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Enhancement of Microstructure and Mechanical Properties of High Nb-TiAl Alloys Prepared Using Laser Additive Manufacturing by Annealing Treatment

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Abstract: The microstructure, phase composition, hardness, and tensile properties of the Ti-48Al-2Cr-5Nb alloy have been systematically investigated using laser additive manufacturing technology. Results indicate that both the as-deposited and annealed microstructures contain both the α_2 (Ti₃Al) and γ (TiAl) phases. As the annealing temperature increased, the structure changed significantly from a large block structure to a fine equiaxed structure and finally to a large lamellar structure. Nevertheless, the amount and distribution of precipitation of α_2 phase are obviously different, especially during the annealing at 1260 °C, where the fine α_2 phases are evenly distributed on the γ phase matrix. The hardness value of the as-deposited sample is the highest, with a HV value of 484 at the room temperature, while the hardness value of the annealed sample at 1260 °C is the smallest, with a HV value of 344. An annealed sample at 1260 °C exhibits the highest tensile strength and elongation at room temperature, with values of 598 MPa and 2.1%, respectively. These values are increased by 1.15 times and 1.4 times compared to the as-deposited sample (519 MPa, 1.5%).

Keywords: laser additive manufacturing; annealing treatment; high Nb-TiAl alloys; microstructure; mechanical properties



Citation: Liang, J.; Liu, Z.; Wang, H.; Yin, G.; Zhu, X. Enhancement of Microstructure and Mechanical Properties of High Nb-TiAl Alloys Prepared Using Laser Additive Manufacturing by Annealing Treatment. *Metals* **2023**, *13*, 1562. <https://doi.org/10.3390/met13091562>

Academic Editor: Abdollah Saboori

Received: 31 July 2023

Revised: 23 August 2023

Accepted: 5 September 2023

Published: 6 September 2023



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1. Introduction

TiAl alloy has the advantages of high melting point, low density, excellent high temperature oxidation resistance, good creep resistance, and high specific strength, and is expected to partially replace the most widely used nickel-based superalloys in the aerospace field. The material will become an important alternative structural material for the aerospace industry, especially the lightweight high-temperature structural material for the new generation of supersonic aircraft, thereby reducing the weight of aviation engines and improving efficiency. However, the working temperature of aeroengine blades and turbofan engines made of TiAl alloy materials is around 650 °C [1–3]. For this situation to be changed, researchers have improved the properties of TiAl alloy by adapting process parameters and annealing techniques, and adding different alloying elements such as Cr, Mn, W, Mo, Si, B, C, Ta, and Nb. Beijing University of Science and Technology uses investment casting technology to add Nb to TiAl alloy to improve the order temperature of TiAl alloy. The working temperature of TiAl alloy is 200 °C higher than that of traditional TiAl alloy. Compared with traditional TiAl alloy, high Nb TiAl alloy shows stronger oxidation resistance and creep resistance at high temperature. Developing high Nb-TiAl alloy represents a new direction in the development of TiAl alloy. In the manufacture of aeroengine blades, there is a great deal of application potential [4,5].

The traditional forming methods of TiAl alloy include powder metallurgy, precision casting, and directional solidification. Due to the poor plasticity and mechanical properties of high Nb-TiAl alloy at room temperature, it is difficult to produce high-quality

alloys with complex shapes by traditional forming methods, which severely limits its wide application [6–8]. Currently, there are significant advantages of laser additive manufacturing (LAM) technology for the high-quality forming of TiAl alloys. It is possible to manufacture parts using laser additive manufacturing technology without the use of molds and assembly processes, which can save material, shorten the manufacturing process, and prepare alloy materials with large sizes, high density, and complex structures. It is now possible to prepare parts using laser additive manufacturing technology according to the required material composition at different locations in accordance with the required material properties. The following is an example: laser additive manufacturing technology can prepare blades with complex shapes directly on the blisk, which eliminates the need to assemble the blade and blisk, thereby improving productivity and product quality and reducing manufacturing costs. It is obvious, therefore, that the preparation of high-performance TiAl alloy has good development potential in several important fields, which will undoubtedly provide a guarantee for the development of superalloys. Currently, scholars have been using laser additive manufacturing technology to prepare high Nb-TiAl alloys and examining their properties and microstructures [9–11]. Di et al. [12] found that laser power can greatly affect the lamellar spacing, while lamellar interfaces and deformation twins can greatly improve the alloy's mechanical properties. Liang et al. [13] discovered that the creep behavior of high Nb-TiAl alloys containing Y_2O_3 was not only related to dislocation slip and climb of the γ phase, but also to activation of the slip system of the α_2 phase. Additionally, dynamic recrystallization can soften the alloy. Kan et al. [14] found that nano-TiC reinforced high Nb-TiAl nanocomposites exhibited a microhardness of 393 HV, which was 24% higher than high Nb-TiAl alloys.

In this study, a laser additive manufacturing technology is used to prepare as-deposited Ti48Al2Cr5Nb alloy samples on TC4 titanium alloy substrates. We then attempt to anneal it and systematically study its microstructure and tensile properties. It is intended to improve the structure and properties of the alloy and to provide experimental support for the development of aerospace blades made of high Nb-TiAl alloy at high temperatures.

2. Experimental Procedures

2.1. Materials and Methods for Conducting Experiments

The material used in this experiment is Ti-48Al-2Cr-5Nb alloy powder with a particle size range of 53–100 μm , purchased from AVIC Maite Powder Metallurgy Technology Co., Ltd., Beijing, China; its chemical composition (wt.%) is: 31.06 Al, 2.49 Cr, 11.35 Nb, 0.06 O, 0.005 N and the rest is Ti. In preparation for the test, the Ti-48Al-2Cr-5Nb alloy powder is dried at 200 $^{\circ}C$ for one hour in order to remove water vapor from the surface of the alloy powder, improve its fluidity during the manufacturing process, and reduce the formation of pores in the deposited layer. This substrate is made of TC4 titanium alloy with dimensions of 100 mm $\times$ 100 mm $\times$ 10 mm. In order to prevent the influence of oxide film and oil on the forming quality of TC4 titanium alloy matrix surface, the surface of the matrix was polished with a grinder before the test to remove the oxide film and surface defects, and then cleaned with anhydrous ethanol and acetone in turn to remove oil and other impurities. In order to prevent cracking when depositing Ti-48Al-2Cr-5Nb powder, it is necessary to ensure that the substrate preheating temperature is not below 350 $^{\circ}C$, and the substrate preheating temperature in this experiment was 400 $^{\circ}C$.

The laser additive manufacturing equipment is manufactured by Nanjing Zhongke Raycham Laser Technology Co., Ltd., Nanjing, China, with the model LDM8060. The system consists of a semiconductor laser LDF-4000, a four-way powder feeding print head, a pneumatic powder feed machine, a water cooler, a three-axis CNC worktable, and an inert gas chamber. 99.99% Ar protective gas is used in the inert gas cabin, in which the water and oxygen content is less than 50 ppm. The laser processing parameters for the as-deposited samples are shown in Table 1. The reciprocating scanning method was used to form a defect-free sample on the TC4 substrate by multi-layer and multi-channel deposition layer by layer. The sample size was 30 mm $\times$ 20 mm $\times$ 8 mm, as shown in Figure 1a. Afterward,

the as-deposited samples were annealed at 1200 °C/30 min/FC, 1260 °C/30 min/FC, and 1320 °C/30 min/FC, respectively. The “FC” in the paper refers to annealing, that is, the specimen is heated to a certain temperature and then reduced to room temperature by furnace cooling.

Table 1. Parameters associated with laser processing.

Material	Laser Power (W)	Laser Scan Speed (mm /min)	Powder Feed Rate (g/min)	Diameter of the Spot (mm)	Thickness of the Layer (mm)
Ti48Al2Cr5Nb	1400	540	5.6	0.4	3

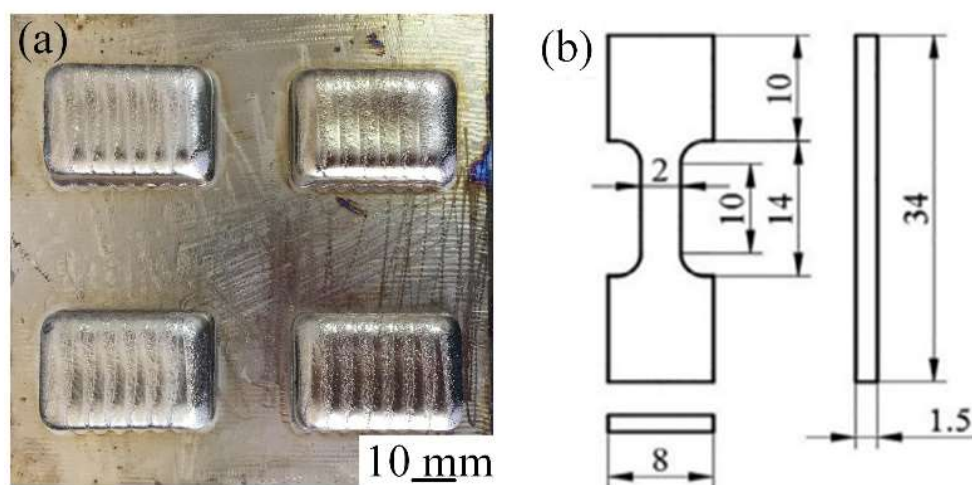


Figure 1. Deposition sample and tensile sample sizes in laser additive manufacturing: (a) as-deposited sample; (b) size of the tensile sample (Unit: mm).

A ZSL1600X heat treatment furnace was used to anneal the samples. Following the annealing treatment, the formed sample was wire-cut along the vertical laser scanning direction in order to prepare it for future metallographic, X-ray diffraction, hardness, and other test purposes. The XRD specimen should be prepared as follows: the cut specimens should be ground in a criss-cross pattern with abrasive papers of different particle sizes (240#, 600#, 800#, 1200#, 1500#, 2000#, 3000#). Diamond spray polishing agent (0.5 μm) should be used to mechanically polish the sample to an optical grade, after which it should be cleaned and dried using acetone. XRD specimen preparation without corrosion. We examined the phase composition of the deposited layer by using the X-7000 X-ray diffraction analyzer (scanning rate 8°/min; scanning range 20°–90°). A metallographic specimen was prepared by inlaying and then grinding and polishing. The grinding and polishing process was the same as the process described above, followed by chemically etching with Kroll reagent (HF, HNO₃ and H₂O are diluted 2:3:10). The corrosion time was about 15 s. A Hitachi S-3400N scanning electron microscope (SEM) was used to analyze the microstructure. The preparation of EBSD samples involved electrolytic polishing. The polishing process was the same as the above-mentioned process, and then electrolyte was used for the final polishing and electrolysis to remove the residual stress layer on the sample's surface. The electrolysis parameters were as follows: the electrolyte volume ratio was 60:34:6 for methanol, n-butanol, and perchloric acid, the electrolyte temperature was −30 °C, the electrolysis voltage was 20 V, and the electrolysis time was 10–25 s. The phase distribution, texture distribution, and grain orientation of the samples were studied by electron backscattered diffraction (EBSD). We measured the Vickers hardness distribution of the sample with the HVS-5 Vickers hardness tester (the loading force was 200 g and the duration was 10 s). Moreover, each sample was randomly measured four times and the average value was taken as the experimental data. During wire cutting of tensile samples

at room temperature, the cutting method should be parallel to the direction of deposition. A water-abrasive paper should be used to grind the tensile sample to 2000# in order to remove any traces of wire cutting. Ti-48Al-2Cr-5Nb alloy tensile properties were tested at room temperature using the WDW-3100 tensile testing machine. Graph the tensile test data as a stress-strain curve. The maximum tensile strength σ_b and the elongation at fracture δ could be calculated from this curve. Using a Hitachi S-3400N scanning electron microscope (SEM), we examined the fracture morphology of the samples after they had undergone the tensile test. The size of the tensile sample was shown in Figure 1b.

2.2. Parameter Selection for Annealing

According to the Ti-Al binary alloy phase diagram, the different microstructures will be produced when the annealing temperature is 1200 °C, 1260 °C, and 1320 °C. The annealing temperature has a significant impact on the microstructure of the alloy when it is in equilibrium. It is important to note that when the annealing temperature is 1200 °C, the temperature is over the eutectoid transformation temperature line, so the structure of the alloy annealed at this temperature is called a near γ structure (NG). As long as the annealing temperature is set at 1260 °C, the alloy surface structure obtained at this temperature is commonly referred to as a dual-state structure (DP). Dual-state structures have excellent comprehensive mechanical properties, and their extensibility will be greatly increased while only a small loss of strength will be experienced. During annealing at 1320 °C, the temperature is below the α transformation line of the alloy, which is known as a near-lamellar structure [15].

3. Results and Discussion

3.1. The Microstructure

As shown in Figure 2, the SEM microstructure images of Ti-48Al-2Cr-5Nb alloy samples with different annealed conditions can be seen. As can be seen from the figure, the microstructure of the as-deposited sample is mainly composed of fine lamellar structures and smaller massive structures [16–18]. The interior of the lamellar structure is composed of lamellar γ phase and α_2 phase, in which γ phase is the matrix phase. The large block structures were observed in the microstructure of the annealed sample at 1200 °C. The microstructure of the sample annealed at 1260 °C showed a large number of fine equiaxed structures [19]. Due to the fact that when the temperature is 1200 °C and 1260 °C, it will be in the $\alpha + \gamma$ two-phase region with about an equal volume fraction on the binary phase diagram of TiAl. This heat treatment range can result in a dual-state structure consisting of γ grains and α_2/γ lamellar. Due to the mutual pinning of α phase and γ phase during heat treatment, the grain growth rate is slow and eventually a dual-state structure with smaller grain sizes develops. Microstructures in the annealed sample at 1320 °C consist of coarse lamellar structures. Due to the use of heat treatment in the $\alpha + \gamma$ two-phase region slightly below the α_2 transition temperature, the near-lamellar structure is formed following furnace cooling, consisting of α_2/γ linear lamellar clusters and a small amount of equiaxed γ grains. The volume fraction of γ grains in the alloy structure at this point is small, and the pinning effect on the growth of α grains is weak; finally, a larger lamellar structure is formed.

3.2. The Phase Composition

The results of the XRD analysis of the as-deposited sample and samples with different annealed conditions are shown in Figure 3. As seen in the figure, the as-deposited Ti-48Al-2Cr-5Nb alloy structure is composed of α_2 phase and γ phase, of which γ phase is the matrix phase. The composition of the phase in the alloy does not change after annealing. Compared with the other three groups of diffraction peaks, the γ (111) diffraction peak of the sample annealed at 1260 °C/30 min/FC is the highest when 2θ is equal to 39° [20]. This indicates that under this annealed condition, the alloy contains a greater percentage of γ (111) crystal plane in the crystalline area. Upon increasing the temperature of the

annealing to 1320 °C, the α_2 diffraction peaks increased at $2\theta = 36^\circ$ and $2\theta = 54^\circ$, and the γ (202) crystal plane diffraction peaks disappeared at $2\theta = 65^\circ$.

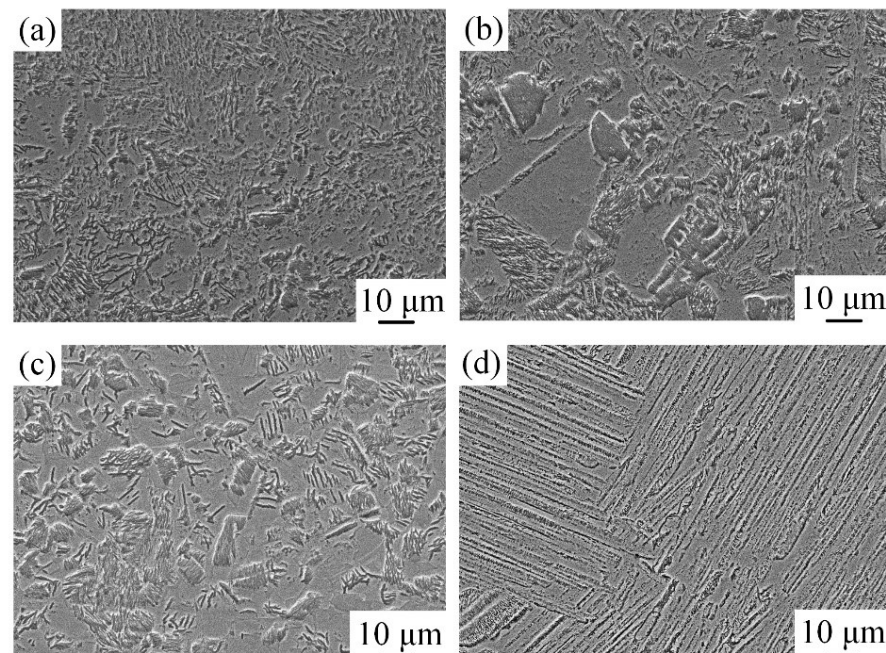


Figure 2. SEM microstructure images of Ti-48Al-2Cr-5Nb alloy samples with different annealed conditions: (a) as-deposited; (b) 1200 °C/30 min/FC; (c) 1260 °C/30 min/FC; (d) 1320 °C/30 min/FC.

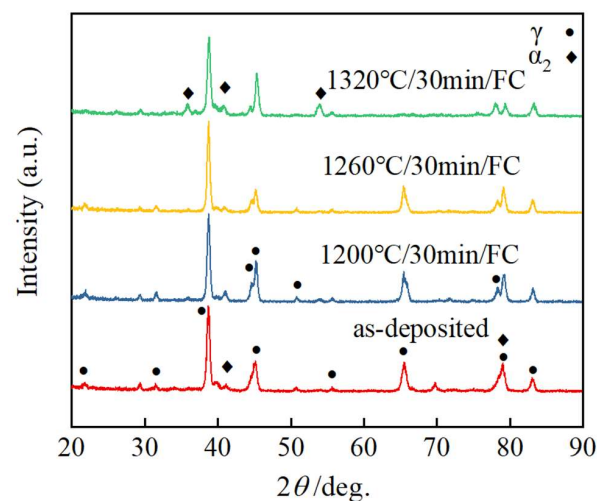


Figure 3. Results of XRD analysis of the Ti-48Al-2Cr-5Nb alloy samples with different annealed conditions.

3.3. The Elemental Distribution

EDS composition analysis results for the as-deposited and annealed samples are shown in Figure 4. According to Figure 4a, the Al, Ti, and Cr elements in the as-deposited Ti-48Al-2Cr-5Nb alloy are evenly distributed without obvious segregation, whereas the Nb element exhibits a small degree of segregation. In spite of the increase in annealing temperature, the distribution of Ti element in the structure does not change significantly. This is primarily due to the fact that the addition of the Cr element reduces the activity of Ti, making it difficult to react or diffuse with other elements [21]. It is also worth noting that Al_2O_3 is more stable on the γ phase surface than on the α_2 phase surface. Therefore, Al elements are enriched in the γ phase [22,23]. Furthermore, the enrichment of Cr at the grain boundary is becoming more and more obvious, mainly due to Cr playing a significant

role in oxygen absorption in the alloy. It is easy to combine with the O element under high-temperature conditions, while the O ion can easily enrich at the grain boundaries. It is for these reasons that Cr elements accumulate as oxides at the grain boundaries [24,25]. It appears that the distribution of Nb elements in the microstructure is becoming more and more uniform, indicating that annealing can promote the diffusion of Nb elements.

3.4. The Phase Distribution

Figure 5 shows the EBSD phase distribution map of as-deposited and annealed samples. In the figure, the blue represents the γ phase and the red represents the α_2 , where the γ phase is the matrix phase of the alloy Ti-48Al-2Cr-5Nb. It has been determined that the volume fraction of the α_2 phase in the as-deposited sample and samples with different annealed conditions is 3.4%, 11.3%, 5.2%, and 6.1%, respectively. At the same time, the volume fraction of γ phase is 96.6%, 88.7%, 94.8%, and 93.9%, respectively. The α_2 phases in the as-deposited sample and sample at annealed 1200 °C/30 min/FC are unevenly distributed on the γ matrix. As the alloy is annealed at 1260 °C/30 min/FC, the fine α_2 phase is evenly distributed in the γ matrix [26]. When annealed at 1320 °C/30 min/FC, the α_2 phase is distributed in a coarse lamellar pattern on the γ matrix. As is well known, the α_2 phase structure belongs to the hexagonal structure that has relatively fewer slip systems and therefore poor plasticity [27,28]. The presence of more α_2 in the microstructure has an adverse effect on the alloy's mechanical properties at room temperature. Moreover, the results of the tensile testing below provide further evidence of this point.

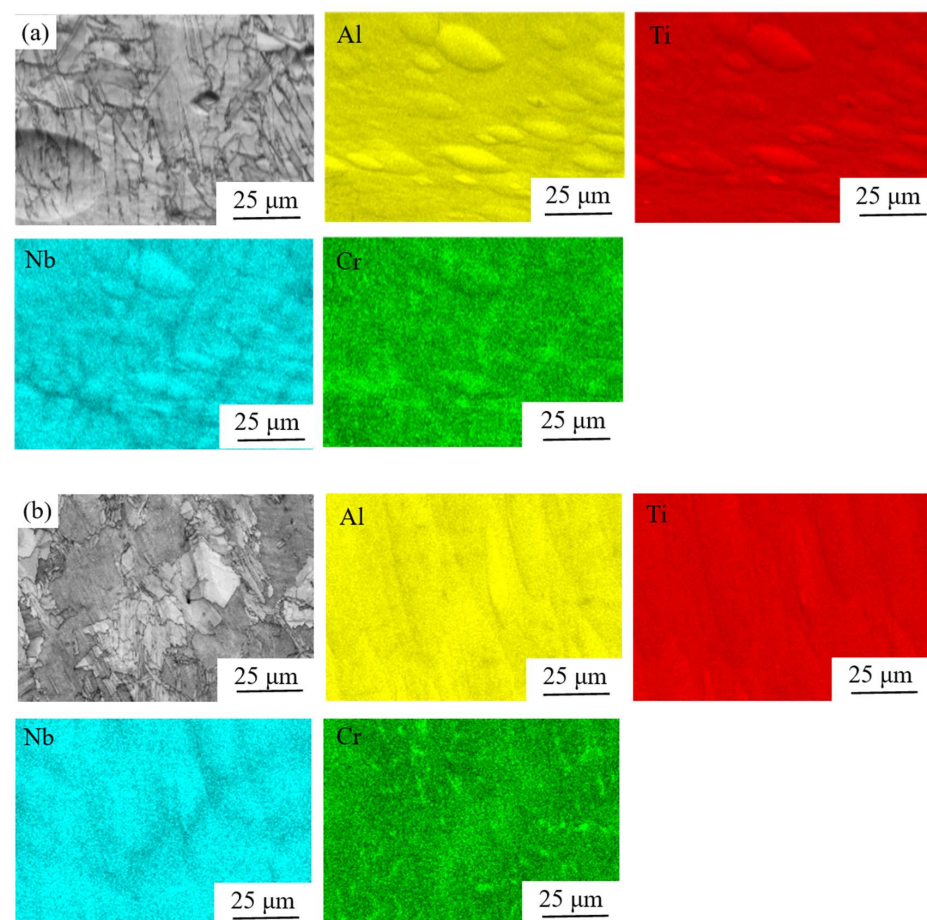


Figure 4. Cont.

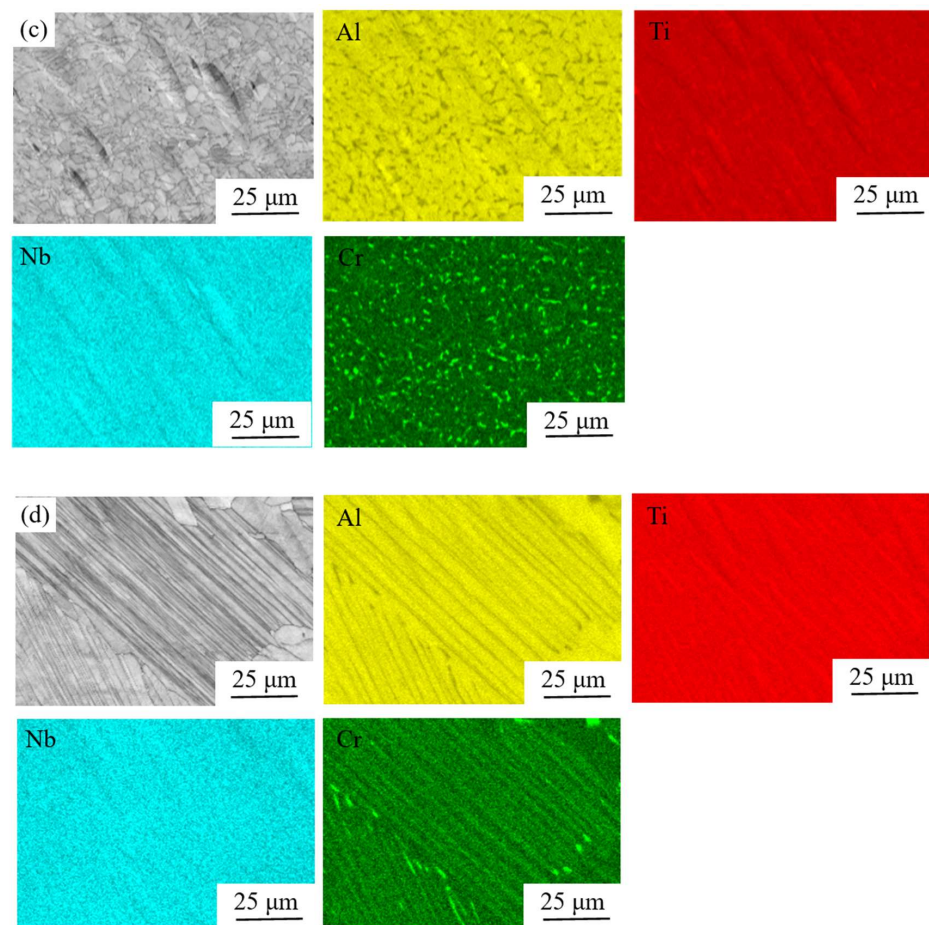


Figure 4. Results of the EDS composition analysis in the as-deposited sample and samples with different annealed conditions: (a) as-deposited; (b) 1200 °C/30 min/FC; (c) 1260 °C/30 min/FC; (d) 1320 °C/30 min/FC.

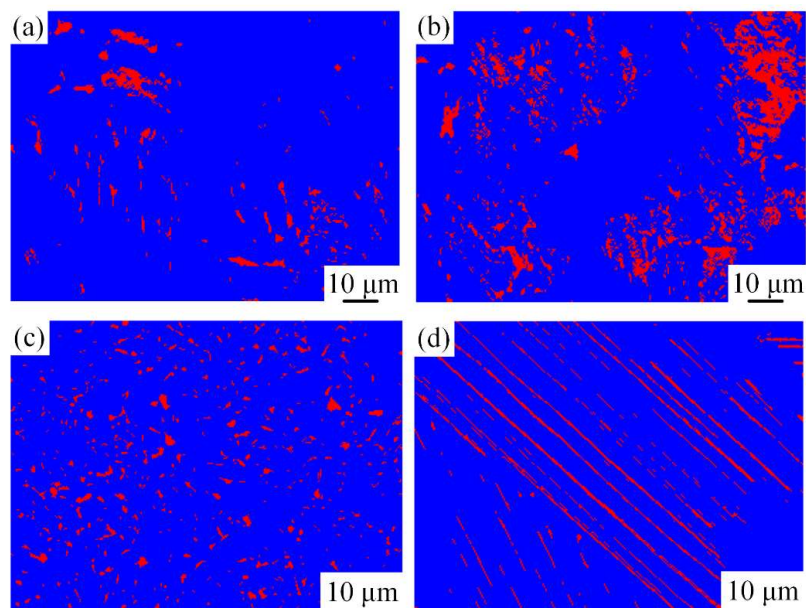


Figure 5. The EBSD phase distribution map of the as-deposited sample and samples with different annealed conditions: (a) as-deposited; (b) 1200 °C/30 min/FC; (c) 1260 °C/30 min/FC; (d) 1320 °C/30 min/FC.

3.5. The Grain Orientation

Figure 6 shows the crystal orientation map for the as-deposited and annealed samples. Since the γ phase is the matrix phase of the Ti-48Al-2Cr-5Nb alloy, the crystal orientation of the γ phase represents the crystal orientation within the alloy. It should be noted that the red in the crystal orientation distribution diagram represents the (001) crystal orientation in the γ phase, the green represents the (010) crystal orientation in the γ phase, and the blue represents the (110) crystal orientation in the γ phase. In the as-deposited sample, the crystal orientations mostly are (010). Compared with the as-deposited sample, some colors in Figure 6b change to purple. Under the annealed condition of 1260 °C/30 min/FC, the grain orientation is (001), indicating that it is beneficial to the tensile properties in the direction of (001). At 1320 °C/30 min/FC, the crystal orientations are distributed evenly in the three directions (001), (110) and (010). This indicates that the number of grains distributed in different orientations is not much different, and neither are the tensile properties in each direction [29].

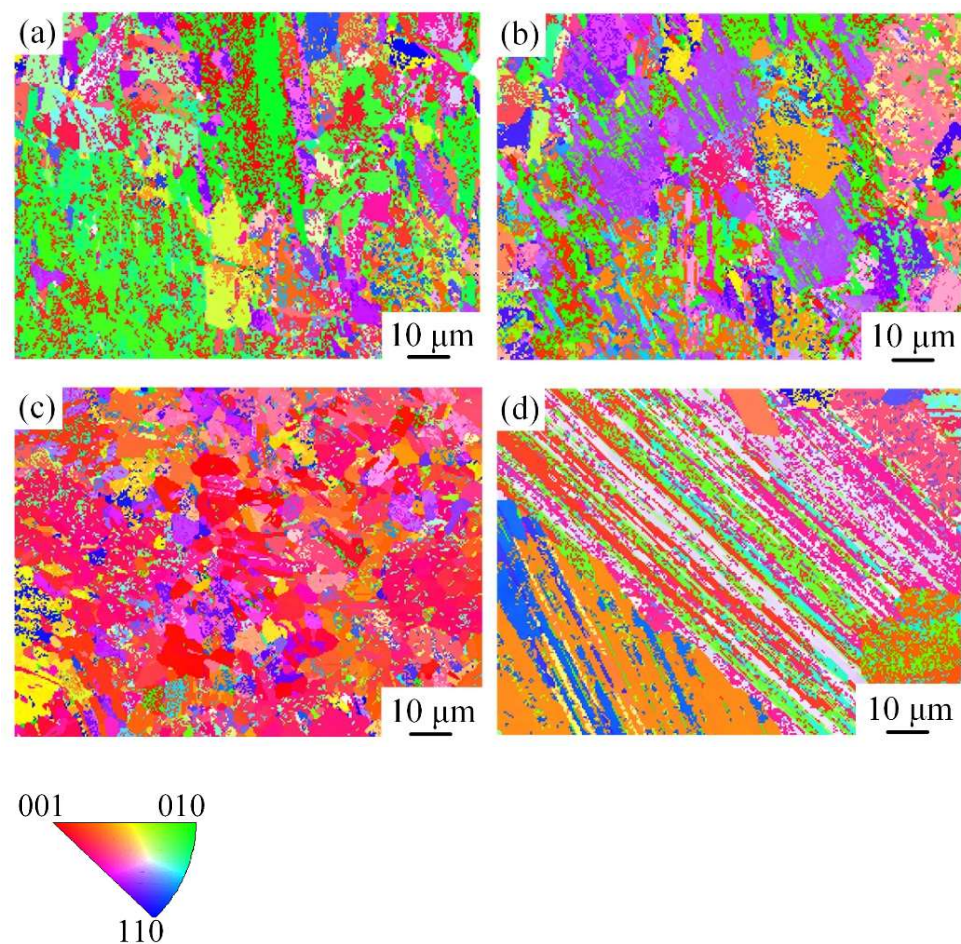


Figure 6. The grain orientation map for the deposited sample and samples with different annealed conditions: (a) as-deposited; (b) 1200 °C/30 min/FC; (c) 1260 °C/30 min/FC; (d) 1320 °C/30 min/FC.

3.6. The Texture Strength

Figure 7 shows the pole figures for α_2 {0001} and γ {111} in the as-deposited and annealed samples. Texture in metal materials can be seen as the crystal orientation or the crystallographic orientation. In this paper, the texture strength refers to the proportion of two phases growing in different directions. As the annealing temperature increases, the texture strength of the α_2 {0001} and γ {111} in the alloy increases first and then decreases. It has been determined that the texture strength of α_2 {0001} in the as-deposited sample and samples with different annealed conditions is 119.9, 61.2, 7.4, and 154.8, respectively. The

texture strength of γ {111} is 9.3, 17.4, 5.9, and 33.4, respectively. The texture strength of the annealed sample at 1260 °C/30 min/FC is the smallest among them. In comparison with the as-deposited state, the maximum texture intensity of α_2 {0001} and γ {111} decreased by 112.5 and 3.4, respectively. The sample annealed at 1320 °C/30 min/FC has the highest texture strength. It can be seen that the annealing temperature has a significant influence on the texture strength of the alloy [30–32].

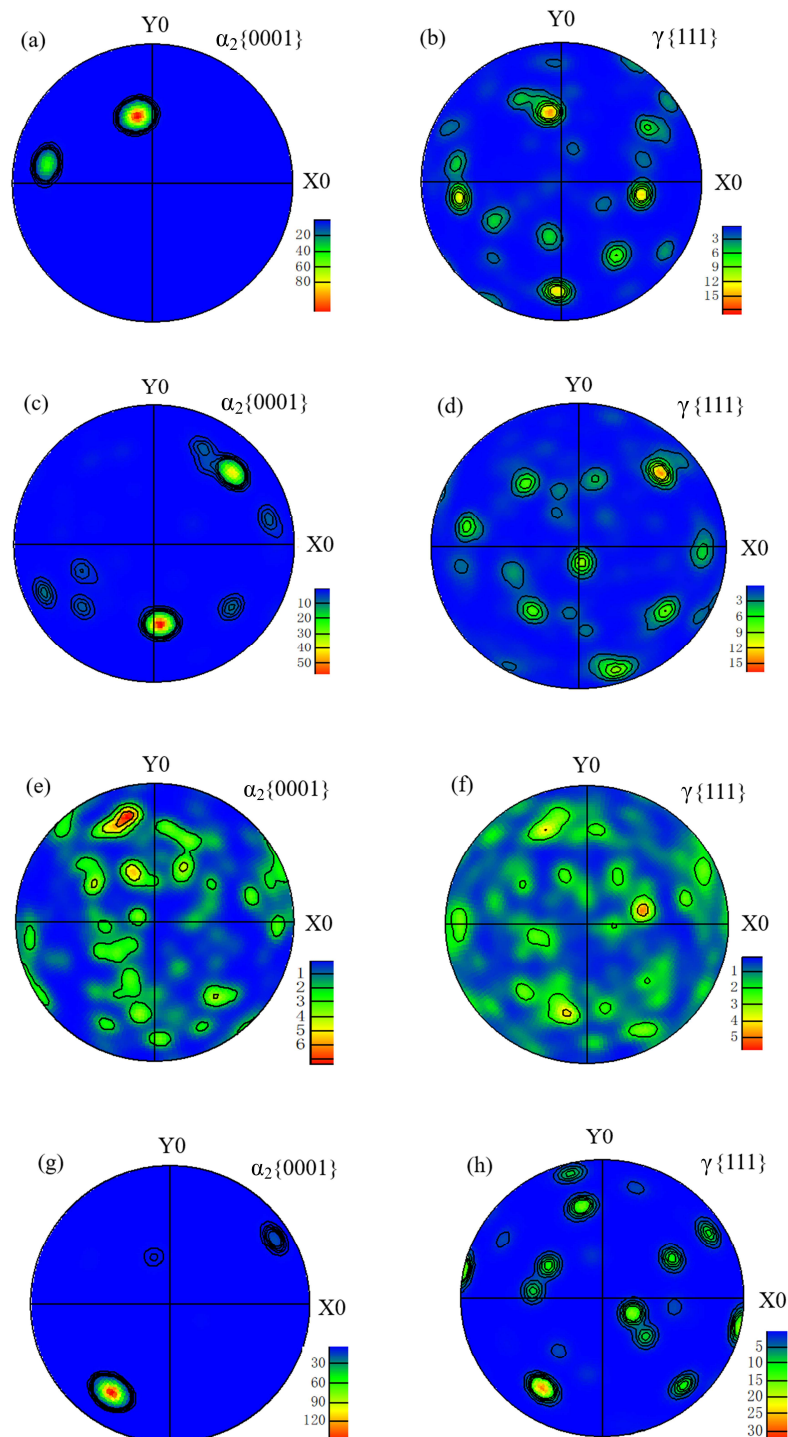


Figure 7. Diagrams depicting the poles of α_2 {0001} and γ {111} in the as-deposited sample and samples with different annealed conditions: (a,b) as-deposited; (c,d) 1200 °C/30 min/FC; (e,f) 1260 °C/30 min/FC; (g,h) 1320 °C/30 min/FC.

3.7. The Mechanical Properties

In Figure 8, the mechanical properties of the as-deposited and annealed samples are shown. Test randomly four points on each sample surface to take the average. The hardness of each point is determined by an average, as shown in Figure 8a. The figure indicates that the average hardness of the as-deposited specimen and specimens annealed at 1200 °C/30 min/FC, 1260 °C/30 min/FC, and 1320 °C/30 min/FC is 484 HV, 384 HV, 344 HV, and 366 HV, respectively. In this comparison, the Vickers hardness value of the as-deposited sample is the highest, while that of the 1260 °C/30 min/FC annealed sample is the lowest. In general, the α_2 phase belongs to the hexagonal structure, and the γ phase belongs to the face-centered crystal structure. The slip system of the hexagonal structure is less than that of the face-centered structure, so the hardness of the α_2 phase is higher than that of the γ phase [27,31,33,34]. The reduction of alloy hardness in this experiment was primarily due to the presence of the α_2 phase. The content of α_2 phase decreases and equiaxed γ phase forms, which are conducive to the dislocation of dislocations, thereby reduce the hardness of the microstructure.

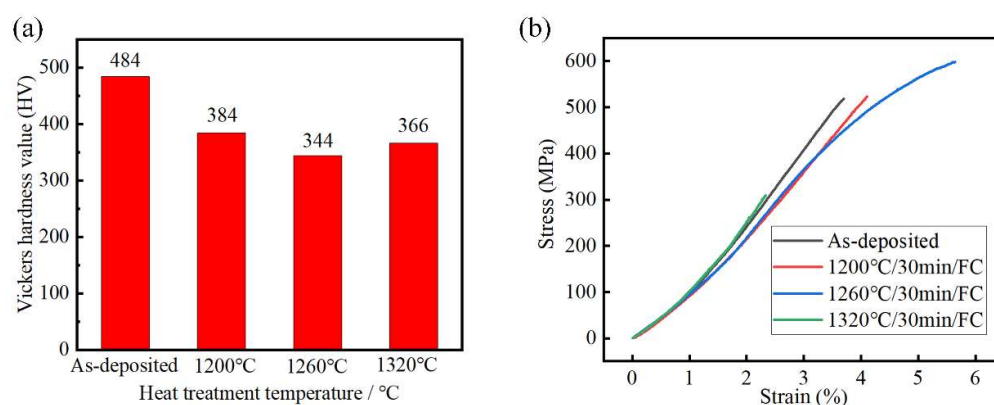


Figure 8. Mechanical properties of the as-deposited sample and samples with different annealed conditions: (a) Vickers hardness; (b) mechanical properties.

According to Figure 8b, the room temperature tensile curves of the as-deposited and annealed samples are shown. Among the as-deposited, 1200 °C/30 min/FC, 1260 °C/30 min/FC, and 1320 °C/30 min/FC annealed samples, the tensile strength is 519 MPa, 523 MPa, 598 MPa, and 309 MPa, and the elongation is 1.5%, 1.6%, 2.1%, and 1.1%, respectively. As the annealing temperature increases, the tensile properties of the alloy at room temperature first increase and then decrease. In light of the above microstructure characterization analysis, it can be observed that excessively high annealing temperatures will result in the growth of lamellar clusters and the coarsening of the α_2/γ lamellar layers within the lamellar clusters. According to the Hall–Petch formula, the larger the grain size, the poorer the alloy’s mechanical properties [35,36]. The annealing temperature is therefore too high and the alloy’s mechanical properties are poor as a result. Furthermore, the strength of the sample after 1260 °C/30 min/FC annealing has been greatly improved compared with the as-deposited sample, and its tensile strength at room temperature increases from 519 MPa to 598 MPa, an increase of approximately 1.15 times. It is found that the maximum elongation is achieved by the sample annealed at 1260 °C/30 min/FC, which is about 1.4 times that of the deposited alloy, increasing from 1.5% to 2.1%. The main reason is the presence of equiaxed γ grains within the microstructure which contribute to crystal deformation. Therefore, tensile deformation is easier, i.e., plasticity is higher.

Based on the above analysis of hardness and strength, the grain of the alloy in this paper has been refined after annealing, and the plasticity has been improved. However, the dislocation density decreases after annealing, eliminating the internal stress of the material, resulting in a decrease in hardness.

3.8. The Fractography

Figure 9 shows the tensile fracture morphology of as-deposited and annealed samples. According to the figure, the fracture mechanisms of the deposited specimen and specimens annealed at 1200, 1260, and 1320 °C for 30 min belong to the quasi-cleavage fracture mechanism [37–39]. It is evident from Figure 9a,b that the fracture surfaces have river patterns both in the deposited sample and the sample annealed at 1200 °C/30 min/FC, and without dimples. The fracture surface in Figure 9c shows that river patterns are significantly reduced and cleavage planes are small under annealing at 1260 °C/30 min/FC. There are mainly small cleavage planes and tear edges on the fracture surface, and no dimples are present. The cleavage surface in Figure 9d is larger and the proportion of the cleavage surface to the fracture surface is greater, which significantly weakens the fracture performance of the sample annealed at 1320 °C/30 min/FC. Generally, the greater the proportion of the cleavage plane to the fracture surface, and the greater the single area of the cleavage plane, the worse the mechanical properties of the alloy at room temperature [40]. Because the alloy consists of larger layer clusters, the loading stress is vertical in the layer direction, resulting in a weakening of the bonding force between the layers, leading to a decrease in strength and plasticity.

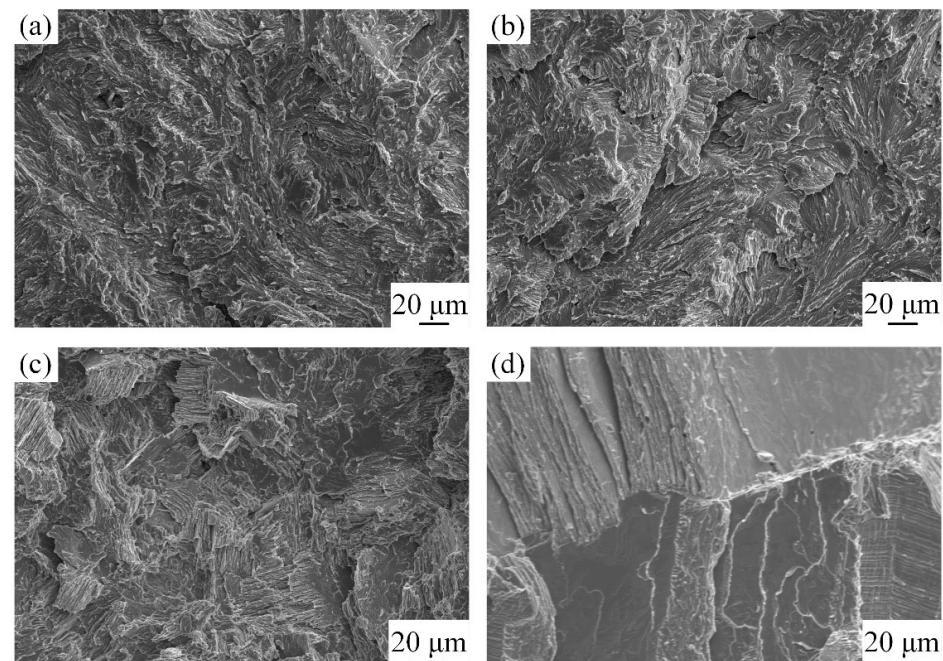


Figure 9. Fracture morphology of the as-deposited sample and samples with different annealed conditions: (a) as-deposited; (b) 1200 °C/30 min/FC; (c) 1260 °C/30 min/FC; (d) 1320 °C/30 min/FC.

4. Conclusions

In this paper, the samples of Ti-48Al-2Cr-5Nb alloy deposited on TC4 titanium alloy are successfully prepared by using laser additive manufacturing technology. Then, we try to anneal it and systematically study its microstructure and mechanical properties. The experimental results are as follows:

- (1) The results show that the annealed microstructures are still composed of α_2 (Ti₃Al) and γ (TiAl) phases.
- (2) With the increase in annealing temperature, the structure changes obviously from large block structure to fine equiaxed structure, and finally to large lamellar structure. However, there are significant differences in the amount and distribution of α_2 phase precipitation.
- (3) The mechanical properties change significantly with the increase in annealing temperature. The results show that the tensile properties at room temperature can be improved and the brittleness of the alloy can be reduced by proper annealing.

Author Contributions: Conceptualization, J.L. and Z.L.; methodology, J.L. and Z.L.; software, H.W.; validation, G.Y.; formal analysis, J.L. and X.Z.; investigation, Z.L.; resources, Z.L.; data curation, H.W.; writing—original draft preparation, J.L. and Z.L.; writing—review and editing, J.L. and Z.L.; visualization, H.W.; supervision, Z.L.; funding acquisition, Z.L., G.Y. and X.Z. All authors have read and agreed to the published version of the manuscript.

Funding: The work was supported by the Doctoral Research Start-up Fund of Liaoning Province (Grant No. 2023-BS-195); Basic Scientific Research Project of Department of Education of Liaoning Province (Grant No. LJKMZ20220960).

Data Availability Statement: Data are unavailable in this study.

Conflicts of Interest: The authors declare no conflict of interest.

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