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# A hierarchical and multiphase nanolaminated alloy with an excellent combination of tensile properties



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# ABSTRACT

Ti alloys that possess a fine fully lamellar structure generally exhibit high strength but limited ductility. In this study, a hierarchical and multiphase nanolaminated structure that consists of large microscale primary  $\alpha_p$  grains (~1.5 µm), sub-microscale  $\alpha$  plates (~200 nm) and nanoscale acicular isothermal (orthorhombic)  $\alpha''$  martensites (~15 nm) is produced in a TiZrAlV alloy with a fine (~12 µm)  $\beta$  grain size through the use of severe plastic deformation (SPD) combined with subsequent recrystallisation annealing and aging treatments. This specific structure results in an excellent combination of tensile properties, e.g., an ultimate tensile strength of  $\sigma_b \sim 1545$  MPa and an elongation to failure of  $\varepsilon_f \sim 7.9\%$ . The present study demonstrates an alternative route for enhancing the mechanical properties of titanium alloys with laminated structures.

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#### 1. Introduction

High strength and good ductility are essential properties of structural materials for their applications. However, the strength and ductility of most materials are often mutually exclusive [1–2]. For example, high strength has already been observed in many nanocrystalline metals and alloys, but in most cases, these materials exhibit very low ductility [3–4], which greatly limits their practical applications. Therefore, enhancing the ductility of nanocrystalline materials is a formidable challenge. To overcome this problem, a hierarchical structure consisting of nanocrystalline (NG, < 100 nm), ultrafine (UFG, 100 nm-1  $\mu$ m) and micrometresized grains (CG,  $> 1 \,\mu m$ ) has recently been demonstrated to be a significant route for improving the ductility of nanostructured metals [5–9]. Similar to nanocrystalline metals, conventional lamellar structures in Ti alloys are generally composed of singlemodal fine lamellae, which usually results in high strength but limited ductility [10–11]. Hence, a hierarchical laminated structure that consists of large primary  $\alpha_p$  grains and fine  $\alpha$  lamellae (bimodal structure) or of lamellae with different sizes, e.g., in width with nanometre and sub-micrometre scales, has also recently been used to enhance the combination of mechanical properties of Ti alloys [10–16]. However, a hierarchical structure

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with extremely fine ( $\sim$ 50–100 nm in length) and coarse  $\alpha$ lamellae ( $\sim 1 \,\mu m$  in length) has been reported to result in a better combination of mechanical properties than that of a hierarchical structure consisting of primary  $\alpha_p$  grains and coarse  $\alpha$  lamellae  $(\sim 1 \,\mu m$  in length) [15]. This result suggests that a hierarchical laminated structure with an appropriate amount of extremely fine  $\alpha$  lamellae may have a more significant effect on improving the combination of mechanical properties than a structure with coarse  $\alpha$  lamellae. The extremely fine  $\alpha$  lamellae can lead to a high strength level [17–18], whereas the formation of an appropriate amount of extremely fine  $\alpha$  lamellae can result in more residual ductile  $\beta$  phase, which contributes to good ductility [11,19]. Therefore, excellent combinations of tensile properties may be obtained in a hierarchical structure that possesses extremely fine  $\alpha$  lamellae. However, the width of fine  $\alpha$  lamellae has generally been reported to be  $\sim$  50–100 nm [10,14–16]. Therefore, obtaining extremely fine lamellae, e.g., < 30 nm in width, for improving the mechanical properties of Ti alloys with hierarchical structures is a challenge. An unusual acicular (orthorhombic)  $\alpha^{"}$  martensite has recently been formed in some  $\beta$ -metastable titanium alloys during aging treatments, which presents an extremely fine lamellar width that is smaller than that of  $\alpha$  lamellas and exhibits a high strength [20–22]. This result implies that the isothermal  $\alpha''$  martensite with an extremely fine lamellar width, e.g., < 30 nm in width, can be introduced into a hierarchical structure in some  $\beta$ -metastable Ti alloys. This hierarchical structure may lead to excellent combinations of tensile properties. In the present study, using a

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 $\beta$ -metastable TiZrAlV alloy as a model system, a hierarchical and multiphase nanolaminated structure consisting of large microscale primary  $\alpha_p$  grains ( $\sim 1.5 \ \mu$ m), sub-microscale  $\alpha$  plates ( $\sim 200 \ n$ m) and nanoscale  $\alpha''$  martensites ( $\sim 15 \ n$ m) was successfully produced in an alloy with a fine ( $\sim 12 \ \mu$ m)  $\beta$  grain size using appropriate thermomechanical processing (TMP) treatments, which led to excellent combinations of high tensile strength and reasonable ductility.

# 2. Experimental details

40.2Ti-51.1Zr-4.5Al-4.2 V (wt%) (henceforth denoted as TiZrAlV) with a composition corresponding to a  $\beta$ -metastable alloy was prepared by melting Ti sponge (99.7 wt%), Zr (Zr + Hf > 99.5 wt%), industrially pure Al (99.5 wt%) and V (99.9 wt%) using a ZHT-001 consumable electrode vacuum arc furnace. Subsequently, the ingot was held at 900 °C for 90 min and then forged. The  $\beta$ -transus temperature of the alloy was determined to be  $\sim$  720 °C by differential scanning calorimetry (DSC) measurements. Alloy plates with a thickness of  $\sim$ 6 mm, which were produced by hot rolling the ingot at 950 °C, were solution treated under vacuum at 850 °C for 1 h and then water quenched. The as-quenched samples exhibited a nearly singlephase  $\beta$  structure with an average  $\beta$  grain size of  $\sim\!200\,\mu m$  (data not shown). The as-quenched samples were subjected to TMP treatments, which consisted of severe cold rolling at room temperature (RT) to over 90% reduction, recrystallisation annealing and aging treatments under vacuum. The recrystallisation annealing was performed in the  $\alpha + \beta$  field, i.e., at 675 °C for 10 min, to form a bimodal structure and a fine  $\beta$  grain size, followed by cooling in air to RT. Subsequently, the recrystallised samples were treated with a one-step aging at a high temperature of 625 °C for 2 h (A1) to precipitate coarse precipitates and with a two-step aging, i.e., A1 followed by direct low-temperature aging at either 450 °C for 2 h or 300 °C for  $0.5 \sim 4.5$  h (A2), to precipitate different types or various amounts of fine precipitates. The microstructures and phase compositions of the samples subjected to TMP treatments were characterised and determined using optical metallography (OM), transmission electron microscopy (TEM) and X-ray diffraction (XRD) with Cu  $K_{\alpha}$  radiation. The volume fraction (vol.%) of each phase was determined using quantitative TEM analysis [23] and the Rietveld refinement method using Maud software, which can take the texture and the shape anisotropy of coherent diffraction domains and of micro-deformations (e.g., lamellae) into account [24–26]. The  $\beta$  grain size was determined by approximating the equivalent sphere diameters of  $\beta$  grains by directly measuring their areas from the OM images using a digital micrograph analysis software. The statistical  $\alpha$  grain/width size distributions were obtained from TEM measurements on more than 150 grains/lamellas. Tensile tests of samples with a gauge dimension of  $5 \times 2.2 \times 0.35$  mm<sup>3</sup> were performed at RT using an Instron 5948 machine under a constant cross-head speed with an initial strain rate of  $1 \times 10^{-3}$  s<sup>-1</sup>. The strain was measured with a non-contacting video extensometer. Four specimens were used for tests under each condition, and the deviations in the ultimate tensile strength and elongation to failure were less than 30 MPa and 1%, respectively. The tensile direction was parallel to the rolling direction of the samples.

#### 3. Results and discussion

Fig. 1 shows the tensile engineering stress-strain curves of TiZrAlV subjected to TMP treatments. After cold rolling, recrystallisation (675 °C/10 min) and A1 aging (625 °C/2 h), sample A (curve A) exhibited an ultimate tensile strength of  $\sigma_b \sim 1150$  MPa and an elongation to failure of  $\varepsilon_f \sim 10.7\%$ . After A2 aging (450 °C/2 h), the  $\sigma_b$  increased to ~ 1500 MPa and  $\varepsilon_f$  decreased to ~ 6.5% in sample B



**Fig. 1.** Tensile engineering stress-strain curves of TiZrAlV after cold rolling, recrystallisation and one- and two-step aging. Curve A, 675 °C/10 min+625 °C/2 h; curve B, 675 °C/10 min+625 °C/2 h+450 °C/2 h; and curve C, 675 °C/10 min+625 °C/2 h+300 °C/15 h.



**Fig. 2.** XRD patterns of TiZrAIV after cold rolling, recrystallisation (675  $^{\circ}C/10$  min) and (a) one-step A1 aging (625  $^{\circ}C/2$  h) and two-step A1 aging followed by (b) 450  $^{\circ}C/2$  h and (c) 300  $^{\circ}C/1.5$  h A2 aging.



Fig. 3. OM microstructure of TiZrAIV after cold rolling, recrystallisation (675 °C/10 min) and two-step aging (625 °C/2 h+300 °C/1.5 h); the inset shows the  $\beta$  grain size distribution.

(curve B). After A2 aging at 300 °C/1.5 h, an excellent combination of a high tensile strength of  $\sigma_b \sim 1545$  MPa and a reasonable ductility of  $\varepsilon_f \sim 7.9\%$  was obtained in sample C (curve C), indicating a simultaneous enhancement in strength and ductility compared with those of sample B ( $\sigma_b \sim 1500$  MPa and  $\varepsilon_f \sim 6.5\%$ ).

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