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Microstructures and mechanical deformation behaviors of ultrafine-grained commercial pure (grade 3) Ti processed by two-step severe plastic deformation

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ABSTRACT

Ultrafine-grained (UFG) commercial pure (CP, grade 3) Ti was produced using two-step severe plastic deformation, eight passes equal channel angular extrusion (ECAE) and cold rolling (CR) at liquid nitrogen temperature (LNT). Microstructural evolution and mechanical behaviors of UFG CP-Ti have been systematically investigated. After eight passes ECAE, the grain size was refined to sub-micron scale, smaller than 0.5 μm. Subsequent CR at LNT or RT for both UFG and coarse-grained (CG) specimens led to further refinement of structure, dramatically intensifying (0 0 0 2) peak, and the preferred orientation along the (0 0 0 2) crystal plane is formed at the expense of other crystal plane. After eight passes ECAE and CR at LNT, the ultimate tensile strength of UFG CP-Ti (grade 3) is 1218 MPa, and an elongation of 12.6%. Strain hardening behaviors of UFG CP-Ti (grade 3) during tensile deformation at RT have been analyzed.

1. Introduction

Equal channel angular extrusion (ECAE) has been successfully used to produce ultrafine-grained (UFG) pure titanium [1–3]. Microstructure refinement and formation of UFG structures in pure titanium result in considerable enhancement in mechanical properties, such as tensile strength limit [4], compressive strength limit [5], and fatigue strength [6]. Stolyarov et al. [7] found that the ultimate tensile strength (UTS) of commercial pure titanium (CP-Ti) was improved to 710 MPa after eight passes ECAE treatment, and the elongation to failure was 14%. But, the UTS is still lower than that of titanium alloys such as Ti–6Al–4V [8]. Combining ECAE with cold extrusion, at a strain of 75%, the UTS of CP-Ti was increased to 1050 MPa, with an elongation of 8% preserved [9]. Stolyarov et al. [10] applied two-step SPD (ECAE followed by cold rolling at room temperature) to CP-Ti, resulting in an UFG CP-Ti with UTS of 1050 MPa and an elongation of 6%. Wang et al. [11] investigated the room temperature (RT) and liquid nitrogen temperature (LNT) tensile deformation behaviors of UFG CP-Ti (processed by two-step SPD, eight passes ECAE plus CR at RT with a strain of 73%). It was reported that at RT, the UTS of UFG CP-Ti was ∼1000 MPa with an elongation of 13.5%, but at LNT, the UTS of CP-Ti was drastically elevated to ∼1400 MPa, and rather than a reduction in ductility, the elongation was increased to 16% [11].

So far, the room temperature tensile mechanical behavior of UFG CP-Ti after two-step SPD, ECAE treatment and CR at LNT, has not been reported. In this investigation, we combined ECAE and CR at LNT to further improve the room temperature UTS of CP-Ti. In the present experiments, after CR at LNT, the room temperature UTS of UFG CP-Ti (grade 3) was remarkably elevated to 1218 MPa, which is higher than that of Ti–6Al–4V [8], and surprisingly, the larger magnitude improvement in strength did not result in the significant reduction of elongation. The relationships between the UFG microstructure after two-step SPD and the tensile deformation behaviors have been investigated.

Very little strain hardening is a typical mechanical behavior for as processed UFG CP-Ti (grades 1 and 2), which results in low ductility as suggested in Refs [12,13]. Annealing at 300 °C or deforming at 77 K restored some strain hardening, delayed the necking and promoted the ductility of UFG CP-Ti (grades 1 and 2) [11,12]. Although the mechanism of little strain hardening for UFG metals is still controversial [8–10,13], the effective way to improve the ductility of UFG metals is to increase the strain hardening and delay the necking [14,15]. Strain hardening and necking behaviors of UFG CP-Ti (grade 3) during tensile deformation have been analyzed in the present paper.

2. Experimental procedure

The commercial pure (grade 3) Ti rods were annealed at 1023 K for 1 h, cooled with furnace, before ECAE. Billets for ECAE treatment with a dimension of 10 mm × 10 mm × 120 mm were cut from the annealed CP-Ti rods. ECAE die was designed to yield an effective

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strain of ∼1 by single pass, with an inner contact angle (Φ) and outer contact angle (Ψ) of 90°, as shown in reference [16]. ECAE processes were performed at 390–400 °C by Bc path (the billets were rotated 90° along clockwise direction, between adjacent passages), at a pressing speed of 8 mm/s. Samples for CR were cut from billets after eight passes ECAE, with a dimension of 2.8 mm × 5 mm × 25 mm, and cold-rolled at RT and at LNT. The direction of CR was consistent with the extruding direction during ECAE. The roller was 130 mm in diameter, with a rotation speed of 33 revolutions/min. During CR at LNT, samples were wrapped with stainless steel sheet, cooled in liquid nitrogen for 5 min before the next pass, and then rolled at a reduction of 0.12 mm in thickness per pass.

Samples for microstructure observation were cut from the longitudinal plane of billets parallel to the extruding direction of ECAE and CR. Specimens for optical microscopy examination were prepared by metallographic polishing and etched by a mixture of hydrofluoric acid, nitric acid and distilled water with a volume ratio of 1:4:20, then investigated with a LeICA-MEF4M optical microscope. TEM and HRTEM studies were carried out with a JEOL-JEM-2010 operating at 200 kV and a JEOL-JEM100CXII Electron Microscope with an accelerating voltage of 100 kV. The specimens for TEM and HRTEM were mechanically thinned to ∼50 μm, followed by a twin jet polishing technique using a solution of 6% perchloric acid, 35% butanol and 59% methanol at an applied potential of 30 V and at −40 °C. XRD patterns were acquired on Ricaku D/max 2550V equipped with a Cu Kα anode (wavelength λ = 1.54 A).

Tensile tests were performed at room temperature (RT) and at a constant strain rate of $1 \times 10^{-3}\,s^{-1}$ using a testing machine Zwick/Roll Z020. Samples with a dimension of 0.8 mm × 2 mm × 50 mm were machined along the direction parallel to the direction of ECAE and CR with a gauge length of 10 mm. The deformation displacement was measured by a strain-extensometer with a measuring error of 0.001 mm.

3. Results and discussion

3.1. Microstructures

Microstructures of CP-Ti (grade 3) annealed and processed by ECAE are shown in Fig. 1. Grains of annealed samples are coarse and not homogeneous, 40–120 μm in size Fig. 1(a). After one pass ECAE, the initial coarse grains were broken and lots of deformation twins were formed (Fig. 1(b)). The width of these twin bands was different, changing within 0.1–0.5 μm. The SAED in Fig. 1(c) was obtained from the area C in Fig. 1(b). The twin plane is determined as $\{1\,0\,\bar{1}\,1\}$, which is in consistence with the result of Refs [17,18]. The plastic deformation of fcc and bcc structured metals is mainly accommodated by the dislocation movement [19,20]. However, the hcp structured Ti has fewer slip systems, therefore the twinning takes predominant role in plastic deformation during first pass ECAE process [17,18,21].

As shown in Fig. 1(d), after two passes ECAE, the microstructure is mainly composed of remnant elongated grains and local dislocation cell structure, no deformation twins observed. During the second pass ECAE process, the dislocation movement, such as slip, climb or cross-slip, becomes prime plastic deformation mode instead of twin shear since dislocation movement needs lower critical shearing stress than twin shear and refined grains provide more slip systems in co-operation, which is in consistence with the results of Refs. [17,21–23].

After four passes ECAE treatment, these elongated grains are partially replaced by finer equiaxed grains, smaller than 0.5 μm in size, some with large-angle grains boundaries, as shown in Fig. 1(e). There is lower dislocation density in the interior of finer equiaxed grains. Lots of dislocations are accumulated and tangled with others around grains boundaries. After eight passes ECAE treatment, microstructure becomes more homogeneous and the fraction of fine equiaxed grains increases, but the size of the fine equiaxed grains changes little, as seen in Fig. 1(f).

Microstructures of CP-Ti (grade 3) processed by ECAE and cold rolling (CR) at RT and LNT are shown in Fig. 2. After CR at RT with an accumulative strain of 76%, the UFG structure produced by ECAE processes was further broken, and no clear grains and boundaries could be observed under TEM, as shown in Fig. 2(a). The microstructure is mainly composed of finer dislocation cell structure. The corresponding SAED pattern in Fig. 2(a) proves that microstructures are further refined after CR treatment. Stolyarov et al. [9,10] have reported that many finer elongated grains are formed, about 100 nm in width, after eight passes ECAE and CR at RT with an accumulative strain of 75%. Close observation of Fig. 2(a), small white and black longitudinal grains were identified, but the contrast was not so sharp.

After cold rolling at LNT with an accumulative strain of 74%, the microstructure was also composed of dislocation cell structure, no clear grains boundaries could be identified, as shown in Fig. 2(b). The corresponding SAED in the right upper corner of Fig. 2(b) also suggested that microstructure was further refined. No microtwins were observed. The SAED pattern and high resolution image of area C in Fig. 2(b) are shown in Fig. 2(c) and (d), respectively. Two-dimensional HRTEM image in Fig. 2(e) was obtained by Fourier and inverse Fourier transformation of Fig. 2(d) with the spots of $(\bar{1}\,0\,1\,1)$ and $(\bar{1}\,1\,0\,\bar{1})$. Many edge dislocations can be seen in Fig. 2(e).

3.2. XRD analysis

XRD patterns of specimens annealed, after ECAE and CR are shown in Fig. 3. The integral intensity ratios of each peak relative to the strongest peak calculated from the XRD patterns are shown in Table 1. The data of the standard pure Ti power sample from Ref. [24] is also shown in Fig. 3(a) and Table 1.

Examination of Fig. 3(b) and Table 1 shows that the intensity of coarse-grained (CG) CP-Ti (annealed) are basically in accordance with that of the standard pure Ti power sample (Fig. 3(a)), except that $(0\,0\,0\,2)$ and $(1\,0\,\bar{1}\,3)$ peaks become stronger, and $(1\,0\,\bar{1}\,0)$ and $(1\,1\,\bar{2}\,0)$ peaks are weaker, which reveals that there is a little grain preferring orientation remained after annealing.

After one pass ECAE (Fig. 3(c) and Table 1), the integral intensities of diffraction peaks $(1\,0\,\bar{1}\,0)$ and $(1\,1\,\bar{2}\,0)$ are intensified obviously, meanwhile the integral intensities of peaks $(0\,0\,0\,2)$ and $(1\,0\,\bar{1}\,3)$ decrease, comparing with the annealed CG CP-Ti sample, which reveals that $(0\,0\,0\,2)$ and $(1\,0\,\bar{1}\,3)$ preferring orientation reduced and instead by $(1\,0\,\bar{1}\,0)$ and $(1\,1\,\bar{2}\,0)$ preferring orientation. This result is reasonably related to the microstructural changes, i.e. twins formed during first ECAE process, as shown in Fig. 1(b) and (c).

With the increase of ECAE passes, the grain size is gradually refined, as shown in Fig. 1, and width of diffraction peak in XRD pattern is broadened, as shown in Fig. 3. When the extrusion pass number reaches to eight, the width of diffraction peak in XRD pattern is broadened. The result is due to the grain refinement and the lattice distortions induced by the severe plastic deformation [25].

Comparing the XRD pattern and integral intensity ratios of CP-Ti after eight passes ECAE with those of standard power and initially annealed CP-Ti, it is found that both peak intensity ratio (Fig. 3) and integral intensity ratios (Table 1) of CP-Ti after eight passes ECAE are basically accordant with those of standard power and initially annealed sample. It reveals that after eight passes ECAE, the preferring orientations of fine grains are reduced, which is identical to TEM observations on CP-Ti after eight passes ECAE, as shown in Fig. 1(f).

The subsequent CR treatment for eight passes ECAE processed samples at RT and LNT dramatically intensify $(0\,0\,0\,2)$ peak, as seen