Effect of solution heat treatment of nitinol (Ni55%-Ti45%)manufactured via L-PBF

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Introduction

The properties of nitinol, an alloy of nickel and titanium, include its good biocompatibility, corrosion resistance, damping capacity, fatigue strength, superelasticity, and shape memory characteristics. With other conventional methods, it has been challenging to achieve high precision and accuracy of the produced parts; however, laser powder bed fusion (L-PBF) has provided a useful new route for the processing of nitinol [1]. While L-PBF offers many advantages, it also has drawbacks, including the potential for the formation of different phases and residual stress during rapid solidification. Post L-PBF heat treatment conditions aid in the generation of targeted stable phases. As reported by Lee et. al. [2], the mechanical properties and transformation temperatures of the manufactured nitinol samples were largely influenced by the heat treatment. Fan et. al. [3] showed an increase in the transformation temperatures by increasing the heat treatment temperatures after a solution heat treatment. Heat treatments that help in achieving the desired properties are two-step heat treatment processes [2,3]. This study investigates the feasibility of applying a single-step solution heat treatment to Ni-rich nitinol and reports its effects on density, transformation temperatures, microstructures and microhardness for intended applications.

Experimental Results

Nickel rich nitinol with a composition of 55%Ni was sourced as feedstock for the laser powder bed fusion (L-PBF), from Fort Wayne Metals Ireland, to fabricate cuboids of dimensions 5 x 5 x 6 mm using a Aconity Mini 3D metal printer. The printer is equipped with a 200W fiber laser of wavelength 1068 nm. Cuboids with dimensions of 5 x 5 x 6 mm were fabricated with the process parameters as in Table 1. Layer thickness was maintained at 60 µm and laser spot size of 60 µm. After fabrication, solution heat treatment was performed on the samples using a Lenton box furnace under atmospheric conditions. Samples were treated at 800°C, 1000°C and 1200°C for varying times of 1, 3 and 5 hours respectively [4,5]. Density measurements were conducted according to ASTM standards [6] with microhardness measurements at 5 random locations on the sample [7]. Phase transformation temperatures were measured using a differential scanning calorimetry (DSC) with optical microscopy being used for microstructural characterisation. Kroll's reagent [8] was used to etch the samples and reveal the microstructures. Chemical composition was studied with the help of Energy Dispersive X-Ray Spectroscopy (EDX) equipped on a Zeiss Scanning Electron Microscope (SEM).

Table 1: Process parameters for fabrication of nitinol cuboids.

Laser Power (W)	Scan Speed (mm/s)	Hatch Spacing (µm)	VED (J/mm^3)
150	500	60	62.50

Samples heat treated at 800°C were found to be very close to that of the as-fabricated samples with density of 6.46 g/cm^3. However, for the samples heat treated at 1000°C exhibited a slight decrease in the density values to an average of 6.08 g/cm^3. Increase of time did not significantly affect the density values of the heat treated samples. It was also observed that samples heat treated at 1200°C showed an increase in density from 5.34 to 5.57 g/cm^3 with an increase in holding time. However, it was much lower than the density values of as-built samples. Vickers microhardness results exhibited an increase in hardness values to an average of 408HV for samples heat treated at 800°C than the as-fabricated samples with hardness of 356HV. It was noted that similar trends were observed for the hardness values treated at different temperatures and times. Nickel content of the samples heat treated for 3 hours at the varying temperatures was measured and reported. Samples heat treated at 800°C and 1000°C did not show significant changesin Ni content from the as-fabricated samples with a nickel content of 50.3 at.% before etching of the samples. However, samples treated at 1200°C reported higher nickel content. A general decrease in the Af

temperature was observed for the heat treated samples from the as-fabricated samples (A_f = -26°C) along with the other phase transformation temperatures including M_S, M_f, and A_S. Samples exhibited an average decrease of 25 to 30°C when heat treated. No transformation temperatures results were obtained for the samples heat treated at 1200°C. Optical microscopy revealed the microstructures after the sample surface was etched using the Kroll's reagent. As-fabricated sample showed a checkerboard pattern according to the scanning strategy of 90° rotation employed during L-PBF. While samples treated for 3 hours at varying temperatures showed similar pattern and further analysis is needed to completely understand the microstructures.

Discussion

The L-PBF process parameters have an influence on the density, microstructures, transformation temperature and microhardness of the Ni-rich NiTi samples [8]. For the heat treated samples at 800°C and 1000°C irrespective of holding times, Ni evaporation during the L-PBF process and the restructuring of the NiTi matrix led to decrease in the A_f when compared to the as-fabricated samples [5]. It is noted that even small variations in the Ni content can affect the transformation temperatures of NiTi samples, as this material is known to be highly sensitive [9]. Optical micrographs exhibited the presence of pores on the sample surface which was an effect of restructuring of the NiTi matrix which also resulted in an increase in the microhardness values. Samples treated at 1200°C need further in-depth analysis to understand why it showed a higher Ni content. This increased Ni content could be due to the formation of a Ti-rich phase, possibly Ti_2Ni.

Conclusions

The current study focused on the influence of process parameter and solution heat treatment on Ni-rich NiTi samples fabricated using the L-PBF technique. Solution treatment parameters were applied with varying temperatures (800°C, 1000°C, & 1200°C) and times (1h, 3h, & 5h). The phase transformation temperatures, density and microhardness were measured. All of the heat treated and L-PBF fabricated samples exhibited an austenitic finish temperature A_f below the room temperature. Therefore, the samples should be superelastic in nature at room temperature. Heat treated samples exhibited a decrease of an average of 25 to 30°C in A_f from the as-fabricated sample. Increased heat treatment time did not significantly affect the transformation temperature. Density values for samples treated at 800°C and 1000°C exhibited a slight decrease from as-fabricated (6.32 g/cm^3) to a lowest of 6.04 g/cm^3. However, a much lower density was found for samples treated at 1200°C (at an average of 5.46 g/cm^3). The microhardness of the heat treated samples slightly increased from 356HV to 446 HV for treatments up to 1000°C whereas samples treated at 1200°C showed hardness values up to 550HV.

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