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Selective Laser Melting of Ni-Rich NiTi: Selection of Process Parameters and the Superelastic Response

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Selective laser melting of Ni-rich NiTi: Selection of process parameters and the superelastic response

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ABSTRACT

Material and mechanical properties of NiTi shape memory alloys strongly depend on the fabrication process parameters and the resulting microstructure. In selective laser melting, the combination of parameters such as laser power, scanning speed, and hatch spacing determine the microstructural defects, grain size and texture. Therefore, processing parameters can be adjusted to tailor the microstructure and mechanical response of the alloy. In this work, NiTi samples were fabricated using $Ni_{50.8}Ti$ (at.%) powder via SLM PXM by Phenix/3D Systems and the effects of processing parameters were systematically studied. The relationship between the processing parameters and superelastic properties were investigated thoroughly. It will be shown that energy density is not the only parameter that governs the material response. It will be shown that hatch spacing is the dominant factor to tailor the superelastic response. It will be revealed that with the selection of right process parameters, perfect superelasticity with recoverable strains of up to 5.6% can be observed in the as-fabricated condition.

Keywords: Shape memory alloys, Ni-rich NiTi, Superelasticity, Additive manufacturing, Selective laser melting, Biomedical implants.

1. INTRODUCTION

In recent years, shape memory alloys (SMAs) have attracted much attention in various fields such as automotive [1, 2], biomedicine [3, 4], and aerospace [5, 6]. NiTi alloys as the most common SMA demonstrate unique functional properties, i.e., superelasticity and shape memory properties. These functional properties cause the NiTi components to recover large strains up to 8% upon unloading and unloading-heating-cooling, respectively [7-9]. In addition, NiTi alloys demonstrate other desirable characteristics such as low modulus of elasticity (~47 GPa) [10, 11], adequate fatigue life ($2N_f$ = 1271 at ε_{max} = 3.0%) [12, 13], biocompatibility, corrosion resistant (corrosion rate < 0.89 mpy), and high damping ratio (0.038 ± 0.004 in austenite; 0.002 ± 0.004 in martensite) [14-16].

In spite of the great interest in NiTi alloys, it is a very challenging task to fabricate them using conventional techniques because of the associated high reactivity and high deformability of these alloys [17, 18]. To name a few, conventional techniques include arc or induction melting, followed by a hot working process and eventually machining to the final shape. Thanks to additive manufacturing (AM) techniques, the fabrication of complex NiTi geometries, such as porous structures, component with hole, or curved samples have been recently realized. The AM techniques for fabrication of NiTi alloys are either powder-bed based (e.g., Selective Laser Melting (SLM)) or flow-based (e.g., Laser Engineered Net Shaping (LENS)). In powder-bed based AM techniques, a roller, blade, or knife deposit a powder layer on top of the building substrate while the powder is provided through one or more nozzles in flow-based AM techniques [19]. SLM is the most common AM technique for fabrication of complex NiTi parts [7, 8].

In SLM technique, the CAD model of the part and the supports are first sliced into subsequent layers with predefined thicknesses typically ranging from 20 to 100 μm. Each layer contains the SLM processing parameters (e.g., laser power-P, hatch spacing-h, scanning speed-v, layer thickness-t, scanning strategy). Next, a powder layer is deposited on top of the building plate with the thickness similar to that of sliced CAD layer. Then, the high-power laser selectively melts the powder layer according to the geometrical information of the corresponding sliced CAD. Once solidified, the building plate goes down by the thickness of a powder layer to allow the deposition of the next powder layer. The procedure is

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repeated until the production of the CAD file is completed. Finally, the surrounding powder and supports are removed to obtain the final geometry. Figure 1 summarizes the steps during the SLM fabrication.

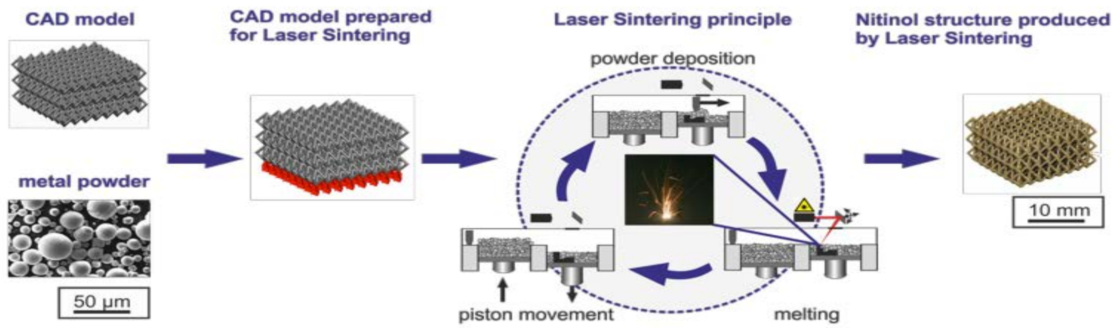

Figure 1. The schematic of SLM fabrication [19].

In biomedical applications, the superelasticity feature of NiTi alloys is an essential factor as it allows the implants to behave similar to bone through recovering a large amount of deformation (up to 8% strain). It has been reported in the literature that both SLM processing parameters and post heat treatments affect the superelasticity properties of SLM NiTi. Haberland *et al.* [20] reported stabilized superelasticity with strain recovery of 3.4% and the recovery ratio ($\varepsilon_{rec}/\varepsilon_{tot}$) of 95% in SLM $Ni_{50.7}Ti$ (at. %) after solution annealing and subsequent aging at 350 °C for 24 h. Saedi *et al.* [21] reported higher strain recovery of 5.5% and recovery ratio ($\varepsilon_{rec}/\varepsilon_{tot}$) of 95% in the solution annealed Ni-rich $Ni_{50.8}Ti$ (at. %) sample aged at 350°C for 18 h. Later, Saedi *et al*. [18] demonstrated that high energy input (E=P/(v.h.t)) of 222 J/mm^3 through low P and low v result in superelasticity. They observed strain recovery of 5.77% in the first cycle in the sample. There is no work in literature focusing on the effect of h on the superelastic response of SLM NiTi structures.

The focus of this research was to evaluate the influence of h on the superelastic response of SLM $Ni_{50.8}Ti$ (at.%). To this aim, the processing parameters of P, v, and t were considered to be 250 W, 1250 mm/s, and 30 µm while h was altered from 80 to 180µm. In this work, microstructure, transformation temperatures (TTs), and the superelastic behavior of all the samples were studied.

2. MATERIALS AND METHODS

2.1 Fabrication

The $Ni_{50.8}Ti$ (at. %) ingots were purchased from Nitinol Devices & Components, Inc. (Fremont, CA). The ingots were atomized to powder by TLS Technik GmbH (Bitterfeld Germany) using an electrode induction-melting gas atomization (EIGA) technique. A range of 25-75 µm powder particle fraction was used as this fraction resulted in appropriate flowability, density, and impurity [22]. A Phenix PXM by 3D Systems (Rock Hill, SC) was used to fabricated cylindrical samples with 4.5 mm diameter and 10 mm length for compression testing. Table 1 indicates the processing parameters implemented for the fabrication of the samples. Alternating x-y scanning strategy was also implemented [19].

Table 1. The implemented SLM processing parameters for fabrication of Ni-rich NiTi structures.

Sample #	Laser power- P (Watt)	Scanning speed- v (mm/s)	Layer thickness- t (μm)	Hatch spacing-h (μm)	Energy input-E (J/mm^3)
1	250	1250	30	80	83.3
2	250	1250	30	100	66.7
3	250	1250	30	120	55.6
4	250	1250	30	140	47.6
5	250	1250	30	160	41.7
6	250	1250	30	180	37.0

2.2 Experimental Procedure

TTs was determined using a Perkin-Elmer DSC Pyris 1 with the heating/cooling rate of 10 ℃/min in nitrogen atmosphere. Superelastic response was evaluated through thermo-mechanical tests using 100 kN MTS Landmark servo-hydraulic test platform. A strain rate of $10^{-4}s^{-1}$ was employed during loading while unloading was conducted under force control at a rate of 100 N/s. The strain was measured by a mechanical MTS high- temperature extensometer attached to the grips.

3. RESULTS

3.1 Microstructural Properties

Figure 2 (a) and (b) show the optical images of two SLM NiTi samples processed with extremely low h (h=80 μm) and high h (h= 180 μm), respectively. It is clear that h=80 sample only contains micro voids as the melting tracks are overlapping each other. On the other hand, porosities and imperfections increase with higher h spacing.

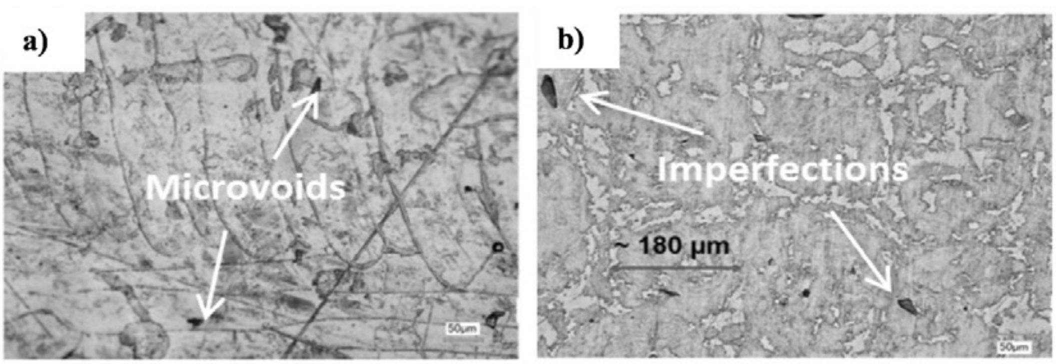

Figure 2. Optical micrographs of SLM $Ni_{50.8}Ti$ (at.%) samples fabricated by (a) h=80 μm and (b) h=180 μm.

3.2 Phase Transformation Response

Figure 3 (a) and (b) show the variation of TTs (A_f -Blue line, M_s -Red line) as a function of h and E, respectively. It is clear that TTs increase as the amount of E increase (lower h). This can be explained by the fact that higher implemented E causes the melt pools to be held at a high temperature for a longer periods of time, which, in turn, result in the higher rate of Nickel (i.e., Ni) evaporation. It has been reported that such Ni depletion is associated with an increase in TTs [23, 24].

a)

b)

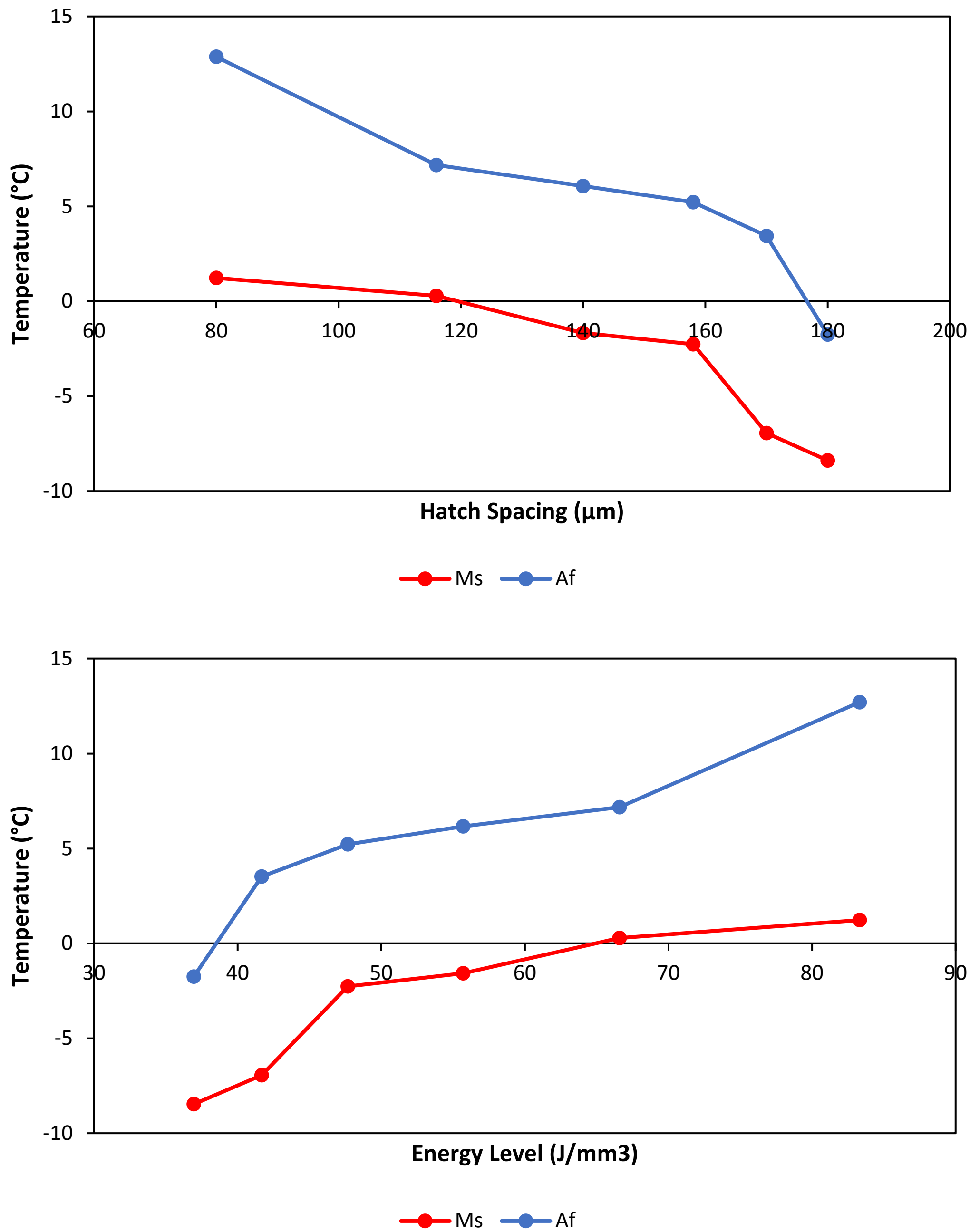

Figure 3. Transformation temperatures of SLM $Ni_{50.8}Ti$ (at.%) as function of (a) hatch spacing-h and (b) Energy level-E.

3.3 Superelastic response

Figure 4 demonstrates the superelastic responses of SLM $Ni_{50.8}Ti$ (at.%) samples tested at a temperature above A_f. The stress-strain plots indicate that lower values for h (higher E) result in the best superelastic response. For a better clarification, the total strain (ε_{tot}), irrecoverable strain (ε_{Irrec}), and recoverable strain (ε_{rec}) of the samples are also demonstrated in Table 2. For example, the sample processed by h=80 demonstrates high ε_{rec} of 5.62 and negligible amount of ε_{Irrec}.

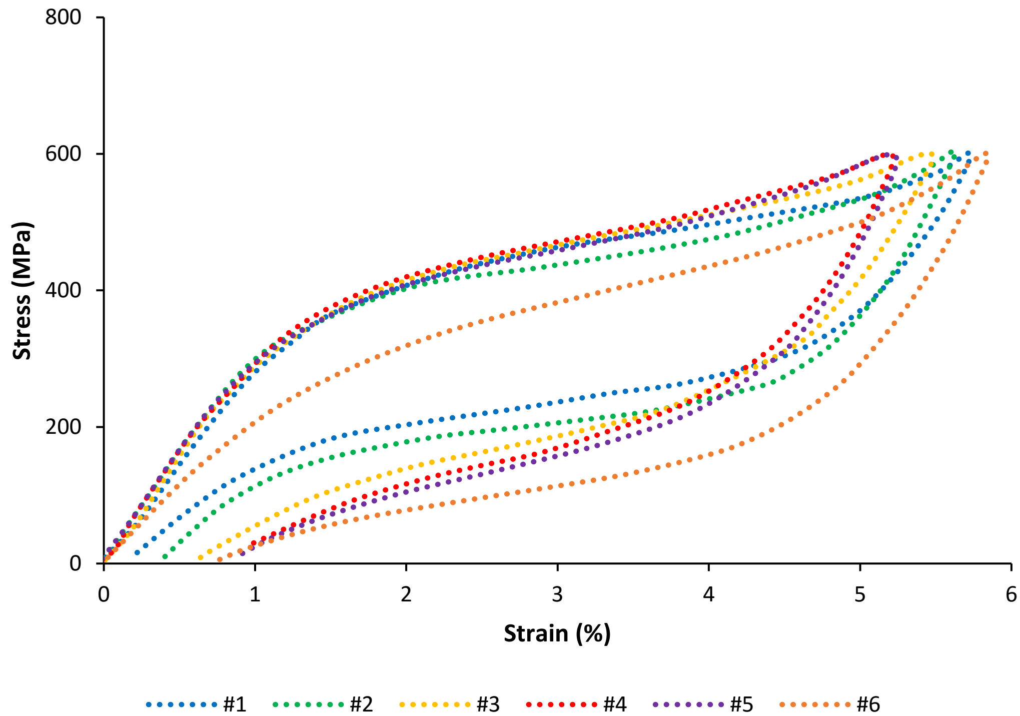

Figure 4. The superelastic response of samples tested at room.

Table 2. The total strain (ε_{tot}), irrecoverable strain (ε_{Irrec}), and recoverable strain (ε_{rec}) of SLM NiTi samples processed with different values of h.

Sample #	ε_{tot} (%)	ε_{Irrec} (%)	ε_{rec} (%)
1	5.72	0.10	5.62
2	5.51	0.38	5.13
3	5.47	0.62	4.85
4	5.12	0.8	4.32
5	5.22	0.87	4.35
6	5.85	0.77	5.08

4. SUMMARY AND CONCLUSION

In this study, we evaluated the influence of h on the microstructure, TTs, and superelastic response of SLM Ni-rich NiTi. To this aim, several cylindrical samples were fabricated using $Ni_{50.8}Ti$ (at. %) powder via SLM PXM by Phenix/3D Systems. The samples were then analyzed through DSC analysis and compression loading-unloading testing. The main findings of the study are outlined as follow:

- TTs were increased as h was decreased. This was attributed to the corresponding higher level of Ni evaporation.

- The samples processed with lower h resulted in better superelastic response. For example, h=80 sample demonstrated strain recovery of 5.62% with negligible irrecoverable strain.

The findings of this research suggest that h factor significantly affect the final properties of the SLM NiTi samples. Further investigations are however required to find the main reason behind such observations, which may help to further optimize the processing parameters.

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