EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE AND PROPERTIES OF Ti-22Al-25Nb ALLOY FABRICATED BY SELECTIVE LASER MELTING

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Abstract

Ti-22Al-25Nb orthorhombic alloy is considered to be an advanced intermetallic alloy for high-temperature applications. In this paper, the effects of Hot Isostatic Pressure (HIP), homogenizing annealing and aging in different phase regions on microstructure and microhardness of the Ti-22Al-25Nb alloy obtained from elemental powders by Selective Laser Melting (SLM) were studied. HIP allowed to reduce the residual porosity in the samples, however high-temperature annealing was needed to dissolve Nb particles and obtain a homogeneous microstructure. Differential Scanning Calorimetry was used to determine phase transition temperatures in the fabricated alloy and allowed to choose appropriate aging temperatures. Aging of the alloy resulted in the formation of intermetallic Ti_2AlNb and Ti_3Al phase depending on the temperature.

Keywords: Additive manufacturing, Ti-22Al-25Nb, heat treatment, microstructure, intermetallic

1. INTRODUCTION

Intermetallic alloys based on TiAl, Ti_3Al, Ti_2AlNb titanium aluminides present a material class with high yield strength, low-cycle fatigue, creep resistance and a high working temperature up to 650 ºC [1]. 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 [2]. Ti_2AlNb-based alloys with orthorhombic crystal structure 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 [3]. Intermetallic titanium alloys are of particular interest for Additive manufacturing (AM) due to their hard machinability and high cost of production by conventional methods.

While in the previous work [4] it was shown that Ti-22Al-25Nb alloy can be synthesized from elemental powder mixtures by Selective Laser Melting technology, it is still important to investigate the effect of heat treatment at different conditions on microstructure and properties of the alloy to achieve optimal performance of the material.

In the recent years, there have been studies devoted to heat treatment of Ti_2AlNb-based alloys obtained by various techniques, e.g. spark plasma sintering [5], forging [6], hot pressing sintering [7]. 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, we study the effect of Hot Isostatic Pressing (HIP), homogenizing annealing, and aging in the range of 700-1100 ºC on microstructure, phase composition, and microhardness of the Ti-22Al-25Nb alloy manufactured by SLM.

2. MATERIALS AND METHODS

Spherical elemental powders of CP Ti Grade 2, Al (purity 99.9 %), Nb (purity 99.7 %) were used as the initial powders for preparing a mix of elemental powders in the composition of Ti-22Al-25Nb (at%). The elemental powder mixture had the following particle size distribution: d_{10} = 17.9 μm, d_{50} = 34.0 μm, d_{90} = 60.2 μm.

The SLM process was carried out using SLM Solutions SLM 280HL machine. The samples with the size of 10x10x10 mm^3 were manufactured at 55.6 J/mm^3 volume energy density with the previously optimized parameters allowing to obtain samples with a relative density 99.5 %.

The phase composition was analyzed with a Bruker D8 Advance X-ray diffraction (XRD) meter using Cu-Kα (λ = 0.15418 nm) irradiation.

Buehler VH1150 testing machine with 10 s dwelling time and 500 g load was used to evaluate the microhardness of the produced samples.

TESCAN Mira 3 LMU scanning electron microscope (SEM) with secondary electrons (SE) and backscattered electrons (BSE) was utilized for the microstructural characterization of the samples.

Hot isostatic pressuring (HIP) of the samples was carried out at 1050 ºC temperature and 100 MPa pressure for 3 hours using argon gas. Heat treatment of the samples consisted of annealing in vacuum at 1350 ºC for 2 h followed by approximately 4-6 ºC/s cooling and aging in vacuum at 700, 800, 900, 1000, and 1100 ºC for 24 h followed by furnace cooling.

Differential Scanning Calorimetry (DSC) was carried out using SETSYS Evolution analyzer with the heating rate of 5 ºC/min and maximum temperature of 1400 ºC in an argon atmosphere.

3. RESULTS AND DISCUSSION

To determine phase transformation temperatures in the synthesized alloy, a thermal analysis of the sample produced at 55 J/mm^3 after homogenizing annealing at 1350 ºC was conducted by way of DSC. The DSC-curve is shown in **Figure 1**. There are two exothermal (at 860 ºC and 1069 ºC) and two endothermal peaks (at 978 ºC and 1238 ºC) found on the DSC-curve. The first DSC-peak at 860 ºC corresponds to O→B2 transition. The B2 phase corresponds to the Ti-Al-Nb solid solution with the ordered body centered cubic lattice, that is typical for titanium intermetallic alloy with high Nb content. The O-phase corresponds to the Ti_2AlNb compound with the ordered orthorhombic lattice. The endothermal peak at 978 ºC corresponds to the formation of α2-phase. The exothermal peak at 1069 ºC corresponds to the dissolution of O-phase. The peak at 1238 ºC corresponds to α2-phase dissolution and transition to a single-phase B2-region. The DSC-curve peaks correspond to typical phase transitions for Ti-22Al-25Nb alloy. However, transition temperatures are shifted towards higher temperatures, which might be the result of the reduced Al content. Therefore, Nb content in the sample is relatively higher than in the powder mixture leading to increased temperature stability of B2-phase.

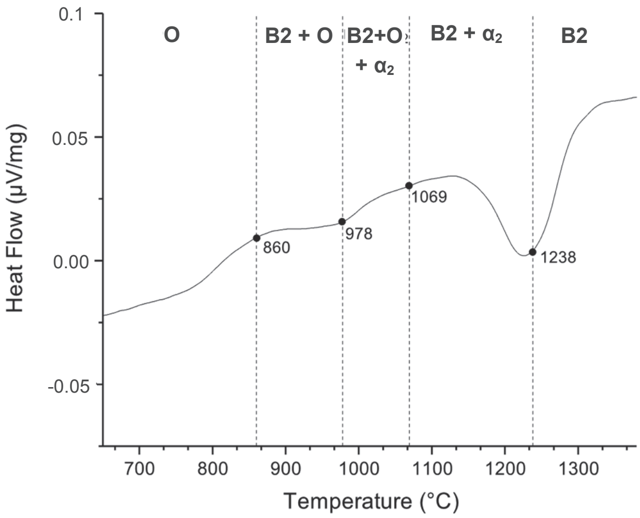

Figure 1 DSC-curve of the sample produced by SLM from Ti-22Al-25Nb elemental powder mixture

The samples obtained by SLM were HIPed at 1050 ºC, 100 MPa for 3 h. SEM-BSE-image of the microstructure and EBSD map of the sample after HIP are shown in **Figure 2**. HIP allowed to obtain almost fully-dense material by closing the residual pores. However, there are still separate undissolved Nb particles in the sample. Thus, the used HIP temperature was not high enough to promote full dissolution of Nb in the sample. However, HIP at 1050 °C followed by slow cooling resulted in the formation of secondary phase precipitates. According to the XRD results (**Figure 3**), these precipitates are α2-phase (Ti_3Al). The EBSD results (**Figure 2 b**) showed that the sample after HIP consists of equiaxed with different crystallographic orientations with a mean grain size of about 50 µm.

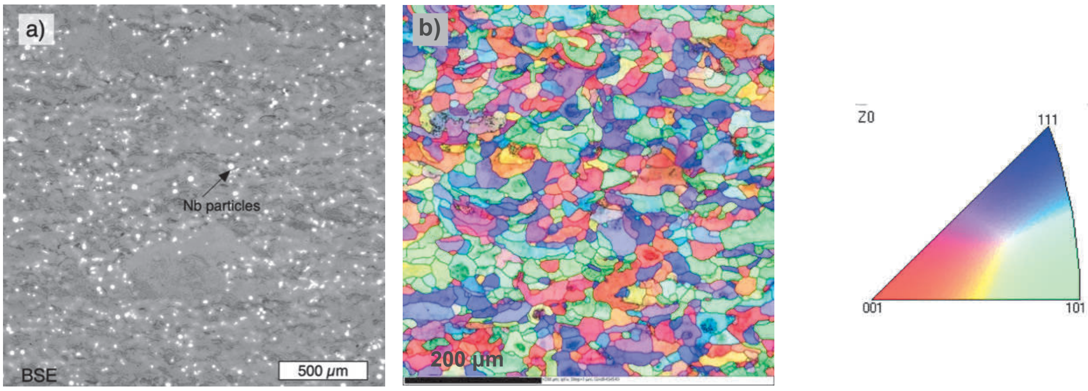

Figure 2 SEM-image of Ti-22Al-25Nb sample's microstructure after HIP (a) and inverse pole figure (IPF) map showing the grain distribution and orientation

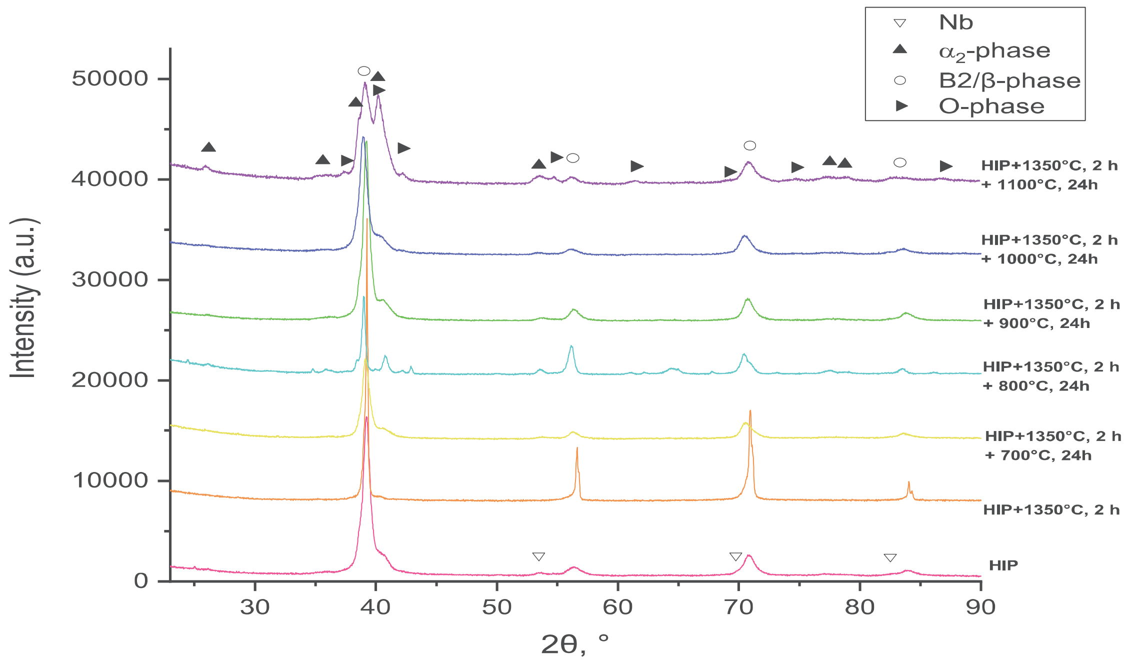

Figure 3 The XRD patterns of Ti-22Al-25Nb samples produced by SLM after HIP and various heat treatment

In order to dissolve Nb particles the HIPed samples were annealed in vacuum at 1350 °C for 2 h. Annealing at 1350 °C resulted in diffusion and dissolution of Nb particles in the alloy. The XRD pattern of the annealed sample shows only the peaks corresponding to B2-phase (**Figure 3**). No secondary precipitates were found in the microstructure, which were the results of a relatively high cooling rate (5-7 °C/s) from the annealing temperature.

To increase the strength of the alloy the amount of intermetallic orthorhombic Ti2AlNb-phase must be increased. For that purpose, the samples after annealing were aged at 700, 800, 900, 1000, 1100 °C for 24 h in vacuum. Based on the DSC-results, these temperatures correspond to different phase regions of the obtained alloy. The microstructures of the samples aged at 700-900 °C are shown in **Figure 4**. Aging at these temperatures resulted in precipitation of fine acicular O-phase of different width inside B2-grains. O-phase precipitates can also be found on the B2-grains borders. The EDX results showed that the composition of O-phase on the grain boundaries after aging at 700 °C is the following (in at. %): Ti - 59.6, Al - 23.0; Nb - 17.4.

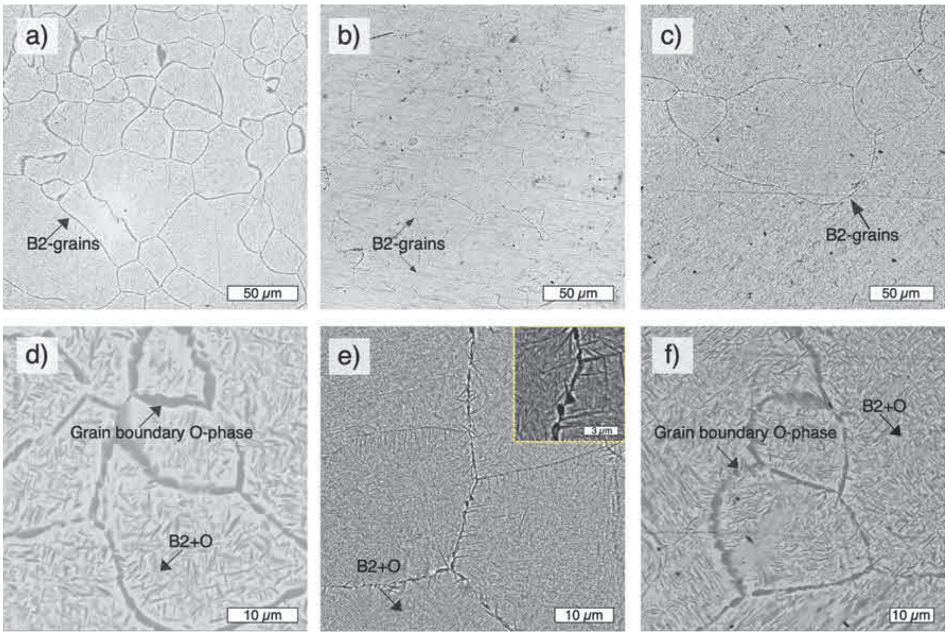

Figure 4 SEM-images of the Ti-22Al-25Nb samples after aging for 24 h at 700 °C (a, d), 800 °C (b, e), and 900 °C (c, f)

Aging at 700 °C resulted in the smallest volume fraction of O-phase, which is 34 ± 3 %. Aging at 800 °C increased the O-phase volume fraction to 46.3 % while aging at 900 °C slightly decreased the O-phase fraction to 42 ± 2 %. Aging at 800 °C resulted to the smallest width of O-phase precipitates (0.12 ± 0.04 μm). **Figure 5** shows the EBSD for the samples aged at 800 °C. The sample has equiaxed grains with different crystallographic orientations. The grains size increased to about 400-500 μm as the result of annealing and aging treatments. Aging of the alloy in (B2+O+α_2)-phase region at 1000 °C results in the formation of fine α_2-Ti_3Al precipitates inside B2-grains, while O-phase can only be found at B2-grain boundaries (**Figures 6 a, c**). Aging at 1100 °C led to the precipitation of a high number of fine α_2-Ti_3Al precipitates with the width of 1-2 μm inside B2-grains and the precipitation of O-phase (**Figures 6 b, d**). The higher amount of O-phase in case of aging at 1100 °C compared to 1000 °C can be attributed to the decomposition of α_2-phase during slow cooling in (B2+O)-region.

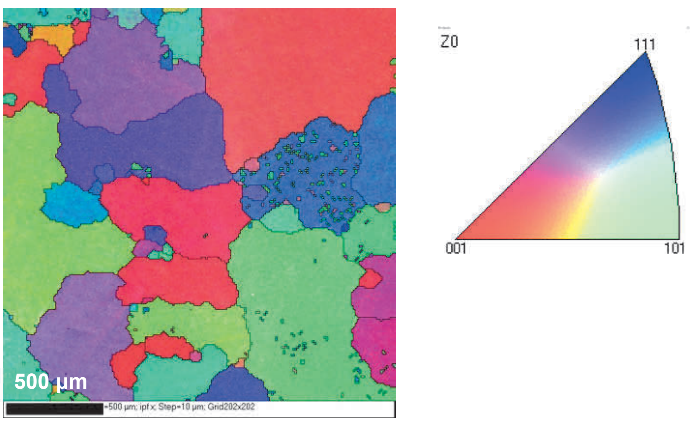

Figure 5 IPF map showing the grain distribution and orientation for the sample aged at 800 °C

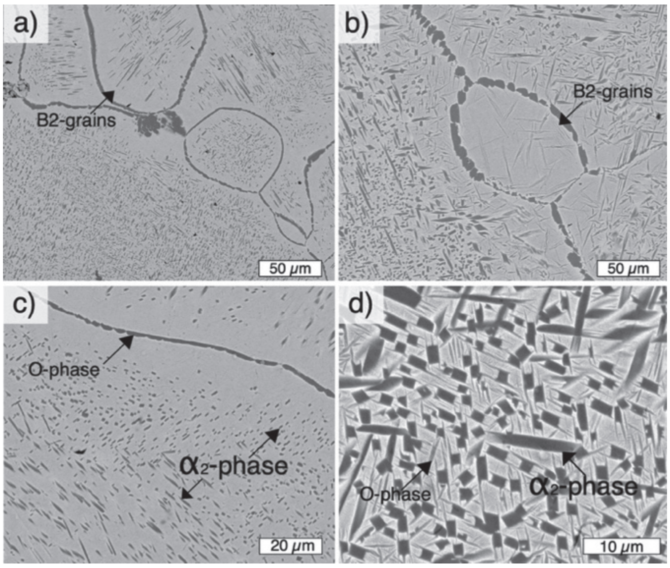

Figure 6 SEM-images of Ti-22Al-25Nb samples after aging for 24 h at 1000 ºC (a, c), and 1100 ºC (b, d)

The microhardness of the samples obtained by SLM at different conditions and after different heat-treatment is shown in **Figure 7**. Homogenizing annealing resulted in lower microhardness due to the formation of B2-phase without any strengthening secondary phase precipitates. Subsequent aging increased the microhardness. The highest microhardness was achieved after aging at 800 ºC provided that the produced alloy featured the largest amount of O-phase precipitates with the smallest width.

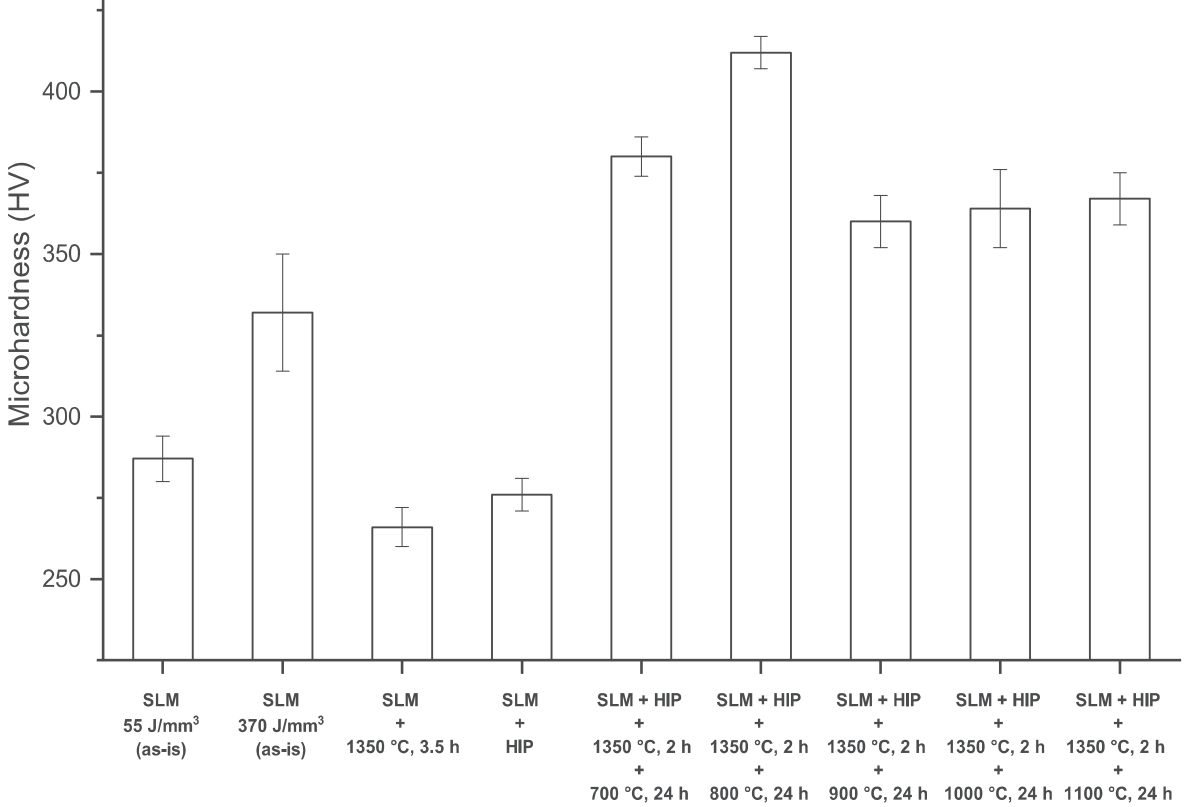

Figure 7 The microhardness of the samples obtained by SLM from Ti-22Al-25Nb elemental powder mixture at different conditions

4. CONCLUSIONS

The paper demonstrates the results of study of HIP and heat treatment effects on microstructure and properties of the Ti-22Al-25Nb alloy obtained by SLM from elemental powders. It was shown that HIP at 1050 ºC resulted in fully-dense material, while Nb particles remained undissolved. α2-Ti_3Al precipitates formed around Nb particles. Annealing at 1350 ºC for 2 h dissolved Nb particles in B2-solid solution. Aging temperature significantly affects the size and the number of secondary precipitates. The highest volume fraction of O-phase precipitates having the smallest size was obtained after aging at 800 ºC in (B2+O)-phase region. Heat treatment conditions significantly affect the microhardness of the alloy. The highest microhardness of the alloy was achieved after aging at 800 ºC.

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