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ARTICLE

Hot Deformation and Dynamic Recrystallization Behavior of Pure Titanium in EB Furnace Process

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Abstract: The hot deformation behavior of pure titanium in EB furnace process (EB-Ti) with centimeter scale original grain size at different deformation temperatures and deformation rates was studied by thermal compression experiment, and the recrystallization mechanism of EB-Ti was discussed based on the electron back scattering diffraction (EBSD) technology. The results show that the hardening behavior of EB-Ti in the process of hot deformation has typical "three-stage" characteristics. In the start stage, the hardening ability is rapidly linearly declined; in the middle stage, it rapidly recovers to a peak; in the final stage, it re-declines from the peak. The three-stage phenomenon is related to twinning in the deformation process. EBSD results also show that the recrystallization mechanism of EB-Ti in the process of hot deformation is mainly a discontinuous dynamic recrystallization.

Key words: pure titanium; EB furnace; hot deformation; dynamic recrystallization; mechanism

Titanium has the characteristics of high toughness, low density, high strength and excellent corrosion resistance, which makes it the most ideal structural material in large-scale equipment in aerospace, marine engineering and other fields[1]. At present, the production of titanium ingots mainly uses vacuum arc smelting (VAR) furnace, but the ingots produced by VAR furnaces cannot completely eliminate segregation and inclusion. With the rapid development of titanium industry, the requirements of metallurgy quality of titanium continue to improve. In order to obtain high purity and uniform composition of pure titanium ingots, electron beam smelting technology, which has the advantages of saving the process for forging the blank, directly rolling the ingots into coils and effectively removing volatile impurities and inclusions, etc, has been widely concerned by researchers. However, compared to titanium ingots produced by the traditional method, ingots produced by electron beam furnaces have a coarser original grain size of centimeter grade, which will affect the properties eventually. To improve the microstructure of titanium ingots, plastic deformation methods such as hot-rolled or cold-rolled annealing can be used to induce the dynamic recrystallization refinement or static recrystallization refinement to obtain a good strength and toughness match.

Salem et al[2,3] studied the work hardening behavior of pure titanium at room temperature, and found that the change of strain hardening rates is correlated with the onset of deformation twinning. Li et al[4] studied the dynamic deformation behavior of ultrafine titanium (average grain size is approximate 120 nm) and indicated that the strain rate sensitivity of ultrafine titanium is similar to that of coarse titanium (average grain size is approximate 72 μm). Yan et al[5] studied the influence of pre-activated twin crystals on dynamic recrystallization of industrially pure titanium at low temperatures and pointed out that the dynamic recrystallization process of pure titanium can be divided into twin-induced recrystallization and discontinuous dynamic recrystallization.

At present, researches of plastic deformation and re-

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crystallization behavior of pure titanium concentrate mainly on micron grade and millimeter grade pure titanium. The research of centimeter grade pure titanium is rarely reported. Hayama et al[6] studied the recrystallization annealing behavior of pure titanium with grain size of centimeter grade after cold-rolling deformation, and indicated that the recrystallization behavior is significantly influenced by deformation and annealing temperature. However, the research of hot-rolling deformation behavior of centimeter grade ultra-coarse pure titanium is almost non-reported.

1 Experiment

The experimental pure titanium ingot produced by EBM was a cuboid with 90 mm in length, 108 mm in width and 63 mm in thickness. Its chemical composition (wt%) was C 0.0072, N 0.0047, H 0.00092, O 0.045, Fe 0.016, and balance Ti. The microstructure is shown in Fig.1 (note the serrated grain boundary). The average grain size reached the centimeter scale. Samples for thermal compression experiments by Gleeble 3800 was taken from ingots with a size of Φ8 mm×15 mm and a surface roughness of R_a=0.7 μm. In order to study the effect of deformation temperature and strain rate on the thermal deformation behavior of the material, the specimen was firstly heated to different deformation temperatures (760, 800, 830, 850, 870 °C), and then started to deform at different strain rates (0.01, 0.1, 1 and 10 s^{-1}) until the true strain reached 0.92. To figure out the microstructure evolution in the thermal compression experiment, the deformation temperature was set at 830 °C and deform was conducted at strain rate of 1 s^{-1} until the true strain reached 0.10, 0.22, 0.36, 0.51, 0.69, 0.92. The temperature increment was 10 °C/s and holding time of deformation temperature was 3 min for all the thermal deformation experiments. The test for each deformation condition was repeated 3 times, and the stress-strain curves of the repeated tests showed a consistent pattern. After the experiments, all the samples were rapidly water-cooled to retain the high temperature microstructure.

The sample was longitudinally cut along the central axis, and the longitudinal section was observed by metallographic and EBSD method. The metallographic observation was carried out with a Zeiss 40MAT digital metallographic microscope. The etching solution was a mixture of hydrofluoric acid, nitric acid and water (1:3:6). The etching time was 10~20 s; the EBSD observation was carried out with Quanta650FEG thermal field emission scanning electron. In the EBSD experiment, the electron microscope voltage was 20 kV, the scanning area was 250 μm×170 μm, and the step size was 0.7 μm. The obtained data was processed by the matching HKL CHANNEL5 software. The solution used for electrolytic polishing was a mixed solution of perchloric acid and methanol, and the volume ratio is 1:9, and electrolytic polishing was performed at room temperature with the voltage of 20 V for 20 s.

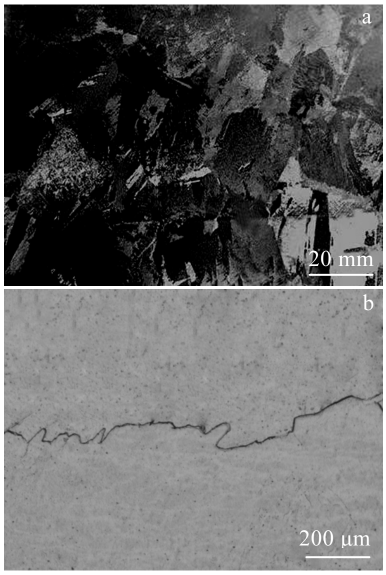

Fig.1 Microstructures of EB-Ti captured by stereo microscope (a) and metallurgical microscope (b)

2 Results and Discussion

2.1 Flow stress behavior and microstructure evolution

Fig.2 shows the true stress-true strain curves of the thermal compression experiment of EB-Ti under different deformation temperature and strain rate conditions. It can be seen that the work-hardening phenomenon of EB-Ti in the compression deformation experiment is quite significant in the case of low deformation temperatures and high strain rate conditions. The curve is fitted with 6-order polynomial. The polynomial is derived to the truth stress so that the work-hardening rate of EB-Ti under different thermal deformation processes can be obtained, as shown in Fig.3.

The work-hardening ability curve of EB-Ti shows an obvious "three-stage" characteristic. In the start stage, with the increase of flow stress, the hardening ability is rapidly linearly declined; in the middle stage, it declines to the bottom and rapidly recovers to a peak; in the final stage, it decreases rapidly from peak as the flow stress increases to a certain extent. It is well known that the finite independent slide system in titanium is not sufficient to coordinate plastic deformation in the deformation process, so the twin becomes the main deformation pattern in hcp metal and plays the key role in plastic deformation[7-9]. Salem[2, 3] and Kumar[10] found a similar phenomenon in the process of pure titanium cold deformation, which is related to the twin deformation mechanism of pure titanium during compression.

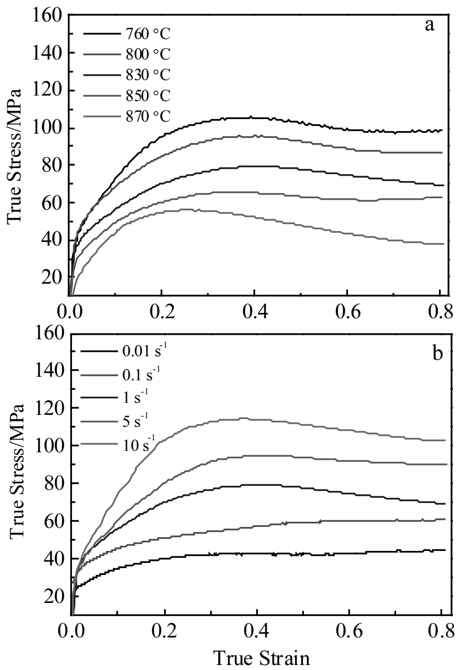

Fig.2 True stress-true strain curves of EB-Ti deformed at strain rate of 1 s^{-1} (a) and temperature of 830 °C (b)

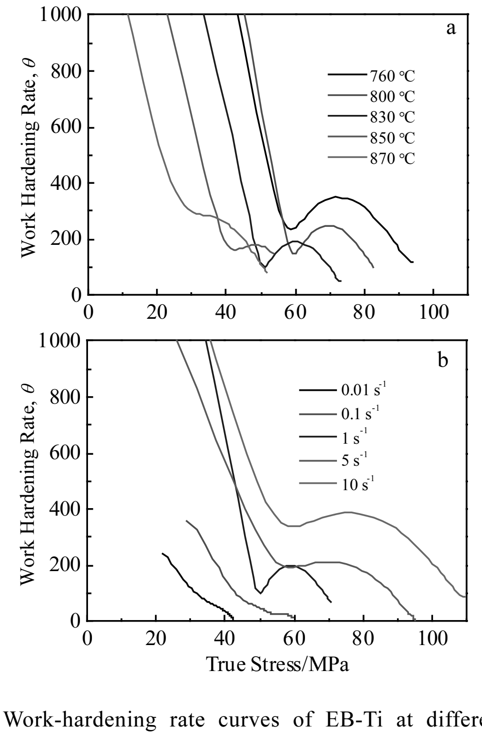

Fig.3 Work-hardening rate curves of EB-Ti at different temperatures (a) and strain rates (b)

In the start stage, there is almost no deformation twin produced from polycrystalline pure titanium, and the slip is the main deformation mechanism. In the middle stage, as the compression strain increases, the grains become flatten along the compression direction, and the orientation of some grains deflects perpendicular to the stress, which is conducive to the production of the twin; as the compression continues, the orientation of more grains deflects perpendicular to the stress, resulting in more twins until saturation. In the final stage, the existence of the twin boundaries makes the original grain fractured and refined. The fine grains hinder the further formation of twinning. Because of the refined grains, a large number of dislocations who blocked slip stood easy slip orientations. The slip of dislocations begins to dominate the deformation mechanism. The true stress-true strain curve alone is not sufficient to confirm the explanation above. A further microstructure evolution is also required (Fig.4). From Fig.2 and Fig.3, when the deformation temperature is 830 °C and strain rate is 1 s^{-1}, the true strain in the middle stage of the work-hardening is just 0.06. A large number of twins are found in the sample microstructure when the true strain is 0.10, as shown in Fig.4a. So the Hall-Petch hardening effect induced by twins will cause an increase in the work-hardening ability. As the strain further increases, the twin gradually disappears, and the refined grains increase, which is consistent with the pattern of reported in Ref.[2,3,7]. It is worth noting that with an increase in deformation temperature and a decrease in strain rate, the character of rapidly increased work-hardening ability in the middle stage is gradually insignificant. The formation of twins is inhibited under the conditions of high deformation temperatures and low strain rates, resulting in less contribution to the plastic deformation of pure titanium.

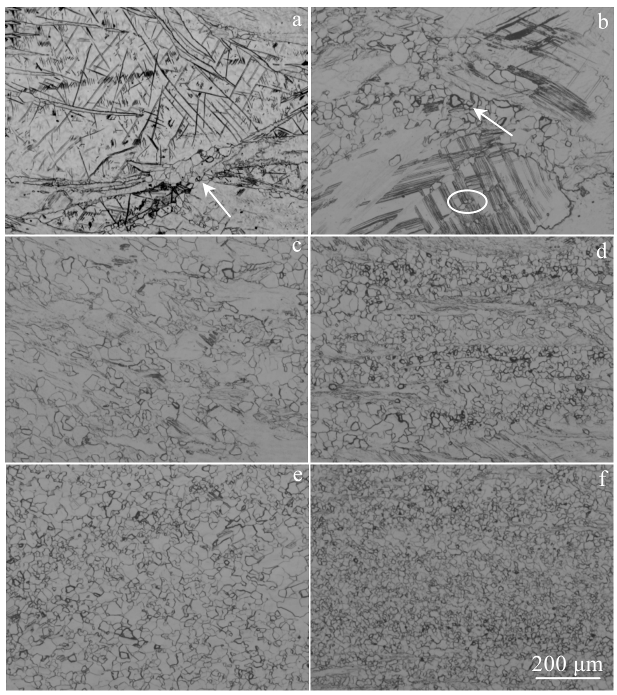

Fig.4 Microstructures of EB-Ti deformed at temperature of 830 °C and strain rate of 1 s^{-1} with different true strains: (a) 0.10, (b) 0.22, (c) 0.36, (d) 0.51, (e) 0.69, and (f) 0.92

2.2 Dynamic recrystallization mechanism

The "double-differentiation" method[11] was used to study the critical stress and critical strain of dynamic recrystallization in the process of thermal deformation of pure titanium, i.e. the strain is critical strain when $|-\partial(\partial\sigma/\partial\varepsilon)/\partial\varepsilon|=0$, as shown in Fig.5. Under deformation conditions of 830 °C in temperature and 1 s^{-1} in strain rate, the critical strain of dynamic recrystallization (DRX) of EB-Ti is about 0.08, which indicates that recrystallization can occur within the strain range set in this experiment.

Fig.6 shows the high and low angle grain boundary graph and average grain misorientation graph (GAM) at different strain, and microstructures in Fig.6a and 6b correspond to the arrows shown in Fig.4a and 4b. Rainbow legend was used to characterize the change in GAM. From blue to green to orange, GAM value gradually increases, representing the increasing density of defects inside the grain[12, 13].

As shown in Fig.1b, the original grain boundary of EB-Ti presents a large number of "bumped" or "jagged" structures, which provide sufficient strain energy and priority core position at the start of deformation. When the true strain reaches 0.10, a large number of twins can be observed, and a few DRX grains are found to be distributed in the original boundary. The GAM graph also shows that there is large strain energy at the original boundary (Fig.4a, Fig.6a). When the strain further increases, the number of DRX grains increases significantly. The DRX grains almost occupy the original boundary and the twin boundary, and developed into the internal of the deformed original grain. The results of the microstructure observed above show that the DRX of centimeter grade coarse titanium grain is nucleated through "bulge", and the fraction of DRX increases with increase of strain, which indicates that the DRX mechanism of EB-Ti in the process of thermal deformation is a typical discontinuous dynamic recrystallization (DDRX).

The "bulge" mechanism can describe the formation of the first recrystallization grains and the first layer of DRX grain from the original grain boundary. But this mechanism does not explain how DRX grains are further extended, eventually covering the entire metamorphic grains. Research suggests that the first layer of new grain on the original boundary will stop growing when it grows to a steady recrystallized size. This grain layer stops growing up. If the deformation continues to make the dislocation density increase, there will be the next round of DRX in this layer of the newly formed grain and the original grain interface nucleates in the "bulge". The process above will continue to loop until the original deformation grain is completely consumed, and eventually replaced by newly recrystallized grains. This is consistent with what Ponge[14] and Mao reported[15]. The entire process

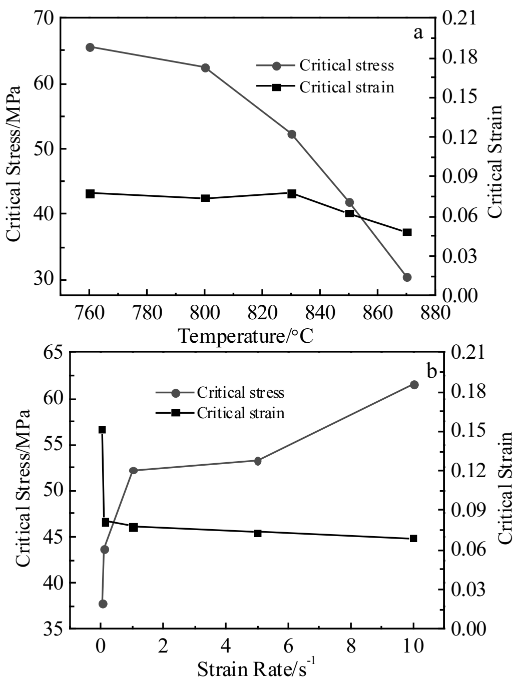

Fig.5 Critical stress and critical strain at different deformation temperatures (a) and strain rates (b)

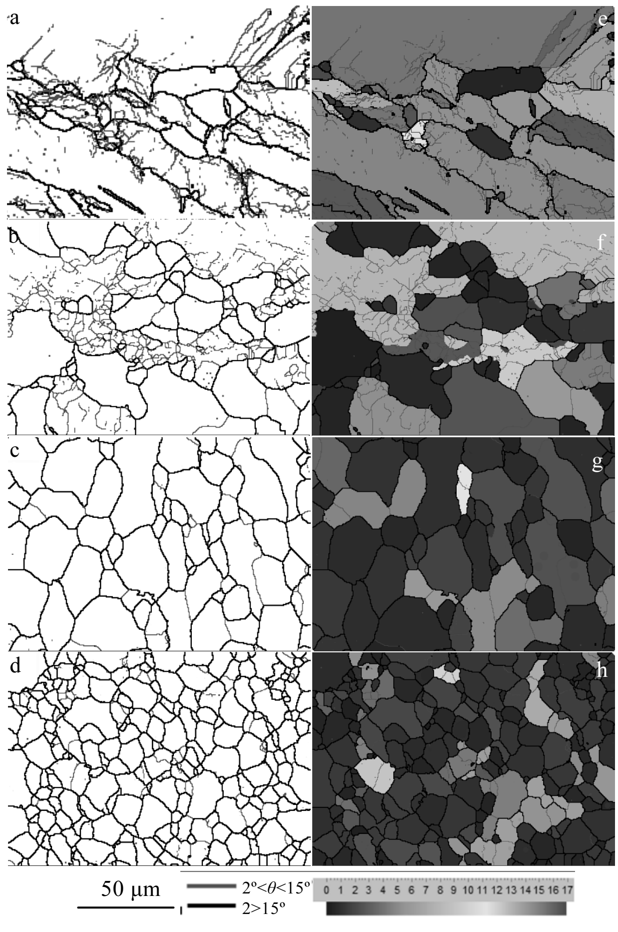

Fig.6 High and low angle grain boundary (a~d) and GAM graph (e~h) at 830 °C and 1 s^{-1} with different strains: (a, e) 0.10, corresponding to arrow in Fig.4a; (b, f) 0.22, corresponding to arrow in Fig.4b; (c, g) 0.51; (d, h) 0.92

of DRX from the beginning of the nucleus to the end can be approximated described in Fig.7.

The nucleation of DRX in the thermal deformation grain basically depends on the formation of movable grain boundary. The ability of grain boundary migration is enhanced with the increase of the misorientation, but the existence of the 10°~15° misorientation in the subcrystalline is a necessary condition for the nucleation of DRX[16]. The DRX nucleation in the subcrystalline structure involves in the rotation of subcrystalline, which is a typical symbol of continuous dynamic recrystallization (CDRX) mechanism. To study the effect of subcrystalline rotation on the DRX nucleation, the pattern of the misorientation range of EB-Ti at different strains was investigated, as shown in Fig.8. It can be seen that the ratio of low angle grain boundaries decreases generally with the increase of deformation, while the ratio of high angle grain boundaries shows the opposite, which indicates that the transition from low angle grain boundaries to high angle boundaries corresponds to the increase of strain. However, the medium angle grain boundary maintains low under any deformation conditions (less than 2%). This phenomenon is also observed in nickel-based alloys and Al-Mg alloys[17,18]. A large number of studies[19,20] have shown that when CDRX of metal material occurs in the process of thermal deformation, the ratio of the medium angle grain boundary will increase significantly. Therefore, it can be considered that CDRX of EB-Ti is not an effective DRX mechanism during thermal deformation process, while DDRX is the main DRX mechanism. It is worth noting that the deformed twins also have some

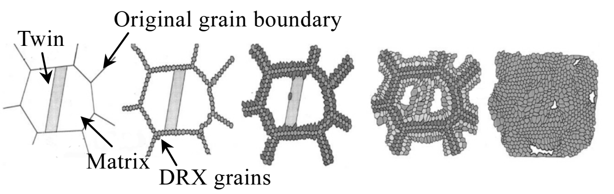

Fig.7 Development process of dynamic recrystallization "necklace" microstructure (increased in volume from left to right)[13]

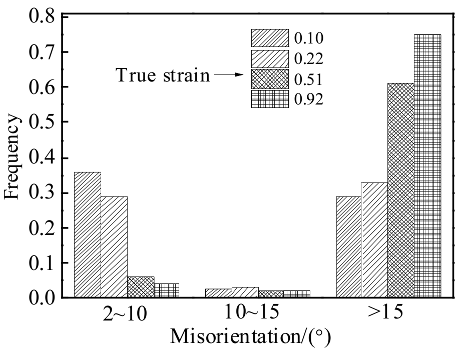

Fig.8 Effect of true strain on low angle (2°~10°), medium angle (10°~15°) and high angle (>15°) grain boundaries at 830 °C and 1 s^{-1}

influences on the recrystallization behavior of EB-Ti, which will be further studied in the future work.

3 Conclusions

1) EB-Ti with a centimeter-grade original grain size has the "three-stage" characteristic of work-hardening behavior in the process of hot deformation. In the start stage, the work-hardening ability is rapidly linearly declined due to dynamic recovery; in the middle stage, it is the recovery because of the existence of the twins; in the final stage, the work-hardening ability decreases rapidly from peak as the stress increases to a certain level.

2) The recrystallized grains have a low average grain misorientation. And the discontinuous dynamic recrystallization is the main recrystallization mechanism of EB-Ti in the process of thermal deformation.

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EB 炉流程纯钛的热变形和动态再结晶行为

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摘 要：通过热压缩试验研究了原始晶粒尺寸为厘米级的 EB 炉流程纯钛在不同变形温度和变形速率下的热变形行为，并基于电子背散射衍射(EBSD)技术对 EB 炉流程纯钛的再结晶机制进行了探讨。结果表明：厘米级的 EB 炉流程纯钛在热变形过程中的加工硬化行为具有典型的“三阶段”特征：第 1 阶段线性快速下降；第 2 阶段迅速回升至一个峰值；第 3 阶段从峰值又开始下降，这与变形过程中的孪生现象有关；EBSD 结果表明超粗晶粒纯钛在热变形过程中的再结晶机制主要是非连续动态再结晶。

关键词：纯钛；EB炉；热变形；动态再结晶；机制

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