Dependency of microstructure and microhardness on withdrawal rate of Ti-43Al-2Cr-2Nb alloy prepared by electromagnetic cold crucible directional solidification

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Abstract: The intermetallic Ti-43Al-2Cr-2Nb (at.%) alloy was directionally solidified in an electromagnetic cold crucible with different withdrawal rates (V) ranging from 0.2 to 1.0 mm·min⁻¹, at a constant temperature gradients ($G=18\text{ K}\cdot\text{mm}^{-1}$). Macrostructures of the alloy were observed by optical microscopy. Microstructures of the alloy were characterized by scanning electron microscopy (SEM) in back-scattered electron mode and transmission electron microscopy. Results showed that morphologies of macrostructure depend greatly on the applied withdrawal rate. Continuous columnar grains can be obtained under slow withdrawal rates ranging from 0.2 to 0.6 mm·min⁻¹. The microstructure of the alloy was composed of α_2/γ lamellar structures and a small number of mixtures of B2 phases and blocky γ phases. The columnar grain size (d) and interlamellar spacing (λ) decrease with an increasing withdrawal rate. The effect of withdrawal rate on microhardness was also investigated. The microhardness of the directionally solidified Ti-43Al-2Cr-2Nb alloy increases with an increase in withdrawal rate. This is mainly attributed to the increase of B2 and α_2 phases as well as the refinement of lamellae.

Key words: TiAl-based alloys; directional solidification; microstructure; microhardness

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TiAl-based alloys recently have been attracting tremendous attention due to their low density, high specific strength, high strength at elevated temperatures, and excellent high temperature burn resistance^[1-3]. Therefore, it has been considered as an attractive candidate material to replace nickel-based superalloys, titanium alloys and other high temperature alloys at the intended service temperature of 600–900 °C. The full lamellar (FL) microstructure in various well-developed TiAl alloys has been widely studied, due to its combination of good toughness at ambient temperature and high strength at elevated temperatures^[4,5]. Furthermore, results deriving from investigations on polysynthetically twinned crystals (PST) revealed excellent room temperature

ductility when the lamellar boundary orientation was parallel or inclined to the loading axis^[6,7]. During the past two decades, extensive directional solidification experiments were carried out to achieve columnar grain with preferred lamellar orientation^[8,9]. Therefore, these studies have focused on the control of lamellar orientation by optimizing solidification parameters during directional solidification of TiAl alloys. However, very little attention has been paid to microstructural characteristics and mechanical properties.

Generally, directional solidification experiments were performed in ceramic mould materials, such as Al₂O₃, Y₂O₃ or CaO^[10-12]. To the best of our knowledge, no refractory materials completely resistant to molten titanium alloy have been developed so far. Furthermore, long term interaction between the melt and ceramic mould due to low withdrawal rates can result in contamination of the melt by ceramic inclusions. These ceramic inclusions have great harm to ductility of directionally solidified (DS) TiAl alloys. When the oxygen content increases from 0.03at.% to 0.08at.%, the elongation decreases from 2.7% to 1.9%^[13]. In order to eliminate the contaminants from

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ceramic crucible, an electromagnetic cold crucible directional solidification technique was developed^[4,8,14]. In this way, the cold crucible directionally solidified (CCDS) TiAl alloy ingots with well controlled lamellar microstructure and lower oxygen content (<200 wt. ppm) were obtained.

The purpose of this research was to study the microstructure and mechanical properties of the Ti-43Al-2Cr-2Nb alloy prepared by electromagnetic cold crucible directional solidification. A detail microscopy study was performed to characterize the microstructure, and the influence of withdrawal rate on microhardness was discussed.

1 Experimental procedure

A master ingot with nominal composition of Ti-43Al-2Cr-2Nb (in at.%) was prepared by placing pure Ti (99.99wt.%), pure Al (99.99wt.%), Al-75wt.%Nb master alloy and pure Cr (99.9wt.%) in a vacuum cold crucible induction melting furnace under argon atmosphere. Master feeder ingots applied for electromagnetic cold crucible directional solidification were cut from the above cylinder ingot into rods with 20 mm in diameter and 200 mm in length by wire electrical discharge machining. The procedure of directional solidification of TiAl-base alloys using a water-cooling electromagnetic cold crucible was described in detail elsewhere^[4,8]. Before directional solidification, the chamber of the furnace was evacuated down to 5×10^{-3} Pa, and then was back-filled with high purity argon to prevent the evaporation of aluminum. CCDS experiments were performed at a constant electric supply power (P) of 50 kW and withdrawal rates (V) varying in the range from 0.2 to $1.0 \text{ mm} \cdot \text{min}^{-1}$. In this case, the alloy was heated to the temperature of $1,710^\circ\text{C}$ and thermally stabilized for 10 min. The temperature gradient (G) in the ingots under different withdrawal rates was measured by PtRh30-PtRh6 thermocouples, and measuring method was described in Ref. [4, 8]. The G value was $\sim 18 \text{ K} \cdot \text{mm}^{-1}$. After directional solidification, the oxygen content of ingots was measured with a LECO TC-436 N/O apparatus as described in Ref. [7, 9]. The test results of the oxygen content were lower than 380 wt. ppm.

After directional solidification, the longitudinal and transverse sections of the specimens were cut, polished and etched using a solution of 5% HF, 5% HNO_3 and 90% H_2O for further analysis. The phases were identified by a Rigaku D/max-RB X-ray diffractometer with monochromatic Cu-K α radiation. Macrostructures of the alloy were observed by optical microscopy. Microstructures were characterized by scanning electron microscopy (SEM) in back-scattered electron (BSE) mode and transmission electron microscopy (TEM). TEM was used to observe different phases at higher magnifications and to determine their individual crystallographic orientations. Thin TEM foils were prepared by mechanical grinding and polishing, followed by dimpling to about $25 \mu\text{m}$ in the central part of the 3 mm disc. Finally, the foils were ion-milled with 3.6 keV Ar ions using a Gatan PIPS until electron transparency occurred. Grain size and volume fractions of α_2 , γ and B2 phases, as well as the interlamellar spacing were statistically measured by a quantitative metallography method.

Microhardness measurements were made with a standardized Vickers measuring test device using a load of 100 g and a loading duration of 10 s on the surfaces of longitudinal section with polished and slightly etched surfaces. Column compression specimens with dimensions of $\Phi 3 \text{ mm} \times 4.5 \text{ mm}$ were cut from CCDS ingot, and the compression axis was chosen to be parallel to the growth direction of the ingots. Statistical evaluation of all compression specimens revealed that the mean angles between lamellar orientation and the compression axis varied in the range $0-18^\circ$. The uniaxial compression tests with an initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ were carried out using an Instron 5569 testing machine in air.

2 Results and discussion

2.1 Macrostructure

Figure 1 illustrates the longitudinal sectional macrostructures of the Ti-43Al-2Cr-2Nb alloy with withdrawal rates of 0.2, 0.4, 0.6, 0.8 and $1.0 \text{ mm} \cdot \text{min}^{-1}$, respectively. Morphologies of

Fig. 1: Optical macrostructures of CCDS Ti-43Al-2Cr-2Nb ingots in longitudinal-section under different withdrawal rates ($\text{mm} \cdot \text{min}^{-1}$) of 0.2 (a), 0.4 (b), 0.6 (c), 0.8 (d) and 1.0 (e)

columnar grains depend greatly on the applied withdrawal rate. From Figs.1 (a)–(c), it can be seen that the long and continuous column grains almost aligned erectly along the growth direction, indicating that the well directionally solidified macrostructure could be achieved at withdrawal rates form 0.2 to 0.6 $\text{mm}\cdot\text{min}^{-1}$. By comparison, the CCDS samples at 0.8 and 1.0 $\text{mm}\cdot\text{min}^{-1}$ have much shorter columnar grains in length, and exhibit discontinuous morphology in Figs. 1(d) and (e). Previous studies

have proved that columnar grains inclined to the longitudinal axis under a higher withdrawal rate, and grains growth was easily disturbed^[16]. So, to obtain continuous columnar grains, applying a slow withdrawal rate is highly necessary and desirable for CCDS processing. Figure 2 shows the transverse sections of CCDS Ti-43Al-2Cr-2Nb ingots. The columnar grain size (d) decreased averagely from 5.8 to 2.3 mm when the withdrawal rate increased from 0.2 to 1.0 $\text{mm}\cdot\text{min}^{-1}$.

Fig. 2: Optical macrostructures of CCDS Ti-43Al-2Cr-2Nb ingots in transvers-section under different withdrawal rates ($\text{mm}\cdot\text{min}^{-1}$) of 0.2 (a), 0.4 (b), 0.6 (c) and 0.8(d)

2.2 Microstructure

The CCDS Ti-43Al-2Cr-2Nb alloy is characterized by α_2/γ lamellar structure and band-like constituent that consists of dual phases concerning bright B2 and grey blocky γ phases, embedding in the lamellar structure, as shown in Fig. 3(a). Figure 3(b) shows the TEM images of α_2/γ lamellar microstructure and the mixture of B2 and γ phase of Ti-43Al-2Cr-2Nb alloy. The morphology of the mixture is almost the same as observed by SEM. The bright B2 phase and grey γ phase in back-scattered electron

images were further identified by subsequent EDS analysis. The EDS results are listed in Table 1. As compared with the blocky γ phase and lamellar matrix, the B2 phase usually contains much higher concentrations of Ti and Cr but lower Al, which can explain the relatively bright contrast of the B2 phase in Fig. 3(a). Meanwhile, EDS analysis provided the evidence that Cr is tendentiously enriched in the B2 phase rather than that in the lamellar phases, confirming that Cr dissolves easily into β phase (B2 phase) during the solidification process.

Fig. 3: SEM (a) and TEM (b) images of CCDS Ti-43Al-2Cr-2Nb alloy

Table 1: EDS results of constituents of CCDS Ti-43Al-2Cr-2Nb

Constituent	Composition (at.%)			
	Ti	Al	Cr	Nb
B2	55.71	34.82	6.16	3.31
γ	45.8	49.8	1.56	2.84
Lamellar (α_2/γ)	52.79	42.58	1.86	2.77

The withdrawal rate exerts significant effects on the volume fractions of the constituting phases and lamellar spacing, as shown in Fig. 4. It can be seen that increasing withdrawal rate resulted in an increase of B2 phase and α_2 phase, but a

decrease of γ phase and lamellar spacing. It is known that the transformation of β to α phase is often an incomplete transformation, which is attributed to β phase stabilizers such as Cr and Nb. The residual β phase is subsequently transformed into the B2 phase through ordering transformation. In this study, withdrawal rate directly affected the cooling rate and transformation time. Therefore, the higher the withdrawal rate, the shorter the necessary transformation time from β to α , and the larger the volume fractions of residual β phases (B2 phase)^[15]. Similarly, the decomposition of the high-temperature α phase to lamellar structure also depends strongly on the withdrawal rate. Under higher withdrawal rate, the residual α phase (α_2 phase) was relatively larger and γ phase did not have enough time to thicken, resulting in the refinement of the α/γ lamellae.

Fig. 4: Dependence of volume fraction of α_2 (a), γ (b), B2 (c) phases and lamellar spacing (d) on withdrawal rate

2.3 Microhardness

The value of microhardness (HV) provides an indication of resistance to deformation, densification and cracking. It is often used to evaluate the mechanical properties of metal materials. The influence of withdrawal rate on the microhardness of CCDS Ti-43Al-2Cr-2Nb alloy is shown in Fig. 5. As the withdrawal rate increased from 0.2 to 1.0 mm·min⁻¹, the HV value increased from 438 MPa to 461 MPa, the highest value. The variation of HV was mainly attributed to the change of phases and interlamellar spacing. Firstly, as reported in references [16,17], B2 phase and α_2 phase are harder than the γ phase. In this case, increase of B2 phase and α_2 phase would result in the increase of Vickers microhardness of CCDS Ti-43Al-2Cr-2Nb alloy. Secondly, the refined lamellae could harden the alloy, because more α_2/γ phase interfaces would be facilitated

Fig. 5: Dependence of Vickers microhardness on withdrawal rate

in restricting dislocation movement and resisting local plastic deformation, hence resulting in higher hardness values.

3 Conclusions

In the present research, the dependency of macrostructure, microstructure and microhardness on the withdrawal rate of directionally solidified Ti-43Al-2Cr-2Nb alloy was investigated. The following conclusions are obtained:

(1) Morphologies of macrostructure depend greatly on the applied withdrawal rate. Continuous columnar grains can be obtained under slow withdrawal rates ranging from 0.2 to 0.6 mm•min⁻¹. The columnar grain size (*d*) decreases averagely from 5.8 to 2.3 mm when the withdrawal rate increases from 0.2 to 1.0 mm•min⁻¹.

(2) With increasing withdrawal rate, the interlamellar spacing and volume fraction of γ phase decrease, but the volume fractions of α_2 and B2 phase increase.

(3) The microhardness increases with increasing withdrawal rate. This is mainly attributed to the increasing of B2 phase and α_2 phase and the refinement of lamellae.

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