Synergistic enhancement of strength and toughness of β-Ti alloy with a fine grain: Effect of deformation temperature

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Abstract: It is difficult for as-cast titanium alloys to achieve high strength and toughness due to the presence of coarse β grains. In this study, multi-directional forging (MDF) was employed to break down the coarse β grains into fine grains and the mechanism of strengthening and toughening was also revealed. Results indicated that nearly all peaks corresponded to the β phase, with the α phase peaks being almost imperceptible. As the MDF temperature increased, the grain size initially decreased and then increased. All the dislocation is concentrated near the grain boundaries. The refinement of the grains can be attributed to the combined effects of mechanical crushing refinement and dynamic recrystallization. The tensile strength and fracture toughness is 1130 MPa and 82 MPa·m1/2. Grain refinement and dislocation proliferation caused by MDF are the main contributors to the improvement of strength and toughness in the alloys. This discovery lays a solid foundation for further research on new ultra-high strength and toughness titanium alloys, while also providing a new design concept for other high-performance alloys.

1 Introduction

High-strength and tough titanium alloys (tensile strength≥1000 MPa, fracture toughness≥55 MPa·$m^{1/2}$) play a crucial role as structural materials in aerospace and other fields, representing one of the most crucial directions in titanium alloys development [1]. However, the practical application of titanium alloys is often limited by their coarse grain size in the as-cast state, necessitating specific thermal processing to refine the grains. According to the Hall-Petch formula, the yield strength of alloys significantly increases as the grain size decreases, leading to a notable increase in elongation [2]. Additionally, grain refinement can significantly enhance the fatigue performance, impact resistance, corrosion resistance, and processing super-plasticity of titanium alloys [3]. Therefore, grain refinement stands out as an ideal method for material strengthening and a key approach to enhancing the overall performance of titanium alloys.

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Forging is the primary hot working method for high-strength and tough titanium alloys. The main objective of forging is to refine grain size and regulate the microstructure to meet the comprehensive performance requirements of the alloys for plasticity and toughness [4]. Among various forging techniques, multi-directional forging (MDF) proves to be a significant approach to achieve microstructure refinement [5]. By continually changing forging directions, the microstructure can be uniformly refined from all angles. The study by Yang et al. [6] showed that the alloy can be refined by MDF after 4 passes with 40% reduction per compression at 1200°C. The results of Zhang et al. [7] also confirmed that the homogeneity of microstructure and mechanical properties were improved with the MDF steps increasing. The choice of MDF temperature plays a critical role in controlling microstructure, encompassing both dynamic (deformation process) and static (heating and insulation process) recrystallization processes during deformation [8]. Understanding the correlation between microstructure and properties of titanium alloy enables the prediction of various mechanical properties in advance. Owing to multiple factors like phase transformation and thermal coupling in titanium alloys, their microstructures exhibit diversity. The relative content, morphology, size, and distribution of the two phases within the microstructure decisively impact the mechanical properties of titanium alloys [9]. When the deformation temperature surpasses the β phase transition point, dynamic recrystallization (DRX) of β grains predominantly occurs [10], emphasizing the importance of refining β grains in hot working. Consequently, conducting hot deformation in the β single-phase region helps avoid the influence of α phase on strength and toughness, allowing for a focused study on the size and distribution of the β phase concerning alloy strength and toughness.

Regarding the aforementioned researches, the control of grain size in titanium alloys plays a critical role in determining their mechanical properties, as well as revealing important strengthening and toughening mechanisms. However, the coarse β grain size and the presence of the α phase in titanium alloys restrict the ability to investigate the relationship between β grains and the strength and toughness independently. To address this issue, this study conducted MDF above the β phase transition point to achieve grain sizes below 100 μm, resulting in a fine-grained microstructure for a high-strength and tough titanium alloy. Tensile performance and fracture toughness are evaluated, and the strengthening and toughening mechanisms are systematically analyzed without the interference of the α phase. This research has established a solid foundation for designing ultra-high strength and toughness titanium alloys, while introducing a novel approach to enhancing performance of other high performance alloys.

2 Experimental materials and procedures

The experimental material was Ti-6Mo-4Al-4Zr-3Nb-2Cr-1Fe (referred to as Ti644321) alloy with a size of 40×40×40 mm. This alloy was melted using vacuum arc furnace for four times. The phase transition temperature of the alloy was measured by quenching metallographic method to be 815°C. The MDF experiment was conducted on the sample under different temperature conditions (820, 860 and 900°C). Before MDF experiment, the mold was heated in the furnace to the designed temperature, and then the samples were placed in the mold to be heated to the deformation temperature and kept warm for 20 min. Then, forging was carried out using a press. The forging process began by applying pressure along one side of the sample, followed by two rotations of 90° and forged it separately. After deformation occurred in all three directions mentioned above, a MDF process was completed. A total of three passes of MDF were carried out, with a deformation amount of 50% in each direction for each pass. The samples of 8×8×5 mm were cut from the center of the MDF sample. Phase composition analysis of MDF samples at different temperatures was performed using an Axios-PW4400 X-ray Spectrometer. The microstructure of the alloys, including phase distribution, composition, and morphology, was characterized and analyzed using electron backscattered diffraction (EBSD, SUPRA55) and scanning electron microscopy (SEM, Merlin Compact). The tensile performance test and fracture toughness test were both conducted on an electronic universal materials testing machine (Instron 5569). The tests were conducted three times to obtain an average value to avoid errors. The tensile specimen had a dog-bone shaped with dimensions of 15×2.2×2 mm while the fracture toughness specimen measured 16×4×2 mm.

3 Results and Discussions

3.1 Microstructural characteristics

Phase composition analysis was conducted on MDF alloys at different temperatures, and the results are shown in Fig. 1(a). Almost all peaks corresponded to the β phase, while the peaks corresponding to α phase are almost invisible, which is due to the transformation from α phase to β phase above the β phase transition point. Observing the internal microstructure showed varying degrees of fragmentation in β grains. The alloy MDF at 820℃ exhibits a significant elongation, while the grains of the alloy MDF at 860 and 900℃ mostly remain equiaxed. Due to forging in all three directions, most of the β grains are equiaxed, but some remained elongated. Additionally, small equiaxed β grains are found distributed along the boundaries of elongated β grains, indicating the occurrence of dynamic recovery (DRV) and DRX of the β phase under these conditions. Compared to the microstructure at low temperatures, the microstructure of the alloy MDF at 900℃ exhibits better uniformity. An increase in deformation temperature and dynamic recrystallization resulted in a more uniform microstructure distribution and more uniform material properties. Besides grain morphology, the size of the β grains also underwent significant changes. Due to the obvious elongation state, the alloy MDF at 820℃ has the largest grain size, while the alloy MDF at 900℃ exhibits a significant growth phenomenon in microstructure. Therefore, the alloy MDF at 860℃ has the smallest β grains with 81 μm among all three temperatures. Observing the morphology and distribution of dislocations, it is found that the dislocation density in all three samples was high and concentrated near the grain boundaries.

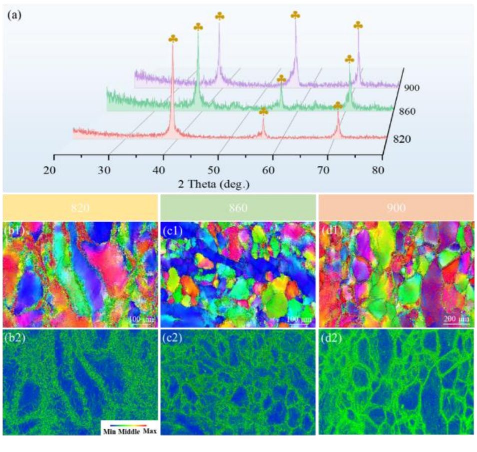

Fig. 1 (a) XRD results of Ti644321 alloys at different MDF temperature, (b1, c1, d1) the IPF images of Ti644321 alloys at different MDF temperature, (b2, c2, d2) the KAM maps of Ti644321 alloys at different MDF temperature.

Fig. 2 shows the enlarged β grains and internal microstructure. Different MDF temperatures resulted in significant differences in the morphology of β grains. MDF at 820℃ results in a significant elongation of β grains and an uneven microstructure with bent and a clear serrated shape β grain boundaries. No α phase is found internally, which is consistent with the XRD results. Many small recrystallized grains are found surrounding the elongated β grains, consistent with the EBSD results. Severe lattice distortion is present at the grain boundaries, resulting in high energy and hindering the movement of dislocations. The dislocations are accumulated and strain energy is increased. Therefore, the recrystallized core will be preferentially generated here [11]. The microstructure of MDF at 860 and 900℃ exhibits good uniformity, clear β grain boundaries, and an equiaxed shape. Similarly, serrated β grain boundaries are observed in both temperatures. However, compared to the microstructure at lower temperatures, the microstructure of MDF at 900℃ exhibits significantly growth. As the deformation temperature increases, there is a phenomenon of grain growth and mutual merging, resulting in relatively coarse grains at high temperatures. This is because the increased deformation temperature accelerated the atomic activity, which accelerates the interface migration rate and grain merging [12].

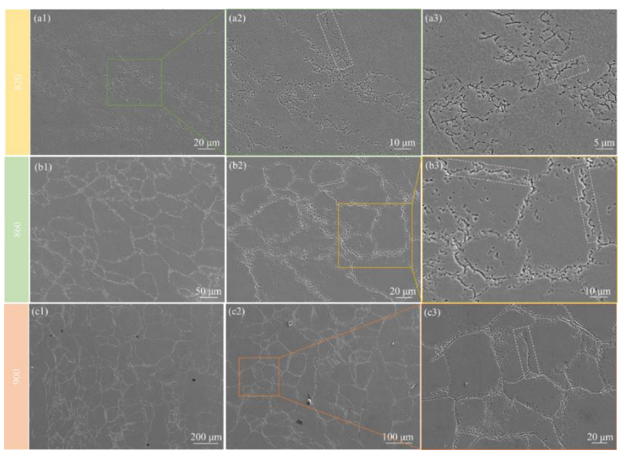

Fig. 2 The morphology of β grains of Ti644321 alloys at different MDF temperature.
(a1-a3) 820℃, (b1-b3) 860℃, (c1-c3) 900℃

3.2 Tensile properties and fracture toughness

Grain refinement significantly improves the strength and toughness of alloys. The tensile properties of the MDF sample are shown in Fig. 3. Compared to the as-cast alloy, the fragmentation of β grains leads to significant improvements in strength and plasticity. However, the difference in strength between MDF samples at different temperatures was not significant, possibly due to the small difference in grain size. Among the three MDF temperatures, MDF at 860℃ resulted in the highest strength value of 1130 MPa, and MDF at 900℃ resulted in the highest tensile strain of 25% for the alloy. For the alloy MDF at 860℃, the strength increased by 34.7%, and the plasticity increased by 262%.

As the MDF temperature increased from 820 to 860°C, the toughness values are the 75, 82 and 77 MPa·m$^{1/2}$. Compared to as-cast alloys, the toughness has increased by 60%. Based on the strength values mentioned earlier, it can be found that the alloys obtained through MDF above the β phase transition point meet the requirements of high-strength and tough titanium alloys. This discovery lays a solid foundation for the research on new ultra-high strength and toughness titanium alloys, and provides a new design concept for other high-performance alloys.

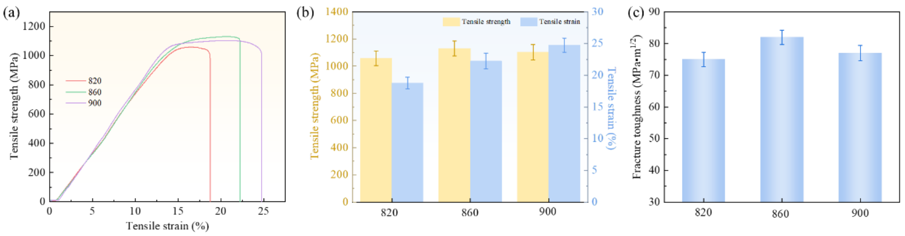

Fig. 3 (a) the tensile curves of Ti644321 alloys at different MDF temperature, (b) the values of tensile strength and tensile strain, (c) the values of fracture toughness.

3.3 The mechanism of grain refinement and strengthening-toughening

During the forging process of MDF, the microstructure is refined from various directions due to continuous changes in loading direction, resulting in uniform deformation. The alloy undergoes deformation within the recrystallization temperature range, with DRX and mechanical crushing refinement mechanisms working simultaneously. Initially, mechanical crushing plays a dominant role, but as deformation progresses, accumulated plastic deformation decreases the DRX temperature. Fine new grains with large angle grain boundaries begin to sprout at the initial grain boundaries, increasing in number as deformation continues. Subgrains elongate transversely and tend towards equiaxation, while the orientation difference between subgrain boundaries and initial grain boundaries increases, promoting the generation of new fine grains continuously. The alloys of MDF at the single-phase region leads to a microstructure mainly consisting of β large grains. During MDF, β large grains are flattened and elongated along the direction of metal flow, with increased temperature from deformation providing a driving force for β grain recrystallization. With the increase of deformation temperature, the migration ability of grain boundaries is enhanced, and the atomic thermal vibration and diffusion rate in the alloy are intensified [13]. The slip, climb and cross slip of dislocations are easier than at lower temperatures, which is conducive to the nucleation and growth of DRX, and the grain size is significantly refined. In addition, an increase in deformation temperature can increase the fluidity of the metal, reduce deformation resistance, and make deformation more uniform [14]. When the temperature is too high, the grains grow rapidly. This is because as the temperature increases, alloy elements or impurity elements on the grain boundaries dissolve into the interior of the grains, reducing the pinning effect on the grain boundaries and leading to grain growth.

Compared with as-cast alloys, the biggest difference between alloys after MDF is the refinement of β grains and the proliferation of dislocations. In the MDF process, as the MDF temperature increases, the coarse original β grains break and the β grains become smaller. Due to the Hall-Petch relationship [15], grain refinement can improve the strength of materials, resulting in the highest strength at 860°C with the smallest grain size. As the temperature rises to 900°C, the β grain size increases and the effective slip length of the alloy increases. However, at this point, the uniformity of the microstructure is good, resulting in a slight decrease in strength. In addition, a large number of dislocations generate at grain boundaries due to deformation. Dislocation strengthening is also one of the most effective strengthening methods in metal materials. High density dislocations are prone to cross each other during their motion, forming cutting steps and causing dislocation entanglement. This creates obstacles to the movement of dislocations, making it difficult to continue plastic deformation, thereby improving the strength of the alloy. The fracture toughness is closely related to the effective crack length and is controlled by the energy principle [16]. The direction of the crack always extends along the path with the minimum propagation resistance. As the MDF temperature in the single-phase region increases, the β grains in the microstructure are refined, the grain boundaries are continuous, and the α/β phase interface increases. Therefore, the hindering effect during crack propagation is enhanced, thereby absorbing greater energy and resulting in higher fracture toughness. On the other hand, the movement of dislocations within the plastic zone at the crack tip can alleviate stress concentration at the crack tip and improve toughness to a certain extent. In summary, grain refinement and dislocation proliferation caused by MDF are the main reasons for improving the strength and toughness of alloys.

4 Conclusions

The coarse β grains of as-cast titanium alloy are broken down into fine grain through MDF. This discovery lays a solid foundation for further research on new ultra-high strength and toughness titanium alloys, while also providing a new design concept for other high-performance alloys. The refinement of the grains can be attributed to the combined effects of mechanical crushing refinement and dynamic recrystallization. The strength and toughness exhibited an initial increase followed by a decrease with temperature, peaking at 860°C. The tensile strength and fracture toughness is 1130 MPa and 82 MPa·$m^{1/2}$, which is increased by 34.7% and 60% compared to as-cast alloy, respectively. Grain refinement and dislocation proliferation induced by MDF are key factors contributing to the enhancement of strength in the alloys.

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