

Effect of the initial grain size on grain refinement in Ti–2Al–2.5Zr alloy subjected to multi-impact process

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ABSTRACT

Effect of the initial grain size on plastic deformation behavior and grain refinement mechanism has been investigated in Ti–2Al–2.5Zr alloy subjected to multi-impact process. The results show that grain refinement is dominated by dislocation slip in the fine-grained sample. In contrast, deformation twinning becomes prevalent in the coarse-grained one, and contributes to grain refinement by means of twin subdivision and fragmentation. Dynamic recrystallization (DRX) takes place in both samples. But, it is delayed in the coarse-grained one due to relative low fraction of grain boundaries and energy dissipation of deformation twinning. Nevertheless, the final grain size in the refined area does not show an obviously difference between the two samples at a large strain, i.e., 1.61. Moreover, grain refinement is inhomogeneous in both samples. The heterogeneity in the coarse-grained sample is much stronger than that in the fine-grained one. It was ascribed to the superimposed effect of large initial grain size and subsequent deformation twinning. A criterion for achieving an optimum grain refinement was finally suggested.

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1. Introduction

Slip and twinning are two predominant plastic deformation modes in metallic materials. Their activity depends on material parameters and deformation conditions [1]. Generally, deformation twinning could be activated in the low-symmetry metals at room temperature due to their insufficient independent slip systems. It can also occur at a high strain rate and/or low temperature condition in the other metals [1]. Moreover, some experimental results demonstrated that it could be affected by the initial grain size [2]. The propensity for twinning was observed to decrease with decreasing the initial grain size in copper [3], brass [4], titanium [5] and magnesium [6]. When grain size decreases to a critical value, twinning could be completely suppressed [7]. In the nanocrystalline materials, twinning is first promoted and then suppressed with decreasing grain size, and displays an inverse grain-size effect [8].

Severe plastic deformation (SPD) has been developed as an effective technique to fabricate bulk nanocrystalline materials [9]. The relationship between initial microstructure, plastic deformation mode, and grain refinement mechanism during SPD has attracted strong research interest [10,11]. The initial grain size was observed to have an obvious influence on microstructural evolution and grain refinement mechanism in some materials. Belyakov

et al. found that a decrease of initial grain size could significantly accelerate the kinetic of grain refinement in 304 stainless steel [12]. Inoue et al. [13] pointed out that the difference of initial grain sizes gave rise to inhomogeneous distributions of microstructure and strain in high-purity aluminum even at the same reduction rate. These investigations, however, have been mainly concentrated on the high-symmetry cubic materials where dislocation slip is the primary plastic deformation mode irrespective of initial grain size. To the author's knowledge, little information is available in the low-symmetry materials where twinning plays an essential role in maintaining a homogeneous plastic deformation and the initial grain size has an obvious effect on deformation mode and microstructural evolution.

Ti–2Al–2.5Zr alloy possesses a hexagonal close-packed crystal structure (hcp) [14]. Twinning usually becomes activated during plastic deformation [14]. The low-symmetric hcp structure leads to the high deformation resistance. As a result, its grain refinement by the conventional SPD techniques, such as equal-channel angular pressing (ECAP), is extreme difficult at ambient temperature. A novel fabrication technique, named as multi-impact process, has been invented by our group recently so as to refine low-symmetry and hard-deformed materials via combining the applied plastic deformation and transformation-induced plasticity [15]. It has been successfully used in Ti–10V–2Fe–3Al alloy, in which extensive nanocrystalline structure and even amorphous were achieved [16]. The significant refinement was ascribed to the subdivision effectiveness of stress-induced martensitic transformation resulting in the initiation and growth of shear bands [16]. The objective of

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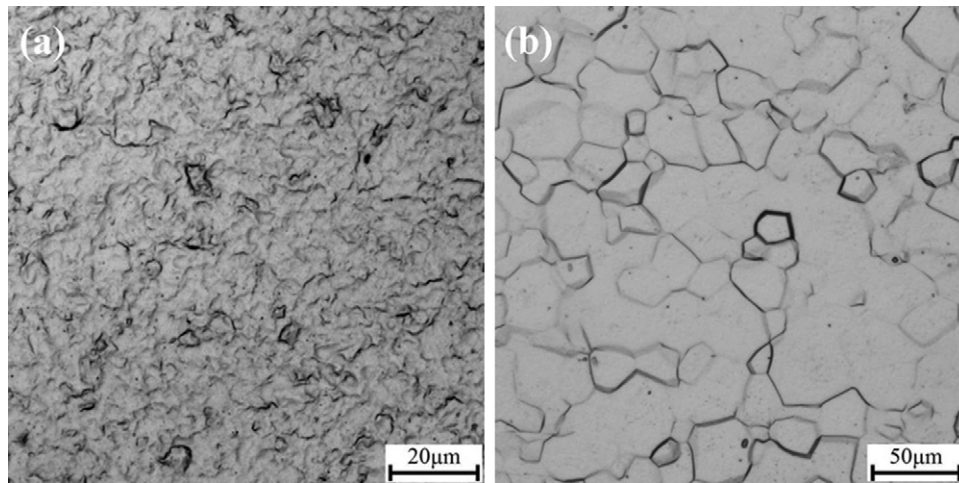


Fig. 1. The initial optical micrographs of Ti–2Al–2.5Zr samples: (a) the fine-grained sample; (b) the coarse-grained sample.

this work is to study grain refinement in Ti–2Al–2.5Zr alloy where plastic deformation-induced martensitic phase transformation is replaced by twinning during multi-impact process. The effect of initial grain size on microstructural evolution and grain refinement mechanism will be systematically analyzed.

2. Experimental procedure

The raw materials are the hot-rolled Ti–2Al–2.5Zr bars with 16 mm in diameter. The actual chemical composition is 1.59 wt.% Al, 1.91 wt.% Zr, the balance Ti. The stress-relief annealing was conducted at 550 °C for 1 h and cooled in air. The annealed microstructure is composed of equiaxed α grains of approximately 5 μm in diameter (Fig. 1(a)). Some segments were cut from the raw bars. They were annealed at 800 °C for 2 h in order to promote grain growth. Coarse grains about 40 μm in diameter were obtained (Fig. 1(b)). Two sets of segments with different initial grain sizes were machined into disc samples with dimensions of 2 mm in thickness and 16 mm in diameter for subsequent multi-impact process. The samples with an initial grain size about 5 μm and 40 μm hereinafter will be simply designated as the fine-grained sample and the coarse-grained one, respectively.

The multi-impact process was executed on the self-made device [15]. A variety of strains of 0.1, 0.35, 0.69, 1.2 and 1.61 were applied to the samples, respectively. The strain is defined as $\varepsilon = \ln(L_0/L_f)$, where L_0 and L_f are the initial and final thicknesses of the deformed samples. The strain rate applied to the sample is about 10^2 – 10^3 s^{-1} . The set-up and procedures of multi-impact process have been described in detail elsewhere [15].

Metallographic analysis was carried out on Olympus PMG3 optical microscope (OM). The samples were etched in a solution of 2% (volume) HF, 8% HNO_3 and 90% H_2O . The thin foils for TEM analysis were first mechanically ground to about 80 μm in thickness and thinned by twin-jet electrochemical polisher in a solution of 60% (volume) methanol, 35% butyl alcohol and 5% perchloric acid at 10–15 V and -30°C . The substructure was observed with a JEM-2100F transmission electron microscope (TEM) operated at 200 kV. Vickers hardness was measured with a 5 kg force held for 10 s. Each hardness value is an average of at least 20 points.

3. Results

3.1. Microstructural evolution in the fine-grained sample

When the fine-grained sample was subjected to a strain of 0.1, there is no obvious difference in the optical microstructure

compared with that in Fig. 1(a). Further TEM examination shows that the dominant deformation mode is dislocation slip. The typical dislocation configuration is dislocation tangle, as shown in Fig. 2(b). The corresponding selected area electron diffraction pattern (SADP) inside the tangled structure shows a set of brilliant spots, which indicate that almost no misorientation occurs in this dislocation configuration. Fig. 2(c) shows another typical TEM image at this strain. High density of dislocations are gradually accumulated and rearranged to form dislocation cells. The cells are roughly equiaxed with an average diameter of 600 nm.

Fig. 3 shows TEM micrograph of the fine-grained sample subjected to a strain of 0.35. The dislocation density is dramatically reduced and cell boundaries become thin in comparison with that in Fig. 2(b). The diameter of dislocation cells slightly decreases to be about 500 nm.

When the sample was deformed to a strain of 0.69, strain contrast becomes strong and the size of dislocation cell further decreases to about 300 nm, as shown in Fig. 4(a). The corresponding SADP shows that the original isolated diffraction spots are spread into small diffraction arcs. This means that small misorientations between the adjacent dislocation cells are formed. High-magnified image at this strain shows that some grain embryos, as marked by arrows, nucleate in the high-strain area (Fig. 4(b)). Careful observation shows that these grain embryos seem to migrate along certain direction. In other words, they grew into the area of high-density dislocation. In addition, some moiré fringes are visible in some local zone, as marked by triangles in Fig. 4(b). They might be caused by the misorientation between neighboring crystalline domains [17].

As the strain increased to 1.2, high-magnification TEM image shows that grain contour with an obvious contrast becomes clearly visible within the severely deformed zone. Equiaxed grains with a size about 100 nm are formed, as labeled by the numbers (1, 2, 3, ..., 7) in Fig. 5. Less dislocation was observed in the grain interior. In contrast, dislocation density is high in the vicinity of grain boundary. In other words, the elimination of dislocation takes place in local area even though there is still high dislocation density in the surrounding area. This indicates that dynamic recrystallization (DRX) has occurred in the highly deformed area.

When the fine-grained sample was deformed to a large amount of strain, i.e., 1.61, well-developed nanograins are produced in some local area (Fig. 6). The corresponding SADP consists of discontinuous rings. This implies the formation of high-angle grain boundaries with random crystallographic orientations inside the selected area. This nanocrystalline structure was identified as alpha single phase. The statistical distribution of grain size obtained from dark-field images is shown in Fig. 6(c). The grain size corresponds to the

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