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Microstructural evolution of precipitation-hardened β-type titanium alloy through high-pressure torsion

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Abstract

In order to elucidate the microstructural refinement mechanism and the effect of secondary phase on the microstructural evolution of β-type titanium alloy, severe plastic deformation was conducted on samples of a precipitation-hardened Ti–29Nb–13Ta–4.6Zr (TNTZ). Specifically, TNTZ that was precipitation-hardened through an aging treatment ($TNTZ_{AT}$) was subjected to high-pressure torsion (HPT) processing ($TNTZ_{AHPT}$). The microstructure of $TNTZ_{AHPT}$, which has been evaluated as a function of the torsional rotation number, N, exhibits ultrafine elongated β grains. The needle-like α precipitates in $TNTZ_{AT}$, which exhibit a diameter of approximately 12 nm, are homogeneously distributed within the β grains. The dislocation density and subgrain diameter, estimated by X-ray line profile analysis, saturate at approximately $4.2 \times 10^{16}\ m^{-2}$ and 12.2 nm, respectively, at $N \geqslant 10$. The β grains contain nanostructured subgrains having non-uniform morphologies surrounded by blurred and wavy boundaries. A saturated hardness distribution at approximately 450 HV indicates that microstructural homogeneity has been achieved at $N \geqslant 10$. The α precipitates enhance the β grain refinement and microstructural homogeneity is achieved in $TNTZ_{AHPT}$ at $N \geqslant 10$, whereas this occurs at later stages ($N > 20$) in TNTZ which is solution-treated and therefore does not contain any α precipitates.

Keywords: Titanium alloys; Precipitation; Nanostructured materials; Dislocation structure; High-pressure torsion

1. Introduction

With respect to processing, microstructure and properties, metastable β-type titanium alloys can fulfill the requirements for biomedical applications, such as a low Young's modulus and high static and dynamic strength [1–3]. These alloys can also exhibit the levels of toughness and fatigue resistance required for aerospace and automotive applications [4,5]. Interestingly, even though the correlation between the microstructure and mechanical properties of these alloys has been well recognized in the literature, most studies have focused on conventional thermomechanical processing in order to control their microstructure, which involves refinement of the single-phase polycrystalline β matrix and controlling secondary phases such as α and ω precipitates [1–5].

Severe plastic deformation (SPD), introduced through processes such as equal-channel angular pressing [6] and high-pressure torsion (HPT) [7], has been recognized as an effective way to improve the mechanical properties of bulk metallic materials by means of microstructural refinement. The large numbers of dislocations introduced by severe deformation gradually become arranged into high-angle boundaries, resulting in an ultrafine-grained and/or

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nanometer-scale microstructure [6,7] because of the increased fraction of grain boundaries, which block sliding dislocations.

In order to achieve a homogeneous microstructural refinement, strain inducement varies in metallic materials according to their stacking fault energy. For example, homogeneous grain refinement for the face-centered cubic pure Al, which has a high stacking fault energy, can be achieved after one rotation ($N = 1$) during HPT and subsequent recovery of dislocations, and grain growth occurs thereafter [8]. In contrast, a higher strain is needed to obtain homogeneous microstructural refinement in metals having low stacking fault energies, such as Mo [9], Cr [10] and W [11], when subjected to SPD.

Recent studies [12–15] have reported that ultrafine-grained or nanostructured β-type titanium alloys can be attained through SPD. The microstructure of single-phase body-centered cubic (bcc) β-type Ti–29Nb–13Ta–4.6Zr (TNTZ) subjected to HPT processing ($TNTZ_{HPT}$) have exhibited ultrafine elongated β grains with nanostructured subgrains surrounded by blurred and wavy boundaries, which indicates a non-equilibrium state as well as the accumulation and rearrangement of dislocations [12,13]. This microstructure has resulted in promising mechanical properties, such as ultimate tensile strength and hardness values of approximately 1100 MPa and 300 HV, respectively. However, in contrast to Al, a homogenous microstructure for $TNTZ_{HPT}$ has been achieved during HPT processing with rotation numbers (N) > 20.

The metastable structure of β-type titanium alloys can be precipitation-hardened and strengthened by controlling the precipitate phases, such as the hexagonal close-packed (hcp) α phase [4,5,16,17]. In this case, precipitation of the α phase occurs during aging, which significantly increases the mechanical strength. Therefore, microstructural control through SPD of β-type titanium alloys having multiple phases, including precipitate phases in the β matrix, has been attracting greater attention [18,19]. Furthermore, the co-deformation behaviour of the α phase in the β structure, where α and β exhibit different flow behaviors during deformation, can influence the resulting microstructure [20–22]. Kim et al. [23] reported that the α phase is three times stronger than the β matrix for titanium. Therefore, the different flow behaviors of the α phase and the β matrix trigger additional shearing processes through SPD.

Even though the α phase has a higher Young's modulus and exhibits less elongation-to-failure behavior than the β matrix [14,15], it can be used as a means to refine the β grains during HPT processing, and the resulting increased volume fraction of boundaries and triple junctions can help maintain a low Young's modulus and provide adequate elongation to failure [11,24]. For example, Horita et al. and Tsuji et al. have reported that secondary phases could increase both the strength and the elongation to failure of Zn–Al [20] and Al–Fe [21] alloys and steel [25]. Hence, it was anticipated that the initial α phase might assist in the microstructural refinement by trapping grain boundaries, and this could also contribute to the enhancement of the strength.

In a recent study, SPD in the form of HPT processing has been employed in order to introduce a large strain in β-type TNTZ, which contains α precipitates after aging. The major goals of the present study are to provide comprehensive information on the microstructural refinement mechanism and the effect of the α precipitates on the microstructural evolution during HPT processing. Hardness distributions in the radial and depth directions on the surface and cross-section were carried out in order to investigate local microstructural changes. Furthermore, in order to identify the microstructural mechanism, an X-ray line profile analysis (XLPA) [26], using a convolution multiple profile (CMWP) fitting [27,28] method, was performed to analyze the dislocation structure and subgrain diameter.

2. Experimental procedures

2.1. Materials

The chemical composition (Table 1) of the hot-forged TNTZ bar that was used in this study was determined by conventional chemical and gas analyses. Namely, the metallic elements were determined using an inductively coupled plasma optical emission spectrometric method, C and O were analyzed using an infrared absorption method after combustion, and N and H were analyzed using a thermal conductivity method. The TNTZ bar was subjected to aging treatment (AT) at 723 K for 259.2 ks ($TNTZ_{AT}$) after solution treatment at 1063 K for 3.6 ks in vacuum, followed by water quenching. The $TNTZ_{AT}$ bar was then machined into coin-shaped specimens with a diameter of 20 mm and a thickness of 0.8 mm. These specimens were then subjected to HPT processing ($TNTZ_{AHPT}$) under a quasi-constrained condition [7], during which the lower HPT anvil was rotated 1, 5, 10 or 20 times (N) with a rotational speed of 0.2 rpm (0.0244 rad s^{-1}) under a pressure of 1.25 GPa in air at room temperature. The equivalent strain, ε_{eq}, was estimated [7] by the following equation:

$$\varepsilon_{eq} = 2\pi rN/t\sqrt{3} \quad (1)$$

where r is the distance from the specimen center, π is the ratio of the circumference of a circle to its diameter, N is the rotation number and t is the specimen thickness.

2.2. Microstructural characterization

The phase constitutions of each specimen was analyzed by X-ray diffractometry (XRD) at the half-radius (r_h) position, where $r = 5$ mm, using a Cu K_α radiation with an operation voltage of 40 kV and a tube current of 40 mA. Furthermore, the microstructure was observed using a transmission electron microscopy (TEM) with an accelerating voltage of 200 kV and high-resolution TEM (HRTEM) with an accelerating voltage of 300 kV at the r_h position on