Material Characterization of Shape Memory Alloys for Morphing Hybrid Composites

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ABSTRACT

Shape memory alloy hybrid composites have promise in realizing the 21st century goal of morphing structures. There is considerable work to be done in the development the characterization and modelling techniques for these materials. The proposed characterization methodology adapts existing standards to include previously omitted factors required for the numerical modelling of shape memory alloys and their integration into end-use applications. A nickel-titanium-copper (NiTiCu) shape memory alloy is characterized using these methods and then numerically modelled. Samples' mechanical behaviour is shown to stabilize after 43 cycles of mechanical loading. Thermomechanical properties measured before and after stabilization are shown to vary inconsistently by up to 72%, demonstrating the need for stabilization for accurate thermomechanical characterizations and consistency in end-use applications. Physical experiments are numerically replicated in Abaqus\Standard using the measured properties. Sufficient correlation is shown for the design of shape memory alloy hybrid composites. The result of this work is a comprehensive thermomechanical characterization approach for shape memory alloys which can be used to develop morphing SMA hybrid composite structures.

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KEYWORDS: *Smart Composites, Hybrid Composites, Shape Memory Alloys, Morphing Structures*

1 INTRODUCTION

An advantage of composite materials, when compared to metals, is the relative ease with which *net-shape* structures are manufactured, thereby reducing assembly complexity. A factor limiting part consolidation is actuation, where structures (i.e. wing flaps) must move to perform their function. While components may be made from composites, assemblies with fasteners and traditional mechanical actuators (i.e. hydraulic motors) are typical for these applications. A review morphing aircraft wing concepts (Li et al., 2018) identifies that shape memory alloy hybrid composites can enable *net-shape* and *net-function* structures because they can be integrated into existing composite structures. Shape memory alloy hybrid composite structures can bring actuation capability without the additional complexity of traditional mechanical systems. This is especially useful for applications such as helicopter rotor blades or sailing hydrofoils where volume and mass limitations preclude the use of assemblies.

A major limit to the application of SMAs is the ability to characterize their unique micromechanics (Hartl et al., 2015). In this research a characterization process for binary and ternary nickel-titanium shape memory alloys is proposed and executed. This process is compatible with well-developed thermomechanical models, testing standards, and numerical simulation tools.

2 SHAPE MEMORY ALLOY MICRO- AND MACRO- MECHANICS

Shape memory alloys undergo a solid-state, reversible, diffusion-less, phase change when thermally or mechanically loaded. Two main phases are observed in nickel-titanium based shape memory alloys, martensite and austenite. Martensite is stable at low temperature and stress, as well as at high temperature and stress. Austenite is stable at high-temperature and low-stress as well as low-temperature and high-stress. An unstable, transient, R-Phase is also possible (Otsuka and Ren, 2005).

For actuation application applications, the resulting mechanical behaviour of interest is *superelasticity*, which occurs above the temperature at which unstressed austenite is stable during heating – the *austenite finish temperature* (A_f). Superelasticity describes the non-linear hysteretic behaviour of SMAs during mechanical loading, an example of which is shown in Figure 2.1. There are a number of phenomenological models for superelasticity – see (Terriault et al., 2006), (Brinson and Lammering, 1993), (Boyd and Lagoudas, 1996), and (Lagoudas, 2008). The Auricchio and Taylor model (Auricchio and Taylor, 1997) is widely implemented in commercial analysis packages such as Abaqus (Dassault Systemes, 2018) and MSC Marc (MSC Software, 2017), and thus followed for purposes of property evaluation.

Note that the loading and unloading plateau stresses are thermally dependent. When SMAs are strained within the plateau region and their temperature varied above the A_f they will exert a tensile stress. It is this thermal dependency that enables the use of superelastic SMAs in actuation applications.

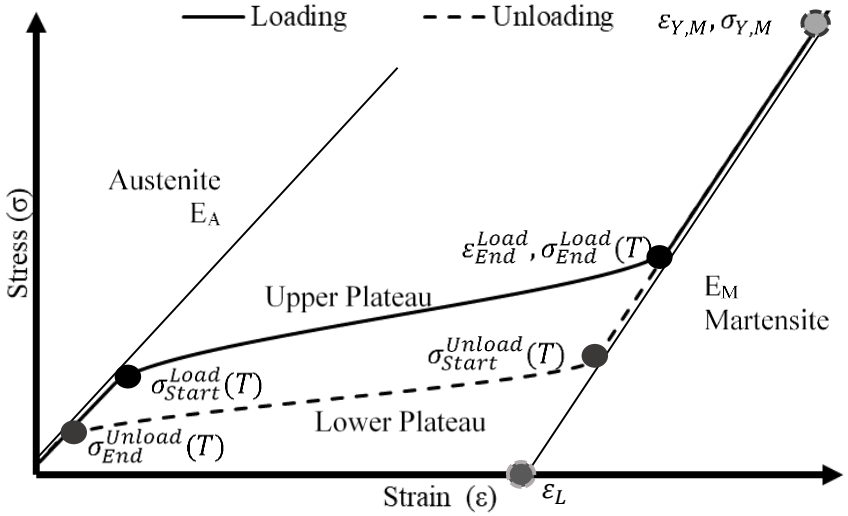

Figure 2.1 Example Superelastic Curve

3 CHARACTERIZATION METHODS & FUNCTIONAL STABILIZATION

Thermomechanical characterization of SMAs for actuation applications is an active field of research with many problems to solve, as discussed by (Hartl et al., 2015). The overarching challenge is the large number of mechanical properties with significant thermal dependencies which must be characterized for superelasticity, regardless of the modelling approach. A common approach is a two-step method based on ASTM F2004-17 (ASTM International, 2017a) and ASTM F2516-18 (ASTM International, 2018), as performed by (Mabe et al., 2004). This method first measures phase transformation temperatures using differential scanning calorimetry, followed by measurement of isothermal mechanical properties using a tensile test. The two-step method omits two important facets of SMA characterization. Firstly, it does not characterize the thermal dependencies properties necessary for actuation applications. Secondly, it does not characterize the well-documented phenomenon of functional stabilization. ASTM recently addressed the thermomechanical response of SMAs in the E3098-17 (ASTM International, 2017b) and E3097-17 (ASTM International, 2017c). Neither of these methods directly measure responses relevant to characterization for the Auricchio and Taylor model and do not provide any insight into functional stabilization.

Functional stabilization[1] refers to the phenomenon that low-cycle mechanical loading below the yield stress – typically less than 100 cycles (Hebda and White, 1995) – stabilizes the thermal and mechanical behaviours (Bo and Lagoudas, 1999). A schematic of this behaviour adapted from experimental work by (Zhang et al., 2016) and an example stress-stain curve is shown Figure 3.1. During the first cycles, there are significant evolutions of all thermomechanical properties. Once stabilized properties may change at a reduced rate due to traditional fatigue (Kang and Song, 2015). The effect of cyclic loading on shape memory alloys is particularly challenging to measure due to the number of thermomechanical properties and variance in their stabilization rates (Auricchio et al., 2003).

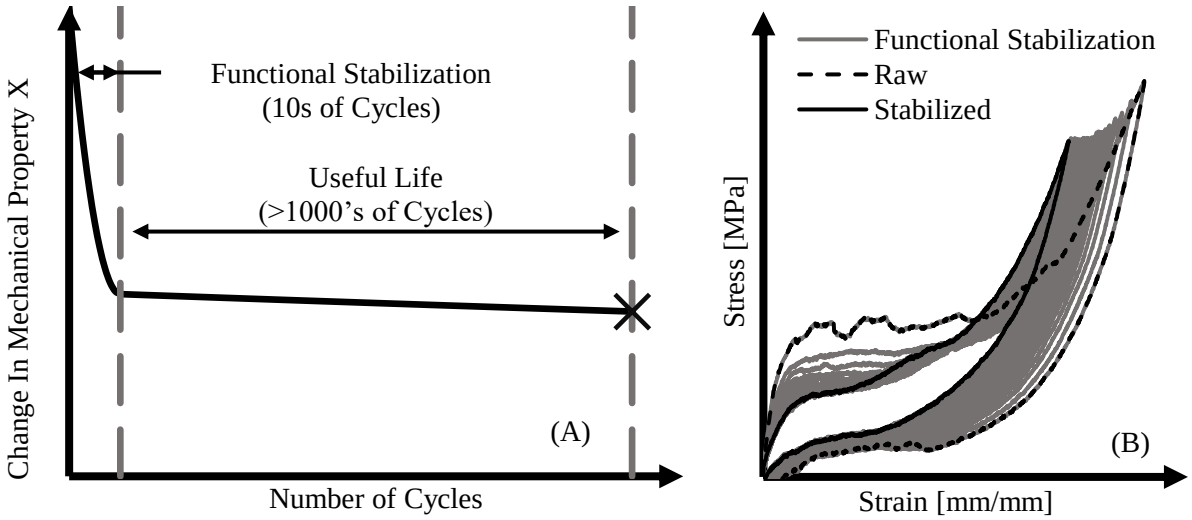

Figure 3.1 Schematic of Functional Stabilization adapted from (Zhang et al., 2016) (A) and Example Functional Stabilization Stress Strain Curve (B)

[1] The literature also refers to "functional stabilization" as "functional fatigue" and "training." Training has another meaning in the context of the Two Way Shape Memory Effect (Hebda and White, 1995) and fatigue is often used in engineering to refer to high-cycle effects. The word "stabilization" is used in this work to prevent confusion with the other phenomena to which fatigue and training refer.

The use of hysteresis energy of the transformation cycle (i.e. the area between the loading and unloading curves) was proposed by (Moumni et al., 2005) and extensively developed by (Morin, 2011). This method allows for the stabilization of all mechanical properties to be captured in a single parameter without introducing error from an intermediate measurement step (i.e. modulus measurement). Furthermore, it is model-agnostic as it is also easily evaluated using true-stress-strain data directly measured in many mechanical tests. Given the large number of phenomenological models available to characterize superelasticity, this is a significant advantage as it allows stabilization results to be easily compared.

Proposed is a six-step method – shown in Figure 3.2 – which includes thermomechanical and functional stabilization characterizations using methods from ASTM F2004-17 and ASTM F2516-18. First, the A_f^{Raw} is measured in step (1) on raw wire to inform the first isothermal yield test in step (2). From the isothermal yield test, the yield strain and end of loading transformation strains are measured. Step (3) characterizes functional stabilization by cyclically loading to a strain between the end-of-loading-transformation and yield strains. The hysteresis energy approach is used to evaluate functional stabilization. ASTM F2004-17 and ASTM F2516-18 are repeated on stabilized samples in steps (4) and (5) to characterize the stable isothermal mechanical behaviour. Lastly in step (6), tests which follow the cyclic portion of ASTM F2004-17 are performed at multiple isotherms to characterize the thermomechanical response. Tests are performed at multiple isotherms rather than using temperature ramps to relieve concern of thermal gradients (ASTM International, 2017c) and ensure constant strain rates, known to have a strong influence on SMA behaviour (Morin, 2011).

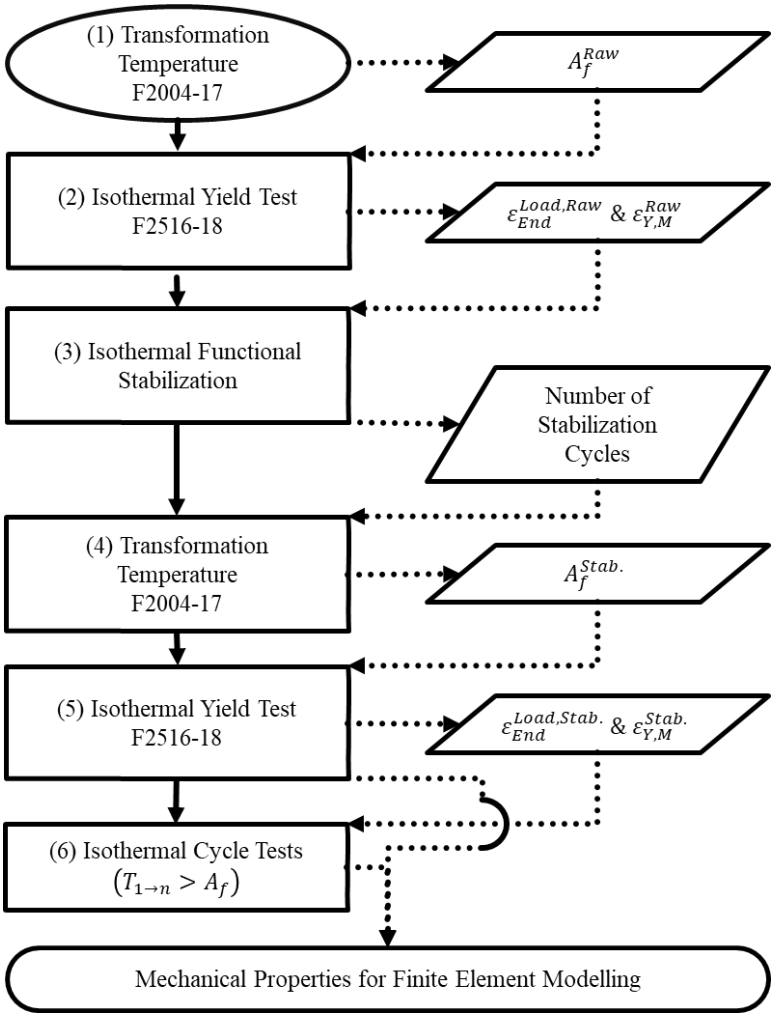

Figure 3.2 Proposed SMA Characterization Process

4 EXPERIMENTAL WORK

4.1 Methods

Samples tested are a NiTiCu wire from Furukawa Electric of 150 [μm] diameter which have been straight annealed and are composed of 44.86 [wt%] Ni and 10.06 [wt%] Cu. The proposed characterization methodology is carried out using a Walter+Bai AG LFM electromechanical testing machine inside a Noske-Kaeser environmental chamber and a TA Q200 DSC. All tensile samples are strained at 0.30 [mm/s] with a minimum length of 225 [mm] under 0.01 [N] of load at the test temperature.

4.2 Results and Discussion

To begin, steps (1) and (2) are carried out to characterize raw wire and inform the functional stabilization experimental methods, the results of which are shown in Table 1. The measured end-of-loading-transformation strain is 40% larger than the ASTM specified 60 [με] for cyclic tests (ASTM International, 2018). Ensuring complete transformation of the sample is important for measuring functional stabilization (Morin, 2011). Therefore, it is important to perform these initial tensile yield tests at a suitable temperature to ensure that the sample is completely transformed during each stabilization cycle.

Table 1: Experimental Results from Initial Characterization of Raw Samples

Austenite Finish Temperature (A_f^{Raw}) [°C]	65.6 (±0.706)
End-of-Loading-Transformation Strain at 80 [°C] ($\varepsilon_{L,E}^{T}$) [με]	86.9 (±0.828)
Yield Strain at 80 [°C] (ε_{Y}^{M}) [με]	121 (±1.92)

Functional stabilization is evaluated by cyclically loading samples between $90 < \varepsilon_{max} < 116$ [με] at 80 [°C]. The exponential form used to evaluate hysteresis energy by (Moumni et al., 2005) is adapted to evaluate as a decay function, which is shown in Figure 4.1. The use of a decay allows for a general criterion which defines a *"stable"* sample. In this case, the criterion is that the change in hysteresis energy reduces by two orders of magnitude which occurs by the 43rd cycle $(\Delta HE_{43,42}/\Delta HE_{2,1} < 10^{-2})$ for stabilization.

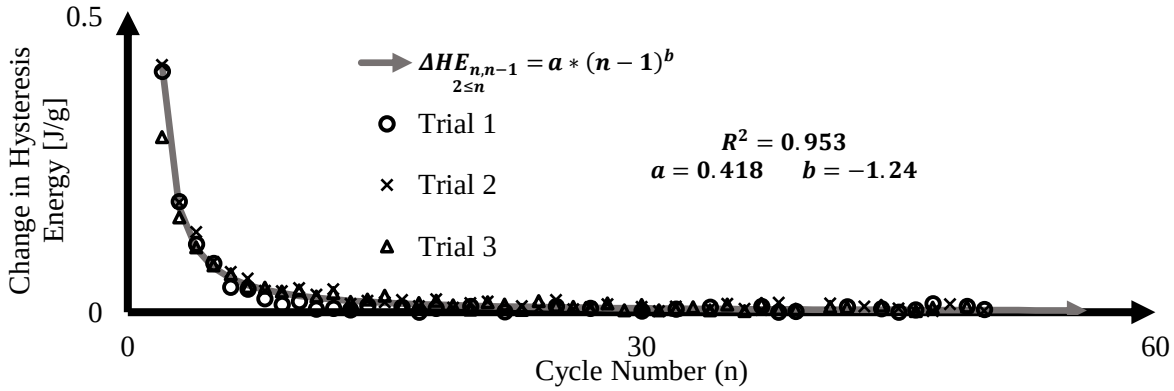

Figure 4.1 Change in Hysteresis Energy per Cycle to Evaluate Functional Stabilization

DSC and tensile yield tests are repeated on stable samples, which are shown with raw sample results in Figure 4.2. Comparisons between raw and stabilized samples' thermomechanical properties are shown in Figure 4.3. There are significant changes to both thermal and mechanical behaviour attributable to the functional stabilization, demonstrating the need for functional stabilization prior to mechanical property measurement or the application of SMAs in end-use applications.

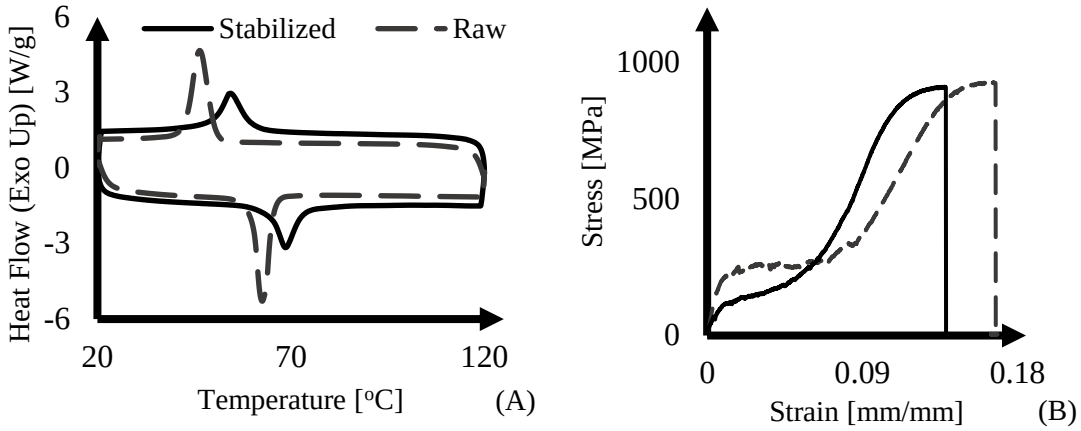

Figure 4.2 Thermal (A) & Mechanical (B) Results for Raw and Stabilized Samples

The A_f is measured to be 12% greater in stable samples at 73.5 [°C]. It is important to consider this when selecting a temperature to measure the raw wire yield behaviour and perform functional stabilization. If the A_f increases beyond the test temperature during stabilization, the results are no longer coherent and the characterization must be restarted at step (2). ASTM recommends testing samples 5 [°C] above their A_f (ASTM International, 2018), which is not enough in this case. It is difficult to predict the A_f change *a priori*, so a more conservative temperature is used, such as the maximum end-use service temperature.

The thermal and mechanical energies required for transformation decrease during stabilization, indicating a reduced amount of austenite available for transformation in the stabilized samples. This is expected as stabilization changes phase distribution within the samples (Lagoudas et al., 2002) which is a root cause of the mechanical property changes.

There are two indications that it is not a simple phase volume fraction redistribution that is occurring because of stabilization. First, there is the inconsistency between the softening and stiffening of the austenite and martensite moduli. Second is that some properties change by more than 70%, while others change less than 15%. This suggests that dislocations and other defects introduced in this process will affect the properties of the stable sample. Given the large variety of factors influencing formation of these defects, it is important to evaluate samples which are representative of both end-use application geometries and chemistries.

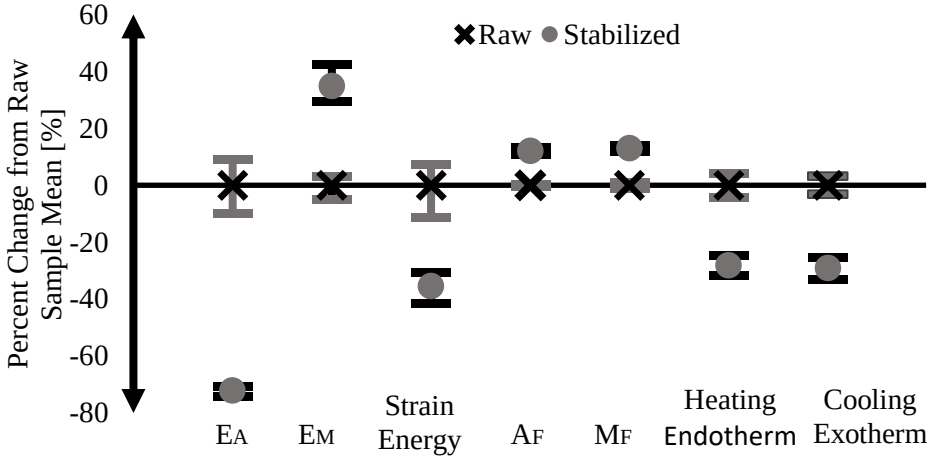

Figure 4.3 Comparison of Thermomechanical Properties for Raw and Stabilized Samples

The final steps, performing yield (5) and cyclic loading (6) tests at multiple isotherms are then performed on stabilized samples. From these results, the mechanical properties for the Auricchio and Taylor model are measured, shown in Table 2.

Table 2: Experimentally Measured Constants for Auricchio and Taylor Model

Austenite Young's Modulus (E_A) [GPa]	10.1	Loading Start of Transformation Stress (σ_S^L) [MPa]	92.8
Martensite Young's Modulus (E_M) [GPa]	15.6	Loading End of Transformation Stress (σ_E^L) [MPa]	240.
Loading Stress-Temperature Slope $\left(\frac{\delta\sigma}{\delta T}\right)_{Load}$ $[\frac{MPa}{^oC}]$	10.8	Unloading Start of Transformation Stress (σ_S^U) [MPa]	120.
Unloading Stress-Temperature Slope $\left(\frac{\delta\sigma}{\delta T}\right)_{Unload}$ $[\frac{MPa}{^oC}]$	6.10	Unloading End of Transformation Stress (σ_E^U) [MPa]	32.7
Reference Temperature (T_0) [°C]	80	Projected Martensite Strain (ε_L) [με]	53.5

4.3 Finite Element Result Comparison

The isothermal cyclic experimental tests are replicated numerically in Abaqus\Standard using the Auricchio and Taylor model for superelasticity and the experimentally measured constants in Table 2. A notable variance between the FE model and experimental data is that martensitic and austenitic regions are not observed to be perfectly linear. The Auricchio and Taylor model assumes complete transformation from austenite to martensite, which is known not to be the case with polycrystalline samples (Brinson et al., 2004), explaining this variance. For the purposes of designing SMA hybrid composites this model adequately captures the behaviour, however end-use applications must be calibrated with experimental data.

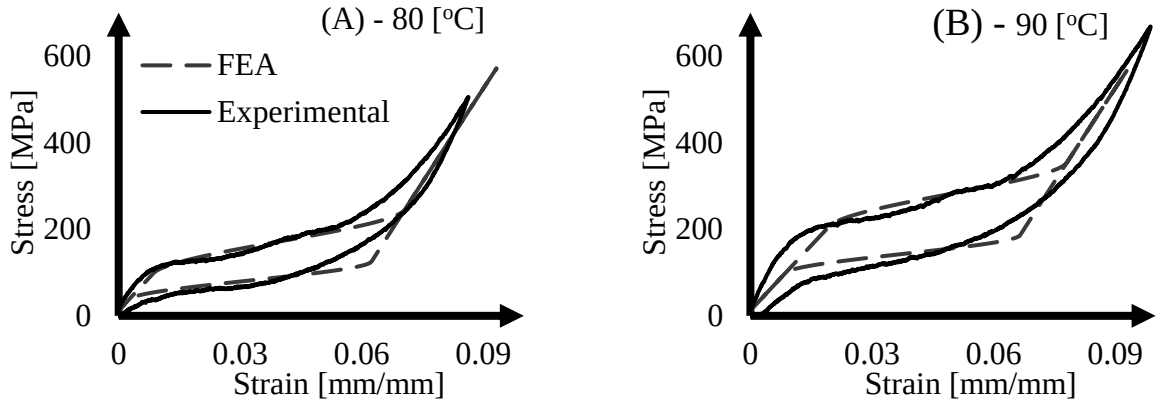

Figure 4.4 Comparison of Finite Element and Experimental Results at 80 [°C] (A) and 90 [°C] (B)

5 CONCLUSIONS

An experimental process for characterization of shape memory alloys is proposed and executed. First, the need for two initial characterization steps is demonstrated as the ASTM standards may under-strain samples and occur at too low a temperature the purposes of functional stabilization. Mechanical properties are then measured to vary by more than 70% due to functional stabilization, demonstrating the need for characterizing this phenomenon. Good agreement between finite element and experimental results are observed, given the assumptions of the model. In adapting ASTM standards, this experimental characterization leverages existing experimental techniques to provide a complete data set for the thermomechanical modelling of shape memory alloys. The ability to characterize the low-cycle stabilization behaviour and non-isothermal response of SMAs is key step in the design and manufacturing of smart SMA hybrid composites.

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