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In-situ investigation of stress-induced martensitic transformation in Ti–Nb binary alloys with low Young's modulus

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ABSTRACT

Microstructure evolution, mechanical behaviors of cold rolled Ti–Nb alloys with different Nb contents subjected to different heat treatments were investigated. Optical microstructure and phase compositions of Ti–Nb alloys were characterized using optical microscopy and X-ray diffractometre, while mechanical behaviors of Ti–Nb alloys were examined by using tension tests. Stress-induced martensitic transformation in a Ti–30 at%Nb binary alloy was in-situ explored by synchrotron-based high-energy X-ray diffraction (HE-XRD). The results obtained suggested that mechanical behavior of Ti–Nb alloys, especially Young's modulus was directly dependent on chemical compositions and heat treatment process. According to the results of HE-XRD, α″-V1 martensite generated prior to the formation of α″-V2 during loading and a partial reversible transformation from α″-V1 to β phase was detected while α″-V2 tranformed to β completely during unloading.

1. Introduction

Ti-based alloys have attracted much attention due to their high corrosion resistance, good compatibility, low Young's modulus [1,2]. Recently, β-type Ti–Nb based alloys consisting of non-toxic elements have attracted considerable interest as a promising biomaterial. Those alloys exhibit a martensitic transformation from β (disordered bcc) to hexagonal martensite (α′) or orthorhombic martensite (α″), which depends mainly on the alloy composition and materials processing [3–5]. Previous sstudies have shown that acicular α″ may form either in isothermal conditions or under tensile tests [6–9]. Reports indicated that martensite formation becomes possible below Ms by supplying an extra amount of energy by mechanical means, which is referred to as stress-induced martensite (SIM) [6]. SIM formation may lead to concomitant improvements in strength and ductility, as has been demonstrated in steel technology [6]. On the other hand, a low Young's modulus of approximately 40 GPa has been observed in an unstable β Ti alloy polycrystal undergoing stress-induced α″ martensitic transformation during cold rolling [8]. The low modulus and superelasticity of metastable β titanium alloys was elucidated by a continuous phase transformation from β to α″ during loading [10]. Those aforementioned results have indicated that the deformation mechanisms in the Ti–Nb system alloys are quite complex, which are closely related not only to the stability of the β phase but also to the detailed scenario of deformation process. Moreover, mechanical behaviors of Ti–Nb alloys are directly correlated to evolution of microstructure during deformation. For the sake of tailoring Ti–Nb alloys for biomaterial application, an in-depth understanding of the microstructure evolution, phase transformation behavior, mechanical properties and relationship between them is required. The aim of this paper is to investigate the relationship between microstructure and mechanical properties of Ti–Nb alloys. Stress-induced α″ martensite transformation was characterized by using in-situ high-energy X-ray diffraction (HE-XRD). The evolution of lattice strain and phase transformation kinetics vs. applied stress in the Ti–30 at%Nb (noted as Ti–30Nb) alloy were in-situ traced during tensile loading and unloading.

2. Experiment

The Ti–20 at% Nb(Ti–20Nb) and Ti–30 at% Nb(Ti–30Nb) ingots were melted in an arc-melting furnace under argon atmosphere. Ingots were melted six times to ensure chemical homogeneity. The as-cast alloys were sealed in a vacuum in a quartz tube and homogenized at 1000 °C for 3.6 ks followed by furnace cooling. Cold-rolling was conducted at room temperature with a reduction

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of 90% in thickness and the final thickness of Ti–Nb sheets was about 1 mm. Specimens for optical microstructure observation, X-ray diffraction (XRD) and tension were cut using an electro-discharge machine. The specimens were cleaned with ethanol and encapsulated in quartz under a 25 Torr partial pressure of high-purity Ar to avoid oxidation during solution treatment. Solution heat treatments of Ti–Nb alloys were conducted at 750 °C, 850 °C and 950 °C for 3.6 ks, respectively and specimens were quenched into water by breaking the quartz tubes. Optical microstructure of Ti–Nb alloys was revealed by using an etchant of HF(5 vol%)+ HNO_3(5vol.)+water(bal.). XRD measurements were conducted at room temperature using a Rigaku D/MAX diffractor with Cu-Kα radiation (λ=1.5406 Å) operating at 40 kV and 30 mA. Tensile tests were carried out at a strain rate of $1.0\times10^{-3}\ s^{-1}$ at room temperature. The gauge length of specimens was 20 mm, and triplicate tests along rolling direction (RD) were conducted. The fracture morphology was observed using scanning electron microscopy (SEM) Quanta FEG 250 operated at 15 kV.

In-situ synchrotron-based HEXRD experiments were performed for studying the mechanical behavior of the Ti–Nb alloy, using the beam-line 11-ID-C at the Advanced Photon Source (APS) at Argonne National Laboratory. An intense monochromatic X-ray beam with the energy of 115 keV (wavelength of 0.10801 Å) and a beam size of $400\times400\ \mu m^2$ was used to map the lattice strain distributions of the specimens under the tensile loading mode. At different loading levels, the diffraction patterns were recorded on a two-dimensional (2D) detector, which was placed at about 2.1 m behind the sample. The CeO_2 powders were attached to the surface of the measured specimen for calibrating the accurate distance between the specimen and the geometry position of 2D detector during deformation. During the in-situ experiments, the CeO_2 powders were removed from the sample for obtaining clear diffraction patterns. The Bragg angle θ_{hkl} of the *hkl* plane was determined by fitting the individual single peak or overlapped peaks in the diffraction spectra with General Structure Analysis System (GSAS) software.

3. Results and discussion

3.1. Microstructure evolution

Fig. 1 shows optical microstructures of Ti–Nb binary alloys obtained by quenching into water from different solution treatment temperatures. As seen in Fig. 1(a)–(c), α″ martensite plates nucleated and distributed homogeneously inside β grains of Ti–20Nb alloys.The average β grain size of Ti–20Nb alloys solution treated at 750 °C, 850 °C and 950 °C was determined to be about 55 μm, 114 μm and 162 μm, respectively. Unlike (β+α″) microstructure of Ti–20Nb, the microstructure of solution treated Ti–30Nb alloys showed single β phase grain structure and the average β grain size of Ti–30Nb after solution treatment at 750 °C, 850 °C and 950 °C was ~51 μm, ~60 μm and ~65 μm, respectively. The results indicated that for both Ti–20Nb and Ti–30Nb specimens, the grain size of β increased with the increasing solution treatment temperature. The stability of bcc β phase in Ti–M alloys (M=trainstition element) can be understood from two aspects [11]: (1) the formation energies of compositions, i.e. decrease in formation energy indicates increase in stability of the phase; (2) the ratio of valence electrons and number of atoms (*e*/*a*), β phase in Ti–M alloys is stablized at the *e*/*a* ratio is 4.2 or more. Karre et al. calculated binary Ti–Nb systms using first-principles calculations with density-functional framework, the theoretical results suggest that the Ti–Nb alloy system have stable bcc (β) phase for Nb content 22 at% and higher [11]. However, according to Bönisch et al., β phase was detected in Ti–29 wt%Nb (Ti–18 at% Nb) alloys solution treated at 670 K+WQ (water quenched), 818 K+WQ, 1420 K+WQ [12], which indicated the stability of β phase was significantly influenced by heat treatment process. For our case, α″ and β phases were both observed in the optical microstructure of cold and solution treated Ti–20Nb alloys, the existence of β was also confirmed by the XRD results, as seen in Fig. 2 (a). Further investigation of optical microstrucutre indicated that the average β grain size of Ti–30Nb was slightly influenced by solution treatment temperature while grain size of Ti–20Nb

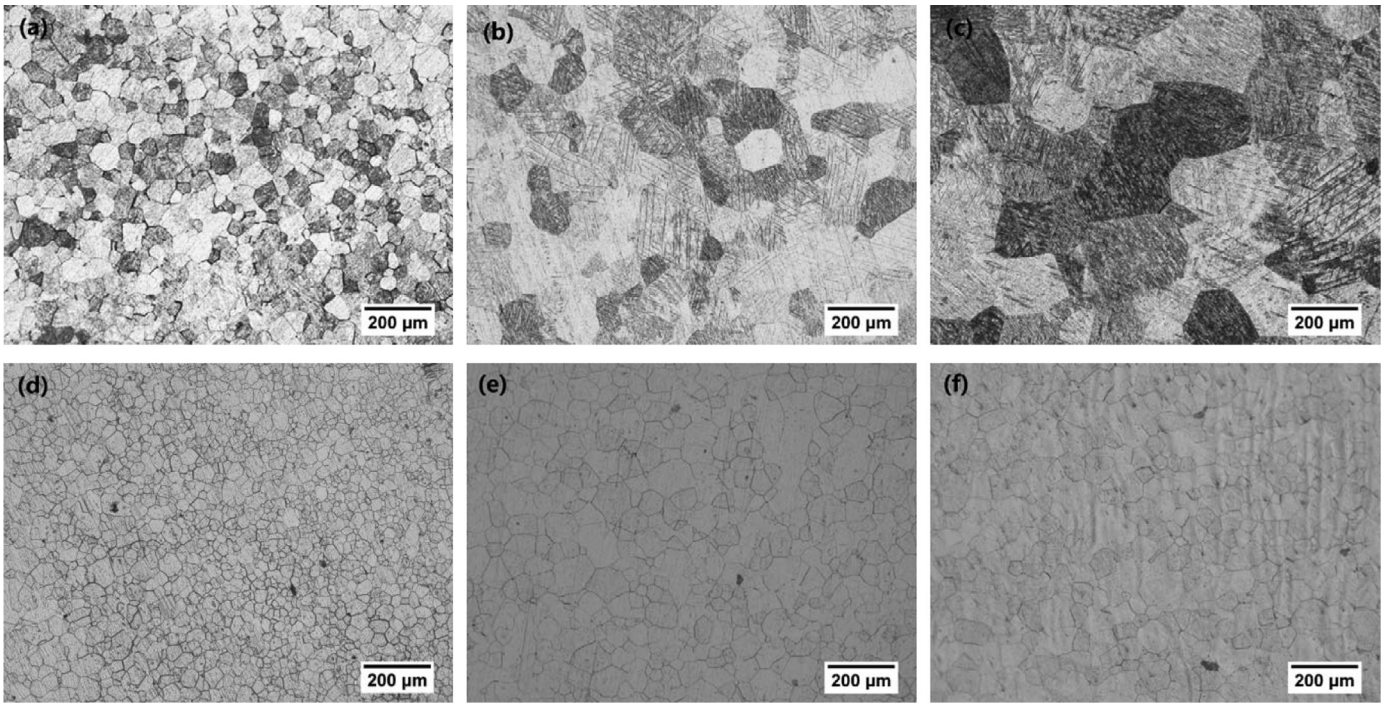

Fig. 1. Optical microstructures of Ti–Nb alloys in solution heat treatment: (a) Ti–20Nb-750 °C; (b) Ti–20Nb-850 °C; (c) Ti–20Nb-950 °C; (d) Ti–30Nb-750 °C; (e) Ti–30Nb-850 °C; (f) Ti–30Nb-950 °C.