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In situ observation of NiTi transformation behaviour: a micro-macro approach

Kasun S Wickramasinghe, Rachel A Tomlinson, Jem A Rongong
Department of Mechanical Engineering, University of Sheffield, Mappin St. S11 3JD, UK

ABSTRACT: A novel experimental investigation is presented of thermally and stress induced transformation behaviour of a Polycrystalline NiTi Shape Memory Alloy (SMA) plate for flexural-type applications: *In situ* techniques are employed to allow simultaneous macroscopic and microstructural observation of the SMA in a 4-point flexural test. Forming part of a wider research towards realising a NiTi SMA Variable Stator Vane assembly for the gas turbine engine, the study explores variables critical to flexural-type morphing NiTi structures: (1) temperature; (2) strain; and (3) cyclic loading. It builds a relationship between the macro and micro response of the SMA under these key variables and lends critical implications for the future understanding and modelling of shape memory alloy behaviour for all morphing applications. This paper presents the methodological aspects of this study.

KEYWORDS: Shape memory; NiTi; In situ; Phase transformations; Micro-macro approach; Cyclic loading

INTRODUCTION

Gas turbine performance development has been governed traditionally by the "worst case" deterioration and operating condition [1]. This leads to severe compromises and large safety margins. Active control of the engine operation using smart materials could potentially improve engine efficiency. Shape Memory Alloys (SMAs), a class of smart materials, exhibit several desirable characteristics exploitable for this purpose. NiTi, based on an equiatomic compound of nickel and titanium is the most widely used SMA in commercial applications [2]. Besides the ability of tolerating relatively large amounts of shape memory strain, NiTi shows high stability in cyclic applications, possesses an elevated electrical resistivity, and is corrosion resistant [3].

An exciting possibility is the incorporation of SMA in plate form into blade structures of the Gas Turbine compressor. Airflow control is introduced into compressor designs through the use of Variable Inlet Guide Vanes and a number of stages incorporating Variable Stator Vanes. They operate by progressively closing as the compressor speed is reduced from the original design value to maintain an acceptable air angle value into the following rotor blades. Traditionally, such a system encompasses a complex structure employing control levers that are actuated through an electrical or bleed air system. Switching this system to a NiTi plate based actuation mechanism is intrinsically very attractive due to its high power density, solid-state actuation, high damping capacity, durability and fatigue resistance.

A concept actuator design using NiTi plates to form a solid-state actuator that replicates the behaviour of the VSV system is depicted in Figure 1. In this concept, the activation of each NiTi plate translates to a deflection at the tip of the actuator. To achieve this, the system exploits a flexural type strain application/recovery using the Shape Memory Effect.

PREVIOUS STUDIES

A thorough understanding of the flexural performance of NiTi plate is required for the implementation of such a system. Previous studies have primarily evaluated material behaviour under tension employing mostly uni-axial loads. Of particular interest are studies that employ in-situ loading and microscopy [4-12]: they offer a rich understanding of SMA response that addresses both microstructural and macromechanical response simultaneously. In their mico-macro study, Brinson et al [12] clarified several aspects of the transformation behaviour under loading at different strain rates and fatigue. This included observations of low-cycle localized plastic deformation, correlations between latent heat release and stress relaxation, and a refined definition of "full transformation" for polycrystalline materials.

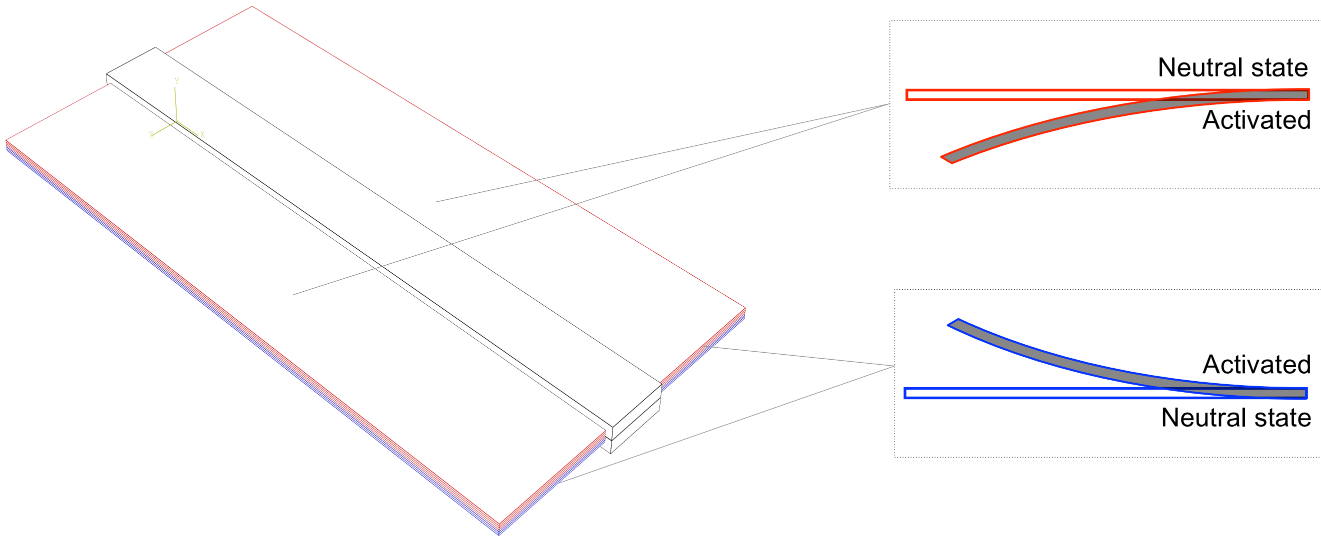

Fig. 1 Concept sketch

However, only a few experimental studies have addressed SMA behaviour under compression or bending, despite the prevalence of these deformation modes in applications. Most studies have restrained from using traditional point-bending fixture tests; instead, they have opted for custom-built pure-bending apparatus. Studies on small diameter NiTi wire naturally restrict point-bending fixtures as a result of small radii of curvature and large displacements - conditions where undesirable axial loads can develop due to the support constraints. The studies conducted by Berg [13] and Bundara et al. [14] have employed moment-controlled experiments to assess the constitutive relationship between applied pure bending moments and the resulting curvatures. These studies, however, suffer a major limitation due to their lack of control over strain rate, leading to a significant impact on the stress/strain response during phase transformations [12]. More recent work by Rejzner et al. [15] and Reedlunn et al. [16] avoid this problem by using displacement-controlled, custom pure bending fixtures integrated into the load frame.

The study by Reedlunn et al. [16] is particularly interesting as they used larger diameter NiTi tube instead of wire, which negates the requirement of a small radius of curvature to incur strain. Additionally, the resulting smaller deformations and larger specimen dimensions allow the use of in-situ imaging techniques in the guise of Digital Image Correlation for strain measurement. Their experimental tests were able to discover a significant asymmetry between the compressive and tensile deformation modes. This resulted in a shift of the neutral axis towards the compressive side to equalise the distribution of tensile and compressive stresses in the cross section. One limitation that was noted, however, was the appearance of the *Brazier effect*; the tendency of thin walled tubes to ovalise in bending due to longitudinal tension and compression [17]. Ovalisation is particularly undesirable if it leads to variant re-orientation along this new deformation path at a granular level as it questions the homogeneity over compressive and tensile responses in their bend tests. This could also result in accelerated development of stress localisations under fatigue, which could lead to premature failure [12]. Another limitation encountered by Reedlunn et al. was through undertaking tests at room temperature without direct measurement of the specimen temperature. This procedure is not ideal because of the high temperature sensitivity of NiTi.

A common theme to most bending studies is the exclusive focus on SMA behaviour in its Superelastic phase. This can be attributed to the myriad of applications that already employ Superelastic SMAs in this deformation mode, making it a more lucrative research area. Moreover, most studies on bending have followed a macro level approach to exploring material behaviour. This has left the investigation of its microstructural behaviour largely unexplored, under this deformation mode.

EXPERIMENTAL TECHNIQUES

Inspired by the above stream of research, the present study explores NiTi behaviour in bending. What distinguishes this research is the analysis of both thermal and stress induced transformation behaviour of NiTi plate. Additionally, in-situ techniques allow simultaneous observation at both macroscopic and microstructural levels. The inclusion of these different aspects, however, presents the study with a new series of hurdles. The methodology employed to conduct this study is detailed below.

Testing stage

Figure 2a illustrates the general experimental set up. The load frame - a tabletop MTS 858 servo-hydraulic test system - uses a cross-head assembly which includes a single moving upper grip, a stationary lower grip and a LVDT position sensor. The system is designed to function in a closed-loop configuration under computer control. The specimen sits within a temperature controlled environmental chamber- a Thermcraft LB-series Box Laboratory oven.

A custom 4-point bend fixture is built and integrated into the uniaxial load frame to perform the flexural tests, as depicted in Figure 2b. The external and internal supports are inverted to limit the deflection of the optically investigated central section of the specimen. The fixture is designed with adequate spatial clearances in mind, allowing upper support deflection of up to 12.5mm (equivalent to a 4% strain at the outer fibre with our specimens).

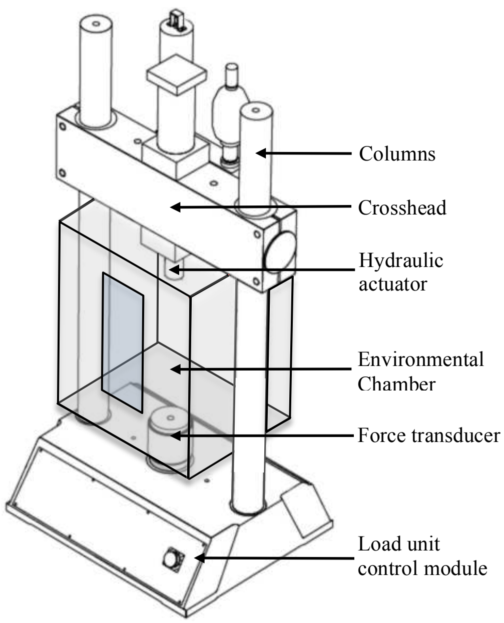

Fig. 2(a) Testing stage

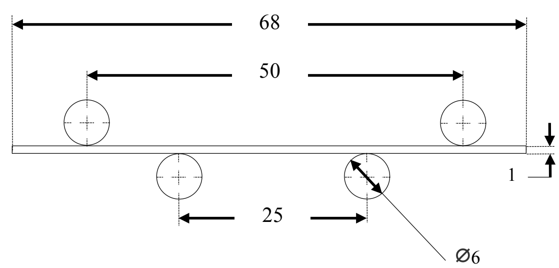

Fig. 2(b) Custom 4-point bend fixture

Optical microscopy

A digital camera is used to observe changes in the surface transformation characteristics at a macroscopic and fine-scale microstructural level as a function of loading parameters. All images are taken during static holds of constant temperature and strain. Figure 3 depicts the unique twin surface preparation employed to conduct this in-situ observation.

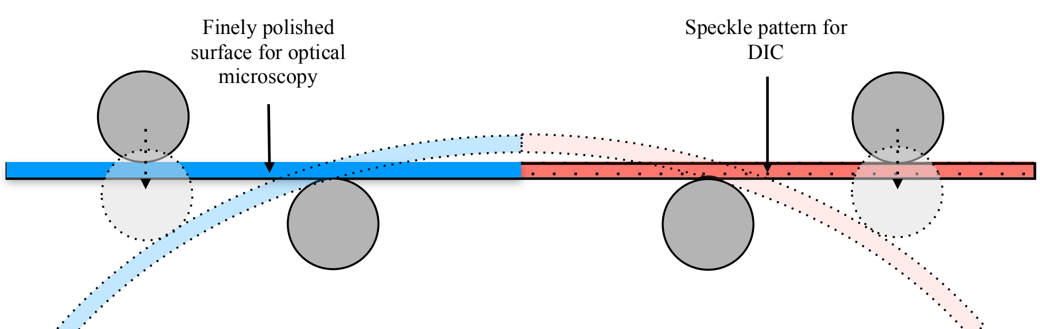

Fig. 3 Preparation of observational surface

Digital Image Correlation (DIC) is employed to obtain local strain measurement without the need to rely solely on grip displacements. The specimen is prepared by applying a random speckle pattern to its surface. DIC starts with a reference image and followed by subsequent images recorded during the deformation process. A strain distribution map is created by calculating the correlation between the reference and deformed speckle patterns. In contrast, the microstructural changes are observed from the finely polished surface on the left hand side of the specimen. A polarized light interference filter is used for higher detection sensitivity of fine topological details. A maximum zoom which focuses on a surface area approximately $1mm^2$ is required to investigate observation of the material at a variant level. This allows the simultaneous viewing of both compressive and tensile deformation modes.

SPECIMEN PREPARATION

Machining - Near-equiatomic 1mm thick NiTi plate was obtained from Memry Metalle GmbH with manufacturer specified Austenite finish (A_f) temperature of 65°C. The plate is machined to dimensions of 66mm x 8mm using a wire cut electrical discharge machining process. This minimizes heat distortion and cold work induced on the specimen. As the tests optically investigate the specimen surface and relate it to the material behaviour as a whole, the homogeneity over their behaviours is important.

Annealing - The specimens are heat treated in an oven at 450°C for 1 hour followed by water quench. This annealing condition ensures a large grain size - the amorphous bands start to crystallize without completely annealing the structural defects in the material, as crystallite nucleation is supressed [18]. This step is essential for the material to recover from any previous cold work or damage it has sustained and to stabilize transformation characteristics [18].

Thermal cycling - Additionally, the specimens are thermally cycled through martensite and austenite phases using a water bath. Miyazaki et al. [6] indicate that M_f and M_s temperatures decrease with increasing number of thermal cycles. The decrease is rapid at first but more gradual with increasing cycles as values stabilize. Their TEM observations reveal the introduction of dislocations from the first thermal cycle onwards, which act as obstacles to dislocation movement and stabilizes behaviour through further thermal cycles. However, their thermal cycling had also shown to activate R-Phase transformation in NiTi SMAs that had a previously suppressed R-Phase. In our study, the specimens are subjected to 30 thermal cycles despite possible R-Phase activation, as it is paramount to obtain stable and repeatable transformation characteristics throughout.

Micropreparation – The thermally prepared specimens are then subjected to several steps of grinding and polishing using the Bueler Automet 250, to allow observation of variant microstructure at the specimen surface. As the surface was initially rough, all sides of the specimen are grinded using P800 and P1200 abrasive discs. The observational side of the specimen is then sequentially polished using 3µm MetaDi Supreme Diamond abrasive for coarse polishing and then finally using 0.02 – 0.06µm MasterMet Silica for fine polishing.

Specimen characterisation

Differential Scanning Calorimetry (DSC) is used to identify phase transformation temperatures of the NiTi plates. This is a thermoanalytical method commonly used in NiTi SMA studies that determines absolute phase transformation temperatures. It measures the difference between the amount of heat required to increase the temperature of a sample and reference, as a function of temperature. Our DSC tests adopt a temperature profile of 15°C to +100°C with a constant scanning rate of 10 K/min. This is considered the most efficient temperature rate to measure the intrinsic transition quantities and also a common value within published literature. The characterisation of the transformation temperatures is essential in establishing temperature ranges for the experimental study.

DSC scans of the NiTi plate specimens in the following conditions are presented: as received (grey), annealed (red), and thermally cycled (blue). Figures 4a and 4b depict thermograms of each of these states under heating (M → A) and cooling (A → M), respectively. The transformation temperatures for each of the specimen states are recorded in Table 1.

Table 1: Phase transformation temperatures

Specimen	M_s	M_f	A_s	A_f
As received	47.8C^o	30.7C^o	28.5C^o	50.9C^o
Annealed	34.9C^o	30.6C^o	33.1C^o	37.4C^o
Thermally cycled	35.6C^o	31.3C^o	33.5C^o	38.2C^o

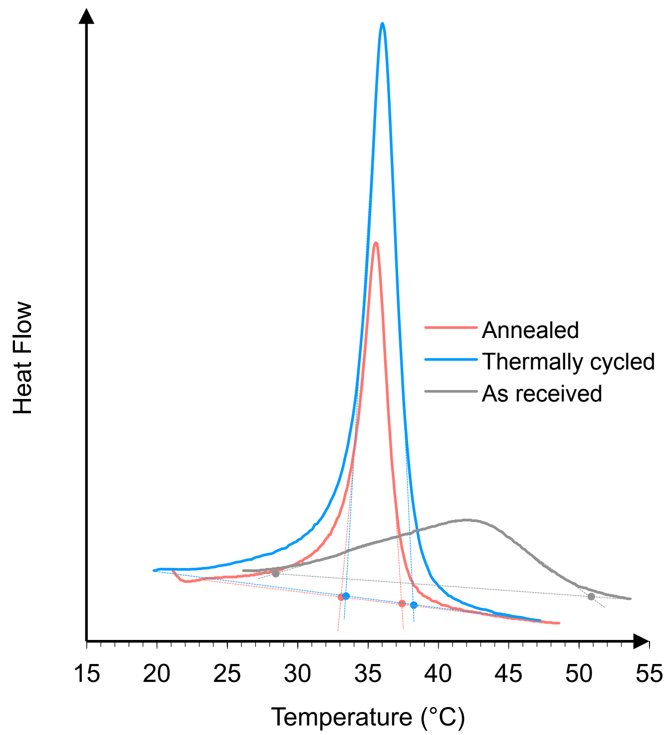

Fig. 4a DSC Thermogram for heating (M → A)

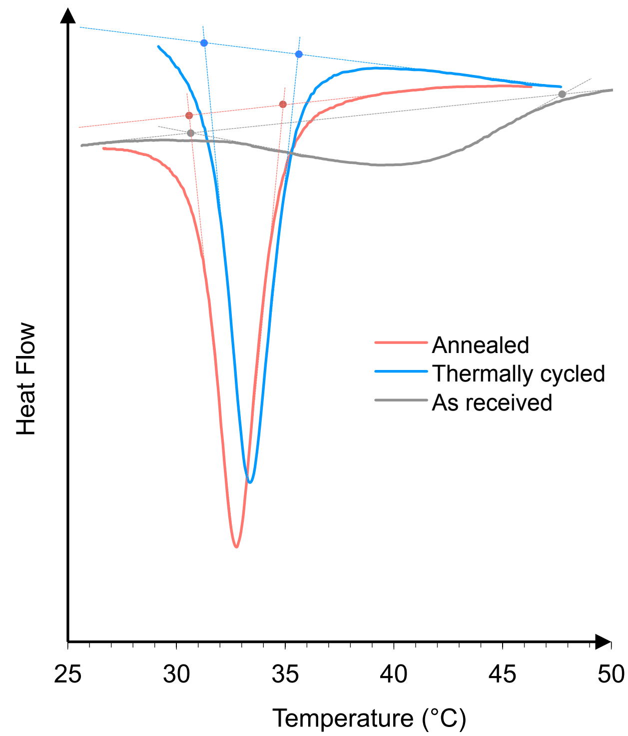

Fig. 4b DSC Thermogram for cooling (A → M)

TEST PLAN

The experimental test study can be subcategorized into two sections: (1) constant temperature tests with load increments, and (2) constant load tests with temperature increments. The tests employ a temperature range between A_f- 25C^o → A_f + 25C^o and a peak flexural strain of 2%. Using these load parameters, the study performs a comprehensive evaluation of the material behaviour within the operating envelope of the planned NiTi SMA actuator.

The extremities of these parameters are established due to the requirement of the maximum strain falling within the de-twinning phase at the lowest temperature, A_f- 25C^o. However, it must be noted that due to the non-linear response of the material, localised strains could be considerably more, especially during the de-twinning phase where large heterogeneities can exist between grain boundaries. An adequate safety margin is thus kept from the end of the strain plateau observed from previous data from tensile tests on this material: ~4% at room temperature. This strain magnitude range ensures that thermo-mechanical cycling effects are minimised and life span of each specimen is improved. Furthermore, the lower strains reflect the behaviour of a high cycle element such as the variable stator vane where uniformity of performance must be established throughout the life cycle.

Three NiTi plate specimens are used in this study. The thermal and fatigue history of each of the specimens is recorded and carefully balanced to enhance comparability between results. In each test cycle, the loading is temporarily paused at planned intervals and the specimen is held at constant displacement, while photographs are taken of the observational surface.

Constant temperature tests with load increments attempt to replicate the deflection behaviour of the NiTi plate actuator concept over a range of static temperatures. Table 2 highlights the parameters used in this study. The tests are sequenced so that strain is always recovered after a cycle that is loaded from the martensitic phase: the subsequent cycle runs above A_f and activates the SMA. Loading sequences for each of the initial 36 tests are repeated in reverse order to account for the temperature history sensitivity of the material. The strain is regulated by a load input to allow strain relaxation in non-isothermal type loading.

Table 2: Constant Temperature Tests				
Strain	Strain rate	Temperature	Optical Imaging	Total tests
2%	1 x $10^{-4}s^{-1}$ → 1 x $10^{-2}s^{-1}$ (3 steps)	A_f - 25C° → A_f + 25C° (12 steps + 12 steps reverse)	Static strain holds 0.25% → 2% → 0% (16 steps)	72

Constant load tests with temperature increments attempt to replicate the actuation behaviour of the NiTi plate actuator concept over a range of static loads. Strain recovery characteristics of the NiTi plate specimens at different load magnitudes are analysed to accomplish this. Table 3 highlights the parameters used in this study, which allows the evaluation of macroscopically observable material characteristics and microstructural changes over the stated temperature range. The strain is regulated by a load input to allow strain recovery. The specimen is heated past its Austenite finish (A_f) temperature prior to each subsequent test to ensure that strains are not carried over between tests. This is executed at zero strain, using an identical temperature ramp rate.

Table 3: Constant Load Tests				
Load	Temperature	Temperature ramp rate	Optical Imaging	Total tests
0.1% → 2% (at A_f - 25C°/ 20 steps)	A_f - 25C° → A_f + 25C° → A_f - 25C°	1 x 10^{-1}C°S^{-1}	Static temperature holds (11 steps/every 5C°)	60

PREDICTIONS AND LIMITATIONS

In this section, we evaluate the possible outcomes of the experimental tests. While the broader aim of the study is to evaluate material behaviour under the possible operating envelope of the NiTi plate actuator, this study also assesses several NiTi SMA behavioural aspects that may have academically significant implications, as discussed below.

Asymmetry between tension and compression - The bend tests allow the simultaneous evaluation of the material behaviour under both tensile and compressive deformation modes. While classical Euler–Bernoulli bending kinematics assumes a linear strain profile across the cross section, the heterogeneous nature of the bending strain fields in NiTi result in a much more complex strain profile. In their bend study, Reedlunn et al [16] report a significant shift of the neutral axis under bending. However, the appearance of the Brazier effect, due to their choice in specimen geometry, could have affected this to a certain extent. In contrast, the use of plate specimens in our study negates such effects. Moreover, this study potentially allows a richer understanding of the two deformation modes as we use broader testing conditions and follow a micro-macro approach.

Relationships between macroscopic behaviour and microstructural observations - This study combines macroscopic and microstructural analysis via DIC and optical microscopy. Most previous studies have either focused on a micro or macro level approach: micromechanical studies are often theoretical and not directly applicable to end applications whereas macro level studies do not contain enough depth to explain the observed phenomena. The employment of both approaches allows relationships to be constructed, explaining material behaviour in greater detail.

A complete map of transformation behaviour – Phase transformations, temperatures and applied stresses are closely intertwined where one affects the other. As this study varies both temperature and load in its tests, a complete phase diagram can be developed, showing the effect of applied flexural deflection on the phase transformation temperatures for the tested specimens.

Effects of loading rates – The constant temperature tests repeat every loading sequence at three loading rates: 1 x $10^{-4}s^{-1}$, 1 x $10^{-3}s^{-1}$ and 1 x $10^{-2}s^{-1}$. Uniaxial studies by Brinson et al. [12] report on slower strain rates following an isothermal behaviour where martensite initiates and grows from a single band, while at higher strain rates, the formation of multiple bands is present. To our knowledge, however, the implications on loading rate for bend tests using full-field, in-situ methods such as DIC are largely unexplored.

Localised phase transformations – Under tension, NiTi is known to exhibit strain localization and propagation during portions of stress induced transformations [19]. Under flexural loading, however, the development of strain localisations can be non-linear due to the presence of compressive and tensile deformation modes in varying magnitudes through the specimen thickness. In their study of Superelastic NiTi tube, Reedlunn et al. highlight this effect, reporting on strain localisations in tension and on the absence of strain localisations in compression [16]. Our study will further evaluate this behaviour, linking the evolution of macroscopic transformation bands to the microstructural changes as a function of both applied load and temperature.

Due the testing techniques employed, our study contains several limitations. These limitations and the extent to which they can affect the outcomes are discussed below.

Bend fixture – The use of 4-point bend fixtures can be somewhat problematic in NiTi studies due to the creation of undesired axial loads at the support constraints. These stress concentrations, if large enough, can lead to the premature transformation to martensite near the constraints, and result in inaccurate displacement measurements. While these issues exist in our study, they are outweighed as a result of the easy integration of the 4-point bend fixture in the loading frame and the subsequent flexibility offered in tests. Furthermore, we try to negate drawbacks associated with point-bending fixtures by employing several measures: (1) a flat contact surface between specimen and constraints to reduce stress concentrations, (2) relatively small peak deformations in tests, (3) 4-point (as opposed to a 3-point) bend fixture as they have a larger region of deflection, and (4) contact surfaces polished to reduce shear.

In-situ material characterisation – This study uses DIC and optical microscopy to capture phase transformation behaviour at a kinematic level. Another characteristic of phase transformation behaviour that can be captured is its thermal signature. This can be accomplished by using an Infrared camera, which can detect the instantaneous local heating of the domains due to the released latent heat during phase transformation. The synchronization of both kinematic and thermal measurements would lead to a highly attractive complete thermo-mechanical characterisation of phase transformation behaviour. However, the integration of such a technique alongside DIC and optical microscopy can be logistically very difficult (antagonist surface texture requirements, imaging devices having different pixel and acquisition rates [20]). A novel technique developed by Maynadier et al. [20] - Infra-Red Image Correlation - synchronizes kinematic and thermal measurements and thus negates many of these drawbacks. This will be explored in future studies.

Fatigue – Previous studies have reported on a continuous change in the functional properties of the NiTi such as the transformation temperatures, transformation hysteresis and strain response under thermo-mechanical cycling [18]. Our experimental work comprises 132 tests, which effectively subject the 3 test specimens to thermo-mechanical cycling throughout the study; hence, the stability of the material response is a concern. Several measures have been taken to dramatically reduce such effects, including careful thermal preparation of the specimens, use of low peak deformation magnitudes and careful workload management between the specimens in the experimental tests. The option to completely ignore thermo-mechanical cycling effects - employing a vast number of specimens - was not explored as it does not truly reflect the required characteristics of the NiTi plate actuator, a high cycle element with uniformity of performance required throughout its life cycle.

Temperature range – All the tests are conducted within a temperature range of A_f - 25C^o → A_f + 25C^o. This range is restricted due to the lack of active cooling in the environmental chamber, which prevents tests being undertaken below ambient temperature. The temperature range should be sufficient to capture forward and reverse transformation at most load levels. Nevertheless, the integration of active cooling in future studies is a vital step, as it would allow recreation of the extreme temperatures that would be experienced by the NiTi plate actuator.

CLOSING REMARKS

A review of previous studies has demonstrated a gap in the knowledge concerning the thermo-mechanical behaviour of NiTi, especially in the bending deformation mode. In this paper, we have presented the methodological aspects of a novel experimental investigation that addresses these issues. Our attempt is to evaluate thermal and stress induced transformation behaviour of NiTi plate for flexural-type applications. We have employed in-situ techniques, which allow simultaneous observation of both macroscopic and microstructural material behaviour in 4-point bend flexure tests. A special emphasis is placed on material preparation not only to attain the desired surface characteristics for in-situ observation, but also to stabilize the thermo-mechanical response of the material for accurate tests. The experimental study is designed to assess several key characteristics of NiTi SMA behaviour. The tests were broadly split into two categories: (1) constant temperature tests to evaluate deflection characteristics, and (2) constant load tests to evaluate strain recovery and relaxation characteristics. The speculated outcomes and their implications were hypothesised. In particular, the following aspects may have academically significant implications: (1) Asymmetry between tension and compression, (2) Relationships between macroscopic behaviour and microstructural observations, (3) A complete map of transformation behaviour, (4) Effects of loading rates, and (5) Localised phase transformations. We were also able to identify several limitations in our study. They include limitations related to the experimental techniques used and to the range of the test study, and limitations universally inherent to NiTi testing. Future work will attempt at addressing these limitations.

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