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A novel experimental method to assess the fatigue behavior of pseudoelastic NiTi wires

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ABSTRACT

A new experimental method for characterizing intrinsic uniaxial pull-pull pseudoelastic fatigue life of commercial NiTi wires is proposed. It consists in the generation of a virtual dog-boned shaped specimen combined with the use of snubbing type of gripping action in order to completely eliminate earlier failures induced by grip-sample interactions. The generation of a virtual dog-boned specimen is based on the localized character of the stress induced martensitic transformation and the evolution of the transformation stresses with further cycling. Reduced pseudoelastic cycles are performed to evaluate functional and structural fatigue with this method. Consistent longer fatigue lives have been obtained compared to traditional gripping methods with fractures occurring inside the region of interest. The present study clearly points to the role of the stress induced transformation in determining the fatigue life. This treatment of the structural fatigue considerable departs from classical approaches but takes into account essential facts like the localized character of the stress induced transformation, the heat effects associated with the first order solid to solid martensitic transformation and its impact on the critical transformation stresses.

1. Introduction

Pseudoelasticity is one of the distinguishing effects exhibited by shape memory alloys (SMA) [1]. The term refers to the capacity exhibited by these materials to be strained up to 8–10% strain in a nearly reversible manner. Responsible for this peculiar behavior is the existence of a stress induced martensitic phase transformation from an austenitic phase (A) to a martensite phase (M). Near equiatomic NiTi alloys belong to this kind of materials. In NiTi alloys the transformation takes place between a high temperature B2-structure and a low temperature B19'-structure [2]. Under uniaxial loading, in single crystalline or polycrystalline wires the stress induced transformation takes place once a critical stress σ_{A-M} is reached. Upon unloading, the transformation reverts, and this occurs at a stress σ_{M-A} lower than σ_{A-M} . The original specimen dimensions are nearly completely recovered in this way [3]. A stress hysteresis $\Delta\sigma$ can be then defined by the difference $\sigma_{A-M} - \sigma_{M-A}$ [4].

NiTi alloys have reached high technological relevance due to the excellent combination of properties such as high transformation stress, high level of strain recovery, adequate fatigue life, and excellent wear and corrosion resistance, including biocompatibility [5]. In many

applications based on the pseudoelastic behavior the martensitic transformation is induced repeatedly (pseudoelastic cycling) and therefore fatigue is an important issue. This is often the case with NiTi shape memory wires which are used in a wide variety of devices ranging from medical tooling to damping systems [6–8].

Pseudoelastic cycling results in a non-even decrease of the direct transformation (more pronounced) and reverse transformation (less pronounced) stresses with a consequent decrease in hysteresis and accumulation of permanent strain [9]. These effects are considered to be a consequence of what is referred to as functional fatigue and are concomitant with microstructural damage that accumulates during cyclic loading eventually leading to fatigue failure (structural fatigue) [10].

Several authors have studied functional fatigue in NiTi alloys under conditions of uniaxial tensile loading paying especial attention to the localized character of the stress induced transformation and the nucleation and movement of transformation fronts [11–14]. However, in most of these previous studies, only approximately 100 cycles were performed and after that it is usually considered that further development of functional fatigue becomes negligible. It is worth mentioning here that the tests just described were performed at a low strain rates in order to avoid the influence of thermal effects associated with latent

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heat of transformation on the critical stresses [15]. These tests are long lasting and for that reason are usually considered not appropriate for the study of structural fatigue.

On the other hand, the structural fatigue of pseudoelastic NiTi wires under high frequency rotary bending conditions has been intensively studied [16–19] in relation with the development of drilling tools for certain medical procedures. However, complex inhomogeneous stress – strain – temperature distributions develop along the radial position in superelastic NiTi wires under rotary bending conditions and therefore, the data generated with these procedures shall only be applied to predict fatigue life for similar geometries and testing conditions, i.e., these tests can be considered application specific.

The previous considerations indicate that there is a lack of an appropriate characterization of the intrinsic structural fatigue life of SMA elements under isothermal, or at least nearly isothermal, uniaxial tensile loading cycling conditions that reflects the intrinsic fatigue resistance of the material. These results could then be used to predict life under more complex loading conditions by considering appropriate modification factors, paralleling classical approaches used for structural materials. The first step in this direction is the development of an appropriate testing procedure.

An additional important aspect deserving further attention in the development of a fatigue testing procedure for pseudoelastic NiTi wires is the preservation of the wire surface. As fatigue cracks initiate in the near surface or surface regions [17] and the surface characteristics (oxidation, residual stresses, defects associated with wire drawing steps, etc.), strongly depends on the details of the previous fabrication procedures, an appropriate characterization should be performed on specimens containing the original wire surface. This requirement leads to discard considering the use of standard dog-bone shaped fatigue specimens, i.e., wires with a central reduced diameter section obtained using machining or spark eroding procedures (see for example, ASTM E466 standard, [20]). Such dog-bone specimens would allow the use of standard wedge devices for gripping. However, the need to characterize the effect of the original wire surface obliges using specimens of uniform cross-section, i.e., specimens with the original (as-fabricated) wire diameter. For such type of specimens, direct wedge gripping cannot be used due to early failure favored by high stress concentration points in that area.

In the present work, a novel method for assessing the intrinsic fatigue of pseudoelastic NiTi wires, including the as-fabricated surface is proposed. It eliminates the need of using geometrically dog-bone specimen by introducing what will be referred to as a virtual dog-bone specimen. Complementary, the acceleration of fatigue damage in the contact area between specimen and grips is prevented by using snubbing type of grips. Preliminary results corresponding to uniaxial pull-pull pseudoelastic cycling are presented to support the viability of the proposed method.

2. Materials and devices

The studied material consisted in commercial Ni rich NiTi (50.9 at. % Ni) 0.5 mm nominal diameter wires provided in the so-called straight-annealed and black oxide surface condition by SAES Getters. The microstructure was similar to the one described in [17], i.e., ultrafine grained with grain size in the range 40–50 nm. The manufacturer specification indicated an A_f temperature of -15°C (fully annealed). Electric resistance vs. temperature measurements determined that the wires were in austenitic phase at room temperature [21]. The specimens had a total length of 140 mm with a free length between grips $L_0 = 60$ mm. All mechanical tests were performed in a dual-column Instron 5567 electromechanical testing machine equipped with a temperature chamber Instron 3119 and instrumented with a 1 kN full-scale Instron load cell and an optical encoder for crosshead displacement determination. An apparent strain ϵ_{CH} (CH-crosshead) was obtained by dividing the crosshead displacement by L_0 . The

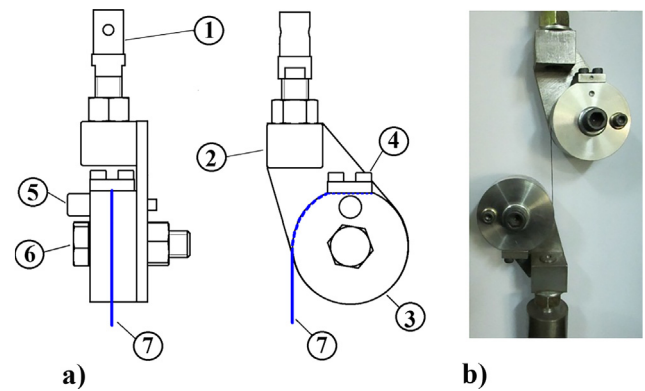


Fig. 1. (a) Diagram of the gripping device used for tests. 1. Connector, 2. Body, 3. Cylinder/Pulley, 4. Clamp, 5. Lock, 6. Screw, 7. Wire rolled over cylinder. (b) Set-up used in the present work.

temperature used for the tests was $T = 37^\circ\text{C}$. Tests were conducted under crosshead displacement rate. The rates considered in the present experiments were 0.1 or 1 mm/min. Indicative equivalent global strain rates can be calculated by dividing these values by L_0 ; the equivalent global strain rates are then $2.77 \cdot 10^{-5} \text{ s}^{-1}$ and $2.77 \cdot 10^{-4} \text{ s}^{-1}$, respectively.

The special gripping device illustrated in Fig. 1, was used. It is similar to the snubbing type grips recommended in standard ASTM E8M [22] for tensile testing thin wires. It was selected here because it provides gentler gripping action in comparison with classical wedge type devices, thus contributing to eradicate early failures and allowing testing of constant cross-section specimens. The diameter of both gripping pulleys is 50 mm which produces an initial strain of 1% in the wires of 0.5 mm of diameter which is just below the characteristic critical value for a stress induced transformation in ultrafine grained pseudoelastic NiTi wires [3,23]. A shallow v-shaped notch precisely machined along the perimeter assures a precise alignment of the specimen and also increase the wire – pulley contacting area favoring the gradual load transfer between them. The cylindrical pulleys are removable to facilitate specimen placement.

3. Experimental method

3.1. Generation of a virtual-dog-bone (VDB) specimen

The experimental method is based on using the localized character of functional fatigue in NiTi in order to study intrinsic fatigue resistance. In this section, the creation of a virtual dog-boned specimen (VDB) will be explained.

Fig. 2(a) and (b) illustrate the behavior of a NiTi wire subjected to 1 and 100 pseudoelastic cycles under quasistatic condition, respectively [13,24]. Red lines (internal cycles) in Fig. 2(a) and (b) represent the expected behavior of the stresses when the sample is cycled between limits ϵ_{\max} and ϵ_{\min} (reduced cycle). It can be observed that after 100 cycles, the stress levels associated with both transformation plateaus decrease and the amount of non-recoverable strain increases. Furthermore, the effects of functional fatigue can be concentrated in a certain zone of the material due to the localized behavior of martensitic transformation [3]. It is worth mentioning that the overstress observed in cycle 1 and cycle 100 is due to the nucleation of a new transforming domain [14,25]. These effects are the basis of the proposal.

The VDB is generated by performing a sequence of cycles in which the transforming zone is carefully controlled. Restricting the transformation to a specific zone results in functional fatigue in that zone. This in turn further favors the transformation activity to take place in that zone due to the decrease in transformation stress associated with cycling [24].

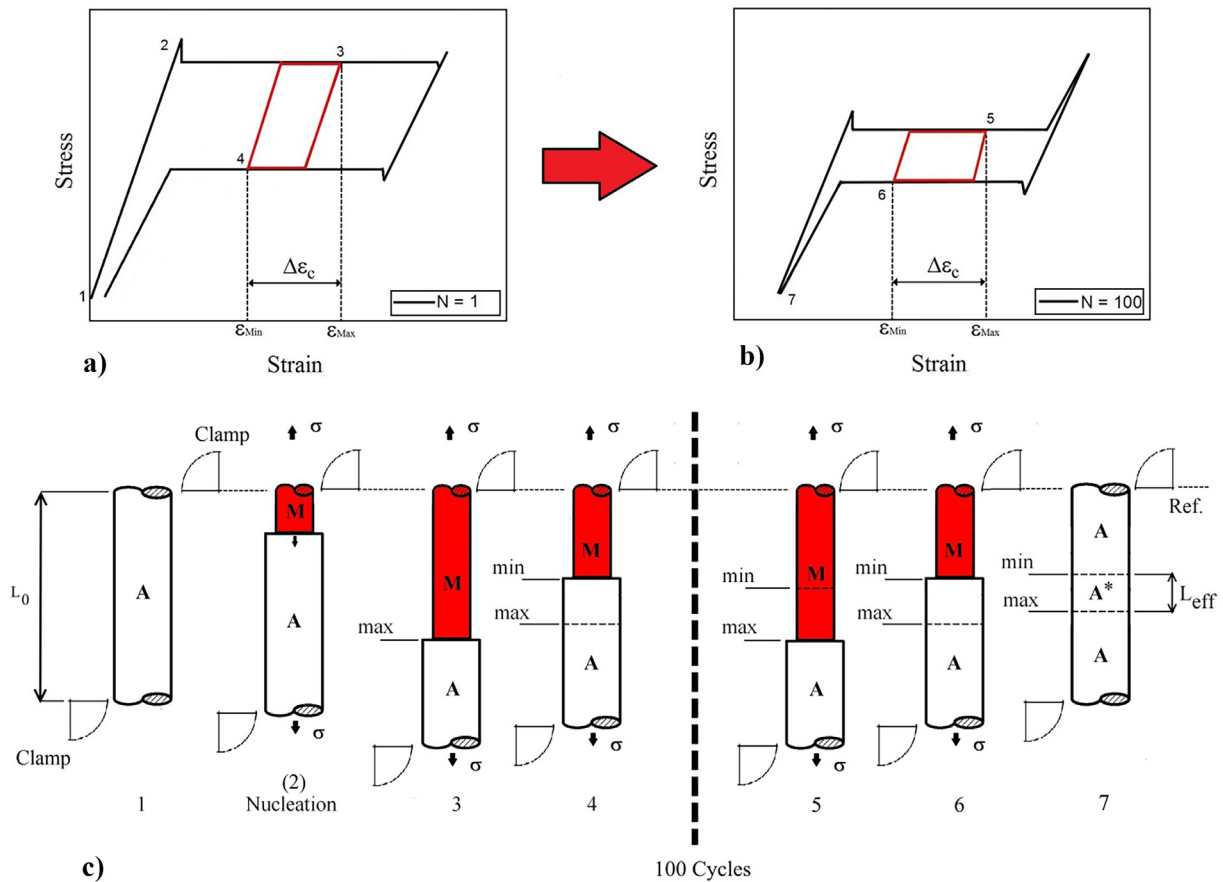


Fig. 2. Schematic drawings of the proposed experimental procedure for the VDB specimen generation: (a) First pseudoelastic cycle and strain limits of the VDB. (b) Pseudoelastic cycle $N = 100$. (c) Evolution of the martensitic transformation.

Fig. 2(c) depicts the evolution of the interface of martensite and austenite along the wire length during the VDB generation. The upper end of the specimen is fixed as a reference to show the relative movements of the other sections of the sample, including those that define the transformation interface. The zones of the wire in austenitic (A) and martensitic (M) phases are indicated. At first, the wire is tensile stressed and the nucleation of martensite takes place (point 2). When the cycle reaches point 3 (ϵ_{Max}) the martensitic transformation front has moved through the material to a maximum position. Then, the specimen is partially retransformed until point 4 (ϵ_{Min}) and the transformation front is located at the minimum position. As a result, the wire has a martensitic region (red zone in point 4 of Fig. 2(c)), and a partially retransformed region between the limit positions of the transformation front (*min*–*max*). The range of strain between points 3 and 4 ($\Delta\epsilon_c = \epsilon_{Max} - \epsilon_{Min}$, see Fig. 2(a)) is applied based on the selected effective test length (L_{eff}) for the VDB.

The next step consists in cycling the sample 100 times between ϵ_{Min} and ϵ_{Max} . The stress range decreases by functional fatigue [3,26] as shown by the red line in Fig. 2 (b). If the crosshead speed is low enough and the test temperature is constant the transformation front will move between positions *max* and *min* (point 5 and 6 of Fig. 2(c)). After these cycles are completed, the decrease in the critical stress for the direct transformation (inside the zone denoted A^*) becomes greater than the overstress necessary to nucleate a new transforming domain [14,25]. Specimen is then unloaded up to point 7.

It is very important to point out here that the relevant parameter for controlling the experiments is the displacement rate and not the strain rate, as might be supposed from the experience with fatigue of conventional materials. In effect, as the stress induced transformation is localized, the velocity at which the transformation fronts propagate is

directly related with the crosshead displacement rate. The front velocity determine the local temperature variation at the austenite-martensite interface and this local temperature in turn determines the critical stress for transformation through a Clausius–Clapeyron type of relationship between temperature and critical transformation stresses. Details can be found elsewhere [12,14,15]. These previous mentioned experience indicates that a displacement rate of 0.1 mm/min assures, on the one hand, that the stress induced transformation proceeds under isothermal conditions and, on the other hand, avoids the nucleation of additional transformation domains outside the region of interest. This is the displacement rate adopted for obtaining the VDB specimen for the present conditions (wire diameter 0.5 mm, still air). Therefore, under this well controlled quasistatic condition, a unique B2/B19' interface can be kept moving forth and back with further cycling while thermal effects that might favor the nucleation of additional interfaces out of the region of interest remains negligible [24].

As a consequence of this type of cycling, a particular region of the sample has been subjected to functional fatigue and has now lower transformation stresses. Thus, further loading will result in the nucleation of a transformation front only within this zone (L_{eff}). The VDB is then created and can be used to study the intrinsic fatigue of the material.

3.2. Verification of the existence of a virtual-dog-bone

Before starting the pseudoelastic fatigue test itself, it is possible to check the correct creation of the VDB. The verification can be done by applying one cycle with a strain amplitude $\Delta\epsilon_c$ at a low crosshead displacement rate (0.1 mm/min). The cycle in Fig. 3(a) ($N = 101$) illustrates the behavior expected when a pseudoelastic cycle of the zone

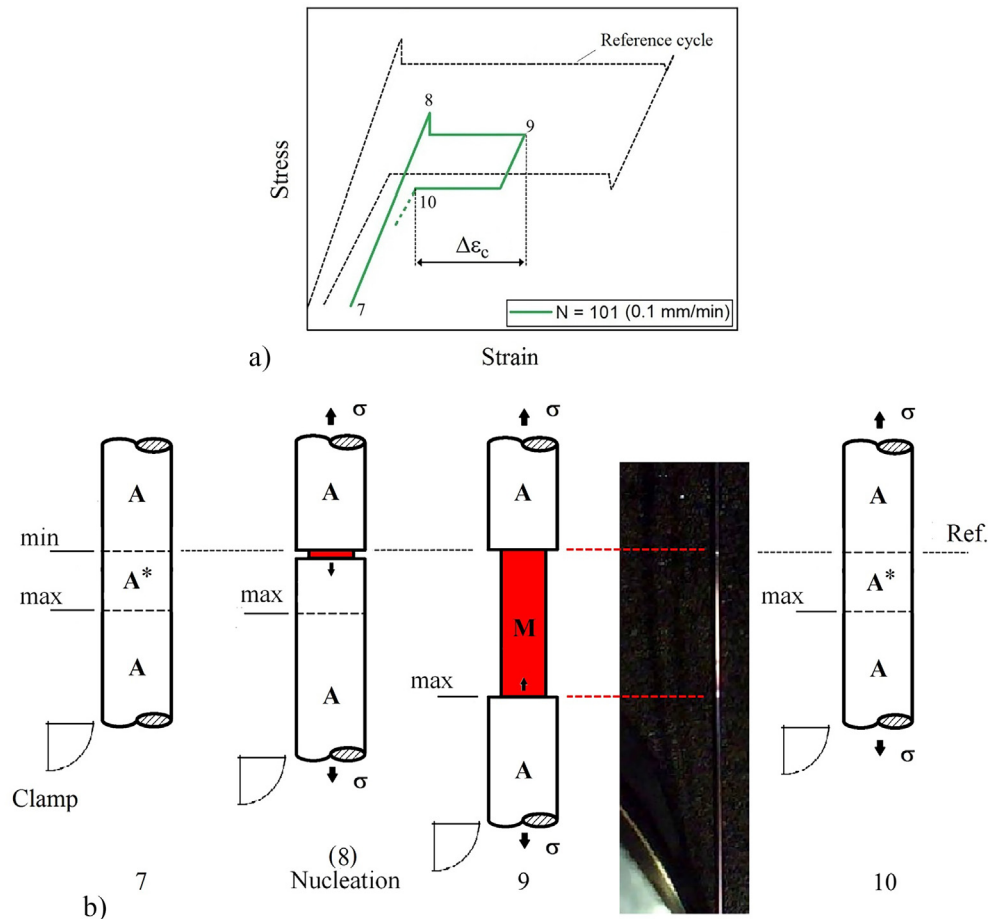


Fig. 3. Verification of the VDB specimen. (a) Applied cycle. (b) Experimental verification of the localization of the martensitic transformation.

A^* takes place. A complete cycle in the wire without previous martensitic transformation can be observed with a black dashed line as a reference. If other martensite domains nucleate outside of A^* several stress peaks would appear in the transformation plateau of the curve. Only one peak (see point 8) would confirm that martensite was nucleated inside the effective test zone.

Fig. 3(b) shows schematically the position evolution of the interfaces of the austenitic-martensitic transformation, where the reference line is now set with the upper limit of the A^* zone. If the procedure for the VDB specimen generation is successful, a single transformation front will move during the entire cycle as shown in Fig. 3(b).

3.3. Considerations for fatigue testing

Once the verification of the formation of the VDB specimen is done, the pseudoelastic fatigue test can be started. There are several possible configurations that can be chosen according to the aim of the study: full or reduced cycling and low or high displacement rates.

In the case of full pseudoelastic cycling, the transformation is performed within the total effective length given by the zone A^* (Fig. 2(c)). This type of approach can be adopted to analyze thermal effects and the presence of multiple transformation fronts. When applying a low speed, a single transformation front appears and moves through the entire cycled zone. Whereas at a high displacement rate, multiple domains of martensite could appear due to thermal effects and if this happens, those multiple transformation and retransformation fronts will move in the test zone [12,15]. For this configuration the displacement rate should not be greater than 10 mm/min in order to prevent the nucleation of martensite outside L_{eff} .

The case of reduced pseudoelastic cycling involves holding a core of

martensite transformed on a certain zone of the sample. Several devices based on NiTi subject the material to this type of loading. It is recommended that the displacement rate in this case does not exceed 1 mm/min in order to ensure that the transformation remains in the same area. Also, if this speed increases, new martensite domains could nucleate along the testing length L_{eff} , thus altering the testing conditions [12,15].

The increase of the displacement rate from the 0.1 mm/min used in the previous stage (generation of the VDB specimen) to 1 or 10 mm/min for the fatigue testing stage results in a slight departure from isothermal conditions with its consequent variation of the transformation stresses. However, this moderate increase in displacement rate is compulsory for performing fatigue testing in realistic practical times (several days or weeks per specimen).

It is important to remark that the maximum stress is controlled through the selected temperature and maximum applied strain. The minimum stress is related to the selected temperature and the evolution of the retransformation stress.

Taking into account these considerations, the characteristics of the effective test zone can be controlled allowing a suitable intrinsic fatigue study of the alloy. Parameters such as applied load and number of cycles can be monitored without external effects.

4. Results

In order to illustrate the correct operation of the proposed test method, three reduced cyclic tests with the same configuration were performed. Fig. 4 shows the procedure in a tested specimen (Test 1). A first complete transformation cycle of the virgin wire (without previous martensitic transformation) can be observed with a black dashed line as

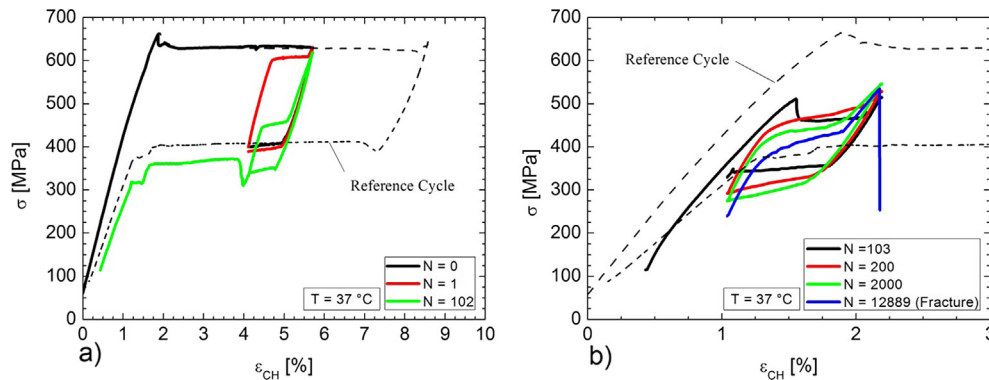


Fig. 4. Test 1: (a) VDB generation (0.1 mm/min). (b) Fatigue Test (1 mm/min).

Table 1

Fatigue tests parameters and resulting fatigue lives N_f .

Test	$\Delta\epsilon$ (%)	σ_{max} (MPa)	σ_a (MPa)	N_f
1	1.16	525	138	12,889
2	1.16	568	135	6418
3	1.15	542	126	11,750

a reference.

Fig. 4(a) presents the generation of the VDB using a displacement rate of 0.1 mm/min. This test had a L_{eff} of 10 mm which results in strain limits of 4.06% and 5.66%. During the final unloading the transformation front advances outside of the cycled zone (the zone denoted A* in Fig. 2 (c)). As a consequence, a stress peak can be observed at about 4% strain ($N = 102$). The outside region has only experienced one pseudoelastic cycle: loading during $N = 0$ and unloading after cycle $N = 102$. This behavior confirms the localized character of the martensitic transformation and the fact that the pseudoelastic cycling was performed only on the selected zone A*.

The experimental verification of the existence of the VDB can be observed in cycle $N = 103$ of the plot presented in Fig. 4(b). This cycle was realized at a displacement rate of 0.1 mm/min (equivalent global strain rate $2.77 \cdot 10^{-5} \text{ s}^{-1}$). As stated previously, the presence of only one transformation peak demonstrates the correct generation of the VDB. Cycle $N = 103$ also shows that no previous formation of martensite occurred in any other part of the sample. Furthermore, the shape of transformation plateau demonstrates that only one transformation front advanced, so that an isothermal behavior can be assumed [13].

Fatigue testing was performed applying a crosshead displacement rate of 1 mm/min (equivalent global strain rate $2.77 \cdot 10^{-4} \text{ s}^{-1}$). The maximum applied strain value for cycling (2.19%) was chosen for the application of a maximum stress of 500 MPa, similar to the nucleation stress for the cycle $N = 103$. The minimum applied strain value (1.04%) was selected in order to avoid the complete pseudoelastic retransformation.

Table 1 shows the experimental conditions and the fatigue lives N_f obtained. In all tests fracture took place in free length between grips in the cycled region, thus indicating that that interaction between grips and specimen does not play any role on the structural life determined with the method here proposed.

Finally, in order to analyze the ability of the snubbing device shown in Fig. 1 to avoid fatigue fracture at the gripping zone, five fatigue test were performed with a VDB specimen using classical wedge type gripping method. The obtained fatigue life was systematically close to 2000 cycles for all tests, a factor between 3 and 6 times shorter than the fatigue lives obtained with the snubbing device for otherwise identical cycling conditions. In addition, it was verified that in these comparative tests, the fatigue fracture took place in the specimen - wedge grip contact zone, far from the effective fatigue zone of the VDB specimen.

This allows concluding that both the generation of a VDB specimen plus the use of a gentle gripping method is essential to perform adequate intrinsic fatigue assessments. Therefore, the VDB method can be considered successful when analyzing intrinsic fatigue of NiTi pseudoelastic wires.

It is worth noticing here that the critical stresses continue to decrease along the whole test (Fig. 4(b)). This behavior would indicate that functional fatigue is still active even besides that the most evident evolutions occur during the generation of the VDB specimen (100 cycles). This is an important aspect that will be addressed in a further work but clearly indicates that functional fatigue effects are not exhausted after performing a couple of hundreds cycles as it is usually assumed in the literature.

5. Final remarks

A new method to study intrinsic pseudoelastic fatigue life of commercial NiTi wires was presented. It consists in obtaining a “virtual dog-bone” shaped specimen and using a special gripping device. This method allows the test zone to be kept away from the grips taking advantage of the localized functional fatigue of the material. Thermal issues related to the martensitic transformation were prevented by controlling the displacement rate. Uniaxial fatigue tests were carried out in order to validate the method. In all cases, fracture took place in the free length between grips and can be considered representing the intrinsic fatigue life of the studied specimens subjected to pull-pull uniaxial cycling conditions through the stress induced martensitic transformation.

By adopting the method here proposed, the true structural fatigue can be assessed. In the present work, few examples were given which clearly demonstrates that longer fatigue lives are obtained in comparison with direct gripping methods where premature failure are induced. An in depth characterization of fatigue of NiTi wires using the method here proposed constitutes the objective of further studies. It is important to mention here that the developed method would also allow characterizing the functional fatigue life until final fracture.

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