Influence of hot isostatic pressing on microstructure, properties and deformability of selective laser melting TC4 alloy

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Received: 2021-04-26

Accepted: 2021-07-21

Abstract: The influence of different hot isostatic pressing regimes on microstructure, phase constitution, microhardness, tensile properties and deformability of TC4 alloy fabricated by selective laser melting (SLM) technology was studied. The results show that the microstructure of SLM TC4 alloy is composed of acicular martensite α' phase, and the sample exhibits high microhardness and strength, but low plasticity. After hot isostatic pressing, acicular martensite α' phase transforms into $\alpha+\beta$ phase, and with the increase of hot isostatic pressing temperature and duration, α phase with coarse lath is gradually refined, and the proportion of α phase is gradually reduced. Because of the change of phase constitution in SLM TC4 alloy after hot isostatic pressing, the grain refinement strengthening is weakened, the density of dislocation is reduced, so that both microhardness and tensile strength are decreased by around 20%, the elongation is increased by more than about 70%, even over 100%, compared with as-deposited TC4 alloy. When the hot isostatic pressing regime is 940 °C/3 h/150 MPa, the tensile strength and the elongation achieve optimal match, which are about 890 MPa and around 14.0% in both directions. The fracture mechanism of alloy after 940 °C/3 h/150 MPa HIP is ductile fracture. Hot isostatic pressing causes concave deformation of SLM TC4 alloy thin-walled frames, and the deformation degree increases with the increase of temperature.

Key words: selective laser melting; TC4 alloy; hot isostatic pressing; microstructure; mechanical properties; deformability

CLC numbers: TG146.23; **Document code:** A; **Article ID:** 1672-6421(2021)04-389-08

1 Introduction

TC4 alloy with the characteristics of light weight, high strength, good corrosion resistance and heat resistance is widely used in the aerospace field, and as an important engineering material for the manufacturing of aircraft parts^[1,2]. At present, with the increasing demand for the integration and complexity of the aircraft parts, it is difficult to produce components with dimensional accuracy and acceptable surface quality by traditional processing technologies like casting and forging^[3]. Selective laser melting (SLM) technology is particularly suitable for the preparation of the metal components with complicated structure^[4-6], which is decided by the unique technological process: using specific software to cut the three-dimensional (3D) model of the components to be processed into a series of layers with a certain thickness, then using a high-energy laser beam to melt metal powder selectively layer by layer according to each layer's data^[7,8]. An attractive feature of this process is that there is no limit for the structural complexity of the parts, and it also has the advantages of high material utilization and no molds^[9-11].

However, SLM TC4 alloy usually retains high residual stress, and random distributed defects such as cracks and holes are easily formed inside, which may affect its mechanical

properties^[12]. In this case, the microstructure and properties are often regulated by heat treatment. Fan et al.^[13] found that after holding at 950 °C for 1 h, part of α phase in SLM TC4 alloy was spheroidized, and other α phase still presented basket weave structure. Under this condition, the strength of the sample reached 1,200 MPa, but the elongation was only 2.42%. Liang et al.^[14] studied the effects of 5 different heat treatment regimes on the microstructure and mechanical properties of SLM TC4 alloy. The results showed that, in maintaining heat treatment duration for 4 h, with the increase of temperature from 650 °C to 900 °C, α' phase gradually decomposed into lamellar $\alpha+\beta$ structure, and almost completely decomposed at 800 °C/4 h, the elongation was more than 12%, and the tensile strength was more than 1,000 MPa. Zhang et al.^[15] found that the microstructure changed from Widmanstatten to basket weave structure when the annealing temperature was above 800 °C. With the increase of annealing temperature, the tensile strength of the alloy decreased and the elongation increased. The SLM TC4 alloy subjected to heat treatment at 800 °C/2 h had excellent comprehensive mechanical properties. It can be seen from the above research that heat treatment can reduce the residual stress of SLM TC4 alloy, and regulate the microstructure and properties. Hot isostatic pressing (HIP) can compact the internal defects through high pressure, thus improving the overall mechanical properties of the components. Xu et al.^[16] studied the effects of HIP temperature on as-cast TC4, the results showed that the shrinkage was reduced as the HIP temperature increased, and even completely

closed above 920 °C. Jiang et al.^[17] found that after HIP, the porosity defects in direct laser deposition Ti60 alloy disappeared, and the elongation increased from 6.5% to 12.0%, while maintaining tensile strength over 1,000 MPa. Therefore, HIP may have more advantages in the post-processing of SLM components compared with annealing heat treatment in terms of eliminating the random hole defects that form in the SLM process^[18], thus improving the performance.

At present, the study on the effect of HIP on the microstructure and properties of SLM TC4 alloy is greatly insufficient. In this study, through the hot isostatic pressing method, the influence of temperature and duration of HIP on the microstructure, phase constitution, microhardness, tensile properties and deformability of SLM TC4 alloy was studied, which can provide theoretical basis and technical support for the manufacturing of aircraft parts.

2 Experimental procedure

The powder selected for the study was processed by high-speed plasma rotating electrode process. Table 1 shows the typical chemical composition of the TC4 powder. The TC4 powder morphology is shown in Fig. 1, showing that the surface of the powder particles is smooth and clean. The powder has high sphericity degree, up to 92.7%. Powder particle size distribution is as follows: $D(10)=26.74\ \mu\text{m}$, $D(50)=40.41\ \mu\text{m}$, $D(90)=52.15\ \mu\text{m}$.

Table 1: Composition of experimental TC4 alloy powder (wt.%)

Ti	Al	Cu	Fe	Mn	Mo	Sn	V	C	H	N	O
Balance	6.02	<0.005	0.062	<0.005	<0.005	<0.005	4.02	0.0035	0.0042	0.038	0.096

Fig. 1: Morphology of TC4 alloy powder

The SLM experiment was carried out with RenAM500 equipment produced by Renishaw Company, and the maximum forming volume of the equipment was 250 mm×250 mm×350 mm. Before SLM processing, the titanium alloy substrate was preheated to 170±2 °C, and the forming chamber was filled with argon, to assure the oxygen content in the chamber was less than 0.05%. The SLM processing parameters are shown in Table 2. A previous experiment showed that the TC4 alloy samples fabricated with these parameters had good formability, and the density of the samples was as high as 99.95%. The frame samples with a length of 50 mm, height of 20 mm, and wall thickness of 1.5 mm were fabricated.

In order to find the difference in microstructure and mechanical properties of samples under different HIP regimes,

Table 2: SLM processing parameters

Layer thickness (mm)	Laser power (W)	Scanning speed (mm·s ⁻¹)	Hatch spacing (mm)
0.04	280	1,800	0.065

Table 3: HIP regimes of TC4 alloy

No.	Temperature (°C)	Time (h)	Pressure (MPa)	Cooling mode
1	900	3	150	Furnace cooling
2	940	3		
3	940	4		
4	960	4		

four sets of HIP regimes were designed, as shown in Table 3. After grinding and polishing, the surfaces of the samples were etched by corrosion solution of 10% HNO₃+5% HF+85% H₂O (in volume). The microstructure of the samples was observed by the Leica DM4000 metallographic microscope and FEI nano450 field emission scanning electron microscope (FESEM). The fracture morphology of SLM TC4 alloy after HIP of 940 °C/3 h/150 MPa was also observed by FESEM. The phase analysis was carried out using D2 phaser X-ray diffraction (XRD). The microhardness was measured by HV-1000AT digital Vickers hardness tester, with 2 N load and 10 s loading time, and the hardness value was the average of 10 measurements. The tensile properties at room temperature were tested by AG-IS250KN universal material testing machine with a tensile rate of 0.45 mm·min⁻¹. An ATO three-dimensional optical scanner produced by German GOM company was employed to measure the deformation of the frames. Finally, through comparing the deformation of TC4 alloy frames before and after hot isostatic pressing, the influence of different hot isostatic pressing regimes on the specimen deformation was studied.

3 Results and analysis

3.1 Effect of HIP on microstructure

The microstructure of SLM TC4 alloy samples is shown

in Fig. 2. It can be seen that the morphology of the sample perpendicular to the building direction (XY direction) is similar to that parallel to the building direction (Z direction): both directions achieve good metallurgical bonding, no cracks or lack of fusion defects are found, but there are few hole defects near β grain boundary with a size about 1 μ m (Fig. 3), which may be related to the residual gas in the powder [19]. The microstructure is composed of fine needle-like phase interlaced with each other. According to TTT curve (isothermal transformation curve) of TC4 alloy [20], different cooling rates can obtain different morphologies and types of microstructures. The cooling rate of cast TC4 alloy is only about 0.5 °C·s⁻¹ [21], the solidification process is so long that the grains have positive growth conditions to form thick strip-shaped α phase with certain orientations. However, the SLM process is a rapid heating and cooling process, and the cooling rate is as high as 10⁴–10⁷ K·s⁻¹ [22], so there is not enough time for the initially formed β phase to transform into α phase, so it can only be transformed into martensite α' phase by shear transformation [23]. Figure 4 shows the XRD diffraction pattern of SLM TC4 alloy, in which no β phase diffraction peaks are found, but martensite α' phase diffraction peaks with close-packed hexagonal structure are seen. The analysis above proves that the fine needle-like structure is martensite α' phase.

Figure 5 shows the microstructure of SLM TC4 alloy after

Fig. 2: Microstructure of SLM TC4 alloy: (a) XY direction; (b) Z direction

Fig. 3: Hole defects in SLM TC4 alloy

Fig. 4: XRD diffraction pattern of SLM TC4 alloy

different HIP regimes. The results show that after HIP, hole defects that can be observed in SLM TC4 alloy are mostly closed, and the acicular martensite α' phase disappears and transforms into α phase and β phase. The microstructure in both directions is similar and there is no obvious difference. When the HIP regime is 900 °C/3 h/150 MPa, α phase appears to be coarse lath or even equiaxed. With the increase of HIP temperature and duration, the morphology of α phase with coarse lath is gradually refined, and the proportion of α phase is gradually reduced. Figure 6 shows the XRD diffraction patterns of SLM TC4 alloy under different HIP regimes. β phase diffraction peak is still too weak to be observed, except a β phase (110) peak (Fig. 6) under 940 °C/3 h/150 MPa. This is because in TC4 alloy, the

content of α phase is dominant, the content of stable β phase is very low, and β phase is distributed in the grain boundaries of α phase in linear or dot shape, so it is difficult to observe the diffraction peak of β phase in the XRD pattern of TC4 alloy. In addition, it can be seen that the diffraction peak intensity of α phase (10-11) in SLM TC4 alloy decreases gradually with the increase of temperature and duration of HIP, which may indicate the content of α phase decrease. This is consistent with the phenomenon observed in Fig. 5.

3.2 Effect of HIP on microhardness

Figure 7 shows the variation curves of microhardness of SLM TC4 alloy under different HIP temperatures and

Fig. 5: Microstructures of SLM TC4 alloy under different HIP regimes: (a) 900 °C/3 h/150 MPa, XY direction; (b) 900 °C/3 h/150 MPa, Z direction; (c) 940 °C/3 h/150 MPa, XY direction; (d) 940 °C/3 h/150 MPa, Z direction; (e) 940 °C/4 h/150 MPa, XY direction; (f) 940 °C/4 h/150 MPa, Z direction; (g) 960 °C/4 h/150 MPa, XY direction; (h) 960 °C/4 h/150 MPa, Z direction

Fig. 6: XRD diffraction patterns of SLM TC4 alloy under different HIP regimes

duration. Because acicular martensite α' phase is a sort of supersaturated solid solution, its microhardness is significantly higher than that of α phase. Therefore, the microhardness of SLM TC4 alloy is the highest, reaching more than 350 HV. After HIP, the microstructure is composed of α phase and β phase, the microhardness decreases. With the increase of HIP temperature and duration, the microhardness decreases from around 320 HV to 300 HV. On the one hand, HIP can effectively reduce the residual stress in the samples; on the

other hand, the relative density of α phase is 0.74 with close packed hexagonal structure with 1 slip plane and 3 slip directions, while the relative density of β phase is 0.68 with its body-centered cubic structure with 6 slip planes and 2 slip directions. Therefore, the plastic deformation ability of β phase is stronger than that of α phase; that is to say, the higher the content of β phase, the lower the microhardness of TC4 alloy^[24]. According to the microstructure shown in Fig. 5, with the increase of HIP temperature and duration, the content of β phase increases, so the microhardness gradually decreases.

Fig. 7: Microhardness of SLM TC4 alloy under different HIP regimes

3.3 Effect of HIP on tensile properties

The strength of SLM TC4 alloys is generally higher than 1,100 MPa, but the elongation can only reach 6%–8%^[25, 26]. After HIP, the changes of tensile properties of SLM TC4 are shown in Fig. 8. It can be seen that after HIP, the tensile strength of the samples perpendicular to the building direction decreases from over 1,100 MPa to 862–902 MPa, and the elongation increases from less than 8.0% to 13.2%–14.3%, compared with SLM TC4 alloy. The similar variation trend can

be observed parallel to the building direction after HIP. The tensile strength decreases from 1,107 MPa to 875–900 MPa, and the elongation increases from 7.3% to 13.5%–14.8%, compared with SLM TC4 alloy, except a slight decrease of elongation under 960 °C/4 h/150 MPa. It can also be seen that after HIP, the strength is decreased by about 20% and the elongation is increased by more than about 70%, and even over 100%, and the strength and the elongation are at the same level in the *XY* and *Z* directions, there is no obvious anisotropy.

Fig. 8: Tensile properties of SLM TC4 alloy with different HIP regimes in different directions: (a) *XY* direction; (b) *Z* direction

The reason for the change of strength and plasticity in SLM TC4 alloy after HIP is that the needle-like martensite α' phase changes to $\alpha+\beta$ phase, so the grain refinement strengthening is weakened, and with the dislocation in martensite α' phase moving, the entangled dislocation is recombined, the density of dislocation decreases to release internal stress^[27], so the plasticity is improved. Moreover, because the strength of β phase is lower than that of α phase, while the plasticity is higher than that of α phase, with the increase of HIP temperature and duration, the ratio of β phase increases so that the strength decreases and the plasticity increases. When the HIP regime is 940 °C/3 h/150 MPa, the tensile strength and the elongation achieve optimal match, are 890 MPa and 13.9% in *XY* direction,

888 MPa and 14.4% in *Z* direction.

The fracture morphology of SLM TC4 alloy after HIP of 940 °C/3 h/150 MPa is shown in Figs. 9 and 10. Figures 9(a) and (b) show the macro fracture morphology of the tensile samples in *XY* and *Z* directions, respectively. The fracture is composed of fiber area and shear lip, and the fracture surface fluctuates greatly. Figures 10(a) and (b) show the micro fracture morphology of the tensile samples in *XY* and *Z* directions, and the circular dimples with different sizes and morphologies can be clearly observed, indicating that the fracture mechanism is ductile fracture, which means good plasticity. The result is consistent with the tensile property testing results shown in Fig. 8.

Fig. 9: Low magnification fractograph of SLM TC4 alloy after 940 °C/3 h/150 MPa HIP: (a) *XY* direction; (b) *Z* direction

Fig. 10: High magnification fractograph of SLM TC4 alloy after 940 °C/3 h/150 MPa HIP: (a) XY direction; (b) Z direction

3.4 Effect of HIP on deformability

For TC4 alloy parts with thin-walled structure fabricated by selective laser melting, deformation may occur in the process of HIP, which would affect the practical application of the parts. Deformation values are based on the first measurement data (before HIP), and evaluating the deviation of the second measurement data (after HIP). The influence of HIP

temperature and duration on the deformation of SLM TC4 alloy thin-walled frames is shown in Fig. 11 and Table 4. It can be seen that after HIP, all four frames have a certain degree of concavity. With the increase of HIP temperature and duration, the maximum concavity gradually increases from 0.154 mm to 0.293 mm. Compared with the wall thickness (1.5 mm), the maximum deformation is between 10.3% and 19.5%.

Fig. 11: Deformation diagram of frames under different HIP regimes: (a) 900 °C/3 h/150 MPa; (b) 940 °C/3 h/150 MPa; (c) 940 °C/4 h/150 MPa; (d) 960 °C/4 h/150 MPa

Table 4: Deformation analysis of frames under different HIP regimes

HIP regimes	Maximum concavity (mm)	Maximum deformation (%)
900 °C/3 h/150 MPa	+0.154	10.3
940 °C/3 h/150 MPa	+0.246	16.4
940 °C/4 h/150 MPa	+0.240	16.0
960 °C/4 h/150 MPa	+0.293	19.5

It can be found that the HIP temperature and duration have different effects on the deformation of the SLM TC4 alloy thin-walled frames. Maintaining HIP duration at 3 h and increasing HIP temperature from 900 °C to 940 °C, the maximum concavity will increase from 0.154 mm to 0.246 mm. Maintaining HIP duration at 4 h and increasing HIP temperature from 940 °C to 960 °C, the maximum concavity will increase from 0.240 mm to 0.293 mm. However, maintaining HIP temperature at 940 °C and extending HIP duration from 3 h to 4 h, the maximum concavity is basically unchanged. From the above discussion, it can be seen that the deformation of the frames increases with the increase of HIP temperature and basically unchanged with the increase of HIP duration.

4 Conclusions

(1) Microstructure of SLM TC4 alloy is characterized by acicular martensite α' phase. After hot isostatic pressing, unstable martensite α' phase decomposes into $\alpha+\beta$ phase, and with the increase of hot isostatic pressing temperature and duration, α phase with coarse lath is gradually refined and the proportion of α phase decreases.

(2) After hot isostatic pressing, microhardness decreases from 355 HV to around 300 HV, tensile strength also decreases from over 1,100 MPa to 862–902 MPa, but the elongation increases from less than 8.0% to 13.2%–14.8%, compared with as-deposited TC4 alloy, and there is no obvious anisotropy. When SLM TC4 alloy is heat treated by 940 °C/3 h/150 MPa hot isostatic pressing, the tensile strength and the elongation in both of XY and Z directions come to about 890 MPa and around 14.0%, which achieves an optimal match. The fracture mechanism of alloy after 940 °C/3 h/150 MPa HIP is ductile fracture.

(3) Hot isostatic pressing can cause concave deformation of SLM TC4 alloy thin-walled frames (wall thickness: 1.5 mm). In this study, the maximum deformation is between 10.3% and 19.5%. The deformation degree increases with the increase of HIP temperature.

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