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On Microstructure Evolution and Mechanical Behavior of Near Equiatomic Nickel Titanium (NiTi) Alloys Fabricated by Laser Deposition of Elemental Powders

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Abstract

Elementary powder of nickel (Ni) and titanium (Ti) were deposited on the surface of a Ti-6Al-4V alloy substrate by direct laser deposition. By varying laser parameters such as power and scan speed, we investigated how these factors affect phase formation, hardness, elastic modulus, and elastic recovery. Microstructural analysis revealed dendritic structure in most specimens, with variations in the formation of second phase precipitates, such as $TiNi_2$ and $TiNi_3$, depending on the processing conditions. Specimens processed with higher laser energy had fewer dendritic structures. A nanoindentation test was carried out to assess the hardness and elastic recovery of the specimens, showing differences in mechanical properties linked to the processing parameters, and it revealed that specimens processed with higher laser energy exhibited superior mechanical properties, with a recovery index reaching 30% in some cases that includes laser power of 1.25 Kw and scan speed of 1.5 m/min. When the optimized laser parameters were used, more homogeneous phases were formed, enhancing both hardness and elasticity. This study shows that direct laser deposition can be adjusted to improve the performance of NiTi alloys for applications that need high strength and elasticity.

Categories: Advanced Materials, Materials Engineering, Design and Manufacturing
Keywords: nitinol, shape memory alloy, additive manufacturing, hardness, microstructure

Introduction

Near equiatomic nickel-titanium alloys, which are referred to as shape memory alloys, have drawn a lot of attention to them for different industrial applications due to their interesting combination of shape memory effect, superelasticity, and perfect biocompatibility [1,2]. These unique properties are mainly due to the material's microstructure and phase transformations, and that made the investigation of the microstructural evolution important for understanding the mechanical behavior of the alloys [3,4]. The shape memory effect and the superelasticity of nickel titanium alloys are a result of a reversible martensitic transformation between the austenite and martensite phases, which can be influenced by different factors such as processing conditions, composition, and thermal or mechanical loading [1,3]. These alloys have applications in various fields, such as biomedical devices, including stents, orthodontic wires, and components for energy-harvesting systems [2,4]. However, getting the desired mechanical performance, thermal stability, and functional reliability needs an understanding of the underlying microstructural mechanisms.

Laser direct metal deposition or laser cladding refers to the additive manufacturing process for building parts from a CAD model without the use of forming dies, tooling, or machining [5]. In contrast to conventional methods, such as casting or powder metallurgy, laser depositions offer a localized control of thermal input, which significantly affects the microstructure and as a result the mechanical properties of the nickel titanium alloys [6,7]. In particular, the direct use of elemental powders for laser deposition offers an opportunity to engineer composition gradients and control phase evolution, and so offering a pathway to customize material properties [8]. This technique also has the potential for reducing material waste and fabrication time, which goes along with the growing demand for sustainable manufacturing practices. Despite its advantages, the fabrication of the near equiatomic nickel titanium alloys by Laser deposition has its own challenges. These involves achieving a precise compositional control to make sure of the needed Ni\Ti ratio, controlling the thermal gradients to minimize the defects like cracks and pores, and understanding the relation between the processing parameters and the resulting microstructure. In addition to that, the rapid cooling rate which is involved in the laser deposition can result in the formation of metastable phases or inhomogeneous composition, which may affect the functional properties of the alloy [9,10]. Recent studies have highlighted the important role of the processing parameters, such as laser power, scanning speed, and powder feed rate in determining the resulted microstructure and phase stability of nickel titanium alloys during laser deposition [7,8]. For example, improved processing conditions can promote the development of a uniform microstructure featuring small grains, which in turn boosts the mechanical characteristics and functional efficiency of the alloy. Additionally, post processing methods like heat treatment and surface modification have been investigated to enhance the microstructure and reduce residual stresses, guaranteeing long-term reliability during operation [10].

In this study, a detailed investigation into the effect of the laser power and the scanning speed on the microstructure and mechanical properties of near equiatomic nickel titanium alloys fabricated using laser deposition of elemental powders was conducted. The main objective is to establish an understanding of how changing these critical process parameters affects key aspects such as phase composition, grain morphology, and mechanical behavior. To achieve this, microstructural analysis techniques, including scanning electron microscopy and energy dispersive X-ray spectroscopy (EDS), are employed to characterize the phase distribution and compositional uniformity of the alloys. In addition to that, nanoindentation testing is carried out to evaluate the mechanical properties, such as hardness and elastic modulus. Particular emphasis is placed on identifying the optimal laser power and scanning speed combinations that promote favorable phase transformations, such as the formation of austenite and martensite phases. By correlating the microstructural findings with mechanical performance data, this study seeks to uncover the interplay between laser parameters, thermal gradients, and the resulting functional properties of nickel titanium alloys. The findings of this research offer significant insights into the role of laser deposition parameters in controlling phase evolution and mechanical behavior, providing a foundation for the design and fabrication nickel titanium components.

Materials And Methods

Materials

Elemental nickel and titanium powder with a particle size of approximately 90 microns were utilized in this study. These powders were selected for their high purity and uniform particle size distribution, ensuring consistent flowability and efficient mixing during the laser deposition process. The substrate material employed was Ti-6Al-4V, a widely used titanium alloy that offers excellent mechanical properties, superior corrosion resistance, and high compatibility with laser deposition techniques. The Ti-6Al-4V substrate ensures good adhesion to the deposited layers and reduces thermal expansion mismatches, preventing residual stresses and delamination. This combination of materials ensures a robust foundation for exploring the effects of laser processing parameters on the resulting microstructure and mechanical properties of near equiatomic nickel titanium alloys [11].

Processing equipment

The specimens were prepared based on the optimum conditions listed in Table *1* which were concluded from our previous research [11]. The Ti-6Al-4V plate was used as a substrate for the deposition of the elemental powder of nickel and titanium. The plates were sand blasted and cleaned with acetone before the process. The equipment used was a 4.4kW Nd; YAG laser system. The laser beam was focused using a series of mirrors that reflect it into the precise location where it was wanted. The laser head was then positioned to a height that gives the laser beam the desired spot diameter for the particular sample. The laser was manipulated using a 5-axis Kuka robot arm shown so that it can be programmed to produce the desired part [11].

The substrate for powder deposition is preheated to 500°C using a ceramic plate equipped with a coil heater and a thermocouple, integrated into a simple circuit for temperature control. For the deposition of nickel and titanium powder on the substrate a 3-way coaxial nozzle system connected to a 5-axis CNC machine and a 1.5 bar GTV multi-hopper powder feed system. The powder feed system regulates the powder feed rate through its rotary speed. To evaluate the flowability, the multi-hopper feeding system was set to a specific rpm and operated for 60 seconds, after which the container was weighed before and after the operation. This measurement was performed for both powder materials at different rpms as part of a calibration process to optimize the required weight percentage by adjusting the rpm [11].

Specimen no.	Power (kW)	Scan speed (m/min)	Ti-powder			Ni-powder		
			rpm	Deposition rate (g/min)	Deposition rate (atom/min)	rpm	Deposition rate (g/min)	Deposition rate (atom/min)
1	1.25	2	1.5	5.09	6.40E+22	1.5	4.74	4.86E+22
2	1	1.5	1.5	5.09	6.40E+22	1.5	4.74	4.86E+22
3	1.15	1.5	1.5	5.09	6.40E+22	1.5	4.74	4.86E+22
4	1.25	1	1.5	5.09	6.40E+22	1.5	4.74	4.86E+22
5	1.25	1.5	1.7	5.87	7.38E+22	1.5	4.74	4.86E+22
6	1.25	1.5	1.9	6.26	7.87E+22	1.5	4.74	4.86E+22

TABLE 1: Laser experimental processing parameters for powder deposition

Materials characterization and mechanical testing

Cross-sections of the laser metal deposited specimens were prepared for microstructural analysis. The samples were mounted, finely polished, and etched using a solution composed of 3.2 mL HF, 12.2 mL HNO_3, and 84.6 mL H_2O. The microstructural analysis was conducted using a Ferox PL trinocular optical microscope. The nano-indentation test was done at the CSIR-National laser center (NLC) in Pretoria, South Africa, using Anton Paar nanoindentor whose indentor is of Berkovich type and made of diamond.

Results

For the purpose of examining the shape memory alloys (SMA) properties, several tests were carried out. Some of them were done to study the metallurgical characteristics such as the type of microstructure and the type of defects present, by inspecting the cross-section of the specimen. Nevertheless, the rest of the tests were performed to study the mechanical properties, such as hardness and elastic recovery.

Microstructural characterization

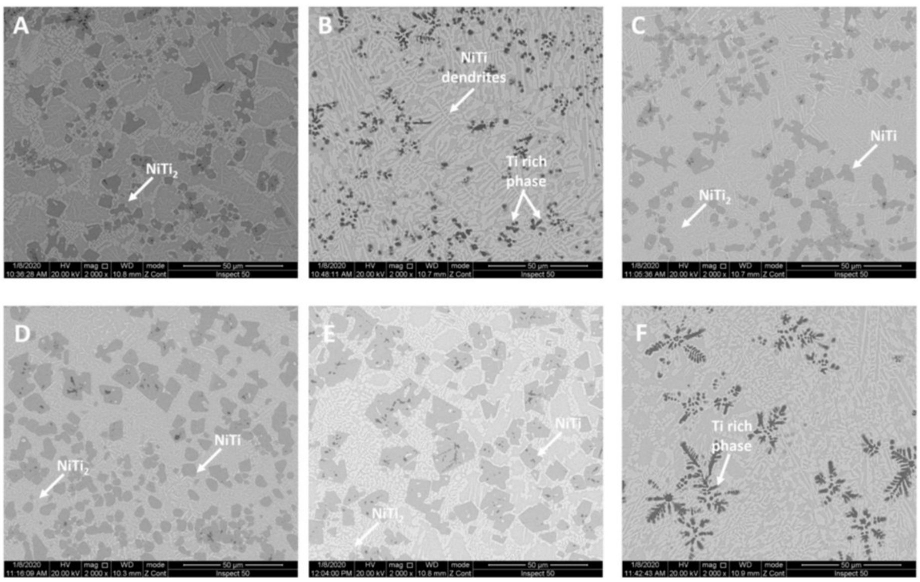

FIGURE 1: Microstructure images of the specimens: (A) specimen 1, (B) specimen 2, (C) specimen 3, (D) specimen 4, (E) specimen 5, (F) specimen 6

Microstructures of the specimens were examined by taking images from the center of each sample to ensure consistency. A common feature across all the specimens was a dendritic structure, which indicates rapid solidification during laser deposition. We also observed intermetallic compounds, such as $TiNi_2$ and $TiNi_3$, which are typical of NiTi alloys produced under these rapid cooling conditions [12]. There were no cracks in any of the specimens, suggesting good structural integrity. The micrographs showed that all specimens had a TiNi matrix, with darker regions representing $NiTi_2$ rich areas. In particular, specimens 2 and 6 exhibited a flower-like morphology, suggesting the presence of a Titanium-rich phase, which likely formed due to excess titanium not fully reacting with nickel during processing. These images are shown in Figure *1*. By comparing the specimens processed with different laser parameters, we found that the dendritic structures were less pronounced in specimen 4, which had the highest laser energy. This indicates that higher energy input allowed for a more complete phase transformation. In contrast, specimens with lower laser energy retained more dendrites and exhibited martensitic phases.

Nanoindentation test

To evaluate the mechanical properties, we performed nanoindentation testing, which helped us assess both the elastic recovery and microhardness of the specimens. Each specimen underwent 10 cycles of loading and unloading, with a constant rate of 600 mN/min and a maximum load of 200 mN per cycle. After each unloading phase, there was a 20 second hold time to ensure stability. The load-displacement curves generated from these tests are shown in Figure *2* and the corresponding elastic recovery values are listed in Table *2*. These results provide insights into the material's ability to recover elastically under cyclic loading and how it responds to repeated stress. Specimens 1 and 2, which were processed under similar parameters, exhibited consistent elastic recovery values of approximately 28.1% and 27.8%, respectively, indicating good repeatability of the deposition conditions. Specimen 3, although it was processed under the same nominal conditions, displayed a lower average recovery of 24.7%. Specimen 4 exhibited the highest maximum indentation depth (993 nm), and the average recovery across 10 cycles was 28.5%. Specimen 5, which had a slightly increased titanium feed rate compared to previous specimens, had an average recovery of 26%. Specimen 6, processed with the highest titanium deposition rate (6.26 g/min), showed the highest elastic recovery, 30.0%, among all samples.

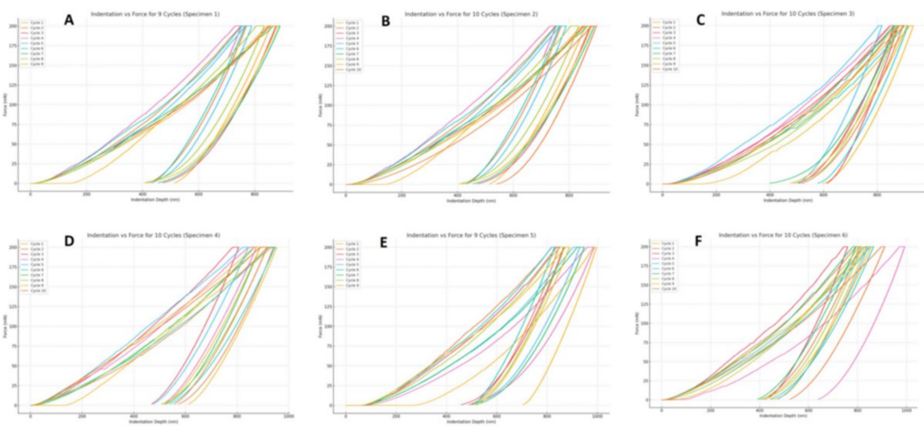

FIGURE 2: Force indentation curves: (A) specimen 1, (B) specimen 2, (C) specimen 3, (D) specimen 4, (E) specimen 5, (F) specimen 6

Specimen no.	1	2	3	4	5	6
Elastic recovery	28.1%	27.8%	24.7%	28.5%	26%	30%

TABLE 2: Elastic recovery values

Mechanical properties

The hardness values of the specimens were determined using the Oliver and Pharr method, and the results are summarized in Table *3*. Specimen 6 exhibited one of the highest hardness values, which is likely due to its higher titanium content. On the other hand, specimen 5 had lower hardness despite undergoing similar processing conditions. The elastic modulus values for the specimens ranged from 239.45 GPa (Specimen 4) to 297.49 GPa (Specimen 1), and these values are also provided in Table *4*. The trends in hardness and modulus, related to the laser energy and processing parameters, are discussed further below.

Specimen no.	1	2	3	4	5	6
Hardness (HVIC)	1865.4 V	1647.57 V	1511 V	1600 V	1480 V	1852 V

TABLE 3: Hardness values of deposited layers

Specimen no.	1	2	3	4	5	6
Elastic modulus (GPa)	297.49	289.12	282.65	239.45	252.23	268.03

TABLE 4: Elastic modulus values of deposited layers

Discussion

Microstructural characterization

Dendritic structures were a common feature across all specimens, indicating rapid solidification during laser deposition [13]. Additionally, intermetallic compounds such as $TiNi_2$ and $TiNi_3$ were identified, which are typical in NiTi alloys formed under these rapid cooling conditions. The images revealed that no cracks were present in any of the specimens, suggesting good structural integrity and processing quality. The contrast in the images allowed for the differentiation of phases within the material. All specimens exhibited a NiTi matrix, with darker areas identified as regions rich in $NiTi_2$. A flower-like morphology, especially noticeable in specimens 2 and 6, indicated the presence of a Titanium-rich phase. This likely resulted from excess titanium that did not fully react with the molten nickel. As the remaining titanium remelted, it segregated to form this distinctive structure [14]. To better understand the effects of laser process parameters such as power, scan speed, and energy density on the microstructure, the results were compared across all specimens. The dendritic structures, which appeared in most of the specimens, represent a metastable phase that ideally should have transformed into the stable NiTi phase with sufficient energy in the melt pool. However, lower laser energy resulted in incomplete phase transformation, as seen in several specimens. In contrast, specimen 4, which was processed with the highest laser energy, had the fewest dendrites, indicating a more complete phase transformation due to the higher energy input [15]. Additionally, specimens produced with lower laser power and scan speed exhibited a martensitic phase in their microstructures, which aligns with findings reported by Dadbakhsh et al. [16]. The lower thermal input likely favored the retention of the martensitic phase, preventing the transformation to a stable austenitic NiTi phase. When comparing specimens 1 and 2, it was observed that decreasing both laser power and scan speed led to an increase in second phase precipitates, such as $TiNi_2$ and $TiNi_3$, within the microstructure. This could be attributed to the insufficient time and heat needed to dissolve these precipitates during processing. Conversely, comparing specimens 2 and 3, where the scan speed remained constant and only the laser power was increased, showed a significant reduction in precipitates. The increase in laser power provided the necessary energy and time for these phases to dissolve, resulting in a more homogeneous microstructure [17,18].

Nanoindentation and hardness evaluation

The elastic recovery behavior observed across the six specimens reveals how sensitive mechanical performance is to small variations in processing conditions especially the titanium deposition rate. One of the most notable trends was seen in specimen 6, which showed the highest average elastic recovery (30%). This can be attributed to the higher Ti feed rate used during its fabrication, which may have resulted in a denser and more uniform microstructure, which also can likely be attributed to a higher concentration of titanium-rich phases and intermetallic compounds. These phases have a significant impact on the mechanical properties, leading to more variability in the material's response under cyclic loading conditions [19]. Interestingly, specimens 1 and 2 were processed under the same conditions, and they showed very similar recovery values (28%). This consistency is reassuring and suggests that the process itself is reliable and repeatable under controlled parameters. On the other hand, specimen 3, despite having the same nominal settings, showed lower elastic recovery (24.7%). This discrepancy may reflect underlying variations in the microstructure, possibly from subtle defects, porosity, or thermal inconsistencies during the build process. Specimen 5, which had a moderately increased Ti feed rate, showed only a slight improvement in recovery compared to previous specimens. This suggests that simply increasing one parameter, like Ti feed, is not always enough. Specimen 4 showed a consistent and stable nanoindentation curves. This consistency indicates an optimized microstructure, characterized by less defects and more uniform phase distribution. The good mechanical properties of this specimen can be attributed to higher laser energy applied during processing which resulted into more homogeneous microstructures. The nanoindentation results suggest that higher laser energy, in conjunction with proper post-processing, leads to improved microstructural stability and mechanical properties [20]. However, mechanical behavior seems to be governed by the interaction of multiple factors, including both Ti and Ni deposition rates, as well as thermal effects during deposition.

The hardness values from the nanoindentation tests showed that specimen 6, which had a higher titanium content, displayed greater hardness. This aligns with the findings of Shiva et al. [21], who observed that an increased titanium ratio promotes the formation of $NiTi_2$, a phase known to enhance hardness. In contrast, specimens processed with lower scan speeds like specimens 1 and 4 exhibited lower hardness due to a reduction in the formation of second-phase precipitates. Conversely, for specimens 2 and 3, reducing laser power led to an increase in hardness. This could be due to the insufficient energy in the melt pool, which prevented the complete dissolution of second phase precipitates, thus contributing to higher hardness values. These findings align with the observations of Marattukalam et al. [22] and Saedi et al. [23], who both reported similar trends linking laser energy and hardness.

Elastic modulus insights

The elastic modulus values ranged from 239.45 GPa for specimen 4 to 297.49 GPa for specimen 1. This variation can be explained by the differences in laser energy used during the process. Higher laser energy generally leads to slower cooling rates, which can promote larger grain sizes and more complete phase transformations, resulting in higher elastic modulus values. Conversely, lower laser energy results in faster cooling, leading to finer grain structures and different phase balances, which is likely why some specimens exhibited lower modulus values [24]. These results further emphasize the importance of controlling processing parameters to optimize the mechanical properties of NiTi alloys.

Conclusions

The present results reveal how sensitive the microstructure evolution and mechanical behavior of nickel titanium alloys are to the processing parameters of laser deposition. From the microstructure investigation, it was clear that changing the processing parameters affects the microstructure and the precipitates deposited. Reducing the laser power and scan speed increases the deposition of second-phase precipitates, as insufficient heat and time are available to melt them. Conversely, increasing the laser power while maintaining a constant scan speed reduces the precipitates. Additionally, decreasing the scan speed while keeping the laser power constant minimizes the occurrence of cracks. For the nanoindentation results, all specimens showed some recovery. However, specimen 6 had the highest recovery values and this is due to the higher amount of titanium deposited which improved the functional property. It was observed that reducing the scan speed resulted in decreased hardness, while lowering the laser power increased hardness. This behavior is attributed to the number of precipitates formed and the influence of laser power and scan speed on their formation.

Additional Information

Author Contributions

All authors have reviewed the final version to be published and agreed to be accountable for all aspects of the work.

Concept and design: Engy M. Zain, Sisa Pityana, Mohamed A. Taha

Acquisition, analysis, or interpretation of data: Engy M. Zain, Ahmed M. Elsabbagh, Mohamed A. Taha, A.F. Youssef, M. Abdelwahed

Drafting of the manuscript: Engy M. Zain, Ahmed M. Elsabbagh, M. Abdelwahed

Critical review of the manuscript for important intellectual content: Engy M. Zain, Ahmed M. Elsabbagh, Sisa Pityana, Mohamed A. Taha, A.F. Youssef, M. Abdelwahed

Supervision: Sisa Pityana, Mohamed A. Taha

Disclosures

Human subjects: All authors have confirmed that this study did not involve human participants or tissue. **Animal subjects:** All authors have confirmed that this study did not involve animal subjects or tissue. **Conflicts of interest:** In compliance with the ICMJE uniform disclosure form, all authors declare the following: **Payment/services info:** This work lies within the frame of a project funded by the African Laser Centre Research (ALC) Grant Collaboration Program 2021/2022. Project PI is Sisa Pityana, CSIR

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