturated value depends on the strain amplitude: the higher is the strain amplitude, the greater is the obtained saturated residual strain. Similar results have been obtained in previous researches [2,36].

3.4. Transformation ratcheting of NiTi wires at stress-controlled mode

Stress-strain curves obtained in the 1st and the 60th cycles when controlling nominal stress at a stress rates of 6.3 MPa.s⁻¹ and 315.6 MPa.s⁻¹ are shown in Figures 11(a) and (b), respectively. It is found that the dissipation energy per cycle (Δw) and the transformation start stress (σ_{Ms}) decrease when the number of cycles increases. Moreover, the peak strain per cycle increases, and the residual strain accumulates progressively during the cyclic loadings. Therefore, an obvious transformation ratcheting occurs in the cyclic tension-unloading tests of pseudoelastic NiTi SMA, and becomes more remarkable at higher stress rate (Figure 12). It is shown that the 'transformation ratcheting' occurring in the case of pseudoelastic NiTi is mainly caused by the cyclic accumulation of remained martensite due to the incomplete reverse transformation from the stress-induced martensite to original austenite. Here, it is to note that this phenomenon and its evolution depend greatly on the applied stress amplitude, mean stress and loading chart as previously reported in the literature [41].

In order to summarize and compare the cyclic behaviour in two modes of control, Figure 13 displays the evolution of cyclic behavior under stress-and strain controlled ($\sigma.c$ and $\epsilon.c$, respectively).

4. Conclusions:

The rate-dependent cyclic deformation of pseudoelastic NiTi wires was investigated by strain- and stress-controlled cyclic loading-unloading tests at various strain rates. A thermal analysis was carried out in order to qualify the studied alloy in terms of characteristic phases (austenite, martensite and the R-phase) and the corresponding phase changes. The initial austenitic microstructure of NiTi as well as the grains' size were revealed too. The following conclusions can be drawn:

(1) during strain-controlled cyclic tensile-unloading, there is a significant pseudoelasticity degradation, i.e., both the transformation start stress and responding peak stress per cycle decrease, the residual strain accumulates and the hysteresis loop becomes narrower and narrower with the increase of the number of cycles;

(2) the saturated value of residual strain depends not only on strain rate, but also on strain amplitude. The stabilization is reached rapidly when the strain amplitude is low, and it is accompanied with a decrease in the martensitic transformation yield stress. A 'flash thermal' test was performed in order to reveal the origin of this residual strain and it is shown that it is principally due to plasticity;

(3) the pseudoelastic NiTi alloy presents apparent transformation ratcheting under stress-controlled cyclic loading. Indeed, the dissipation energy per cycle and the martensitic transformation yield stress decrease, and the residual strain accumulates progressively during the cyclic loadings.

This experimental investigation is primordial for a better understand of mechanical behaviour of NiTi wires especially under repetitive loadings. Results can be taken further to examine and model localization phenomenon depending on strain rate under cyclic tensile loadings.

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