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## Influence of Mn addition on the microstructure and mechanical properties of a directionally solidified  $γ$ -TiAl alloy



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### ABSTRACT

TiAl alloys with nominal compositions of Ti-47Al-2Nb-2Cr-(0, 1, 3, 5)Mn were prepared via electromagnetic cold crucible directional solidification (ECCDS), and the influence of Mn addition on the microstructure and mechanical properties of the alloys was investigated. The experimental results showed that the Mn atoms were dissolved within the TiAl alloy, and no Mn compounds were formed. Based on the X-ray diffraction (XRD) analysis results, the peak angle of the Mn-containing sample shifted to a higher value, indicating lattice shrinkage. The Mn addition also increased the volume fraction of the B2 phase. The yield strength of the TiAl alloy monotonically increased as the content of the substitutional-solute Mn atoms increased. The ultimate strengths of the TiAl alloys containing 1 at.%, 3 at.%, and 5 at.% Mn consequently decreased because microcracks were preferentially formed in the B2 phase during room-temperature deformation. The introduction of substitutional-solute Mn atoms could result in a reduction of the stacking-fault energy of the γ phase, and facilitate the operation of twinning-controlled plastic-deformation during the intermediate stage of the plastic deformation process. The interaction between the deformation twins and movable dislocations led to a continuous increase in the work-hardening rate.

#### 1. Introduction

TiAl-based alloys have attracted extensive attention because of their low density, high specific strength, and excellent oxidation and creep resistance [1–[4\]](#page--1-0). They are regarded as the most promising structural materials with regard to the replacement of Ni-based superalloys used for weight-saving applications over the temperature range of 650–750 °C [[5](#page--1-1),[6](#page--1-2)]. Referring to Ref [\[5\]](#page--1-1), Chen and his co-workers have successfully prepared TiAl single crystals with controlled lamellar orientations and the room temperature tensile ductility, elevated strength at 900 °C and creep resistance have been significantly improved. This TiAl single-crystal alloy has provided expanded opportunities for higher-temperature applications. However, the inherent disadvantages of intermetallic compounds, such as poor fracture toughness etc., still restrict the extensive use of TiAl alloys in the aviation industry [\[7\]](#page--1-3). The mechanical properties of TiAl alloys depend on their microstructures, and over the past few decades many methods have been applied to modify the microstructure of the TiAl alloy. Yang et al. has reported that the solidification-segregation and β-segregation are considerably reduced by controlled post-solidification isothermal holding and cooling [\[8\]](#page--1-4). They also find that a novel method termed mushy zone cooling control can effectively tailor the microstructure [[9](#page--1-5)]. In addition,

numerous studies have been conducted in the fields of solidification technology and composition design to control the microstructures and improve the mechanical properties of γ-TiAl alloys  $[10-13]$  $[10-13]$ . Directional solidification (DS) is an effective technique, which is used to improve the strength and ductility of TiAl alloys with  $(α<sub>2</sub> + γ)$  lamellae [\[14](#page--1-7)–16]. The columnar grains and lamellar structures of DS TiAl alloys grow unidirectionally. TiAl alloys exhibit an excellent combination of strength and ductility when the tensile axis is parallel to the lamellar interface [\[17](#page--1-8)]. In contrast to the conventional DS technology, the electromagnetic cold crucible directional solidification (ECCDS) method, used in the present study, does not require a ceramic crucible. Slight contact exists between the molten TiAl and copper crucible because of the constraint effect of the electromagnetic force. ECCDS technology can be used to prevent contamination owed to oxide particles, such as  $Y_2O_3$ , and CaO [18-[20\]](#page--1-9). This is of great benefit with regard to the purification of the TiAl melt and the improvement of the mechanical properties of the resultant material [[21\]](#page--1-10). In addition to the continuous development of solidification technology, alloying is also considered an effective method with regard to improving the performance of TiAl alloys [\[22](#page--1-11)–25]. By considering phase stabilization, site occupation, and the gamma-to-alpha volume fraction, there have been various efforts to determine why alloying elements result in

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strengthening and ductilizing [\[26](#page--1-12)]. Mn is proven to be an efficient alloying element with regard to improving the mechanical properties of TiAl alloys [27–[29\]](#page--1-13). Researchers have reported that adding Mn into TiAl alloy could make the  $c/a$  ratio of  $L1_0$  structure closer to 1 from 1.02 and improve the symmetry of  $L1_0$  structure, thus leading to better room temperature ductility. However, there is still a lack of research regarding the influence of Mn addition on the strength of TiAl alloys. In the present study, we will compare the microstructural evolution and mechanical properties of DS Mn-free and Mn-containing TiAl alloys to elucidate the strengthening mechanism owed to the addition of Mn.

#### 2. Experimental Procedures

Alloys with nominal compositions of Ti-47Al-2Cr-2Nb-(0, 1, 3, 5) Mn, termed TNC-xMn  $(x = 0, 1, 3, 5)$ , were prepared via ECCDS in the present study. Unless otherwise mentioned, alloys contents were shown in at.%. The alloys were solidified with a growth rate of 1.0 mm/min at a constant temperature gradient of 25 K/mm. The details of the ECCDS technology could be found in Ref [\[30](#page--1-14)]. The ECCDS ingots were cut in two along axial direction, followed by grinding, polishing and etching. The macrostructures were composed of columnar grains which were continuous and aligned along the axial direction of the ingots. The samples for microstructure observation and performance testing were cut from the same position of each ingot. According to the previous study [\[31](#page--1-15)], the performances of TiAl alloys strongly depended on the lamellar orientation. In the present paper, the influence of solidification parameters on lamellar orientation could be neglected because the growth rate and temperature gradient were the same. Then the lamellar orientation depends on the chemical composition of the alloy. The lamellar orientations of the four alloys were counted through metallographic observation and statistical results were displayed in [Fig. 1](#page-1-0). It could be seen that introduction of Mn atoms showed little influence on the lamellar orientation.

X-ray diffraction (XRD) with CuK<sub>α</sub> radiation ( $\lambda = 1.54056$  Å) was used to determine the phase constitution of the experimental alloys. Scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS) and transmission electron microscope (TEM) were employed to investigate microstructure. The samples for SEM observations were cut from the ingots by wire cut electrical discharge machining (WEDM). The samples were then prepared by standard metallographic procedures involving grinding by emery paper and electro-polishing in a solution of 5 vol% perchloric acid, 30 vol% Nbutanol and 65 vol% methanol. TEM samples were manually ground to approximately 50 μm, followed by cutting into wafers with a diameter of 3 mm. Finally, the foils were ion-milled with 3.6 keV Ar ions using a Gatan PIPS until electron transparency occurred.

The samples for compression tests were cut along the growth direction. According to the standard test methods of compression testing of metallic materials at room temperature (ASTM E9-89a(2000)), the size of the compression samples was  $\Phi$ 6 mm × 9 mm. Prior to compressive testing, the two ends of the samples were ground to keep parallel. Room temperature compressions were carried out on the Instron 5569 machine and the strain rate was  $1.0 \times 10^{-3}$  s<sup>-1</sup>. The mean value of compression strength was derived from 3 individual compression tests. In order to characterize the microstructural evolutions during compression, the samples for SEM observations were cut along the compression axis.

#### 3. Results and Discussion

#### 3.1. Phase Constitution

The phase constitutions of the Ti-47Al-2Nb-2Cr-(0, 1, 3, 5)Mn alloys were characterized using XRD. [Fig. 2](#page--1-16) shows the resultant XRD patterns. In the cases of both the Mn-free and Mn-containing alloys, the Bragg diffraction peaks of the  $\alpha_2$ -Ti<sub>3</sub>Al and γ-TiAl phases can be observed, while in the case of the Mn-containing samples, no Bragg diffraction peaks attributed to the Mn-containing phases can be observed. The XRD results indicate that the incorporation of Mn does not change the phase constitution of the TiAl alloys; in addition, no Mn compounds are formed. However, by comparing the diffraction pattern of the Mn-free sample with that of the Mn-containing sample, it can be observed that the corresponding diffraction peak positions of the  $\gamma$  phase vary with the Mn addition. [Fig. 2\(](#page--1-16)b) and (c) show magnified images of the corresponding peak positions of the  $(110)_{\gamma}$  and  $(111)_{\gamma}$  planes of the four samples. The diffraction peaks shift to higher 2θ values as the Mn content is increased. According to the Bragg diffraction equation  $2d \sin \theta = n\lambda$  (where d means interplanar spacing,  $\theta$  is the diffraction angle,  $\lambda$  means the wavelength of the X-ray and *n* is integer), the interplanar spacing is inversely proportional to the diffraction peak angle (θ). The increase in 2θ value indicates a reduction in the interplanar spacing.  $\gamma$  (TiAl) phase has the L1<sub>0</sub>-structure (in f.c.t. notation). The top and bottom layers of an f.c.t. unit cell are occupied by Al atoms and Ti atoms reside on the remaining face center, forming a Ti-layer at  $Z = 0.5$ . The change in the interplanar spacing is closely related to the occupancy of Mn atoms. According to a previous study, Mn atoms preferentially substitute the Al sublattice sites of the  $\gamma$  phase. Since the atomic radius of Mn is smaller than that of Al ( $r_{\text{Mn}} = 0.127$  nm and  $r_{\text{Al}}$  = 0.143 nm), lattice contraction can occur when Mn atoms occupy Al sublattice sites.

<span id="page-1-0"></span>

Fig. 1. (a) Metallographic images of the lamellae, (b) statistical results of lamellar orientation.

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