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Formation of an ultrafine-grained structure during equal-channel angular pressing of a β-titanium alloy with low phase stability

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A uniform, well-defined ultrafine-grained structure was attained by equal-channel angular pressing in a metastable $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$ β-titanium alloy with low phase stability. In contrast, coarser β grains of several micrometers were yielded upon equivalent plastic strain in a relatively stable $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$ alloy. The pronounced deformation-induced grain refinement is proposed to mainly result both from the stress-assisted β → α″ martensitic and α″ → β backward transformations during severe plastic deformation, and from the interaction between α″ and dislocations in the β phase.

Keywords: Ultrafine-grained microstructure; Equal-channel angular pressing; β-titanium alloys; Grain refinement

Bulk ultrafine-grained and nanostructured metals and alloys exhibit unique mechanical properties and have attracted growing interest during the past decade [1,2]. As a promising process, equal-channel angular pressing (ECAP) is widely utilized to fabricate fully dense ultrafine-grained and nanostructured metallic materials [2–12]. The extent of grain refinement by ECAP is strongly dependent on the imposed strain, the processing route as well as on the mechanism of plastic deformation [2]. In most cases, grain refinement by ECAP relies on intensive dislocation generation and slip [3,4]. Relatively limited effort has been devoted to complex metal and alloy systems involving phase transformation or twinning during ECAP processing such as in NiTi [9,10], stainless steel [11,12] and Ti [8].

Depending on phase stability, the dominant deformation mechanism of body-centered cubic (bcc) structured β-titanium alloys may vary from dislocation slip to twinning/martensitic transformation [13,14]. The interaction between these mechanisms is believed to greatly facilitate grain refinement [10,12,15–17]. Recently, nanosized grains less than 50 and 100 nm were readily attained in β-titanium alloys deformed by cold rolling [18,19] and compression [18,20,21], respectively. The cold-rolled nanostructure was found to be thermally unstable with quick grain growth to the ultrafine scale after annealing between 773 and 923 K [19]. On the other hand, the nanostructure formed during compression was highly localized with some regions containing micrometer-sized grains [18,20] or even amorphous structure [21]. Stimulated by pronounced grain refinement assisted by martensitic transformation in NiTi [9,10] and stainless steel [11,12], the present work is thereby aimed at tailoring β phase stability to yield a uniform and well-defined ultrafine-grained structure in β-titanium alloys by means of ECAP.

Based on the bond order/*d*-orbital energy level map (Bo/Md) developed by Morinaga et al. [14], two β-titanium alloys were designed, including $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$ (TNTC, at.%), which has high stability (Bo = 2.866, Md = 2.423) with dislocation slip as the main deformation mechanism, and $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$ (TNZS, at.%), which exhibits low stability (Bo = 2.866, Md = 2.455) with a high tendency for martensitic transformation and twinning upon deformation. Master alloys were prepared by arc-melting a mixture of high-purity elements (99.99%) under an argon atmosphere with a Ti getter. Cylindrical rods 10 mm in diameter and about 80 mm long were fabricated by cold crucible casting followed by heat treatment at 1273 K for 24 h and subsequent water quenching. Samples of both alloys

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were inserted into a preheated 90° die at 903 K, held for 20 min and subjected to ECAP for four passes following route C [2]. Structural characterization was carried out using X-ray diffractometry (XRD, CuK_α), optical microscopy and transmission electron microscopy (TEM, FEI Tecnai F20, 200 kV). Thin slices for TEM analysis were prepared by mechanical grinding and polishing followed by argon ion milling. In order to verify the β phase stability, some samples after quenching were compressively deformed at a strain rate of 3.0×10^{-3} s^{-1} to a strain of 30% at room temperature.

Prior to ECAP processing, both $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$ and $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$ alloys showed coarse and equiaxed β grains 200–400 μm in size. No other phases besides bcc β were detectable by XRD and microscopic analyses for both alloys after heat treatment (Fig. 1a) and for compressively deformed $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$ (not shown here). In contrast, apart from the β phase, β twins and the orthorhombic α″ phase due to the β → α″ martensitic transformation were present in the compressively deformed $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$, as indicated in Figure 1b. The bright-field TEM micrograph of the deformed $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$ (Fig. 1c) shows a typical microstructure composed of β phase (bright contrast) as verified by the selected-area diffraction (SAD) pattern (top right inset in Fig. 1c) and α″ martensite (dark contrast) as corroborated from the fast Fourier transform (FFT) of the corresponding high-resolution TEM (HRTEM) image, which is in agreement with XRD analysis (not shown here). In addition, the weak and diffuse streaks featured in the SAD pattern (top right inset of Fig. 1c) implied the presence of the athermal ω phase in the β phase of the compressively deformed $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$.

After the ECAP processing, the β grains in $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$ were refined to an average size of 1.4 μm with near equiaxed shape (Fig. 2a). The SAD pattern (bottom right inset in Fig. 2a) of individual grains gave no hint for the presence of the α″ phase. As a result of severe plastic deformation, a high density of dislocations in the form of dislocation tangles was found in a large number of grains (top right inset in Fig. 2a), indicating that the formation and evolution of dislocation cells were the primary mechanism for grain refinement in $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$ [22]. It also implies that further reduction in grain size may be possible when more strain is imposed.

On the other hand, $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$ with lower phase stability experienced more pronounced refinement after undergoing the same ECAP processing as that done to $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$, which has an average grain size of 412 nm (Fig. 3), less than a third of the size of ECAPed $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$. Moreover, the narrower grain size distribution in $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$ indicated a more uniform grain structure. The corresponding SAD pattern (inset in Fig. 3a) showed dotted circles, confirming the presence of a large fraction of high-angle grain boundaries (HAGBs). The interior of a large number of grains in $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$ exhibited a much higher density of dislocations (Fig. 3a) with a complicated structure, as further elucidated in detail in Figure 4. The ultrafine grain shown in Fig. 4a was com-

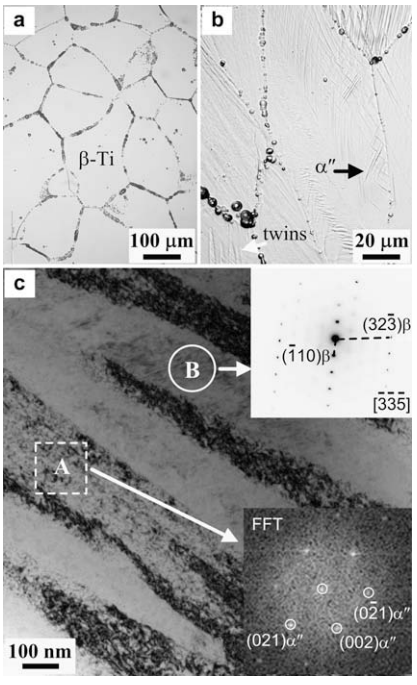

Figure 1. Optical images of (a) heat-treated and (b) compressively deformed $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$. (c) TEM bright-field image showing α″ martensite formed in the β parent phase in the deformed $Ti_{67.4}Nb_{24.6}Zr_5Sn_3$; the top right inset displays the SAD pattern from region B and the bottom right inset shows the FFT of the corresponding HRTEM image for region A.

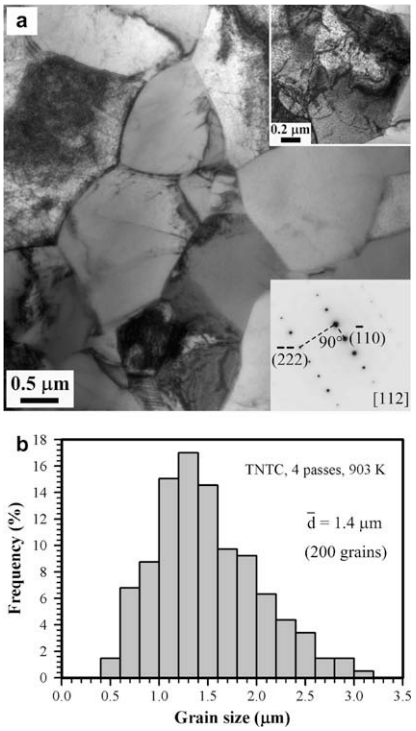

Figure 2. (a) TEM bright-field image showing refined equiaxed β grains with the presence of dislocation tangles (top right inset) in the ECAPed $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$; the bottom right inset shows an SAD pattern for an individual β grain. (b) Distribution of grain sizes in the ECAPed $Ti_{73.5}Nb_{19.6}Ta_{4.5}Cr_{2.4}$.