

MICROSTRUCTURAL CHARACTERISTICS AND MECHANICAL PROPERTIES OF A Nb/Nb₅Si₃ BASED COMPOSITE WITH AND WITHOUT DIRECTIONAL SOLIDIFICATION

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ABSTRACT

The Nb/Nb₅Si₃ based composites were fabricated by conventional casting (CC) and directional solidification (DS) methods. Microstructural characteristics, compressive properties and fracture toughness of the CC and DS composites were investigated by SEM, XRD, TEM, bending and compression tests. The results demonstrate that in the CC Nb/Nb₅Si₃ based composite, the intergrowth of fine (Nb,Ti)₃Si and α -(Nb,Ti)₅Si₃ phases leads to the formation of eutectic structure and the coarse α -(Nb,Ti)₅Si₃ dendritic phase prefers to grow along eutectic cell boundary. The (Nb,Ti)₃Si, (Ti,Nb)₅Si₃ and Dy₂O₃ phases mainly segregate along the eutectic cell boundary and moreover there is an orientation relationship between the (Nb,Ti)₃Si and (Nb,Ti)₅Si₃ phases: $[001]_{(Nb,Ti)_3Si} // [112]_{(Nb,Ti)_5Si_3}$ and $(110)_{(Nb,Ti)_3Si} // (110)_{(Nb,Ti)_5Si_3}$. The DS processing promotes the formation of coarse primary α -(Nb,Ti)₅Si₃ phase, (Ti,Nb)₅Si₃/(Nb,Ti)₅Si₃ eutectic and α -(Nb,Ti)₅Si₃/(Nb,Ti)₅Si₃ eutectic in the DS Nb/Nb₅Si₃ based composite. Moreover, the (Nb,Ti)₅Si₃ and α -(Nb,Ti)₅Si₃ phases are aligned paralleling to the DS direction and exhibits strong crystal orientation preference. In addition, an orientation relationship between the (Nb,Ti)₅Si₃ and α -(Nb,Ti)₅Si₃ phases is observed: $[310]_{\alpha-(Nb,Ti)_5Si_3} // [110]_{(Nb,Ti)_5Si_3}$. Compared with the CC Nb/Nb₅Si₃ based composite, the DS Nb/Nb₅Si₃ based composite possesses the higher yield strength and fracture toughness, which should be ascribed to the microstructure optimization.

Key words: Nb/Nb₅Si₃ composite; Directional solidification; Microstructure; Mechanical properties

1. INTRODUCTION

In the past decades, the intermetallic compound has drawn so much attention, because of its excellent advantages in some aspects [1-3]. As a kind of intermetallic compound, niobium silicide (Nb-Si) has many advantages, such as high melting point (about 2520°C), excellent creep resistance and high strength [4,5]. Once, the Nb-Si intermetallic compound had been considered as a promising material to use as structural component in ultra-high temperature environment [6]. However, the undesirable fracture toughness at room temperature and poor oxidation resistance intermediate temperature handicap its application [7,8]. In order to conquer these problems, many technologies have been used and many Nb-Si based alloys have been developed [9-11].

The S. Qu et al.[12] investigated the influence of alloying element on microstructure of Nb-Si binary alloy and exhibited that the Ti and Hf could stabilize Nb₃Si phase, while the Al and Cr lead to the forming of Nb₅Si₃ phase. While, the research of Li exhibited that the addition of Ti could result in the formation of (Nb,Ti)₅Si₃ phase which contributed to increase

the strength of the Nb-Si alloy. The of N. Sekido et al [14] investigated the Nb-Ti-Si alloy and binary Nb-Si alloy prepared by unidirectional solidification and revealed that the unidirectional solidification could arrange the Nb and Nb₃Si phases along solidification direction regularly. Such a microstructure could make the utmost of the ductility of Nb solid solution (Nbss) and be beneficial to the increase of the fracture toughness greatly. However, the presence of massive Nbss was harmful to the high-temperature strength, because of its low deformation resistance at high-temperature. Moreover, the oxidation resistance of the Nb-Si alloy still needs to be improved. Therefore, the further alloying has been applied on the Nb-Si-Ti based alloy.

The previous researches [15,16] had demonstrated that the oxidation resistance of Nb-Si based alloy could be enhanced by adding of Cr and Al. In addition, the research [17,18] indicates that the stable Nb₅Si₃ phase is more helpful to enhance the strength of Nb-Si based alloy. Thus the Nb-Si-Ti-Al-Cr-Hf alloy was thought as the most potential one containing the ductile Nb solid solution (Nbss) phase and

the Nb₅Si₃ strengthening phase [19-21]. Based on the recent investigations [22,23], the appropriate rare earth addition could increase the cohesion of phase boundary. Moreover, the regular and fine arrangement of ductile and strengthening phases could be helpful to the ductility and strength simultaneously [24,25]. Therefore, in the present research the dysprosium (Dy) was added in the Nb-Si-Ti-Al-Cr-Hf alloy and fabricated by the conventional casting (CC) to obtain the CC Nb/Nb₅Si₃ based composite. The directional solidification (DS) was performed by using the optical floating zone melting method to prepare the DS Nb/Nb₅Si₃ based composite. The microstructural observation and mechanical properties of the CC and DS Nb/Nb₅Si₃ based composite were investigated.

2. EXPERIMENTAL PROCEDURE

The alloy with nominal composition of Nb-16Si-22Ti-3Al-7Cr-2Hf-0.1Dy (at.%) was prepared by the vacuum arc melting technology from niobium (99.9%), silicon (99.9%), titanium (99.9%), aluminum (99.9%), chromium (99.9%), dysprosium (99.8%) and hafnium (99.8%) to obtain the CC Nb/Nb₅Si₃ based composite. A part of CC Nb/Nb₅Si₃ based composite was investigated at as cast state and the other was ready for the DS process. The rods for DS process with dimension of $\Phi 8 \times 80$ mm were cut from the CC Nb/Nb₅Si₃ based composite by the electro-discharge machining (EDM). The op-

tical floating zone (OFZ) melting furnace was used to perform the DS process, which adopted the high-energy visible light to remelt the rod with small fusion zone. In addition, the synchronous rotation of the original and melted rods guaranteed the uniform and orderly crystal growth. The growth rate of the composite was set as 8 mm/h.

The specimens for microstructural observation and phase identification were cut from the CC and DS Nb/Nb₅Si₃ based composites and prepared by the conventional metallographic method. Microstructural observation on the CC and DS Nb/Nb₅Si₃ based composites were carried out by a JEOL 6340 Field Emission Gun Scanning Electron Microscopy (FEGSEM) and Hitachi S-3400N scanning electron microscopy (SEM). The Philips X-ray diffractometer with Cu-K α radiation ($\lambda = 0.15418$ nm) was employed to analyse the phases at 40 kV and 40 mA. The foils for transmission electron microscopy (TEM) observation were cut from the CC and DS Nb/Nb₅Si₃ based composites and sectioned and grounded. The foils with diameter of $\phi 3$ mm and thickness of 30 μ m were twin-jet electropolished in a solution of 90% alcohol and 10% perchloric acid at -20°C with the twin-jet current of 40mA. The JEOL-2100 high-resolution transmission electron microscope (HRTEM) was employed to observe the precipitate in the composites.

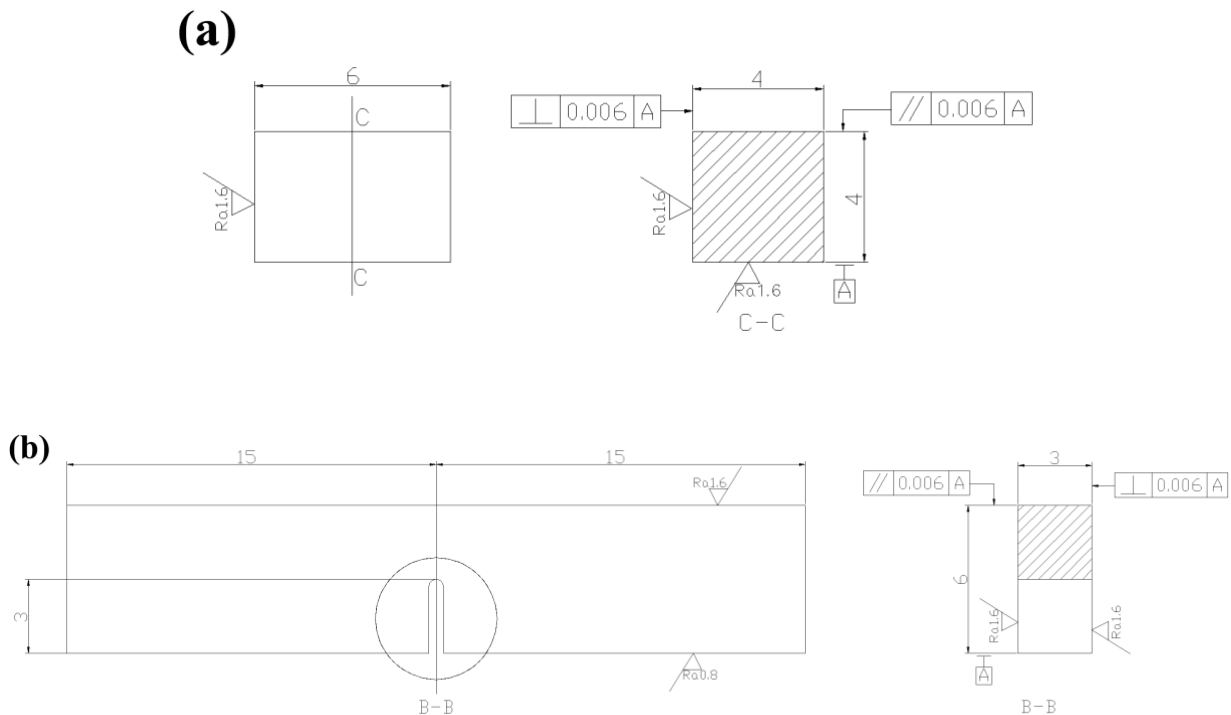


Fig.1: The Schematic diagrams of the compression and bending test specimens: (a) Compression test specimen, (b) Bending test specimen

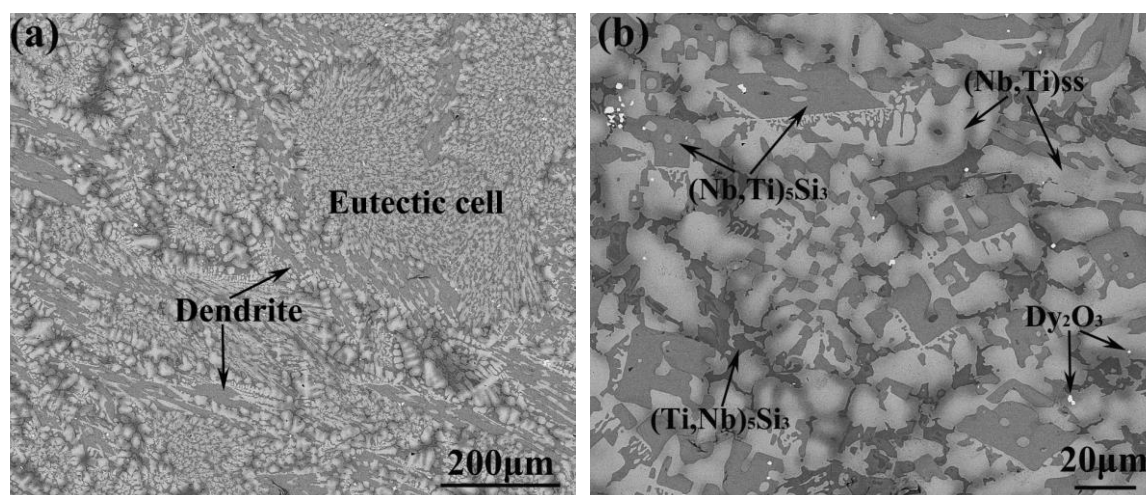


Fig.2: SEM micrograph of the CC Nb/Nb₅Si₃ based composite: (a) Morphology of the eutectic structure, (b) Precipitates distributed along eutectic cell boundary

Compression specimens with the size of 4mm × 4mm × 6mm were cut from the CC and DS Nb/Nb₅Si₃ based composites and grounded by 1000-grit SiC abrasive, as shown Fig.1 (a). The compression was tested on the Gleeble 3800 based on the GB/T 7314-2005 standard. The compression test was carried out in air with the initial strain rate of 2×10^{-3} /s at room temperature. Three compression tests of the same composite were performed to evaluate the mechanical properties. The three-point bending test based on the GBT 4161-2007 standard was performed to obtain the fracture toughness. The specimens for bending test were cut from the CC and DS Nb/Nb₅Si₃ based composites with the size of 3 mm × 6 mm × 30 mm with a 3 mm notch in middle, as shown in Fig.1 (b). For the DS specimens, the composite growth direction is perpendicular to the force direction. The bending tests were performed on the Instron type testing machine with the cross-head speed of 5×10^{-3} mm/s and the loading direction was perpendicular to the solidification direction. Three bending tests were performed to evaluate the fracture toughness of the composite.

3. RESULTS AND DISCUSSION

3.1. Microstructure

The SEM observations on the CC Nb/Nb₅Si₃ based composite are presented in Fig.2 (a) and (b). Based on the backscattered electron SEM micrograph, it can find that the microstructure of the CC Nb/Nb₅Si₃ based composite is mainly composed of relative fine eutectic structure and coarse gray phase dendrite along the eutectic structure boundary. The further observation reveals that the CC Nb/Nb₅Si₃ based composite comprises of four kinds of phases. Based on recent researches [15,19], grey phase should be α -(Nb,Ti)₅Si₃ and the grey-white matrix

should be (Nb,Ti)ss phase, which form the main eutectic structure and grows into eutectic cell. Moreover, the black (Ti,Nb)₅Si₃ phase tends to precipitate and grow along eutectic cell boundary forming the semi-continuously necklace shape. Additionally, the white particles mainly distribute along phase and grain boundary randomly.

XRD analysis on the CC Nb/Nb₅Si₃ based composite exhibits that the (Ti,Nb)₅Si₃, (Nb,Ti)ss and α -(Nb,Ti)₅Si₃ phases are main constituents, as seen in Fig.3. From the XRD pattern, it can confirm that the α -(Nb,Ti)₅Si₃ phase has the Cr₅B₃ type crystal structure (I4/mcm). Furthermore, the amount of α -(Nb,Ti)₅Si₃ phase is more than that of the (Ti,Nb)₅Si₃ phase. The different crystal orientations of phases indicate the random of crystal growth in the CC Nb/Nb₅Si₃ based composite.

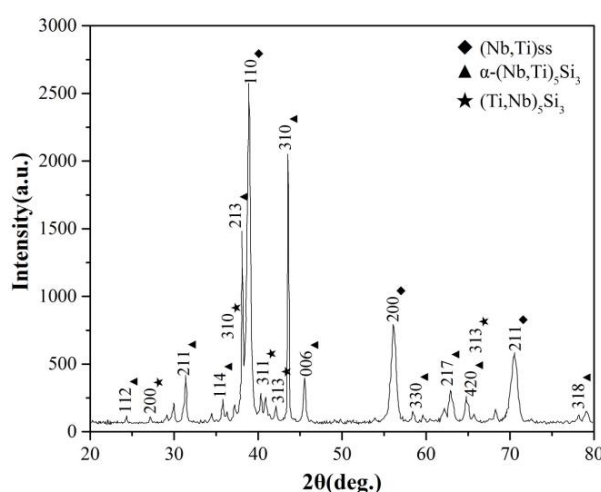


Fig.3: XRD pattern of the CC Nb/Nb₅Si₃ based composite

TEM observation and selected area diffraction were

carried out to analyse the phase in the CC Nb/Nb₅Si₃ based composite. It reveals that the (Ti,Nb)₅Si₃ and α -(Nb,Ti)₅Si₃ phases inter-grow near eutectic cell boundary, as shown in Fig.4 (a). The selected area diffraction patterns (SADPs) confirm that the (Ti,Nb)₅Si₃ and α -(Nb,Ti)₅Si₃ phases have the hexagonal and tetragonal crystal structure, respectively. TEM observation still finds the (Nb,Ti)₃Si precipitates near the eutectic cell boundary, as shown in Fig.4 (b). According to the previous researches [16,20], the (Nb,Ti)₃Si is metastable in the Nb-Si-Ti based alloy. Moreover, TEM observation also reveals that there is an orientation relationship between (Nb,Ti)₃Si and (Nb,Ti)ss: $[001]_{(Nb,Ti)_3Si} // [112]_{(Nb,Ti)ss}$ and $(110)_{(Nb,Ti)_3Si} // (110)_{(Nb,Ti)ss}$. The formation of this type of phase may be attributed to the element segregation and relative high interface supercooling. Moreover, some small oxides have been observed by the TEM observation, as presented in Fig.4 (c). The SADP confirms that it is Dy₂O₃ which has the P-3m1 space group and hexagonal crystal structure ($a=b= 3.82$ nm, $c=6.11$ nm). The Dy₂O₃

oxides should be the white particle observed in the SEM. The presence of Dy₂O₃ oxide should be ascribed to the high activity of rare earth Dy which could capture the oxygen and impurity to form the oxide. This formation of Dy₂O₃ oxide is beneficial to the cohesion of phase boundary and grain boundary.

SEM observations on the DS Nb/Nb₅Si₃ based composite are shown in Fig.5. It can find that the microstructure of the Nb/Nb₅Si₃ based composite is changed by the DS process greatly. The observation on the DS Nb/Nb₅Si₃ based composite perpendicular to the DS direction shows the coarse primary α -(Nb,Ti)₅Si₃ exhibits rectangle shape and intergrow with the (Nb,Ti)ss, which forms the core of eutectic cell, as shown in Fig.5(a). The (Ti,Nb)₅Si₃/(Nb,Ti)ss and α -(Nb,Ti)₅Si₃/(Nb,Ti)ss eutectic structures grow on the core of the eutectic cell. The observation on the DS Nb/Nb₅Si₃ based composite parallel to the DS direction shows that the (Nb,Ti)ss and α -(Nb,Ti)₅Si₃ phases grow alternatively forming the lamellar structure, as exhibited in Fig.5 (b). How-

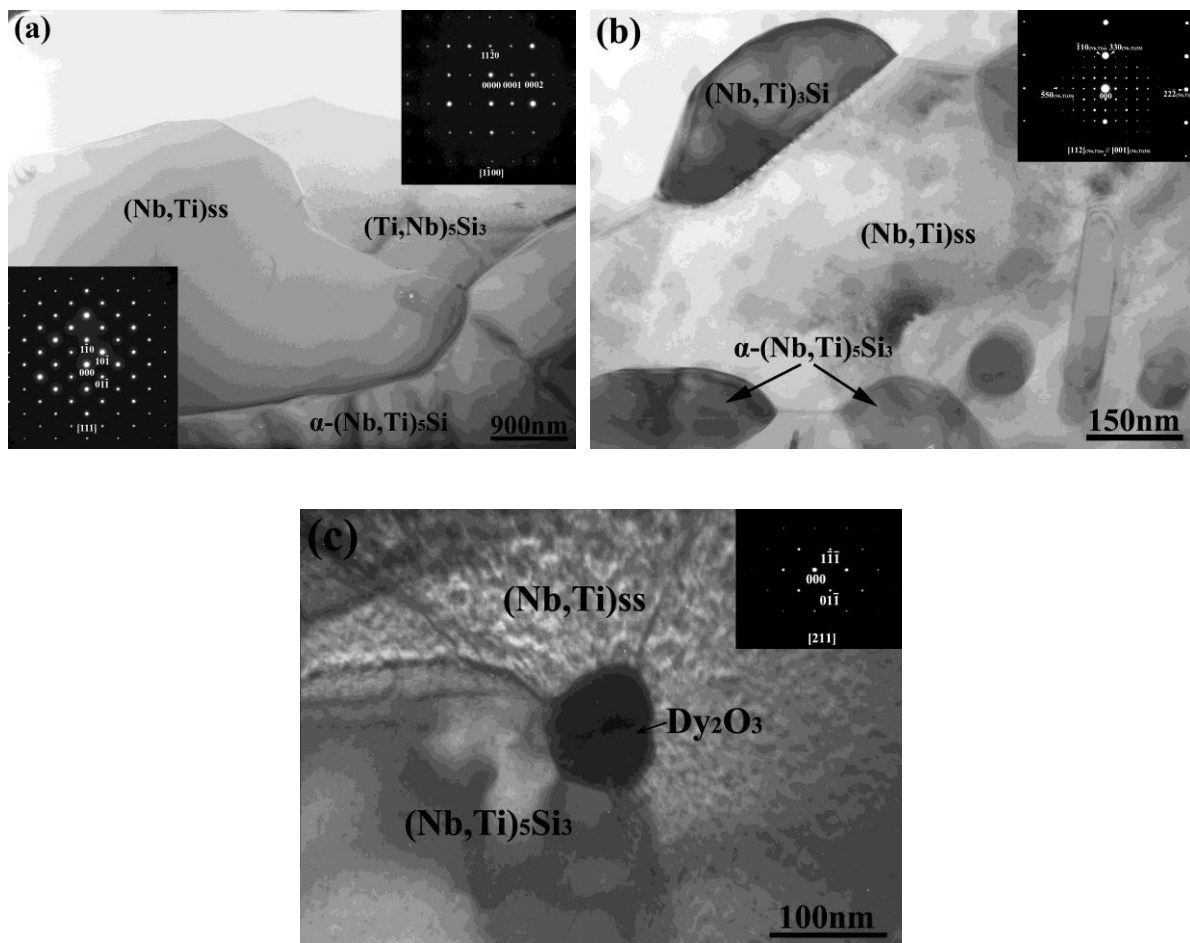


Fig.4: TEM observation on the CC Nb/Nb₅Si₃ based composite: (a) Morphology and SADPs of the (Ti,Nb)₅Si₃ and α -(Nb,Ti)₅Si₃ phases (The inset top right micrograph showing the SADP of (Ti,Nb)₅Si₃ phase and the bottom left image showing the SADP of α -(Nb,Ti)₅Si₃), (b) Precipitate of (Nb,Ti)₃Si phase along the eutectic cell boundary (Inset image showing the compound SADP of (Nb,Ti)₃Si phases and (Nb,Ti)ss), (c) Precipitate of Dy₂O₃ oxide (Inset image showing the corresponding SADP of oxide)

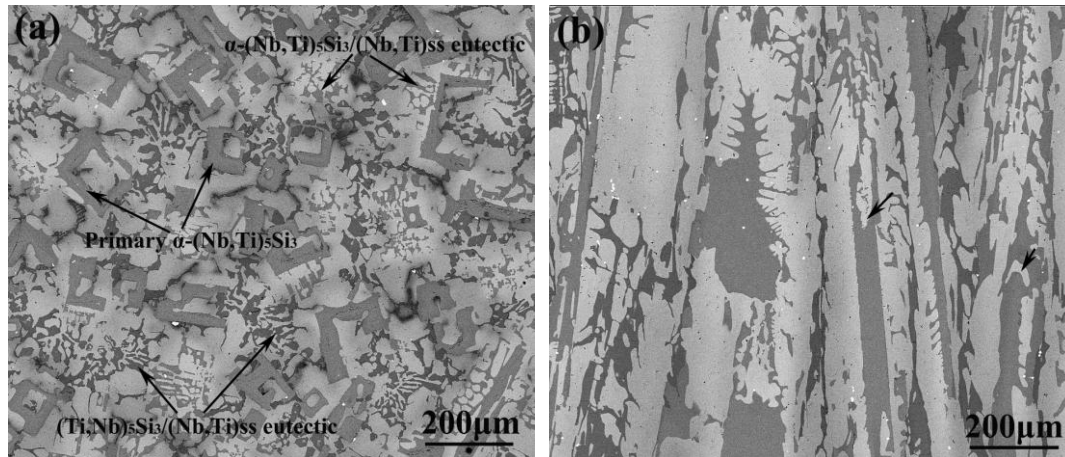


Fig.5: SEM image of the DS Nb/Nb₅Si₃ based composite fabricated by 8 mm/h growth rate: (a) Microstructure perpendicular to the DS direction, (b) Microstructure parallel to the DS direction

ever, it can be found that the α -(Nb,Ti)₅Si₃ phases are broken periodically by the eutectic structure. According to the recent researches [26,27], the low growth rate is beneficial to the element diffusion, which would reject the redundant solute elements that the equilibrium eutectic growth does not need. The increased solute element concentration in front of liquid/solid interface would lead to great constitutional supercooling which could result in the formation of crystal nucleus and growth of fine eutectic structure. And then the growth of the (Nb,Ti)ss and primary α -(Nb,Ti)₅Si₃ lamellar structure would be interrupted, as indicated by arrowed position in Fig.5 (b). Moreover, the narrow melting region in the FOZ method promotes the solute element concentration, due to its limited element diffusion region. Compared with the CC Nb/Nb₅Si₃ based composite, the DS could coarsen the primary α -(Nb,Ti)₅Si₃ phase but refine the eutectic cell, which should be attributed to the low growth rate. In addition, the amount of (Ti,Nb)₅Si₃ has been increased and the distribution of Dy₂O₃ particle becomes more uniform.

The XRD analysis on the DS Nb/Nb₅Si₃ based composite demonstrates that the DS processing has changed the crystal growth greatly, as shown in Fig.5 (a). The DS Nb/Nb₅Si₃ based composite has obvious crystal orientation preference. Compared with the CC Nb/Nb₅Si₃ based composite, the α -(Nb,Ti)₅Si₃ phase replaces the (Nb,Ti)ss as the strongest peak and it prefers to grow along (310) crystal plane greatly. Moreover, the amount of the α -(Nb,Ti)₅Si₃ phase in the DS Nb/Nb₅Si₃ based composite is more than that in the CC Nb/Nb₅Si₃ based composite. And the microstructure observation also confirms such a phenomenon. Different from the relative balanced growth preference of (Nb,Ti)ss phase along (110),

(200) and (211) crystal plane, the DS processing promotes the (Nb,Ti)ss phase mainly grow along the (110) crystal plane. And the (310) crystal plane also becomes the preference of (Ti,Nb)₅Si₃ phase in the DS Nb/Nb₅Si₃ based composite. Based on the previous researches [21,28], the (Ti,Nb)₅Si₃ and α -(Nb,Ti)₅Si₃ phases almost have the similar lattice parameters, which indicates they would choose the same crystal growth preference under the same crystal growth condition. Moreover, the (Nb,Ti)ss matrix may promote the (Ti,Nb)₅Si₃ and α -(Nb,Ti)₅Si₃ phases grow along the same crystal plane.

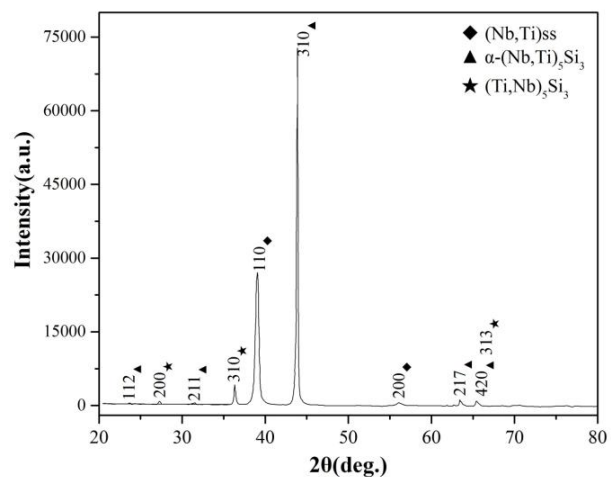


Fig.6: XRD pattern of the DS Nb/Nb₅Si₃ based composite

TEM observation on the DS Nb/Nb₅Si₃ based composite was carried out to analyse the microstructure evolution. The TEM observation exhibits that the (Nb,Ti)ss and primary α -(Nb,Ti)₅Si₃ phases have the straight and clear interface, as shown in Fig.7(a). Moreover, the compound SADP indicates that there is an orientation relationship between the (Nb,Ti)

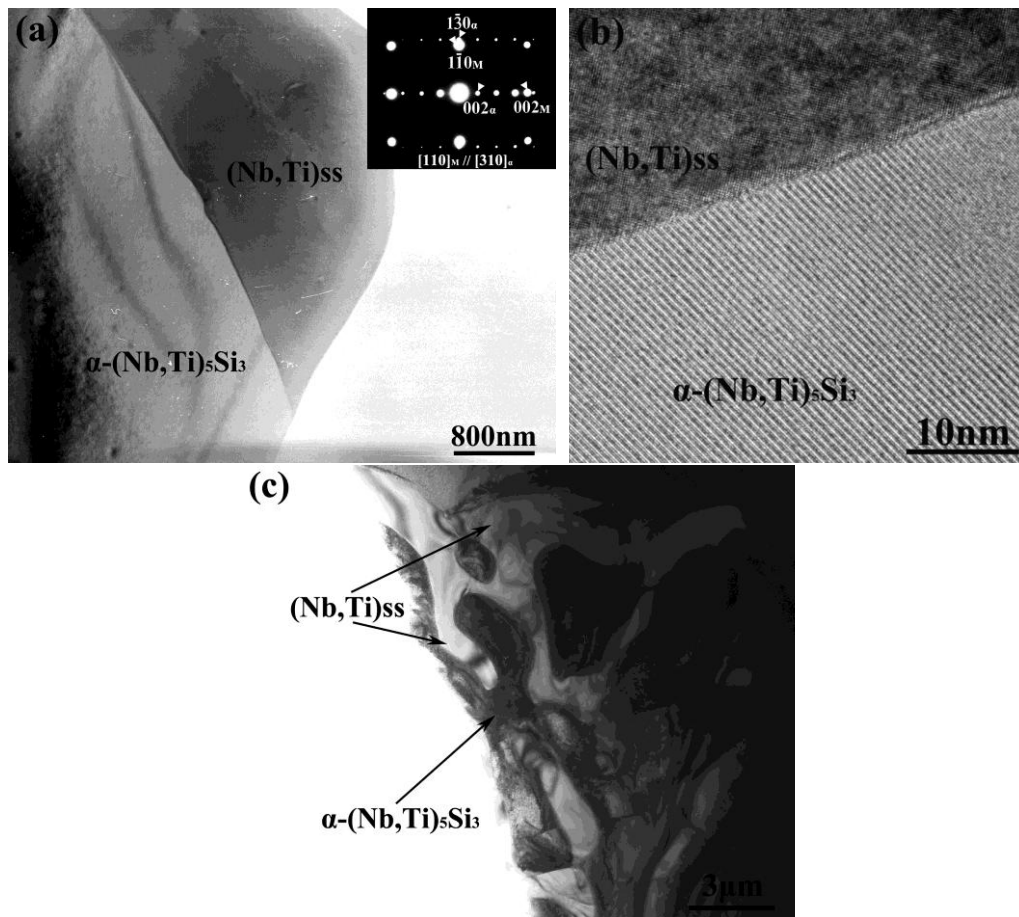


Fig.7: TEM observation on the DS Nb/Nb₅Si₃ based composite: (a) Morphology of intergrowth α -(Nb,Ti)₅Si₃ and (Nb,Ti)ss (The inset image showing the compound SADP), (b) HRTEM micrograph of the phase interface of (Nb,Ti)ss and α -(Nb,Ti)₅Si₃, (c) Morphology of fine α -(Nb,Ti)₅Si₃/(Nb,Ti)ss eutectic structure

ss and α -(Nb,Ti)₅Si₃ phases: $[310]_{\alpha-(Nb,Ti)_5Si_3} // [110]_{(Nb,Ti)ss}$. Further high resolution TEM (HRTEM) observation along $[110]$ zone axis of (Nb,Ti)ss phase demonstrated that the atom array of (Nb,Ti)ss phase could be observed clearly but in the α -Nb₅Si₃ phase just atom array of some crystal plane can be observed, as exhibited in Fig.7(b). Further analysis on the compound SADP can find that the diffraction spots of the two phases have not overlapped well and there is a little distance. Such a phenomenon should be ascribed to the great difference of crystal parameters between them. Further observation finds the fine α -(Nb,Ti)₅Si₃/(Nb,Ti)ss eutectic structure near the eutectic cell boundary. Inside eutectic structure, the (Nb,Ti)ss and α -(Nb,Ti)₅Si₃ aligns alternately. The width of the (Nb,Ti)ss lamella is $1.5 \sim 3$ μm and that of α -(Nb,Ti)₅Si₃ lamella is $2 \sim 3$ μm.

3.2 Mechanical properties

In order to investigate the effect of DS process on the mechanical properties of the Nb/Nb₅Si₃ based composite, the compression and three-point bending tests at room temperature were carried out. The

room-temperature yield strength and fracture toughness of the CC and DS Nb/Nb₅Si₃ based composites are exhibited in Table 1. Obviously, the mechanical properties of the Nb/Nb₅Si₃ based composite has been improved obviously by the DS process. The yield strength and fracture toughness of the DS Nb/Nb₅Si₃ based composite are both higher than those of the CC Nb/Nb₅Si₃ based composite, especially the fracture toughness. It almost 30% higher than that of the CC Nb/Nb₅Si₃ based composite. The improved yield strength and fracture toughness of the Nb/Nb₅Si₃ based composite should be attributed to the microstructure optimization by the DS process. According to the previous investigations [30-32] on the lamellar composite, the morphology of constituent phase and interface are very important to the strength and ductility of the composite. The fine lamellar structure with alternative strengthening phase and ductile phase is beneficial to strength and ductility, because the phase interface could handicap the movement of dislocations and deflect the cracks. However, the increase of eutectic cell boundary would be detrimental to ductility due to the segrega-

Table 1: Yield strength and fracture toughness of the CC and DS Nb/Nb₅Si₃ based composites

Composite	Yield strength (MPa)	K _Q (MPa·m ^{1/2})
CC	1750±15.6	10.7±0.6
DS	1980±14.1	13.6±0.3

tion of impurity here. Furthermore, the fine lamellar structure is harmful to the high-temperature strength. While in the present study, the CC Nb/Nb₅Si₃ based composite has relative fine eutectic structure, which contributes to the strength and fracture toughness. However, the CC Nb/Nb₅Si₃ based composite also contain many bulk dendrite of α -(Nb,Ti)₅Si₃ phase, which is harmful to its fracture toughness. Moreover, the obvious eutectic cell boundary and the segregated (Ti,Nb)₅Si₃ phase decrease the fracture toughness further. By the DS processing, the DS Nb/Nb₅Si₃ based composite exhibits relative uniform combination of primary α -(Nb,Ti)₅Si₃ phase and eutectic structures. Though the coarse primary α -(Nb,Ti)₅Si₃ phase is harmful to the fracture toughness, it is helpful to the strength. And moreover, the well-aligned (Nb,Ti)_{ss} and primary α -(Nb,Ti)₅Si₃ paralleling DS direction could make full use of the strengthening and ductile effect. In addition, the dimming of eutectic cell boundary also contribute to the field strength and fracture toughness. Therefore, the mechanical properties of the DS Nb/Nb₅Si₃ based composite are higher than that of the CC Nb/Nb₅Si₃ based composite.

4. CONCLUSIONS

(1) In the CC Nb/Nb₅Si₃ based composite, the intergrowth of fine (Nb,Ti)_{ss} and α -(Nb,Ti)₅Si₃ phases forms the eutectic structure and the coarse α -(Nb,Ti)₅Si₃ dendritic phase prefers to grow along eutectic cell boundary. The (Nb,Ti)₃Si, (Ti,Nb)₅Si₃ and Dy₂O₃ phases mainly segregate along the eutectic cell boundary and moreover there is an orientation relationship between the (Nb,Ti)₃Si and (Nb,Ti)_{ss} phases: $[001]_{(Nb,Ti)3Si} // [112]_{(Nb,Ti)ss}$ and $(110)_{(Nb,Ti)3Si} // (110)_{(Nb,Ti)ss}$.

(2) The DS processing promotes the formation of coarse primary α -(Nb,Ti)₅Si₃ phase, (Ti,Nb)₅Si₃/(Nb,Ti)_{ss} eutectic and α -(Nb,Ti)₅Si₃/(Nb,Ti)_{ss} eutectic structures in the DS Nb/Nb₅Si₃ based composite. Moreover, the (Nb,Ti)_{ss} and α -(Nb,Ti)₅Si₃ phases are aligned paralleling to the DS direction and exhibits strong crystal orientation preference. In addition, an orientation relationship between the (Nb,Ti)_{ss} and α -(Nb,Ti)₅Si₃ phases is observed:

$$[310]_{\alpha-(Nb,Ti)5Si3} // [110]_{(Nb,Ti)ss}$$

(3) Compared with the CC Nb/Nb₅Si₃ based composite, the DS Nb/Nb₅Si₃ based composite has the higher yield strength and fracture toughness, which should be attributed to the microstructure optimization.

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