Effects of Heat-Treatment on Microstructures and Mechanical Properties of Hot Deformed Ti-6Al-4V Alloy

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Abstract. In this paper, Ti-6Al-4V alloy were successfully fabricated using a Spark Plasma Sintering (SPS) process, thermal deformation and heat treatment process. SPS sintering is divided into two steps: The first step is low-temperature molding at 600°C and 300 MPa, and the second step is thermal deformation at 800°C, with a deformation of 70%. The effects of solution treatment and aging process at temperatures above and below the \( \beta \) transformation point on the microstructure and mechanical properties of Ti-6Al-4V are investigated. The study found that the mechanical properties were better after treatment (900°C, 30 min+ water quenching +550°C, 4 h).

1. Introduction
Spark plasma sintering technology is a new type of powder metallurgy technology developed in recent years. It is heated by plasma generated by pulse current, so compared with traditional hot-pressing sintering and pressureless-sintering, it has the advantages of fast heating rate, short sintering time, high production efficiency and fine and uniform product structure\textsuperscript{[1-2]}. Spark plasma sintering technology has been widely used in the preparation of functionally graded materials, thermoelectric materials, ferroelectric materials and amorphous alloys. As a typical \( \alpha+\beta \) two-phase titanium alloy, Ti-6Al-V alloy has been produced by various processing methods, such as casting, selective laser melting, powder metallurgy and especially spark plasma sintering\textsuperscript{[3-4]}.

According to previous reports, the microstructure of Ti-6Al-V alloy includes equiaxed structure, Widmanstatten structure, acicular structure and basket-like structure\textsuperscript{[5]}. Among them, the equiaxed structure and Widmanstatten structure depend on the processing temperature, and the acicular structure and the basket-like structure are closely related to the subsequent heat treatment process\textsuperscript{[11]}. Widmanstatten structure is composed of lath \( \alpha \)-Ti and intergranular \( \beta \) phase\textsuperscript{[5-6]}. When the processing temperature exceeds the \( \beta \) phase transition temperature, the Widmanstatten structure will form after
Ti-6Al-V alloy is cooled to room temperature. The Widmanstatten structure usually leads to poor ductility of the alloy[7,8].

In this study, in order to improve the mechanical properties of Ti-6Al-4V alloy, we successfully prepared Ti-6Al-4V alloy by combining spark plasma low temperature and high pressure sintering and spark plasma thermal deformation. The effects of different heat treatment methods on the structure and mechanical properties of Ti-6Al-4V alloy were also investigated.

2. Material and method

Ti-6Al-4V powder (99.8%, Beijing Xingrongyuan Technology) is used to produce bulk Ti-6Al-4V alloy. The size of Ti-6Al-4V powder is 15–53 μm. Use a carbide alloy dies with an inner diameter of 20mm to consolidate the powder in the spark plasma sintering system (Sojitz Machinery Corporation, Japan). Sintering temperature, pressure and holding time are 600°C, 300 MPa and 5 min respectively. The heating rate is 100°C/min. After being consolidated into a bulk with a diameter of 20 mm and height of 10 mm, a C-C dies with an inner diameter of 30 mm is used for thermal deformation in the SPS system. The deformation temperature is 800°C and the loading speed is 20 kN/min. Then, the samples were heated to 900°C and held for 5 minutes. The heating rate of the deformation process was 50°C/min. We performed heat treatment on the deformed samples, and the heat treatment process is shown in Table 1.

Use X-ray diffraction (XRD) to analyze the phase composition of the sample (X’ Pert PRO MPD, PANalytical B.V., Netherlands). The relative density of the samples is measured by Archimedes principle. The morphology of the sample was observed by Scanning electron microscope (SEM, Hitachi S-4800N, Hitachi, Japan) and Optical microscope (OlympusPME-3). The tensile test was done on the YHS-229WJ-30 kN universal testing machine with a strain rate of 10^{-3} s^{-1}. In order to ensure the accuracy of the experiment, 3 samples in each group are measured. Additionally, the fracture surfaces of the tensile specimen were also observed by SEM.

Table 1. Heat treatment process of specimen

| Sample | Solution treatment | Cooling way | Aging (annealing) treatment | Cooling way |
|--------|-------------------|-------------|----------------------------|-------------|
|        | Temperature (°C)  | Time (min)  | Temperature (°C) | Time (h) |
| S1     | -                 | -           | 550               | 4         | AC         |
| S2     | 900               | 30          | WQ                | 550       | 4         | AC         |
| S3     | 1000              | 30          | WQ                | 550       | 4         | AC         |

3. Results and discussion

The RDs of the annealed and solution-aged Ti-6Al-4V are shown in Figure 1a. As shown in the figure, the RDs of the bulk material prepared for the 600°C/300 MPa/5 min sintering process has reached 95%. Due to the high RDs obtained under this condition, the bulk material will not be broken during the subsequent thermal deformation treatment. The RDs of Ti-6Al-4V that has been thermally deformed increases significantly and is greater than 99.6%. Because thermal deformation can eliminate the cavities inside the sintering bulks, the relative density of the material after deformation are greatly improved, which has a great impact on the improvement of material properties. Figure 1b shows the XRD spectra of the annealed and solution-aged Ti-6Al-4V alloys respectively. The XRD results show that the Ti-6Al-4V alloy after annealing treatment and solution aging treatment has no
obvious β diffraction peak, indicating that the β phase has been transformed into the secondary α' phase during the above treatment.

![Figure 1](image)

**Figure 1.** a: the relative density of Ti-6Al-4V alloy under different process conditions and b: X-ray diffraction patterns of the annealed and solution-aged samples.

The SEM micrographs of the original powder of Ti-6Al-4V alloy and the preformed bulk material are shown in Figure 2. It can be seen from Figure 2a that the Ti-6Al-4V alloy powder is spherical. The morphology of the bulk material prepared for the conditions of lower sintering temperature and higher axial pressure is shown in the Figure 2b. In the preformed bulk material, the atomic amplitude increases and diffuses, forming a sintered neck and gradually growing. The holes shrink as the sintering neck grows, and the density of the material increases.

![Figure 2](image)

**Figure 2.** SEM micrograph of a shows the original Ti-6Al-4V powder and b: the preformed Ti-6Al-4V bulk.

The temperature $T_{β}$ of Ti-6Al-4V phase transition was determined to be 955°C by metallographic calibration method, so 900°C and 1000°C were selected as the solution temperatures. As shown in Figure 3a, after solution treatment by 900°C for 30 min and water-quenching, the microstructure is composed of primary equiaxed α phase and fine acicular α' martensite. As shown in Figure 3b, after solution treatment by 1000°C for 30 min and water-quenching, the microstructure of the coarse
original β-grains was distributed with a single fine acicular martensite, and the martensite was interlaced in the grain.

![Figure 3](image1.png)

**Figure 3.** OM micrograph of a: 900°C for 30 min, water-quenching, and b: 1000°C for 30 min, water-quenching.

Figure 4 shows the microstructure of Ti-6Al-4V under an optical microscope after solution treatment of different temperatures for 30 minutes and aging at 550°C for 4 hours. As shown, after aging at 550°C for 4 hours, short rod-shaped precipitates with different degrees of dispersion appeared inside the microstructure. According to previous reports, the short rod-shaped precipitates were β-phase\(^5\).

![Figure 4](image2.png)

**Figure 4.** OM micrograph of a: 900°C for 30 min+ quenching water + 550°C for 4 hours and b: 1000°C for 30 min+ quenching water + 550°C for 4 hours.

Figure 5 shows the room temperature tensile results of Ti-6Al-4V after different heat treatment processes. The elongation of the sample after solution treatment decreased, which may be attributed to the transformation of β phase into acicular secondary α' phase after water quenching. The strength of the material increases after solution aging treatment for the following reasons: (1) The secondary α' transformed after solution treatment contributes to the strength of the material; (2) The nucleation position of α' phase is in β phase instead of at the interface of primary α and primary β, which reduces stress concentration and reduces the possibility of abnormal fracture of the material.

![Figure 5](image3.png)
Figure 5. Tensile properties at different heat treatment processes.

Figure 6 shows the room temperature tensile fracture of Ti-6Al-4V under different heat treatment processes. As shown, after the heat treatment of S1, S2, and S3, the network dimples are evenly distributed over the fracture surface. It can be seen that these fractures are ductile fractures, and the size and depth of the dimples of the samples of different heat treatment processes is not much different.

Figure 6. Tensile fracture morphology at room temperature a: 550°C/4 h, b: 900°C/30 min+550°C/4 h and c: 1000°C/30 min+550°C/4 h.

4. Conclusions

(1) The relative density of the bulk material prepared under the condition of 600°C/300 MPa/5 min is 95%, and the relative density of Ti-6Al-4V processed by different heat treatment processes is greater than 99.5%.

(2) After solution treatment in the α+β dual-phase region for 30 minutes, and then water quenching, the microstructure is the primary equiaxed α phase and fine acicular secondary α' phase. After solution treatment in the β single-phase region for 30 minutes, and then water quenching, the microstructure is a single fine acicular secondary α' phase distributed inside coarse original β grains.

(3) Tensile experiments showed that Ti-6Al-4V, which was solution treated at 900°C for 30 min, water quenched, and aged at 550°C for 4 h, had good mechanical properties, with a yield strength of 1043 MPa, a tensile strength of 1101 MPa and an elongation of 13.5%.
5. References

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