GRAIN REFINEMENT IN Ti-6Al-4V ALLOY DURING THERMO-MECHANICAL PROCESSING AND INVESTIGATION OF FLOW PROPERTIES

Digvijay J. Sheed[1], Bhagawati P. Kashyap[2], Rajkumar P. Singh[1]

[1]Bharat Forge Limited, Pune, 411036, India
[2]Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Mumbai, 400076, India

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Abstract

The flow properties of thermo-mechanically processed Ti-6Al-4V alloy were investigated in this study. Two samples from as received Ti-6Al-4V alloy plate with consisting coarse β grains of ~ 170 μm size were rolled at 940°C and 550°C respectively. In hot rolled (940°C) sample fully equiaxed microstructure with average grain size ~ 2.3 μm was produced. In warm rolled (550°C) sample, heterogeneous microstructure was produced, which consists of elongated and partially equiaxed grains. To investigate flow properties of hot and warm rolled Ti-6Al-4V alloy samples, differential strain rate tests were performed. Strain rate sensitivity (m) values and apparent activation energy (Q) for were calculated for both hot and warm rolled samples.

Introduction

Titanium and titanium alloys, especially Ti-6Al-4V alloy has wide applications in aerospace, marine, chemical, automobile engineering and biomedical science due to high strength to weight ratio and good corrosion resistance properties [1]. In many applications, mechanical properties and ability to produce intricate shapes are important properties of materials. It is well known from Hall-Petch equation that the decrease in the grain size will increase the yield strength of material significantly at room temperature. In addition to this, the fine grain size (< 10 μm) is a necessary condition for superplastic forming. In recent years various severe plastic deformation (SPD) methods were developed like equal channel angular processing (ECAP), high pressure torsion (HPT), accumulative roll bonding (ARB) etc. [2-4], to achieve ultrafine and nano grain size materials. These SPD methods have limited industrial applications, due to small sample size and they require special tooling which is costly and necessitates special preparation during processing. Generally, thermo-mechanical process is suitable for mass production in industries, consists of high temperature deformation and subsequent heat treatments like annealing, results in the grain coarsening. It is very difficult to achieve fine grained structure with conventional thermo-mechanical processes. In order to produce fine grain material and reduce grain coarsening at high temperature, warm-working of material is generally preferred. Flow properties of Ti-6Al-4V alloy at high temperature can be affected, by the microstructures which include α phase morphology, volume fraction of β phase etc. In the current study, significant grain refinement in Ti-6Al-4V alloy achieved as well as the effect of deformation temperature and resultant microstructure on flow properties was investigated.

Material and Experimental Procedure

In the present study, a commercial two phase (α/β) Ti-6Al-4V alloy plate with thickness of 4 mm was used. The chemical composition (weight) of the alloy is 6.03%Al, 4.02%V, 0.32%Fe, 0.016%C and balance Ti. The β transus temperature of the alloy was ~ 995°C. Two samples of 150 mm length and 35 mm width were cut from as received Ti-6Al-4V alloy. Rolling was done with roll mill of 75 tones capacity and 320 mm roll diameter. In order to prevent the oxidation at deformation temperature, the samples were coated with deltaglaze. After rolling the samples were water quenched. Processing parameters employed for rolling are tabulated in the Table I.

Table I. Processing parameters employed for hot and warm rolling

Processing parameters	Hot Rolling	Warm rolling
Deformation temperature	940°C	550°C
Initial plate size- 150 x 35 x 4 mm^3		
Final reduction	2 mm (50%)	2.2 mm (45%)
Number of passes	2	6
Intermediate heating	Yes	Yes
Soaking Time	40 Min	40 min for 1-4 pass 60 min for 5-6 pass
Rolling speed	15 MPM	14 MPM - for 1-4 pass 10 MPM - for 5-6 pass

Samples were cut from rolled Ti-6Al-4V plate for metallographic sample preparation. Kroll's reagent, consist of 5% HNO_3, 10% HF and 85% H_2O, was used as etchant. Microstructures of rolled samples were observed with optical microscope (OM) and scanning electron microscope (SEM) (Hitachi S-3400N) along longitudinal direction (LD), transverse direction (TD) and normal direction (ND). For orientation image microscopy (OIM) analysis electro polishing was done with 600 ml methanol, 360 ml ethylene glycol and 60 ml perchloric acid electrolyte.
Tensile samples of gauge length 10 mm and gauge width 2 mm were machined from rolled Ti-6Al-4V alloy plate. Differential strain rate tests were performed at high temperature by using 100 kN capacity, Zwick-Roell Amsler Universal Testing Machine attached with triple zone split furnace. The tensile samples were deformed for ten different strain rates between $2x10^{-5}$ s^{-1} to $1x10^{-2}$ s^{-1} and at different deformation temperatures of 927°C, 900°C, 850°C, 800°C and 750°C. The samples were soaked for 20 minutes at the test temperatures before deformation. After differential strain rate tests the microstructures of gauge and shoulder section of deformed samples were observed under OM and SEM.

Results and Discussion

Microstructural evolution

In the present study, β hot rolled and water quenched Ti-6Al-4V alloy, consisting coarse β grains of ~ 170 μm size and α' martensite microstructure, appears as starting microstructure (Fig. 1). The β grains are elongated in ND. The microstructures of hot rolled and warm rolled samples examined in OM and SEM are presented in Fig. 2

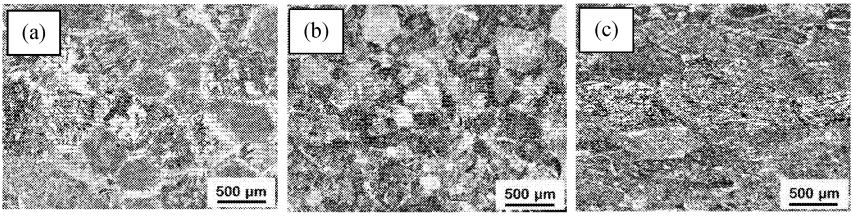

Figure 1. OM micrographs of as-received Ti-6Al-4V alloy along LD (a), TD (b), ND (c)

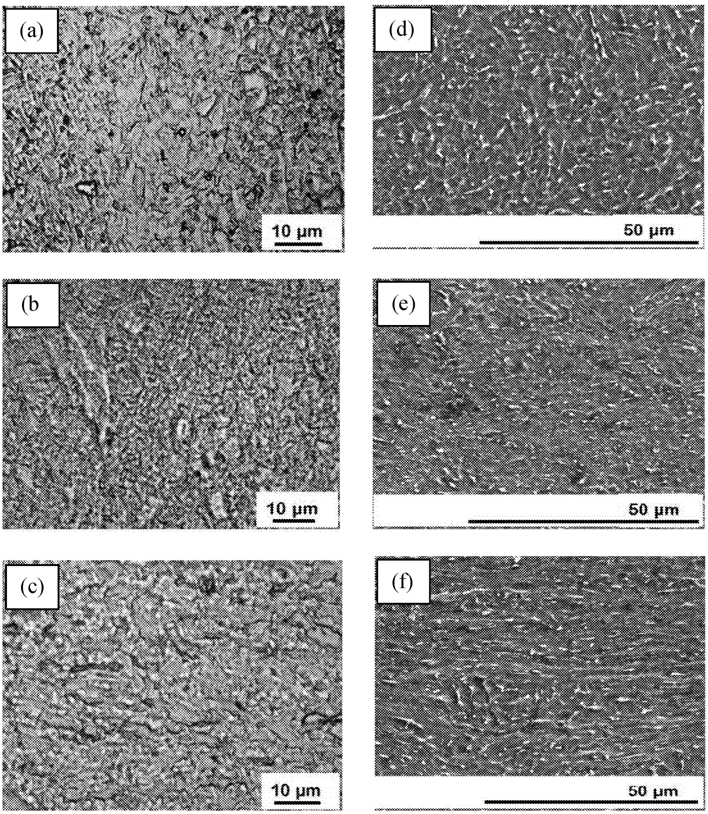

Figure 2. OM micrographs (a-c) of hot rolled sample along LD (a), TD (b), ND (c), and SEM microstructures (d-f) of warm rolled sample along LD (d), TD (e), and ND (f)

The grain size, aspect ratio and volume fraction of hot and warm rolled Ti-6Al-4V alloy samples are tabulated in Table II.

Table II. Grain size, aspect ratio and volume fraction of rolled Ti-6Al-4V alloy sample

	Hot rolled after second pass	Warm rolled after six pass
α morphology	Equiaxed (Mixture of fine and coarse grains)	Partially equiaxed and elongated grains
Grain size	LD – 2.2 μm TD – 2.2 μm ND – 2.5 μm	LD – 4.4 μm TD – 2.2 μm ND – 2.4 μm
Aspect ratio	LD – 3.4 ND – 2.7	LD – 4.7 ND – 4.5
Volume fraction of β phase	37%	25%
Fraction of high angle grain boundaries	0.96	0.93

In hot rolling process, Ti-6Al-4V alloy plate was deformed to effective strain of 0.69 in two passes. Under OM, a mixture of fine and coarse equiaxed grains was observed. The elongated grains in the first pass were transformed to equiaxed grains because of globularization [5], which consists of static as well as dynamic globularization contributions. In hot rolling, the effective strain of 0.69 is sufficient to initiate globularization, as in beta annealed and water quenched sample the globularization initiated at lower effective strains of 0.5 to 1 depending on temperature [6]. The fraction of globularization increases with temperature and also affected by static heat treatment prior to rolling [7, 8]. In warm rolling the sample was deformed to effective strain of 0.59 which is very low to initiate the globularization at 550°C deformation temperature, hence the mixture of elongated and partially equiaxed grains were present. Due to high deformation temperature close to β transus temperature the volume fraction of β phase is more in hot rolled sample than that in warm rolled sample. The rolled Ti-6Al-4V alloy samples were not annealed because the material with as deformed condition shows good superplasticity rather than the annealed material. [9]. From OIM analysis it is observed that in present study there is less influence of deformation temperature on the number fraction of high angle grain boundaries in deformed sample. High angle grain boundaries are important as they favor the grain boundary sliding (GBS) process which is important for superplasticity [10].

<u>Investigation of Flow Properties</u>

In differential strain rate test, from load-elongation data at varying strain rates, ln(flow stress) vs ln(Strain rate) plot for hot and warm rolled samples are derived as shown in Fig. 3.

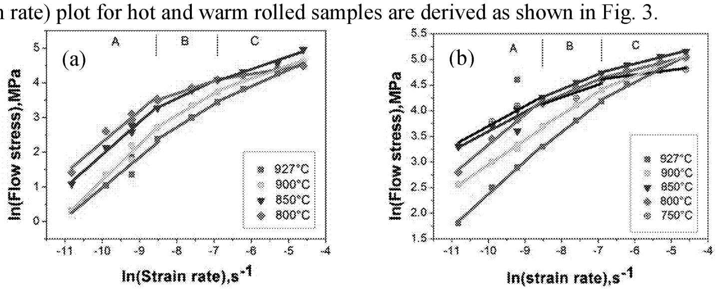

Figure 3.ln(flow stress) vs ln(strain rate) plot for hot rolled (a) and warm rolled (b) samples

It is observed from ln(Flow stress) vs ln(Strain rate) plot for hot and warm rolled samples, that flow stress increases with decrease in deformation temperature and increase in strain rate. However, at 800°C flow stress is seen to be less in hot rolled sample than that at 850°C, especially towards higher strain rates. This appears to be the result of neck formation at 800°C. In warm rolled tensile sample, flow stress is less at 800°C than that at 850°C, at higher strain rates but then it is followed by greater flow stress at 750°C deformation temperature towards lower strain rates. The successive varying nature of flow stress as a function of strain rate at 800°C and 750°C in warm rolled sample may be the result of change in grain morphology during deformation at high temperature. The strain rate sensitivity (m) value, which is measure of superplasticity ($m \geq 0.3$), are calculated from ln(Flow stress) vs ln(strain rate) plot for regions A, B and C in Fig. 3 and same are listed in Table III.

Table III. Strain rate sensitivity (m) values for hot and warm rolled samples

	Hot rolled			**Warm rolled**		
Temperature, °C	A	B	C	A	B	C
927	0.88	0.66	0.48	0.64	0.55	0.37
900	0.99	0.63	0.38	0.48	0.43	0.28
850	0.94	0.51	0.37	0.38	0.30	0.18
800	0.89	0.36	0.17	0.59	0.30	0.18
750	--	--	--	0.39	0.25	0.10

The higher m values appear at lower strain rates for both hot and warm rolled samples. For hot rolled sample, m values are greater as compared to that for the warm rolled sample. This is because of difference in α grain morphology and β phase fraction between both samples. In hot rolled sample, equiaxed grains facilitates grain boundary sliding (GBS). In warm rolled sample, for elongated grains, there can be difficulty in accommodation of deformation at triple point junction [12].

Microstructure of hot rolled Ti-6Al-4V alloy sample has equiaxed grains and 37% of β phase fraction. The β phase fraction has significant effect on the strain rate sensitivity (m) of Ti-6Al-4V alloy. The second phase β has body centered cubic structure and having two orders of magnitude more self-diffusion coefficient than α phase, so it has tendency of grain coarsening at high temperature. The grain coarsening of β phase is prevented by α phase, which results stable microstructure during deformation. The presence of β phase, also decreases α/α grain contacts as α/α grain boundary has less contribution to the superplastic deformation [13]. In hot rolled sample, deformation is controlled mostly by grain boundary sliding ($n= 1/m=2$) whereas in warm rolled sample deformation is mostly controlled by dislocation climb mechanism with m being around 0.2 to o.25 ($n = 5$ to 4).

Analysis towards constitutive relationship

Mechanism during deformation is studied by using Arrhenius type rate equation. From the ln(flow stress, σ) and inverse absolute deformation temperature (1/T) K^{-1}, Arrhenius plot is made for hot and warm rolled samples as shown in Fig. 4.For hot rolled sample, the value of activation energy (Q) is ~170 -137 kJ/mole at lower strain rate, in region A and for region B (Fig. 3) it is ~ 188 – 152 kJ/mole, which are in good agreement with the values reported earlier [14]. This Q values are much closer to the self-diffusion in α-Ti (150 kJ/mole), which is approximately the activation energy of grain boundary diffusion [15]. In warm rolled sample at lower strain rate in region A, Q ~ 345-321 kJ/mole are found which changed to ~185 kJ/mole in region B. In present study, the activation energy value in warm rolled sample is noted to vary significantly with strain rate in warm rolled sample.

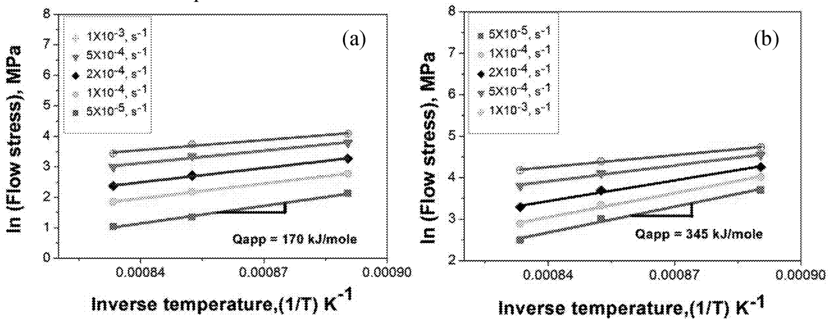

Figure 4. Arrhenius plot for hot rolled sample (a) and warm rolled sample (b) showing variation of flow stress vs inverse of deformation temperature.

Metallography of deformed samples

The differential strain rate test samples were deformed up to failure and allowed to furnace cool. Microstructures of the gauge and shoulder sections of such samples are shown in Fig. 5.

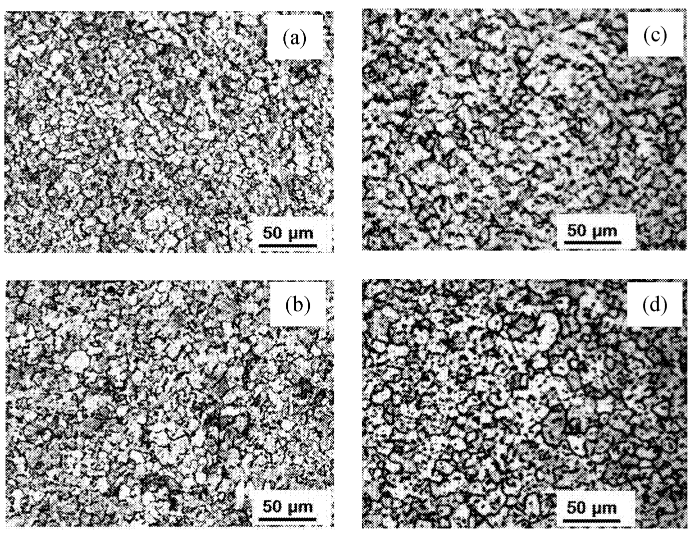

Figure 5.Microstructures after differential strain rate tests for hot rolled tensile sample: gauge section (a), shoulder section (b); and for warm rolled tensile sample: gauge section (c), shoulder section (d)

It is observed from the micrographs that, in hot rolled tensile sample, grains remain equiaxed after deformation in gauge as well as in shoulder sections, which is very important feature of the superplasticity [15]. In warm rolled sample, the grain boundaries of β phase are distorted and slightly elongated in gauge section as compared fully equiaxed and stable grains in shoulder section. As discussed earlier, in elongated grains there can be difficulty in accommodation of deformation by grain boundary sliding at triple point junction and so deformation is controlled by dislocation climb mechanism.

Conclusions

In present study grain refinement up to ~ 2.3 μm from the initial grain size of ~170 μm was obtained in Ti-6Al-4V alloy with conventional thermo-mechanical processing.

Superplasticity was investigated in both hot and warm rolled samples. The flow properties were affected by α grain morphology and β volume fraction in hot and warm rolled samples. In Hot rolled sample, equiaxed grains and 37% β phase fraction showed superior flow properties and higher strain rate sensitivity index (m) values in comparison with that by elongated grain structure and lower β phase fraction in warm rolled sample.

The apparent activation energy (Q) calculated for hot rolled and warm rolled Ti-6Al-4V alloy samples respectively, suggest that in hot rolled sample, deformation by grain boundary sliding is accommodated by grain boundary diffusion. In warm rolled sample, the same is accommodated by dislocation climb mechanism.

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