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# Hot deformation behavior and dynamic recrystallization of melt hydrogenated Ti-6Al-4V alloy



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# ABSTRACT

The effect of hydrogen on hot deformation behavior of Ti-6Al-4V alloy was investigated. Ti-6Al-4V alloy was hydrogenated by melting alloy in gas mixture of hydrogen and argon (melt hydrogenation). Experimental results of hot compression at same strain rate showed hydrogen decreased flow stress at higher deforming temperature, which was attributed to hydrogen induced dislocation movement and dynamic recrystallization (DRX). At lower temperature, peak stress firstly decreased and then increased with increasing hydrogen content. Hydrogen decreased the peak stress and improved the hot workability of alloy deformed at same temperature and different strain rates. Microstructure observation of as deformed alloy indicated hydrogen promoted DRX on both  $\alpha$  and  $\beta$  phase, and encouraged the decomposition of residual lamella. Electron back-scattered diffraction results indicated that hydrogen mainly encouraged discontinues DRX and decreased the dislocation density in  $\alpha$  phase. Compared to unhydrogenated alloy, when hydrogen content was  $5.31 \times 10^{-2}$  wt.%, volume fraction of DRX increased from 42% to 53% and 41.8%–72.1% at strain rate of 0.01 and 0.001 s<sup>-1</sup>, respectively.

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# 1. Introduction

Ti-6Al-4V alloy (Ti64) with high strength, low density and high corrosion resistance, has been widely used in the industry and/or science of aerospace and aviation [1,2]. It has been reported that over the half titanium products [3] are Ti64 alloys, however, due to the poor workability, manufacturing the Ti64 alloy by cold working induces serious cracks which decides the Ti64 alloy must be hot worked before used.

Titanium alloy including Ti64 alloy with high deforming resistance which means improving the hot workability of Ti64 alloy is meaningful and important. Hydrogenation is an effective method to improve the hot workability of titanium alloys. It has been reported [4] that hydrogen as temporary alloying element can increase the volume fraction of softer  $\beta$  phase and dynamic recrystallization (DRX), and then decrease the flow stress of titanium alloy deformed at high temperature. Senkov [5] investigated the thermo-hydrogen processing of titanium alloys, and found that hydrogen enhanced the interaction between dislocation and obstacles, and then increase the dislocations mobility which was beneficial to the high temperature deformation. Chen [6] and Ma [7] hydrogenated the titanium aluminides alloys and found that hydrogen decreased the flow stress and encouraged the discontinuous dynamic recrystallization (DDRX).

Most researchers [8–10] completed the hydrogenation process by holding the materials in hydrogen included environment at high temperature (750 °C) for several hours. Its low efficiency and longtime cost restricted the wide use. In this study, hydrogen was added into the materials by melting the alloys in gas mixture of hydrogen and argon, hydrogenation and fabrication were completed at the same time. In others work [11–14], this method was called melt hydrogenation, Wang [15,16] investigated the microstructure evaluation, hot deformation behavior and hydride formation in Ti64 alloy. Liu [17–20] studied hydrogen absorption, hot compression and mold filling behavior of TiAl alloy fabricated by melt hydrogenation. The acquired experimental results indicated that hydrogen decreased the flow stress and improved the hot workability of hydrogenated alloys, which was induced by improvement of softer  $\beta$ phase, DRX and dislocation mobility. However, the effect of melt



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hydrogenation on high temperature deformation of Ti64 alloy has not been systematically studied. Therefore, this study focused on the effect of deforming temperature, strain rate and hydrogen content on hot compressing behavior of melt hydrogenated Ti64 alloy, besides, the mechanism of evaluation on DRX and dislocations were also discussed.

### 2. Materials and methods

Ti64 alloy was synthesized by vacuum arc melting which was performed on vacuum arc furnace with tungsten gun and watercooled copper plate. Raw materials were titanium sponge (purity 99.9%), AIV alloy (58.14 wt.% vanadium) and aluminum (purity 99.999%). The samples were melted in gas mixture of hydrogen and argon, hydrogen percentage was controlled by JF-2200 system which was able to show the hydrogen partial pressure and total pressure of gas mixture in real time. Based on the results shown on JF-2200 system, hydrogen percentage of 10%, 20% and 30% in gas mixture were obtained. Each specimen was re-melted in gas mixture for 5 times to keep chemical composition homogenous, meanwhile, melting time and input power were kept all the same. Hydrogen content in as received Ti64 alloy was measured by weighting the specimen before and after dehydrogenation process to the accuracy of 0.01 mg, dehydrogenation process was performed on the vacuum annealing furnace at 750 °C for 6 h, working pressure was lower than  $5 \times 10^{-3}$  Pa. The relative experimental details of dehydrogenation had been published in the previous work from our group [21], verification of hydrogen content and other information can be found elsewhere [16]. This paper will not show the further details, and the relative hydrogen content were  $3.64 \times 10^{-2}$  wt.%,  $5.31 \times 10^{-2}$  wt.% and  $6.39 \times 10^{-2}$  wt.% when hydrogen percentage in gas mixture were 10%, 20% and 30%, respectively.

Samples for X-ray diffraction (XRD) analyze were pieces with size of  $10 \times 10 \times 2$  mm, which were cut from as cast alloys, after mechanical grind, the tests were conducted on D/max-RB X-ray diffractometer with scanning angle ranging from 20° to 80°. The as cast alloys were cut into cylinders with 6 mm in diameter and 9 mm in height, after mechanical grind and cleaned by ultrasonic wave, the cylinder samples were used for high temperature compression experiment. Before hot compression, all the samples were coated with glass lubricant which is a kind of silicone gel with good heat resistance and lubricity at high temperature, to prevent the escape of hydrogen from samples. Hot compression of Ti64 alloy was performed on Gleeble-1500D thermal simulation machine. High purity argon gas was put into the working chamber as protective gas during the hot compression after environmental pressure vacuumized to the lower than  $5\times 10^{-4}$  Pa. The samples were heated from room temperature to evaluated temperature in 10 °C/s and then hold at evaluated temperature for 3 min. Hot compressing temperature of 700, 750, 800 and 850 °C and strain rate of 0.001, 0.01 and 0.1  $s^{-1}$  were chosen in this study, after hot compression, the samples were quenched into water immediately to preserve the high temperature microstructure. The specimens for microstructure observation were cut from the center of as compressed alloy, after mechanical grind and polished, they were etched by solution of hydrofluoric acid, nitric acid and water (HF:HNO<sub>3</sub>:H<sub>2</sub>O is 1:1:8, volume fraction) for 10 s. Scanning electron microscopy (SEM) of as compressed alloy was conducted on FEI Quanta 200F scanning electron microscope in secondary electron mode. Specimens for transmission electron micrograph (TEM) were cut from the center of as compressed alloy, after mechanical grind to 60 µm, they were electrochemical polished by twin-jet polisher in solution of 6% perchloric acid, 34% n-butyl alcohol and 60% methanol (volume friction) at  $-25 \degree$ C for 45s, and same solution was used to prepare the samples for electron back-scatter diffraction (EBSD) observation, TEM observation was conducted on FEI Talos F200X transmission electron microscope, bright field images on observation of dislocations were taken under the same diffraction condition of  $\alpha$  tilt is 0° and  $\beta$  tilt is 0°, and EBSD observation was conducted on FEI Quanta 200F scanning electron microscope.

## 3. Results and discussion

#### 3.1. Hot compression at same strain rate

Fig. 1 shows the XRD results of as cast alloys with increasing hydrogen content, no hydride was detected due to the low hydrogen content, and the peaks of  $\beta$  phase move to lower angles. Hydrogen is interstitial atom, and the solubility of hydrogen in  $\beta$  phase is much higher than in  $\alpha$  phase. In this study, the solutes hydrogen atoms in  $\beta$  phase leading to the lattice expansion which decrease the Bragg angles of  $\beta$  phase. For example,  $5.31 \times 10^{-2}$  wt.% hydrogen decreases the Bragg angles of  $\beta$  phase from 39.318° to 38.566°. In comparison, the Bragg angles of  $\alpha$  phase keep in the same position. This may be the proofs that hydrogen dissolves into the  $\beta$  phase in as received alloys, as shown in Fig. 1.

Fig. 2 shows the true strain-stress curves of unhydrogenated and hydrogenated Ti64 alloy deforming at increasing temperature and same strain rate (0.01 s<sup>-1</sup>). According to the results shown by Zong [22], the  $\alpha/\beta$  transition point of unhydrogenated Ti64 alloy is 980 °C. In this study, the highest hydrogen percentage in gas mixture is 30%, and hydrogen content in Ti64 alloy is only 6.39  $\times$  10<sup>-2</sup> wt.%, therefore, hydrogen can decrease the  $\alpha/\beta$  transition point, but the deforming temperatures (700–850 °C) chosen in this paper are still in  $\alpha+\beta$  phase region.

According to the results shown in Fig. 2, work hardening effect can be found at the starting stage of compression in which flow stress increases and reaches the peak point. Working hardening is mainly induced by dislocation tangles and piles up. After the peak point, flow stress decreases gradually with increase of strain, which is induced by DRX. The flow stress is sensitive to the change of deforming temperature, flow stress decreases with increase of deforming temperature, which is induced by the increase of softer  $\beta$ phase content and decline of bonding force between atoms.

The peak stress of Ti64 alloy with increasing hydrogen content deformed at same strain rate (0.01 s<sup>-1</sup>) and increasing temperature (750 and 850 °C) is plotted as Fig. 3. When deformed at higher temperature (850 °C), peak stress decreases with increase of



**Fig. 1.** XRD results of as received Ti64 alloys with increasing hydrogen content. (1) Unhydrogenated; (2)  $3.64 \times 10^{-2}$  wt.% hydrogen; (3)  $5.31 \times 10^{-2}$  wt.% hydrogen; (4)  $6.39 \times 10^{-2}$  wt.% hydrogen.

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