

# Preparation of the ZrTiAlV alloy with ultra-high strength and good ductility

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## ABSTRACT

The 30Zr–62Ti–5Al–3V (wt%) alloy with ultra-high strength and good ductility was prepared via consumable electro-arc melting, forging, and heat treatment. The phase transition and structure evolution of the alloy during heat treatment were also investigated. X-ray diffraction (XRD) results show an orthorhombic martensite  $\alpha''$  phase in the specimen solution-treated at 825 °C. The orthorhombic martensite  $\alpha''$  phase decomposed into  $\alpha$  and  $\beta$  phases during the subsequent ageing treatment. No intermetallic compound peak was found in the XRD patterns of the specimens under different conditions. Microstructural analysis shows that the fine short-bar-shaped  $\alpha$  phase precipitated along the original  $\beta$  grain boundary together with the ultrafine dot-shaped  $\alpha$  phase that presented inside the original  $\beta$  grain when the ageing temperature was lower than 550 °C. As the ageing temperature increased, the dot-shaped  $\alpha$  phase inside the original  $\beta$  grain gradually grew into the short-bar shape. The different microstructures resulted in varied mechanical properties. After ageing at 500 °C, the tensile strength reached 1622 MPa; at the same time, the 2.1% ductility in terms of elongation was retained. As the ageing temperature increased to 650 °C, the ductility (in terms of elongation) increased to 9.3% and the strength remained above 1400 MPa. The ultra-high strength and good ductility of the studied alloy make it a good candidate for a wide range of applications.

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## 1. Introduction

Zirconium (Zr) and Zr alloys are gaining increased attention because of their small neutron absorption area and good corrosion resistance. A series of Zr alloys, from the Zr–Sn [1] and Zr–Nb [2] series to the new Zr–Sn–Nb [3], Zr–Ti [4], and Zr–Mo [5] alloys, among others, have been developed. As nuclear materials, the structure [6,7], deformation behavior [8], and corrosion resistance [9] of Zr alloys have been extensively studied. However, such studies have not focused on improving the mechanical properties of these alloys. To promote a safer and more extensive use, the mechanical properties of Zr-based alloys need to be significantly improved. The commonly used methods for improving the mechanical properties include composition design and structural adjustment. Hsu [10] demonstrated an obvious change in the bending strength as the alloy composition changed from pure Zr to Zr–40Ti. Oh [11] also showed that trace addition of Fe, V, Sb, and/or Mn into the Zr–0.8Sn alloy dramatically improved the alloy hardness. Regarding structural adjustment, Yang [12] reported that the precipitation of the  $\alpha$  phase from the  $\beta$  phase matrix in the  $\text{Ti}_{50}\text{Zr}_{30}\text{Nb}_{10}\text{Ta}_{10}$  alloy resulted in increased hardness. In general,

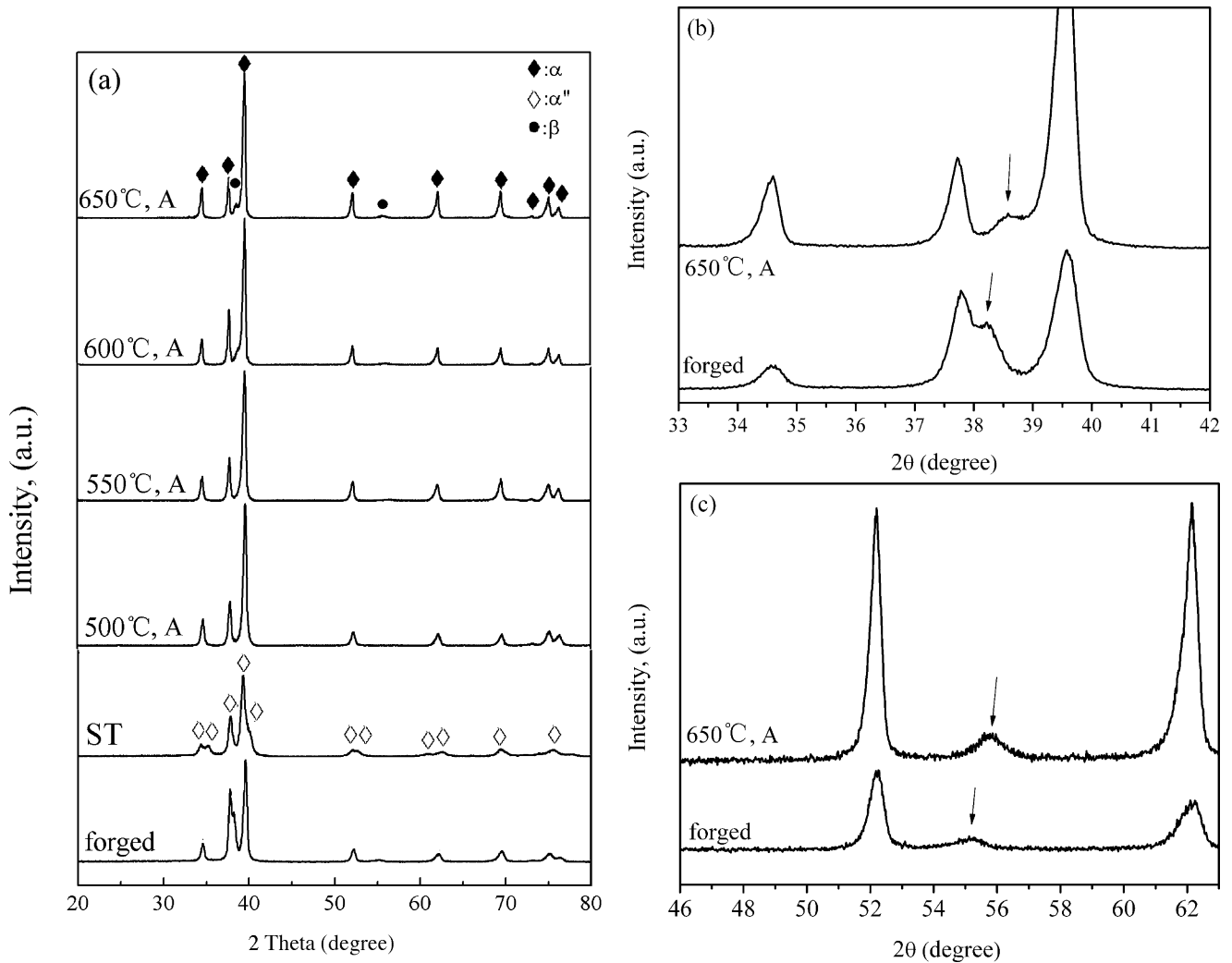
microstructure and mechanical properties can be widely modified via heat treatment [13–15]. Dehghan-Manshadi [16] reported that the size, morphology, volume fraction, and distribution of the  $\alpha$  and  $\omega$  phases in the Ti–5Al–5Mo–5V–3Cr alloy greatly depend on the heat treatment process and affect the alloy hardness. Thus, the effect of heat treatment on the mechanical properties is mainly due to the formation of a wide range of microstructures resulting from the size, morphology, volume fraction, and distribution of the  $\alpha$  phase precipitating in the  $\beta$  phase matrix.

In a previous work by our group [17], a series of ZrTiAlV alloys exhibiting excellent mechanical properties have been obtained in a laboratory environment. In the current paper, a 30Zr–62Ti–5Al–3V alloy was prepared using conventional industrial methods. The obtained alloy exhibited ultra-high strength and good ductility. The effect of heat treatment on the microstructure and mechanical properties was also investigated.

## 2. Experimental procedure

Sponge Zr (Zr + Hf  $\geq$  99.5 wt%), sponge Ti (99.7 wt%), industrially pure Al (99.5 wt%), and V (99.9 wt%) were used to prepare the 30Zr–62Ti–5Al–3V (wt%) alloy. The alloy was melted three times using a vacuum consumable electro-arc furnace to ensure a uniform chemical composition. The ingot underwent multiple breakdowns after being held at 1050 °C above the  $\beta$  transit temperature for

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**Fig. 1.** XRD patterns of forged and heat treated specimens (a) and (b), (c) are partial XRD patterns of forged and 650 °C aged specimens. ST is short for solution treated. A is short for aged.

1.5 h to completely break the coarse grain. The ingot was then held at 930 °C for 1.5 h. The final heat forging was performed, and the ingot was lathed into bars 43 mm in diameter. The bars were sectioned into 10 mm × 10 mm × 70 mm pieces and used as heat-treatment specimens. The heat treatments were performed in a tubular vacuum heat treatment furnace with a protective argon atmosphere and consisted of a solution at 825 °C for 30 min, followed by quenching in water. The ageing treatments were performed at 500, 550, 600, or 650 °C for 4 h. Bone-shaped plate specimens with an original gauge length of 21 mm and a cross-sectional dimension of 3 mm × 2 mm were prepared for the tensile tests.

X-ray diffraction (XRD) was used to determine the crystal structure of the as-forged and heat-treated specimens; optical microscopy (OM) and transmission electron microscopy (TEM) were used to determine the microstructures; and scanning electron microscopy (SEM) was used to analyze the tensile fracture. The TEM specimens were prepared via twin-jet electrochemical polishing in a solution containing 10% perchloric acid and 90% methanol at 13 V and −35 °C. Tensile tests were performed on an Instron 5982 mechanical test system at room temperature at a strain rate of  $5 \times 10^{-4} \text{ s}^{-1}$ . The strain during the entire testing process was monitored using an extensometer with a 12.5 mm gauge length.

### 3. Results and discussion

#### 3.1. Phase transformation

Fig. 1 shows the XRD patterns of the forged and heat-treated specimens exhibiting the phase composition of the 30Zr–62Ti–5Al–3V alloy under different conditions. The XRD pattern of the forged specimen consists of  $\alpha$  phase peaks as well as obvious  $\beta$  phase (1 1 0) and (2 0 0) peaks near 38° and 55°, respectively. When the alloy was solution-treated at 825 °C for 30 min followed by water quenching, typical  $\alpha''$  phase peaks [18–20] clearly appeared in the XRD pattern. Cheng [21] also reported that the addition of Zr into a Ti alloy promotes orthorhombic martensite  $\alpha''$  phase formation. Generally, when Zr or Ti alloys are quenched from a two-phase or  $\beta$  phase temperature region, three kinds of martensite phases containing different kinds of structures, namely, a hexagonal close-packed structure ( $\alpha'$  phase), an orthorhombic structure ( $\alpha''$  phase), and a distorted hexagonal close-packed structure ( $\omega$  phase), can appear in order as the  $\beta$  phase stability increases. The appearance of the  $\alpha''$  phase in the solution-treated specimen indicates that the  $\beta$  phase of the 30Zr–62Ti–5Al–3V alloy is more stable than that of the Ti–6Al–4V alloy, in which only the  $\alpha'$  phase was detected after quenching from the  $\alpha + \beta$  two-phase or  $\beta$  phase temperature region. The martensite phase in the Zr

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