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Heat-activated Prestressing of NiTiNb Shape Memory Alloy Wires

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Abstract:

Pre-strained shape memory alloys (SMAs) can develop significant level of recovery stress (prestress) when heated above their transformation temperature in the constrained conditions. In this study, thermo-mechanical tests were conducted to investigate heat-activated prestressing (HAP) of NiTiNb SMA wires. The tests were conducted to investigate: (1) the effect of pre-strain on reverse transformation temperatures (A_s and A_f) of the NiTiNb SMA; (2) the effect of strain rate and the pre-strain level on HAP; (3) post-HAP behaviour under monotonic and cyclic loading; and (4) reusability of the NiTiNb SMA wires for HAP applications. The digital image correlation technique was used to gain deeper insight into micro-mechanical behaviour of the SMA. The study highlights the complex material behaviour of the NiTiNb SMAs. The results from this study suggest that NiTiNb SMAs can be effectively used for HAP. Recommendations on optimal strain rate for pre-straining NiTiNb SMAs and the optimal pre-strain level for achieving maximum recovery stress in NiTiNb SMAs are given in this paper.

Keywords: SMA, NiTiNb, NiTi, recovery stress, shape memory effect, active confinement, heat-activated prestressing

1. Introduction

The growing demand for high performance, adaptive and reliable structural systems is continuously driving the scientific community to explore for new, more effective and resilient materials. A class of smart materials that has recently started to receive increased attention in civil engineering are the shape memory alloys (SMAs). SMAs possess several unique features which makes them a highly desirable functional material for many civil engineering applications. The two prominent features of SMAs are the superelasticity (SE), which is the ability of the material to recover large 'pseudoelastic' strain through removal of the applied load; and the shape memory effect (SME), which is the ability of the material to recover large 'pseudoplastic' strain by heating. Both SE and SME are related to martensitic transformation of SMAs, which is a diffusionless phase transformation caused by reversible shear lattice distortion [1,2]. Although SMAs exhibiting SME were discovered as early as 1963 [3], their practical applications have been realised only in the past 20-25 years. Furthermore, due to the high cost and narrow transformation hysteresis of the earlier compositions, their application was restricted to only few specialised areas, such as biomedical, aerospace and robotics. The reduction in their cost [4] in the past decade or so, and the introduction of new SMAs with wide transformation hysteresis [5] has widened the domain of SMA to civil engineering.

Since the discovery of Nitinol in 1963, many different compositions exhibiting shape memory and super elastic effects have been synthesised. However, depending upon the chemical composition and thermo-mechanical treatment, SMAs can significantly differ in their transformation temperatures (thermal hysteresis), transformation strains and the overall mechanical behaviour. Among other SMAs, NiTi, NiTiNb and Iron based SMAs (Fe-SMAs) are the most widely used SMAs in civil engineering. The selection of a particular SMA depends on application it is used for. SMA can be used in a wide variety of applications in civil engineering. In general, they are selected either to make use of their *pseudoelastic* feature (i.e. superelasticity), for example, superelastic bracing systems for seismic applications [6,7] or the SME, which includes applications such as active confinement [8,9] and heat-activated prestressing [10–13] This study focuses on heat-activated prestressing (HAP) of SMAs.

SMAs have the ability to develop a significant level of recovery stress. The term recovery stress in the literature is used to refer to the prestress developed in SMAs by means of pre-straining and heating. In this study, the process of prestressing SMAs in this manner is defined as heat-activated prestressing (HAP). Martensitic SMAs (either pre-strained or not) and pre-strained austenitic SMAs when heated above their austenite finish temperature, A_f in constrained conditions develop a significant level of recovery stress [12]. SMAs that are martensitic at room temperature, whether they are pre-strained or not, lose all the recovery stress when the temperature is lowered to room temperature. As a result, they have a very limited application in civil engineering. On the contrary SMAs that are austenitic at room temperature retain a significant level of recovery stress at room temperature when pre-strained and heated in constrained condition. These types of SMAs have been widely used for HAP in civil engineering, for example [8,9,12,13]

Several researchers in the past have investigated recovery stress in SMAs. Proft & Duerig [14] investigated constrained phase transformation in NiTiFe (47%Ni, 50%Ti and 3%Fe (at. wt.)) SMA. They showed that the recovery stress in NiTiFe SMAs depends on the applied pre-strain and increases with the increase in the level of pre-strain up to a certain

point after which it reduces. Proft & Duerig achieved maximum recovery stress of 700 MPa at a pre-strain of 9%. Li *et al.* [15] investigated the effect of pre-strain on the recovery stress in thin NiTi (50.2%Ti, 49.7%Ni (at. wt.)) wires. A pre-strain range of 0 to 6.6% was investigated by them. They also showed that recovery stress in NiTi SMAs depend on the applied pre-strain. The maximum recovery stress equal to 425 MPa was achieved by them at a pre-strain of 6.6%. Researchers for example, [13,20–23] have investigated recovery stress in other types of SMAs also. Dommer & Andrewes [16] investigated recovery stress in NiTiNb SMA. They reported that recovery stress of 556 MPa is retained in NiTiNb SMA wires at room temperature when a pre-strain of 6% is applied to the NiTiNb SMA. Suhail *et al.* [13] reported that a recovery stress of only 438 MPa is retained at room temperature in NiTiNb SMA wires when a pre-strain of 6% is applied. In contrast, Choi *et al.* [17] achieved a recovery stress of only 275 MPa in NiTiNb SMA wires when a pre-strained of 7% was applied to the SMA.

In the recent past a lot of research has been carried out on Fe-SMAs [19–30]. Fe-SMAs have started to gain popularity in the Civil Engineering due to their low cost and thus bears a good potential to be used in bulk. Hosseini *et al.* [22] carried out a case study on the cost comparison of equivalent Fe-SMA and CFRP end anchorage strengthening systems and found that the overall cost of the strengthening procedure, which included the material and labour cost tends to be similar for the two materials, but when considering only material cost, the cost of Fe-SMA (strips) was reported to be approximately double the cost of CFRP (plates). Another important material related aspect of Fe-based SMAs is the corrosion resistance of Fe-SMAs. Although, Fe-SMAs are not as good corrosion resistant materials as NiTi-based SMAs, but studies have shown that due to the presence of chromium in the composition of Fe-SMAs, the corrosion resistance of Fe-based SMAs is found to be better than conventional structural steel used in civil engineering applications [22].

Several researchers have also investigated the recovery stress in Fe-based SMAs. Lee *et al.* [18] investigated recovery stresses in FeMnSiCrNi at two pre-strain levels, 2% and 4%. A maximum recovery stress of 370 MPa and 400 MPa was reported for pre-strain level of 2% and 4%, respectively. Lee *et al.* reported that no loss in recovery stress occurs in Fe-SMAs on the removal of heat after HAP. Instead, as the specimen cools down, the recovery stress continues to increase until room temperature is reached. Interestingly, subsequent heating resulting in loss of recovery stress. Shaverdi *et al.* [11] used Fe SMAs for pre-stressing concrete beams. The Fe-SMA were pre-stained by 4%. The maximum recovery stresses developed in the Fe-SMAs strip was reported to be ~350 MPa. A more detailed material characterization of Fe-SMAs can be found elsewhere [19].

Existing studies [15,27,28] have shown that pre-straining SMAs not only has an effect on the recovery stress, but also the phase transformation temperatures. Li *et al.* [15] investigated the effect of pre-strain on the reverse transformation temperature of NiTi (Ti-50.2 at.% Ni) SMA. They showed that reverse transformation temperature of NiTi SMA increases gradually with the increase in the level of applied pre-strain. The increase in transformation temperature has been interpreted as a result of relaxation in the internal strain energy stored during pre-straining [33]. He & Rong [31] investigated the effect of pre-strain on reverse transformation temperatures (A_s and A_f) of NiTiNb (44%Ni, 47%Ti, 9%Nb (at.% wt.)) SMA. No change in the reverse transformation temperatures was reported by them up to a pre-strain of 8%, but when the pre-strain was increased above 8% a significant increase of the reverse transformation temperatures

was observed by them. He & Rong showed that increasing the pre-strain from 8% to 16.5% in NiTiNb SMA increased A_f of the SMA from approximately 40 °C to 85 °C, respectively. Takagi et al. [32] also reported increase in reverse transformation temperature of NiTiNb SMA when a pre-strain of more than 8% was applied to the SMA. For HAP, phase transformation temperatures of SMAs are critical parameters. For example, having a prior knowledge of the reversed transformation temperature, A_f is important. To ensure maximum recovery stress is achieved on heating, a pre-strained SMA must be heated above its A_f . Likewise, having a prior knowledge of forward transformation temperature, M_s (martensite start temperature) is important because the drop in the ambient temperature below M_s will result in a significant drop in the recovery stress.

A review of the literature shows that most of the existing studies have considered only a limited range of pre-strain, and in many cases only one or two arbitrarily chosen pre-strain levels are investigated. Moreover, a great deal of scatter in the results can be seen in the existing studies. To date, no study has been conducted to investigate the effect of strain localisation and/or strain rate on the recovery stresses in SMAs. Literature has shown SMAs exhibit strong strain localisation [12,30,31] and high strain-rate sensitivity [12,32,33]. For a comprehensive understanding on recovery stresses in SMAs, a more detailed and systematic investigation is required.

In this study, an experimental programme was carefully planned to carry out a systematic investigation of recovery stress in NiTiNb SMA wires. The following tests were carried out:

1. A set of thermo-analytical tests using differential scanning calorimetry (DSC) to study the effect of pre-strain on transformation temperatures of the NiTiNb SMA.
2. A set of uniaxial tensile tests (Tests 1–7) to study the effect of strain rate on the pre-strain retained on unloading (i.e. the residual strain retained after pre-straining the SMA) and the associated recovery stress. The effect of strain rate was investigated within the quasi-static range which varied from 2×10^{-5} /s to 2×10^{-1} /s.
3. A set of uniaxial tensile tests (Tests 8–15) to investigate the effect of pre-strain on recovery stress. These tests were aimed to find the optimum pre-strain level for maximum recovery stress. A wide range of pre-strain values, ranging between 0–18%, were investigated at an optimum strain rate (identified in Point 2 above).
4. To investigate the mechanical behaviour of the NiTiNb SMA wires in the prestressed state (i.e. post-HAP) under monotonic loading, specimens in the above two sets of tests (Point 2 and 3) were loaded monotonically after the HAP until their fracture.
5. One additional test (Tests 16) was performed to investigate the post-HAP mechanical behaviour of the NiTiNb SMA wires under cyclic loading. The specimen was cyclically loaded after HAP until a complete loss of recovery stress was recorded.
6. Lastly, one test (Test 17) was performed to investigate the re-usability of NiTiNb SMAs for HAP applications. The same specimen was pre-strained and heated in constrained conditions 10 times.

2. Experimental procedures

The transformation temperatures of the pre-strained SMA wires were determined using Perkin Elmer diamond differential scanning calorimetry (DSC). The SMA wire used in this study had a diameter of 2 mm. NiTiNb wire specimens were first pre-strained by different levels in a Zwick Roell universal testing machine at a strain rate of 3.3×10^{-3} /s. The strain rate of 3.3×10^{-3} /s was chosen based on the previous studies conducted by the authors [12]. From each pre-strained wire, small specimens of 1 mm length were cut along the longitudinal axis of the wire using wire electrical discharge machining (EDM) for DSC tests. EDM was used to avoid introducing deformation (at the cutting ends) and heating-up of the specimens during the cutting. Both of which can affect the end results. The weight of the DSC specimens varied from 22 mg for specimens pre-strained by 0% to 19.6 mg for samples pre-strained by 16.2%. Heating and cooling in all the DSC measurements was carried out at rate of ± 10 °C/min, following the recommendation given in standard test method ASTM F-2004 [38]. The start and finish of the reverse of transformation temperatures were determined from the intersection of the tangents to the slope of enthalpy peaks and the corresponding baseline. Two different heating procedures were adopted. In the Series-I, DSC specimens were heated from room temperature (RT) onwards, up to a temperature of 200°C. In the Series-II, DSC specimens were first cooled to -150 °C and then heated to 200°C. DSC specimens were cooled in Series-II to investigate the effect of cooling on pre-strained specimen. In a situation where NiTiNb is pre-strained at a factory and then transported in cold cambers or freezers to the place of installation, the results from the Serie-II may be used.

Tensile tests of all NiTiNb wires specimens (see Table 1 for summary of tests) were carried out in a Zwick Roell universal testing machine installed with a load cell of 100 kN capacity. The accuracy of the load cell as per the manufacturer's calibration chart is given as $\pm 0.02\%$ between a load range of 0 to 5 kN. Body over wedge grips were used to clamp the specimens in the machine. Both grips were supported with pivots to minimise the effect of bending in the specimens. The grip-to-grip distance of all specimens used in this study was equal to 80 mm. In order to avoid errors in strain measurements due to slip, strain in the specimens was measured using either the digital image correlation (DIC) (Tests 1–15 in Table 1) or video extensometer (Tests 16–17 in Table 1). The DIC is an in-situ non-contact optical correlation technique which is used to obtain full-field strain contour maps by tracking random pattern on the specimen's surface. By comparing two digital images (comprised of pixels) of a specimen's surface taken at different times, a relative displacement in the pixel coordinate system is obtained through correlation analysis of grey-scale intensity values. The displacement is then converted into the physical coordinate system. The strain field is obtained through differentiation of the displacement field. In this technique, a good quality surface pattern is of crucial importance. A random surface pattern could be an artificially applied pattern or an inherent material feature such as ribs and dents on a rebar surface. In this study, a fine discrete speckle pattern was achieved by spraying black and white spray-paint on the surface of the specimens. A Canon EOS 5D Mark III fitted with Canon EF - 24-105mm - F/4.0 lens was used to record the images of the specimen during the tests. Depending on the strain rate used, images were captured at rates varying between 1–5 Hz. The accuracy of the DIC measurement depends on number of parameters such as speckle size, image resolution and field of view (FOV) and the facet size used in the DIC [39]. More detailed information on DIC can be found elsewhere [35,36]. Post processing of the image was carried out using GOM correlate software developed by GOM [41]. The speckle size in this study varied between 3–5 pixels. The accuracy of the DIC measurements in this study is estimated equal to ± 5 micro strain.

A high precision non-contact single camera Zwick Roell video extensometer was also used in this study. To measure strains using the video-extensometer, two methods were attempted: 1) the front-light method in which the video-extensometer and the light are directed in the same direction, towards the specimen and; 2) the back-light method in which the lighting device and the video-extensometer are directed in opposite direction with the specimen placed in between. The back-light method produces higher contrast black and white images. Trial tests showed that the back-light method produced less noise in the strain readings and was more stable for high-strain rate tests, therefore this method was adopted in this study. In this study, the FOV of video-extensometer was set between 100–200 mm which gives a minimum accuracy of 1 μ m according to the user manual of the extensometer used. A 500 mm long light with light-emitting diodes was used to ensure constant luminosity. The gauge length on each specimen was specified by two target pieces directly attached to the specimen. The target pieces were specifically designed to cater for cross-sectional reduction of specimens during the tests and stability during the high strain rate tests. As suggested in the video-extensometer user manual, the target pieces were slightly inclined (between 2°–5°) from the horizontal.

For HAP tests, each specimen was first deformed by a specific amount and then fully unloaded to zero stress. In this study, this step is referred to as pre-straining step. Upon unloading, specimens were held in position (by restraining the movement of the crosshead) and heated above the A_f until a stress plateau in the stress-temperature plots was observed. The stress plateau in the stress-temperature plots indicated that the maximum recovery stress was achieved. Specimens were then allowed to naturally cool down to room temperature while maintaining the constrained boundary condition. At room temperature, specimens were left undisturbed for about 30 minutes – 1 hour. After which, each specimen was loaded either monotonically or cyclically until the rupture took place.

Heating of the specimens was carried out using 2500 W fan-heaters and 2400 W heat-gun. The temperature of the wire during the tests was monitored using a Type-T thermocouple which was attached directly on the surface of the specimens. Ideally, an environment chamber should have been used to heat the specimens however, this was neither possible in the DIC nor in the video extensometer set-up used in this study as both the set-ups required specimens to remain exposed throughout the test. The use of heat-gun although resulted in non-uniform heating however, it is reported in the literature [16,38] that non-uniform heating does not affect the maximum recovery stresses developed in the SMAs.

1

Table 1. Summary of the tensile tests carried out in this study

Test number	Test type	Loading method	Average Pre-strain applied (%)	Strain measurement method	Strain rate (1/s)
Test 1	Strain-rate tests	Displacement control	4.95	DIC	2.0×10^{-5}
Test 2			3.61		2.0×10^{-4}
Test 3			2.90		2.0×10^{-3}
Test 4			5.10		2.0×10^{-2}
Test 5			5.18		5.2×10^{-2}
Test 6			6.19		1.0×10^{-1}
Test 7			4.80		2.0×10^{-1}
Test 8	Pre-strain tests	Displacement control	1.31	DIC	2×10^{-4}
Test 9			3.85		2×10^{-4}
Test 10			6.49		2×10^{-4}
Test 11			8.69		2×10^{-4}
Test 12			10.51		2×10^{-4}
Test 13			12.59		2×10^{-4}
Test 14			14.59		2×10^{-4}
Test 15			17.54		2×10^{-4}
Test 16	Post-HAP (cyclic)	Strain control	8.0	Video extensometer	2×10^{-4}
Test 17	Re-usability test		8.0		3.3×10^{-3}

2

3. Results and discussion

3.1. Material properties

The chemical composition of the SMA used in this study was determined by energy dispersive spectroscopy (EDS) using a FEI Quanta600 scanning electron microscope. The wire consisted of 47.1% Ni, 43.8% Ti and 9% Nb (at.%). The characteristic phase transformation temperatures of as-received NiTiNb wire were provided by the supplier (Xi'an Saite Metal Materials Development Co., Ltd) and are given in Table 2.

Table 2. Transformation temperature of as-received NiTiNb wire (°C)

	M_f	M_s	A_s	A_f
Data from the supplier	-108	-80	-24	2

10

The wire was fully annealed at 850°C for 1.8 ks. No information on the amount of cold rolling was available. A more detailed information on the material properties of the SMA wires can be found in [12].

3.2. Effect of pre-strain on transformation temperatures

Specimens for DSC tests were pre-strained by 0 to 16.2%. The summary of the DSC measurements is given in Table 3. The results in Table 3 only show the reverse transformation temperatures (A_s and A_f). The forward transformation temperatures (M_s and M_f) could not be determined from the DSC curves and it is assumed that they exceeded the measurement range of the DSC machine. A similar observation was reported by Dommer *et al.* [16] for their NiTiNb specimens. For the purpose of comparison, the data from the SMA supplier is also given in Table 3. The minimum pre-

strain given in Table 3 is ~8%. Pre-straining of austenitic NiTiNb SMA involves stress induced martensitic (SIM) phase transformation. The phase transition undergoes through discrete events of nucleation and growth of transformation bands (TBs) [12]. The nucleation of a TB always produces an instantaneous strain of ~8%, which is equal to the transformation strain, ε_t of NiTiNb SMAs [12], which implies that the minimum strain in the NiTiNb SMA (in the transformed region) is 8%. A more detailed discussion on this material behaviour is given in the following sections [12].

Table 3 provides the values for the applied pre-strain as well as the residual pre-strain of the DSC specimens, i.e. pre-strain applied to the specimen just before unloading and pre-strain retained in the specimen on complete unloading. The pre-strain retained in the specimen on unloading is strain-rate sensitive [12]. For higher strain-rates, the pre-strain retained on unloading may be less even when the maximum pre-strain applied are same. Consequently, the DSC results may also be different.

Table 3. Transformation temperatures of NiTiNb wire from supplier and two separate DSC tests.

Data from supplier			Series-I				Series-II			
Pre-strain	A_s	A_f	Pre-strain (%)		A_s	A_f	Pre-strain (%)		A_s	A_f
(%)	(°C)	(°C)	applied	retained	(°C)	(°C)	applied	retained	(°C)	(°C)
0	-24	2	-	-	-	-	-	-	-44	0
-	-	-	8.1	5.7	39	80	9.4	6.9	43	62
-	-	-	12.7	9.6	76	123	12.2	9.2	52	86
-	-	-	16.2*	12.5	96	135	15.1	11.8	65	117
-	-	-	16.2*	12.5	90	133	15.4	11.6	78	124

*Samples taken from the same wire but tested in different DSC machines

The A_s of the as-received SMA wire was measured equal to -44°C , which is about 20°C less than the A_s given in the supplier's data. However, the measured A_f of the specimen was close to the value given in the supplier's data. The difference in A_s of the as-received SMA may be due to difference in the rate at which the DSC tests were conducted or the shape of the enthalpy peaks and the baseline chosen for estimating temperature values or the combination of all the above. As no additional information was available, the exact reason for this difference could not be ascertained.

For the pre-strained specimens, with the increase in the pre-strain level, the reverse transformation temperatures of the SMA increased almost linearly, see Figure 1(a) and (b). The A_f of a specimen pre-strained by 8.1% in Series-I was measured equal to 80°C . The A_f of the specimen pre-strained by 16.2% in Series-I was $\sim 135^\circ\text{C}$. Table 3 shows two specimens pre-strained by 16.2% in Series-I. The two specimens were obtained from the same SMA wire but tested in two different DSC machines. The DSC measurements for the two specimens are in close agreement.

As in Series-I, the reverse transformation temperatures of the SMA in Series-II also increased with the increase in pre-strain. The increase in transformation temperature with the increase in applied pre-strain is attributed to the influence of permanent slip (plastic deformation) in β -Nb particles and TiNi matrix [31], which increases the resistance to strain recovery upon heating. The influence of the heating regime adopted in the two series can be clearly seen in Figure 1(a)

and (b), where it is found that the transformation temperature interval (TTI) ($A_s - A_f$) in Series-I is nearly same at all pre-strain level. However, for Series-II, the TTI is narrower at lower pre-strain level and widens as the pre-strain level is increased. The explanation for difference in TTI in the two series although pre-strained by approximately same amounts is not fully understood.

Figure 1: Reverse transformation temperatures of pre-strained NiTiNb specimens; (a) Serie-I (room temperature → 200°C) (inset: typical measurement); (b) Series-II (-150 → 200°C).

By comparing the results of this study with the existing studies [27,28] it is found that the increase in the reverse transformation temperatures (A_s and A_f) of an SMA, as a results of pre-straining, is significantly influenced by the thermo-mechanical treatment. For example, Takagi *et al.* [32] showed that a pre-strain of 18% increased the A_f of the NiTiNb SMA by 66°C while as in this study for the same type of SMA, a pre-strain of 16.1%, ~2% less than the former, increased A_f of the SMA by 135°C. This difference in reverse transformation temperature is believed to be due to the different thermo-mechanical treatment of SMAs. The SMA used in this study was annealed at 850°C, which is well above the recrystallization temperature of NiTiNb SMAs. Annealing of SMA above the recrystallization temperature results in SMA exhibiting partial superelasticity [43], and is accompanied by a significant level of plastic deformation at all strain levels above the elastic limit. The SMA used by Takagi *et al.* was aged at 400°C and cold rolled by 30%. Their SMA was superelastic at room temperature and retained significantly less residual plastic strain as compared to SMA used in this study.

3.3. Effect of strain rate on recovery stress

To investigate the effect of strain rate on the recovery stress, σ_{recov} , specimens from the as-received NiTiNb SMA wires were deformed in Tests 1–7 at strain rates varying from 2×10^{-5} /s to 2×10^{-1} /s. The tests were conducted in displacement-control at room temperature. All specimens were subjected to the same crosshead displacement, which corresponded to specimen elongation of 8%. Figure 2 shows the loading and unloading of the specimen in the pre-straining step, σ_{recov} achieved in the constrained conditions during the heating step and the post-HAP loading of the specimens in Tests 1–7.

The strain rate adopted in each test is indicated on the plots. Due to slip at the grips, the elongation response given in Figure 2 could significantly differ from the local/average strain in the material.

Figure 2: Nominal stress vs elongation response of virgin NiTiNb specimens in Tests 1–7. (where, Δ is the crosshead displacement in mm and L is the total length of the specimen between the grips in mm).

In Figure 2(a – e), several stress-drops are observed over the stress plateau region. The stress drops occur due to the nucleation of localised martensite transformation bands [12,40]. Furthermore, in Figure 2(d – g), as the strain rate increases, it is observed that the deformation retained in the specimens on unloading is reduced. In Test-1, the average apparent deformation retained on unloading is about 5.2% (Point j in Figure 2(a)) and in Test-6 Figure 2(f)) it is only about 2%. The reduction in the residual deformation becomes significant when the strain rate is increased above $2 \times 10^{-3}/s$. The reason for this material behaviour is the thermo-mechanical coupling in the material. The phase transformation in NiTiNb SMA is accompanied by the localised release/absorption of latent heat [31,40,41], which significantly influences the material behaviour both at micro- and macro-level. At strain rate of $1 \times 10^{-1}/s$ (Test 6) the released heat in the loading process was fast such that it did not have enough time to transfer out due to high strain rate (i.e. loading time is much shorter than heat convection time) and therefore it was carried to unloading process, resulting in specimen's temperature much higher than the ambient temperature. It is most likely that the temperature of the specimen was either very close to $80^\circ C$ (A_f of the deformed NiTiNb specimen based on results given in Table 2) or well above it which consequently resulted in pseudoelastic behaviour of the material. In Figure 2 one can also see that the reduction in residual deformation results in the reduction of recovery stress.

The elongation response (deformation) given in Figure 2 includes the contribution from the slip at the grips and cannot be regarded as strain. To eliminate this from the strain measurements, strains were measured directly from the full-field strain contour maps of the specimens given in Figure 3. For brevity, strain contour maps of each test are presented only for the maximum applied pre-strain (PS_{Max} ; Point i in Figure 2) i.e. at a point just before unloading and residual pre-

strain (PS_{res} ; Point j in Figure 2) i.e. at a point when the specimen is completely unloaded. The strain contour maps are obtained by correlating the image of specimen taken at instants corresponding to PS_{Max} and PS_{res} with that the reference image, taken at the beginning of the test, respectively. Except for 3–6 mm of specimen's length near the grips, the entire length of the specimens was monitored using DIC.

Figure 3: Full-field strain contour maps of NiTiNb specimens deformed at different strain rates; (a) strain distribution at maximum applied pre-strain, (PS_{Max}); (b) pre-strain retained on unloading, (PS_{res}).

The full-field strain measurements of the specimens given in Figure 3 provide an insight into the complex material behaviour of SMAs. As can be seen in Figure 3, the strain applied to the specimens during pre-straining is localised in the form of transformation bands (TBs). The propensity of SMAs to form localised bands of large strain during the stress induced martensitic (SIM) phase transformation has been reported by several other authors as well. A more detailed information on this phenomenon can be found in [12,37,31]. Due to the non-uniform strain distribution, the results from Tests 1–7 need to be interpreted in conjunction with the effect of local strain. The relationship between recovery stress (σ_{recov}), strain rate, maximum applied pre-strain on loading and pre-strain retained upon unloading in Tests 1–7 is plotted in Figure 4(a) and (b). In Figure 4(a), both the applied pre-strain and pre-strain retained on unloading were measured locally within the TBs using the strain contour maps given in Figure 3. The strain in TBs was measured over

a gauge length of 10 mm (indicated by virtual strain gauges in Figure 3) for all tests, following the recommendation given in ASTM F E8/E8M-09 [46]. For Test-3, a gauge length of only 6 mm was used because the maximum length of the TB formed in Test 3 was only 6 mm. In Figure 4(b) strain given is the average strain in the specimen measured using DIC strain contour maps. The average strain was measured by dividing the total deformation of the specimen (measured using DIC) by the total length of the specimen between the grips. The strain measured this way is the total deformation averaged over a gauge length and not actually the ‘strain’ due to strain localisation. The “strain” obtained this way could differ from the local strain in the material. However, for the sake of simplicity, we will just use the term average strain. Recovery stresses in Figure 2 were measured at 25°C from the stress-temperature plots.

Figure 4: Effect of strain rate on maximum pre-strain, pre-strain retained on unloading and recovery stresses at 25°C; (a) local strain in transformed detwinned martensite bands (TBs); (b) average strain (measured over the entire length of the specimen). Strain given in the figure is measured using DIC.

In Tests 1–7, the maximum applied pre-strain within the TBs varied between 7–8%. The pre-strain retained within the TBs on unloading varied significantly between Test 1 and Test-6. At higher strain rates, less pre-strain was retained in TBs. A TB in the specimen tested at a strain rate of 2×10^{-5} /s (Test-1) retained maximum strain of about 5.5%, while a TB in specimen tested at strain rate of 1×10^{-1} /s (Test-6), retained a strain of only 1.4% upon unloading. Less pre-strain retained in TBs meant less shape recovery on heating as a result less recovery stress was developed during heating of the specimens (in fully constrained conditions). The recovery stress significantly decreased from 454 MPa in Tests 1 to 160 MPa in Tests 6.

In Figure 4(a), the pre-strain retained locally within the TBs in Tests 1–3 is almost same, about 5.4%, but the average pre-strain (calculated over the total length of specimen between grips) is different, see Figure 4(b), due to the different

length of the TBs, see Figure 3(b). In Tests 1–3 as the average pre-strain applied and retained reduced, recovery stress in NiTiNb wires also reduced.

In Tests 4, the average pre-strain retained on unloading was more or less same as in Tests 5 and 6, see Figure 4(b). The pre-strain retained in the TBs was also similar except in one small region of the top TB in Test 4, which is indicated by dashed box in Figure 3(b). In the region the TB had slightly higher concentration of strain which resulted in significantly higher recovery stress in Test 4 as compared to Test 5 and 6. Therefore, both the average pre-strain and the local concentration of the pre-strain retained within the TBs influence the recovery stress in the NiTiNb SMA. To achieve the maximum recovery stress, pre-straining of SMAs should be carried out at the lowest possible strain rates. From the above results strain rates lower than $2 \times 10^{-3}/s$ appears to have negligible effect on the residual strain.

3.4. Effect of pre-strain on recovery stress

To study the effect of pre-strain on the recovery stress in NiTiNb SMA wires, virgin specimens taken from as-received NiTiNb SMA wires were pre-strained by different amounts in Tests 8–15. The specimens were subjected to crosshead displacements which corresponded to elongation of 3–25% of specimen's length. The actual average strain in the specimen measured using DIC varied between 1.3–17.5%, which is significantly less than the crosshead measurements. The difference in the two measurements as discussed earlier is due to the contribution of slip at the grips in the crosshead measurements. Based on the results presented in the previous section, the strain rate chosen for these tests was $2 \times 10^{-4}/s$, 10 times less than the minimum recommended strain rate ($2 \times 10^{-3}/s$) in the previous section.

Figure 5 shows the loading and unloading of the specimens in the pre-straining step, the σ_{recov} achieved in the constrained conditions during the heating step and the post-HAP loading of the specimens in Tests 8–15.

Figure 5: Nominal stress vs elongation response in virgin NiTiNb specimens in Tests 8–15 (where, Δ is the crosshead displacement in mm and L is the total length of the specimen between the grips in mm; Pre-strain (PS) is average pre-strain in the specimen estimated using DIC)

Full-field strain contours maps of the specimens investigated in Tests 8–15 are given in Figure 6. As per the previous section, strain contour maps of each specimen are presented only for the point of maximum applied pre-strain, PS_{Max} (Point k in Figure 5) and the point corresponding to the complete unloading of the specimen PS_{res} (Point l in Figure 5) in the pre-straining step.

Figure 6: Full-field strain contour maps of NiTiNb specimens subjected to different pre-strain levels; (a) maximum average pre-strain on loading, PS_{Max} ; (b) pre-strain retained on unloading, PS_{res} .

In Figure 6, it can be seen that the specimens in Tests 8–10 were partially transformed and the strain in the specimens was concentrated locally within the TBs. It is found that maximum strain in the TBs in these tests is limited to ~8% until the whole specimen is fully transformed. With the exception of Test 8, local strain in the TBs was measured over a gauge length of 10 mm marked on the transformed region of the specimens as indicated in Figure 6(a). In Test-8, the TB was formed partially outside the strain contour map therefore, virtual strain gauge in Test 8 was relatively small (4 mm) as shown in Figure 6. In Tests 8–10 the applied deformation after the nucleation of TBs was accommodated by the growth (broadening) of the TBs. It is clear in Figure 7 the difference in the average pre-strain in the specimen and the local strain in the TBs is significant only up to complete propagation of the TBs until which the strain in the TBs is

limited to 8% – a value of strain that corresponds to the transformation strain ε_t of the NiTiNb SMA. After the specimen is fully transformed the strain distribution in the specimens appears to be more or less uniform. A similar observation was reported by in [12] and [36].

Figure 7: Difference between the local strain in TBs and average strain in specimen; (Tests 8–15). Strain given in the figure is measured using DIC.

Figure 8 shows the maximum average pre-strain applied, average pre-strain retained on unloading, maximum recovery stress, (σ_{recov}) developed on heating, and the recovery stress retained at 25°C in Tests 8–15. The strain given in Figure 8 is measured from the strain contour maps given in Figure 6. In Figure 8, the maximum recovery stress in Test-8 is equal to 230 MPa. The corresponding maximum average pre-strain is equal to 1.3% and the average pre-strain retained on unloading is equal to 0.3%. A relatively high recovery stress with respect to average pre-strain applied in Test 8 (i.e. only 0.3% average strain produced a 230MPa recovery stress) is believed to be due the high concentration of local strain (~8%) in the TB formed near the bottom grip, see Figure 6 and Figure 7. In Tests 9 and 10, the average pre-strain applied to the specimens is equal to 3.8% and 6.5%, respectively and the maximum recovery stress recorded is equal 470 MPa and 632 MPa, respectively. In Test 11, the entire length of the specimen was transformed before unloading of the specimen was carried out. The maximum average pre-strain in the specimen was 8.7% and the maximum recovery stress developed on heating was about 700 MPa of which 550 MPa was retained at 25°C.

Figure 8: Stress-strain plot showing the relationship between average pre-strain (PS) applied, average PS retained on unloading, maximum recovery stress developed on heating (σ_{recov}^{Max}), and the recovery stress retained at 25°C ($\sigma_{recov}^{Ret, T=25^{\circ}C}$). Strain given in the figure is measured using DIC.

Further increase in the pre-strain in Tests 12–15 resulted in lower recovery stresses. Although the maximum recovery stresses developed in Test 11 and Test 12 were almost the same, the recovery stress retained at 25°C was about 25 MPa less in Test-12. In Tests 13 – 15 the average pre-strained applied was significantly higher than ϵ_t of the NiTiNb SMA. The recovery stress recorded in these tests was considerably lower than the recovery stress recorded in tests (Test 11 and 12) whose average pre-strained was close to ϵ_t . The primary reason for this is the accumulation of the plastic strain, also referred to as *amnesia* strain [14], which increases the resistance to strain recovery upon heating and which in turn reduces the shape recovery and the recovery stress.

From the above results it is found that the maximum recovery stress for the NiTiNb SMA used in this study is achieved when the entire specimen is fully transformed and an average pre-strain level close to the transformation strain, ϵ_t is applied.

Dommer *et al.* [16] investigated recovery stresses in NiTiNb SMA pre-strained by 6%. On heated the specimen in constrained conduction a maximum recovery stress of 540 MPa was achieved by them. The residual recovery stress retained in the specimen at room temperature (26°C) was reported equal to 556 MPa. Comparing these results with the results obtained in this study, the maximum recovery stress measured for a similar pre-strain level (6.5% in Test 10) is found considerably higher (632 MPa) however, the residual recovery stresses retained at room temperature are comparable. There could be several reasons for the difference in peak recovery stress, for example; 1) their specimens were heated only up to 185°C so, it could be due to partial activation; 2) their specimen had considerably higher reverse transformation temperatures as compared to the specimens used in this study, which implies they were subjected to different thermo-mechanical treatment. In another study, Choi *et al.* [17] reported a significantly lower peak recovery stress for NiTiNb SMA. A maximum recovery stress of only 275 MPa was achieved by Choi *et al.* in the specimen pre-strained by 7%. This could be due to the cold rolling which their specimens were subjected to prior to pre-straining, which in turn may have increased the total plastic strain in the specimens. It could also be due to the lower strain concentration in TBs or higher strain rate used during the pre-straining, which may have resulted in lower residual pre-strain.

The maximum recovery stress achieved in NiTiNb SMA used in this study wire is found to be considerably higher than in other types of SMA. For example, Chen *et al.* [42] reported an optimum recovery stress of 416 MPa in Iron based FeNiCoTi SMA at a pre-strain level of 5.5%.

From the above discussion, it is observed that there are many parameters that can influence the recovery stress in SMAs. Most of these parameters depend on the thermo-mechanical treatment carried out during the production process. To increase the confidence of engineers in this material and to encourage them to use it in real structures, a need for the standardisation of the material and its manufacturing process is required as discussed by *Suhail. R* [12].

3.5. Stress-strain behaviour of NiTiNb SMA in prestressed state

3.5.1. Stress-strain behaviour under monotonic loading

The stress-strain behaviour of NiTiNb wire specimens loaded monotonically after heating in constrained condition (i.e. after HAP) in Tests 1–7 and Tests 8–15 is presented in Figure 9(a) and (b), respectively. Post-HAP loading of the specimens in all the tests was carried out at the same strain rate as in their respective pre-straining steps. For simplicity, the plots Figure 9(a) and (b) only show the post-HAP stress-strain curves with strain initialised to zero. The strain given is the average ‘strain’ obtained using the crosshead data. As the specimen, at the start of the post-HAP loading, were already under some level of stress, the contribution of slip in the crosshead data is assumed to be negligible. Strain measurements using DIC were obtained only for Test 4. The speckle pattern in all other tests was extensively damaged during the heating process, therefore, correlation of post-HAP test images in these tests could not be performed.

Figure 9: Fracture strain and post-HAP stress-strain behaviour of specimens in: (a) Tests 1-7; and (b) Tests 8-15. Strain given in the plots is the average strain measured using crosshead displacement. (where, Δ is the crosshead displacement in mm and L is the total length of the specimen between the grips in mm)

In Figure 9(a) and (b), it can be seen that the post-HAP loading of the specimens initially resulted in a rapid increase of stress until the yield stress (the critical stress for SIM phase transformation to begin) is reached. Yielding of the specimen is followed by the stress plateau, which in some tests contains peaks and stress-drops and is smooth in some. To

investigate the stress-plateau region in more detail, stress-strain curves for each test is re-plotted for a strain range of only 0 – 16% in Figure 10 and Figure 11.

In Figure 10 and Figure 11, it is found that those specimens which were partially transformed during pre-straining (Tests 1–10; see Figure 3 and Figure 6) their stress plateaus consisted of two different parts: (1) smooth portion (between points g – h); and (2) portion with spikes and stress drops (between points h – i) (see Figure 10 and 11). Points g , h and i were identified from the intersection of tangents drawn on the stress-strain curves at locations where the direction of the slope of the curve changed. Strain between Points g – h is assumed to be corresponding to the deformation (reorientation) of those transformation bands (TBs) which were formed during pre-straining. The reason for this assumption is that the deformation of the NiTiNb SMA that has already undergone SIM phase transformation results in a smoother stress-strain curve when loaded the second time due to “training effect” [12]. Strain between Points h – i is assumed to correspond to the formation of new TB during the post-HAP loading. The jaggedness in region h – i of the stress plateau is believed to be due to the nucleation of new transformation bands. The nucleation of new TBs in NiTiNb SMAs results in a stress-drops which depends on the strain rate [12]. For partially transformed specimens with small pre-strain, the strain within Points h – i is found to be larger than specimens with large pre-strain.

For the specimens which were fully transformed during pre-straining (Tests 10–15) the post-HAP loading resulted in a stress plateau which consisted of only a smooth region g – i (see Figure 11 and Figure 6). As no new TBs were formed in these specimens, no significant stress-drops took place and the stress-strain curves were smooth.

Figure 10: Post-HAP stress-strain behaviour of prestressed NiTiNb specimens under monotonic loading (Tests 1–7); strain given in the plots is the average strain measured using crosshead data and PS (pre-strain) given in the plots is the applied PS before heating and measured using DIC.

Figure 11: Post-HAP stress-strain behaviour of prestressed NiTiNb specimens under monotonic loading (Tests 8–15); Strain given in the plots is the average strain measured using crosshead data and PS (pre-strain) given in the plots is the applied PS before heating measured using DIC.

In Figure 10 and 11, it is further observed that for the specimens partially transformed during pre-straining (i.e. Tests 1–10) the smooth region of the stress plateau always precedes the region with peaks and stress-drops, which indicates that the SIM phase transformation first takes place in the TBs which are formed during pre-straining. This phenomenon is demonstrated in the strain contour maps given in Figure 12.

Figure 12: Full-field strain measurement of specimen in Test 4; (a) pre-straining step; (b) post-HAP loading: loading up to failure (with upper-limit of the strain contour maps equal to 35%); (c) post-HAP loading: only frames 0'– (with upper-limit of the strain contour maps limited to 8%).

In Figure 12, full-field strain measurement of the specimen used in Test 4 is given for the point of maximum applied pre-strain (PS_{Max}), residual pre-strain (PS_{res}) retained on complete unloading, and the temporal evolution of strain produced in the specimen during the post-HAP loading. As can be seen in Figure 12(a), two TBs (TB1 and TB2) were formed in the specimen during the post-HAP loading. Strain contour plots in Figure 12(b) were obtained by correlating the images taken at various stages of test during the post-HAP loading with the reference image taken at the start of post-HAP loading (i.e. at PS_{res}). PS_{res} was chosen as reference image so that only the post-HAP strain in the specimen is obtained. By comparing Figure 12(a) and (b), it is evident that the post-yielding strain in post-HAP loading is first produced in those TBs which were already formed during pre-straining i.e. in TB 1 and TB 2, see Frame 1 and 2 in Figure 12(b). This is because the front propagation stress of the TBs is lower than nucleation stress of a new TB. More details on the mechanics of the SIM phase transformation in SMAs can be found elsewhere [35]. To keep discussion simple, here, only the macroscopic behaviour of the material is discussed. To distinguish the difference in strains in transformed and untransformed bands, Frames 0'–5 are re-plotted in Figure 12 (c) with lower upper-limit of the strain contour maps. In Figure 12(c) it is clear that those regions of the specimen which were not transformed during pre-straining remained untransformed until the strain in the TB 1 and TB 2 reached a strain of $\sim 5\%$ (Frame 2 in Figure 10(b)). This value of

strain is regarded as the limiting strain $\varepsilon_{t,p}$ of TBs transformed 2nd time. Once the strain in TB 1 and TB 2 exceeded $\varepsilon_{t,p}$, the nucleation of the new TBs (TB3 and TB4) took place. The new TBs formed during the post-HAP loading appear to have same features as the TBs formed during pre-straining (i.e. in virgin specimens). The nucleation of TB 3 and TB 4 resulted in the local strain of 8% within the TBs, see Frame 3 in Figure 12(c). As the loading continued, the newly formed TBs broadened but the maximum local strain in the TBs was limited to 8% until they merged with previously formed TBs. At this stage, the strain in the previously formed TBs began to increase while the strain in the newly formed TBs remained limited to 8%. At a point when the strain in the entire specimen was approximately 8%, the strain in the specimen began to increase almost, but not exactly, uniformly along its length.

$\varepsilon_{t,p}$ measured from DIC gives an indication of the extent of stress plateau expected after HAP of the NiTiNb SMA. For specimens fully transformed during pre-straining, the extent of stress plateau is equal to $\varepsilon_{t,p}$. The range of the post-HAP stress plateau varied considerably among the specimens which were partially transformed (Tests 1–7 and Tests 8–10) during pre-straining. Specimens with lower average pre-strain possessed longer stress plateau in the post-HAP loading. For specimens that were fully transformed during pre-straining (Tests 11–15, see Figure 6), the post-HAP stress plateau varied between 4.2%–5% strain (~4.5% average), which is the value close to $\varepsilon_{t,p}$ obtained from strain contour maps in Figure 12(c). The difference between the $\varepsilon_{t,p} = 5\%$ measured using DIC and extent of stress plateau (~4.5% average) obtained using crosshead data in Tests 11–15 could be due to different strain measurement method and strain rate.

The effect of strain rate on the post-HAP stress-strain behaviour of NiTiNb SMAs, can be seen in Figure 10 and 11. The stress-strain plots of Tests 1–7, see Figure 10, shows that the slope of the stress plateau increases with the increase in strain rate. The increase in the slope of stress plateau is due to accumulation of the latent heat which in turn increase the front propagation stress [35]. Similar observations were made in virgin specimens during the pre-straining process. In Figure 11, all test (Tests 8–15) were carried out with the same strain rate, the slope of the stress plateau was approximately the same.

Figure 9(a) and (b) suggest that the fracture strain of the specimens in post-HAP loading depends on pre-strain history. Generally, for specimens that were pre-strained by less than 8% (Test 1–10), the fracture strain was more than 30%. For specimens that were pre-strained by more than 8% (Test 11–15), the fracture strain ranged between 22–25%.

3.5.2. Stress-strain behaviour under cyclic loading

Under cyclic loading, the extension of pre-stressed SMAs during the post-HAP loading tends to cause the loss of the recovery stress [17]. This is because, the reoriented martensites formed during the post-HAP SIM transformation (i.e. during the post-HAP loading) are retained in the specimen due to which the specimen is extended (elongated). When the specimen is unloaded, the elongated specimen resulted in the loss of recovery stress. The loss of recovery stress could diminish the practical applicability of using SMAs for applications such as active confinement. For example, loss of recovery stress in SMAs used for active confinement imply that SMA confinement would essentially act as passive confinement after a certain number of loading cycles. Passive confinement is less effective than active confinement [47] and can be achieved by other means which are more convenient and less expensive to use, such as fibre reinforced polymer (FRP) wrapping and steel jacketing.

To investigate the post-HAP mechanical behaviour of NiTiNb SMA wire under cyclic loading, Test 16 was conducted in a strain-controlled mode at a strain rate of 2×10^{-4} /s using a video extensometer. The test was conducted in strain rather than displacement control for two reasons: (1) to control the actual strain applied to the specimen during loading and unloading; and (2) to accurately measure the strain while performing the test. To ensure accuracy of the strain applied to the specimen during the test, real time strain measurement was necessary which could not be achieved using DIC. The downside of using a video extensometer set-up is that it was not possible to achieve a full-field strain measurement.

Figure 13(a) shows the stress-strain plot obtained from Test 16. Strain in the test is measured over the gauge length (lg) of 50 mm which was specified on the specimen using specially designed target pieces (see Figure 13(b)). The SMA specimen was first pre-strained by 8% (fully transformed) and then heated above the austenite finish temperature in the constrained conditions as shown in Figure 13(a). During heating, a small strain recovery (Point $d - e$ in Figure 13(a)) within the gauge length was recorded even though the crosshead was constrained (held at constant position). This is due to non-uniform heating which resulted in non-uniform shape recovery in the SMA specimen. A similar observation was reported in [12] for NiTi SMAs also.

During the heating process, a maximum recovery stress of 652 MPa was developed and approximately 550 MPa of recovery stress was retained at room temperature. The maximum recovery stress in Test 16 was approximately 50 MPa less than the maximum recovery stress recorded in Test 11 even though the specimens in both tests were subjected to same pre-strain level. The difference in the maximum recovery stress is due to the difference in the way heating was carried out in the two tests. In Test 11, in addition to heating the exposed part of the specimen, heat was also applied inside the jaws of the grip through the front slit. In Test 16, heating inside the jaws could not be carried out because the opening in the jaws was in the line of sight of the video extensometer and therefore it prevented heating of the specimens inside the jaws. The recovery stress retained at room temperature was, however, approximately same in the two tests.

After heating (HAP), the SMA specimen was cyclically loaded as shown in Figure 13(a). The cyclic loading was carried out in such a way that unloading in each cycle was restricted to a strain of 5.1%, the strain corresponding to Point *d* (see Figure 13(a), which corresponds to the deformed length of the specimen after pre-straining).

Figure 13: (a) Behaviour of prestressed NiTiNb specimens under cyclic loading; (b) photograph of target pieces attached to the specimen for measuring strain (where l_g is the gauge length).

In Figure 13(a), it is observed that any deformation caused in the loading step of the post-HAP loading causes some loss of the recovery stress upon unloading. The recovery stress is gradually lost as the post-HAP deformation increases. The magnitude of strain between the Points $f-i$ in Figure 13(a) is the range for which recovery stress is effective and is found to be approximately 3.5% in this study. Choi *et al.* [17] conducted similar tests on the NiTiNb SMA wires subjected to similar pre-strained level (7% vs 8% in this study). They reported recovery stress is completely lost as the post-HAP deformation (strain in SMA wire) exceeds a value of $\sim 1.8\%$. The recovery stress for the NiTiNb SMA used in this study is found to be effective for a much larger strain range. There are several parameters that can influence this range, for example, the level of pre-strain applied, strain rate used (both during pre-straining and post-HAP), the unloading modulus of the SMA wires and thermo-mechanical treatment of the material.

“Ghafoori *et al.* [48] investigated low and high cycle fatigue behaviour of pre-stressed Fe-based SMAs. The specimens were subjected to small strain amplitude, $\Delta\epsilon_0 = 0.07\%$ at different strain rates ($2.8 \times 10^{-4}/s$ and 1.4×10^0) and loading frequencies (0.002 Hz and 10 Hz). Ghafoori *et al.* reported that residual recovery stress (on unloading) in Fe-SMA also decrease when subjected to post-HAP deformation. Furthermore, they reported that at high strain rate ($1.4 \times 10^0/s$), the maximum stress on loading and minimum stress on unloading (basically the residual recovery stress on unloading), at a given strain amplitude, are slightly higher than the values recorded for low strain rates. This behaviour may be attributed to the thermo-mechanical coupling in SMAs.” Due to latent-heat release/absorption, specimens’ temperature oscillates during the cyclic loading. The mean specimen temperature increases with loading frequency until it reaches a saturated value in the high frequency range [49].

3.6. Re-usability of NiTiNb wires for HAP application

To investigate the effect of repeated thermo-mechanical loading on the recovery stress, one NiTiNb wire specimen was prestressed 10 times by heat-activation (i.e. in 10 steps of HAP) in Test 17. In each step, the specimen was first pre-strained by 8% (Point A–D in Figure 14) and then heated above the austenite finish temperature in constrained conditions until the maximum recovery stress was achieved (Point D–E in Figure 14). After achieving maximum recovery stress, the specimen was allowed to cool down to room temperature (Point E–F in Figure 14). Any loss in the recovery stress during cooling process was recorded. Once at room temperature, the specimen was held in position for approximately 30 mins – 1 hour. After this, the specimen was unloaded to zero stress (Point F–G in Figure 14) and the net residual strain was measured. The whole process was repeated 10 times (in 10 steps) on the same specimen. The aim of this test is to see if the same wire could be re-used for different application. For example, in one application an NiTiNb SMA wire is used for tightening of a joint and, in another application, the same SMA wire is re-used in prestressing of concrete beams. These two applications could be in two different set-ups and on two different occasions.

Test 17 was conducted at a strain rate of $3.3 \times 10^{-3}/s$, which corresponds to maximum crosshead speed of 0.2mm/min per mm of original gauge length, as recommended by ASTM F 2516-07 [50]. The strain was monitored using video extensometer over a gauge length (lg) of 50mm at the mid height of the specimen. The pre-strain chosen for this test was equal to 8% because the optimum level of pre-strain for achieving maximum recovery stress in the NiTiNb SMA (used in this study) is $\sim 8\%$.

Figure 14 presents the stress-strain curve obtained (using the video-extensometer) during the Step-1 of Test 17. Also given in the figure (see inset) is the average stress-‘strain’ curve for the same step obtained using crosshead data. While the crosshead was constrained in position (see path D–E in the inset in Figure 14) during entire heating process a considerable amount of strain recovery within the gauge length was observed. A similar observation was made in Test 16 discussed in the previous section. The strain recovered upon unloading (Point C–D), strain recovered within the gauge length on heating due to shape memory effect (Point D–E) and the residual strain on unloading after HAP, $\epsilon_{res}^{PS=8\%}$ are indicated in Figure 14. $\epsilon_{res}^{PS=8\%}$ retained at the end of each step is believed to unrecoverable plastic strain.

Figure 14: Stress-strain plots of NiTiNb wire specimen tested in the 1st step of HAP, Test-17.

In Figure 14, one can see that a significant amount of unrecoverable residual strain, $\epsilon_{res}^{PS=8\%}$ is retained in the specimen in each step. The specimen extended axially and reduced in cross sectional area in each step. The nominal stress in the wire was therefore calculated using the actual reduced cross-sectional area of the specimen measured in each step. The reduced diameter in each step was obtained using the following equation:

$$\varphi_j = \varphi_i \cdot (1 - \nu \cdot \epsilon_{res,i}) \quad \text{for } i \geq 1 \quad \dots (1)$$

where, φ_j is the diameter of the specimen in the j^{th} step, where $j = i+1$, φ_i is the diameter of the wires in i^{th} step, ν is the Poisson's ratio that was assumed equal to 0.3, and $\epsilon_{res,i}$ is the longitudinal residual strain retained in i^{th} step. The above method assumes that the cross-sectional area of the specimen is uniformly reduced over the gauge length however, when measured physically, a minor variation along the length of the specimen was noted.

The effect of repeated thermo-mechanical loading (multiple thermal activations) on the recovery stress is presented in Figure 15(a). Also given in Figure 15(a) is the cumulative residual strain retained in the specimen. A maximum recovery stress of about 600 MPa was recorded in Step-1. Both the maximum recovery stress and the residual recovery stress at room temperature reduced gradually in each step. By the end of Step-10, a reduction of ~26% in the maximum recovery stress and 31% in residual recovery stress retained at room temperature was recorded. Considering that by the end of Step-10 a cumulative residual strain (plastic strain) of about 25% is introduced in the specimen the effect of repeated HAP appears does not appear reduce the recovery stress by much. It is interesting to note that even after retaining a significant level of permanent residual strain, the SMA specimen continued to exhibit the shape memory effect.

The maximum recovery stress developed in Test-17 (Step-1) was about 100 MPa less than what was recorded in Test 11 even though in both the tests the specimens were pre-strained by same amount, equal to 8%. This difference is because in this test (Test 17) heating a portion of the specimen inside the grip was not possible. The reason for this is discussed in detail in the previous section.

Hosseini *et al.* [28] carried out two thermal activations on a Fe-based SMA specimens. The first heating step (thermal activation) was carried out after pre-straining the specimen and second after subjecting the specimen to two million loading cycles of very small strain amplitudes $\Delta\epsilon_0 = 0.07\%$ and $\Delta\epsilon_0 = 0.105\%$. They found that heating Fe- SMA specimens (second thermal activation) could help regaining considerable portion of the recovery stress lost during the cyclic loading.

Figure 15: Effect of repeated use of NiTiNb wire on recovery stress; (a) recovery stress and cumulative residual strain and; (b) corresponding stress-temperature plots.

Figure 15(b) presents the plots of recovery stress vs temperature obtained in each step in Test-17. The purpose of plotting this graph is to demonstrate that repeated pre-straining of the SMA has a little effect on its the reverse transformation temperatures when compared to first cycle of HAP. As can be seen in Figure 15(b), the recovery stress in each step increases immediately after turning the heating on, which indicates that the A_s of the specimen in each step remains close to the room temperature in each HAP step. In each step, the increase in recovery stress is rapid up to ~100°C, after which recovery stresses increases only gradually until the maximum recovery stress is achieved (which can be confirmed by stress plateau in Figure 15(b)). This indicates that A_f of the specimens in each step is close to 100°C in each step. As the heating was carried out in a relatively non-uniform manner, the exact A_s and A_f values are hard to determine from these plots however, these plots provide a useful information on the effect of repeated pre-straining and heating on reverse transformation temperature (A_s and A_f) of the NiTiNb SMAs.

4. Conclusions

This paper discussed the results obtained from a series of experimental tests conducted on heat-activated prestressing of the NiTiNb SMA wires. The following conclusions are drawn from this study:

1. Pre-straining the NiTiNb SMA wire above its transformation strain limit ($\sim 8\%$) results in an increase of its reverse transformation temperatures, A_s and A_f . The increase in the reverse transformation temperatures depend on the level of pre-strain applied and the thermo-mechanical treatment of the SMA.
2. The reverse transformation temperatures of the NiTiNb SMA after pre-straining in each heat-activated prestressing (HAP) cycle are maintained more or less in the same range.
3. The strain rate is an important factor to consider while pre-straining SMAs wires for HAP. Pre-strain retained on unloading reduces significantly when a high strain-rate ($> 2 \times 10^{-3}/s$) is used. The reduction in the residual pre-strain could be as high as $\sim 75\%$ when strain rates $> 1 \times 10^{-1}/s$ is used. Therefore, in order to achieve maximum recovery stress, NiTiNb SMA should be pre-strained at low strain rates (preferably $< 2 \times 10^{-3}/s$).
4. The local strain retained in the transformation bands significantly influences the recovery stresses developed on heating. Two specimens with same average residual pre-strain but one with relatively lower local strain in the transformation bands could develop lower recovery stresses than the specimen with higher strain concentration in the transformation bands.
5. The optimum level of pre-strain for NiTiNb SMA is found to be close to transformation strain (8%) of the SMA. A substantial level of recovery stress is developed (~ 700 MPa) in the SMA when an optimum pre-strain is applied. Most of the recovery stress (~ 550 MPa) developed on heating is retained at ambient temperature and could be exploited in many civil engineering applications.
6. The range of post-HAP stress plateau in a stress-strain curve depends on the pre-strain history of the SMA. For a fully transformed NiTiNb SMA wire (i.e. the SMA pre-strained by $> 8\%$) the stress plateau is found to be considerably shorter than in virgin specimen (i.e. $\sim 5\%$ in post-HAP as compared to 8% in virgin specimen).
7. Under post-HAP cyclic loading, recovery stress is reduced progressively as the applied strain is increased. For an optimum pre-strain level ($\sim 8\%$ for SMA used in this study), the range of strain over which recovery stress is effective is found to be relatively large ($\sim 3.5\%$) in NiTiNb SMA.
8. NiTiNb SMA wires can be repeatedly used for HAP with only a minor reduction in maximum recovery stress in each subsequent step. After 10 pre-straining/heating cycles, a reduction of 26% was recorded in the maximum recovery stress. The effect of cumulative residual strain (plastic strain) appears to have relatively small effect on recovery stress.
9. To increase the confidence of engineers in this material and to encourage them to use it in real structures, a need for the standardisation of the material and its manufacturing process is required.

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