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## Strength and grain refinement of Ti-30Zr-5Al-3V alloy by Fe addition



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#### ABSTRACT

The influence of various Fe contents on the microstructure and mechanical properties of Ti-30Zr-5Al-3V (ZTAV) alloy was investigated. After Fe addition, the grain refinement is obvious and the phase composition changes as  $\alpha'+\beta\to\beta$ . Ti-30Zr-5Al-3V-0.5Fe exhibits a better mechanical property ( $\sigma_b\approx1420$  MPa,  $\epsilon_f\approx5.34\%$ ) than Ti-30Zr-5Al-3V ( $\sigma_b\approx1302$  MPa,  $\epsilon_f\approx4.28\%$ ). Highest ultimate tensile strength ( $\sigma_b\approx1667$  MPa,  $\epsilon_f\approx2.34\%$ ) have been achieved in Ti-30Zr-5Al-3V-1.5Fe. The mechanical properties change owe to the phase transformation, grain refinement, and solid solution strengthening caused by Fe addition.

#### 1. Introduction

Titanium alloys are very attractive materials for aerospace and automotive industries due to their high strength-to-density ratio, good hardenability, excellent fatigue performance, and environmental resistance [1,2]. Recently, many investigations have been performed on the development of high strength Zr-Ti based alloys. Ti and Zr are belonging to the same main group (IVB), their physical and chemical properties are similar. They both have two kinds of crystal structure ( $\alpha$ -and  $\beta$ -phase).  $\alpha$ -phase (hcp) can be stable when the temperature is lower than  $\alpha/\beta$  transus temperature, otherwise,  $\beta$ -phase (bcc) is stable phase. Due to their similar chemical and physical properties, Ti and Zr are infinitely miscible solid solution. Solution strengthening effect is significant in Zr-Ti based alloys, such as Zr-Ti-Al [3,4], Ti-Cu-Al-Zr [5], and Zr-Ti-Al-V (ZTAV) [6,7] etc. Zr-Ti based alloys have shown great potential to be structural materials in aerospace and other fields.

It has been found that ZTAV alloys exhibit good mechanical property, such as Ti-30Zr-5Al-3V, it shows high strength  $(\sigma_b \approx 1258 \text{ MPa}, \, \epsilon_f \approx 8.4\%, \, \text{forged state}) \, [8]$ . This result is owing to the addition of Al and V. Though their content is limited, the influence on mechanical property is very obvious. It indicates that the addition of alloying element is an effective way to change the mechanical properties, phase component and phase transformation temperature [9,10]. The previous study demonstrated that the Fe is an effective grain refinement element for Al and Mg alloy [11]. However, grain refinement and strength enhancement of ZrTi alloy with addition of Fe has not yet been reported. In this work, the grain refinement and strength enhancement of Ti-30Zr-5Al-3V (ZTAV) with Fe addition as the solid

solution element was systematically studied. The results reveal that the grain size changes with the Fe content, which is one of the main factors to enhance the mechanical properties of the TiZr-based alloys. Also, the strength of alloys as a function of the concentration of the solute atom Fe was achieved with the solid solution strengthening scheme.

#### 2. Experimental

All ingots of Ti-30Zr-5Al-3V-xFe (x=0,0.5,1.0,1.5,2.0,2.5) (wt%) (denoted as TZAFV) were prepared by arc-melting technique in a Ti-gettered argon atmosphere. The raw material is prepared by a mixture of sponge Zr (Zr+Hf $\geq$ 99.5 wt%), sponge Ti (99.7 wt%) and industrially pure Al (99.5 wt%), Fe (99.9 wt%), V (99.9 wt%). To ensure compositional uniformity, each ingot was re-melted at least ten times. The actual compositions of each ingot are shown in Table 1. The ingots were heated to 1000 °C, held for 0.5 h, and then hot rolled into plated samples with a thickness of 5.2 mm. The plated samples were cooled in the air after rolling. The total deformation was 70%. Hot rolled samples were removed about 1.5 mm on each rolled surfaces for following tests and analysis.

To analyze the phase structure of hot rolled plated samples, the centers of plated samples were tested by X-ray diffraction (XRD) using copper  $K\alpha$  X-radiation (ASTM: D5380-93(2014)) at a diffraction angle (2 $\theta$ ) range of 20–100° and at an accelerating voltage of 40 kV and a current of 40 mA. The specimens for XRD were mechanically polished by SiC waterproof emery papers up to 2500 grit. Differential scanning calorimetry (DSC) was used to test the phase transition temperatures in heating velocity 10 °C/min adopting the standard of ASTM: F2004-

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Table 1
Actual alloy compositions of the examined ZrTiAlVFe alloys, wt%.

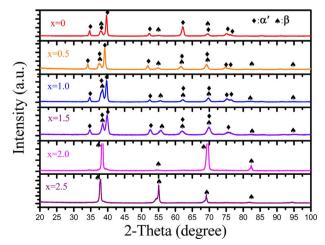
Ti	Zr	Al	V	Fe
62.00	30.00	5.00	3.00	0
61.48	30.01	5.00	3.01	0.50
61.00	30.00	5.00	3.00	1.00
60.49	30.00	5.00	3.00	1.50
60.00	30.00	5.00	3.00	2.00
59.50	30.00	5.00	3.00	2.50
	62.00 61.48 61.00 60.49 60.00	62.00 30.00 61.48 30.01 61.00 30.00 60.49 30.00 60.00 30.00	62.00     30.00     5.00       61.48     30.01     5.00       61.00     30.00     5.00       60.49     30.00     5.00       60.00     30.00     5.00	62.00     30.00     5.00     3.00       61.48     30.01     5.00     3.01       61.00     30.00     5.00     3.00       60.49     30.00     5.00     3.00       60.00     30.00     5.00     3.00

05(2010). Microstructure of the plated samples was observed through Olympus DSX500 type optical microscopy (OM). For TEM, the specimens were mechanically polished with SiC waterproof emery papers up to 2500 grit to a thickness of approximately 30  $\mu m$ . After that, they were twin-jet electrochemically polished in a solution containing perchloric acid (10 vol%) and methanol (90 vol%) at 15 V and below  $-30~{\rm ^oC}$ . For OM, the specimens were mechanically polished using SiC waterproof emery paper of up to 4000 grit and to a final level of approximately 0.04  $\mu m$  with colloidal silica suspension. Each mirror-polished specimen was etched using aqueous solutions of hydrofluoric acid (5 vol%) and nitric acid (15 vol%).

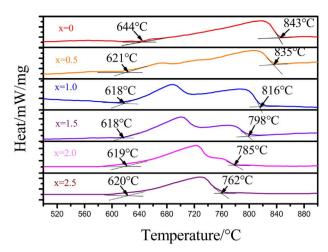
Tensile samples were machined from hot-rolled plates after removing 1 mm thick portions from both sides of the rolled surface. The size of tensile samples is shown in Fig. 1. Uniaxial tensile tests (ASTM: E8/E8M-11) were performed on an *INSTRON* 5982 machine at a strain rate of  $5\times10^{-4}~\rm s^{-1}$ . The strain during the entire testing process was monitored using an extensometer with a 12.5 gauge length. The tensile direction was parallel to the rolling direction of the sample. The microhardness of the polished alloys is measured with a load of 200 gf for 10 s.

#### 3. Results and discussion

Fig. 1 shows the XRD patterns of ZTAV and ZTAFV alloys. The XRD results show that the phase constitution and lattice parameters are changing with Fe content. Without Fe addition, all peaks match well with those of  $\alpha'$ - and  $\beta$ - phases. With the Fe content increases from 0 wt% to 1.5 wt%, the XRD patterns show similar curve. It indicates that they are all compose of  $\alpha'$  and  $\beta$  phases [12]. The intensity of the peak of  $\beta$ -phase increases with the Fe content, which indicates that the content of  $\beta$  phase increases with the Fe content. Meanwhile, as the atomic radius of Fe (0.126 nm) is smaller than those of Ti (0.147 nm) and Zr (0.160 nm), the addition of Fe results in the lattice distortion that causes the lattice parameters of  $\alpha'$ - and  $\beta$ - phase to decrease. It is shown in the XRD patterns that the peaks of  $\alpha$ - and  $\beta$ - phases shift toward to high angle orientation with the addition of Fe. When the Fe



**Fig. 1.** The XRD patterns of the Ti-30Zr-5Al-3V-xFe (x=0,0.5,1.0,1.5,2.0,2.5).



**Fig. 2.** DSC curves of Ti-30Zr-5Al-3V-xFe (x=0,0.5,1.0,1.5,2.0,2.5).

content increase further from 1.5wt% to 2.5 wt%, the XRD patterns show only  $\beta$  phase. This is due to the reason that the eutectoid transformation of  $\beta{\rightarrow}\alpha'$  phase is wholly hindered when the sample cooled from hot rolling state with the increase of Fe content.

Fig. 2 shows the DSC curves of TZAFV alloys. The DSC results indicate that the martensite transformation temperature of the alloys is changing with Fe content. The end temperature of martensite transformation of TZAFV reduces with the increasing of Fe content. This results can be attributed to the  $\beta$ -stabilizer element of Fe in Ti and Zr alloy from previous researches [13] and Ti-Fe, Zr-Fe binary phase diagrams.

Fig. 3 shows the size of optical micrographs of ZTAV and ZTAFV alloys with different Fe contents. The size of  $\beta$  grain changes with Fe content increasing. When Fe content reaches 0.5 wt%, the smallest size of  $\beta$  grain can be obtained. However, the size of  $\beta$  grain would increase with increasing Fe content. The growth restriction factor (GRF) was proposed to measure the effect of solute concentration, such as Fe and other solute elements, on grain size and crystal morphology. The grain size reaches a minimum at a critical GRF [14]. This explains why the size of  $\beta$  grain reaches the smallest value when Fe content is 0.5 wt%. Beyond the critical GRF, the transition from cellular to dendrite growth would occur, and the grain size would increase with increasing Fe content. The constitutional supercooling in ZTAFV alloys would increase with Fe content increasing. With the increase of Fe content in ZTAFV alloys, the foreign clusters, which consist of Fe atoms, would tend to grow as the flake eutectoid phase. This means that they could not serve as the potential nucleation of primary β grains. In other words, the number of potential nucleation of primary  $\beta$  grains plays a key role in the grain refinement of ZTAFV alloys with low content of Fe. Furthermore, the pinning effect of intermetallic compound on grain boundaries can be another factor which also plays an important role in the grain refinement of ZTAFV alloys with low content of Fe. The pinning effect of intermetallic compound can hinder the growth of primary  $\beta$  grains. In the whole process of the crystallization of ZTAFV alloys, the way of grain grows, the number of potential nucleation of primary  $\beta$  grains and the pinning effect on grain boundary, could contribute to the peak-valley relationship between Fe content and grain

Fig. 4 shows the TEM images and the corresponding SAED patterns of ZTAV and ZTAFV alloys with Fe content of 0.5 wt% (ZTAFV-0.5), 1.0 wt% (ZTAFV-1), 1.5 wt% (ZTAFV-1.5), respectively. The SAED patterns proves that the structure of the lath or acicular grain is hcp, namely  $\alpha'$  phase. It shows different morphology of  $\alpha'$  phase existed in the alloys. The  $\alpha'$  phase exists as lath in the ZTAV alloy, while it exists as acicular martensite with different width for Fe content of 0.5, 1.0, and 1.5 in ZTAFV alloy. It reveals that the morphology of  $\alpha'$  phase is dependent on the Fe content. The reported results demonstrated that

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