

Article

Processing, Microstructure, and Mechanical Properties of Laser Additive Manufactured Ti₂AlNb-Based Alloy with Carbon, Boron, and Yttrium Microalloying

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Abstract: In this work, Ti-22Al-23Nb-0.8Mo-0.3Si-0.4C-0.1B-0.2Y (at. %) alloy powder was used to fabricate the Ti₂AlNb-based alloy samples using Laser powder bed fusion (L-PBF) Additive Manufacturing with a high-temperature substrate preheating. L-PBF process parameters, including laser power, scan speed, hatching distance, and preheating temperature, allowing for obtaining fully dense (99.9% relative density) crack-free samples, were determined. The effects of substrate preheating temperature during the L-PBF process on microstructure, phase composition, and properties of the obtained Ti₂AlNb-based alloy were investigated using X-ray diffraction, scanning electron microscopy, electron backscatter diffraction analysis, and microhardness testing. The results obtained for the material with C, B, and Y microalloying were compared to the Ti₂AlNb-based alloy fabricated by L-PBF from the powder not alloyed with C, B, and Y. The results revealed that the microalloying reduced the number of solidification cracks; however, no significant microstructural changes were observed, and high-temperature substrate preheating was still necessary to suppress cold cracking of the alloy. The microstructure of the alloy varied from fully-β/B2, B2 + O, to fully-O depending on the preheating temperature. Effects of hot isostatic pressing and heat treatment conditions on microstructure and mechanical properties were investigated.

Keywords: additive manufacturing; powder bed fusion; titanium aluminides; microalloying; microstructure; Ti-22Al-25Nb



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1. Introduction

Advanced titanium intermetallic alloys based on titanium aluminides are of great interest to aviation, aerospace, and automotive industries due to their high specific strength, heat resistance, and the potential to replace heavy Ni-based superalloys [1,2]. Orthorhombic Ti₂AlNb-based (O-phase) alloys have shown great potential for their high-temperature application due to improved ductility compared to Ti₃Al- and TiAl-based alloys [3–5].

Traditional ways of producing Ti₂AlNb-based alloys involving casting and thermo-mechanical processing involve multiple labor-consuming stages significantly increasing the cost of intermetallic parts [6,7]. Moreover, poor machinability and brittleness of orthorhombic alloys limit the geometrical complexity of the final product. Powder metallurgy methods, such as hot isostatic pressing (HIP) or spark plasma sintering, have been demonstrated to be of possible use to fabricate Ti₂AlNb-alloy components [8,9]. However, these methods have severe limitations in terms of geometry complexity, adequate cost, and decent level of mechanical properties [10].

Welding of intermetallic titanium alloys using laser or electron beam welding remains challenging due to their proneness to cracking [11,12]. Controlling the cooling rate during the welding process or applying post-weld heat treatment are typical ways to prevent cold cracking of titanium aluminide alloys during the welding.

Additive Manufacturing (AM) is a promising method for producing metal parts with a high degree of geometrical complexity and high level of mechanical properties [13]. Selective Laser Melting (SLM), also known as laser powder bed fusion (L-PBF), is a widely used AM technique to fabricate parts from metal alloys powders using laser beam as an energy source [14,15].

The susceptibility to cracking, brittleness, and limited weldability of titanium aluminide alloys make it hard to produce defect-free intermetallic parts using the L-PBF process [16,17]. In the previous research [18], the authors showed that preheating the substrate above 600 °C during the L-PBF process is necessary to obtain crack-free Ti₂AlNb-based Ti-24Al-25Nb-1Zr-1.4V-0.6Mo-0.3Si (at. %) alloy. While high-temperature substrate preheating allowed to successfully suppress cold cracking of the alloy and achieve good room- and high-temperature strength values after subsequent HIP treatment [19], the alloy exhibited poor plasticity, which might be attributed to the non-optimal microstructure and oxygen pickup during the L-PBF process.

One of the possible ways to improve the properties and AM processability of titanium aluminide alloys is to tailor the chemical composition of the feedstock powder [20].

Ti-22Al-25Nb (at. %) alloy is considered a base orthorhombic alloy and is one of the most investigated Ti₂AlNb-based alloys [21]. Additional alloying elements are usually added to improve properties of the alloy. For example, a combination of Mo and Si is added to improve creep strength and oxidation resistance of the material [22–24]. The addition of several atomic percent of other β -stabilizing elements, such as V, Hf, Ta, and W, is often used to increase the β /B2 phase-field resulting in improved workability of the alloy but an increased density [25,26].

The titanium aluminide alloys are currently used in many studies of AM processability research [18,27,28], which were originally developed for conventional processes such as casting and hot working. This results in issues concerning solidification cracking, impurities pickup, grain boundaries embrittlement, and texture and negatively affects the AM processability and mechanical properties of the alloys. Thus, a Ti₂AlNb-based alloy with a tailored chemical composition has been proposed in the current work to use in the L-PBF process with high-temperature substrate preheating. Based on the experimental results and literature data on titanium aluminide alloys design, the addition of several alloying elements to the base Ti-22Al-25Nb composition has been proposed and experimentally evaluated.

As mentioned earlier, Mo and Si are usually added simultaneously to enhance the oxidation resistance and creep strength [22] of the Ti-22Al-25Nb alloy by lowering the diffusion rate, introducing silicides, and forming beneficial protective oxides [29]. Alloying titanium aluminides with small amounts of Si and C results in improved tensile strength and creep resistance and leads to higher operating temperatures [20]. Microalloying with Si and C can result in solid solution strengthening or precipitation strengthening by forming secondary silicides and carbides, which would decrease dislocation mobility and stabilize microstructure at elevated temperatures [20,30].

Boron is added in small amounts to introduce boride precipitates during the crystallization that further act as nucleation sites and result in refined primary β -phase grains [20,30] and thus an enhanced ductility [31]. As shown in [32] for the Ti-22Al-20Nb-2W alloy, the addition of 0.2 (at. %) of B improved the alloy's room temperature ductility as well as creep behavior.

Another promising way of improving the mechanical properties of titanium alloys is adding small amounts of rare-earth elements [33–37]. Ti-based and titanium aluminide alloys are usually alloyed with rare-earth elements, such as Y and Gd [33,34,38]. Sc, Ce, and Er are used less frequently [36]. Rare-earth elements form refractory compounds with oxygen and nitrogen that promote grain refinement, and slow diffusion processes, and result in an improved microstructural thermal stability. Microalloying titanium alloys with rare-earth elements is also an effective way of scavenging oxygen at grain boundaries in titanium alloys, which results in enhanced ductility. For example, an addition of 0.4–0.8 (at. %)

Er to Ti-22Al-25Nb alloy resulted in a refined microstructure due to the formation of Er_2O_3 oxides and Al_3Er precipitates acting as heterogeneous nuclei [36]. Microalloying of Ti-22.5Al-23.5Nb alloy with 0.1 (at. %) Gd improved both room- and high-temperature tensile strength and ductility [33]. Addition of Y to Ti-22Al-25Nb alloy improved its grain size stability at elevated temperature [37], while a small amount of Y to Ti-47Al alloy led to grain refinement and improved tensile properties [38]. Y is a reasonable choice among the rare-earth elements to mitigate the oxygen content as it has a high oxygen scavenging capability and relatively low cost [39,40].

Based on this knowledge, the study presents a Ti_2AlNb -alloy with a tailored chemical composition microalloyed with carbon, boron, and yttrium. The resultant alloy had the following composition (in at. %): Ti-22Al-23Nb-0.8Mo-0.3Si-0.4C-0.1B-0.2Y. Gas atomized powder of this alloy was used in the L-PBF process to fabricate the samples for microstructural analysis and mechanical properties evaluation. The results were compared to the Ti_2AlNb -based alloy obtained by the L-PBF using the atomized powder with the composition originally developed for conventional processing by hot working [41,42].

2. Materials and Methods

2.1. Feedstock Powder

Ti-22Al-23Nb-0.8Mo-0.3Si-0.4C-0.1B-0.2Y alloy spherical powder obtained by electrode induction gas atomization (EIGA) method with a mean particle size $d_{50} = 33 \mu\text{m}$ was used in the L-PBF process. Before the atomization process, the alloy was microalloyed with carbon, boron, and yttrium and will be further referred to as Ti_2AlNb -CYB powder. The resultant chemical composition of the powder after gas atomization is presented in Table 1. As can be seen in Figure 1, the particles mostly have a spherical shape and dendritic microstructure typical for gas atomized powders. The X-ray diffraction (XRD) results (Figure 1c) showed that the Ti_2AlNb -CYB powder consists of a single phase corresponding to the $\beta/\text{B2}$ phase. This is in agreement with the previously reported results for Ti-22Al-25Nb alloys [10,43]. Precipitation of the secondary phase is inhibited during the atomization process due to high cooling rates typical for gas atomization.

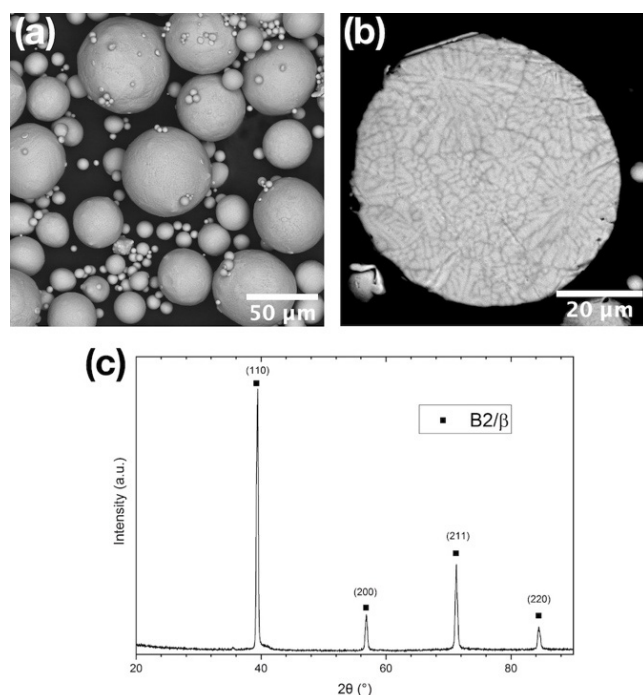


Figure 1. SEM images of the Ti_2AlNb -CYB powder showing (a) particles' surface morphology and (b) their cross-section. The XRD pattern of the powder (c).

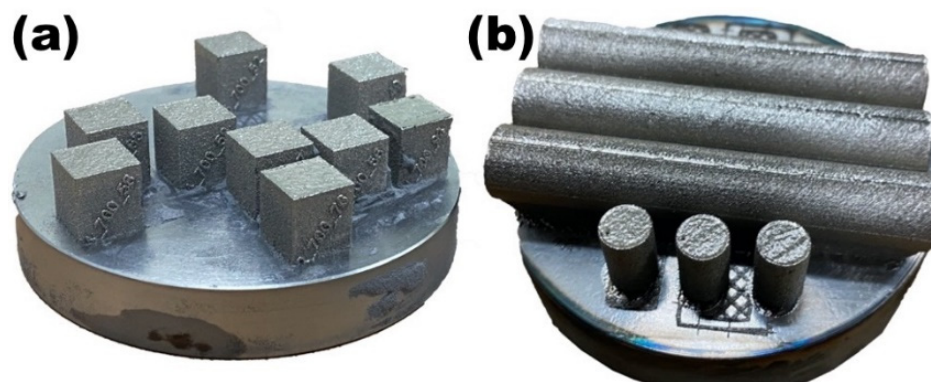
Table 1. Chemical composition of the Ti₂AlNb-CYB powder (in at. %).

Element	Ti	Al	Nb	Mo	Si	Y	B	C	O, wt. %	N, wt. %
Method	ICP-OES						C/S analyzer		O/N/H analyzer	
Content	Bal.	22.6	23.32	0.83	0.31	0.55	0.12	0.20	0.082	0.016

For comparison of microstructures and mechanical properties, a similar titanium orthorhombic alloy powder with Ti-24Al-25Nb-1Zr-1.4V-0.6Mo-0.3Si (at. %) nominal composition and without boron, yttrium, and carbon microalloying was used to fabricate the samples using the L-PBF process. This powder was used in the previous research [18] to evaluate optimal L-PBF parameters with high-temperature substrate preheating allowing for obtaining crack-free fully-dense samples. The powder was produced by the same supplier as the Ti₂AlNb-CYB powder; however, no microalloying was used. The powder without microalloying will be further referred to as Ti₂AlNb-noCYB powder. A detailed characterization of the Ti₂AlNb-noCYB powder can be found in [18].

2.2. Laser Powder-Bed Fusion

A L-PBF AconityMIDI (Aconity3D GmbH, Herzogenrath, Germany) system was used to manufacture the intermetallic alloy samples for further investigations. Cubic 10 × 10 × 10 mm³ samples were produced for microstructural investigation as well as defect analysis (Figure 2a). The L-PBF system is equipped with an inductive preheating module allowing for preheating the substrate up to 1200 °C. The Ti-6Al-4V alloy substrate was used in the study, and it was preheated to the specific temperature before starting the laser processing [19]. The L-PBF process was carried out under a protective argon atmosphere to prevent the material from oxidation.

**Figure 2.** The samples fabricated by L-PBF for (a) microstructural characterization and (b) tensile tests.

Based on the preliminary results obtained for the Ti₂AlNb-noCYB powder, several sets of L-PBF process parameters with varied substrate preheating temperature, scanning speed (SS), and hatching distance (HD) were chosen for manufacturing of the samples (see Table 2). Laser powder (LP) and layer thickness (L) were fixed. The volume energy density (VED) (Equation (1)) varied from 49 to 93 J/mm³. The platform preheating temperature (T_{PH}) was 300, 700, and 850 °C:

$$VED = \frac{LP}{SS \cdot HD \cdot L} \left[\frac{J}{mm^3} \right] \quad (1)$$

Table 2. The process parameters used for manufacturing of the samples.

#	LP, W	SS, mm/s	HD, mm	L, mm	VED, J/mm ³	T _{PH} , °C
1	140	500	100	30	93	300, 700, 850
2	140	600	100	30	78	
3	140	700	100	30	67	
4	140	800	100	30	58	
5	140	900	100	30	52	
6	140	600	120	30	65	
7	140	700	120	30	56	
8	140	800	120	30	49	

Horizontal cylindrical samples (12 mm diameter and 63 mm length) with solid supports (Figure 2b) were fabricated for the tensile tests. After the fabrication process, the samples were machined to achieve a dog-bone shape with 20 mm gauge length and 4 mm diameter by ISO 6892-1. Three samples per point were tested.

2.3. Characterization

Relative density of the fabricated specimens was evaluated using a metallographic method by taking optical images of the polished samples' surfaces and processing the images using ImageJ 1.53 software by calculating the area of pores relative to the whole area.

Mira 3 (TESCAN, Brno, Czech Republic) scanning electron microscope (SEM) in backscattered electrons (BSE) mode was utilized to study the microstructure using polished sections of the specimens. An electron backscatter diffraction (EBSD) module with a step size of 0.5 µm was used to analyze phase features.

A Bruker D8 Advance X-ray diffraction (XRD) meter (Bruker, Bremen, Germany) with Cu-Kα ($\lambda = 1.5418 \text{ \AA}$) irradiation was used to analyze phase composition of the samples.

A LECO TC-500 analyzer was used to evaluate oxygen and nitrogen content in the samples by the inert-gas fusion-infrared method.

Phase transformation temperatures were analyzed using a 404 Pegasus Netzch (Selb, Germany) differential scanning calorimetry (DSC) analyzer.

HIP was conducted at 1160 °C, 160 MPa pressure, and 3 h holding time.

Heat treatment was carried out using a vacuum furnace and a muffle furnace under the following conditions:

- (1) vacuum solution annealing (SA) the samples at 1015 °C for 1.5 h followed by furnace cooling (FC);
- (2) vacuum SA the samples at 1015 °C for 1.5 h followed by FC to room temperature and subsequent aging at 800 °C for 6 h followed by FC;
- (3) SA at 1015 °C for 1.5 h followed by water cooling (WC);
- (4) SA at 1015 for 1.5 h followed by WC and subsequent aging at 800 °C for 6 h followed by FC.

Tensile tests were carried out using a Z100 testing machine (Zwick/Roell, Ulm, Germany). The tensile direction was perpendicular to the BD. Three tensile coupons per point were used to evaluate average values.

3. Results and Discussion

3.1. L-PBF Processability

Figures 3 and 4 show the effects of substrate preheating temperature during the L-PBF process on crack susceptibility and relative density of the samples. As can be seen in Figure 3a, using a low substrate preheating temperature of 300 °C resulted in severe cracking of the samples. The cracks formed from the side surfaces and are mostly transverse with a length up to several millimeters. High thermal stresses during the L-PBF process coupled with the brittleness of the Ti₂AlNb-alloys led to cracking of the sample indicating that 300 °C preheating temperature was not sufficient to reduce the thermal stresses. Similar results were obtained for the samples fabricated using the Ti₂AlNb-noCYB powder [18], suggesting that the microalloying of Ti₂AlNb-alloy could not prevent the severe cracking during the L-PBF at low substrate preheating temperatures. When the preheating tem-

perature was increased to 700 °C, mostly crack-free samples were obtained (Figure 3b). However, some small occasional surface cracks still could be found in the case of 700 °C preheating temperature. Further increasing the preheating temperature to 850 °C prevented the cracking as can be seen in Figure 3c and allowed to obtain crack-free Ti_2AlNb -alloy samples. Thus, the samples fabricated using 700 °C and 850 °C preheating temperatures were further used for porosity evaluation. Cold cracking of titanium aluminide alloys has been observed for various welding techniques [41,44–46]. For example, laser beam welding of Ti_2AlNb -based joints resulted in the formation of transverse cracks due to high residual stresses; however, preheating the joints above the brittle-to-ductile transition temperature reduced the cooling rate after welding and eliminated the crack formation [41].

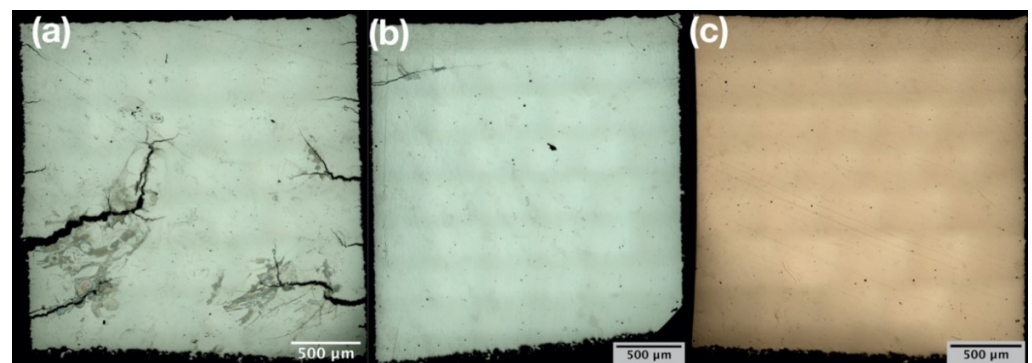


Figure 3. Typical OM images of the polished samples fabricated from the Ti_2AlNb -CYB powder showing (a) severe cracking in case of 300 °C, (b) small occasional cracks in case of 700 °C, and (c) no cracking in case of 850 °C preheating temperature.

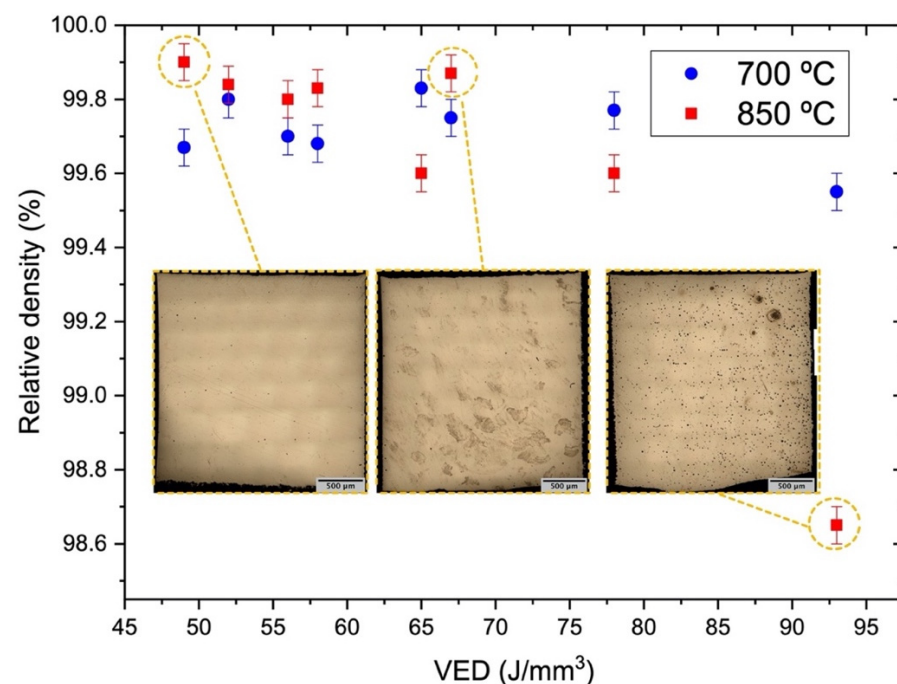


Figure 4. Effect of VED and substrate preheating temperature on the relative density of the samples fabricated from the Ti_2AlNb -CYB powder. The insets show the representative OM images of the polished samples corresponding to different VED values.

As shown in Figure 4, the fully-dense samples with a relative density of $99.9 \pm 0.1\%$ were fabricated using VED of 49 J/mm^3 and 850 °C preheating temperature. Applying high VED values resulted in an increased porosity due to the formation of spherical pores. This effect was specifically visible in the case of a higher preheating temperature. For example,

$98.6 \pm 0.1\%$ relative density was achieved using 93 J/mm^3 VED at 850°C preheating temperature, while applying the same VED at 700°C preheating resulted in $99.5 \pm 0.1\%$ relative density. This can be attributed to local overheating of the melt pool during the L-PBF process and partial evaporation of the alloy, which would result in the formation of spherical gas pores [47,48].

As shown in Figure 5a, when the $\text{Ti}_2\text{AlNb-noCYB}$ powder was used to fabricate the samples at 700°C preheating temperature, some microcracks could be found in the samples. These microcracks have a length of approximately $50 \mu\text{m}$ and are located at the prior $\beta/\text{B2}$ -phase grain boundaries and parallel to the building direction. In contrast, no microcracks were observed in the case of the $\text{Ti}_2\text{AlNb-CYB}$ powder (Figure 5b) for the samples produced using the same preheating temperature. During the L-PBF of Ti_2AlNb -alloys at high preheating temperatures, brittle intermetallic O-phase (Ti_2AlNb) precipitates at the $\beta/\text{B2}$ grain boundaries [18]. Precipitation of the O-phase at the prior $\beta/\text{B2}$ grain boundaries has also been observed for conventionally produced Ti_2AlNb -based alloys during heat treatment with the O-phase rim's thickness increasing with the temperature and/or holding time [49–51]. Prior $\beta/\text{B2}$ grain boundaries can act as nucleation sites for the O-phase promoting the formation of the grain boundary O-phase rim. Moreover, even though the L-PBF process is carried out under an inert atmosphere, some oxygen pickup by the alloy is still possible due to high temperatures. It is also known that the diffusion of oxygen at the phase boundary is faster as a result of the presence of phase/grain boundary energy [52]. This results in increased oxygen content in the grain boundary O-phase leading to its further embrittlement. Under the residual stresses during the L-PBF process, crack formation of the material occurs.

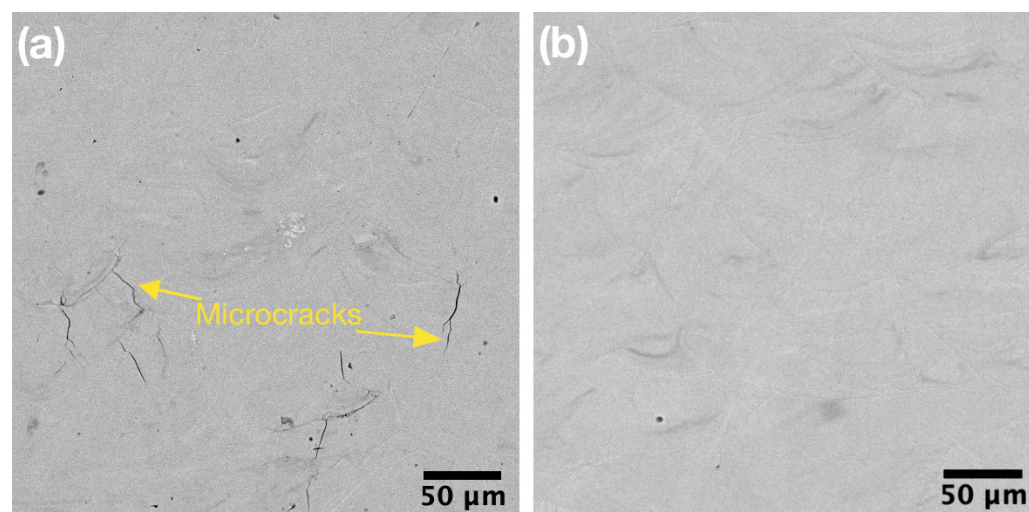


Figure 5. BSE-SEM images of the samples produced from (a) $\text{Ti}_2\text{AlNb-noCYB}$ and (b) $\text{Ti}_2\text{AlNb-CYB}$ powders at 700°C preheating temperature showing the presence of microcracks in the case of the $\text{Ti}_2\text{AlNb-noCYB}$ powder.

At the same time, the addition of rare earth elements either as a powder modifier or an alloying element has been shown to be beneficial in eliminating cracks during the L-PBF of various alloys [53–55]. Yttrium can act as an effective oxygen scavenger during both gas atomization and L-PBF processes resulting in reduced oxygen content in the alloy. As can be seen in Table 3, the microalloying of the powder significantly reduced the oxygen content in both the initial powder and the fabricated alloy. It can be noted that, while the oxygen in the sample produced by the L-PBF process increased in the case of $\text{Ti}_2\text{AlNb-noCYB}$ powder, using the $\text{Ti}_2\text{AlNb-CYB}$ powder resulted in almost the same oxygen level in the fabricated alloy, which is notably lower compared to the alloy without microalloying. It is known that an oxygen level of 1000–2000 ppm has a detrimental effect on the processability of titanium aluminide alloys. In addition, yttrium oxides generated during the processing of the alloy

can have a pinning effect on the grain boundaries preventing grain coarsening and improve ductility [55,56]. Additionally, introduction of yttrium can result in the formation of nano-sized oxides inside the β , O grains. These oxides might act as pinning centers for the defects and impede dislocation movement enhancing strength and ductility of the material [57]. As can also be seen from Table 3, the N content of the L-PBF alloys decreases compared to the as-atomized powders. This might be attributed to partial nitrogen evaporation during the laser melting in argon atmosphere as discussed in [58–60].

Table 3. Oxygen and nitrogen content in the initial powders and fabricated samples.

Sample	O, wt. %	N, wt. %
Ti ₂ AlNb-noCYB powder	0.137	0.020
Ti ₂ AlNb-CYB powder	0.082	0.016
Ti ₂ AlNb-noCYB (L-PBF)	0.200	0.003
Ti ₂ AlNb-CYB (L-PBF)	0.085	0.002

Thus, the presence of yttrium in the Ti₂AlNb-CYB powder as a microalloying element improved the processability of the alloy by L-PBF with high-temperature substrate preheating and eliminated grain boundary microcracks.

3.2. Microstructure and Phase Composition

Figure 6 shows microstructures of the samples fabricated from the Ti₂AlNb-CYB powder using different substrate preheating temperatures. Using a relatively low preheating temperature of 300 °C resulted in a single-phase β /B2 microstructure (Figure 6a) suggesting that precipitation of secondary phases was suppressed due to high cooling rates during the L-PBF process and the preheating temperature of 300 °C was not enough to promote the precipitation of Ti₂AlNb O-phase. The XRD results shown in Figure 6 confirmed that the samples fabricated using 300 °C preheating temperature consists only of the β /B2 phase.

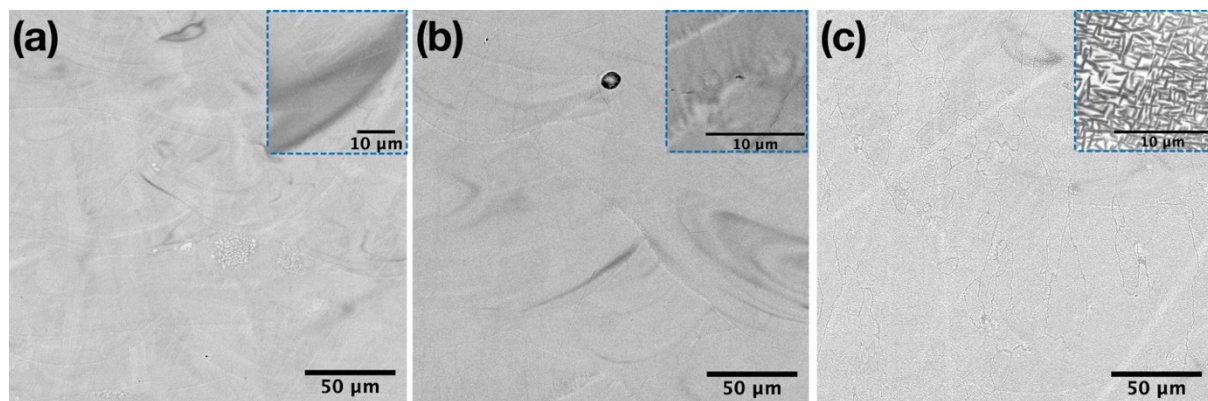


Figure 6. Microstructures of the samples fabricated from the Ti₂AlNb-CYB powder at (a) 300 °C, (b) 700 °C, and (c) 850 °C substrate preheating temperature. Insets show microstructures of the corresponding samples at higher magnifications.

Crescent-shaped melt pool boundaries typical for the L-PBF process could be seen in the BSE-SEM image (Figure 6a) showing a melt pool width of approximately 100–120 µm, which is slightly bigger than the laser spot size. The preheating temperature of 700 °C resulted in an almost fully-O microstructure in accordance with BSE-SEM images (Figure 6b) and the XRD results (Figure 7). Small amounts of residual β -phase could form near melt pool boundaries similar to the Ti₂AlNb-based alloy fabricated from the Ti₂AlNb-noCYB powder at 700 °C preheating temperature [18].

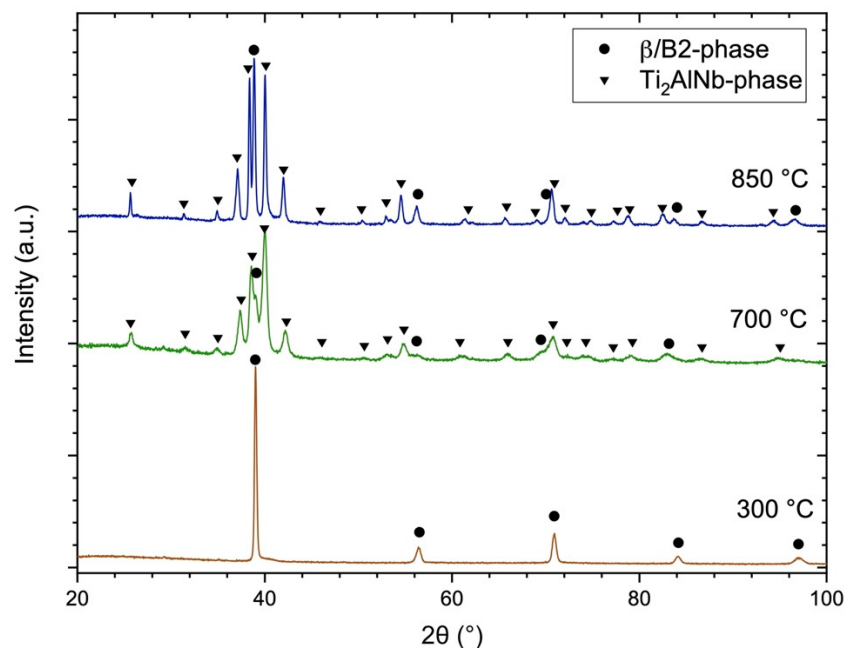


Figure 7. XRD patterns of the samples produced from the Ti_2AlNb -CYB powder at 300, 700, and 850 °C substrate preheating temperatures in the as-fabricated condition.

The BSE image of the sample also shows that there are dark bands near some melt pool boundaries indicating that there are some chemical inhomogeneities in the produced alloy. According to the EDS-mapping results shown in Figure 8, these dark bands correspond to Nb-lean and Ti-rich areas. These micro-inhomogeneities could result from solidification segregation during the L-PBF process. According to the Ti-22Al-xNb phase diagram [61], a decrease in Nb content might result in the formation of α_2 (Ti_3Al) phase and reduced B2-phase content. However, due to the small size and fraction of these areas, the XRD results did not show the peaks corresponding to the α_2 phase. At the same time, as can be seen from Figure 6, an increased preheating temperature led to a more homogeneous microstructure due to an increased diffusion rate at higher temperatures.

Further increasing the preheating temperature in the B2 + O region to 850 °C, which is closer to the single B2 phase region, resulted in an increased β /B2 phase volume fraction and the formation of B2 + O microstructure as can be seen in Figure 6c. The estimated volume fraction of the O-phase in the case of 850 °C preheating temperature was approximately 50%. The solidification of the alloy during the L-PBF process takes place through the B2/ β region with the formation of primary B2/ β grains. The subsequent holding of the alloy at the preheating temperature coupled with additional local heating by the laser exposure promoted the participation of the acicular O-phase inside the B2/ β grains as well as the rim O-phase along the grain boundaries due to its continuous precipitation. According to the DSC results shown in Figure 9, the 850 °C preheating temperature corresponds to the B2 + O region. Additional possible heating of the material into the α_2 + B2 + O or α_2 + B2 region during the laser exposure did not promote the formation of α_2 phase due to the long time required for its formation. It can also be noted that, according to the DSC results for the alloy obtained from the Ti_2AlNb -CYB powder, no significant changes in phase transformation temperatures were observed compared to alloy without microalloying. In general, the microstructure morphologies and phase compositions of the samples obtained from the Ti_2AlNb -CYB powder are similar to the alloys fabricated using the Ti_2AlNb -noCYB powder at the corresponding preheating temperatures [18].

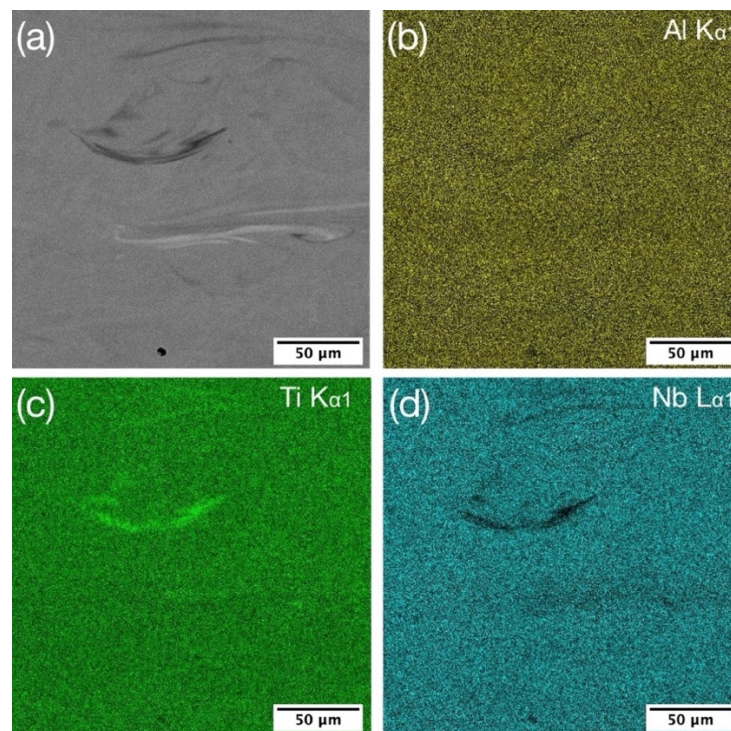


Figure 8. EDS maps of the sample produced from the Ti_2AlNb -CYB powder at 300 °C substrate preheating temperature: (a) BSE-SEM image, distribution of (b) Al, (c) Ti, and (d) Nb.

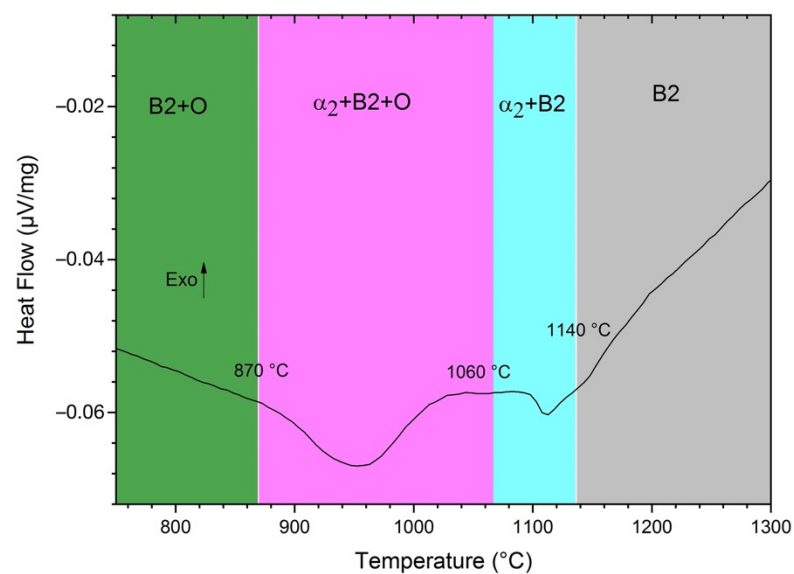


Figure 9. DSC curve of the sample fabricated from the Ti_2AlNb -CYB powder.

Figure 10 shows the inverse pole figure (IPF) maps of the as-fabricated alloys using the microalloyed powder and the powder without additions of carbon, yttrium, or boron. It can be seen that both alloys have columnar β /B2 grains along the BD. The alloy obtained from the Ti_2AlNb -CYB powder (Figure 10a) features refined β /B2 grains compared to the alloy without microalloying (average grain size of 31 μm and 48 μm for Ti_2AlNb -CYB and Ti_2AlNb -noCYB, respectively).

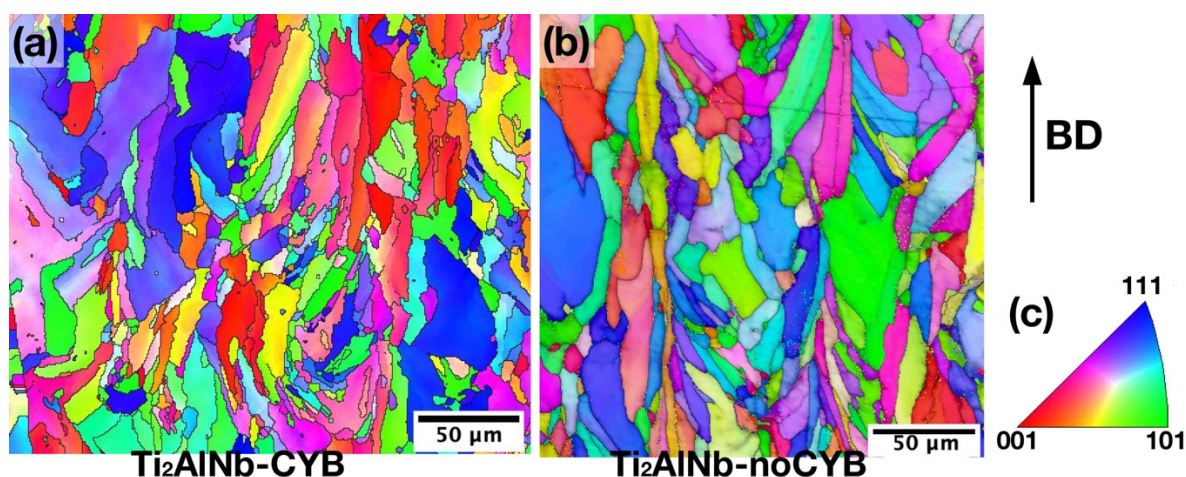


Figure 10. EBSD orientation maps along the BD of the samples fabricated at 300 °C preheating temperature from (a) the Ti_2AlNb -CYB and (b) the Ti_2AlNb -noCYB powders, (c) inverse pole figure of β -phase.

Grain refinement by the addition of rare-earth elements has been known as an effective method to reduce the cracking of additively manufactured alloys [34,54,56,62]. Firstly, the addition of rare-earth elements is associated with the reduction of oxygen content at the grain boundaries enhancing the ductility of the alloy. Secondly, grain refinement leads to a larger grain boundary area per unit volume reducing the accumulation of thermal stresses as demonstrated in [62]. As a result, the microalloying of the Ti_2AlNb -based alloy powder resulted in a reduced possibility of cracking formation during the L-PBF process.

3.3. Heat Treatment and Hot Isostatic Pressing Effects on Microstructure

The Ti_2AlNb -alloy samples fabricated by L-PBF using 850 °C preheating temperature were used to investigate the effects of heat treatment and HIP on the microstructure of the alloy.

Figure 11 shows the microstructure of the Ti_2AlNb alloy samples after HIP. For both Ti_2AlNb -CYB and Ti_2AlNb -noCYB alloys, HIP resulted in dual-phase B2 + O microstructure consisting of equiaxed prior B2 grains and fine lamellar O phase precipitates within the B2 grains. B2 grain boundaries are characterized by the presence of rim O phase in case of both alloys. The main difference between the microstructures of the alloys after HIP is a considerably smaller grain size in the case of the Ti_2AlNb -CYB alloy ($110 \pm 40 \mu\text{m}$ and $70 \pm 50 \mu\text{m}$ for Ti_2AlNb -noCYB and Ti_2AlNb -CYB alloys, respectively). Thus, microalloying resulted in reduced grain size after HIP. This can be attributed to the formation of submicron and nanosized secondary precipitates in the case of the microalloyed sample which prohibits grain growth during HIP or heat treatment. It should also be noted that, after HIP, the residual porosity in the sample was mostly eliminated and only a few occasional micropores could be found by the microscopic evaluation.

Figure 12 shows the microstructures of the Ti_2AlNb -CYB alloy after various heat treatments. After SA followed by FC without preceding HIP (Figure 12a), the microstructure of the alloy consisted of B2 and O phases similar to the as-fabricated condition. However, the annealing resulted in an increased B2 phase content due to the decomposition of the O phase and, correspondingly, a decreased O phase fraction. Specifically, SA followed by FC resulted in a decreased O phase fraction from approximately 50% to 40%. At the same time, the acicular O phase precipitates inside the B2 grains and grain boundary rim O phase visibly increased in thickness after annealing and aging heat treatment. Additionally, the subsequent aging (Figure 12b) resulted in an increased O phase fraction to approximately 50% compared to the annealed condition. While the annealing temperature of 1015 °C corresponds to the $\alpha_2 + \text{B2} + \text{O}$ phase region according to the DSC results, the α_2 phase precipitates were not identified in the annealing alloy. This might be explained by a

relatively short annealing time, while the precipitation of the α_2 phase as a result of B2 phase decomposition requires long holding times due to the slow diffusivity of Nb in titanium orthorhombic alloys [63].

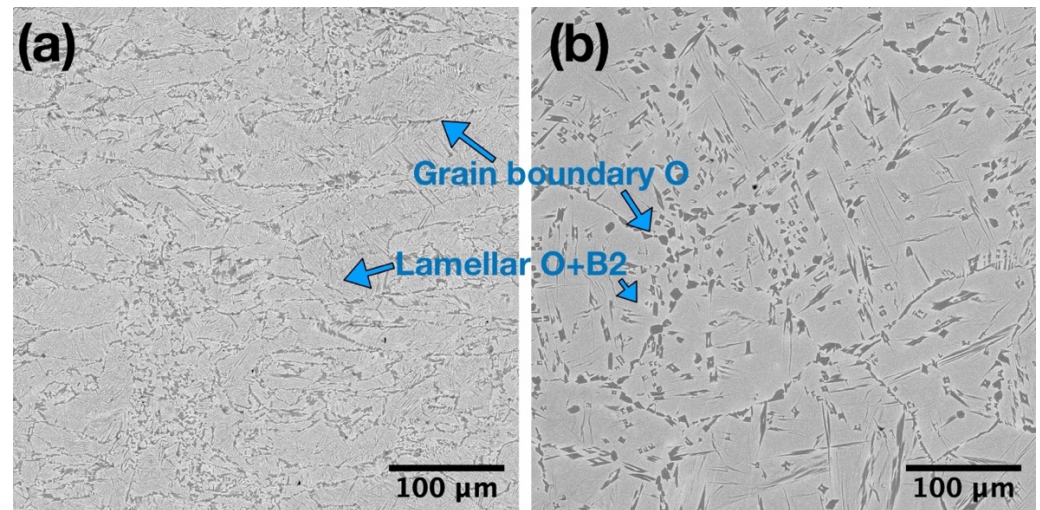


Figure 11. Microstructure of the samples produced from (a) the Ti_2AlNb -CYB and (b) the Ti_2AlNb -noCYB powders after HIP.

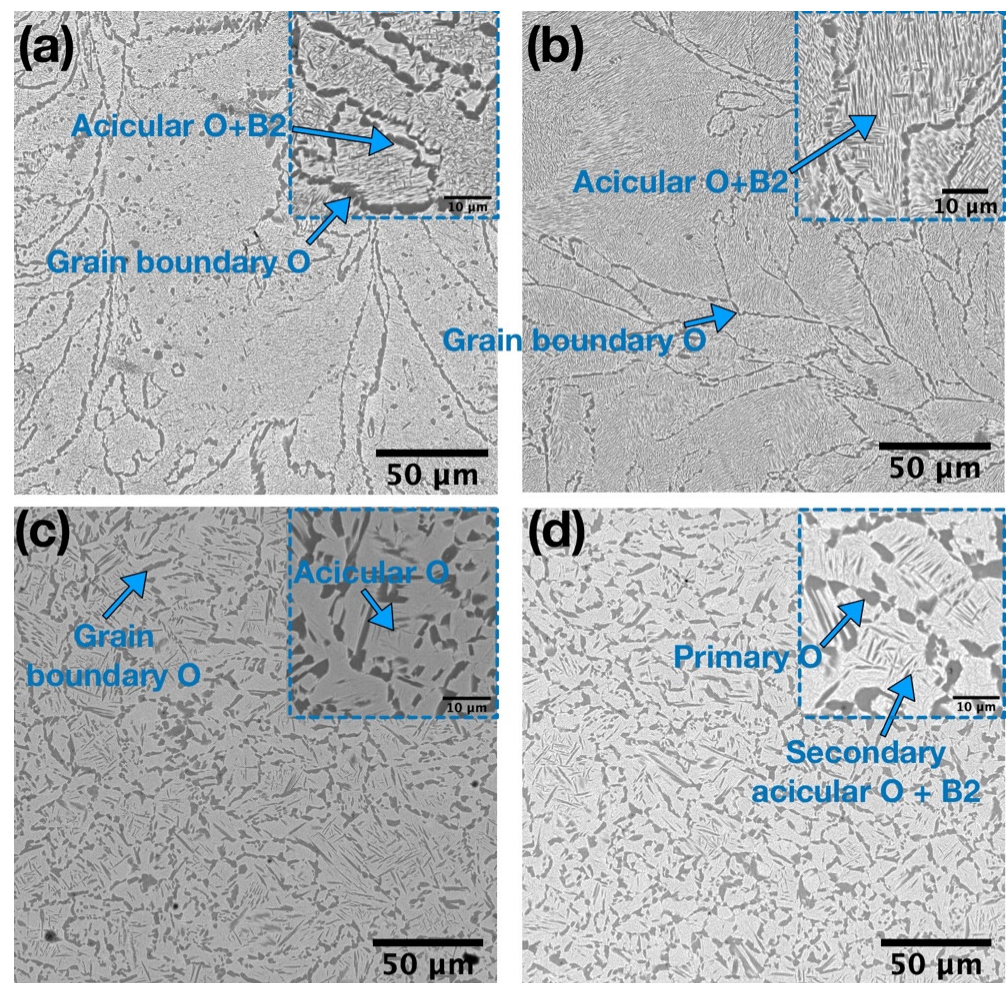


Figure 12. Cont.

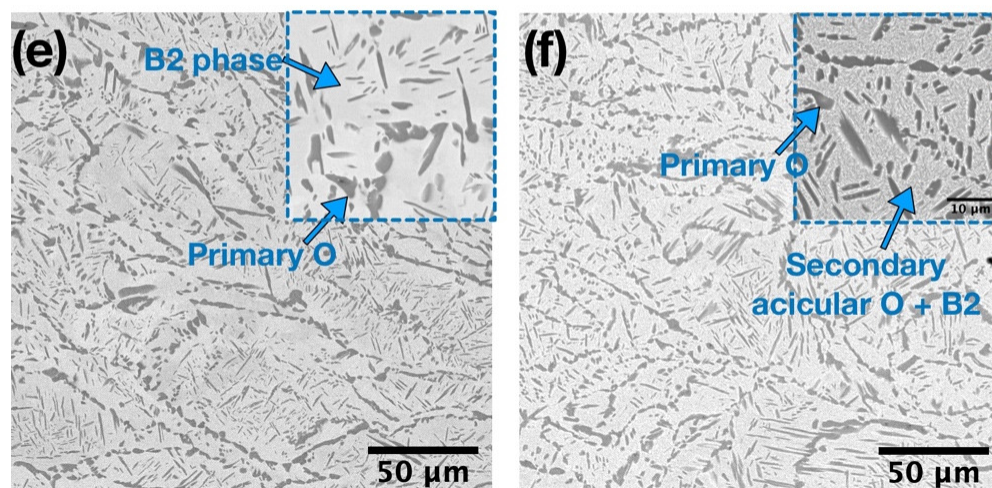


Figure 12. Microstructures of the as-fabricated samples after heat treatment: (a) SA followed by FC, (b) SA followed by FC plus aging. Microstructures of the HIPed samples after heat treatment: (c) SA followed by FC, (d) SA followed by FC plus aging, (e) SA followed by WC, (f) SA followed by WC plus aging. Insets show microstructures of the corresponding samples at higher magnifications.

HIPing of the alloy prior to SA (FC) resulted in a decreased number of acicular O phase precipitates within the primary B2 grains (Figure 12c) compared to the annealed non-HIPed alloy. At the same time, the grain boundary O phase transformed from continuous to discontinuous precipitates. The HIP temperature of 1160 °C corresponds to the B2 phase field, while the SA temperature is 1020 °C corresponding to the $\alpha_2 + B2 + O$ phase field. This leads to a partial dissolution of O phase grain boundary precipitates during HIP resulting in a discontinuous grain boundary O phase.

After SA followed by WC (Figure 12e), the fraction of the O phase was significantly reduced due to a rapid cooling preventing precipitation and growth of secondary phase precipitates. The subsequent aging allowed for achieving fine nano-sized secondary acicular precipitates of O phase along with coarse primary O phase (Figure 12f).

3.4. Mechanical Properties

Table 4 shows the microhardness values for the Ti_2AlNb -alloys fabricated by the L-PBF process under various conditions. The preheating temperature of 700 °C resulted in the highest microhardness for both alloys since it corresponds to the highest fraction of the intermetallic O-phase. Preheating temperature effects on the microhardness are identical for both powders. In general, the samples produced using Ti_2AlNb -CYB and Ti_2AlNb -noCYB powders exhibited similar microhardness values in the corresponding conditions; however, the Ti_2AlNb -noCYB alloy showed slightly higher microhardness. This might be attributed to a higher oxygen content for the sample fabricated from the powder without microalloying.

Table 5 shows the results of tensile tests for the L-PBF fabricated samples after various post-processing. In the as-fabricated condition, the Ti_2AlNb -alloy samples showed low tensile strength (TS) values at room and elevated temperatures as well as brittle deformation with low elongation at break (EL) values. Low mechanical properties in the as-fabricated condition might be attributed to the presence of residual defects acting as stress concentrators as well as brittle intermetallic O phase grain boundary precipitates.

Table 4. Microhardness of the samples fabricated using Ti₂AlNb-CYB and Ti₂AlNb-noCYB powders by L-PBF process with different preheating temperatures (TPH) and post-processing.

Powder	Ti ₂ AlNb-CYB				
Condition	300 °C TPH, as-is	700 °C TPH, as-is	850 °C TPH, as-is	850 °C TPH, annealed (FC)	850 °C TPH, HIPed
Microhardness, HV0.5	365 ± 10	492 ± 10	405 ± 10	370 ± 10	350 ± 5
Powder	Ti ₂ AlNb-noCYB				
Condition	300 °C TPH, as-is	700 °C TPH, as-is	850 °C TPH, as-is	850 °C TPH, annealed (FC)	850 °C TPH, HIPed
Microhardness, HV0.5	380 ± 10	505 ± 15	425 ± 15	385 ± 10	360 ± 10

Table 5. Tensile mechanical properties of Ti₂AlNb-based alloys.

Alloy	Condition	TS (RT), MPa	TS (650 °C), MPa	EL (RT), %	EL (650 °C), %
Ti-22Al-23Nb-0.8Mo-0.3Si-0.4C-0.1B-0.2Y (at. %) (this study)	L-PBF	652	647	0.6	3.0
	L-PBF + HIP + SA (FC)	1090	801	1.0	4.5
	L-PBF + HIP + SA (FC) + Aging	1120	875	1.2	3.5
	L-PBF + HIP + SA (WC)	1160	600	3.4	2.1
	L-PBF + HIP + SA (WC) + Aging	1290	772	1.7	3.7
Ti-24Al-25Nb-1Zr-1.4V-0.6Mo-0.3Si (at. %) [19]	L-PBF + HIP	1027	860	1.1	5.8
Ti-11Al-40Nb-5(V + Mo + Zr + Ta + W) + 0.2Si + 0.3Y (wt. %) [5]	Wrought and annealed	1110	850 (at 700 °C)	9.0	5.5 (at 700 °C)
Ti-22Al-25Nb (at. %) [64]	Spark plasma sintered	1105	797	9.4	12.8

Carrying out HIP allowed for achieving high tensile strength at room temperature and 650 °C; however, the elongation values were low compared to the wrought Ti₂AlNb-based alloy. At the same time, heat treatment conditions affected the mechanical properties of the fabricated alloy. Annealing followed by WC allowed for achieving the highest elongation due to the discontinuous grain boundary O phase precipitates. Subsequent aging increased the tensile strength of the alloy up to 1290 MPa. This is 25% higher compared to the alloy fabricated from the Ti₂AlNb-noCYB powder and 16% higher compared to the wrought Ti₂AlNb-alloy with Y microalloying. The alloy produced from the Ti₂AlNb-CYB powder exhibited elongation at a break of 3.4%, which is notably higher compared to the alloy obtained from the Ti₂AlNb-noCYB powder. While the achieved tensile strength is on par or higher compared to conventionally produced Ti₂AlNb alloys, the elongation of the L-PBF-produced alloy is lower. Thus, further optimization of heat treatment conditions might be carried out to improve the plasticity of the alloy.

4. Conclusions

In this work, a Ti-22Al-23Nb-0.8Mo-0.3Si-0.4C-0.1B-0.2Y orthorhombic alloy atomized powder was used to fabricate Ti₂AlNb-alloy samples using various L-PBF processing parameters. The following conclusions have been drawn:

- Addition of microalloying elements such as yttrium, boron, and carbon did not lower the alloy's susceptibility to cracking during the L-PBF despite the reduced oxygen content. High-temperature substrate preheating during the L-PBF process

with temperatures above 700 °C is necessary to prevent the cold cracking of the alloy. At the same time, the microalloying of the Ti₂AlNb-based alloy improved its L-PBF processability and resulted in a reduced number of grain boundary microcracks.

- It was shown that using VED of 49 J/mm³ and 850 °C preheating temperature during the L-PBF process allowed for fabricating fully dense (relative density of 99.9 ± 0.1%) crack-free Ti₂AlNb-based alloy samples.
- Microstructure of the titanium orthorhombic alloy is highly sensitive to the substrate preheating temperature used during the L-PBF. A low preheating temperature of 300 °C resulted in a single β/B2 phase microstructure. Increasing the preheating temperature to 700 °C led to the formation of a single O phase microstructure with the highest microhardness. Further increasing the preheating temperature led to a dual-phase microstructure consisting of B2 + O phases. The microalloying resulted in a slightly refined β/B2 grain size compared to the reference alloy. At the same time, no significant changes in microstructures were observed for the microalloyed and reference powders. Microalloying allowed for achieving a smaller grain size after HIP is compared to the reference Ti₂AlNb-alloy.
- Heat treatment conditions affected the microstructure and mechanical properties of the fabricated alloy. The highest room temperature tensile strength of 1290 MPa was achieved after HIP with subsequent annealing followed by water cooling and aging resulting. Applying HIP post-treatment for the Ti₂AlNb alloy obtained by L-PBF is essential to improve its mechanical properties.

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