Study of microstructure and mechanical properties of pure titanium processed by new route of equal channel angular pressing

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Abstract. In this study, samples of pure titanium were processed by equal channel angular pressing (ECAP) up to four passes at 573 K to produce a refined microstructure under different deformation routes. A new route B₁₃₅ was developed by defining 135° clockwise rotation of samples in each pass. In order to study the deformed structures, optical micrograph (OM) and electron backscatter diffraction (EBSD) analysis were performed, and the mechanical property was evaluated by the microhardness tests. The OM and EBSD results reveal that the mean grain size of ECAPed samples is much finer than that of the initial sample. Furthermore, it also reveals that B₁₃₅ is much effective in terms of grain refinement. The microhardness test results show that ECAP process via both route B₁₃₅ and Bₖ causes a much increase in hardness.

1. Introduction

Equal channel angular pressing (ECAP) technique has been successfully used to produce sub-micro or even nano-grained bulk materials [1, 2]. ECAP die is consisted two channels that were intersected by the angles of $\Phi$ generally greater than or equal to 90°. During ECAP, a sample is first pressed into the entrance channel and then exited from the export channel after simple shear deformation [3, 4]. In this process, the cross-sectional dimensional values of samples are identical to the initial state between the consecutive pressing so that it is able to be extruded repetitively to accumulate large plastic strains [5]. The grain refinement and deformed structures are strongly dependent on the processing route. There are four recognized routes, which are defined by the rotation angle of samples around its longitudinal axis in each pass [6-9]: route A, not rotation; route $B_\lambda$, 90° clockwise and counterclockwise alternatively rotation; route $B_\lambda$, 90° clockwise rotation; route $C$, 180° rotation.

Many of the early studies on the effect of ECAP route on refining grain size and microstructures devoted to cube structure metals including Cu [10], Al [10, 11], Ni [4], iron [12]. More recently, there is an increasing interest in applying ECAP to process hard deformation materials including Ti, Mg and its alloys [13, 14, 15]. Especially, ECAP of pure Ti has attracted considerable interest because it has an extensive application prospect in the area of medicine and aerospace. However, there are only a few studies in refining grain size and microstructure of pure Ti that was processed with different routes.

In the present study, a new route $B_{135}$ was developed by defining 135° clockwise rotation of samples in each pass. Samples were processed by the two different routes, e.i. $B_{135}$ and $B_\lambda$, at 573K. The OM and EBSD analysis was made on the ECAPed samples with different routes to investigate the
change of the microstructure with passes number and the most effective route in grain refinement. Mechanical property of the original sample and ECAPed samples was studied by hardness measurements.

2. Experimental procedure

Pure titanium samples used for testing of ECAP was 70 mm in length by 15 mm in diameter. The average grain size of the original is about ~2 mm. Table 1 shows the chemical composition of the applied pure titanium material. ECAP pressing was performed at 573K using a split die made of H13 steel with channel angles $\Phi = 120^\circ$ and arc of curvature $\Psi = 25^\circ$. According to the two angles of $\Phi$ and $\Psi$, the imposed strain is estimated as $\sim$0.63 for a single pass [16]. All samples were pressed through a maximum pass of 4 under routes $B_{135}$ and $B_C$. The lubricant of MoS$_2$ based grease was used to reduce the friction between the sample surface and die channels.

Deformed microstructure was observed using optical micrograph (OM) and electron backscatter diffraction (EBSD) on a plane that is parallel to the pressing direction. Specimens for OM study were first prepared by mechanical polishing and then etched by the solution with 25% HNO$_3$, 20% HF and distilled water. OM images were obtained by utilizing a Leica DFC320. The specimens for EBSD observations were mechanical polishing and followed by electrolytically polishing with a solution of 20% HClO$_4$ and 80% CH$_3$OH at 17 V (15 s) and a temperature of 295 K. EBSD measurements were performed by utilizing a Zeiss Ultra Plus equipment with a NordlysNano EBSD detector from Oxford Instruments. The Vickers microhardness, HV, were measured using the equipment of KB3000BURZ-SA, and a load of 100 gf and loading time of 15 s were applied. The hardness was obtained from a mean value of the total of 12 points.

Table 1. Chemical composition of the titanium samples.

|   | Fe   | C    | N    | H    | O    | Ti   |
|---|------|------|------|------|------|------|
|   | 0.0014 | 0.0017 | 0.001 | 0.0002 | 0.014 | balance |

3. Results and discussion

The microstructures of pure titanium after ECAP processing under the both routes $B_C$ and $B_{135}$ exhibit a severely deformed, as shown in Fig. 1. The typical microstructure after 1 pass is shown in Fig. 1(a). It is clearly shown that the microstructures exhibit significant inhomogeneity with elongated coarse grains alternating with a large volume fraction of deformation twins. Deformation twins were the typical deformation microstructure in ECAPed titanium after one ECAP pass [14, 17]. This suggests that twinning is crucial in accommodation of plastic deformation at the initial stage during ECAP.

The microstructures after 2 passes through route $B_C$ and $B_{135}$ were showed in Fig. 1(b) and (c), respectively, the elongated coarse grains were broken up into smaller subgrains and a small number of deformation twins can still be observed. With the further increase of ECAP passes, the microstructure is further refined and there is no evidence that any deformation twins structure is still existing. It should be noted that the sample exhibits a more homogeneous microstructure through 4 ECAP passes under route $B_C$ when compared with that of through 4 ECAP passes under route $B_{135}$. 
For the deformed microstructure after 4 passes using route BC and B135, EBSD observations are carried out. The orientation maps after ECAP for 4 passes with different route are shown in Fig. 2. It is clearly shown that the microstructure after route BC processing is more homogeneous than that of after route B135 processing. The EBSD results are consist with the OM observation. After route B135 processing, the microstructure is composed by elongated grains and band structures that consisting of nanometer and submicrometer grains.

The grain size distribution histograms of ECAPed samples via route BC and route B135 are shown in Fig. 2(c) and (d) corresponding for the images of Fig. 2(a) and (b), respectively. The mean grain size through 4 passes is about 2.82 μm and 2.36 μm for route BC and B135, respectively. Therefore, it can conclude that route BC is less effective than B135 in the grain refinement.

Figure 1. Optical microstructures of pure titanium processed by ECAP using route BC after 1 pass (a), 2 passes (b), 3 passes (d), 4 passes (f) and using B135 after 2 passes (c), 3 passes (e), 4 passes (g).
Figure 2. The EBSD pattern of the titanium ECAP samples after 4 passes using two different routes (a) route BC; (b) route B135; (c) and (d) are the grain size distribution histograms corresponding for the images of (a) and (b), respectively.

The values of Vickers hardness of the sample processed under route BC and B135 are plotted against with the pass number, as shown in Fig. 3. Prior to ECAP process, the hardness value for original titanium sample is about 107 HV. After the first pass, the hardness is increased significantly and has a value of 154.8. With increasing number of passes, the hardness of samples processed by route BC and B135 underwent virtually the same development. For route BC, the hardness increase from 154.8 to 156.4 after two passes and eventually to 164.3 after four passes. For route B135, the hardness increase from 154.8 to 162.1 after two passes and eventually to 172.7 after four passes. The increased hardness of the ECAPed samples due to grain refinement can be interpreted in terms of the well-known Hall–Petch relation [18] as $H_v = H_0 + kd^{-1/2}$, where $H_v$ is the Vickers hardness, $d$ is the average grain size, $k$ and $H_0$ are material constants. It should be noted that the hardness of samples after one pass is slightly increased with the further increase of ECAP passes, but samples processed under route B135 has a higher hardness than that of under route BC in each pass. This may be attributed to that route B135 has a smaller grain size.
Figure 3. Variation of Vickers hardness of the sample processed via route $B_C$ and $B_{135}$ as a function of ECAP pass number.

4. Summary
In the present work, pure titanium was severely deformed by ECAP at 573 K. A new route was developed by defining $135^\circ$ clockwise rotation of samples in each pass, which was defined as $B_{135}$. Microstructures and hardness property of the deformed samples processed by both routes of $B_C$ and $B_{135}$ were studied in detail. The experiments results reveal that route $B_C$ is less effective than route $B_{135}$ in refinement of grain size. The hardness measurements suggest that the hardness is significantly increased after ECAP processing via both route, but sample processed via route $B_{135}$ has a higher hardness than the sample processed under route $B_C$ in each pass. The high hardness of samples processed via route $B_{135}$ can be caused by the fact that route $B_{135}$ has a smaller grain size.

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