Comparison of the Production Processes of Nickel-Titanium Shape Memory Alloy through Additive Manufacturing

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ABSTRACT

Shape memory alloys are a group of intermetallic materials that exhibit distinctive properties in biomedical, aerospace, robotics, mechatronics and many other engineering applications. Among them, the nickel-titanium (NiTi) shape memory alloys (SMA) are the most attractive materials due to their significant physical and mechanical properties including one-way and two-way shape memory effect, superelasticity and high damping coefficiency. Development of these materials using conventional production methods is still a challenging task due to the high chemical sensitivity and thermomechanical characteristics. However, the recent technological innovations provide a new manufacturing technique which is called additive manufacturing. Additive manufacturing has attracted much attention and can provide more effective, lower cost and higher productivity solutions rather than the conventional melting and powder metallurgical methods for the NiTi components. As part of AM, the electron beam melting (EBM) or a direct selective laser melting (SLM) processes provide new opportunities for direct melting of parts using a high-power electron beam or a laser beam. In this study, a comparison is made on the processing of NiTi alloy via AM. Furthermore, the effect of powder properties on the production of NiTi shape memory alloy is investigated.

Keywords: *Additive Manufacturing, Nickel-Titanium Alloy, Shape Memory Alloys*

ÖZET

Şekil bellekli alaşımlar, biyomedikal, havacılık, robotik, mekatronik ve diğer birçok mühendislik uygulamasında ayırt edici özelliklere sahip olan intermetalik malzeme grubudur. Bunların arasında, nikel-titanyum (NiTi) şekil bellekli alaşımlar (ŞBA), tek yönlü ve iki yönlü şekil bellek etkisi, süperelastik davranış ve yüksek sönümleme katsayısı gibi önemli fiziksel ve mekanik özelliklerinden dolayı en etkin kullanılabilen malzemelerdir. Bu malzemenin geleneksel üretim yöntemleri kullanılarak geliştirilmesi, yüksek kimyasal hassasiyet ve termomekanik özelliklerden dolayı halen zor bir süreçtir. Bununla birlikte, son teknolojik gelişmeler, katmanlı imalat olarak adlandırılan yeni üretim tekniğini ortaya çıkarmıştır. Katmanlı imalat, NiTi bileşenleri için geleneksel ergitme ve toz metalürjisi yöntemlerinden daha etkili, düşük maliyetli ve yüksek üretilebilirlik çözümleri sağlamaktadır. Katmanlı imalat yöntemleri içerisinde yer alan, elektron demet ergitme (EBM) ve direkt seçmeli lazer ergitme (SLM) işlemleri, parçaların yüksek güçlü elektron ışını veya lazer ışını kullanılarak doğrudan ergitilmesi ile yeni fırsatlar ortaya çıkarmaktadır. Bu çalışmada, katmanlı imalat ile NiTi alaşımın üretim süreçleri arasında karşılaştırma yapılmıştır. Ayrıca, kullanılan toz özelliklerinin NiTi şekil bellekli alaşım üretimine etkisi araştırılmıştır.

Anahtar Kelimeler: *Katmanlı İmalat, Nikel-Titanyum Alaşım, Şekil Bellekli Alaşımlar*

1.Introduction

Additive Manufacturing (AM) used to be referred to as rapid prototyping (RP) and 3D printing in the industry for many years until the actual users noticed that the meaning of these terms is limited to describing the prototyping aspect and not the manufacturing capabilities that these technologies provide [1]. According to the terminology which is defined by the ASTM subcommittee, the additive manufacturing (AM) is defined as "a process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies". Additionally, synonyms are expressed as additive fabrication, additive processes, additive techniques, additive layer manufacturing, layer manufacturing, and freeform fabrication [2]. In the AM process, material is added layer by layer according to the original CAD data in order to produce the actual part. Thus, the description of the desired part geometry as a 3D design, formation of the geometrical information of layers and generation of the physical part are the fundamental key factors to be considered. Despite the fact that the type of the production process or raw material characteristics may be different in AM techniques, the fabrication consists of eight similar stages of all types throughout the development process as described in Fig.1.

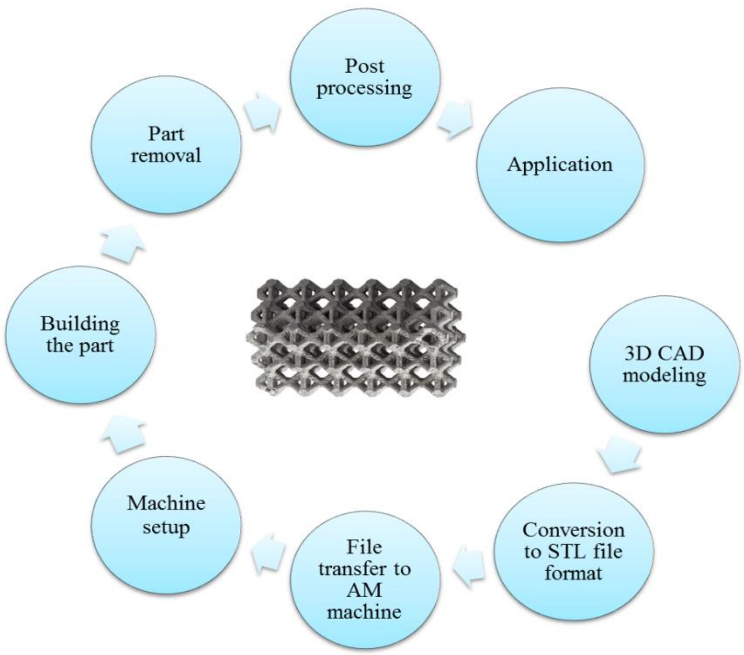

Figure 1.General AM processing steps

The term shape memory alloys (SMA) refers to a group of intermetallic materials that exhibit reversible shape change upon heating above the austenite final temperature after applying the required thermomechanical treatments [3,4]. After the discovery of the shape memory and superelastic effects in NiTi alloys, its applications have been expanded in both commercial and industrial fields including biomedical, aerospace, automotive, robotics, soft actuators, and micro-electromechanical systems [5-9].

2. Additive manufacturing of nickel-titanium shape memory alloys

Although NiTi SMAs are promising materials, the difficulties of the processing, melting and machining limit its potential applications. Particularly, due to the induced martensitic transformation and poor thermal conductivity properties, the adhesion and the tool wear can easily occur during machining. The grinding and laser cutting applications which involve crucial challenges create intermetallic phases and lead to crack formation in the heat affected zone [10-12]. The intermetallic compounds cannot be removed with conventional processing methods and require post-processing heat treatments. Additionally, fully dense materials cannot be achieved even with PM processes owing to the capillary forces coming from these compounds [13]. Thus, the production of NiTi alloy with high chemical homogeneity and good shape-memory properties especially for complex shapes and geometries is still challenging. The microstructural properties of the Ni-Ti alloys have significant effect on the static and dynamic mechanical behavior. The average diameter of the equiaxed grains should be maximum of 90 μm according to the related ASTM standard and Hall-Patch relation. Layer-based melting techniques provide anisotropic microstructures with elongated grains in the building direction and lead to that the crystallization depends on the process parameters including the powder layer thickness, the laser/ beam power, and the scanning velocity [14]. Thus, the additive manufacturing technique provides an attractive processing method for NiTi SMAs to overcome the manufacturing challenges by using layer by layer fabrication principles and the design flexibilities [15-17].

3. Comparison of the production processes

NiTi alloys can be produced using different AM techniques depending on the heating source for the melting of the powder materials; such as electron beam and laser beam. The main difference between the EBM method and laser-based methods is the usage of photons instead of electrons as a source of energy to melt the powder particles as seen in Figure 2.

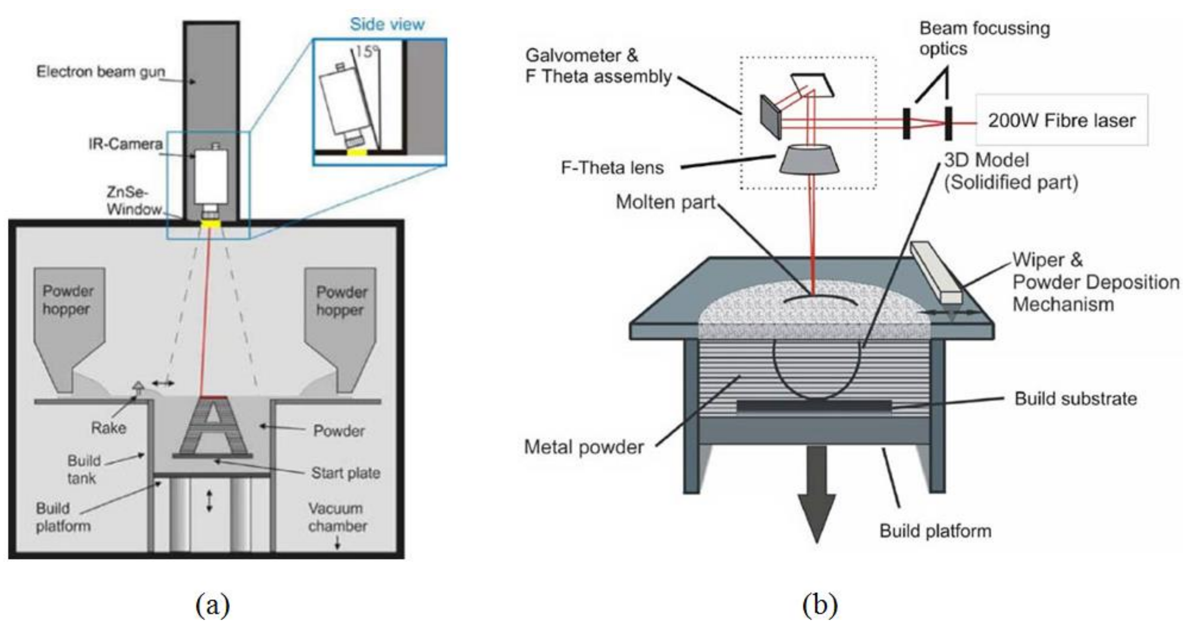

Figure 2. Schematic illustration of working principles; a) Electron beam melting [18], b) Laser beam melting [19]

There are many advantages of the EBM process compared to laser systems as described in Table 1; such as higher power efficiency, superior material properties, and 98% recyclability of metal powders used as raw materials. The control of process parameters in the EBM system is similar to the laser based systems. However, the electron beam control system does not contain any optical or moving reflectors as in laser based control systems. In addition, in order to obtain maximum efficiency in the laser system, it is necessary to match the wave lengths to different materials. While very high energy levels can be achieved with a narrow beam in electron beam systems, the power efficiency is reduced due to the excessive reflection of the photons in the laser systems. The penetration depth of an electron beam into the irradiated material is obviously greater than a laser beam. The EBM method does not require moving parts in order to deflect the beam, resulting in high scan and production speeds [20]. While the lifetime of the laser is limited, there is no restriction on the duration of use of the electron beam other than the life of the filament. The vacuum system in the EBM machines ensures a clean environment and helps to outgas impurities during processing which is highly important for the reactive metals such as Ni and Ti [21]. In contrary to EBM process, the selective laser melting (SLM) process with its inert argon atmosphere leads to higher oxygen content. Another significant advantage of the EBM system is the high scanning capability of the electron beam to preheat the powder. These advantages are important for the build process selection and the final physical properties of NiTi alloys [22, 23]. However, no previous report was found to date in the literature associated with the production of NiTi alloys via electron beam melting. In the scope of our future work, the additive manufacturing of NiTi alloys via the EBM process is going to be investigated including development of process parameters and observation of their effects on the physical properties of the alloy.

Table 1. Comparison of the main features of laser beam melting and electron beam melting

Features	**Laser Beam Melting**	**Electron Beam Melting**
Thermal Source	Fiber Laser [24]	Electron Beam [25]
Environment	Inert Gas (Argon/Nitrogen) [24, 26]	Vacuum $<5 \times 10^{-2}$ Pa chamber pressure, $<5 \times 10^{-4}$ Pa column pressure [25, 26]
Building Volume	250x250x325 mm [24]	200x200x180 mm [24]
Max. Beam Power	200/400 W [24]	3000 W [25]
Layer Thickness	20-100 µm [27]	50-200 µm [27]
Powder Fractions	25-75 µm [28]	45-105 µm [21, 29]
Operation Temperature	100-200 ºC [26]	400-1000 ºC [26]
Main process parameters	Melting: Laser power, scan speed, hatch spacing, and layer thickness [30]	Preheating, melting stages: Scanning speed, beam current, focus offset, line order, layer thickness, speed function [31]
Residual Stresses	Significant [26]	Minimum due to the preheating [26]

Several research groups have studied the production of Ni-Ti alloys using AM processes. These research projects have focused on the laser-based AM systems such as selective laser sintering (SLS), selective laser melting, direct metal laser sintering and LaserCUSING to produce NiTi components [28, 32-34]. In this regard, while fabricating dense parts via any of the AM systems, the process parameters should be optimized by minimizing the impurity pick-up without affecting the phase transformation temperatures with correct energy input in order to improve the functional properties of the alloy. For instance, Hamilton et al. fabricated Ni-Ti alloys by laser-based directed energy deposition system on a CP-Ti substrate after preheating up to 390 ºC as seen in Figure 3. This established setup diminished the thermal excursions and residual stresses [35].

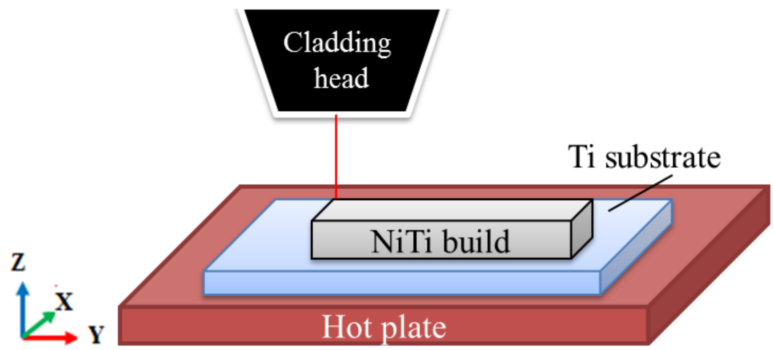

Figure 3. The build plan for laser-based directed energy deposition of NiTi SMAs [35]

Xu et al. investigated the effect of laser solid forming process parameters on the microstructure of Ti-50wt%Ni alloy. After setting the laser power to 2-3 kW, the scanning speed of 3-7 mm/s and the beam diameter of 4 mm, TiNi dendrites are obtained as the main phase. Additionally, increasing laser energy density caused the coarsening of dendrites. Furthermore, increasing the scanning velocity and decreasing the laser power, decreased dendritic arm spacing and caused the formation of two-phase TiNi + B2 eutectic dendrites and finally to TiNi + Ti_2Ni anomalous eutectic microstructures from the primary TiNi dendrite [36]. In another study by Shishkovsky et al. Ti-55wt%Ni alloy fabricated with 97 % of relative bulk density via SLM process preheating up to 900 ºC with the process parameters following as: the hatch distance of 100 µm, laser power of 50 W, laser beam diameter of 60 µm and the scan velocities 100 and 160 mm/s under the additional heating up to nearly 500 ºC. At lower energy input, the monolayers dispersion was indicated and grains were segregated after a high-speed laser recrystallization from the melt [37].

4. The effect of powder properties on the production

The EBM process begins with the formation of the first layer by raking spherical metal powders of 45-100 µm in size onto a steel platform serving as an initial substrate that has been preheated by the beam. After the accelerated electron beam has melted the metal powder by scanning a layer respectively, new powder is spread to form the second layer at the determined thickness. Preheating is carried out by a defocused beam, between the layers to ensure that the powder is added to the previous layers with low thermal stress and sintered. After a layer is melted, new metal powders are added to form the other layer, so that the cycle continues until the final part is built, based on the CAD design [31, 38, and 39]. Therefore, the feedstock quality of the powder is seen as one of the most important process inputs which is identified by the powder morphology, size, distribution, composition, conductivity and evaporation [29, 40]. In this regard, some powder characterization methods are utilized within the AM processes as classified in Table 2.

Table 2. Powder characterization types and their measurement methods

Characterization type	**Application method**
Powder Density and Flow Rate Measurements	Tap Density Apparent Density Flow Rate
Particle Size, Morphology and Distribution Analysis	Optical Microscopy (OM) Laser Diffraction X-Ray Computed Tomography
Chemical Analysis	Energy Dispersive Spectroscopy (EDS) X-Ray Photoelectron Spectroscopy (XPS) Auger Electron Spectroscopy Inductively Coupled Plasma-Atomic Emission Spectrometry Inert Gas Fusion
Microstructural Characterization	Scanning Electron Microscopy (SEM) Transmission Electron Microscopy (TEM) X-Ray Diffraction

A variety of techniques are used for the production of powders including; Plasma Atomization, Plasma Spheroidization, Vacuum Induction-melting, Inert Gas Atomization (VIGA) and Electrode Induction-melting Inert Gas Atomization (EIGA) methods [32]. The elected powder production technique plays an important role on the final material characteristics. Depending on the atomization methods, the powder shape (irregular, spherical or satellites etc.), size and porosity amount in the built material indicate some variations. Spherical particles and smaller particle sizes improve the viscosity and apparent density [26]. Fine and midfraction powders provide a large surface area which leads to absorb high energy and higher sintering rate. The ideal powder for additive manufacturing is completely spherical with Gaussian type particle size distribution and low oxygen content due to its good flowing characteristics, high packing density and new appearance after recycling in the process. Among these methods, the gas atomization is generally preferred for additive manufacturing due to the highly spherical shape of the powder. In contrast, the water atomization is very economical to produce economical irregular shaped powder. In this process, the water imparts more energy into the molten metal stream leading to the rough shape of the powder particles with rapid cooling. A spherical powder with a Gaussian particle size distribution can be optimized for additive manufacturing [41].

For the layer-based manufacturing methods, the management of the melt pool geometry, which is highly dynamic becomes necessary to achieve a successful result. The powder packing density is also an effective parameter for the melt pool geometry. Low packing density leads to the formation of a high initial porosity in the powder bed and shrinkage takes place largely after the melting stage of the material. When the powder packing density is reduced, the thermal conductivity between the layers tends to decrease due to the high amount of porosity. As a result of the reduction in the packing density the liquid pool deepens and spreads to a wider area. Thus, the high packing density is inevitable to have the desired melt pool geometry. Regarding the determination and the development of EBM parameters, the power density in the direction of the beam and the powder density distribution should be evaluated as significant parameters. Assuming that the acceleration is constant, in order to define powder density distribution, the electron beam density measurement becomes necessary [42, 43]. Additionally, the electron beam movement shows a Gaussian distribution (normal distribution). The schematic illustration of the Gaussian-shaped electron beam in the EBM machines can be seen in Figure 4 [44].

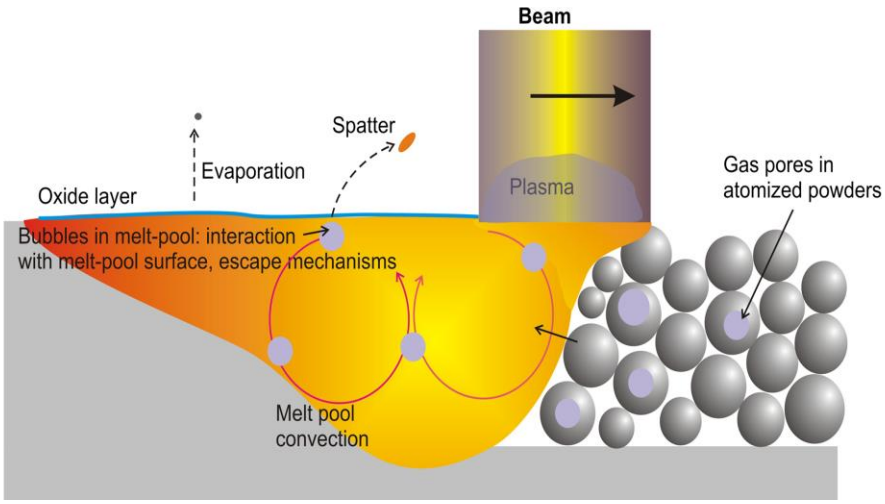

Figure 4. Schematic illustration of beam-powder interaction during powder bed melting stage [44]

The layer thickness is the other main process parameter in AM. The deposition rate of the material is associated with the z-axis layer height or layer thickness. Layer thickness is identified by the powder raking amount. During the 'raking' stage, powder is distributed to the built surface with a metal rake bar. Powder layers should be quite thin in order to obtain bulk structures with a tighter bonding. Thus during the scanning, the required energy can be easily transferred to the surface by an electron or laser beam to melt the previous layer. Eventually, the value of the layer thickness should be as low as possible in addition to powders with smaller particle size and bimodal size distribution in order to improve the surface quality of the built parts as seen in Figure 5 [45].

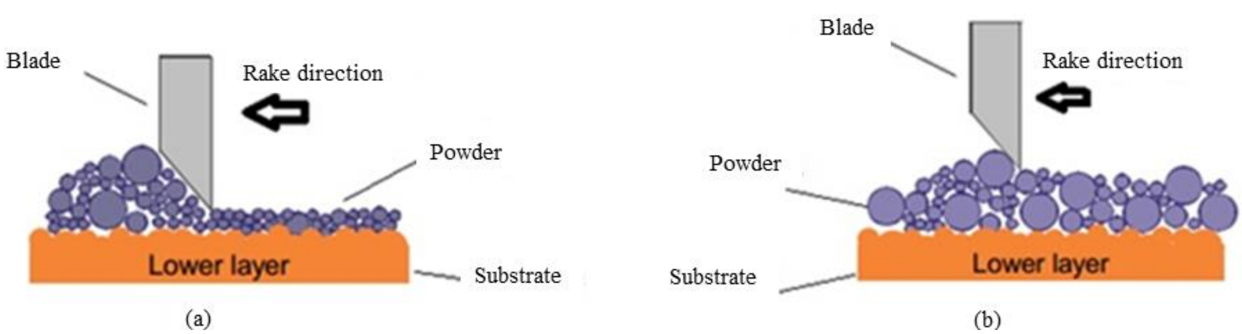

Figure 5. a) 30 µm layer thickness, high density of powder particles; b) 70 µm layer thickness, low density of powder particles [45]

5. Conclusion

The layers are provided in powders and melted by energy sources using the laser or electron beams sources for the AM of the metals. In comparison with the laser beam melting, the electron beam melting have a greater energy density, faster scan speed, and higher temperatures of the powder bed. Moreover, the higher surface quality on finished parts can be obtained in addition to the eliminated secondary processing and reduced manufacturing costs. Final manufactured material properties are highly affected by the physical properties of the powder particles in EBM and DSLM as well as other AM techniques. In general, powder structures used in AM are supposed to be nominally spherical. They should have a homogenous particle size distribution for the purpose of obtaining fully dense final manufactured parts with desired physical and mechanical properties. In the future work, the experimental results which will be obtained from electron beam melting will be released.

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