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Thermo-mechanical characterisation of NiTi-based shape memory alloy wires for civil engineering applications

Raj Suhail¹, Giuseppina Amato¹ and Daniel McCrum²

Abstract
Shape Memory Alloys (SMAs) exhibit a complex material behaviour due to thermo-mechanical coupling. At present, the understanding of their material behaviour under different thermo-mechanical loading conditions relevant to civil engineering applications is lacking even though they have been widely used in the past decade or so, for example mechanical behaviour at constant stress but variable temperature loading has not received much attention in civil engineering. In this study a comparative analysis of the mechanical behaviour of various NiTi-based SMAs is carried out to investigate: (1) stress-elongation and stress-temperature response under direct tensile loading; (2) thermo-mechanical behaviour under constant stress condition but under variable temperature loading (to investigate their ability to exhibit shape memory effect, if any, while carrying load); and (3) the ability to develop and retain recovery stress at room and sub-zero temperatures. Essentially, all these tests aim at investigating the potential/limitations of the SMAs in civil engineering in particular re-centring and heat-activated prestressing applications. The results from this study showed that at a constant stress of 600 MPa a large forward transformation strain, in the range of 5%–11%, is produced in NiTi-based SMAs when the temperature is lowered to or below the room temperature. This material behaviour of NiTi-based SMAs could be detrimental in many civil engineering applications of NiTi-based SMAs. However, all NiTi-based SMAs used in this study were found to have a good potential for re-centring applications, but only austenitic NiTi-based SMAs were found suitable for long-term prestressing applications. The maximum recovery stress developed on heating with constrained boundary conditions ranged between 230–750 MPa.

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

1. Introduction
Over the past few decades, many different types of shape memory alloys (SMAs) have been developed. Due to their unique material properties, SMAs have attracted the attention of many civil engineer researchers. In civil engineering, however, mostly NiTi and Fe-based SMAs have been used. NiTi-based SMAs exhibit the most effective memory and superelastic properties among all known polycrystalline SMAs (Shaw et al., 2008). The selection of a particular SMA type depends on the intended application. SMAs 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, in superelastic bracing systems for seismic applications (Dolce et al., 2000; McCormick et al., 2007) or the shape memory effect (SME), which includes applications such as active confinement (Choi et al., 2008; Shin and Andrawes, 2009; Suhail et al., 2020a) and heat-activated prestressing (Czaderski et al., 2006; Shahverdi et al., 2016; Sinha et al., 2020; Suhail, 2018; Suhail et al., 2015, 2018, 2020b). As SMAs have started to gain wide acceptance as a civil engineering building material, understanding the complex material behaviour of SMAs from a civil engineering perspective is imperative. Although many studies have been conducted on material characterisation of SMAs in the past (Dolce and Cardone, 2001; Dommer and Andrawes, 2012; Ozbulut et al., 2015, Suhail et al., 2016, 2020c) however, one important aspects of the material behaviour of

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SMAs that has been overlooked or received only minimal attention is the effect of external stress/applied pre-strain on the transformation temperatures and the subsequent consequences, due to shift in the transformation temperatures, on the safety and efficacy of SMA application.

Literature has shown that the unique material behaviour of SMAs is accompanied by a highly complex material behaviour and as stated by Shaw et al. (2008) “It is not an amateur sport”. Depending upon the alloying elements and thermo-mechanical treatment SMAs exhibit different behaviour in different conditions and are highly sensitive to variation in temperature, phase in which it is used, loading pattern, strain rate and pre-strain conditions. Literature (Dagdelen et al., 2020; Daly et al., 2007; Funakubo, 1987; Hartl et al., 2006; Otsuka and Wayman, 1999; Saburi, 1999; Zhao et al., 1990) shows that even a slight variation in the percentage content of the alloying elements of SMA can result in significantly different thermo-mechanical properties. For example, a small increase in Ni content from 50% in equiatomic Ni$_{50}$Ti$_{50}$, to 51% in Ni$_{48}$Ti$_{49}$, the transformation temperatures reduces from $M_t = 15^\circ$C and $A_t = 89^\circ$C to $M_t = -153^\circ$C and $A_t = -40^\circ$C, respectively (Funakubo, 1987; Otsuka and Wayman, 1999), where $M_t$ and $A_t$ are the martensitic finish and austenitic finish temperatures, respectively. Similarly, with an addition of a tertiary element such as Niobium to Ni-Ti matrix, for example, in Ni$_{47}$Ti$_{44}$Nb$_{9}$, the transformation temperature range of the SMA can be increased significantly. For example, Zhao et al. (1990) reported transformation temperature range of 140°C with $M_t = -175^\circ$C to $A_t = -35^\circ$C in NiTiNb SMAs, which is significantly higher than typically found in binary NiTi SMAs. Studies (Duerig et al., 2013; Otsuka and Wayman, 1999) have further shown that the phase transformation temperature not only depend on the composition but also thermo-mechanical treatment. Thermo-mechanical treatment such as cold rolling and annealing alter the mechanical behaviour of the SMA quite significantly. Cold rolling alone leads to lower recoverable strains due to the introduction of random dislocations into the material but the subsequent annealing restores shape memory effect by rearranging the dislocations (Duerig et al., 2013; Mitwally and Farag, 2009).

This paper does not intend to present a complete review of the materials science and mechanics of the SMAs as the literature regarding this is extensive. For a comprehensive review on the thermo-mechanical behaviour of SMAs and the parameters that effect the thermo-mechanical behaviour of an SMA, readers are directed to ref. (Churchill et al., 2009a, 2009b, 2010; Kumar and Lagoudas, 2008; Otsuka and Wayman, 1999; Shaw et al., 2008; Suhail, 2018). In this paper, focuses is laid on the effect of external stress/applied pre-strain on the transformation temperatures and subsequent consequences on the civil engineering application such as in bracing systems and heat-activated prestressing. Several studies (Brinson, 1993; Liang and Rogers, 1997; Todoroki, 1990; Wu et al., 2003) have been carried out in the past to investigate the dependence of transformation temperatures on applied stress (constant stress). It is well established that under the external stress there occurs a shift in the transformation temperature of SMAs. Wu et al. (2003) showed that transformation temperatures under applied stresses vary non-linearly. They found that transformation temperatures are linear function of applied stress up to at a stress level of $\sigma_s$ and above the stress level of $\sigma_f$, where $\sigma_s$ is the critical stress for the start of martensite reorientation (sometimes also referred to as yield stress) and $\sigma_f$ is the stress corresponding to completion of martensite reorientation that is, stress corresponding to end of stress plateau in a stress-strain plot. The transformation temperature, within the stress range between $\sigma_s$ and $\sigma_f$ is found to rise sharply giving an overall non-linear behaviour. Studies (Wu et al., 2003) have further showed that the microstructure of martensite is different at different external stress levels, that is, oriented martensite form at high stress level, self-accommodated martensite form at low stress level and partially oriented martensite forms at intermediate level. As the transformation temperatures depend on chemical composition and treatment history, SMAs with different compositions subjected to different thermo-mechanical treatment may exhibit a different relationship between transformation temperature and external stress. The total shift in the transformation temperature under the influence of an external load would determine whether an SMA is applicable for a given civil engineering application or not.

The shift in the phase transformation temperature due to applied pre-strain has implications on the recovery stress also. Pre-strain, transformation temperature and recovery stress share a complex relationship. Suhail et al. (2020b) showed that forward transformation temperatures follow a linear relationship with applied pre-strain that is, greater the applied pre-strain, greater in the shift in the forward transformation temperatures. Other studies (Choi et al., 2013; Li et al., 2003; Prof and Duerig, 1990) have shown that recovery stress not only depend on the chemical composition and transformation temperatures of an SMA but also only the level of pre-strain applied. Prof and Duerig (1990) showed that the recovery stress in NiTiFe (47%Ni, 50%Ti and 3%Fe (at. wt.%)) SMA varied significantly with the applied pre-strain. The recovery stress in NiTiFe first with the increase in the level of pre-strain up to a certain point and then it decreases. The same trend was observed by Suhail et al. (2020b) for NiTiNb SMAs. For a safe an effective use of SMA
in heat-activated prestressing applications, the shift in the phase transformation in SMAs should be such that the forward transformation temperatures are well above the room temperature so that applied pre-strain (deformed shape) is retained at room temperature and the shift in reverse transformation temperatures does not cross the minimum temperature expected in the proposed application.

The dependency of phase transformation temperature on applied stress or applied pre-strain could be a crucial piece of information required for deciding the suitability of an SMA for a particular civil engineering application. Therefore, in this study, an experimental programme was carefully planned to investigate this behaviour. A discussion on the implications of applied stress dependency of transformation temperatures, and on the thermo-mechanical behaviour of prestressed SMAs is also presented. To present a comparative analysis of different SMA types, five different types of NiTi-based SMAs commonly used in civil engineering are investigated in this study.

### 2. Materials and experimental procedures

Five different types of NiTi-based SMA wires were procured for this study. SMA were either binary NiTi composition or ternary with Niobium added to the NiTi matrix. The SMAs were bought from two different suppliers, Xi’an Saite Metal Materials Development Co., Ltd, China (Supplier 1) and Intrinsic Devices, Inc., USA (Supplier 2). The diameter of the SMA wires varied between 2.0 and 2.2 mm. No information of the thermo-mechanical treatment or heat treatment history was available. SMAs were however said to retain the pre-strain on unloading.

#### 2.1. Material characterisation

Chemical composition of the SMAs used in this study was determined using Energy-Dispersive X-ray (EDX) analysis. An FEI Quanta 600 scanning electron microscope (SEM) equipped with an energy dispersive X-ray analyser was used to obtain the EDS spectrum of the samples. The samples, 2 mm in height, and diameter equal to the diameter of the wire, were cut from the as-received SMA wires using wire electrical discharge machining (EDM) and cast in an electrically conductive resin. Samples were then polished before placing them in the SEM for analysis. The phase transformation temperatures of the SMA wires were determined by differential scanning calorimetry (DSC) using a Perkin Elmer diamond DSC. The start and finish temperatures of the phase transformations were determined from the upright and inverted bell-shaped peaks found in the DSC curves of the SMAs.

#### 2.2. Thermo-mechanical tests

SMA specimens, taken from the as-received SMA wire, were tested under different thermal and mechanical boundary conditions to study their thermo-mechanical behaviour at room temperature, see Table 1 for the summary of all thermo-mechanical tests conducted in this study. The original undeformed length of the specimens in all thermo-mechanical tests was kept same, equal to 110 mm (clear distance between the grips).

At first, direct tensile tests, in which SMA specimens were subjected to monotonic tensile loading, were conducted at room temperature (RT). Two virgin specimens from each SMA type were stretched in Zwick Roell universal testing machine (UTM) until their failure. Specimens were deformed, in a strain-controlled mode at a strain rate of 3.3x10⁻³/s. The strain rate corresponds to maximum crosshead speed of 0.2 mm/min per mm of original gauge length, as recommended by ASTM F 2516-07 (Standard A.S.T.M ‘F 2516–07’, 2007). A high precision non-contact single camera video-extensometer was used to measure the strain in this study. The field of view (FOV) of the video-extensometer was set between 100 – 200 mm in this study, which resulted in a minimum accuracy of 1 μm in strain measurements. Due to the small diameter of SMA wires, the back-light method which produces a high contrast black and white images of the specimen, was preferred for strain measurement in this study. ‘Strain’ in the specimens was measured over a gauge length, l = 50 mm) specified at the mid-height of the specimen using custom-made target pieces, which were directly attached to the specimen. The gauge length
was chosen to be less than the maximum value recommended by ASTM F E8/E8M (Standard A.S.T.M `E8/ E8M’, 2009) for wires of uniform sections (80% of the specimens distance between the grips) but long enough to minimise the strain localisation effect. ‘Strain’ was estimated as $\delta/L$, where $\delta$ is the total deformation measured within the gauge length $L$. Due to strain localisation in the material, readers must note that the strain measured is the elongation response averaged over a gauge length and could differ from the local strain at a point in the material. Elongation response measured as $\Delta/L$, where $\Delta$ is the total crosshead displacement and $L$ is the total length of the specimen between the grips may include the contribution of the slip within the grips and thus may not be accurate enough. The temperature of the specimens was monitored throughout the tests using a Type-T thermocouple attached directly on to the surface of the specimens. The stress (load) in the specimen was obtained from the load cell attached to the UTM, which was calibrated to achieve an accuracy of $\pm 0.02\%$ of the applied load, in the range of 0 – 5 kN.

Following the direct tensile tests, thermo-mechanical tests were conducted to study the effect of temperature variation on the forward/reverse transformation strains in the SMAs held at constant stress of 600 MPa. Loading procedure adopted for investigating the effect of temperature variation on the forward/reverse transformation strains in the SMAs held at constant stress of 600 MPa. Table 2 provides the details on loading protocol followed in these tests along with the thermal and mechanical boundary conditions adopted in each loading step. The tests in this section were conducted in a force-control mode. Loading and unloading of the specimens was carried out at 50N/s. Temperature in these tests was varied between $-50^\circ C$ and $300^\circ C$.

In the last set of thermo-mechanical tests, tests were conducted to study recovery stress in the SMAs. Recovery stress in the SMA specimens was investigated before applying the pre-strain and also after applying a pre-strain. A pre-strain between 6% and 9% was applied to the specimens. The tests in this section were conducted in a strain-control mode. Loading and unloading of the specimens was carried out at a strain rate of $3.3\times10^{-3}/s$. Temperature in these tests was varied between $-50^\circ C$ and $250^\circ C$.

In some of the above tests, that is, Tests 6–15, heating and cooling of the specimens was required. Heating of specimens was carried out using two 2400 W fan heaters supplemented with a 2400 W heat gun. With this method, a temperature of about $300^\circ C$ was easily achieved, which is well above the $A_f$ of all SMAs used in this study. In some tests, that is, Tests 6–15, SMA specimens were required to cool down below zero degrees Celsius. Generally, liquid nitrogen is preferred to cool the temperature however, in this study a freezing spray containing fluorinated greenhouse gases HFC-134a was used to enable use of video-extensometer, which required the specimen to remain exposed throughout the tests. Using this spray, it was possible to reduce the temperature of wire specimens to approximately $-50^\circ C$. The disadvantage of this cooling method is that the temperature is brought down to sub-zero levels very quickly. The rate of cooling of the specimen was of the order of only few seconds as opposed to 30 – 45 minutes under natural conditions. Cooling spray was used only after the specimen had natural cooled down to room temperature ($\sim 18^\circ C$). Figure 1 shows the test set-up used for carrying out various thermo-mechanical tests in this study.

| Thermal/mechanical load step* | Mechanical and/or thermal boundary condition |
|-----------------------------|--------------------------------------------|
| Step-1 Point $a\rightarrow$ Point $b$ | Specimen is heated above its $A_f$. The crosshead of the UTM is allowed to move freely to monitor strain recovery or thermal expansion. (The initial heating step was carried out to ensure each specimen was in the stress-free austenite phase before the application of the load) |
| Step-2 Point $b\rightarrow$ Point $c$ | Specimen is loaded to a stress of 600 MPa while maintaining the temperature of the specimen above its $A_f$. |
| Step-3 Point $c\rightarrow$ Point $d$ | Specimen is held at a constant stress of 600 MPa but allowed to cool down naturally to room temperature. Strain produced is recorded. |
| Step-4 Point $d\rightarrow$ Point $e$ | Specimen is held at a constant stress of 600 MPa while the specimen is further cooled down sub-zero temperature. Strain produced is recorded. |
| Step-5 Point $e\rightarrow$ Point $f$ | Specimen while maintained at the constant stress of 600 MPa is heated above its $A_f$. Strain recovery (due to SME) during the heating process is recorded. |
| Step-6 Point $f\rightarrow$ Point $g$ | Specimen is unloaded to zero stress while maintaining the specimen at elevated temperature. |
| Step-7 Point $g\rightarrow$ Point $h$ | Specimen is allowed to cool down to room temperature while the crosshead of the testing machine is allowed to move freely to monitor any change in the strain. |

*See Figure 5 for Points a–h.
3. Results and discussion

3.1. Material characterisation

The EDX spectrums of the SMA samples are given in Figure 2. The chemical composition of the SMAs in atomic percentage (at.%) is given in Table 4. For the convenience of the readers, the equivalent wt.% of the compositions is also given in the Table 4. The results ignore the trace amounts of other interstitial elements. In the binary compositions, the nickel content varied from 48.8% (at.%) in Type-5 to 50.1% (at.%) in Type-2. In the tertiary compositions, nickel content was lower and varied from 44.2% (at.%) in Type-4 to 45.5% (at.%) in Type-1 SMA. The percentage content of Niobium was about 9.1% (at.%) in Type-1 while in Type-4, the percentage content of Niobium was 12.5% (at.%), about 3.5% higher than in Type-1.

Phase transformation temperatures of the SMAs are given in Table 4. Type-1, -2 and -4 SMAs had there $A_f$ well below the room temperature, RT ($\sim 18^\circ$C), and
were therefore in austenite phase at RT. Type-3 and -5 SMAs were in martensite phase at room temperature with their $A_f$ well above the RT.

### 3.2. Direct tensile tests

Two virgin specimens (Sp-1 and Sp-2) from each SMA type given in Table 4 were tested under monotonic tensile loading. The objective of these tests was to understand the overall thermo-mechanical behaviour of the wires at room temperature under monotonic tensile loading and to determine engineering parameters such as modulus of elasticity ($E_{RT}$), stress ($\sigma_f$) and strain (\(\varepsilon_f\)) corresponding to the onset of yielding (i.e. when strain localisation and detwinning of the specimens starts), stress ($\sigma_f$), and strain (\(\varepsilon_f\)) corresponding to the point of complete propagation of the localised reorientation bands (i.e. end of stress plateau), ultimate stress ($\sigma_{ult}$), and the fracture strain (\(\varepsilon_{fr}\)). The stresses $\sigma_f$ and $\sigma_{ult}$ and the corresponding strains $\varepsilon_f$ and $\varepsilon_{ult}$ were obtained by drawing tangents on stress-strain curves. The summary of the test results is tabulated in Table 5.

From Figure 3 and Table 5, it is evident that all SMAs used in this study exhibited a great deal of difference in their mechanical behaviour. The modulus of elasticity, $E_{RT}$ of the SMA specimens varied between 18 and 83 GPa. The elastic modulus of an SMA depends primarily on the phase of the SMAs, which in turn depends on the alloying elements and the thermo-mechanical treatment of the SMA. Generally, SMAs in martensitic phase exhibit a considerably lower elastic modulus, typically ranging from 20 to 40 GPa, than in austenitic phase, which typically ranges between 70 and 90 GPa. For austenitic NiTiNb SMAs used in this study, that is, Type-1 and -4 SMAs, the average modulus of elasticity was 79 and 68 GPa, respectively. Type-2 NiTi SMA, had an average modulus of elasticity equal to 55 GPa. The average modulus of elasticity of martensitic NiTi SMAs that is, Type-3 and -5 SMAs was 19 and 21 GPa, respectively.

Like $E_{RT}$, the yield stress of the SMAs also varied significantly, from $\sim 630$ MPa in Type-1 SMAs to $\sim 180$ MPa in Type-3 SMA (see Table 5). Type-3 and -5 SMAs, being martensitic at room temperature, exhibited considerably lower yield strength. The yield stress in martensitic SMAs is controlled by the friction of the martensite twin interfaces (Drexel et al., 2006) which leads to lower stress values than the austenitic yield stress values which takes place through material transformation from austenite to martensite. Studies have shown that variation in the composition of the SMAs can also lead to significant variation in yield stress even when the two SMAs are in the same phase. The general trend, in binary NiTi SMAs, is that a decrease in nickel content decreases the yield stress (Duerig et al., 2013). However, when the same SMA is subjected to different thermo-mechanical treatment, the yield stress of the NiTi SMAs may not follow the general trend for example, NiTi SMA with lower Ni content but higher degree of cold working may result in higher yield strength. In fact, the mechanical behaviour of NiTi-based SMAs greatly depends on the heat-treatment, amount of cold
working, processing history, transformation temperatures, test-temperature and small number of ternary additions. Addition of third or fourth element to NiTi leads to increase in the austenite yield strength and widening of the hysteresis width that is, lengthening of the martensite plateau as is found in Type-1 and -4 NiTiNb SMAs used in this study. Fu et al. (2009) reported that the effect of Nb content on the yield strength depend on the coaction, \(\beta\)-Nb phase composition strengthening mechanism and the Nb solid solution weakening mechanism. The yield strength first decreases and then increases with the increase in percentage Nb content.

From Figure 3 and Table 5, the martensitic plateau in the SMAs is found vary between 3% and 8% strain. The characteristic transformation strain, \((\varepsilon_t - \varepsilon_a)\) of an SMA depends on several parameters such composition of alloy, testing direction (whether longitudinal to transverse to rolling direction), deformation mode (whether tension, compression, or torsion) and thermo-mechanical treatment (Kumar and Lagoudas, 2008; Otsuka and Wayman, 1999). Studies (Mitwally and Farag, 2009) have shown that cold working alone dramatically affects the martensitic plateau, although it may increase the strength, but it also reduces the ductility. Cold working introduces a high density of random dislocations which impede the mobility of the twin boundaries. Generally, annealing step after the cold working is employed to restore the shape memory effects. Therefore, the amount of cold work and the annealing temperature (and annealing time) will dictate the trade-off between shape memory property and the strength of the SMA. Among all SMAs, the ductility of Type-5 was found to be the greatest. The rupture strain in Type-3 SMAs was just under 10%. Type-5, on the contrary failed at strain larger than 50%.

In Figure 4, two distinct temperature-strain elongation trends were obtained from the monotonic tensile tests of the SMAs. In Type-1, Type-2 and -4 SMAs, a sudden increase in the temperature took place as the specimens crossed the yield stress, see Figure 4(a), (b) and (d). While in Type-3 and -5 SMAs, the rise in temperature was gradual, see Figure 4(c) and (e). Yielding of SMAs indicate the start of either stress induced martensitic (SIM) phase transformation, as in the case of austenitic SMAs, or reorientation of martensitic variants from twinned to detwinned structure in martensitic SMAs. Both the mechanisms are accompanied by the localised release/absorption of latent heat (Churchill et al., 2009a; Leo et al., 1993; Shaw and Kyriakides, 1995; Zhang et al., 2010). In Type-1, -2 and -4 SMAs, due to the SIM phase transformation a sudden generation of large strain takes place resulting in large stress drops. Under the quasi-static tensile loading, the SIM phase transformation takes place through the manifestation of macroscopically observable domain(s) (or transformation bands; TBs), the evolution of which is strongly coupled with heat transfer within the material and the surrounding environment (Suhail et al., 2020c). The amount of latent heat produced depends on the number of local transformation bands, their active propagation fronts and the speed of the propagation fronts (Churchill et al., 2009a; Leo et al., 1993; Shaw and Kyriakides, 1995; Zhang et al., 2010). From the stress-elongation plots of Type-1, -2 and -4 SMAs, it appears that several TBs were formed in these specimens over the stress plateau region. The nucleation of a new TB is typically identified by stress drop in the stress-elongation plots (mostly in strain-controlled tests). The larger the stress drop, the larger is the strain produced and larger the latent heat released. The sharp increase in the temperature in Type-1, -2 and -4 SMAs was observed only up to a strain of approximately 10%, which is very close to the strain corresponding to the end of stress plateau in stress-elongation plots, which also marks the complete transformation of specimen into martensite phase. Type -1, -2 and -4 SMAs, resulted in an overall rise in temperature of about 15°C. In Type-3 SMAs, due to reorientation of martensite variant, a temperature rise just under 4°C was recorded. Type-5 SMA, also assumed to have undergone only martensite reorientation, resulted in the total temperature rise of 14°C which is considerably higher than Type-3 SMA. The reason for this is believed to be total heat accumulation over a significantly large deformation range. In type 5, heat accumulation took place gradually over a strain of 0.55 (almost five times the Type-3 SMA). When compared with Type-3 SMA, at 10% strain the rise in temperature in Type 5 is more or less the same as Type 3 SMA (≈4°C). The slope temperature-elongation (exothermic rate) curve was also the same in the two types of SMA.

From the above discussion, it is found that thermomechanical behaviour of SMAs can vary quite significantly, and depends on the number of parameters. For the safe use in civil engineering applications, civil-structural designers need to be aware of the influence of different parameters on the overall mechanical behaviour of SMAs.

### 3.3. Effect of variable temperature on transformation strains at constant non-zero stress

SMAs to be used in civil engineering applications will most likely be under some kind of load (stress). Under stressed conditions, the effect of temperature variation on the transformation strains (forward/reverse transformation strain) in SMAs may be a critical information required in deciding the feasibility of an SMA for a given civil engineering application. In order to investigate the effect at variable temperature on transformation strains of the SMA wires at constant non-zero
stress, specimens from each SMA type given in Table 4 were tested as per the loading protocol discussed in Table 2. A typical stress-elongation response and temperature-elongation response of austenitic and martensitic SMAs is given in Figure 5(a), (c) and (b), (d), respectively.

Figure 6(a) and (b) shows the bar plot of total strain recorded during Step-1 (Point a → Point b) and Step-3 and -4 (i.e. Point c → Point d and Point d → Point e) of the tests. The symbol ⊙ and ⊙ on the bars in Figure 6 indicate the number of times the specimen was tested. ⊙ indicates the test was conducted on the virgin specimen and ⊙ indicates the test was conducted on a reused SMA, after completing all the steps in test ⊙. All results are reported but for brevity only virgin specimen results that is, test ⊙ are discussed in detail. As expected, those specimens (i.e. Type-1, -2 and -4 SMAs) which were originally in their austenite phase at room temperature did not recover any strain during the initial heating step (Step-1) instead, a small positive strain due to thermal expansion was recorded in these specimens, see Figure 6(a). For Type-1 and -2 SMAs, a maximum thermal expansion strain of approximately 0.23% was recorded, which corresponds to a thermal co-efficient of approximately 11x10⁻⁶/°C. On the contrary, specimens (i.e. Type-3 and -5) which were in the thermal martensite phase at room temperature recovered a relatively large reverse transformation strain during the initial heating step; see Point a → Point b in Type-3 SMA in Figure 5(b) for instance. For Type-3 and -5 SMAs, maximum reverse transformation strain, (ε_{Max,rev, α = 0}) of approximately 2.45% and 2.80% was recorded in stress-free state in test ⊙, respectively.

In Step-4, at a constant stress of 600 MPa, when the temperature was lowered, a considerable amount of forward transformation strain was recorded in all SMAs. Among the martensitic NiTi SMAs, a maximum forward transformation strain (ε_{fwd,600}) of 11.6% was recorded in Type-5 SMAs (refer to Figure 6(b)). As the M_f of the Type-5 SMA was above room temperature, almost all the forward transformation strain was produced before reaching room temperature (~18°C) that is, between the Points c → Point d. Austenitic SMAs also produced some forward transformation strain due to the shift in the phase transformation temperatures. In Type-1 austenitic SMA, having the original Ms well below the room temperature, only a small forward transformation strain was generated at room temperature. A forward transformation strain of only 0.1% (ε_{fwd}, RT) was generated in the Type-1 SMA at room temperature. However, as the temperature was brought down further, below the RT, a large forward transformation strain, equal to 6.83%, was rapidly generated at 9°C, see Figures 5(c) and 6(b). Clearly, M_s of Type-1 SMA appears to have shifted to approximately 9°C
under constant stress of 600 MPa. In Type-2 and -4 austenitic SMA, a relatively large forward transformation strain was produced at RT, which indicated $M_s$ of the SMA was shifted above the RT. An additional amount of forward transformation strain was produced when the temperature was brought down below RT.

On heating SMA specimens in Step-5, that is, from Point e → Point f, only partial recovery of strain was achieved during reverse transformation of the SMAs. Figure 6(c) presents the summary of results obtained in Step-5 of the tests. A maximum reverse transformation strain ($\varepsilon_{\text{rev}, \sigma = 600}^{\text{Tr}}$) of 2.63%, was recorded in the Type-3 SMA. In the Type-1 SMA, $\varepsilon_{\text{rev}, \sigma = 600}^{\text{Tr}}$ of approximately 2% strain was recovered. The Type-5 SMA on the contrary expanded and produced a small extension of 0.6%. Clearly, all SMAs used in this study showed only a limited strain recovery. The main reasons for this behaviour are: (1) thermo-mechanical treatment of an SMA; and (2) the effect of constant stress during the phase transformation. Thermo-mechanical treatment such as annealing temperature and annealing time could be responsible for limited strain recovery during reverse transformation if annealing is carried out at temperature greater than the recrystallisation temperature of an SMA. Temperatures higher than 600°C refines SMAs in a fully annealed conditions and imparts poor mechanical properties and leads to significant level of irrecoverable plastic strain during the reverse transformation (Duerig et al., 2013; Saburi, 1999). The behaviour becomes complicated

Figure 5. Effect at variable temperature on transformation strains (forward/reverse transformation strain) at constant single stress of 600 MPa: (a) typical stress versus elongation response of austenite SMA (Type-1 SMA), (b) typical stress versus elongation response of martensite SMA (Type-3 SMA), (c) typical temperature vs elongation response of austenite SMA (Type-1 SMA) and (d) typical temperature vs elongation response of martensite SMA (Type-3 SMA).

Note the difference in Step-1 (Point a → Point b) in martensitic and austenitic SMAs.
The presence of external stress may also impede the strain recovery, for example Jin et al. (2001) reported that strain recovery in NiTiNb SMA would be incomplete due to plastic deformation of Nb-rich phase if the external stress exceed by 160 MPa. Pobedennaya et al. (2015) further showed that higher the level stress lesser is the strain recovery in NiTiNb SMA.

All specimens upon unloading in Step-7 resulted in a significant residual strain. The net residual strain retained by the SMAs at the end of tests is given in Figure 6(d). A maximum net residual strain ($\varepsilon^{\text{net}}_{\text{res}}$) of 10.4% was recorded in Type-5 SMA. Type-1 and -4 retained a lowest $\varepsilon^{\text{net}}_{\text{res}}$ of approximately 4.8%.

The above test results highlight the difference in the thermo-mechanical behaviour of different SMAs under...
stressed conditions. More importantly, the results highlight the consequences of large shift in the phase transformation temperature of some SMAs under the influence of external stress. The shift in the phase transformation temperature can be significant to an extent that even an austenitic SMAs having stress free $M_s$, well below the room temperature (e.g. Type-1 SMA) could result in a significantly large forward transformation strain at temperature close to RT. This material behaviour of SMAs could jeopardise the safety of a structures subjected to seasonal ambient temperature that may vary from $-20^\circ$C to $50^\circ$C. A forward transformation strain of even 1% due to temperature fluctuation could result in a significantly large deformation for example, in a beam retrofitted with SMA, 1% forward transformation strain can lead to large midspan deflections which in turn may lead to failure in fulfilling the serviceability criteria. Similarly, an SMA fastener in a beam-column joint connection exposed to low temperatures, could experience a release of sudden large strain (forward transformation strain) that could compromise the integrity of the structure. It must be noted that the stress level chosen in the above tests is relatively high and very close to the yield strength of the Type-1 SMA. At lower stresses, within the elastic region, no or significantly lower forward transformation strain may be generated in the austenite SMAs (Types-1, -2 and -4). For real-world applications, the amount of forward transformation strain $\varepsilon_{\text{Max.fwd,600}}$ produced due to temperature variation will dictate the feasibility of the SMA for a particular application.

SMAs used in this study although exhibited only partial strain recovery but this material feature can be effectively used in many civil engineering applications (short term) for example, in self-centring of a structural system or closing of cracks in reinforced concrete elements. However, for these types SMAs, a relatively larger pre-straining may be required as a result of partial strain recovery. Shape recovery when enforced in constrained conditions could be used in heat-activated prestressing in civil engineering. A more detail on this is presented in the next section.

### 3.4. Constrained shape recovery (heat-activated prestressing)

To study the constrained shape recovery (heat-activated prestressing) of SMAs used in this study, specimens from each SMA type given in Table 4 were tested as per the loading protocol discussed in Table 3. Specimens were evaluated in terms of: (1) the level of recovery stresses developed before and after applying a pre-strain; and (2) recovery stresses retained at room temperature and at sub-zero temperate range.

Figure 7 shows the nominal stress-elongation response and nominal stress-temperature response of the different types of the SMAs tested for constrained shape recovery. Two different plots of nominal stress-elongation responses are given in this figure. In the first set of plots, Figure 7((a), (d), (g), (j) and (m)) elongation response was obtained using the crosshead readings as $\Delta/L$. These figures are plotted to demonstrate the boundary condition at each stage of the tests. In the second set of plots, Figure 7((b), (e), (h), (k) and (n)) the elongation response, $(\delta/L)$ of the specimens were obtained using the video-extensometer measuring. The third set of figures, Figure 7((c), (f), (i), (l) and (o)) present the nominal stress-temperature response of the SMAs. For simplicity, only heating and cooling cycles are plotted in these figures that is, only Step-1 and -2 (first heating and cooling cycle) and Step-5 and -6 (second heating and cooling cycle) are plotted in these figures.

As expected, the specimens which were already in austenite phase (i.e. Type-1, -2 and -4 SMAs) did not develop any recovery stress during the initial heating

**Table 3.** Loading procedure adopted for heat-activated prestressing of various shape memory alloy wires used in this study.

| Thermal/mechanical load steps | Boundary conditions and thermal conditions |
|------------------------------|-------------------------------------------|
| **Step-1**  | Point $a \rightarrow$ Point $b$ | Specimen is heated above its $A_r$ in constrained conditions, that is, by restraining the crosshead movement. The recovery stress or the stress developed due to thermal expansion on heating in this step is recorded. |
| **Step-2**  | Point $b \rightarrow$ Point $c$ | Specimen is allowed to naturally cool down to room temperature. Any loss or gain in the stress developed in Step-1 is recorded. |
| **Step-3**  | Point $c \rightarrow$ Point $e$ (through Point $d$) | Specimen is pre-strained between 6% and 9% elongation at room temperature. |
| **Step-4**  | Point $e \rightarrow$ Point $f$ | Specimen is unloaded to stress free state at room temperature. |
| **Step-5**  | Point $f \rightarrow$ Point $g$ | Specimen is heated in the constrained condition. The recovery stress developed on heating due to shape memory effect is recorded. |
| **Step-6**  | Point $g \rightarrow$ Point $h$ | Specimen is allowed to naturally cool down to room temperature and further cooled down to sub-zero temperature using the freezing spray. Any loss or gain in the stress in Step-6 is recorded. |
| **Step-7**  | Point $h \rightarrow$ Point $i$ | Specimen is unloaded to stress free state (zero-stress). The net residual strain retained on unloading is recorded. |

*a* See Figure 7 for Points $a$–$i$. 
Table 4. Material specification of the SMAs used in this study.

| SMA type | Alloying elements | Approx. chemical composition | Transformation temperatures | Phase at room temperature (~18°C) | Diameter [mm] |
|----------|-------------------|------------------------------|-----------------------------|----------------------------------|--------------|
|          |                   |                              | $M_f$ | $M_s$ | $A_s$ | $A_f$ |               |             |
|          |                   |                              | [°C] | [°C] | [°C] | [°C] |               |             |
| Type-1   | Ni, Ti, Nb        | Ni$_{45}$Ti$_{45}$Nb$_{10}$ | –180 | –80  | –24  | 2    | Austenite     | 2.0         |
| Type-2   | Ni, Ti            | Ni$_{50}$Ti$_{45}$Nb$_{5}$  | –32  | –20  | 4    | 12   | Austenite     | 2.0         |
| Type-3   | Ni, Ti            | Ni$_{49}$Ti$_{45}$Nb$_{12}$ | 12   | 20   | 72   | 77   | Martensite    | 2.0         |
| Type-4   | Ni, Ti, Nb        | Ni$_{48}$Ti$_{45}$Nb$_{12}$ | –140 | –90  | 0    | 0    | Austenite     | 2.2         |
| Type-5   | Ni, Ti            | Ni$_{48}$Ti$_{51}$Nb$_{12}$ | 52   | 65   | 94   | 108  | Martensite    | 2.2         |

Table 5. Experimentally derived material parameters for as-received SMA wires.

| Material properties   | Type-1 (Sp-1) | Type-1 (Sp-2) | Type-2 (Sp-1) | Type-2 (Sp-2) | Type-3 (Sp-1) | Type-3 (Sp-2) | Type-4 (Sp-1) | Type-4 (Sp-2) | Type-5 (Sp-1) | Type-5 (Sp-2) |
|-----------------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| $E_{RT}$ (GPa)        | 75            | 83            | 54            | 57            | 20            | 18            | 72            | 65            | 21            | 22            |
| $\sigma_{f}$ (MPa)    | 630           | 635           | 565           | 544           | 183           | 177           | 604           | 589           | 292           | 297           |
| $e_s$                 | 0.0086        | 0.0077        | 0.0110        | 0.0107        | 0.0090        | 0.0095        | 0.0084        | 0.0113        | 0.0137        | 0.0135        |
| $\sigma_{g}$ (MPa)    | 782           | 805           | 587           | 573           | 210           | 209           | 663           | 657           | 322           | 330           |
| $e_f$                 | 0.083         | 0.083         | 0.032         | 0.0296        | 0.0426        | 0.043         | 0.084         | 0.077         | 0.043         | 0.047         |
| $e_f - e_s$ (%)       | 7.4           | 7.5           | 2.1           | 1.9           | 3.4           | 3.3           | 7.6           | 6.6           | 2.9           | 3.4           |
| $\sigma_{ult}$ (MPa)  | 1067          | 1059          | 947           | 929           | 1057          | 1073          | 867           | 875.86        | 1012          | 1018          |
| $\varepsilon_{ult}$   | 0.22          | 0.24          | 0.312         | 0.288         | 0.084         | 0.092         | 0.2791        | 0.3281        | 0.518         | 0.543         |
| $\varepsilon_{f}$     | 0.22*         | 0.26          | 0.312*        | 0.288*        | 0.084*        | 0.099         | 0.2791*       | 0.3281*       | 0.518*        | 0.543*        |

*Specimens in which the fracture took place outside the gauge length.

step, see Figures 7 and 8(a), instead of a small negative stress (approximately 100 MPa) due to thermal expansion was recorded. The results are consistent with the previously obtained results in section 3.3. Martensitic SMAs (i.e. Type-3 and -5 SMAs) on the contrary, underwent reverse transformation on heating and therefore produced a considerable amount of recovery stresses in the specimens. A maximum recovery stress ($\sigma_{PS}^{Max, recov}$) of approximately 380 MPa was developed in the Type-3 SMA on heating, and approximately 150 MPa in the Type-5 SMA. As $M_f$ of the martensitic SMAs was considerably higher than the room temperature, the recovery stress developed during the initial heating in Type-3 and -5 was completely lost when the specimens were allowed to cool down to RT. The summary of the results obtained from initial constrained shape recovery (i.e. without applying any pre-strain (PS = 0); Step-1 and -2) is given in Figure 8(a).

In Step-3, when the specimens were pre-strained, between 6% and 9% and subsequently heated to ~250°C in Step-5, a significant level of recovery stress was developed in all specimens, see Figures 7 and 8. A maximum recovery stress of approximately 750 MPa was developed in Type-3 SMA. The lowest level of recovery stress was observed in Type-2 SMA, approximately 238 MPa. The level of recovery stress developed during the constrained shape recovery depend on number of parameters such as shape memory setting, which in turn depends on the composition and thermal-mechanical treatment, pre-strain level, ambient temperature and the activation temperature. SMA with greater characteristic transformation strain range (i.e. martensitic stress plateau range) and better shape recovery tend to produce greater recovery stress while as SMAs with high density of random dislocations at molecular level recover less strain and therefore produce less recovery stress. The summary of the results obtained from second constrained shape recovery of the SMAs (i.e. after applying pre-strain (PS); Step-5 and 6) is given in Figure 8(b).

In Step-6, when the specimens were allowed to cool down to room temperature, the recovery stress in the martensitic SMAs was completely lost, just like in the first cooling cycle. However, austenitic SMAs (Type-1, -2 and -4) retained most of the recovery stress developed in Step-5; meaning that the shift in the $M_f$ did not exceed the room temperature. A recovery stress ($\sigma_{PS}^{Max, recov}$) of approximately 500 MPa was retained in the Type-1 SMA at room temperature, which is the maximum recovery stress retained among all SMAs discussed in this section. A summary of the recovery stresses developed and retained by the SMAs is given...
Figure 7. Nominal stress-elongation of the SMAs before and after the pre-strain. Left column plots (a), (d), (g), (j) and (m): crosshead displacement over total length of the specimen between the grips; Middle column plots (b), (e), (h), (k) and (n): deformation in the gauge region using video-extensometer measurements; Right column plot (c), (f), (i), (l) and (o): nominal stress-temperature responses of the SMAs before and after the pre-strain.
in Table 6. A significant level of permanent residual strain (strain measured between Point i and Point a) was retained in all the SMAs. Figure 9 shows the bar plots comparing the net residual strain retained by different SMAs used in this study.

The above test results highlighted the difference in the behaviour of martensitic and austenitic SMAs. Although, martensitic SMAs lose all the recovery stress when the temperature is lowered to room temperature, they can still be used in many civil engineering applications that requires temporary pre-stressing for example, in re-centring applications, tightening of joint connections etc. For applications where recovery stress needs to be retained at ambient temperature for example, active confinement or heat-activated pre-stressing of structural members, austenitic SMAs like Type-1, -2 and -4 SMAs can be effectively used within a wide range of ambient temperature. In Figure 7 it is found that even when the temperature is brought down to \(-20^\circ C\) (see Figure 7(c)) a minimum of 320 MPa was still retained in Type-1 SMA. The recovery stress lost due to lowering of the temperature was naturally gained again when the temperature rose to room temperature again. Therefore, in most civil engineering applications, the SMAs like Type-1 SMA used in this study, are expected to perform safely and without the risk of losing of recovery stress completely. Further, combing the results from section 3.3 and 3.4, it can be concluded that the behaviour of SMA could be significantly different, under the application of external load and the under the recovery stresses developed internally. Unlike in section 3.2 in which Type-1 SMA under the influenced of external stress (of 600 MPa) produced a significantly large forward transformation strain at \(9^\circ C\), the SMA did not lose much of the recover stress developed internally at this temperature.

Table 6. Summary of the results obtained from heat-activated prestressing of the SMAs.

| SMA type | Recovery stress (or thermal expansion) before pre-strain | Net residual strain (%) |
|----------|--------------------------------------------------------|-------------------------|
|          | At Max. temp. Retained at room temp. | Pre-strain applied | At Max. temp. | At room temp. | |
|          | [MPa] | [MPa] | [%] | [MPa] | [MPa] | |
| Type-1   | \(-115^*\) | 0 | 6 | 550 | 497 | 2.5 |
| Type-2   | \(-117^*\) | 0 | 6 | 236 | 221 | 2.7 |
| Type-3   | 374 | 0 | 7 | 750 | 0 | 4.2 |
| Type-4   | \(-107^*\) | 0 | 6 | 296 | 284 | 3.2 |
| Type-5   | 146 | 0 | 9 | 455 | 0 | 5.7 |

*Negative sign is due to thermal expansion of SMAs held in constrained conditions.

Figure 8. Recovery stress developed in different types of SMAs used in this study: (a) before pre-straining and (b) after pre-straining.
PS: pre-strain; PS = X, where X varied from 6% to 9%
Figure 9 shows a comparison of net pre-strain applied \( \left( PS_{\text{net}} \right) \), strain recovered on unloading \( \left( e_{\text{recov Unl}} \right) \) and net residual strain retained \( \left( e_{\text{net res}} \right) \). Clearly, austenitic SMAs used in the study retained low residual strain than martensitic SMA. This is due the applied pre-strain being very close or above the transformation strain of the SMA.

### 4. Conclusions

In this study, a series of experimental tests were conducted to investigate the thermo-mechanical behaviour of shape memory alloy wires for civil engineering applications. Tests were conducted to investigate stress-elongation response under direct tensile loading, shape memory effect at constant stress of 600 MPa and recovery stresses developed in constrained conditions at different pre-strain levels. The following conclusions can be drawn from the test results:

1. The stress-strain behaviour of SMAs depend on a number of parameters. The chemical composition, phase of the material, thermo-mechanical treatment during the production phase, all have a significant effect on the overall mechanical behaviour of SMAs. Therefore, it is important to have complete information on these parameters before the selection of an SMA for a particular civil engineering application.

2. In general, the addition of Nb to NiTi results in favourable effects on the yield strength, stress plateau \( \left( \varepsilon_s - \varepsilon_y \right) \), ductility, phase transformation temperatures and the recovery stress at room temperature. However, the percentage content of Nb can significantly alter these properties.

3. Under a constant stress (600 MPa), temperature fluctuation could result in a large shift in the transformation temperature of an SMA which in turn could risk the safety of many civil engineering applications.

4. A considerable amount of recovery stress (up to \(~700\) MPa) can be developed in NiTi-based SMAs. The phase transformation temperature and the thermo-mechanical treatment of an SMA are found to be the key parameters for the selection of an SMA for a civil engineering application.

5. At room temperatures, only the pre-strained austenitic SMAs retained much of the maximum recovery stress developed on heating. Depending on the pre-strain level and the consequent shift in the phase transformation, recovery stress retained at sub-zero level can be relatively high \((\sim 55\%)\) of maximum recovery stress developed on heating), for example Type-1 SMA retained 308 MPa at \( -20^\circ \text{C} \).

At present, the number of alloys that exhibit shape memory effect is large, and this number is continuously evolving. Each SMA has different material properties. Even SMAs with similar composition can have significantly different thermo-mechanical behaviour if subjected to different thermo-mechanical treatment. The reliability of the material behaviour is the most important factor for the safe use of SMAs in civil engineering applications. Therefore, a need for standardisation of the material specification of SMAs including the manufacturing process (i.e. thermo-mechanical treatment) is required for consistent and safe design in civil engineering applications.

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