Stress Serration and Arch-shaped Lüders Stress Plateau Behaviour of Ti-50.8 at.% Ni Wire Prepared by Selective Electrical Resistance Over-aging

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Abstract

Joule heating of NiTi shape memory alloy wires is a commonly applied technique for heat treatment and shape setting in many applications. Another innovative use of this method is to produce functionally graded NiTi. In this study, NiTi wires with spatially varied shape memory characteristics along the length were created by electrical resistance over-aging of a Ni-rich superelastic NiTi alloy. The stress-strain behaviour of such wires exhibited some new and unique characteristics during the stress-induced martensitic transformation, including two discrete stress plateaus, stress serration during transition between the two stress plateaus and an arch-shaped stress plateau in the over-aged section. These unique features have direct implications to design using NiTi alloys and the underlying mechanisms are explained in this study.

Keywords: Shape memory alloy (SMA); NiTi; Joule heating; Martensitic transformations; Pseudoelasticity; Lüders deformation

1. Introduction

Near-equiatomic NiTi alloys are attractive for a wide range of smart designs and innovative applications owing to their unique functional properties, including the shape memory effect and pseudoelasticity [1-3]. These functional properties are related to the martensitic transformation in these alloys [4, 5]. It is well established that tensile deformation of slender NiTi components exhibits distinctive Lüders-type deformation over the stress-induced martensitic transformation [6-8]. The Lüders-type deformation is characterised by some unique features, including an upper-lower yielding phenomenon, a flat stress plateau and a definitive strain range [9-11]. These features provide unique design opportunities for certain applications, whereas in some other cases it can be undesirable and requires modifications [12]

For a given NiTi alloy, the deformation behavior and transformation behavior are known to be sensitive to heat treatment conditions [5, 13, 14]. The general effects of heat treatment have been well understood and documented in the literature. For near-equiatomic NiTi alloys, the dominant effect is the temperature of anneal after cold work [15, 16]. These alloys generally exhibit limited pseudoelastic behavior under partially annealed conditions and perfect shape memory behavior when fully recrystallized. For Ni-rich NiTi alloys, the dominant effect is related to the process of precipitation ageing, which is influenced by alloy composition, ageing temperature and ageing time [6, 17-20]. These alloys generally exhibit superior pseudoelasticity under well aged conditions. Precipitates that may form in these alloys include Ti_3Ni_4 , Ti_2Ni_3 and TiNi_3 , with Ti_3Ni_4 being coherent with the matrix and the most important in causing the R phase transformation and pseudoelasticity [5, 6, 17, 18]. Over ageing at higher

temperatures encourages conversion of the coherent Ti_3Ni_4 into the incoherent Ti_2Ni_3 and in extreme cases into TiNi_3 , causing the loss of pseudoelasticity [21].

Whereas most of the fundamental understanding of the effects of heat treatment has been achieved historically for homogeneous heating conditions, as in the case of inside a furnace, some more complex heat treatment conditions are becoming more frequently used, for example joule heating of thin wire materials [22-24], laser scan for surface heating of thin films [25] and thin plates. Such heat treatments impose unique characteristics, for example extremely high heating rates up to $3500\text{ C}^\circ/\text{s}$ [26] and gradient temperature profiles along the length of a wire [22] or through the thickness of a plate [27]. It has also been reported that laser welding of austenitic NiTi can cause the occurrence of martensite in the heat-affected zone and in the fusion zone at room temperature, due to the compositional variation and change of local transformation temperatures in these sensitive regions during the welding process [28, 29]. The superelastic behaviour of laser welded NiTi was also studied recently [30]. Joule heating of NiTi wires is also used for some specific applications, such as shape setting for artificial biometric fingers [31] and microelectromechanical system (MEMS) devices [32, 33]. Such unconventional heat treatment techniques also offer opportunities to create novel properties, such as functionally graded NiTi materials [22, 34].

In an effort to design self-navigable smart biopsy rotating needles [35], we envisaged a design of NiTi thin tubular device with varied properties along its length. The tip or certain section of the thin tube may need sufficient flexibility for easy bending, in order to achieve active insertion direction control through soft tissues. In the meantime, the reminder of the device must remain high strength and superelasticity to fulfil its functions. This is achieved by locally heat treating a pseudoelastic NiTi thin wall tube by joule effect to cause over-ageing to suppress locally the pseudoelasticity. Such heat treatment is anticipated to cause some unique material property characteristics, such as discrete and non-flat stress plateaus for stress-induced martensitic transformation upon loading [36]. In this paper, we report an analysis of these phenomena to delineate the mechanisms. Such knowledge is of generic interest for other treatments and applications where local heating is involved.

2. Experimental procedure

A commercial pseudoelastic Ti-50.8 at.% Ni alloy wire of 0.9 mm in diameter was used. The wire was cut into samples of 60 mm in length. Local heat treatment was performed by means of electrical resistance heating (joule heating) using a DC laboratory power supply. The heating section between the electrodes was 30 mm. The temperature profile along the length within the heating section was measured by thermocouples spot welded on the surface of the sample. Deformation behaviour of the samples was characterized in tension using an Instron 4301 universal testing machine at a strain rate of $3.33 \times 10^{-5}\text{ s}^{-1}$ at room temperature (300 K). The propagation of martensite band during the tensile deformation was recorded using a digital camera.

3. Results and discussion

3.1 Temperature gradient of joule heating

Figure 1(a) shows a schematic view of the dimensions of the wire samples. The heating length

(30 mm) is a middle section of the wire sample defined between the two electrodes used for joule heating. The gauge lengths (GL) indicated are the gauge lengths for tensile deformation testing. It is seen that the samples were tested under three different gauge length conditions (GL=20 mm, GL=30 mm and GL=40 mm), to be within, equal to and wider than the heating length (30 mm), to delineate the contributions of different sections to the mechanical behavior.

Figure 1. (a) Schematic view of the dimensions of the wire samples used for selective electrical heating and for tensile test; (b) temperature distribution profiles along the heating gauge length of samples; (c) distribution of the upper yielding stress, σ_u , and the plateau stress, σ_p , along the heating length; (d) schematic view of the initiation and propagation of martensite bands within the heating length at 6 A and 7 A.

Figure 1(b) shows the temperature profiles along the heating length as measured by a number of thermocouples attached to the sample at various locations. The temperatures were measured during electrical aging and the values shown in the figure represent the maximum temperatures reached at two different current levels (6 A and 7 A). The distance values, denoted x , were measured from the center of the wire. The temperature profiles showed a bell shape at both 6 A and 7 A current levels, with the peak temperature T_p being in the middle, indicating the dominant 1D heat dissipation along the length towards the electrodes. It is obvious that the highest temperature gradient occurred at near the ends of the heated section. The sharp gradient originated from the thermal sink effect of the electrodes (two blocks of brass).

3.2 Unique deformation characteristics of joule heated wire

Figure 2 shows the deformation behaviour of two NiTi wires after electrical over-aging at 6 A and 7 A with a 40 mm deformation gauge length. The stress-strain curves of an as-received and a solid solution treated sample (at 1123 K for 1.8 Ks) are also shown for comparison. The as-received sample exhibited perfect pseudoelasticity at room temperature, with a forward stress plateau at $\sigma_{SIM} = 430$ MPa and a plateau strain of 7.6 % (including the elastic strain). Upon unloading, the sample exhibited a permanent plastic strain of 0.3%. The solid solution treated wire exhibited a shape memory effect at room temperature, with the forward stress plateau at $\sigma_{SIM} = 317$ MPa. For the electrically heated wires (at both 6 A and 7 A), the stress-induced martensitic transformation occurred over two stress plateaus. It is obvious that the upper stress plateau was accompanied by pseudoelastic recovery and the low stress plateau showed no pseudoelastic recovery upon unloading.

It is also evident that the electrically heat treated samples showed a clear upper-lower yielding behavior at the onset of the first stress plateau, whereas such phenomenon was absent for the solution treated and the as-received samples. This phenomenon is commonly observed in Lüders type deformation of NiTi in tension [9]. The upper yielding is associated with the initiation of the Lüders band due to the stress-induced martensitic transformation. The lower stress level, i.e., the plateau stress, is associated with the propagation of the transformation Lüders band. Its occurrence demonstrates that a higher stress is required to initiate the localized Lüders type deformation band than to propagate it [37, 38]. This upper-lower yielding and flat plateau behavior is also closely related to the strain rate of the testing. Tobushi et al. has reported that when the strain rate is small (≤ 1 %/min), there is enough time for the interface (between martensite and austenite) to move after the creation of the nucleus and therefore the high stress required to move the interface is relaxed. However, in the case of large strain rates, the interface moves quickly after the creation of the nucleus with very little time to relax the stress at the interface. The internal friction resistance against movement of the interface increases, and thus the upper yield at the start point of the transformation does not appear [39]. The strain rate of the current testing is considered very small ($3.33 \times 10^{-5} \text{ s}^{-1}$) thus exhibiting the upper yield point and flat plateau on the stress-strain curve. For the electrically heated samples, the band was nucleated in the middle of the sample, which is much weaker than the unheated sections at the ends of the sample, thus showing the expected upper-lower yielding behavior. For the as-received and the solution treated samples, which had uniform properties over the entire length of the wire, the stress-induced martensitic transformation had already occurred within the grips during sample mounting prior to the tensile test, thus showing the absence of the upper yielding point but only the lower stress plateau behaviour.

Figure 2. Deformation behaviour of the as-received, solid solution treated and electrically heated NiTi wires at 6 A and 7 A. The tensile deformation gauge length is 40 mm.

3.3 Critical stress for inducing the martensite

For the sample heat treated at 6 A, the first stress plateau occurred at $\sigma_{SIM}^1 = 260$ MPa and the second one at $\sigma_{SIM}^2 = 420$ MPa. The higher stress plateau was at the same level as that of the as-received sample and was associated with the unheated section (outside the heated section) but within the deformation gauge length. The deformation over the lower stress plateau did not show any pseudoelastic recovery. This is attributed to over-aging which has led to conversion of the coherent Ti_3Ni_4 precipitates into incoherent precipitates (Ti_2Ni_3 , $TiNi_3$) [18, 40-42]. The stress level of the lower plateau was below that of the solution treated sample. This is attributed to the higher Ni content in the matrix of the solution treated sample, which had been heated to a higher temperature. Higher Ni content implies lower M_s temperature, thus higher stress for inducing the martensitic transformation at the room temperature.

After unloading, this sample exhibited a residual strain of $\varepsilon_r^6 = 4.6$ %, as seen in Figure 2. Assuming a small plastic deformation same as for the as-received sample ($\varepsilon_{plastic} = 0.3$ %), the residual strain associated with unreverted martensite can be estimated to be 4.3 %. It is also evident that this sample showed a spontaneous strain recovery of 4.9 % upon unloading, which includes both elastic recovery and pseudoelastic recovery. The elastic recovery strain may be estimated using the Young's modulus of the martensite (being estimated as $E=100$ GPa) [43-46] as $\varepsilon_{elastic} = \frac{\sigma_{SIM}^2}{E} = \frac{420}{100 \times 10^3} \approx 0.4$ %. This leaves 4.5 % as the pseudoelastic recovery strain. Based on this, the effective length of the over-aged section can be estimated as $L_{over-age}^6 = \frac{4.3}{4.3+4.5} \times 40 \text{ mm} \approx 19.5 \text{ mm}$, or approximately $-9.7 \text{ mm} < x < 9.7 \text{ mm}$.

A similar calculation was also applied to the 7A sample, which gave the effective length of the over-aged section $L_{over-age}^7 = 22.2 \text{ mm}$, or $-11.1 \text{ mm} < x < 11.1 \text{ mm}$. Applying these to Figure 1 (b), the critical temperature for the suppression of pseudoelasticity by over-ageing is estimated to be $T_c^o = 815 \text{ K}$. This temperature is consistent with the previously reported values (e.g., 833 K for the Ti - 50.8 at.% Ni [17]) for over-aging of NiTi. Considering that the section of the sample outside the heated section has a higher (propagation) stress for inducing the

martensitic transformation and the heated section has a lower stress for inducing the transformation, the plateau stress profile along the length of the wire can be expected to be as expressed by the black curve, denoted σ_p^{6A} , seen in Figure 1 (c). The σ_p^{6A} level within the heated section is 260 MPa and that in close vicinity to the electrodes is 410 MPa, as obtained from the lower and higher plateau stresses of the sample heated at 6A shown in Figure 2.

It is also seen that the sample exhibited an upper yielding stress associated with the initiation of the deformation Lüders band. This stress, denoted σ_u^{6A} , is higher than σ_p^{6A} , and is thus schematically shown as the black dashed curve above σ_p^{6A} in Figure 1 (c). The σ_u^{6A} level within the heated section was 330 MPa as obtained from the upper yielding stress of sample heated at 6A shown in Figure 2. The σ_u^{6A} of the unaffected regions close to the electrodes was considered higher than 430 MPa which was the plateau stress of the as received sample. Due to the absence of the upper yielding stress during the tensile test of the as received sample (initiation of martensite band had occurred during gripping prior to testing), the maximum value of σ_u^{6A} was assigned an estimated value of 480 MPa, i.e. 50 MPa above σ_p^{6A} as for the over-aged section.

For the sample heated at 7 A, the first stress plateau proceeded in an unusual arched shape. This is a common observation of a few samples heated within a narrow current range. The maximum point of the arch was close to that of the solution treated sample. Referring to Figure 1 (b), it is seen that the middle section of the wire had been heated to higher temperatures, and is thus expected to have higher Ni contents due to the increased dissolution of the Ni-rich precipitates. Higher Ni content implies lower M_s temperature, thus higher stress to induce the martensitic transformation at the room temperature. Considering this, σ_p of the 7 A heated sample (σ_p^{7A}) can be expressed in the red curve, which exhibits two minimal stress valleys of 260 MPa, as shown in Figure 1 (b). The red dashed curve shows the initiation stress (upper yielding stress) for the deformation band, σ_u^{7A} . The σ_u^{7A} at the stress valleys was 330 MPa obtained from the upper yielding stress of the sample heated at 7A shown in Figure 2.

3.4 Flat and arched stress plateaus for stress induced martensitic transformation

For the sample heated at 6 A, upon loading in tension, the deformation band for stress induced martensitic transformation will nucleate when the stress reaches σ_u^{6A} within the over-aged section and then propagate across the over-aged length, as shown in Figure 1 (d). Thus, a stress plateau at σ_p^{6A} is observed.

For the sample heated at 7 A, upon loading, the deformation band for stress induced martensitic transformation will nucleate when the stress reaches σ_u^{7A} . The first band will nucleate at either of the two minima on the σ_u^{7A} curve, and then propagate from low to high along the σ_p^{7A} curve. It is seen in Figure 1 (c) that the maximum value of σ_p^{7A} is below the minimum value of σ_u^{7A} . That means when the first formed band is propagating, even with increasing stress over the arch, no other band can nucleate. The propagation continues until the band reaches the second minimum on the σ_p^{7A} curve. Thus, an arch-shaped stress-strain curve is observed over the lower stress plateau associated with the stress induced martensitic transformation within the

heated section.

3.5 Stress serrations

As seen in Figure 2, both samples showed serrated stress-strain curves at the transition between the two stress plateaus, as indicated by the arrow. To investigate this phenomenon, several samples were treated at 6 A. The samples were subjected to tensile deformation with different gauge lengths symmetrical about the centre of the samples, as schematically illustrated in Figure 1 (a). The stress-strain curves of the tests are shown in Figure 3. It is seen that stress serrations occurred for samples with 30 and 40 mm deformation gauge lengths. The sample deformed with 20 mm gauge length showed only one stress plateau with no stress serration. Therefore, it is evident that the stress serrations were associated with a narrow section along the length of the sample on each side of the over-aged zone. These are the regions where the temperature gradient is the greatest.

Figure 3. Deformation behaviour of wires electrical heated at 6 A of different gauge lengths

To observe the phenomenon further, the nucleation and propagation of martensite Lüders band during the tensile deformation of the wire was recorded by video. Snapshots at 10-second intervals of the loading process of the 6 A heated sample were extracted and shown in Figure 4 (a). The dashed curve in the centre across all the snapshots indicates the trajectory of the middle point of the gauge length. The arrows indicate the fronts of the martensite transformation Lüders band. It is seen that the martensite nucleated at the lower middle section of the wire. The fronts of the transformation band propagated towards both ends of the wire during loading. The total length of the Lüders band, measured as the distance between the two transformation fronts, was determined from the snapshots and shown as a function of time of the tensile deformation in Figure 4 (b). The time positions of six characteristic moments (1-6) are also indicated on the stress-strain curve shown in Figure 3. It is seen that the length of the Lüders band increased linearly initially with the time (for a cross-head speed controlled tensile deformation) before 130 s. This section corresponds to the initiation and propagation of the primary Lüders band within the over-aged section. Upon further loading (time increases from 130 to 160 s), the rate of expansion of the primary Lüders band is reduced, displaying a small plateau on the curve. Under the condition of constant strain rate tension, the reduced strain contribution from the slower expansion of the primary Lüders band is compensated by the increase of elastic strain throughout the wire sample due to the increase of stress between

points (3) and (4) on the stress-strain curve of 6A sample (black curve in Figure 3). The continuous but slower propagation of the primary Lüders band proceeded for a short distance beyond the over-aged section with the rapid increase of σ_p^{6A} and stopped until σ_u^{6A} was reached, which triggered the nucleation of a new Lüders band just in front of the primary band and caused the first stress serration on the stress-strain curve (Figure 3, point (4)). At this moment the total length of the Lüders bands suddenly increased again upon loading. Due to the fact that this region where the new Lüders band is formed has a very high anneal temperature gradient (Figure 1 (b)), the newly formed Lüders band has a very limited space to propagate before the stress is becoming too high again and a second new Lüders band is nucleated on the other side of the primary Lüders band, causing the formation of the second stress serration (Figure 3, point (5)). Such process may continue as seen from 170 to 210 s on the stress-strain curve until the Lüders band deformation has reached the original portion of the wire sample outside the heated section. It is also visually evident in Figure 4 (a) that at the moments of stress serration, new Lüders bands had nucleated and propagated for a very short distance within the narrow sections close to the electrodes. Referring to Figure 1 (b), these sections have the highest temperature gradient. The inset of figure 4(a) shows an enlarged image of the new Lüders band (from the lower end of the wire) emerging and propagating within this high temperature gradient region.

Given the above evidences, it is easy to understand the occurrence of the stress serrations and the arched stress plateau phenomena. As discussed above, transformation Lüders band nucleates within the heated zone in the middle of the sample and propagates towards both ends. With the decrease of the heating temperature towards the electrodes, as seen in Figure 1 (b), the stress for transformation band propagation, σ_p^{6A} , increases, till reaches σ_u^{6A} when the propagation of the current Lüders band is stopped and a new Lüders band nucleates. The serrated stress-strain behavior seen in Figures 2 and 3 is in fact the upper-lower yielding behaviour of the new Lüders band nucleation.

Figure 4. (a) Nucleation and propagation of Lüders bands during tensile deformation upon loading of the sample heat treated at 6 A; inset: an enlarged image of tracking of the new Lüders band formation within the high temperature gradient region towards the lower end of the wire (b) Total length of Lüders bands as the increase of time.

4. Conclusions

- (1) Pseudoelastic NiTi wires with spatial variations of mechanical properties can be created by selective over-aging via joule heating. Joule heating can selectively heat treat targeted sections along the length. The heated short section may exhibit symmetric temperature gradient due to 1D heat dissipation. The temperature gradient causes property gradient along the length of the wire.
- (2) Such functionally graded NiTi wires showed several unique characteristics in their tensile deformation behaviour, including two discrete stress plateaus, arch-shaped stress plateau at the lower stress level, and stress serrations in the transition section between the two stress plateaus, and clear upper-lower yielding at the onset of the lower stress plateaus.
- (3) The stress plateau at the lower stress level corresponds to the stress induced martensitic transformation within the over-aged section caused by joule heating, and the stress plateau at the higher stress level corresponds to the deformation of the unaffected length of the wire outside of the heated zone.
- (4) The arched shape of the lower stress plateau is ascribed to the gradient variation in Ni content along the length within the over-aged section caused by the partial dissolution of Ni-rich precipitates back to the matrix. The Ni content is expected to be higher in the middle of the heated section where the joule heating temperature was the highest. This causes the gradual lowering of the martensitic transformation temperature from the two ends of the heated section towards the middle, which in turn results in a the gradual increase of the critical stress for martensitic transformation from the two ends of the heated section towards the middle, thus the arch-shaped stress plateau.
- (5) The stress serrations occurring between the two stress plateaus are ascribed to the

nucleation of new martensitic transformation Lüders bands within the heated section. These new Lüders bands are formed at locations in close vicinity to the boundaries of the heated section where the temperature gradient of the joule heating was very sharp. The low heating temperature and the sharp temperature gradient create narrow sections of rapidly increasing transformation stresses, thus, very short Lüders bands which have effectively only the upper-lower yielding process.

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