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High-pressure torsion of titanium at cryogenic and room temperatures: Grain size effect on allotropic phase transformations

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Abstract

Titanium in the form of bulk and powder was processed by severe plastic deformation using high-pressure torsion (HPT) at cryogenic and room temperatures to investigate the influence of grain size on allotropic phase transformations. Almost a complete α (hexagonal close-packed, hcp) to ω (hexagonal) phase transformation occurred under a pressure of 6 GPa at room temperature until the grain size reached the submicrometer level, while the formation of β (body-centered cubic, bcc) phase was not detected. The ω-phase fraction and the ω → α transition temperature decreased with processing at cryogenic temperatures and/or with using powders, i.e. with decreasing the grain size to the nanometer scale during the deformation. First-principles calculations found the β phase to be dynamically unstable (neither stable nor metastable), while both α and ω phases are dynamically stable at 0 and 6 GPa. This explains why the β phase was not detected in this study using different methods such as X-ray diffraction analysis, high-resolution transmission electron microscopy, automated crystal orientation mapping and electrical resistivity measurements. Mechanical properties of the HPT-processed Ti were also examined.

Keywords: Severe plastic deformation (SPD); Ultrafine-grained (UFG) materials; First principles calculations; Omega phase; Beta phase

1. Introduction

Titanium exhibits a phase transformation from the hexagonal close-packed (hcp) α structure to the body-centered cubic (bcc) β structure at temperatures above 1155 K under ambient pressure [1]. It also exhibits a phase transformation from α to the hexagonal ω phase under pressures well above 2 GPa at ambient temperature [1–4]. Two other phases of Ti have been found under high pressures: distorted hcp [5] and distorted bcc [6]. There are also several reports that Ti with the face-centered cubic (fcc) structure is formed at ambient conditions when the grain size is well below 10 nm [7–9]. It is important to examine the possible effects of grain size on the stability of different phases under high pressures.

High-pressure torsion (HPT) appears to be an ideal process for controlling grain size under high pressures in dynamic conditions (e.g. during deformation) [10–14]. Recently, much attention has been paid to grain refinement [15–17] and phase transformations [18–20] in pure Ti during severe plastic deformation using HPT because of the

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enhanced mechanical properties [20], wear resistance [15], corrosion resistance [21] and biocompatibility of nanostructured Ti [22]. Previous work has determined that processing by HPT affects phase transformations in a number of ways: (i) accelerating the stress-induced phase transformation such as the $\alpha \rightarrow \omega$ transformation in Ti [18–20]; (ii) reducing the effective temperature for phase transformation such as β–α–ω transitions in Zr-based alloys [23,24]; (iii) introducing grain size-induced phase transformation such as hcp–fcc transitions in Co [25]; and (iv) creating non-equilibrium phases such as $\omega + \beta$ in Zr [26,27].

In this study, pure Ti is torsionally deformed by HPT under a pressure of 6 GPa and the effect of grain size on phase transformations is investigated. Experimental analyses and first-principles calculations are employed to examine whether bcc is formed in the HPT-processed Ti as reported for Zr [26,27]. Note that both Zr and Ti belong to the group IV of the transition metals, and have similar relative energies for the formation of different phases [28].

2. Experimental

In order to control the grain size under high pressures, this study used discs of high-purity Ti (99.9%) 10 mm in diameter and 0.8 mm thick (annealed for 1 h at 1073 K), powders of Ti with particle sizes <45 μm and powders of Ti mixed with 20 wt.% Al_2O_3. The Ti–Al_2O_3 powder mixture used in this study matches the material used in an earlier study using HPT [29]. HPT was carried out on the bulk discs, powders and mixed powders with Al_2O_3 for 10 turns with a rotation speed of 1 rpm under a pressure of 6 GPa at room temperature using the facility illustrated in Ref. [30]. In order to ensure reduction of the grain size to the nanometer scale, HPT was also carried out on the samples at cryogenic temperatures in liquid nitrogen, as attempted earlier [14,17,31]. It should be noted that before processing at cryogenic temperatures the Ti powders and mixed powders with Al_2O_3 were first consolidated at room temperature using HPT for 10 turns under a pressure of 2 GPa.

A thermocouple located at the center of the upper anvil 10 mm away from the surface of the disc was used to record the temperature during HPT processing. As shown in Fig. 1, the temperature gradually increased from 280 K (temperature in winter season in Fukuoka) to 320 K during room temperature processing because of heat generation [30,32] (average temperature: ~300 K). However, the temperature reached ~100 K using a liquid nitrogen cooling system and did not increase during the processing, despite significant heat generation.

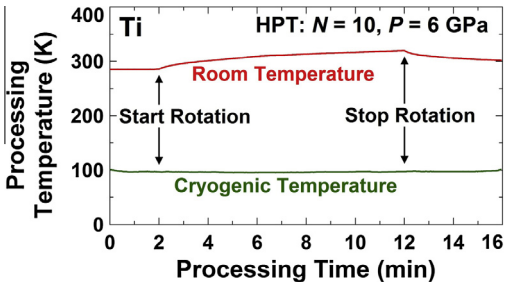

Fig. 1. Processing temperature vs. processing time for bulk Ti samples processed at room and cryogenic temperatures.

The HPT-processed disc-shaped samples 10 mm in diameter were first polished to a mirror-like surface and the Vickers microhardness was measured with an applied load of 500 g for 15 s at 3–4 mm away from the disc center.

X-ray diffraction (XRD) analysis was performed using the Cu Kα line to examine the phase transformations. The ω-phase fraction was calculated using XRD analysis by taking the ratio of the intensity of $(10\bar{1}1)_\omega$ peak to the intensity of $(10\bar{1}1)_\omega + (10\bar{1}1)_\alpha$ peaks.

For transmission electron microscopy (TEM), thin foils were prepared from positions 3–4 mm away from the disc center with either a focused ion beam system followed by ion milling or a twin-jet electrochemical polisher with a solution of 5% $HClO_4$, 25% $C_3H_3(CH_2)_2CH_2OH$ and 70% CH_3OH. TEM was performed at either 200 or 300 kV for microstructure observation in bright-field and dark-field modes and for recording selected-area electron diffraction (SAED) patterns from regions ~1 μm in diameter. The grain size was measured by averaging the two orthogonal axes of each bright area in the dark-field images. Between 90 and 140 grains were randomly selected for each group of samples.

Crystal orientation analysis and phase mapping were performed using scanning transmission electron microscopy (STEM) with an automated crystal orientation mapping system at a voltage of 200 kV.

In order to examine the transition temperatures as well as the activation energies for phase transformations [33], electrical resistivity measurements were conducted during heating at heating rates of 2, 5 and 22 K min^{-1}. Miniature rods with a 0.5 mm square cross-section were cut from the discs 2 mm away from the disc center and four-point electrical resistivity measurements were carried out using an AC current of 100 mA. The electrical resistivity was determined by Ohm's law by measuring the voltage generated within a 5 mm central length of the rods.

Tensile testing was performed using miniature tensile specimens (1.5 mm gauge length, 0.7 mm gauge width and 0.7 mm thickness), as described in detail in Ref. [20].

3. Theoretical calculations

For first-principles calculation, Vienna Ab initio Simulation Package (VASP) code was employed [34–36]. The plane-wave basis projector-augmented wave method [37] was used in the framework of density functional theory within the generalized gradient approximation in the Perdew–Burke–Ernzerhof form [38]. A plane-wave energy cut-off of 300 eV was applied. The Brillouin zones were sampled using $17 \times 17 \times 23$, $26 \times 26 \times 14$ and $20 \times 20 \times 20$ meshes for the ω, α and β unit cells,