Effect of high temperature ECAP process on microstructure and mechanical properties of Ti-5553 alloy

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Abstract. Excellent mechanical properties are the key to improve the erosion resistance of materials. Superplastic deformation process ECAP is a new heat treatment method which can refine grain size, improve the microstructure of materials and the comprehensive mechanical properties of materials. In this paper, the microstructure of Ti-5553 alloy treated by ECAP process and Ti-5553 alloy without ECAP process were observed, and the effect of high temperature ECAP process on the microstructure of Ti-5553 alloy was discussed. The effect of high temperature ECAP process on the mechanical properties of Ti-5553 alloy was studied by tensile test, and the effect of high temperature ECAP process on refining microstructure and improving mechanical properties of Ti-5553 alloy was discussed.

1. Introduction

Near beta titanium alloy has been widely used in aerospace, marine and other fields due to its high specific strength, good fracture toughness, good corrosion and oxidation resistance[1-3]. Compared with other near beta titanium alloys, Ti−5Al−5Mo−5V−3Cr(Ti-5553) is a new near beta titanium alloy[4,5]. The alloy has the advantages of good hardenability, high strength and high fracture toughness. It is an ideal material for manufacturing high strength fuselage forgings[6,7]. Qin et al.[8] studied the mechanical properties, damage behavior and fracture mechanism of different Ti-5553 alloy structures (equiaxed, lamellar and bimodal structures) in tension and compression experiments. In Ti-5553, Fan et al.[9] studied the properties of unique compressive yield behavior and the dynamic response of three different initial microstructures (single crystal, rod like β and sheet α).Welk et al.[10] study shows that when the alloy is heated from low temperature to 838 °C, the isomorphic transformation from dense hexagonal α lattice to BCC β lattice occurs, and when the alloy is cooled at high temperature, the transformation phenomenon of BCC β lattice to dense hexagonal α lattice occurs.

The strength of the alloy can be further improved by severe plastic deformation (SPD)[11,12]. The most widely used SPD method is equal channel angular pressing (ECAP)[13]. Tang et al.[14] applied ECAP extrusion process to Mg-Zn-Y-Zr alloy to study the effect of ECAP extrusion temperature on microstructure, texture evolution and mechanical properties of Mg alloy. The results show that when the extrusion temperature is 473K, the grain size of Mg-Zn-Y-Zr alloy extruded by ECAP is much smaller than that at 623K. Radim Kocich et al.[15] carried out ECAP treatment on TiNi alloy at high temperature. The results show that the microstructure of TiNi alloy is less than 10um after one or two passes of ECAP extrusion. Xu et al.[16] studied the Ti-20Mo alloy prepared by ECAP and recorded the precipitation of α phase during aging in shear band in severely deformed materials. Jiang et al.[17] studied the effect of equiaxed α phase formation of Ti-5Al5Mo-5V-3Cr (Ti-5553) alloy after HPT on the improvement of material ductility. In this paper, the microstructure and mechanical properties of Ti-5553 alloy treated by ECAP process and Ti-5553 alloy without ECAP process were compared and
studied. The effect of high temperature ECAP process on the microstructure and mechanical properties of Ti-5553 alloy was discussed.

2. Experimental

2.1. Materials and preparation
A new metastable near beta Ti-5553 alloy ingot (200 mm × 200 mm × 150 mm) was provided by Baosteel Co., Ltd. Its nominal composition is shown in Table 1. The α→β transformation temperature (Tβ) of the ingot is determined and provided by the manufacturer, and the range is 830 °C ~ 840 °C.

Table 1. Chemical composition of Ti-5553 alloy [values were in Wt %]

|   | Al  | Mo  | V   | Cr  | Zr  | Ti   |
|---|-----|-----|-----|-----|-----|------|
|   | 5.00| 5.00| 5.00| 3.00| 0.65| Bal. |

After the surface of the ingot was removed by 3 mm, glass lubricant was coated on the surface to prevent high temperature oxidation. It was heated at 1100 °C for three hours and forged repeatedly from different directions with a 500 ton hydraulic press, resulting in a large deformation of 60%. The ingot is finally forged into a thin plate with a thickness of 11 mm, and then air cooled to room temperature. A rectangular billet with the size of 10 mm×10 mm×80 mm was processed by wire electrical discharge machining (WEDM). Finally, all samples were ground into 9.7 mm×9.7 mm×80 mm blocks to remove the damaged layer caused by spark.

2.2. ECAP process treatment
Before ECAP extrusion, glass lubricant was applied on the surface of Ti-5553 sample and ECAP mold channel to reduce the friction between the sample and the mold; After that, the ECAP mold was heated to 550 °C at 840 °C for 20 min, which was higher than Tβ. ECAP mold is heated to 550 °C. After that, the Ti-5553 sample was immediately taken out from the preheating furnace and sent to the ECAP mold. The sample was subjected to one pass equal channel angular pressing on the 300t universal vertical hydraulic press produced by Huzhou machine tool factory, as shown in Figure 1. The whole extrusion process is completed in about 3 s. After ECAP treatment, solid solution was performed at 820 °C and aged at 620 °C for 4 h [18] to obtain uniform microstructure. The sample treated by ECAP at high temperature, then solution treated and aged treated, is named E840, and the sample without ECAP treatment is named NE.

2.3. Characterization of microstructure
The microstructure of E840 and NE was observed by OLYMPUS BX51ME optical microscope after ECAP extrusion.
2.4. Mechanical property test
In order to evaluate the mechanical behavior of E840 and NE, according to GB/T 228.1-2010 [19] standard, the two kinds of specimens were processed into tensile specimens, and their shapes are shown in Figure 2. The length of parallel part is 30mm, the width is 10mm and the thickness is 2mm. The uniaxial tensile test at room temperature was carried out with Zwick BTC-T1-FR020 testing machine at the strain rate of 0.005s⁻¹.

3. Results and discussion

3.1. Microstructure of Ti-5553 alloy
As shown in Figure 3, after ECAP treatment at 840 °C and subsequent solution and aging treatment, the optical microstructure of E840 sample is composed of numerous fine α phases dispersed in β matrix (as shown by black spots); while the sample NE without ECAP treatment is forged at 840 °C and then air cooled, its optical microstructure is metastable β polygon coarse grain with diameter of 800 μm, There are few large black spots in the microstructure.

The microstructure of NE of Ti-5553 alloy forged at 840 °C and then air cooled is mostly metastable β - polygon coarse grains. It can be seen that there are enough stable elements of β phase in the original Ti-5553 sample (in the original forging structure) [20]. The existence of a large number of β phase stable elements in Ti-5553 alloy is beneficial to ECAP extrusion, which is also the reason why ECAP will not lead to the rapid growth of β grains when the extrusion temperature is higher than the α→β transformation temperature [21]. In the microstructure of sample E840, there are many fine α particles in β grain. This phenomenon can be explained by two reasons: the first reason is that the ECAP extrusion process of sample E840 is completed in a short time, and the phase composition of sample E840 remains a single β phase without α - grain precipitation [23]; The second reason is due to the large deformation of E840 in ECAP process, and its equivalent effect is as high as 0.91. High density crystal defects, especially dislocations, have been shown in the sample. These crystal defects do not disappear with the rapid water cooling, but become the potential nucleation points of α particles precipitation in the solid solution aging process. Therefore, with the increase of nucleation of α particles and the vacancy of primary α phase, a lot of fine α grains are produced in β matrix of sample E840.
3.2. Study on mechanical properties of Ti-5553 alloy

The tensile stress-strain curves of specimen NE and E840 were obtained. It can be seen from the figure that the maximum stress and fracture strain of E840 are higher than those of sample NE. The yield strength of sample NE is 1100 MPa, and that of E840 is 1500 MPa. After ECAP treatment, the yield strength of specimen is increased by 36%. The fracture failure of specimen NE occurred at 9% strain, while that of E840 specimen occurred at 15% strain. The strain of E840 increased by 66%. The fracture toughness of sample NE is 47.8 MPa·m\(^{1/2}\), while that of E840 is 71.4 MPa·m\(^{1/2}\). The fracture toughness of the sample after ECAP treatment is increased by 49.4%.

3.3. Mechanism of ECAP process on mechanical properties of Ti-5553

The yield strength and elongation of E840 are higher than those of NE. This is due to the introduction of fine dispersed second phase (α phase) into the microstructure of E840 sample after ECAP treatment, and the solid solution element Al is produced. In addition, the grain size of β phase becomes smaller after solution treatment and aging treatment. The second dispersed phase in Ti-5553 alloy is harder than that in β matrix phase, so the strength of E840 treated by ECAP is higher. According to the Orowan mechanism, the volume fraction of the second phase is a certain amount, and the strength increment generated by the second phase is proportional to R\(^{-1}\), where R is the radius of the second phase. The fine, dispersed and hard α phase is strengthened by high mass fraction of Al (5.03%), which can effectively block the dislocation and increase the strength of the sample. Therefore, the yield strength of E840 increases after ECAP treatment. These large amount of β phase stabilizer elements can effectively reduce the growth of β phase, which is helpful to obtain smaller grain size of β matrix in E840 sample. Reducing the grain size can not only improve the strength and fracture toughness of the alloy, but also reduce the stress concentration in the tensile test, so the tensile elongation of sample E840 is increased.

Obviously, ECAP plays a key role in improving the mechanical properties of E840. The main principle of ECAP deformation of the sample is shown in Figure 6, which can be summarized as follows: the sample extruded by ECAP produces large deformation strain, and the coarse β grain changes into a large number of sub grains. After solution treatment, these defective β-grains are stored at room temperature, and then aged. At the initial stage of aging treatment, the defective β-grains recrystallize, and the small equiaxed β-grains begin to nucleate. Due to the existence of a
large number of $\beta$-phase stabilizer elements in Ti-5553 alloy, the $\beta$-grains did not grow rapidly and remained in a small size. In the solution treatment stage, ECAP introduces enough crystal defects to promote the nucleation of $\alpha$-particles, which results in the decrease of $\alpha$-grain size and the increase of $\alpha$-particle precipitation. The probability of fine $\alpha$-phase penetrating into the grain boundary is higher. $\alpha$-phase plays a fixed role in the grain boundary like nails. Therefore, the comprehensive mechanical properties of E840 of Ti-5553 alloy after ECAP extrusion are improved.

Figure 6. The role of ECAP and subsequent heat treatments.

4. Conclusion
In this paper, the mechanical properties of Ti-5553 alloy treated by high temperature ECAP, followed by solution treatment and aging treatment, and Ti-5553 alloy without ECAP treatment were tested. The test results were analyzed. The influence of high temperature ECAP process on the microstructure and mechanical properties of Ti-5553 alloy was clarified.

1. The microstructure of Ti-5553 alloy treated by ECAP at 840 °C followed by solution treatment and aging treatment is deformed, and numerous fine $\alpha$ phases are dispersed in the $\beta$ matrix, and the grains are significantly refined.

2. ECAP is a very effective way to improve the mechanical properties of Ti-5553 alloy. The yield strength, elongation and fracture toughness of Ti-5553 alloy can be effectively improved by ECAP extrusion at high temperature higher than $\alpha \rightarrow \beta$ transformation, followed by solution treatment and aging treatment.

3. The microstructure of Ti-5553 alloy treated with ECAP followed by solution treatment and aging treatment has very fine $\alpha$ particles dispersed in the $\beta$ matrix.

4. High temperature ECAP heat treatment can significantly improve the yield strength and tensile elongation of Ti-5553 alloy, which is mainly due to the introduction of $\alpha$ phase in ECAP heat treatment process is harder than $\beta$ matrix phase, which can effectively block dislocation movement. In addition, Ti-5553 alloy has a large number of $\beta$ phase stabilizer elements, which can effectively reduce the growth of $\beta$ phase.

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