Effect of extrusion routes on thermal stability of pure titanium processed by equal channel angular pressing

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Abstract. Thermal stability of samples deformed by equal channel angular pressing (ECAP) using route B_C and B_135 was studied. The new route B_135 was developed by defining 135° clockwise rotation of samples in each pass. After ECAP processing, samples were heat treated at different temperatures for a period of time. The effect of the two different routes on thermal stability of the ECAPed samples was investigated in detail. Electron backscatter diffraction was used to detect the processes of both recovery and recrystallization during thermal processing. Also, the effect of extrusion route on thermal stability of the deformed samples was further investigated by microhardness measurements.

1. Introduction

Equal-channel angular pressing (ECAP) has been used successfully to fabricate submicrostructured bulk material with advanced properties [1]. Briefly, sample was pressed through the die which have two equal cross section and intersected with angle Φ ranging generally from 90° and 120°. The deformation of samples is considered by simple shear under ideal conditions [2, 3]. Because the cross-sectional dimensions of samples were less changed, the samples can be pressed repetitively. Thus, a large plastic strain are easy to be produced in the samples [4]. It was suggested that a lower angle of die should be applied during ECAP because the strains introduced into the samples in each pass correlate with the inner angle, Φ [5]. According to the rotation scheme of samples around it longitudinal axis between consecutive passes, four basic routes of ECAP were defined [6,7]. As shown in Fig. 1, route A: without rotated; route B_C: rotated 90° in the same direction; route B_A: rotated 90° alternately; and route C: rotated 180° in each pass.
Figure 1. Scheme of four ECAP routes defined by rotating of samples around its longitudinal axis.

Thermal stability of the samples after ECAP processing has received lots of attention since the maximum temperature in practice application appears to be related to the deformed microstructure. It has been reported by several authors that a duplex structure after heat treatment was obtained [8, 9]. The duplex structure can be caused by the discontinuous recrystallization or the abnormal grain growth. It is well established that structures in the deformed samples were determined in thermal stability. Furthermore, microstructure characteristics obtained by ECAP are related to the specific extrusion routes [10, 12]. Thus, it suggests that the extrusion routes may have an important influence on the thermal stability. However, there was less reported in the effect of extrusion routes on thermal stability.

In this paper, the evolution of microstructure and hardness of pure titanium processed by different routes was investigated with respect to the effect of extrusion routes (route Bc and B135) on the thermal stability of the deformed samples. B135 as a new route was developed by defining 135° clockwise rotation of samples in each pass. The deformed and heat treated states were characterized by optical microscopy (OM), electron backscatter diffraction (EBSD) and microhardness measurements. The change of microstructure and hardness of samples after heat treatment was analyzed and compared with that of the ECAPed samples.

2. Experimental procedure

Pure titanium as experimental material was applied and has a purity of 99.99 wt.%. The original material has a mean grain size of ~2 mm. All experimental samples with dimensions of 70 mm in length x 15 mm in diameter were pressed up to 4 passes at 573 K via route Bc and B135. A 120° die made of H13 steel was used. For this die, the equivalent strains are about ~0.63 for a single pass, thereby giving a total equivalent strains of ~2.52 after 4 ECAP passes [13]. To ensure the success of extrusion, MoS2 based grease was used as the lubricant. After ECAP processing, samples were heat treated at either 773 K or 873 K for a period of 1 hour.

The observation of all ECAPed samples was carried on the transverse plane and by OM and EBSD. For OM observation, the etch was performed on the polished samples by a mixture solution of 25% HNO3, 20% HF and distilled water for 12s. Optical images were obtained by utilizing a Leica DFC320 digital camera. All specimens for EBSD observation were mechanical polishing and followed by electrolytically polishing by the solution of 20% HClO4 and 80% CH3OH at 17 V for 15 s at 295 K. High resolution EBSD was carried on Zeiss Ultra Plus with a NordlysNano EBSD detector from Oxford Instruments. The Vickers microhardness was measured with loading time of 15 s. Microhardness was measured on a KB3000BURZ-SA microhardness testing machine. The mean value of hardness was obtained from 12 points of each sample. A load of 100 gf and loading time of 15 s were applied.
3. Results and discussion

Fig. 2 shows the optical microstructures of the ECAPed samples and isochronal annealing at either 773 K or 873 K for a period of 1 hour. The deformed microstructures of the samples after 4 ECAP passes via route BC and B_135 are shown in Fig. 2(a) and (b), respectively. It is clearly shown that after ECAP processing the original coarse grains were significantly refined, but the sample exhibits a more homogeneous microstructure after 4 ECAP passes via route BC when compared with that of via route B_135. The microstructure presented in the samples through 4 ECAP passes via route B_135 exhibits a mixture of ultra-fine and elongated coarse grains, in contrast to the samples deformed via route BC, where a homogeneous fine-grained microstructures was formed. Fig. 2(c) and (d) show the typical microstructures of the material after isochronal annealing at 773 K via route BC and B_135, respectively. For samples processed via route BC, after isochronal annealing for 1 hour at temperatures of 773 K caused much change for the microstructure when compared to the deformed microstructure, and still some of the deformed structures can be observed. For samples processed via route B_135, annealed behavior is similar in the respect of the microstructures change. During annealing for 1 hour at temperatures of 873 K for both routes the grains continued to grow and the deformed structures has almost completely disappeared.

![Figure 2. Optical microstructures of ECAPed samples through 4 passes via route BC (a) and route B_135 (b); microstructure of the samples via route Bc after isochronal annealing at 773 K (c), 873 K (e) and via route B_135 after isochronal annealing at 773 K (d), 873 K (f).](image)

Examples of EBSD micrographs are shown in Fig. 3 through 4 passes and isochronal annealing at either 773 K or 873 K for a period of 1 hour. It is clearly shown that the microstructure through 4 passes of ECAP processing via route BC is more homogeneous than that of processed via route B_135. The EBSD results are consist with the OM observation. The isochronal annealing result in the evolution of similar microstructures in ECAPed samples, which can be identified by the presentation of new coarse grains. In the course of annealing the proportion of these larger grains increases with increasing of annealing temperature, so that isochronal annealing is the formation of new grains by expending the deformed microstructure at this temperature.

Inferential statistics of EBSD data confirm that the average grain size after 4 passes is ~2.82 μm and 2.36 μm for route BC and B135, respectively. For the samples after 4 passes via route BC, the
average grain size increase to 3.13 μm after isochronal annealing at 773 K, and that is further increase to 23.61 μm after annealing at 873 K. For the samples after 4 passes via route B135, a similar change of the average grain size was observed during annealing and the mean grain size is 4.25 μm and 17.62 μm during annealing at 773 K and 873 K, respectively.

The above analysis reveal that route BC is faster than route B135 grain growth during isochronal annealing. This indicates that the pure titanium processed by route B135 has excellent thermal stability, as compared with that of processed by BC. The thermal stability is strongly affected by the fraction of defect density introduced during ECAP processing [14]. It has been reported that the materials processed by ECAP are usually thermally-unstable if it contains a higher density of defects. We therefore conclude that route BC appears to be more conducive than route B135 to introduce defects into the samples during ECAP processing, which leads to its low thermal stability.

Figure 3. EBSD images of ECAPed samples through 4 passes via route BC (a) and route B135 (b); microstructure of the samples via route Bc after isochronal annealing at 773 K (c), 873 K (e) and via route B135 after isochronal annealing at 773 K (d), 873 K (f).

The mechanical properties of those ECAPed samples after isochronal annealing are important since it determines the application temperature in practice. Sections of the pure titanium samples processed by both route BC and B135 were annealed with different temperatures for 1 hour, and the values of microhardness were recorded by measuring on the annealed transverse sections. Fig. 4 shows the relationship of microhardness with the annealing temperature. It can be seen that the hardness of the samples processed via route B135 has a value of 175.0 HV that is higher than that of processed by route BC (164.3). This can be caused by that route B135 has a small grain size. During isothermal annealing the samples processed via route BC and B135 after 4 passes have a similar trend for microhardness development. For route BC, a comparison of annealing material with ECAP deformed samples reveals that during annealing at 773 K the hardness is significantly decreased from 164.3 to 135.4 and eventually to 119.5 after annealing at 973 K. For route B135, the hardness is significantly decreased from 175.0 to 130.3 during annealing at 773 K and eventually to 122.7 after annealing at 973 K. Analysis of the microstructure of the annealed samples reveal that the decreased microhardness is mainly caused by the normal grain growth of the samples during isothermal annealing.
Figure 4. Variation of Vickers hardness of the sample processed via route BC and B_{135} as a function of extrusion temperature.

4. Summary

Pure titanium has been processed by ECAP using either route BC or B_{135} with a 120° die at 573 K. After ECAP process, the isochronal annealing of the deformed samples was performed at the temperature range of 773 – 973 K. The effect of extrusion routes on the thermal stability of the samples was studied. OM and EBSD results reveal that route BC is faster than route B_{135} in grains growth during isochronal annealing. This indicates that pure titanium processed by route B_{135} has excellent thermal stability, as compared with that of processed by BC. Furthermore, microhardness measurements of the annealed samples show that a significant decrease in microhardness was observed in both routes BC and B_{135} when annealed at temperatures above 773 K.

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