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Tensile actuation response of additively manufactured nickel-titanium shape memory alloys

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A B S T R A C T

In the present work, we characterize the tensile shape memory actuation behavior of NiTi shape memory alloys (SMAs) fabricated using laser powder bed fusion (L-PBF) additive manufacturing process. The samples were fabricated using two different sets of processing parameters. While reversible tensile shape memory behavior was observed in both cases, the samples fabricated with a shorter hatch spacing exhibited higher transformation temperatures, lower actuation strain, and lower irrecoverable strain compared to the samples fabricated with wider hatch spacing. The actuation strain and ductility of the L-PBF samples were lower than that of the conventionally manufactured NiTi SMA samples.

Nickel-Titanium (NiTi) is a class of shape memory alloys (SMAs) that exhibits unique properties such as the shape-memory effect (SME) and superelasticity (SE) [1,2]. Due to these properties, NiTi is a suitable candidate for many critical applications; including, but not limited to those in aerospace and biomedical industry [3,4]. The application potential of NiTi is somewhat limited by the challenges and cost of manufacturing it using conventional methods such as casting, machining, or powder metallurgy, especially when the complex shaped parts are needed. This is attributed to many factors, including high reactivity, high strength, and poor formability [5–10]. To date, the majority of manufactured NiTi parts have been limited to simple geometries such as wires, tubes, and sheets [5]. Additive Manufacturing (AM) techniques can help address these challenges and simplify the fabrication of NiTi parts with complex geometries, that cannot be easily or cost-effectively produced using conventional methods, such as porous scaffolds for biomedical applications [3]. In addition to providing a viable alternative for the production of complex geometries, AM offers the unique capability of tailoring properties in different locations within the same part [11,12]. For example, since shape memory properties are dependent on transformation temperatures, by controlling the AM processing parameters, a part can be built with different transformation temperatures in different locations of the part, eventually leading to location specific properties [11].

There is a substantial amount of studies in literature on the fabrication of NiTi SMAs using AM technologies, with emphasis on laser powder bed fusion (L-PBF) processes [5,7,9,11–17]. Many aspects of AM fabricated NiTi have been investigated, including mechanical properties [7], functional properties [12], and microstructure [15,16], among others. In terms of mechanical properties, the majority of prior studies have focused on studying compressive shape memory actuation or superelastic responses [12,13,16,17]. In contrast, tensile actuation of AM-fabricated NiTi has not been adequately addressed [17]. Characterizing tensile actuation is important since many applications of NiTi shape memory actuators operate under at least partial tension (such as bending) and the remnant porosity is one of the defects that may persist in AM parts and negatively affect the tensile properties, which may not have a notable effect on the properties under compression. The present study is among the first attempts in characterizing tensile actuation response of nickel-rich NiTi SMAs produced using L-PBF AM, commercially known as selective laser melting (SLM). In addition, we demonstrate how shape memory characteristics such as transformation temperatures and actuation strain can be controlled through varying AM process parameters.

Conventionally manufactured $Ni_{50.9}Ti_{49.1}$ (at.%) was obtained from SAES-Getters as a 1.5″ diameter rod in hot-rolled condition. Some of this NiTi rod was used to fabricate tension and Differential Scanning Calorimetry (DSC) test samples to compare shape memory properties between the AM and the conventionally manufactured samples. This Ni-rich NiTi composition was selected since Ni-rich side of the stoichiometry allows the control of transformation temperatures through the

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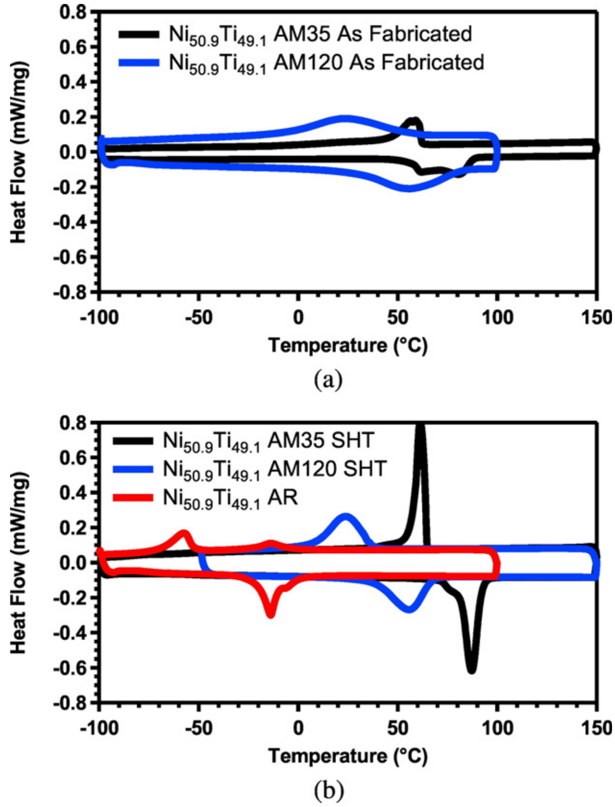

Fig. 1. DSC results of (a) the as-fabricated AM NiTi samples; (b) the same samples after solution heat treatment (SHT) at 900°C for 1 h compared to the as-received $Ni_{50.9}Ti_{49.1}$ alloy. AM35: the samples fabricated using 35 μm hatch distance, AM120: the samples fabricated using 120 μm hatch distance. For the other processing parameters, please refer to the text.

control of precipitates upon heat treatments. The rest of the material was used to produce NiTi in powder form using gas atomization by Nanoval GmbH ($d_{50} = 18.5$ μm). Tension and DSC samples were fabricated on a 3D Systems ProX DMP 100 L-PBF AM system. Two sets of samples were produced. For both sets, the laser power, scanning speed, and layer thickness were kept constant at 50 W, 80 mm/s, and 30 μm, respectively. These parameters were earlier shown to result in macro porosity/macro crack free samples [11]. Sample set 1 (referred to as AM120) was fabricated with 120 μm hatch spacing, while sample Set 2 (referred to as AM35) was fabricated with 35 μm hatch spacing. Hatch spacing refers to the distance between two adjacent passes of the laser beam within the same layer, and was chosen as the primary variable in this study because it was reported in our previous works that it has a significant effect on the transformation temperatures [11, 18]. However, the effects of these parameters on the shape memory/functional properties under tension have not been reported. The laser scanning pattern was alternated between perpendicular directions every other layer. Rectangular blocks with dimensions 4 mm × 8 mm × 30 mm were fabricated, and samples for DSC and isobaric heating-cooling experiments were cut from each block using wire electric discharge machining (EDM) to eliminate surface effects. Additionally, as-received $Ni_{50.9}Ti_{49.1}$ and a $Ni_{49.7}Ti_{50.3}$ alloys (acquired from ATI in hot-rolled form) were similarly prepared as controls.

A TA Instruments Q2000 DSC instrument was used to measure the transformation temperatures. Flat squares with 3 mm × 3 mm × 1 mm dimensions were cut using EDM. The samples were subject to three DSC cycles at a heating/cooling rate of 10 °C/min, either from −100 °C to 100 °C or from −50 °C to 150 °C. Transformation temperatures were determined using the tangent line intercept method in the second cycles to eliminate the first cycle effects.

Local compositional analysis was conducted on some AM and conventionally manufactured DSC samples using Wavelength Dispersive Spectroscopy (WDS) in a Cameca SXFive Scanning Electron Microscopy. Ten measurements were taken from each sample, and all samples were measured in the same session under the same beam conditions to minimize bias error between samples.

Dog-bone shaped miniature tension samples with a gage length of 8 mm were EDM-cut from both the conventionally manufactured NiTi samples and the AM-fabricated blocks. For the AM samples, the tension axis was perpendicular to the build direction and oriented either parallel or perpendicular to the laser scanning direction. The samples were cut at least 0.5 mm away from the surface of the as-built block to eliminate the effect of surface defects. Both the AM and conventional NiTi samples were cycled from approximately −50 °C to 150 °C under constant tensile stress levels in an MTS servohydraulic testing system, and the applied stress was increased incrementally after each heating-cooling cycle up to 500 MPa. For the AM NiTi samples, the heating-cooling experiment was repeated on three separate samples to ensure reproducibility.

Fig. 1 compares the DSC results for AM samples (AM35 and AM120) in both the as-fabricated and the solution heat-treated (SHT) cases, and the corresponding transformation temperatures are presented in Table 1. The transformation temperatures of the as-fabricated NiTi AM samples are considerably higher than that of conventionally manufactured $Ni_{50.9}Ti_{49.1}$ alloy. The AM35 samples show higher transformation temperatures than AM120 samples in both the as-fabricated and SHT conditions. Since NiTi transformation temperatures are inversely proportional to nickel content, these two observations indicate that L-PBF AM causes significant nickel loss. The nickel loss has been reported in the literature, and is attributed to nickel evaporation during L-PBF process, since nickel has a lower boiling point than titanium [13].

The effect of SHT on the DSC results is reported in Fig. 1b. SHT of the AM samples at 900 °C for 1 h did not result in notable changes of the martensite (58 °C) or austenite (86 °C) peak temperatures, but it reduced the width of the peaks (M_s-M_f or A_s-A_f), indicating that the solution heat-treatment made the samples more homogeneous or eliminated AM-induced defects. This suggests that the as-built samples have local inhomogeneity in their composition or microstructure. The as-fabricated samples also show shallower transformation peaks (or

Table 1
Transformation temperatures, transformation enthalpies and the results of the WDS analysis of the chemical compositions for four different materials: conventionally fabricated $Ni_{50.9}Ti_{49.1}$ and $Ni_{49.7}Ti_{50.3}$ samples, and AM35 and AM120 samples fabricated in this study, all in as-received/as-fabricated and solution heat treated conditions. The results show the average values of at least 10 measurements at different locations in a single sample. SHT: Solution heat treatment at 900°C for 1 h. AR: As-received.

	M_f (°C)	M_s (°C)	A_s (°C)	A_f (°C)	ΔH_M (J/g)	ΔH_A (J/g)	Ni content (at.%)	Ti content (at.%)
$Ni_{49.7}Ti_{50.3}$ SHT	52	71	84	106	33	35	48.87 ± 0.13	51.13 ± 0.13
AM35 as fabricated	47	62	56	88	14	14		
AM 35 SHT	58	65	81	92	28	27	47.95 ± 0.91	52.05 ± 0.91
AM120 as fabricated	−2	57	33	82	23	24		
AM 120 SHT	8	37	39	67	22	23	49.09 ± 0.4	50.91 ± 0.4
$Ni_{50.9}Ti_{49.1}$ AR	−70	−52	−22	−8	8	15		
$Ni_{50.9}Ti_{49.1}$ SHT	No transformation						50.08 ± 0.08	49.92 ± 0.08