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ScienceDirect
Trans. Nonferrous Met. Soc. China 21(2011) 1728−1734
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Effect of hot working and post-deformation heat treatment on microstructure and tensile properties of Ti-6Al-4V alloy

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Received 23 September 2010; accepted 20 January 2011

Abstract: The effects of hot compression, hot rolling and post-rolling annealing on microstructure and tensile properties of Ti-6Al-4V were analyzed. Hot compression tests were conducted in the temperature range of 800−1 075 °C and at strain rates of 0.001−1 s^{-1}, and the relations between the characteristic points of flow curve and processing variables were developed. Two passes of hot rolling test with total reduction of 75% were performed in the temperature range of 820−1 070 °C and at constant strain rate of 2 s^{-1}. After hot rolling, some specimens were subjected to heat treatment at 870 °C and 920 °C for 2 h followed by air cooling. Hot rolling in beta phase field resulted in coarse beta grains transforming to martensite by cooling. Otherwise, rolling in the alpha/beta phase filed gave rise to a partially globularized alpha microstructure. The post-rolling heat treatment completed the partial globularization of alpha phase in two-phase region and otherwise broke down the martensitic structure of beta-rolled samples. Tensile tests showed that the strength characteristics as well as elongation decrease significantly with increasing the rolling temperature from the two-phase to the single-phase region. Increasing heat treatment temperature contributed to lower strength for the specimens rolled in two-phase region and higher strength characteristics for the beta-rolled specimens.

Key words: recrystallization; hot rolling; hot compression; heat treatment; globularization

1 Introduction

Hot deformation parameters and post-deformation heat treatments are often selected in a way to control the microstructure and properties of many industrial alloys. The desired strength characteristics and corrosion resistance in titanium alloys are achieved by the precise control of processing route and therefore microstructure. Ti-6Al-4V is a very well known alloy for its desirable mechanical properties and corrosion resistance. The application of this alloy is particularly attractive to aerospace and biomaterial industries [1]. The mechanical and microstructural characteristics of Ti-6Al-4V depend actually on the variables of hot deformation and/or heat treatment. The previous researchers have tried to investigate the microstructural and mechanical behavior of this alloy during hot working. SESHACHARYULU et al [2] studied the hot deformation behavior and damage mechanisms in an extra low interstitial grade Ti-6Al-4V. A number of studies got involved in the effect of texture or morphology of α-phase on the hot deformation characteristics of the alloy [3−6]. Other investigators proposed the occurrence of dynamic globularization of α-phase during hot deformation or static globularization during post-deformation heat treatment [7−11]. However, fewer contributions analyzed the hot deformation behavior of the alloy in the β-phase region and proposed the occurrence of dynamic recrystallizations (DRX) [12]. Some other researchers attempted to probe the mechanical characteristic and microstructural evolutions of this alloy during industrial processes. The deformation stability, plastic anisotropy and cavity formation during hot forging were analyzed in detail [13−14]. Although investigations on hot working behavior of this alloy are numerous, but very few attempts have been made to correlate hot rolling conditions to the mechanical properties [15−16]. Moreover, only limited data on the effect of actual processing conditions on tensile properties are available. Hence, in this investigation thermomechanical processing parameters−microstructure−tensile property relations for Ti-6Al-4V are studied using hot compression, hot rolling and post-deformation heat treatment.

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DOI: 10.1016/S1003-6326(11)60922-9

2 Experimental

The studied Ti-6Al-4V alloy with the composition of 6.66% Al, 5.13% V, 0.21% Fe, 0.03% Mo, 0.02% Mn, 0.02% Si and the balance of Ti was received as hot rolled strips with 12 mm in thickness. The beta transus temperature was approximated 970 °C by thermal dilatation method. The as-received material was primarily subjected to beta annealing treatment at 1 050 °C for 35 min followed by air cooling. The microstructure developed by beta annealing treatment consisted of a lamellar α within the prior β grains along with grain boundary α (Fig. 1). The prior β grain size in the annealed material was measured to be about 350 μm. Cylindrical compression samples of 15 mm in height and 10 mm in diameter were prepared with the axis along the rolling direction of the as-received plate. An INSTRON 8502 testing machine equipped with a fully digital and computerized control furnace was employed to perform hot compression tests under constant strain rates, ranging from 10^{-3} s^{-1} to 1 s^{-1} at an interval of an order of magnitude and at temperatures of 800, 850, 900, 950, 1 000, 1 025, 1 050 and 1 075 °C. Rolling samples with size of 40 mm×60 mm were cut from the as-received plate and subjected to hot rolling at temperatures of 820, 870 and 920 °C in two-phase alpha/beta region and at temperatures of 970, 1 020 and 1 070 °C in single-phase beta region. All rolling samples were reheated prior to testing to simulate actual industrial hot rolling process. The rolling strain rate was 2 s^{-1} and a total reduction of 75% was performed in two-passes. All samples were air cooled after rolling to simulate actual production practice. Some hot rolled specimens were subjected to post-rolling heat treatment at temperatures of 870 °C and 920 °C for 2 h followed by air cooling. All specimens were then prepared according to the standard procedures and subjected to microstructural observations by optical microscopy and tensile testing.

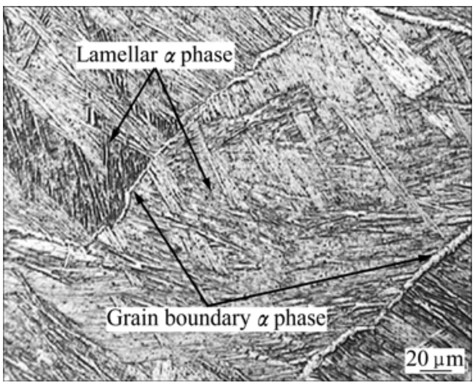

Fig. 1 Microstructure of as-received sample after β annealing treatment at 1 050 °C for 35 min followed by air cooling

3 Results and discussion

Hot compression tests in this research were performed to identify the general flow characteristics of the studied alloy in hot working conditions. Indeed, hot compression is widely used as a simulation technique for actual industrial hot working processes in order to develop practical relationships between critical strains for microstructural changes and processing variables. The typical flow stress curves obtained in $\alpha+\beta$ region at 900 °C, and in single-phase β region at 1 000 °C, are shown in Fig. 2. As seen, the flow stress level actually increases with the strain rate increasing and decreases with temperature increasing. Figure 3 shows the dependence of the characteristic strain and stress on Zener-Hollomon parameter which incorporates the effects of strain rate and temperature as follows:

$$Z = \dot{\varepsilon}\exp(\frac{Q}{RT}) \quad (1)$$

where Q denotes the apparent activation energy, and R and T are the gas constant and absolute temperature, respectively. The values of Q for single-phase β and $\alpha+\beta$

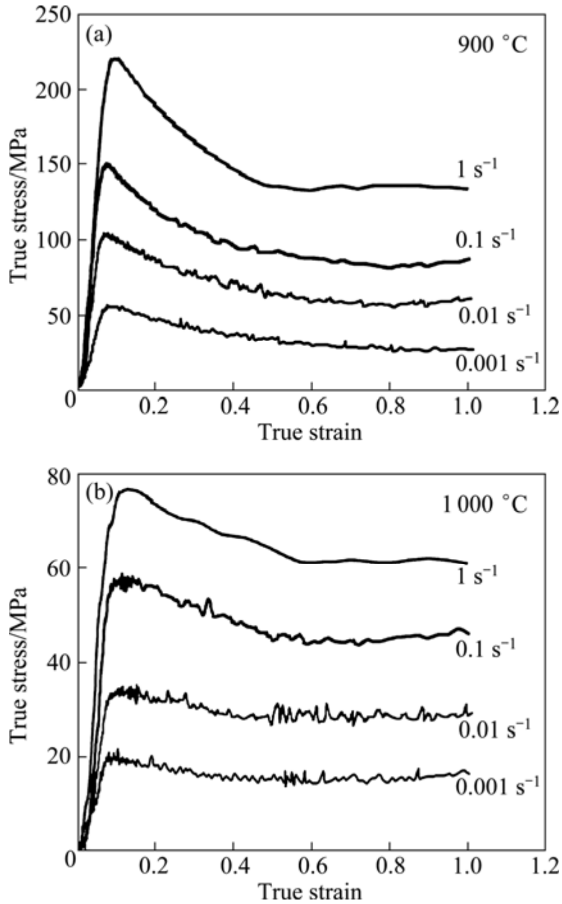

Fig. 2 Typical flow stress curves obtained in two-phase $\alpha+\beta$ region at 900 °C (a) and in single-phase β region at 1 000 °C (b)

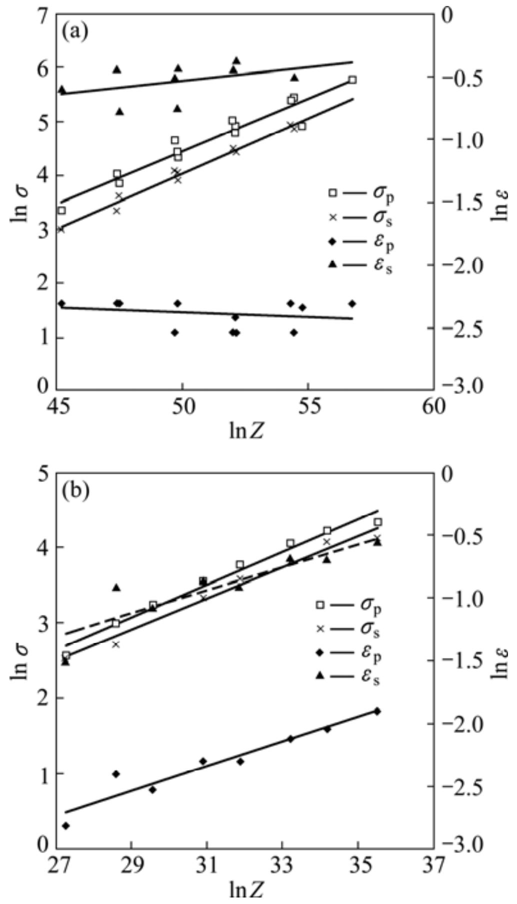

Fig. 3 Linear relations between both strain and stress of characteristic points of flow curve with Zener-Hollomon parameter in two-phase (a) and single-phase regions (b)

regions were determined elsewhere as 530 kJ/mol and 376 kJ/mol, respectively [17]. It is clearly seen in Fig. 3 that the peak strain, ε_p, and the stable flow strain, ε_s, in β region are strongly Z-dependent. In $\alpha+\beta$ region, ε_s shows a slight dependence on Z; however, ε_p is nearly independent of Z. This observation can be attributed to the different dominant dynamic restoration mechanisms in the different phase fields. According to the linear curves in Fig. 3, ε_p in $\alpha+\beta$ region is about 0.09 and other characteristic points bear the following expressions:

$$\varepsilon_s^{(\alpha+\beta)} = 0.2Z^{0.021} \quad (2)$$

$$\sigma_p^{(\alpha+\beta)} = 0.005Z^{0.2} \quad (3)$$

$$\sigma_s^{(\alpha+\beta)} = 0.002Z^{0.2} \quad (4)$$

$$\varepsilon_p^{(\beta)} = 0.005Z^{0.098} \quad (5)$$

$$\varepsilon_s^{(\beta)} = 0.058Z^{0.063} \quad (6)$$

$$\sigma_p^{(\beta)} = 0.039Z^{0.22} \quad (7)$$

$$\sigma_s^{(\beta)} = 0.047Z^{0.21} \quad (8)$$

In fact, the expression of characteristic stress and strain in terms of deformation variables facilitates the design of actual hot working processes such as hot rolling. The microstructures after hot rolling in the two-phase region are illustrated in Fig. 4. According to the hot compression results, the strain of 0.1 is required for starting dynamic globularization which is accompanied by dynamic softening in this region. As the strain performed in each pass of hot rolling was about 0.53, therefore, dynamic globularization (DG) was expected to occur. However, the low strain value applied in each pass and concurrent deformation especially at low temperatures decelerated the progress of DG. Indeed, the lower the deformation temperature is, the less the dynamic globularization is. It is clearly seen that at 820 °C the layers of α are highly elongated and hardly seen at higher temperatures, e.g. 870 °C and 920 °C, the elongated α grains are more distinct. It is worth mentioning that increasing rolling temperature from 870

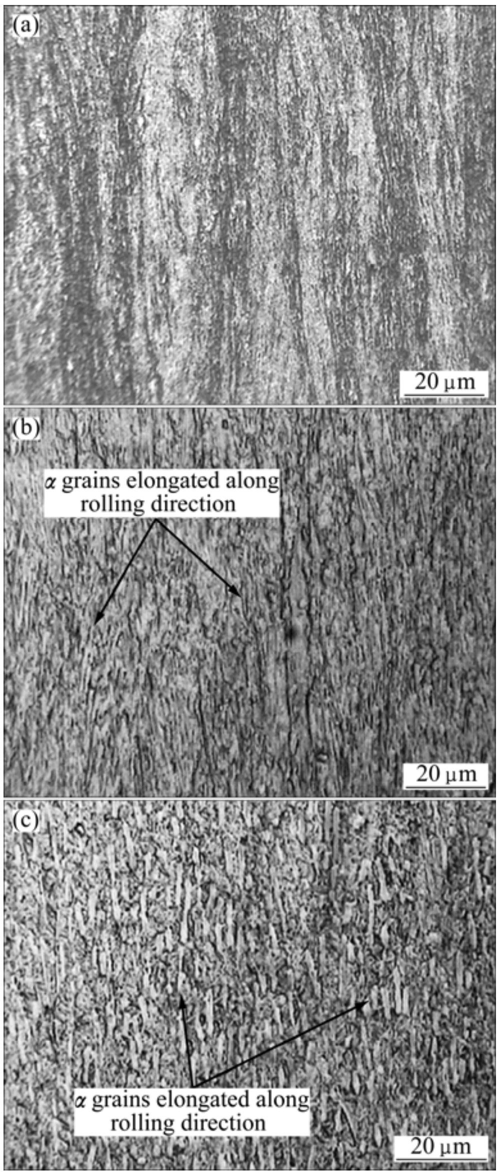

Fig. 4 Microstructures of hot rolled samples to total reduction of 75% through two passes at 820 °C (a), 870 °C (b) and 920 °C (c)

°C to 920 °C decreases the aspect ratio of alpha grains from 10:1 to 7:1, suggesting a greater amount of spheroidization via dynamic globularization.

Figure 5 shows the microstructures of samples hot rolled and immediately annealed at temperatures 920 °C for 2 h. Comparing with the as-rolled microstructures shown in Fig. 4, heat treatment after rolling gives rise to more globularized and coarser α grains. It manifests that static globularization of partially globularized α grains is apparently responsible for microstructural evolution during post-rolling heat treatment. Previous researchers stated that the required strain value for the completion of DG within the studied temperature range is about 2 [18]. As the applied strain during hot rolling is not high enough to complete the globularization dynamically, the post-rolling heat treatment is needed to complete it statically. It is also obvious from Fig. 5 that the size of static spheroidized α grains increases with heat treatment temperature increasing from 870 °C to 920 °C.

Figure 6 shows the microstructures obtained after hot rolling at temperatures of 970 °C, 1 020 °C and 1 070 °C in β region. According to Eq. (5), the peak strains for the studied deformation temperatures are calculated to be 0.2, 0.165 and 0.145, respectively. The values of corresponding steady state strains can be calculated using Eq. (6) as 0.6, 0.55 and 0.5, respectively. The calculated peak and steady state strains indicate that the rolling strain is enough, especially at higher temperatures, to complete dynamic recrystallization (DRX) in β region.

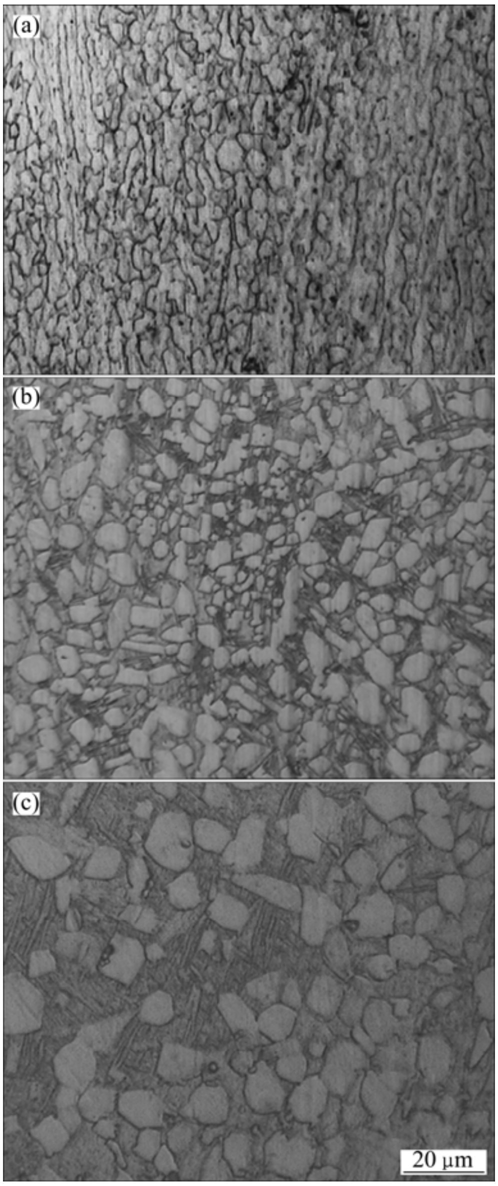

Fig. 5 Microstructures of samples hot rolled at 820 °C (a), 870 °C (b) and 920 °C (c) to total reduction of 75% and then heat treated at 920 °C for 2 h

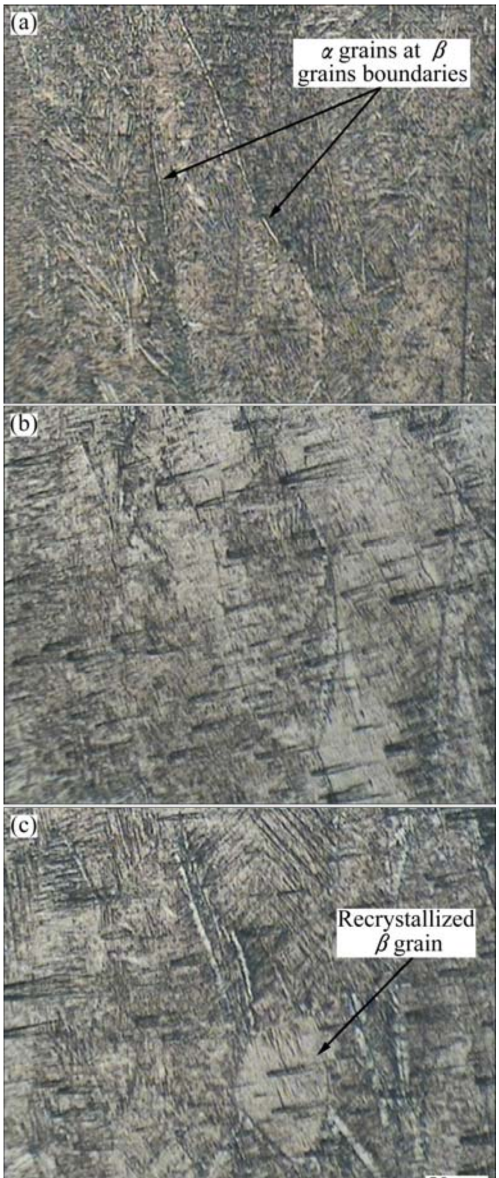

Fig. 6 Microstructures of samples hot rolled in β region to total reduction of 75% at temperatures of 970 °C (a), 1 020 °C (b) and 1 070 °C (c)

In contrary to the expectation, Fig. 6(a) shows that at 970 °C the as-rolled structure includes pancaked grains of *β*. The nucleation of strain induced fine grains of *α* at the original grain boundaries of *β* around 970 °C, determined as *β* transus, can be responsible for the retardation of DRX. Otherwise, in Figs. 6(b) and 6(c), the dynamically recrystallized *β* grains transformed to martensite during following air cooling are clearly observed.

In order to break down martensite in the structure of *β*-rolled samples, post-deformation heat treatment was carried out at 870 °C and 920 °C for 2 h. Figure 7 demonstrates the microstructures of the heat treated samples. As observed all microstructures are characterized by a layer of *α* grains formed at prior *β* grain boundaries and a lamellar structure inside the grains.

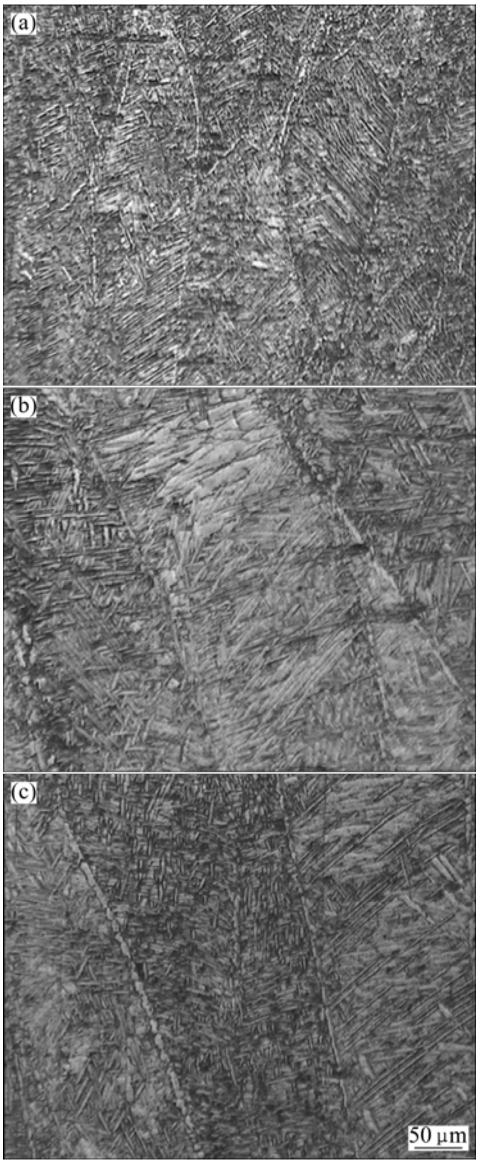

Fig. 7 Microstructures of samples hot rolled in *β* region to total reduction of 75% at temperatures of 970 °C (a), 1 020 °C (b) and 1 070 °C (c) and then heat treated at 920 °C for 2 h

Figure 8 shows the tensile properties of the hot rolled and annealed specimens at different temperatures. Although the yield strength of samples hot rolled in two-phase region increased with rolling temperature, the change in ultimate strength was negligible. Typically, there is also an adverse relation between elongation and strength characteristics. Increase in yield strength with increasing rolling temperature in *α*+*β* region is attributable to the decrease in the volume fraction of *α* and increase in *β*. Therefore, yield strength increases as a

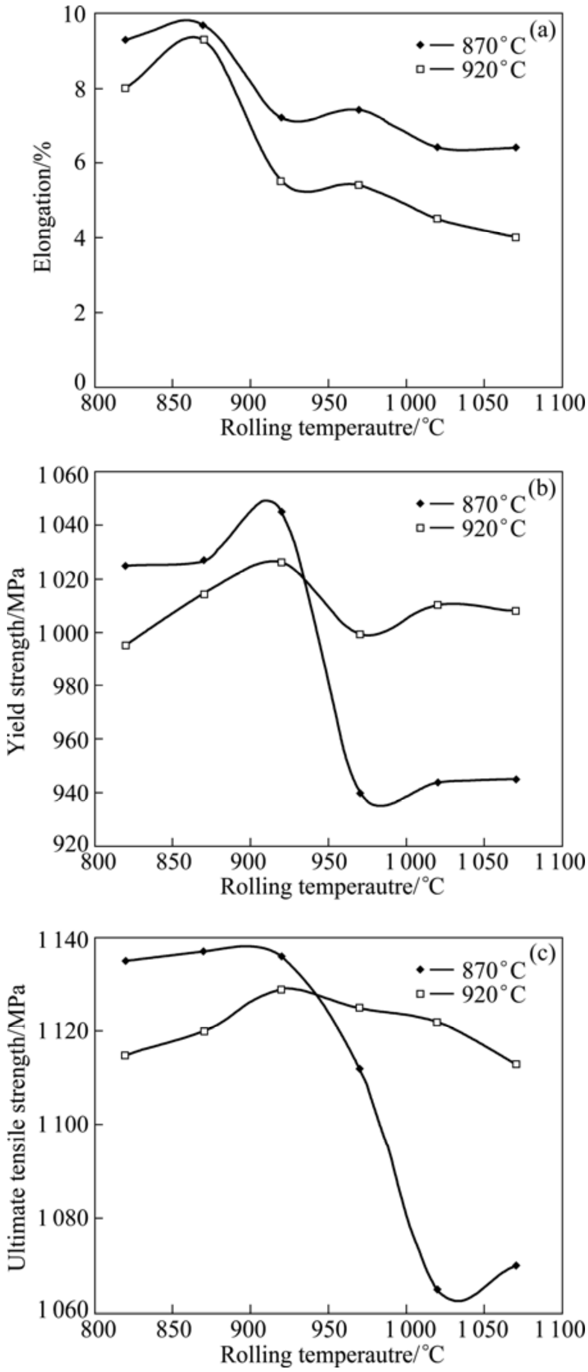

Fig. 8 Tensile properties of samples hot rolled at different temperatures and post-rolling heat treated at 870 °C and 920 °C for 2 h

result of the transformation of β to martensite during cooling. Although rolling at higher temperature increases the volume fraction of β and therefore more martensite on cooling, higher heat treatment temperature increases the thickness of martensite platelets and degrades their strengthening potential. In addition, higher volume fraction of coarse martensite can be responsible for the lower ductility at higher heat treatment temperatures. It is worth mentioning that random martensite platelets with acicular α structure have lower deformation barrier spacing than a common colony structure. Therefore, the strength increase is associated with a decrease in barrier spacing.

The decrease in the strength characteristics of β-rolled samples is likely associated with the growth of β grains during reheating. It is obvious that there is a great difference between the average grain size of specimens hot deformed below and above the β transus, as shown in Figs. 4 and 6. As a matter of fact, a coarse martensitic structure ensues from coarse β grains, resulting in a low strength level. Further, during subsequent heat treatment α grains preferentially nucleate at the β grain boundaries and martensitic structure begins to be replaced by a globularized α structure. It is worth noting that, for the limited number of slip modes the hexagonal crystal structure of titanium exhibits a very strong grain boundary or Hall-Petch strengthening at hot-working temperatures [6]. Because the strength of the α phase is much greater than that of the β phase at hot-working temperatures [19−20], the Hall-Petch effect is conjectured to be controlled by the properties and thickness of α laths or platelets. Therefore, the dislocation pile-up/slip transmission processes that characterize the Hall-Petch phenomena are likely to occur in the α phase, but not in the β phase.

It is important to note that for β-rolled specimens, heat treatment at higher temperature caused higher level of strength. This is because the vanadium content of β phase increases as temperature decreases. The vanadium-rich β phase at lower heat treatment temperature, therefore, depresses the martensite start temperature, M_s, and reserves greater amount of β phase at the ambient temperature that leads to lower strength level.

4 Conclusions

1) From hot compression tests in two-phase region, it was found that peak and steady state strains were almost independent of Zener-Hollomon parameter. Otherwise, in single-phase region, simple power-law equations were used to explain the strong dependence of peak and steady state strains on Z parameter. On the whole studied conditions the flow stress was directly dependent to Z.

2) Dynamic globularization of the α-phase did not complete during hot rolling with conventional strain values. Therefore, it needed post-rolling heat treatment to complete statically.

3) Hot rolling in single-phase β region led to coarse β grains which transformed to coarse martensitic structure during subsequent air cooling.

4) The post-rolling heat treatment completed the globularization of α-phase and broke down the martensitic structure of β-rolled samples in two-phase and single-phase regions, respectively.

5) Tensile tests on hot rolled and heat treated samples clarified that both ultimate and yield strength and also elongation decrease significantly with increase in rolling temperature from the single-phase to the two-phase region. Although for the specimens rolled in two-phase region higher heat treatment temperature caused lower strength characteristics, the observations for the β-rolled specimens were quite adverse.

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热加工和后处理对 Ti-6Al-4V 合金显微组织和拉伸性能的影响

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摘 要：研究了热压缩、热轧制和后续轧制退火处理对 Ti-6Al-4V 合金显微组织和拉伸性能的影响。热压缩实验在温度 800~1 075 °C 和应变速率 0.001~1 s^{-1} 下进行，得到了流变曲线与加工过程参数之间的关系。然后，样品在温度 800~1 070 °C 和恒应变速率 2 s^{-1} 下进行 2 道次热轧制，总变形量为 75%。热轧后，样品分别在 870 °C 和 920 °C 下保温热处理 2 h，随后空冷。在 β 相区的热轧导致粗大的 β 相冷却时转变为马氏体相，而在 α/β 两相区的热轧会导致生成部分球化的 α 相组织。后续的热轧处理能使在两相区部分球化的 α 相得以完成球化，然而，在 β 相区轧制的样品会导致马氏体结构被破坏。拉伸实验表明，随着轧制温度从两相区升高到单相区，合金的强度及伸长率会显著降低。升高热处理温度会降低两相区轧制合金的强度性能，而在 β 相区轧制合金的强度会得到提高。

关键词：Ti-6Al-4V 合金；热轧；热压缩；热处理；拉伸性能

(Edited by YUAN Sai-qian)