

EFFECTS OF HEAT TREATMENT AND HOT ISOSTATIC PRESSING ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Ti₂AlNb-BASED ALLOY FABRICATED BY SLM

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Orthorhombic Ti₂AlNb-based alloys are considered as promising materials for high-temperature applications in aero-engines due to their high yield strength, low-cycle fatigue, creep resistance. Additive manufacturing is a promising way of producing intermetallic alloy since it allows obtained complex-shaped parts without significant machining. At the same time, additive manufacturing of intermetallic alloys involves the use of high-temperature preheating and requires post-processing by hot isostatic pressing or heat treatment. In this paper, Ti₂AlNb-based intermetallic alloy was fabricated by SLM process with a high-temperature substrate preheating and subjected to various heat treatments and hot isostatic pressing to study their effects on microstructure and properties of the alloy.

Keywords: Selective laser melting, additive manufacturing, orthorhombic alloy, heat treatment

1. INTRODUCTION

Intermetallic alloys based on titanium aluminides present a material class with high yield strength, low-cycle fatigue, creep resistance and a high working temperature up to 650 °C [1,2]. Intermetallic titanium alloys are considered to be excellent candidates for replacing nickel-based heat-resistant materials in aerospace, automotive and energy industries due to their combination of physical and mechanical properties. Ti₂AlNb-based alloys with orthorhombic crystal structure(O-phase) have enhanced ductility and corrosion resistance compared to the alloys based on other aluminides and are considered as advanced alloys for manufacturing of gas-turbine engine parts. Intermetallic titanium alloys are of particular interest for Additive manufacturing (AM) due to their hard machinability and high cost of production by conventional methods.

AM of titanium aluminides usually requires utilization of high-temperature preheating, either by scanning with an electron beam for Electron Beam Melting process [3] or by preheating a substrate for Selective Laser Melting (SLM) process [4] to reduce residual stresses and suppress crack formation. High-temperature preheating coupled with high cooling rates typical for AM processes might lead to inhomogeneous or metastable microstructure of alloys. Hence, a proper post processing including heat treatment or hot isostatic pressing might be required to achieve an optimal phase composition and microstructure of Ti₂AlNb-based alloys.

In the recent years, there have been studies devoted to heat treatment of Ti₂AlNb-based alloys obtained by various techniques, e.g. spark plasma sintering, forging, hot pressing sintering [5, 6]. They showed that by varying annealing and aging temperature and cooling rates it is possible to influence a morphology, quantity, and type of intermetallic phases as well as grains size. At the same time, microstructures of the alloys obtained by AM are generally significantly different from conventionally obtained alloys. Thus, it is needed to investigate effects of different heat treatment conditions on microstructure of the Additive Manufactured intermetallic alloy.

In this paper, Ti₂AlNb-based intermetallic alloy was fabricated by SLM process with a high-temperature substrate preheating and subjected to various heat treatments and hot isostatic pressing to study their effects on microstructure and properties of the alloy.

2. MATERIALS AND METHODS

Gas atomized powder (**Figure 1**) of Ti-24Al-25Nb-1Zr-1.4V-0.6Mo-0.3Si (at%) alloy produced by electrode induction gas atomization (EIGA) and supplied by AMC Powders (China) was used in the SLM process. The particle size of the powder ranged from 14 to 52 μm with a mean particle size d_{50} = 29 μm .

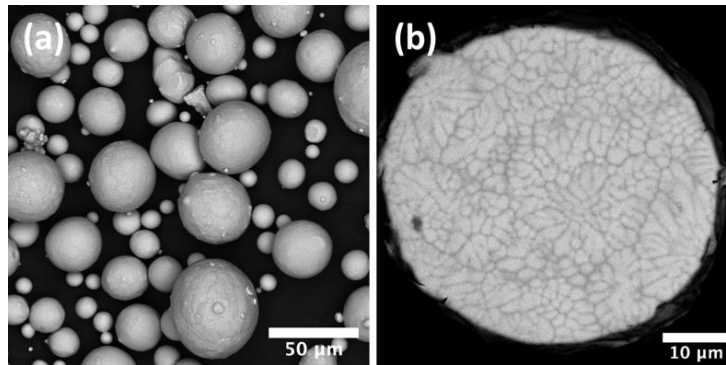


Figure 1 SEM images of the gas atomized powder (GA) showing (a) surface morphology and (b) cross-section of a particle

The samples were manufactured by the SLM process using AconityMIDI (Aconity3D GmbH, Germany) system equipped with a 1070 nm wavelength fiber laser with a maximum power of 1000 W. Cylindrical samples with 8 mm diameter and 15 mm height were fabricated for the investigation. The samples were fabricated on a Ti-6Al-4V substrate, which was put on a molybdenum platform. The molybdenum substrate was inductively preheated to a set temperature, which was continuously controlled by a thermocouple under the molybdenum platform. The titanium substrate was then conductively heated by the molybdenum substrate before starting the SLM process. The process chamber was continuously flooded with high purity argon gas to achieve oxygen content in the chamber below 20 ppm. After the build process was finished, the platform and the samples were cooled down to room temperature with a cooling rate of approximately 5 $^{\circ}\text{C}/\text{min}$. The SLM parameter sets for different preheating temperatures that provide high densities of Ti_2AlNb -alloy samples were established in the previous work [7].

Heat treatment at different temperatures was carried out in a vacuum furnace followed by furnace cooling.

To measure phase transition points differential scanning calorimetry (DSC) analysis was performed using a SETSYS Evolution 18 analyzer. The heating was carried out in an argon flow (30 ml/min) in the temperature range from 200 $^{\circ}\text{C}$ to 1300 $^{\circ}\text{C}$ at a rate of 5 $^{\circ}\text{C}/\text{min}$.

The samples were cut and polished along the build direction (BD) for the microstructural characterization. Mira 3 LMU TESCAN scanning electron microscope (SEM) in backscattered electrons (BSE) mode was utilized to evaluate the microstructures. Energy Dispersive Spectroscopy (EDS) was used for the chemical analysis of the samples and powders on the polished cross-sections. The phase composition of the powders and the fabricated samples was analyzed with a Bruker D8 Advance X-ray diffraction (XRD) using Cu-K α (λ = 0.15418 nm) irradiation.

The microhardness of the samples was measured using a Buehler VH1150 testing machine with 1000g load 10 s dwell time and tensile tests were carried out using an universal testing machine (Zwick/Roell Z100, Germany) with a tensile strain of 0.3 mm/min.

3. RESULTS AND DISCUSSION

Figure 2 shows the microstructure of the Ti_2AlNb -based alloy samples in the as-fabricated condition. The samples were fabricated using 700 $^{\circ}\text{C}$ substrate preheating temperature to obtain crack-free material. The

microstructure of the as-fabricated alloy consists of intermetallic Ti_2AlNb -phase. Melt pool boundaries can be distinguished in the BSE-images since they show slightly lighter contrast which might be attributed to a slightly higher Nb content and correspond to residual β -phase.

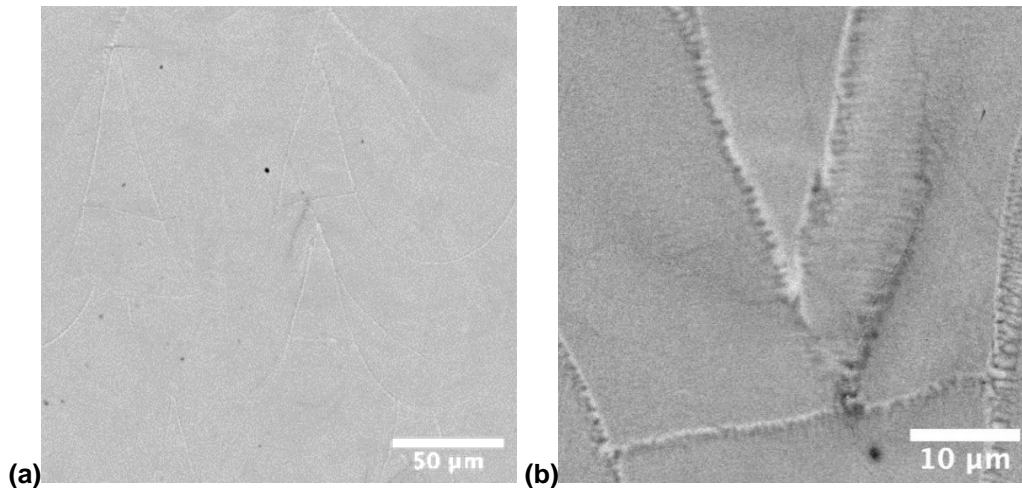


Figure 2 BSE-SEM-images of the Ti_2AlNb -alloy sample fabricated by SLM using 700 °C substrate preheating

According to the DSC-results (**Figure 3**), the preheating temperature of 700 °C is slightly above O→B2 transformation temperature and the microstructure should consist mostly of intermetallic O (Ti_2AlNb) phase. Additional holding at this temperature during the SLM process might induce B2→O transformation and result in a fully-O microstructure.

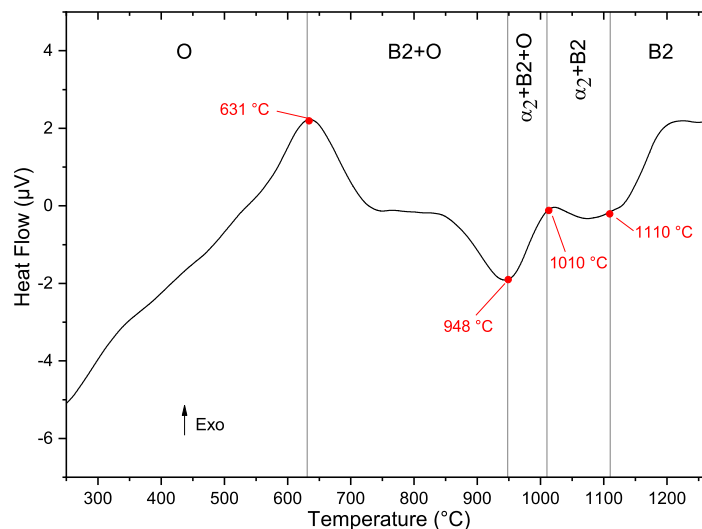


Figure 3 The DSC-curve of the Ti_2AlNb sample fabricated by SLM

Fully-O microstructure is known to have low ductility and fracture toughness since it fully consists of brittle intermetallic compound [8]. For optimal mechanical performance it is recommended to have B2+O microstructure. In order to promote O→B2 transformation and obtain dual B2+O microstructure, heat treatment of the alloy is needed. **Figure 4** shows SEM-images of the alloy microstructure after annealing at 950 °C. It can be seen that annealing of the alloy resulted in B2+O microstructure with lamellar morphology. Lamellar B2/O-phase colonies can be seen inside the prior B2/ β -phase grains. When the annealing temperature was

increased to 1050 °C, B2+O microstructure was also obtained for the Ti₂AlNb-based alloy. However, the volume fraction of O-phase significantly decreased.

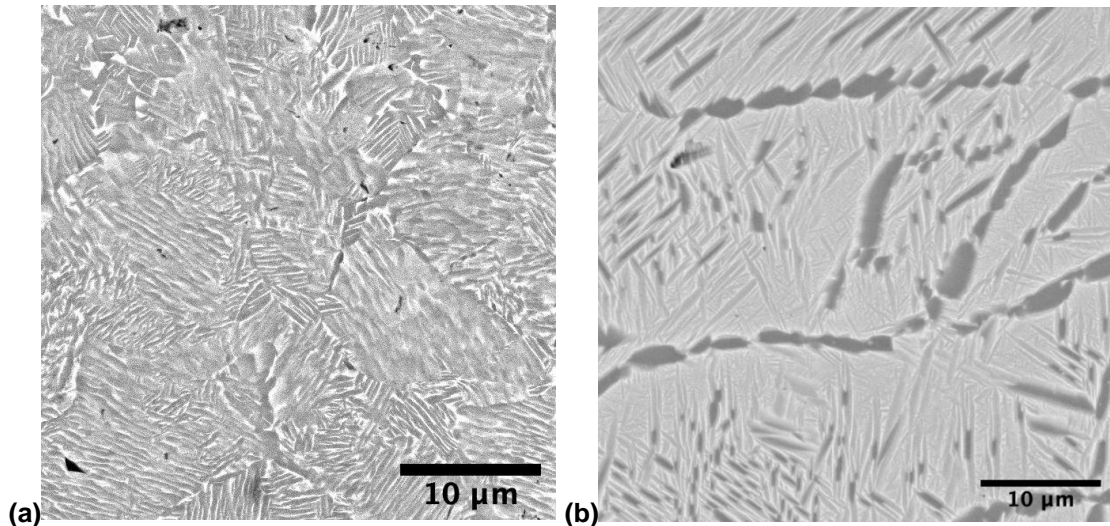


Figure 4 SEM-images of the Ti₂AlNb alloy after heat treatment at (a)950 °C and (b) 1050 °C

Additionally, hot isostatic pressing (HIP) was carried out using 1160 °C temperature, 3 h holding time, and 160 MPa pressure. The microstructure of the alloy after HIP is shown in **Figure 5**. After HIP, B2+O microstructure was obtained. Equiaxed B2-phase grains can be distinguished in the images. The B2-grain borders are characterized by the presence of O-phase precipitates formed during the cooling stage of the HIP. Fine acicular precipitates of O-phase can be seen inside the B2-grains.

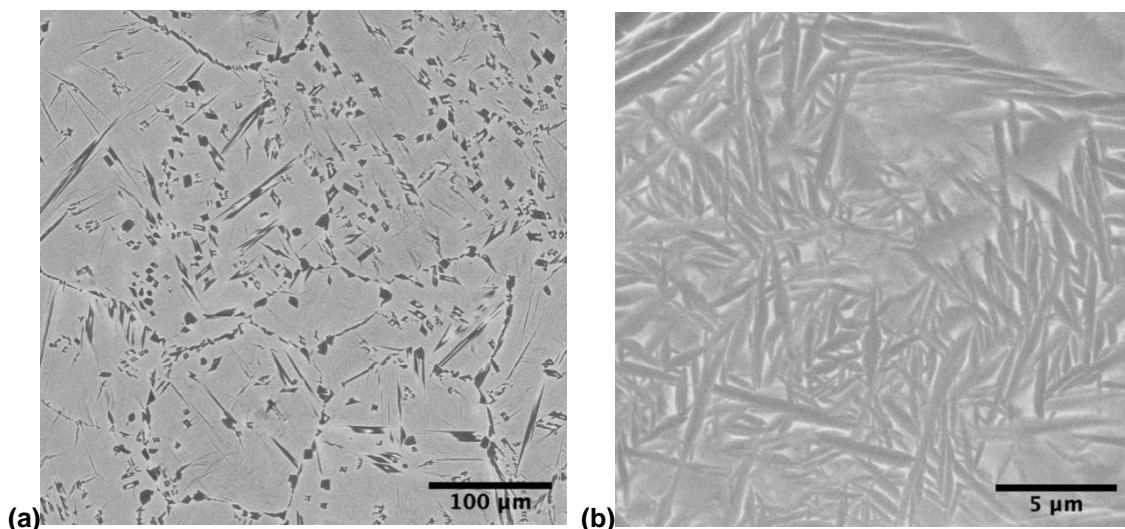


Figure 5 SEM-images of the Ti₂AlNb alloy after HIP at 1160 °C

As shown in **Table 1**, heat treatment and HIP can significantly affect the mechanical properties of the Ti₂AlNb alloy fabricated by SLM. The as-fabricated condition showed the poorest mechanical properties for the tensile tests, but the highest microhardness since the alloy exhibited fully-O microstructure. Annealing resulted in a slightly improved tensile strength, but the alloy still had low strength and brittle deformation. After HIP, the tensile strength significantly improved and reached 1030 MPa without additional heat treatment. This might be attributed to decreased number of defects (pores, microcracks) during the HIP.

Table 1 Results of room temperature tensile tests of Ti₂AlNb samples

Sample	Tensile strength(MPa)	Elongation(%)	Microhardness (HV1)
As-SLM	450	-	568
SLM + annealing at 950 °C	690	-	395
SLM + annealing at 1050 °C	-	-	384
SLM + HIP	1030	1,2	360

4. CONCLUSION

In this paper, Ti₂AlNb-based intermetallic alloy was fabricated by SLM process with a high-temperature substrate preheating and subjected to various heat treatments and hot isostatic pressing to study their effects on microstructure and properties of the alloy.

Heat treatment allowed to transform fully-O microstructure of the alloy to B₂+O and improve its mechanical properties. Depending of the annealing temperature, volume fraction of O-phase can be changed.

HIP treatment of the SLM-ed alloy significantly improved the tensile properties due to decrease in internal defects and transformation of fully-O microstructure to B₂+O microstructure.

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