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Microstructure and anisotropy of the mechanical properties in commercially pure titanium after equal channel angular pressing with back pressure at room temperature

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ABSTRACT

In this work, we report on the anisotropy of the mechanical properties and the results of in-depth microstructural analysis of commercially pure (CP) grade 2 titanium after severe plastic deformation. CP-Ti was successfully processed at room temperature via four consecutive passes of equal channel angular pressing (ECAP) with very high back pressure (BP). An ECAP-BP die with circular channel cross-section, channel angle $\varphi = 90°$ and arc curvature angle $\psi = 0°$ was used. A sub-microcrystalline structure with a grain size of ~150 nm exhibits promising mechanical properties, as determined by hardness measurements and tensile and compression tests in different directions. We observed a significant mechanical anisotropy related to the strong texture. Considering the ID, ED and TD to be the insert, extrusion and transverse directions of the ECAP die, respectively, the highest compression strength was attained for samples with the major axis in the ID and in a direction inclined 22.5° from the ID toward the TD (σ_{max} ~ 1150 MPa). In contrast, the lowest strength was observed in the ED and at 45° from the ID toward the ED (σ_{max} ~ 940 MPa). Although a fracture occurred during compression of the samples tested along the ID, compression along the ED exhibited perfect plasticity with balanced hardening and softening mechanisms. Transmission electron microscopy (TEM) examination after ECAP-BP revealed a small amount of high-pressure hexagonal ω-phase. The occurrence of this phase was induced by a combination of severe plastic deformation and high pressure.

1. Introduction

Commercially pure (CP) titanium is used in highly corrosive environments and in a variety of biomedical applications [4,15,22]. However, deficiencies in the mechanical strength of CP-Ti represent one of the primary obstacles to its widespread usability and often lead to its replacement with other Ti alloys. Ti alloys do not suffer from insufficient strength due to second-phase and/or solid solution strengthening, but commonly used alloying (notably Al and V) is often controversial in other aspects, such as biocompatibility [4]. The mechanical properties of CP-Ti can be enhanced to the same level by grain refinement, which is usually performed via severe plastic deformation (SPD).

Among SPD methods, equal channel angular pressing (ECAP) is especially viable because it can be scaled up fairly easily to produce reasonably large billets and demonstrates very effective grain refinement with negligible contamination during processing [19]. Four standard sequences of strain path changes have been developed since the conception of ECAP, referred to as routes A, Ba, Bc, and C [19]. The four processing routes differ by the rotation around the longitudinal axis of the sample applied after each pass: 0° in route A, ±90° in route Ba, 90° in route Bc, and 180° in route C. Most of the investigations linking ECAP and CP-Ti were performed over a temperature range from 250 to 450 °C [5]. Although increasing the processing temperature usually promotes undesirable grain growth, its necessity is a consequence of three factors: (i) hexagonal close packed (hcp) structure classifies Ti as a difficult-to-work material prone to segmentation at ambient temperatures, (ii) in contrast with other materials unusually high friction between Ti and the walls of the die makes any extrusion process hardly feasible, and (iii) the very high strength of sub-microcrystalline CP-Ti, which can reach > 1 GPa. These facts make the processing of Ti at ambient temperatures technically difficult.

The use of room-temperature ECAP processing is justified by enhanced grain refinement due to suppressed grain growth. However, an excessively low temperature, such as that for cryogenic ECAP processing of Ti, may exhibit suppression of

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recrystallization and consequently does not lead to the desired grain refinement [12]. In addition to several studies dealing with room temperature ECAP of Ti in dies with an inter-channel angle of $\varphi \geq 105°$, which limits the imposed strain during each pass to ensure easier processing, the first successful attempts for multi-pass ECAP of Ti with $\varphi = 90°$ appeared very recently [12,24]. Zhao et al. successfully pressed CP-Ti in four passes via the C route, but used grade 1 Ti, which is easier to process due to the lower content of impurities than is present in grade 2 [24]. In contrast, Podolskiy et al. used grade 2 Ti, but the maximum number of passes used was three [12].

In our work, we propose the SPD processing of grade 2 Ti via a 90° die at room temperature by incorporation of back pressure (BP) during ECAP. Several authors have reported that BP improves the microstructure homogeneity, decreases the grain (cell) size, suppresses the undesirable shear localization and prevents cracking [6,7,9,21]. However, BP imposes further demands on die endurance because it increases the overall loading of the experimental rig. Following these results, we studied the formability of grade 2 Ti via ECAP with very high BP at room temperature (RT) and analyzed the anisotropy of its mechanical properties. The anisotropy plays a crucial role in critical load-bearing applications, such as dental implants, for which ultra-fine grained CP-Ti is preferred [15]. In addition to anisotropy of the mechanical properties, the aim of this work is to analyze the severely deformed microstructure using a transmission electron microscope together with X-ray diffraction.

2. Experimental section

2.1. Material

Commercially pure titanium rods (grade 2) 10 mm in diameter were purchased from Xian Aerospace New Materials Co., Ltd. The chemical composition, listed in Table 1, was verified by glow-discharge optical emission spectroscopy (GDOES) using a Spectruma GDA 750 HR apparatus. The microstructure consisted of equiaxed hcp α-Ti grains with an estimated grain size of ~12 μm (Fig. 1a).

2.2. Processing

An ECAP die with an inter-channel angle of $\varphi = 90°$ and corner angle of $\Psi = 0°$ was used, which leads to an imposed strain of ~1.15 per pass (Fig. 1b). Due to the extreme pressures during processing, we used a die with a circular channel cross section and a diameter of 11 mm to eliminate the stress concentrations at the edges of the square channel cross section. To reduce the frictional effects, a CP-Ti sample with a length of ~52 mm was enclosed in a phosphated thin steel container made from DIN St 37.0 with a wall thickness of 0.3 mm, and a composite lubricant containing approximately 50% graphite powder and 50% MoS_2 grease was used between the container and the die walls. The experiment was conducted at room temperature at a ram speed of 0.12 mm s^{-1} with the applied BP following route A for up to 4 passes. The BP was regulated between 270 and 590 MPa for all the passes to maintain an average plunger load of ~2.8 GPa. The BP value determined the loading limit of the tungsten carbide used as the plunger.

2.3. Microstructure characterization

Microstructural analysis was performed using a FEI TF-20 X-Twin transmission electron microscope (TEM). Texture and phase measurements were conducted using an X-ray diffraction (XRD) PANalytical X'Pert PRO diffractometer equipped with a Co-Kα radiation source ($\lambda = 1.789$ Å). Complete orientation distributions and recalculated full pole figures were determined. Samples for TEM and XRD were cut from the center of the ECAP billet along the ID–TD and ID–ED planes (see Fig. 1b) to verify the microstructural homogeneity. Samples for XRD were mechanically polished to mirror-like finishes using SiC paper, followed by mechanical–chemical polishing with colloidal silica. The thin surface layer, affected by the mechanical preparation, was removed via etching with Kroll's reagent. The samples for TEM observation were cut by wire electro-discharge machining (EDM) into disks with a diameter of 3 mm and thickness of 300 μm. The disks were consequently thinned by grinding to 100 μm and finished by electrolytic polishing using Struers Tenupol 5 with an applied voltage of 30 V at −45 °C. The electrolytic solution was 8 vol% perchloric acid, 7 vol% butanol, and 85 vol% methanol.

Table 1
Composition of the CP-Ti used in this study. The nominal composition is in accordance with ASTM standards, and the proclaimed composition is specified by the producer.

Composition in wt%	Fe	C	N	O	H	Ti
Nominal	≤ 0.30	≤ 0.08	≤ 0.03	≤ 0.25	≤ 0.015	Remainder
Proclaimed	0.19	0.01	0.01	0.15	0.001	Remainder
As measured	0.165	0.009	< 0.001	0.049	< 0.001	Remainder

2.4. Mechanical properties

The mechanical properties were characterized by uniaxial tension and compression on an Instron 5882 testing machine. Flat tensile specimens with dimensions of $4 \times 1.5 \times 10$ (width × thickness × length) mm^3 and cylindrical compression specimens with dimensions of Ø3 mm × 6 mm were deformed at room temperature (RT) and at a strain rate of 1.1×10^{-3} s^{-1}. At least two measurements were performed for each orientation to verify repeatability. Tensile testing was performed only for the ED oriented specimens. The nomenclature used for the description of the orientation between the compression axes and the coordinates of the ECAP-BP billet is shown in Fig. 1c. Provided that compression testing of a sample did not fail under compressive strain up to ~35%, the deformation was intentionally interrupted because the conditions no longer allowed for uniaxial deformation. For stress–strain curves with monotonically increasing strength during compression, the maximum stress was determined at the point of interruption. Vickers microhardness was measured with load of 5 N and a dwell time of 10 s. The average hardness value was calculated from at least seven indents.

3. Results and discussion

3.1. Processing

It is generally accepted that the microstructure and texture varies with the strain path during multiple passes through an ECAP die [1,19]. In this study, specimens were pressed using processing route A. Although route Bc offers better grain refinement [6,19], our preliminary trials reveled tendencies for cracking of the sample towards the end of 2nd pass via route Bc (i.e., close to the transition between the sheared and unsheared parts after 1st pass). Route C has been used for ECAP processing of Ti at RT via a 90° die by Zhao et al. [24]. However, this route leads to a repeating of the shear along the same planes after each pass [19]. Because route A changes the shear plane in a billet after each pass,