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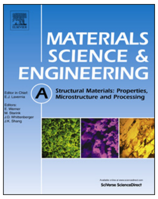

Hot deformation and grain refinement mechanisms of commercially pure titanium processed via three-directional cryo-compression

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ABSTRACT

Commercially pure titanium specimens were processed via three-directional compression at cryogenic temperature to activate homogeneous high-density deformation twins, and subsequent hot compression at 500 °C was applied to induce homogeneous grain refinement. Thus, homogenous and refined grains with an average grain size of 1.6 µm were achieved. During three-directional compression, four type of twins were activated, namely, parallel $\{11\bar{2}2\}$ twins, parallel $\{10\bar{1}2\}$ twins, intersectional $\{10\bar{1}2\}$ twins and parallel $\{11\bar{2}1\}$ twins. In the first pass, $\{10\bar{1}2\}$, $\{11\bar{2}1\}$ and $\{11\bar{2}2\}$ twins were activated; in the second pass, $\{10\bar{1}2\}$ and $\{11\bar{2}2\}$ twins were activated; and in the third pass, $\{10\bar{1}2\}$ twins were activated. During hot compression, the dynamic recrystallization (DRX) process was divided into twin-active DRX stage and continuous DRX (CDRX) stage. In the first stage, the DRX mechanisms were twinning induced CDRX (TCDRX) and twinning induce DDRX (TDDRX) in parallel $\{11\bar{2}2\}$ twins, TCDRX and TDDRX in parallel $\{10\bar{1}2\}$ twins, TCDRX and intersectional twins induced DRX (ITDRX) in intersectional $\{10\bar{1}2\}$ twins and TCDRX in parallel $\{11\bar{2}1\}$ twins. Nine types of new texture components formed in this stage. In the last stage, the DRX mechanism was CDRX. The $< a >$ basal slip, $< a >$ pyramidal slip and 1st $< c + a >$ pyramidal slip were the major deformation mechanisms during hot deformation.

1. Introduction

Commercially pure titanium (CP-Ti) is a promising candidate as components in medical equipment, chemical devices and heat-exchangers [1–4] due to its extraordinary properties, such as excellent biocompatibility, superior corrosion resistant performance and high heat resistance [5–8]. As a typical hexagonal close packing metallic material, CP-Ti mainly deforms by dislocation slip [9] and twinning [10]. The major slip systems for CP-Ti are $< a >$ prismatic slip and $< a >$ basal slip [3] which cannot accommodate $< c >$ axis plastic deformation [11]. Therefore, twinning plays an important role in deformation by accommodating $< c >$ axis deformation, especially at high strain rate [11] and/or cryogenic deformation conditions [12]. The frequently observed twins in CP-Ti are $\{11\bar{2}2\} < \bar{1}\bar{1}23>$ compressive twins and $\{10\bar{1}1\} < 10\bar{1}2>$ compressive twins, which are activated under compression parallel to $< c >$ axis or tension perpendicular to $< c >$ axis [13,14], and $\{10\bar{1}2\} < \bar{1}011>$ tensile twins and $\{11\bar{2}1\} < \bar{1}\bar{1}26>$ tensile twins, which are activated under tension parallel to $< c >$ axis or compression perpendicular to $< c >$ axis [13,14].

Apart from coordinating deformation, twins can promote recrystallization and grain refinement during deformation or annealing [15–18]. Levinson et al. [15] investigated the effect of twinning on annealing of Mg–3Al–1Zn alloy and found that compressive twins were preferred sites for recrystallization, whereas tension twins had little effect on static recrystallization (SRX). Molodov et al. [16] studied the deformation of magnesium single crystal and related dynamic recrystallization (DRX) mechanism. The results showed that DRX-ed grains, identified in $\{10\bar{1}1\}$ twins, were rotated about $< c >$ axis by a degree of 30°. Dogan et al. [17] performed equal channel angular processing experiment on Mg-3Al-1Zn alloy with two different textures at elevated temperature. It was concluded that $\{10\bar{1}1\}$ and $\{10\bar{1}2\}$ twins could promote DRX, which led to deformation localization and the formation of shear bands. As is proved by numerous researchers, grain refinement is an effective method for improving the mechanical properties of metallic materials [19–21]. However, for CP-Ti, few researchers reported the method of achieving fine and homogeneous microstructure via utilizing twinning as recrystallization sites. In fact, only Ahn et al. [22] performed channel die compression experiment to activate deformation twins and annealing experiment to induce SRX for achieving homogeneous grains with grain size of ~ 2 µm. Additionally, the deformation and grain refinement mechanisms have not been revealed in detail.

Several key factors may be considered to achieve fine and homogeneous grains. Firstly, temperature for activating twinning should be

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set as cryogenic temperature rather than ambient temperature, so that higher density twins may be activated [23]. Secondly, as twinning is a polar deformation mechanism, for metallic materials without intensified texture, being subjected to uniaxial deformation may not activate twinning in all grains, and thus result in inhomogeneous DRX-ed grains during later hot deformation [24,25]. In contrast, multi-directional compression at cryogenic temperature may activate relative homogeneous twins. Thirdly, hot deformation rather than static annealing should be utilized for grain refinement because DRX is a coupled process of deformation and recrystallization, which may lead to finer grains than SRX.

In this paper, a novel grain refinement method was proposed. CP-Ti with equiaxial grains was processed via three-directional compression at cryogenic temperature to activate high-density relatively homogeneous deformation twins, and subsequent hot deformation was applied to induce DRX. The fine and homogeneous grains were achieved by the DRX process. Discussion on twinning activated by three-directional compression, grain refinement mechanisms and hot deformation mechanisms may enrich the theory on deformation and recrystallization, and provide practical guidance for plastic deformation technology.

2. Material and methods

A Grade 2 CP-Ti rolled plate with a thickness of 30 mm was used in the experiment. The nominal chemical composition was Fe 0.20, O 0.06, C 0.04 and balance Ti (in wt%). The initial microstructure is presented in Fig. 1 , which is derived from the same sample as our previous results [25]. Fig. 1(a) shows a homogeneous equiaxial microstructure, and Fig. 1(c) illustrates the average value of grain size is 40.4 µm. The $\{0001\}$, $\{10\bar{1}0\}$ and $\{11\bar{2}0\}$ Pole Figures (PF) (Fig. 1(d)) of the plate depict a typical rolling texture. The $\{0001\}$ pole intensity aggregated at ~ ± 35° from normal direction (ND) to transverse direction (TD), and $\{10\bar{1}0\}$ $\{11\bar{2}0\}$ pole intensity was dispersedly distributed. The texture can be expressed by Euler angle of {0°, 15°, 30°}.

The experiment procedure is detailed in Fig. 2. The cuboid samples (8(RD) × 8(TD) × 12(ND) (mm)) (samples S_C) were cut from the initial plate by an electric discharge machine. The ND of the samples was parallel to that of the initial plate. The samples S_C were firstly submerged into liquid nitrogen for 20 min so that a uniform cryogenic temperature of −196 °C was achieved, and then were compressed to a strain of 0.13 at a strain rate of $0.01\ s^{-1}$ on CMT 5205 tester along the ND, RD and TD successively (samples were named Samples S_{C1}, S_{C2} and S_{C3} respectively). Afterwards, the samples S_{C3} were compressed to a series of strains (0.2, 0.5 and 1) at a strain rate of $0.01\ s^{-1}$ at 500 °C, and the corresponding samples were named S_{H1}, S_{H2} and $S_{H3,}$ respectively. The heating rate was 3 °C/s, and the holding time for temperature uniformity was 1 min. After hot compression, the samples were cooled in the air.

The microstructure was examined in the inner RD–TD plane by optical microscope (OM, Zeiss Axio 5 m) and electron back-scattered diffraction (EBSD, FEI Quanta 450). The EBSD files were processed by HKL Channel 5 software package. The samples for microstructural observation were electropolished in a unique solution of 12 mL perchloric acid, 68 mL butanol, and 120 mL methanol with a current of 1 A at −35 °C for 50 s.

3. Results and discussion

3.1. Twinning activated by three-directional compression

The microstructure of CP-Ti subjected to compression at cryogenic temperature is shown in Fig. 3. High-density twins were activated during cryo-deformation and the width of some twin lamellas was relatively thin (< ~ 1 µm) (Fig. 3(a)–(b)). As clarified by researchers, twinning and slip are the two major deformation mechanisms of titanium [12,26,27]. The critical resolved shear stress (CRSS) of slip increases significantly with decreasing temperature, whereas the CRSS of twinning is nearly constant with temperature variation in metallic materials [12]. In our study, decreasing the deformation temperature to cryogenic temperature arbitrarily led to higher CRSS of slip relative to that of twinning. Therefore, high-density twins were activated during deformation. Moreover, the microstructure of CP-Ti subjected to one-

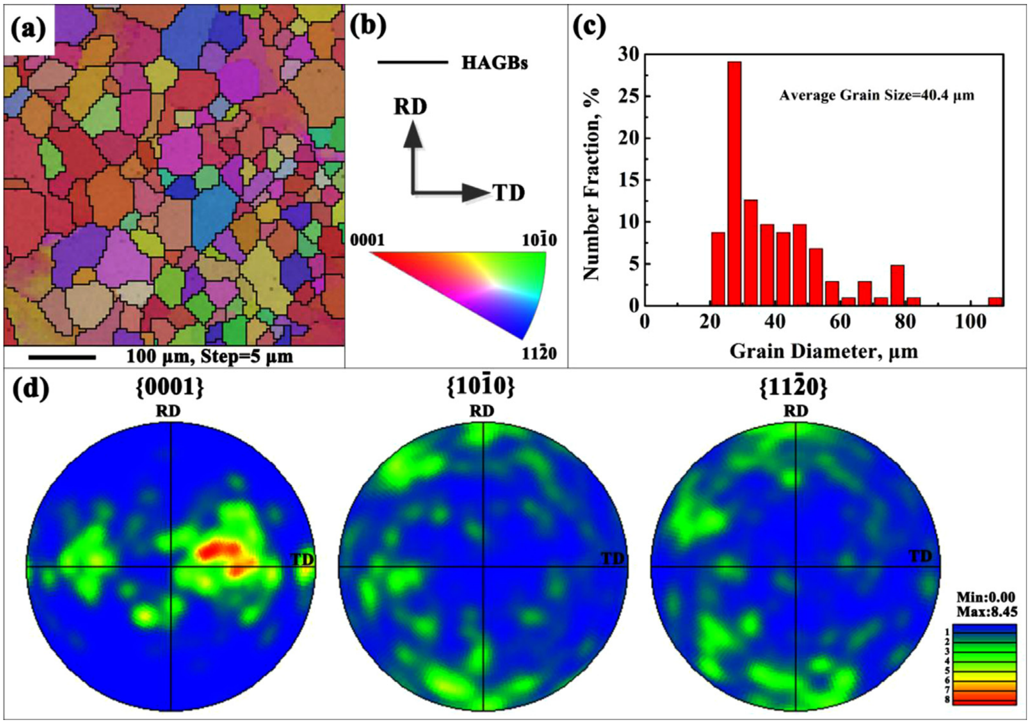

Fig. 1. EBSD maps of the initial microstructure: (a) inverse pole figure (IPF) plus band contrast (BC) map with high angle grain boundaries (HAGB) larger than 15°; (b) IPF legend; (c) grain size distribution; (d) (0001),$(10\bar{1}0)$and$(11\bar{2}0)$ pole figures (PF).