

Investigation of microstructure evolution and mechanical properties of cold crucible directional solidification Nb-Si alloy

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Abstract. In this work, the microstructure and properties of Nb-16Si-24Zr alloy directionally solidified by electromagnetic cold crucible under different process parameters were investigated. As the pulling rate increases, the size of both primary Nbss and eutectic Nbss decreases, resulting in a straighter primary dendrite γ -Nb₅Si₃ and a fishbone like Nbss/ γ -Nb₅Si₃ coupling structure of secondary eutectic. As the power supply increases, the size of the nascent Nbss and γ -Nb₅Si₃ increases, and the coupling growth trend of Nbss and Nb₅Si₃ weakens. As the pulling rate and power supply increase, the toughness shows a trend of first increasing and then decreasing, while the room temperature compressive strength gradually increases.

1 Introduction

The use of directional solidification technology is an effective means to improve the mechanical properties of Nb-Si based alloys [1-3]. At present, relevant research has been conducted on several directional solidification technologies. Bewlay et al. have applied the vertical pulling method to prepare Nb-Si alloys [4-6], and the fracture toughness is about 50% higher than that of arc melted Nb-Si alloys. Li et al. investigated the microstructure and mechanical properties of Nb-Si Mo alloy prepared by directional solidification through optical floating zone (OFZ) [7]. Although these directional techniques significantly improve the mechanical properties of Nb Si based alloys, it is still necessary to optimize the process flow. The pulling rate and power supply have a significant impact on the microstructure and properties of Nb-Si based alloys. The pulling rate and power supply not only affect the microstructure size, but also the morphology of the eutectic, and these factors correspondingly determine the mechanical properties [8-10]. In order to elucidate the strengthening mechanism of directional solidification microstructure, more details about the structure varying with the pulling rate and temperature need to be clarified.

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In this work, the microstructure and properties of Nb-16Si-24Zr alloy directionally solidified by electromagnetic cold crucible under different process parameters were investigated. The directional growth of Nb-16Si-24Zr during directional solidification in an electromagnetic cold crucible was analyzed based on the power supply and pulling rate.

2 Experimental procedure

The alloys with a nominal composition of Nb-16Si-24Zr (at.%) were prepared by vacuum non consumable arc melting in a water-cooled copper crucible. Cut a rod with a diameter of 13 mm and polish it with a grinding wheel to remove the oxidized surface. Directional solidification is carried out using an electromagnetic water-cooled copper crucible. Directionally solidified alloys are prepared at different temperatures (48-64 kw) and pulling speeds (0.1-0.4 mm/min).

The microstructure of the alloy was analyzed using the BSE-SEM (Merlin Compact). The single-sided notch bending test was conducted on the Instron 5569 universal electric testing machine to obtain the fracture toughness. The size of the test sample is $20 \times 4 \times 2$ mm³, with a notch depth of 2 mm. The fracture toughness K_Q value is calculated using the formula mentioned in reference [11]. The fracture toughness test of each alloy shall be conducted at least 3 times at a loading rate of 0.2 mm/min. The specimens with dimensions of $\Phi 4$ mm \times 6 mm were cut by EDM for room temperature compression test, respectively. The room temperature compression tests were carried out on the electronic universal testing machine (Shimadzu, AGX-plus) with a loading rate of 0.2 mm/min.

3 Results and discussion

3.1 Directionally solidified microstructure and primary phase selection

Fig. 1 shows the directional solidification microstructure of Nb-Si alloy with different process parameters and shapes of cold crucibles. It can be seen that the crucible has a good overall orientation, indicating a good overall orientation. The purpose of directional solidification is to obtain directionally grown tissues, thereby improving the unidirectional mechanical properties of materials. However, in the early stage of directional solidification, the directional solidification structure cannot be obtained. There always exists a paste like zone from the original structure of the mother material to the directional solidification structure. For the convenience of research, the paste zone and directional solidification were analyzed separately.

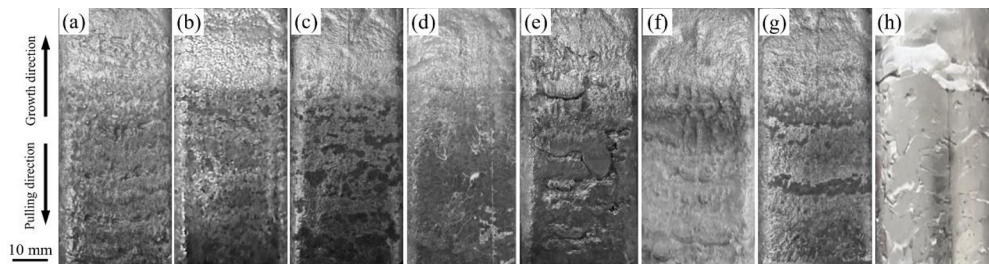


Fig. 1. Directional solidification microstructure of Nb-Si alloy with different process parameters and different shapes of cold crucibles: (a-g) circular crucible; (h) Square crucible; (a) 48 kw, 0.1 mm/min; (b, h) 48 kw, 0.2 mm/min; (c) 48 kw, 0.4 mm/min; (d) 48 kw, 0.8 mm/min; (e) 40 kw, 0.4 mm/min; (f) 56 kw, 0.4 mm/min; (g) 64 kw, 0.4 mm/min.

Fig. 2 shows the paste like microstructure of Nb-Si alloy with different process parameters and different shapes of cold crucibles. The paste like microstructure is retained at room temperature due to sudden power outage during directional solidification. As shown in Fig. 2(a), its primary phase is Nb_5Si_3 phase, which exhibits small planar growth characteristics, resulting in a more chaotic microstructure in the paste like area without obvious dendritic features. When the growth rate increases to above 0.4 mm/min, the paste like zone exhibits obvious dendritic growth characteristics. However, when the power reaches 64 kW, its primary phase transforms again into Nb_5Si_3 phase, as shown in Fig. 2(g). The power supply and pulling rate affect the selection of primary phases in the directional solidification process. The phase selection trend of Nb-16Si-24Zr alloy directionally solidified in a cold crucible is shown in Fig. 3. Under low pulling rate conditions, the solidified primary phase is Nb_5Si_3 . As the pulling rate increases, the primary phase undergoes a transformation into Nbss phase. The temperature gradient has a smaller impact on the primary phase compared to the pulling rate. However, under the condition of a pulling rate of 0.4 mm/min, as the power supply increases sufficiently high, the primary phase will transform back into Nb_5Si_3 phase.

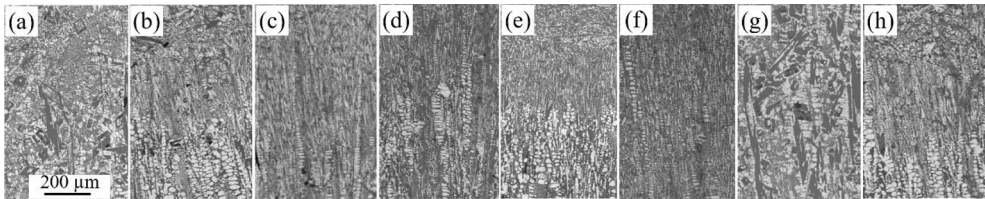


Fig. 2 Paste like microstructure of Nb-Si alloy with different process parameters and shapes of cold crucibles: (a-g) circular crucible; (h) square crucible; (a) 48 kW, 0.1 mm/min; (b, h) 48 kW, 0.2 mm/min; (c) 48 kW, 0.4 mm/min; (d) 48 kW, 0.8 mm/min; (e) 40 kW, 0.4 mm/min; (f) 56 kW, 0.4 mm/min; (g) 64 kW, 0.4 mm/min.

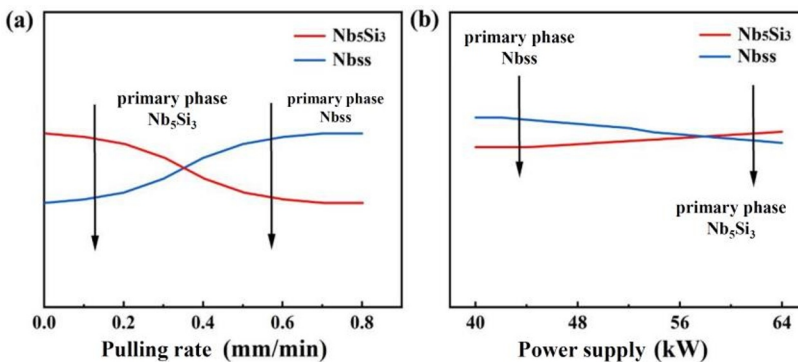


Fig. 3. Phase selection of directionally solidified Nb-Si alloy.

Fig. 4 shows the microstructure of Nb-16Si-24Zr alloy directionally solidified in cold crucibles with different process parameters and shapes. The directional solidification ingot prepared by a circular cold crucible is shown in Fig. 4(a-g). When the power supply is 48 kW and the pulling rate is 0.1 mm/min (Fig. 4), the Nbss phase and Nb_5Si_3 phase in the tissue grow in the opposite direction of the heat dissipation temperature, showing a directional growth trend. As the pulling rate increases, the size of both primary Nbss and eutectic Nbss decreases; The aspect ratio of Nbss and $\gamma-Nb_5Si_3$ both increased, and finally a relatively straight primary dendrite $\gamma-Nb_5Si_3$ appeared. Coupled growth of Nbss and $\gamma-Nb_5Si_3$. In addition, secondary dendrite A appeared around some primary dendrites Nb_5Si_3 , presenting

a fishbone like Nbss/ γ -Nb₅Si₃ eutectic structure around the primary dendrites. When the pulling rate reaches 0.8 mm/min, as shown in Fig. 4(d), the primary dendrite spacing decreases while the fishbone like eutectic structure increases. There are two obvious eutectic structures present in the organization: first, the coupling growth of primary dendrite γ -Nb₅Si₃ and primary Nbss; The second is the alternating arrangement of secondary dendrites γ -Nb₅Si₃ and Nbss along the primary dendrite γ -Nb₅Si₃. The increase in pulling rate leads to an increase in temperature gradient at the front of the solid-liquid interface and a decrease in melt temperature, which is not conducive to atomic diffusion. The coupled growth of Nbss and γ -Nb₅Si₃ depends on the vibration frequency and diffusion rate of Nb atoms, Nb atomic clusters, and Nb+Si atomic clusters in the melt, and both vibration frequency and diffusion rate are closely related to the temperature gradient at the solid-liquid interface front and the temperature of the melt. At a higher pulling rate, the solidification cooling increases, and the longitudinal growth trend of the alloy increases, while suppressing lateral growth, resulting in a decrease in the axial spacing of primary dendrites. When the pulling rate is 0.4 mm/min, as the power supply increases, as shown in Fig. 4(b, e-g), the size of the nascent Nbss and γ -Nb₅Si₃ increases; The coupling growth trend of Nbss and Nb₅Si₃ weakens. In addition, the fishbone like eutectic tissue gradually disappears with the increase of power supply.

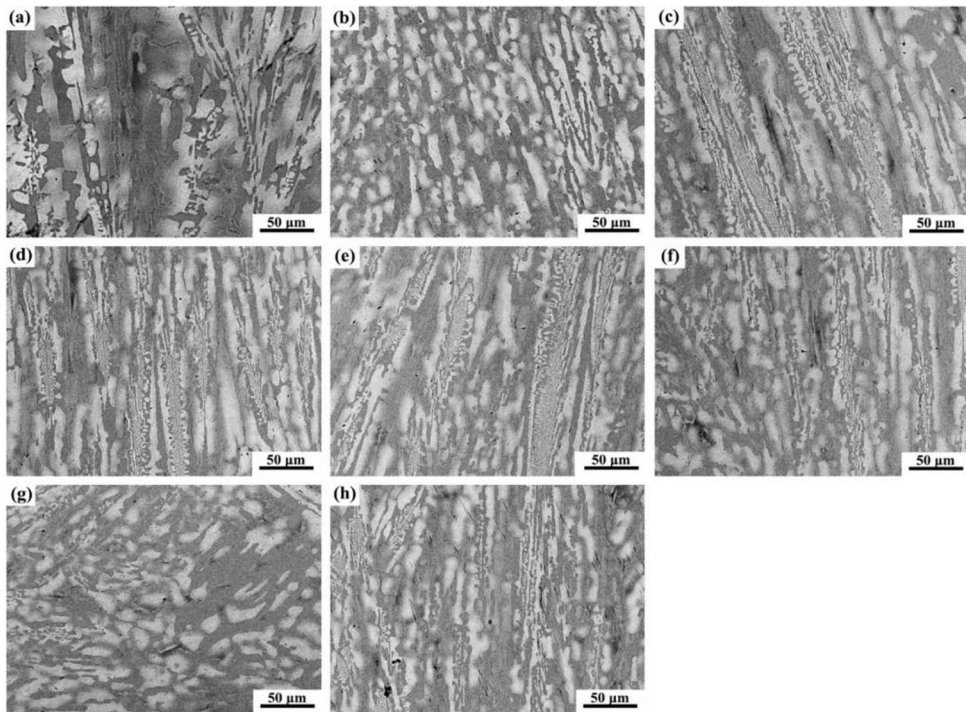


Fig. 4. Directional solidification microstructure of the alloys: (a-g) circular crucible; (h) Square crucible; (a) 48 kW, 0.1 mm/min; (b, h) 48 kW, 0.2 mm/min; (c) 48 kW, 0.4 mm/min; (d) 48 kW, 0.8 mm/min; (e) 40 kW, 0.4 mm/min; (f) 56 kW, 0.4 mm/min; (g) 64 kW, 0.4 mm/min.

3.2 Mechanical properties of directionally solidified alloys

Fig. 5 shows the room temperature fracture toughness test results of directionally solidified alloys. When the power supply is 48 kW and the pulling rate is 0.1 mm/min, the room temperature fracture toughness value (K_{Ic}) of the directionally solidified alloy is 15.84 MPa·m^{1/2}. As the pulling rate increases, the K_{Ic} of directionally solidified alloys shows a trend

of first increasing and then decreasing. When the pulling rate is 0.4 mm/min, the K_{IQ} of the directionally solidified alloy reaches its optimal value of $22.35 \text{ MPa}\cdot\text{m}^{1/2}$. This is mainly attributed to the reduction of the spacing between primary dendrites under higher pulling rates, and the alternating arrangement of Nbss/ γ -Nb₅Si₃ coupling is conducive to hindering crack propagation, which improves the room temperature toughness of directionally solidified alloys. However, at excessively high pulling rates, the number of fishbone-like structures in the secondary eutectic structure increases, and secondary dendrite A penetrates into the Nbss phase perpendicular to the primary dendrite axis, providing a fast channel for crack propagation, leading to a decrease in the room temperature toughness of directionally solidified alloys. When the pulling rate is 0.4 mm/min, with the increase of power supply, the room temperature toughness of the alloy also shows a trend of first increasing and then decreasing. As the power increases, the axial temperature gradient of the directionally solidified alloy increases, which is beneficial for the directional growth of Nbss and γ -Nb₅Si₃. However, at excessively high temperatures, it can cause thermal equilibrium and solute redistribution, interfere with the directionally grown structure, weaken the coupling degree between Nbss and γ -Nb₅Si₃, and is not conducive to the room temperature toughness of directionally solidified alloys.

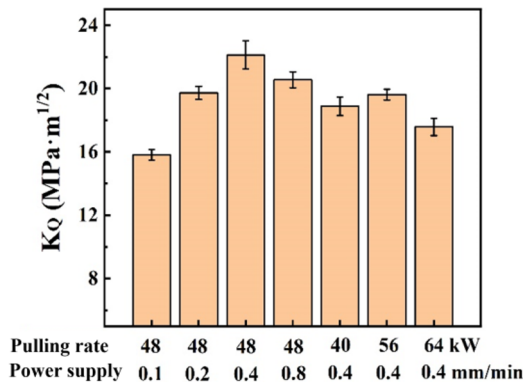


Fig. 5. Room temperature fracture toughness of directionally solidified alloys.

The room temperature compression performance test results of directionally solidified alloys are shown in Fig. 6, with the compression direction perpendicular to the directional growth direction. When the directional solidification conditions are power supply of 48 kW and pulling rate of 0.1 mm/min, the room temperature compressive strength of the directional solidification alloy reaches 2105 MPa. As the pulling rate increases, the room temperature compressive strength of the alloy gradually increases. When the pulling rate is 0.4 mm/min, the room temperature compressive strength of the directionally solidified alloy reaches 2278 MPa. This is attributed to the effect of a decrease in the axial spacing of primary dendrites, and an increase in the aspect ratio of primary eutectic is beneficial for the room temperature compressive strength of directionally solidified alloys. As the power supply increases, the room temperature compressive strength of directionally solidified alloys gradually decreases. When the power supply is 48 kW, the room temperature compressive strength of the directionally solidified alloy reaches 2123 MPa. Coupled arrangement of primary eutectic Nbss/ γ -Nb₅Si₃ is beneficial for increasing compressive strength, while the increase in power supply weakens the coupling effect, leading to a decrease in room temperature compressive strength. When the power supply is 48 kW and the pulling rate is 0.8 mm/min, the compressive strength of directionally solidified alloys prepared by circular and square crucibles is both around 2200 MPa.

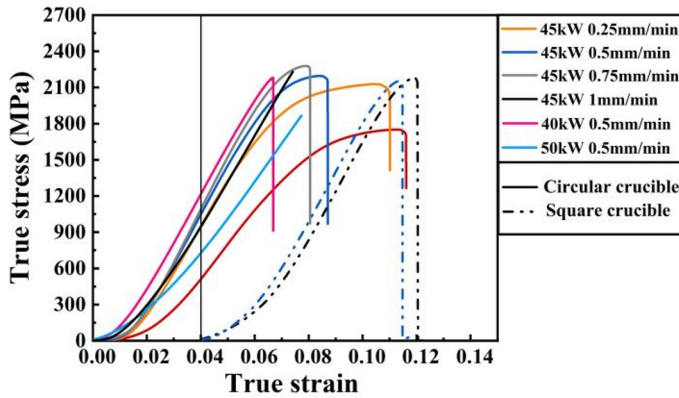


Fig. 6. Room temperature compression of directionally solidified alloys.

4 Conclusion

- 1) The size of both primary Nbss and eutectic Nbss decreases with the pulling rate increasing, resulting in a straighter primary dendrite γ -Nb₅Si₃ and a fishbone like Nbss/ γ -Nb₅Si₃ coupling structure of secondary eutectic.
- 2) The size of the nascent Nbss and γ -Nb₅Si₃ increases with the power supply increasing, and the coupling growth trend of Nbss and Nb₅Si₃ weakens.
- 3) As the pulling rate and power supply increase, the room temperature fracture toughness shows a trend of first increasing and then decreasing. The KQ of the directionally solidified alloy reaches its optimal value of 22.35 MPa·m^{1/2} at 48 kW and 0.4 mm/min.
- 4) The room temperature compressive strength of the alloy gradually increases with the increasing of pulling rate increases. When the pulling rate is 0.4 mm/min, the compressive strength of the directionally solidified alloy reaches 2278 MPa. As the power supply increases, the room temperature compressive strength of directionally solidified alloys gradually decreases.

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