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Original Article

Design of uniform nano α precipitates in a pre-deformed β-Ti alloy with high mechanical performance

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ABSTRACT

A strategy for the microstructural design to achieve a critical upper limit of uniform nano α precipitates by controlling the amount of dislocations in the pre-deformed matrix after short time aging treatment was proposed in Ti–10Mo–8V–1Fe–3.5Al (all in wt.%, TB3 alloy) β alloy. The optimal processes focused on the interaction of defects, mainly point defects and dislocations that are generated during cold rolling (CR). Texture evolution of α and β phases was also specified. Amount of nano-scaled α precipitates was characterized by the number density of precipitates observed by SEM, which is saturated at ∼204.1 μm^{-2}. For the CR 20% sample, a good combination of tensile strength (∼1400 MPa) and elongation (∼12%) was achieved after secondary aging at 550 for 1 h, which provides an enlightenment for the re-engineering of traditional precipitate-hardening alloys.

1. Introduction

Near-β Ti alloys, including Beta-C, Beta III, and Ti-8823, possess high specific strengths, high toughness, and good corrosion resistance and are excellent choices not only for structural components, but also as materials to replace Ni-based fasteners and steel fasteners in aerospace and automotive industries [1]. High strength of this type of age-hardenable alloys is obtained by aging treatment to precipitate fine, uniformly distributed incoherent α platelets of 12 orientation variants in metastable β matrix [2–6]. However, direct aging for such commercially available age-hardening alloys is supposed to induce non-uniform dispersion of coarse α precipitates and precipitate-free zones (PFZs) along grain boundaries (GBs). The occurrence of PFZs generally, explained in terms of either vacancy depletion or solute depletion, is considered

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by Thomas et al. [7] as deleterious layers due to preferential plastic deformation and fracture in the zones along GBs. Such unfavorable factors could induce limited strength and ductility of β-Ti alloys even after long aging duration.

Three main methodologies [2,3,8–10] for achieving finer and more uniform dispersion of α precipitates were explored by researchers in different β-Ti alloy systems: (1) ω-assisted α nucleation: the heterogeneous nucleation of the α phase from the precursory ω precipitates; (2) thermally induced compositional fluctuations within the β matrix via the pseudo-spinodal decomposition mechanism to assist α nucleation; (3) a high density of dislocations, grain, or sub-GBs by hot/cold deformation prior to aging to accelerate the kinetics of precipitation. Based on the third mechanism, Xu et al. [9,10] and other researchers [11–13] have studied different aging and mechanical responses to the corresponding pre-deformation degree as well as passes through cold/hot deformation. Furthermore, severe pre-deformation prior to a thermal treatment is often applied in the fastener process of β-Ti alloys to achieve the desired strength. The accumulation of dislocations during cold deformation leads to low ductility. The enhanced solutes transport is considered to induce undesirable consequences, e.g., creep in materials, which associates with edge-dislocation climb [14,15]. This raises a question: will the precipitate density continue to increase with increasing deformation degree? Alternatively, is there an optimum dislocation density, which could yield the saturation of precipitates and properties? The answer to these questions can help improving properties of the work-pieces with control of defect density by deformation [16].

Thus, the major objective of this study is to improve the precipitate dispersion and refine precipitates to achieve better comprehensive mechanical properties via cold deformation and subsequent heat treatment. The selected processes can avoid the disadvantages of other severe plastic deformation methods such as equal channel angular pressing (ECAP), in which the ECAP-dies have limited bearing capacity to process high-strength alloys. By selecting parameters of pre-deformation and subsequent aging process, a series of combination of processing variables were explored in the presented β-Ti alloy.

2. Experimental procedure

In this paper, a β-Ti alloy was selected. It is the Ti–10Mo–8V–1Fe–3.5Al (wt.%), known as TB3 alloy with [Mo]eq = 15–17, often used as fastener material due to its high strength and good formability. This β-Ti alloy was prepared by arcmelting of a mixture of high-purity elements (99.99%) under a Ti-gettered argon atmosphere, with subsequent cogging, intermediate forging, and hot rolling into cylindrical rods of 15 mm in diameter. Subsequently, solution treatment (ST) was carried out at 830 °C for 0.5 h followed by water quenching. This procedure resulted in β grains with an average size of 50 μm. The alloy composition is shown in Table 1. Cold rolling was conducted on 6 × 6 × 10 mm plates at room temperature using the reduction of 10, 20, 30, 50, 70, 80, and 90%. Plate samples were then sealed in evacuated quartz tubes and aged at 500 °C for 1–12 h. The sample (CR20%/500 °C for 1 h) was subsequently annealing-treated at 550 °C for 1 h. The cooling method of all the aging treatment is water cooling. And the general processes were summarized in Fig. 1.

X-ray diffraction method (XRD; Maxima_X XRD-7000) and transmission electron microscope (TEM; JEM-2100) were used to characterize the microstructure of the processed samples. The samples were subjected to orientation imaging microscopy using a field emission scanning electron microscope (JSM-7100F) with electron back-scattered diffraction (EBSD) facility. Field-emission scanning electron microscopy (FESEM; HysitronSU6600) was conducted in the morphology characterization of precipitates and energy spectrum analysis. The Vickers microhardness (HV) was measured by digital microhardness (HVS-50Z070702) under a load of 300 gf for 10 s. Tensile tests were performed using the Instron 1195 testing machine at a strain rate of 1 mm/min.

3. Results

3.1. *Microstructure of single β phase by cold rolling*

3.1.1. *XRD and TEM characterization*

The evolution of microstructure with CR reduction can be observed in TEM images and reflected by XRD measurements (Fig. 2). Fig. 2(a)–(d) of CR specimens show dislocation tangles and cells in different slip systems with increasing CR percentages. This can be associated with the elongated diffraction patterns of CR20% sample shown in the inset of Fig. 2(c). A distinct dislocation structure of subgrain interiors (lower dislocation density) and boundaries (dislocation tangling with higher density) was achieved at large strain (CR90%). XRD tests (Fig. 2(e)) of ST and CR samples at room temperature show that all the samples tested are composed of a single β phase. Moreover, {1 1 0} texture increases, which can be associated with the change of relative diffraction intensity $I(1\,1\,0)/I(2\,0\,0)$, calculated as 4.04 at ST and 39.4 at CR40%. Microstrain of the CR samples was determined by means of XRD measurements [17]. The XRD pattern shows that the measured Bragg reflection profile is a convolution of the functions representing both the instrumental and the physical broadening profile. The instrumental broadening profile was subtracted as a Gaussian type in the present work. According to XRD measurement, the mean microstrains in those samples of ST (CR0%), CR10%, CR20%, CR30%, and CR40% conditions are given as 0.04, 0.33, 0.42, 0.58 and 0.56%, respectively, as shown in Fig. 2(f).

3.1.2. *Texture development*

Fig. 3(a)–(c) shows the texture data in the form of orientation distribution function (ODF) of ST, CR20%, CR40%, CR50% and CR 80% samples. The data were obtained by EBSD measurements scanning on the transverse direction (TD) plane parallel to both the RD and normal direction (ND). Fig. 3(a) indicates that there exists prominent α-fiber component of (0 0 1) [1 1 0] and also strong (1 1 0) [1 1 0] in the ST sample. The orientation density along α-fiber increases with CR reduction up to 20% (Fig. 3(b)) and then decreases at CR40% and CR50% as shown in Fig. 3(c) and (d). When the CR reduction increases to 80%, the main texture component is shifted to (1 1 0) [1 1 0]. Besides, the texture component (0 0 1) [0 1 0] occurs in all the

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