Effect of multi-pass deformation on microstructure and flow behavior of Ti-6Al-4V alloy fabricated through hot isostatic pressing

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Abstract

Ti-6Al-4V titanium alloy specimens fabricated through hot isostatic pressing (HIP) were made by multi-pass thermal deformation. The temperature was 950 °C, 850 °C and 900 °C for one pass, two passes and three passes, respectively, and each pass corresponded to three strain rate (i.e. 0.01 s\(^{-1}\), 0.1 s\(^{-1}\) and 1 s\(^{-1}\)), in addition, the total reduction in height was 70%. When the true strain was greater than 0.51 and the strain rate was 1 s\(^{-1}\), the stress–strain curves showed the phenomenon of flow softening. X-ray diffraction (XRD) patterns indicated that the diffraction peak of α lattice plane of (002) increased first and then declined with the strain under the strain rate of 0.1 s\(^{-1}\). Besides, the Vickers hardness increased with the strain at the same strain rate and was not sensitive to strain rate. Moreover, when the strain rate was 0.1 s\(^{-1}\), the fraction of LAGBs decreased from two passes to three passes and the microstructure results showed that the fraction of equiaxed α phases increased with the passes increasing. This is due to the strain energy increased as the increasing passes which could provide driving force for dynamic recrystallization (DRX). At the same amount of deformation, increasing the strain rate, the slender lamellar α bended and the equiaxed α grains gradually took place of the discontinuous lamellar α. Additionally, the spheroidization mechanism of lamellar α phase was mainly through grain boundaries bulging. After completing three passes deformation, the average grain size was approximately 12 μm.

1. Introduction

Due to its outstanding performance of high strength, low density and high corrosion resistance, Ti-6Al-4V alloy has been extensively used in the fields of aviation and arms manufacture [1, 2]. The performance of titanium alloy is more demanding in extreme conditions. Generally, the excellent performance is determined by the microstructure of titanium alloy [3–5]. So reliable forming methods and near net forming technique have become the development direction for titanium alloy components. Nevertheless, the usage of titanium alloys is restricted owing to their poor formability or processability, such as limited plasticity and high hardness [6]. Because of poor thermal conductivity of titanium alloy, during forming or thermal processing, high local temperatures can lead to coarse grains. Particularly in casting products, it is easy to see segregation, coarse grain, impurities and other defects, so the casting method would not satisfy high performance requirements. As for forging titanium alloys, though exceptional mechanical properties are attained, serious waste of raw materials and high cost are the major factors blocking their extensive use [7]. Therefore, powder metallurgy (PM) methods have become the focus for researchers.

As a method of powder metallurgy of titanium alloy, hot isostatic pressing (HIP) method provides a shortcut for obtaining high performance titanium alloy because of microstructure uniformity and low cost [8]. Compared to casting and swaging products, HIP methods combine the salient features of complete densification and full utilization of raw materials [9]. In addition, most researches focus on the effect of HIP parameters/powder diameter on the microstructure evolution or mechanical properties. Zeng et al [10] studied the powder diameter on the microstructure variation and pointed out that smaller powder diameter correspond to better microstructures macro-performance. Abu-Issa et al [11] pointed out that the best mechanical properties...
(YS:0.9GPa/UTS:1.0GPa/EI:14%) could be acquired with a moderate cooling rate from 850 °C HIP. Benzing et al [12] obtained excellent mechanical properties by a new HIP method and found that the porosity was reduced remarkably. Nonetheless, few researches concentrate on the effect of thermomechanical processing after HIP on the microstructures and mechanical performance. In addition, thermal processing is necessary in the actual production process for removing the porosities and void caused by HIP, and the uniform microstructure could be obtained.

Multi-pass deformation is an effective route to refine grain and enhance uniformity of microstructure by breaking up the primitive microstructures [13, 14]. So desirable properties of titanium alloy components could be got. Tang et al [15] found that the grain refinement could reach through multi-pass high-pressure sliding. Liu et al [16] pointed that multi-pass compression played an important role with respect to microstructure evolution. Wang et al [17] discovered that heterogeneous microstructure could be weaken with the increase of forming pass. Based on above, multi-pass thermal deformation was conducted to investigate the microstructure of Ti-6Al-4V titanium alloy prepared by HIP process. The multi-pass deformation experiments with the temperature ranged from 850 °C to 950 °C and the strain rate ranged from 0.01 s⁻¹ to 1 s⁻¹ were implemented and the total deformation amount was 70%. The variation of α phases and dynamic restoration feature were systematic studied. The purposes of present work is to study the evolution of α phases during multi-pass deformation, such as the size and amount of α phase variation. Which could provide a better understanding on the microstructures change of Ti-6Al-4V alloy during thermal deformation.

2. Experiment method

Ti-6Al-4V alloy powder for the hot isostatic pressure experiment was produced by Sino-Euro Materials Technologies of Xi’an Co., Ltd. The nominal composition of Ti-6Al-4V powder are given in table 1. A scanning electron microscope (SEM) image of the alloy powder is shown in figure 1. It could be seen that the powders size varies from 80 to 100 μm, and the mean diameter of Ti-6Al-4V alloy powder approximates to 90 μm. HIP device was used to fabricate Ti-6Al-4V specimens with the temperature of 920 °C and the pressure of 120 MPa. After finishing the HIP process, the samples were retained in the furnace for 5 min to make the compositions more evenly distributed and then cooled with the furnace.

The samples with the size of 8 mm in diameter and 12 mm in height were extracted from the HIPed parts, and the β-transus temperature (the temperature which β transforms to α + β absolutely) was 950 ± 5 °C determined by metallographic method and differential scanning calorimetry (DSC), as shown in figure 2. Then the Gleeble-1500 apparatus was used for multi-pass thermal compression experiment with the temperature ranging from 850 °C to 950 °C and the strain rate ranging from 0.01 s⁻¹ to 1 s⁻¹. Before hot compression tests, the specimens were heated to the respective deformation temperature and soaked for 2 min to heat evenly.

In figure 1, the total reduction in height was 70%. Besides, after the one pass deformation at the temperature of 950 °C, 40% (4.8mm) height reduction was reached. The height reduction for two passes and three passes was 20% (2.4mm) and 10% (1.2mm) respectively to the temperature of 850 °C and 900 °C, respectively. Water quenching was selected as the cooling model when the hot compression experiments completed. The specimens were sliced along the compacted axis to observe the microstructure. X-ray diffraction (XRD) experiments were performed using Rigaku smartLab-3kw to research the structures of initial and deformed samples. Using optical microscope (OM) to observe metallographic structures, electron backscatter diffraction (EBSD) tests were conducted on the Hitachi SU5000 scanning electron microscope (SEM) which equipped with EBSD system to study the microstructure evolution. The EBSD data were processing by the orientation imaging microscopy (OIM) software. Before observation, grit papers were used to grind the samples following by polishing and then etching solution comprised of a blended acid of HF, HNO₃ and H₂O₃:6:91 in volume ratio was used to corrode the specimens. Vickers microhardness of HIPed Ti-6Al-4V samples test was implemented by using UHL VMHT. 200gf load was set and the dwell time was 15 s. Each sample was tested 15 times and the average value was taken.

3. Results and discussions

3.1. Stress and strain analysis

The true stress-strain curves for Ti-6Al-4V alloy deformed at different strain rate are shown in figure 3. It could be found that the true stress increased with the strain rate increasing. Formula (1) could explain this phenomenons which was used for BT3-1 titanium alloy by Balasundar [18].

\[ \dot{\varepsilon} = b \rho \upsilon \]  

(1)

In formula (1), \( \dot{\varepsilon} \) expresses the strain rate, \( b \) is burgers vector, \( \rho \) is dislocation density and \( \upsilon \) is sliding velocity of dislocation. Increasing the strain rate, the sliding velocity for dislocations would increase accordingly. But the
slip velocity does not increase indefinitely because of the existence of lattice friction or the influences of solute atoms. In other words, there is a maximum sliding velocity for dislocations glide. When the sliding velocity reached maximum value, a state of equilibrium was reached because of the combination of the strain rate effect and the lattice hindrance effect. So when the strain rate increased continuously and $\nu$ stayed the same, the dislocations density would rise sharply and the true strain increased corresponding.

Another feature could be seen in two passes or three passes that, when the strain rate was $1 \text{s}^{-1}$, there was an obvious dynamic softening in stress-strain curves. This is because the dynamic softening effect is larger than the work hardening effect. Rising the strain rate, there was not enough time here for dislocation sliding to annihilate, so dislocation density would increase. In the meantime, the distortion energy would be greater than that at low strain rates and the degree of dynamic softening also increased correspondingly. The same results were also observed by Bobbili et al [19]. Besides, in the curves where the strain rate was less than $1 \text{s}^{-1}$, after the peak, the curves showed no significant dynamic softening. This is due to the joint effect of thermal effect and distortion energy [20].
3.1. Microstructure evolution

3.1.1. Microstructure of sintered Ti-6Al-4V alloy

Figure 4 were the metallographic image, image quality (IQ) picture, EBSD images and corresponding misorientation angles of sintered Ti-6Al-4V alloy before multi-pass deformation. In figure 4 (c), the difference in

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**Figure 3.** True stress-strain curves of compression specimens.

**Figure 4.** The initial microstructures of Ti–6Al–4V alloy: (a) optical image, (b) image quality (IQ) picture, (c) and (d) EBSD micrograph and corresponding misorientation angles.
color indicates the different orientation of the crystal, and the coloring principle is displayed in figure 4 (c). The grain boundaries with misorientation less than 2° does not take into consideration because of the data noise [21]. The white and black lines represent the low angle grain boundaries (LAGBs) and high angle grain boundaries (HAGBs). The grain boundaries with misorientation angles of 2°–15° are defined as LAGBs while the grain boundaries with misorientation angles large than 15° are described as HAGBs. It could be found that initial microstructure consisted of coarse lamellar α and equiaxed α grains, and some α colonies could be found. The volume fraction of LAGBs was high, as shown in figure 4 (d). Besides, There were no visible pores appeared, so the higher densification samples could be obtained by means of HIP process.

3.1.2. Effect of deformation amount on microstructure

The microstructure images of Ti-6Al-4V alloy deformed at strain rate of 0.1 s⁻¹ under different deformation passes were shown in figure 5, and the observation Point was insert in the top right corner. The compression direction of the following pictures was vertical. Figure 5(a) was the metallographic diagram of the sample deformed at 950 °C, with a height reduction of 40% (4.8 mm). It composed of acicular α grains and a small amount recrystallized grains, shown in the red dotted rectangle. Due to the deformation temperature was above the β-transus temperature and the supercooling was high, some acicular martensite formed during cooling. Moreover, the lamellar α wove with each other and the grain boundary α was un conspicuous, so the microstructure after one pass was weave-basket structure. Figures 5(b), (c) were microstructure images of samples deformed at two passes and three passes, respectively. The temperature and the amount of deformation were 850 °C and 20% for two passes and 900 °C and 10% for three passes. In figures 3(b) and (d), the bright silver represented the β phases and the black indicated the α phases in BSE image of (d). It could be found that some dynamic recrystallization (DRX) grains and equiaxed α increased markedly compared with figure 5(a), shown in the red ellipse. The mean size of DRX grains was about 3.47 μm measured by line intersection method. Besides, the lamellar space of part α was decreasing and the merger of lamellar α phases would appear. As the amount of deformation continued to increase, shown in figure 5(c), more equiaxed α and short rod-like α appeared and the grain size reduced simultaneously. Additionally, the amount of lamellar α reduced sharply.

Figure 5. Optical micrographs of HIPed Ti-6Al-4V alloy deformed at strain rate of 0.1 s⁻¹: (a) one pass, (b) two passes, (c) three passes and (d) BSE image of two passes.
The XRD patterns of the Ti-6Al-4V alloy deformed at different compression amount under the strain rate of 0.1 s\(^{-1}\) and their corresponding microhardness versus true strain were shown in figure 6. In figure 6(a), the diffraction peak intensity at the \(\alpha\) lattice plane of (0002) and (10–11) increased first and then decreased with the deformation while the other diffraction peak intensity changed little. The variation of peak intensity signified the change of corresponding phases. The diffraction peak of \(\alpha\) lattice plane of (0002) correspond to the \(\alpha + \alpha'\) phases structures. In other words, the content of \(\alpha\) phases increased first and then declined. This is due to the lowest deformation temperature of the two passes, for 850 °C. The temperature descended and the deformation increased during the process from one pass to two passes, so the amount of \(\alpha\) phases would increase accordingly and the phase transition \(\beta \rightarrow \alpha/\beta \rightarrow \alpha'\) could explain the shift of the diffraction peak of \(\alpha\) lattice plane of (0002). In addition, during the process from two passes to three passes, rising temperature would promote the transition of \(\alpha \rightarrow \beta\), so the diffraction peak decreased corresponding. The diffraction peak of \(\alpha\) lattice plane of (10–11) consisted of \(\alpha'\) and \(\alpha' + \beta\) phases. \(\alpha'\) was an intermediate phase during the phase transition \(\beta \rightarrow \alpha'\) and \(\alpha'\) distortion was related to V element [22]. Figure 6(b) is the relationship of Vickers hardness against true strain. It could be found that the microhardness increased with the strain at the same strain rate. After finishing the three passes deformation, the maximum hardness values were 318 HV, 309 HV and 312 HV corresponding to strain rate of 0.01 s\(^{-1}\), 0.1 s\(^{-1}\) and 1 s\(^{-1}\), respectively. Apparently, microhardness value was less sensitive to strain rate. It is known that Ti-6Al-4V alloy mainly consists of hard \(\alpha\) phase and soft \(\beta\) phases. So this might be related to the decrease of grain size and the content of \(\alpha\) phases. Reducing grain size would increase the amount of grain boundaries and the resistance to dislocation motion increased. Besides, solute atoms entering the lattice would also prevent the dislocation from moving [23]. So the microhardness would improve as the true strain increased.

The EBSD maps and misorientation angles of the Ti-6Al-4V alloy deformed at 850 °C and 900 °C with the strain rate of 0.1 s\(^{-1}\) were shown in figures 7(a), (c) and (b) (d) corresponding to two passes and three passes, respectively. In figures 7 (a) and (c), the grain size was larger and the volume fraction of LAGBs accounted for a larger proportion. In addition, it could be seen that, after finishing the three passes deformation, more DRX grains appeared and the grain size was obviously refined, about 12 μm, moreover, the volume fraction of LAGBs decreased as the strain increased. Obviously, the change of the fraction of LAGBs is associated with degree of dynamic restoration. This is due to that, during the processes of thermal deformation, a critical strain is required for DRX. In the process of two passes deformation, the temperature of thermal compression was relatively low and the driving force was not enough. The restoration mechanism was mainly dynamic recovery (DRV). So, when the three passes deformation completed, the strain energy was larger enough to trigger DRX. Besides, formation of the grains without distortion needed to consume the dislocation. Finally, the fraction of LAGBs declined.

The OIM map of grain 1 in figure 7(a) was used to illuminate the formation process of DRX grains, as shown in figure 8. In figure 8(a), there are some different gray levels in the same grain and this is associated with subgrain formation. The becoming of low angle grain boundaries was due to the accumulation of dislocations [24]. Increasing the amount of deformation, the low angle grain boundaries would turn into high angle grain boundaries by consuming the growing dislocations. The subgrains rotation could be seen by the three-dimensional hexagons in figure 8(a). Further, the subgrains could chane into DRX grains which was the typic
3.1.3. Effect of strain rate on microstructure
Since the strain rate has little influence on one pass deformation and the microstructures are mostly Widmannstätten structure and basket-weave microstructure, the deformation characteristics of one pass are not discussed here. Optical micrographs of Ti-6Al-4V alloy deformed at two passes were shown in figure 9, and the continuous dynamic recrystallization (CDRX) mechanism. The line graph of misorientation angle along the AB in figure 7(a) firmly confirmed this.

3.1.3. Effect of strain rate on microstructure
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observation point was superimposed in the upper right corner. Figures 9(a) and (b) corresponded to strain rates of 0.01 s$^{-1}$ and 1 s$^{-1}$, separately. The amount of deformation and hot compression temperature were 60% (7.2 mm) and 850 °C, respectively. In figure 9(a), there are a small amount of DRX grains occurred. The lamellar α gradually transformed into ellipsoidal grains. Additionally, the necklace-like grain boundary α (GBα) appeared, shown by the red dotted line in figure 9(a). With the increase of strain rate, the DRX grains increased, indicated by ellipse in figure 5(b) and grain size decreased simultaneously. Besides, the lamellar space of α phases decreased and the GBα was not obvious. In figure 9(b), Obviously spheroidizing and the discontinuous α grains could be found. This could be explained as the dislocations density increased with the strain rate. Thus, more nucleation location appeared, and the increased distortion energy also provided the driving force for nucleation [25]. In addition, more α phases occurred and the martensite decomposition or the β phase transition should be responsible for the result.

Figure 10 was the OM images of Ti-6Al-4V alloy deformed at three passes under different strain rates. The amount of three passes was 70% (8.4 mm) and the thermal deformation temperature was 900 °C. Figures 10(a) and (b) corresponded to strain rates of 0.01 s$^{-1}$ and 1 s$^{-1}$, respectively. In figure 10(a), the amount of deformation increased (increased by 10%), in the same time, the compacted temperature also increased, and the volume fraction of spheroidized α showed significant change compared with figure 9(a). The reason for this phenomenon was that increasing the temperature would provide driving force for recrystallization grains nucleation and growth, meanwhile, the increase of deformation amount also contributed to the increase of distortion energy. So the extent of DRX strengthened. Meanwhile, compared with figure 5(c), there are many slender lamellar α grains and discontinued α grains in figure 10(a), shown in the red rectangle and the arrow. This could be explained that, for the three passes deformation, increasing the strain rate, the slender lamellar α

![Figure 9](image_url)

**Figure 9.** OM images of Ti-6Al-4V alloy deformed at two passes: (a) 0.01 s$^{-1}$, (b) 1 s$^{-1}$.

![Figure 10](image_url)

**Figure 10.** OM micrographs of Ti-6Al-4V alloy deformed at three passes: (a) 0.01 s$^{-1}$, (b) 1 s$^{-1}$.
with the same misorientation would merge with each other. In addition, the bending discontinuous lamellar α grains could be found in figure 10(b).

There are two mechanisms for spheroidization of lamellar α phase. One way of spherification was through termination migration and another was via boundary grooving, as noted in Zherebtsov [26]. The termination migration phenomenon mainly occurred during heat treatment while the boundary grooving phenomenon could appear in hot compression and heat treatment, reported by Jiang et al [27]. In figures 10(a) and (b), the deformation temperature was 900 °C. There were a lot of discontinuous short rod-like α, so the spheroidization mechanism might be boundary grooving. It was known that, in the plane of lamella α phases, there were many defects such as dislocation, grain boundary and subgrain boundary. During thermal deformation process, elements in the β grains diffused into these defects and the grooves deepened gradually. Moreover, the diffusion of elements intensified with the increase of deformation temperature or by dislocation that acted as diffusion pipe [28]. This could be verified that the volume fraction of short rod-like α grains was high in three passes deformation compared with two passes. Eventually the grooves were pinched off and short rod-like α appeared. Increasing the strain rate, the DRX grains increased significantly and some bending discontinued α grains occurred, shown in figure 10(b). This is due to flow softening during hot compression, and the phenomenon of flow softening could be seen in three passes with the strain rate of 1 s\(^{-1}\) in figure 3. The kinking was formed when α colonies were 45° from the compression axis, and the bending of lamellar α was associated with crystal orientation of lamellar α and the stress state around them. The same result was observed by Xiao et al [29]. It could be speculated that, during the thermal deformation process, the lamellar α underwent bending deformation and then gradual fragmented. Finally, the α grains with smaller size were formed.

3.1.4. Microstructure evolution during multi-pass deformation

The evolution laws of microstructure of Ti-6Al-4V alloy under the condition of multi-passes deoreation were shown in figure 11. The initial microstructures consist of equiaxed and lamellar α phases, shown in No.1. After finishing the one pass compression, the equiaxed α grains were elongated but part of the grain boundary α still existed. In addition, the colonies α paralleled or wove with each other that were described as Widmannstatten structure. In the meantime, the dislocations gathered around grain boundaries and the substructures could appear near the vicinity of grain boundaries, shown in No.2 and 3. When the two passes deformation completed. The grain boundary α disappeared gradually and the rod-like α grains took place of part of lamellar α. This could be found in figure 9 and figure 10. Meanwhile, some kinking α grains would appear. This may be related to the orientation of the grains and the stress state around them. When the three passes deformation finished. Most of rod-like α transformed into finer equiaxed α and the metal flow line were apparente, shown in figure 10(b).
4. Conclusion

(1) When the strain rate was 1 s\(^{-1}\) and the true strain was greater than 0.51, the stress-strain curves showed distinct softening phenomenon. Besides, in the curves where the strain rate was less than 1 s\(^{-1}\), after the peak, the curves showed no significant dynamic softening.

(2) X-ray diffraction patterns indicated that the diffraction peak of \(\alpha\) lattice plane of (0002) increased first and then declined with the strain under the strain rate of 0.1 s\(^{-1}\). Moreover, the Vickers hardness increased with the strain at the same strain rate and was not sensitive to strain rate. The EBSD images showed that the fraction of LAGBs decreased from two passes to three passes. Besides, the equiaxed \(\alpha\) phases content increased with deformation amount with the strain rate of 0.1 s\(^{-1}\).

(3) The strain rate has a great influence on the microstructure. The slender lamellar \(\alpha\) bended and the equiaxed \(\alpha\) grains gradually took place of the discontinuous lamellar \(\alpha\). The spheroidization mechanism of lamellar \(\alpha\) phase was mainly through grain boundaries bulged. After the three passes deformation completed, the average grain size was approximately 12 \(\mu\)m.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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