Softening Behavior and Mechanism of Laser Rapid Forming Ti-6Al-4V Alloy Deformed at the High Temperature

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Abstract. Using the gleeble-1500 thermal simulator, the high temperature quasi-static compression experiment on cylindrical samples of Laser Rapid Forming Ti-6Al-4V(LRF TC4) titanium alloy have been conducted. Softening behaviour and mechanism of LRF TC4 alloy deformed at different high temperatures were investigated by means of optical microscope (OM) and transmission electron microscope (TEM). It shows that, the strength of LRF TC4 was observably reduced with the rise of the deforming temperature, showing stronger thermal softening effect and superplasticity ability. Microstructure analysis on the samples deformed at the temperature from 400°C to 900°C showed that the original acicular microstructure of LRF TC4 has two significant changes: Acicular microstructure bends at the temperature from 400°C to 600°C, while acicular microstructure bends drastically, fragmented, spheroidized at the temperature from 700°C to 900°C. No matter bending, fragmentation, or spheroidizing of acicular microstructure, stress in the deformed sample is released. Therefore, these three factors and their superimposition action were the source of thermal softening of LRF TC4 deformed at the high temperature.

1. Introduction

The ti-6al-4v (TC4) alloy is a type of α+β double-phase titanium alloy. Due to its strong specific strength, high corrosion resistance, low elastic modulus and good thermal strength, TC4 alloy is widely used in aerospace, weapon equipment and medical devices. TC4 alloy demonstrates high strength although at the temperature range of 400~500°C. It is an excellent material for manufacturing important parts of aero-engine blades and engine nozzles. There are two main methods for the production of TC4 alloy, one is the traditional casting and forging methods, and the other is the most advanced laser rapid forming method up to date. Laser rapid forming method is a rapid material fabrication method has developed for the past three decades. Due to its near net forming, Laser Rapid Forming (LRF) has advantages in almost no waste and fast fabrication of components with complex shapes [3]. In addition, the fabricated material properties are comparable, or even better than those produced by traditional casting and forging methods [4-6]. Laser Rapid Forming (LRF) technology has become an effective method for the fabrication of components with complex shapes and rare materials.

Currently, there are many studies on the high-temperature mechanical behavior and microstructure evolution of TC4 fabricated by traditional methods [7-10], for the LRF TC4, many researches focus on the control and optimization of its forming process parameters, room-temperature mechanical properties and its microstructure [11-18], very few research involving its softening behavior and softening mechanism at high temperature. Therefore, the study of the high-temperature mechanical behavior and softening mechanism of LRF Ti-6Al-4V (LRF TC4) has high academic impact and engineering application value.
In this paper, a gleeble-1500 thermal simulator was used to conduct a quasi-static high-temperature compression experiment on the cylindrical sample of LRF TC4 titanium alloy, in order to understand the influence of temperature on the mechanical behavior of LRF TC4 titanium alloy, and to explore the softening behavior and mechanism of the material when deformed at high temperature, which has significant value to the engineering application of LRF TC4 titanium alloy.

2. Experimental Materials and Methods

2.1. Experimental Materials and Treatment Methods
The LRF TC4 alloy used in the experiments were purchased from Xi’an Platinum Laser Forming Technology Co., Ltd., and the forming material was TC4 spherical powder with particle size of -100+150 mesh. The composition of powder is listed in the Table 1. The powder is dried before forming under vacuum at about 120°C to remove moisture to reduce the influence of water on the rapid laser deposition process. The rapid prototyping laser system is Rofin-Sinar CO2, including the following functions/parameters: The laser power was 7 KW, the scanning speed was 10-15 mm/s, the powder rate was 15-30 g/min, the protection gas flow was 9-12 L/h and the spot diameter was 6 mm. The scanning was zigzag along the direction of 45° with the X-axis (the direction of the red line arrow in Figure. 1), and the Z-axis direction was the deposition direction. The size of the forming material was 110 mm×110 mm×40 mm, as shown in Figure. 1. The microscopic morphology is shown in Figure 2. Figure 2a shows the low-magnification metallographic micromorphology, forming coarse columnar crystals nearly parallel to the deposition direction. Figure 2b shows the morphology of scanning electron microscopy (SEM). The parallel α bundles in the coarse columnar crystals form a mixture of the Weiss structure and the basket structure. There are defects as well such as holes left by the escape of bubbles and incomplete melting of the powder during the TC4 material rapidly formed by laser.

| Table 1. Chemical Constitution of TC4 powder (ω / %) |
|---|---|---|---|---|---|---|---|---|---|
| Al  | V  | C  | Si | Fe | H  | N  | O  | Ti |
| 6.02 | 4.00 | 0.056 | 0.039 | 0.15 | 0.005 | 0.033 | 0.14 | Bal. |

Figure 1. Schematic diagram of LRF TC4 formed and direction of the LRF TC4 sample

Figure 2. Microstructure of the LRF TC4 sample. (a) Optical microscope (OM) image with low-magnification; (b) SEM image shows the microstructure in better detail.
2.2. Experimental Methods
The deposited TC4 alloy without any subsequent treatment after laser rapid forming, was wire-cut to form the quasi-static compression cylindrical sample along the 45° that is away from the laser scanning direction (as shown in Figure 1). The size of the sample is φ8 × 12 mm.

The gleeble-1500 thermal simulation machine was used to conduct quasi-static compression test on the cylinder sample with the size 8 mm × 12 mm at the temperature of 400°C-900°C, and the strain rate was 0.01 s⁻¹. The sample was quickly cooled by water after deformation.

The original samples were cut along the axis by a wire cutting method. After sandpaper grinding with different particle sizes, they were mechanically polished to the mirror surface and corroded with klingdt reagent (5% HF, 20% HNO₃, 75% H₂O). The samples were then characterized for microscopic morphology using a LEICA DMi5000 M metallographic microscope (OM) and a Quanta 200 scanning electron microscope (SEM).

Small pieces (2×2×0.3 mm) of the central deformed recovery sample profile were cut with wire cutting. After mechanical grinding to 50 μm, and were further reduced to 5~20 nm thickness by ion to prepare TEM films. The microstructural characteristics and changes of the deformed sample were measured with Tecnai G2 TF30 s-twin field emission transmission electron microscope (TEM) system.

3. Experimental Results and Analysis

3.1. High Temperature Mechanical Behavior of LRF TC4
Figure 3 shows the quasi-static compressive stress-strain curve of LRF TC4 alloy at the strain rate of 0.01 s⁻¹ and the temperature of 400°C-900°C. Under quasi-static compression, the material strength decreases with the increase of temperature, while the plasticity increases. Meanwhile, it is clear that the material strength is about 850 MPa when the material is quasi-static compressed at 400°C, which is 220 MPa less than the strength at room temperature. This indicates a strain-strengthening effect at such temperature. This demonstrates that the material has a high softening resistance at 400°C although it is fabricated by laser in a rapid three-dimensional manner. At 500°C, the strain-strengthening effect of the material disappears, and the flow stress nearly shows a plateau steady change. This indicates that the strengthening effect caused by dislocation multiplication and dislocation interaction due to deformation counteract the softening effect caused by temperature rise, which makes the flow stress of the material present steady-state change in the plastic deformation stage. When the deformation temperature exceeds 500°C, the material shows an obvious softening effect and the material strength decreases significantly. Especially when the temperature exceeds 700°C, the material strength decreases more remarkably, but when the temperature is over 800°C, in particular when the deformation temperature is 900°C, the gradient of strength decreasing with the increase of strain becomes smaller, and it even enters into the steady-state change mode from the beginning of deformation.

Another remarkable feature can be observed from the compression stress-strain curves of LRF TC4 samples under the condition of quasi-static compression and different temperature: With the increase of deformation temperature, the plasticity of materials increases significantly compared with that of the quasi-static compression at room temperature (about 0.42), showing a strong superplastic deformation ability, which also implies that the microstructure of the materials has changed significantly, leading to a large increase in the plasticity of the material.
3.2. High Temperature Softening Mechanism of LRF TC4

The analysis in Section 2.1 shows that the softening effect of the materials is different with different deformation temperature. In addition, the remarkable improvement of the superplastic deformation ability of the material at high temperature indicates that the microstructure of the material changes differently, and the mechanism of controlling the thermal softening of the material is different as well.

Figure 4 shows the microstructure of the LRF TC4 sample deformed at 400°C - 900°C. The figure shows that when the LRF TC4 material is deformed at different temperatures, the needle structure changes mainly have two significant characteristics. When the material is deformed at 400°C - 600°C, the needle structure is obviously bent under stress, and the bending becomes more intense with the increase of deformation temperature. Figure 4b displays that the original β grain boundary is subjected to severe bending deformation. At 600°C, the needle structure tends to be arranged in the direction perpendicular to the pressure when it is subjected to severe bending deformation. The reorientation of the needle structure is conducive to generate larger strain and increase the material plasticity [19]. At 700°C-900°C, spheroidization occurred when the needle structure was seriously bent and fractured. In addition, with the increase of temperature, the proportion of spheroidized structure increases. When the temperature is 900°C, the spheroidized structure gets bigger.

Figure 5 is the TEM microstructure of the LRF TC4 sample deformed at 600°C. It shows that under the pressure, a large number of dislocations are generated in the grains, and the straight grain boundary (Figure 2b) becomes distorted before the original deformation (Figure 5a). Figure 5b shows the accumulation of dislocations at the grain boundary. Due to a large number of dislocations accumulate at the grain boundary, stress accumulation occurs at the accumulation position. The grain boundary shifts with external force (if no the grain boundary shifts, the grain boundary fractures will be the consequence), such that the stress is released, and the material exhibits softening of the rheological stress. Due to the non-uniformity of the dislocation density and the unsynchronized motion, the grain boundary is bent, as shown in Figure 5c.

Figure 6 is the TEM microstructure of the LRF TC4 sample deformed at 700°C. The bending deformation of needle structure is more severe, and the inner grain boundary is bent (Figure 6a), while the outer grain boundary is broken under the action of tensile force and dislocation (Figure 6b), resulting in the fracture of the needle structure into smaller equiaxed grains. This is consistent with the research results of Hong-Wu Song et al[19]. Figure 7 shows the TEM microstructure of the LRF TC4 sample deformed at 800°C. At 800°C, the spheroidized structure is formed due to the dynamic recrystallization under the combined action of temperature, external force and grain strain energy.
Figure 8 shows the TEM microstructure of the LRF TC4 sample deformed at 900°C. At this temperature, the spheroidized structure formed by the dynamic recrystallization gradually increases size to form larger equiaxed grains.

**Figure 4.** OM microstructure of the LRF TC4 sample (a) 400 °C; (b) 500 °C; (c) 600 °C; (d) 700 °C; (e) 800 °C; (f) 900 °C

**Figure 5.** TEM microstructure of the LRF TC4 sample deformed at 600°C.

**Figure 6.** TEM microstructure of the LRF TC4 sample deformed at 700°C.
Figure 7. TEM microstructure of the LRF TC4 sample deformed at 800°C.

Figure 8. TEM microstructure of the LRF TC4 sample deformed at 900°C.

The above TEM analysis shows that when the LRF TC4 sample is deformed at 600°C, the needle structure undergoes bending deformation, and this leads to the release of stress concentration, which makes the material exhibits the flow-stress softening effect shown in the Figure. 3 (when the deforming temperature is below 600°C, the degree of grain boundary bending is limited, and the softening effect is also limited, and with the increase of deformation temperature, the grain boundary bending increases and the softening effect increases as well). This conclusion is consistent with the research results of R. M. Miller et al. [8]. When the LRF TC4 sample is deformed at 700°C, the grain boundary first relieves stress caused by bending deformation, then the stress is relieved once more by the fracture of the bent grain boundary. Therefore, the softening effect of the material at 700°C is more obvious.

Due to the twice stress relief before 800°C, when the material is deformed at 800 °C, the appearance of recrystallized spheroidized structure further exacerbates the degree of stress release and the degree of softening effect is larger. During the deformation of the material at 900°C, due to the three times of stress relief previously, the softening effect reached the limit of all deformation temperatures in this experiment with the growth of the recrystallized grains.

4. Conclusions
Using the gleeble-1500 thermal simulator, the high temperature quasi-static compression experiment on cylindrical samples of Laser Rapid Forming Ti-6Al-4V(LRF TC4) titanium alloy have been conducted. Softening behaviour and mechanism of LRF TC4 alloy deformed at different high temperatures were investigated by means of optical microscope (OM) and transmission electron microscope (TEM). The conclusions are as follows:

(1) The strength of the LRF TC4 was significantly reduced with the rise of the deforming temperature, showing stronger thermal softening effect.

(2) Under the high temperature quasi-static condition, the material still has the strain strengthening effect at 400°C, and the rheological stress shows steady change in the plastic deformation stage at 500°C. When the temperature exceeds 500°C, the strength of the material decreases significantly.

(3) When the material is deformed at high temperature, the superplasticity ability of the LRF TC4 alloy is also stronger with the increase of deformation temperature.

(4) When the material is deformed at the temperature from 400°C to 900°C, the original acicular microstructure of LRF TC4 has two significant changes: Acicular microstructure bends at the temperature from 400°C to 600°C, spheroidization occurred while the needle-shaped structure was severely bent and fragmented at the temperature from 700°C to 900°C.

(5) During deformation at high temperature, the softening of the LRF TC4 alloy results from the bending, fragmentation, spheroidizing and their superimposition action of the needle structure.
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6. References
[1] Ji Sik Kim, Young Won Chang, et al. 1998 Metall. Mater Trans. A 29 (1) 217-226
[2] Chan Hee Park, Kyung-Tae Park, et al. 2008 Materials Transactions 49 (10) 2196-2200
[3] Lore Thijs, Frederik Verhaeghe, et al. 2010 Acta Materialia (58) 3303–3312
[4] Hang W.D, LIN X. 2010 MATERIALS CHINA 129(16) 12-27
[5] Wang H.M. 2002 ACTA AERONAUTICA ET ASTRONAUTICA SINICA 23(5) 473-478
[6] Zhao J.F, MA Z.Y, Xie D.Q, Han X.Q, Xiao M. Metal Additive Manufacturing Technique [J], Journal of Nanjing University of Aeronautics & Astronautics, 2014,46(5):675-683.
[7] E.B. SHELL, S.L.1999 SEMIATIN. METALLURGICAL AND MATERIALS TRANSACTIONS A (30A) 3219-3229
[8] R.M. Miller, T.R. Bieler, S.L. Semiatin. 1999 Scripta Materialia. 40(12) 1387–1393
[9] S. L. SEMIATIN, T. R. BIELER. 2001 Acta mater, (49) 3565–3573
[10] C.H. Park, Y.G. Ko, et al.2007 Mater. Sci. Forum. (551–552): 365-372
[11] L. Facchini, E. Magalini, et al. 2010 Rapid Prototyp. J.16(6) 450–459
[12] T. Vilaro, C. Colin, et al.2011 Metall. Mater. Trans. A. (42) 3190-3199
[13] A. Mertens, S. Reginster, H. Paydas, et al.2014 Powder Metallurgy 57(3) 184-189
[14] Jin Yao, TaoSuo, et al. 2016 Materials Science & Engineering A (677) 153–162
[15] ZhaO J.F, MA Z.Y, Xie D.Q, Han X.Q, Xiao M. Metal Additive Manufacturing Technique [J], Journal of Nanjing University of Aeronautics & Astronautics, 2014,46(5):675-683.
[16] Marco Simonelli, Chris Tuck, et al.2015 Metallurgical and Materials Transactions A 46(9):3842–3851
[17] L.Y. Chen, J.C. Huang, et al. 2017 Materials Science & Engineering A(682) 389–395
[18] Ming-Wei Wu, Pang-Hsin Lai, 2016 Mater.Sci.Eng. A.(650) 295–299
[19] Hong-Wu Song, Shi-Hong Zhang, Ming Cheng. 2009 Journal of Alloys and Compounds (480):922–927