Static recrystallization of pure titanium after cryo-deformation

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Abstract. Commercially-pure (CP) titanium was first processed via cryogenic deformation to activate high-density deformation twins, and was subsequently annealed at 500°C to induce static recrystallization (SRX). Two types of twins were mainly activated during cryogenic deformation, i.e., \{11\bar{2}2\} contraction twins and \{10\bar{1}2\} extension twins. Multiple point-like twins were also present because the growth of twins was remarkably impeded at the low temperature. During annealing the recrystallization mechanisms were identified to be \{11\bar{2}2\} and \{10\bar{1}2\} twinning induced SRX. The point-like twins could effectively promote the occurrence of SRX. The grains were significantly refined from ~40 to ~2.7 μm, while a few \{11\bar{2}2\} and \{10\bar{1}2\} TBs still retained with the same twinning angle/axis relationship during annealing.

1. Introduction

The Commercially pure titanium (CP-Ti) is a promising hexagonal close-packed (hcp) metals for medical devices and shipping equipment thanks to its excellent corrosion resistant performance [1, 2]. In addition to dislocation slip, deformation twinning is also a major deformation mechanism for hcp metals due to their limited slip systems [3]. The commonly-observed twinning systems in CP-Ti are \{10\bar{1}2\} and \{11\bar{2}1\} extension twinning, and \{1\bar{2}2\} contraction twinning [4, 5]. The extension twinning is activated under tension parallel to c axis or under compression perpendicular to c axis of the hcp unit cell, while the contraction twinning is activated under tension perpendicular to c axis or under compression parallel to c axis [4, 5].

Except for coordinating plastic deformation, twinning in CP-Ti can promote dynamic recrystallization during hot deformation [6, 7]. Yan et al. [6] applied pre-cryo-deformation to activate twinning and hot deformation to induce dynamic recrystallization (DRX). Three types of twins were active in inducing DRX. It was found that the DRX stage included twin-active stage and discontinuous...
DRX stage and the DRX mechanisms were twinning-induced DRX and discontinuous DRX. Yan et al. [7] also applied three-directional compression at cryogenic temperature to activate homogeneous deformation twins, followed by hot deformation to induce homogeneous fine grains. As a result, homogeneous and equiaxed grains with an average grain size of ~1.6 μm was achieved. The DRX mechanism also involved twinning-induced DRX stage and continuous DRX stage. However, till now, few researchers have focused on the effect of twinning activated at cryogenic temperature on static recrystallization (SRX).

Therefore, in this paper CP-Ti was firstly deformed at cryogenic temperature to activate twins, then was annealed at hot temperature to induce SRX. The study on twinning activated at cryogenic temperature and twinning-induced static recrystallization would enrich the theory on static recrystallization.

2. Experimental

A CP-Ti rolled plate with a thickness of 30 mm was utilized in our experiment. The cylindrical samples (Φ8×12) were machined from the as-received plate, where the axis of the cylinder was parallel to the normal direction (ND). The cylindrical samples were immersed in liquid nitrogen for 30 min, and then were compressed to a true strain of 0.30 at a strain rate of 0.01 s⁻¹ on CMT 5205 testing machine to activate twins. After cryo-deformation, the samples were annealed in a muffle furnace at a temperature of 500°C for a series of time of 3 min and 14 min. After annealing, the samples were cooled in the air.

The microstructure was examined on the RD-TD plane in the central section of the samples by optical microscopy (OM, Zeiss Axio 5.m) and scanning electron microscopy (SEM) equipped with electron backscatter diffraction (EBSD). The samples for OM and EBSD characterization were electro-polished in a solution consisting of perchloric acid, butanol and methanol (3:17:30 in volume ratio) with a voltage of 30 V at a temperature of -35°C. The EBSD data were analysed via HKL Channel 5 software package.

3. Results and discussion

3.1. Twinning activated at cryogenic temperature

The microstructure of the as-received CP-Ti plate, characterized via EBSD, is shown in Figure 1 [6]. A homogeneous and equiaxial microstructure without deformation twins can be seen Figure 1(a). As shown in Figure 1(b), the grain size of the equiaxial grains was ~40.7 μm. The {0001} pole figure revealed a typical split basal texture, where the peak pole intensity of c-axis was split by about ±(25-35°) from ND toward TD (Figure1(c)).

Figure 2 shows the microstructure of CP-Ti after deformation at cryogenic temperature to a strain amount of 0.30 at a strain rate of 0.01 s⁻¹. The low magnification (100×) optical micrograph shows that twins were activated in almost all grains, however, the distribution of twins was inhomogeneous (Figure 2(a)). The morphology of twins could be divided into parallel type, intersectional type, and densely-distributed type, which are denoted by arrows in Figure 2(a). The parallel type twins present a parallel morphology, while the intersectional type twins are inter-crossed each other. The morphology of some high-density twins was not clear in the low magnification observations. Figure 2(b) is high magnification (2500×) OM image, where the widths of most twins were seen to be smaller than 1 μm. The densely-distributed type twins (in Figure 2(b)) exhibit an accumulative morphology. The twin morphology was further characterized via EBSD method. The {1122} twin boundaries (64°<1010>) were colored green and the {1012} twin boundaries (85°<1120>) were colored red in our study. It is seen that the parallel type twins and
intersectional type twins were \{11\overline{2}2\} twins while the densely-distributed twins were \{11\overline{2}2\} and \{10\overline{1}2\} twins (Figure 2(c)). Figure 2(d) is the misorientation angle distribution, where three peaks fell in the ranges of 2-10°, 60-70° and 80-90°, respectively (The peak in the ranges of 2-10° reflects low angle grain boundaries). Inverse pole figures shown in Figure 2(e) were utilized to decide the rotation axis corresponding to the peak in Figure 2(d). The peak fell in the ranges of 60-70° was around the \langle10\overline{1}0\rangle axis and the peaks fell in the ranges of 80-90° was around the \langle11\overline{2}0\rangle axis, which confirmed that \{11\overline{2}2\} and \{10\overline{1}2\} twins were activated, respectively. Typical grains named as A and B in Figure 2(c) are depicted in Figure 2(f) together with their corresponding pole figures for a more detailed analysis. It is seen that in grain A shown in Figure 2(f), multiple intersectional \{11\overline{2}2\} twins were activated, indicating the presence of \{11\overline{2}2\} twin variants. Three twin variants were activated in the pole figure of Figure 2(f). The c axis of grain A matrix was nearly parallel to ND, then the compressive direction was along c axis, leading to the activation of \{11\overline{2}2\} contraction twins. In grain B shown in Figure 2(f), two sets of parallel twins met so as to form new HAGBs, as revealed by the relevant pole figure, where two twin variants were seen to be activated. The reason for the activation of \{11\overline{2}2\} in grain B is the same as that for the activation of \{11\overline{2}2\} twins in grain A. In addition, multiple point-like \{11\overline{2}2\} twins and \{10\overline{1}2\} twins were activated in grain C in Figure 2(c). Twinning process is generally characterized by twin nucleation and growth, where twin nucleation is a process of formation of point-like nucleus [8]. During compression at cryogenic temperature, the growth of twins was remarkably impeded, thus numerous twin nuclei retained the point-like morphology.

3.2. Twinning activated at cryogenic temperature

Recrystallization mode of software Channel 5 was utilized to identify SRX grains. In the recrystallization mode, grains with an average misorientation of smaller than 3° is deformed microstructure, grains with an average misorientation of more than 15° is recrystallized microstructure, and grains with an average misorientation ranging from 3° to 15° is substructure, which means grains with crystal defects. IPF map and recrystallization map of the cryo-deformed CP-Ti after annealing at 500°C for 6 min are shown in Figure 3(a) and Figure 3(b), respectively. The blue-colored subset (with an average misorientation of more than 15°) in Figure 3(b) represents the static recrystallization grains. SRX mechanisms are commonly classified into continuous SRX (CSRX) and discontinuous SRX (DSRX) [9]. CSRX is an evolution process of transforming low angle grain boundaries (LAGBs) into high angle grain boundaries (HAGBs), while DSRX is a process of the nucleation of novel grains by bulging of HAGBs [9]. In addition, twinning-induced static recrystallization (TSRX) is regarded as a novel static recrystallization mechanism. In our study, the SRX grains frequently occurred in the interior of twins or twin boundaries, indicating that the SRX mechanism may be TSRX. During annealing, the
SRX grains resided on the TBs, and TBs evolved into HAGBs. This would confirm the TSRX mechanism, which will be discussed.

Figure 2 – Microstructure of CP-Ti after deformation at cryogenic temperature to a strain of 0.30 at a strain rate of 0.01 s⁻¹: (a)-(b) OM; (c) IPF map; (d) distribution of misorientation angles; (e) IPF; (f) local IPF maps.
Three typical zones named A, B and C are selected from Figure 3(a) for more detailed discussion. SRX grains in zone A evolved from grain A or B in Figure 2(f) since the SRX grains still had \{11\overline{2}2\} TBs. In fact, this was the initial stage of SRX since \{11\overline{2}2\} GBs had not evolved into ordinary GBs. The recrystallization mechanism was \{11\overline{2}2\} twinning induced SRX. By comparing grain C in Figure 2(c) and zone B in Figure 3(a), it is deduced that the SRX grains in Figure 3(a) were induced by the point-like twins in Figure 2(c) for the SRX grains mainly resided on the TBs of the point-like \{11\overline{2}2\} and \{10\overline{1}2\} twins. Hence, the SRX mechanism was \{11\overline{2}2\} and \{10\overline{1}2\} twinning induced SRX. Moreover, the recrystallization rate of zone B was significantly higher than other zones, suggesting that the high density point-like twins could predominantly promote the occurrence of SRX. The grain size in zone C was \sim 10\ \mu m, which was significantly smaller than the average grain size of the as-received CP-Ti, meaning that the incubation period of the grain was short and the grain was SRX grain in the stage of grain growth. The grain was surrounded by \{11\overline{2}2\} and \{10\overline{1}2\} TBs, therefore it could be inferred that the recrystallization mechanism was \{11\overline{2}2\} and \{10\overline{1}2\} twinning induced SRX.

**Figure 3 – Microstructure of cryo-deformed CP-Ti after annealing at 500°C for 6 min: (a) IPF map; (b) recrystallization map.**

IPF map and recrystallization map of the cryo-deformed CP-Ti after annealing at 500°C for 14 min are shown in Figure 4(a) and Figure 4(b), respectively. The grains were significantly refined in comparison with the as-received microstructure. However, a few \{11\overline{2}2\} and \{10\overline{1}2\} TBs still retained the same twinning angle/axis relation during annealing process. The average grain size was \sim 2.7\ \mu m, and the fraction of high angle grain boundaries was \sim 83\% (Figure 3(c-d)).
4. Conclusions

The microstructure evolution of CP-Ti during cryo-deformation and subsequent annealing at 500°C was characterized. The following conclusions can be drawn:

1. During cryogenic deformation, high-density deformation twins were activated, mainly \{11\overline{2}2\} contraction twins and \{10\overline{1}2\} extension twins. Multiple point-like twins were activated because the growth of twins was remarkably impeded at the low temperature.

2. During annealing, the recrystallization mechanisms were \{11\overline{2}2\} and \{10\overline{1}2\} twinning induced SRX. The point-like twins could effectively promote the occurrence of SRX.

3. The grains were significantly refined from ~40 to ~2.7 μm during annealing. A few \{11\overline{2}2\} and \{10\overline{1}2\} TBs still retained the same twinning angle/axis relation in the annealing process.

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