

Effect of Transition Element Ni on Microstructure and Mechanical Properties of Nb-22Si-20Ti Alloy

Xu Qin¹, Ma Xiaohang¹, Chen Dezhi², Cai Yanhui¹, Yin Yajun³, and *Chen Ruirun²

1. School of Mechanical and Electrical Engineering, Henan University of Technology, Zhengzhou, 450001, China

2. National Key Laboratory for Precision Hot Processing of Metals, Harbin Institute of Technology, Harbin, 150001, China

3. State Key Laboratory of Material Processing and Die and Mould Technology, Huazhong University of Science and Technology, Wuhan, 430074, China

*Corresponding address: e-mail: ruirunchen@hit.edu.cn

Abstract: Nb-22Si-20Ti-xNi (x=0, 1, 2, 3, 4at.%) alloys with different content of Ni were prepared by vacuum non-consumable arc melting method, and effects of Ni content on phase constitution, microstructure and mechanical properties were investigated. Results show that addition of Ni will change the phase composition of Nb-22Si-20Ti alloy, and NST-0Ni alloy is mainly consisted of Nbss phase, Nb₃Si phase and a small amount of β -Nb₅Si₃ phase. Addition of Ni will lead to phase transformation from β -Nb₅Si₃ phase to α -Nb₅Si₃ phase and formation of Ti₂Ni phase. Ni is mainly dissolved in silicides, especially in α -Nb₅Si₃ phase which is the highest of solubility. Microhardness of silicide increases from 1298.5 HV to 1450.1 HV which is increased by 10.5% when addition of Ni is increased from 0 to 4at.%, and microhardness of Nbss increases from 645.5 HV to 718.3 HV which is increased by 11.2%. Compressive strength of Nb-22Si-20Ti alloy at room temperature is improved by addition of Ni, but fracture strain of the alloys is reduced. Compressive strength of NST-0Ni alloy is 1972.5 MPa, and the fracture strain is 7.5%. Compressive strength of NST-2Ni alloy reaches its maximum of 2295.9 MPa, which is 16.4% higher than that of NST-0Ni alloy. Fracture strain of NST-2Ni alloy is the lowest of 7.3%. Solid solution strengthening effect caused by addition of Ni can improve microhardness and compressive strength for Nb-22Si-20Ti-xNi alloys, but decrease fracture strain of alloys. In addition, formation of brittle and low strength Ti₂Ni phase will lead to decrease of strength.

Keywords: Nb-Si alloys; alloying effect; microstructure; mechanical property

1 Introduction

Nb-Si alloys exhibit many excellent properties, such as high melting temperature (1750°C), low density (6.6~7.2 g/cm³), high strength at high temperature (~310 MPa at 1350°C) and good creep resistance^[1], and they are considered as promising candidates for the next generation gas turbine blades. Nb-Si alloys are consisted of Nbss and silicide, of which Nbss is the main solid solution phase and silicide contains Nb₃Si and Nb₅Si₃ compounds. Nbss can provide good plasticity and toughness for Nb-Si alloys at room and high temperature and play a key role in improving toughness of Nb-Si alloys. While silicide phase with high hardness and strength can improve high temperature strength and deformation resistance. Synergistic effect of Nbss and silicide phase grant high mechanical properties and good oxidation resistance for Nb-Si alloys.

However, comprehensive mechanical properties of Nb-Si alloys still need to be further improved by various methods^[2-4], of which alloying by different elements has

been proved a very effective method. Alloying elements added to Nb-Si alloys include Ti^[5,6], V^[7], Hf^[8], Cr^[9], Fe^[10,11], B^[12], Re^[13,14], Zr^[15,16] etc, and addition of alloying elements to Nb-Si alloys significantly improve their mechanical properties, therefore promote its further development. Element Ti has been proved to improve toughness and oxidation resistance of Nbss phase. Sekido et al^[17,18] found that addition of 10at.%Ti into Nb-18.7Si alloy can increase eutectoid transformation kinetics, but reduce strength of alloys for addition of Ti can increase dislocation mobility of Nbss phase. Adding appropriate amount of Ti into Nb-Si alloys can reduce the unstable stacking energy and P-N potential barrier energy of dislocation motion and improve ductility of the alloys^[19], therefore, researchers developed ternary Nb-Si-Ti alloys^[20-22].

Miura et al^[23] found that element Ni can promote eutectoid decomposition of Nb₃Si phase in binary Nb-Si alloys. Research by Huang et al^[24] showed that addition of element Ni into ternary Nb-Si-Ti alloy can introduce new Ti₂Ni phase into the alloy. Chen et al^[25] investigated

effect of element Ni on Nb-Si-Ti-Cr-Al-Hf-Zr multiple component alloy and found that addition of Ni can change solidification path of alloy, primary α -(Nb, X)₅Si₃ phase can be observed in the alloy with element Ni, and fracture toughness and compressive strength of the alloy are both improved. However, effect mechanism of element Ni on ternary Nb-Si-Ti alloys still need to be further systematically investigated.

Therefore, Nb-22Si-20Ti ternary alloy was selected as the research object in the present paper, and alloys with different amount of Ni were prepared by non-consumable arc melting furnace to systematically study effect mechanism of Ni on microstructure and mechanical properties of the alloy. Effect of Ni on phase, microstructure, hardness, compressive strength and fracture strain of the alloy will be investigated, and the mechanism of microstructure on mechanical properties of Nb-Si alloys with element Ni will also be explored.

2 Experimental procedures

Nb-22Si-20Ti-xNi alloy ingots with different amount of Ni ($x=0, 1, 2, 3, 4$ at.%) were prepared by non-consumable arc melting furnace under protection of inert gas Ar. To simply expression of alloys, Nb-22Si-20Ti-xNi alloy with addition of Ni by 0, 1at.%, 2at.%, 3at.% and 4at.% were referred to as NST-0Ni, NST-1Ni, NST-2Ni, NST-3Ni and NST-4Ni, respectively. The alloy ingots were taken through five re-melting process to ensure homogeneity of chemical composition for the alloys.

Specimens for microstructure observation were cubic blocks with dimensional size of 10mm×10mm×10mm, which were cut from the button ingots by wire electrode discharge machining (EDM) system. X-ray diffraction (XRD, Panalytical X'Pert, Cu K α , 20KV, 20-90°, 8°/min) were used to determine phases of different alloys. Scanning electron microscopy (SEM, FEI, Quanta 200FEG) was used to conduct microstructure observation in backscattering (BSE) mode. Energy dispersive spectroscopy (EDS, INCA x-sight 7426) equipped on SEM was used to analyze chemical compositions of different phases.

Specimens for compressive test were cylinder blocks with dimensional size of $\phi 4\text{mm} \times 6\text{mm}$ cut from alloys ingots. Compression experiments were conducted at room temperature using Instron 5569 Mechanical Tester at a loading rate of 0.2 mm/min, and the test were conducted three times for each alloy. Fracture surface of the compressive specimens were observed by SEM in secondary electron imaging mode. Microhardness of different phases was measured by FM-ARS900 hardness

tester with load of 100 gf for 10 s, and it was tested five times for each phase and the average was taken as microhardness of the phase for each alloy.

3 Results

3.1 Phase composition

Fig.1 show XRD patterns of Nb-22Si-20Ti-xNi ($x=0, 1, 2, 3, 4$ at.%) alloys with different content of Ni. Results show that NST-0Ni alloy without addition of Ni is consisted of three phases, Nbss, Nb₃Si and β -Nb₅Si₃. Diffraction peaks of β -Nb₅Si₃ phase cannot be observed at XRD patterns of Nb-22Si-20Ti alloy after addition of Ni, and diffraction peaks of α -Nb₅Si₃ can be observed in Ni-containing Nb-22Si-20Ti alloys. Therefore, Nb-22Si-20Ti-xNi alloys with different addition of Ni are consisted of Nbss, Nb₃Si and α -Nb₅Si₃ phase.

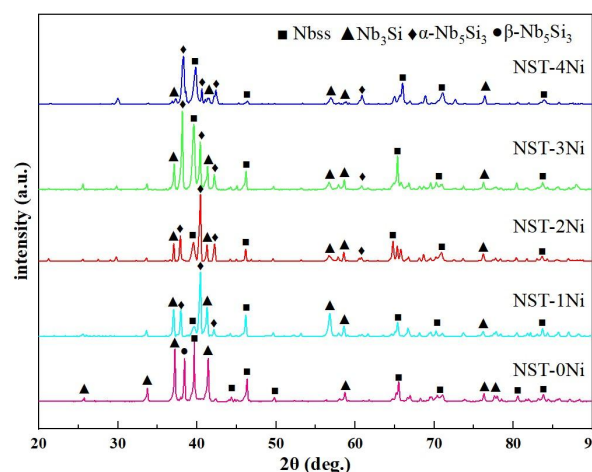


Fig. 1: XRD patterns of Nb-22Si-20Ti-xNi alloys

3.2 Microstructure characteristics

Fig.2 shows microstructure of Nb-22Si-20Ti-xNi ($x=0, 1, 2, 3, 4$ at.%) alloys with different addition of Ni observed by SEM in backscattering mode. Results show that three different phases with distinguished contrast can be observed in the alloys, white phase, light grey phase and dark grey phase. White phase in the alloys is Nbss solid solution, light grey phase is Nb₃Si phase and dark grey phase is confirmed to be Nb₅Si₃ phase according to XRD results and EDS results of distinct phases listed in Table 1. NST-0Ni alloy without any addition of Ni is mainly consisted of Nbss phase, Nb₃Si phase and β -Nb₅Si₃ phase, shown in Fig.2 (a). There are large blocks of Nb₃Si phase, small amount of Nbss phase and some dark grey β -Nb₅Si₃ phase which is surrounded by light grey Nb₃Si phase. The β -Nb₅Si₃ phase in NST-0Ni alloy is primary phase formed directly from solidification of the liquid, other than decomposition of Nb₃Si phase. During solidification of the liquid NST-0Ni alloy, primary

β -Nb₅Si₃ phase first form from the liquid phase, then the primary β -Nb₅Si₃ phase will peritectically react with the liquid phase, $L+\beta$ -Nb₅Si₃→Nb₃Si, and form Nb₃Si phase. However, the peritectic reaction between primary β -Nb₅Si₃ phase and liquid phase is not complete, therefore, phenomenon that dark grey β -Nb₅Si₃ phase surrounded by light grey Nb₃Si phase can be observed in NST-0Ni alloy. Similar phenomenon was also observed in researches by Sekido et al [17] and Guo et al [26]. EDS results listed in Table 1 indicate that content of Nb in β -Nb₅Si₃ phase is the lowest, that of Si in β -Nb₅Si₃ phase is the highest. And content of Ti in Nb₃Si phase and β -Nb₅Si₃ phase (15.13at.% and 15.56at.%) is lower than that in Nbss phase (27.55at.%).

NST-1Ni alloy with addition of 1at.% Ni is mainly consisted of Nbss phase, Nb₃Si phase and α -Nb₅Si₃ phase, and some black phase form on the phase boundaries of Nbss phase and Nb₃Si phase. EDS results of the black phase shows that content of Ti in the black phase is 40.46at.% and that of Ni is 19.28at.%. Therefore, the

black phase is determined as Ti₂Ni phase based on previous research, and there is no diffraction phase in XRD pattern in Fig.1 for the amount of the black Ti₂Ni phase is very tiny. In addition, there are no β -Nb₅Si₃ phases and α -Nb₅Si₃ phase can be observed in the Ni-containing Nb-22Si-20Ti alloys, which is attributed to peritectic reaction $Nb_3Si+\beta$ -Nb₅Si₃ → α -Nb₅Si₃ in hypereutectic Nb-Si alloys. Content of element Ni in Nbss is the lowest of 0.79at.%, while that in Nb₃Si phase is 1.52at.% and that in α -Nb₅Si₃ phase is the highest of 1.62at.%.

NST-2Ni alloy with addition of 2at.% Ni is mainly consisted of Nbss phase, Nb₃Si phase and α -Nb₅Si₃ phase, and some black Ti₂Ni phase can also be observed in NST-2Ni alloy. Amount of Nb₃Si phase in the alloy is the highest, and Nbss phase with shape of long strip or sphere is distributed between Nb₃Si phases. Content of element Ni in Nbss is the lowest of 1.26at.%, while that in Nb₃Si phase is 1.97at.% and that in α -Nb₅Si₃ phase is the highest of 2.11at.%.

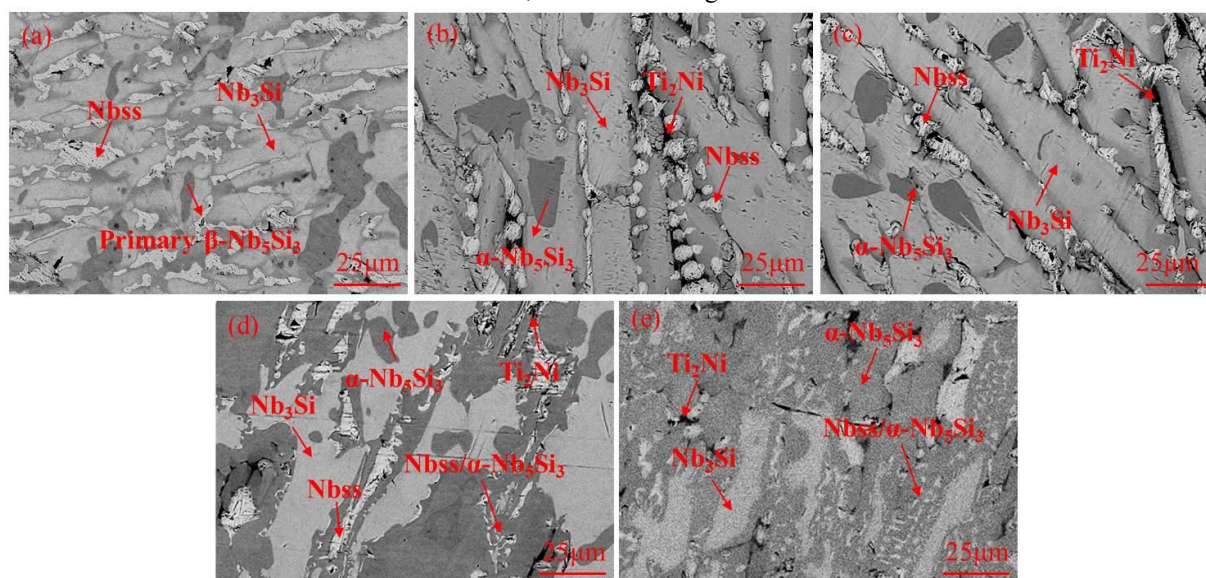


Fig.2 Microstructure of the Nb-22Si-20Ti-xNi alloys
(a) NST-0Ni; (b) NST-1Ni; (c) NST-2Ni; (d) NST-3Ni; (e) NST-4Ni

Table 1. Compositions of different constituent phases in Nb-22Si-20Ti-xNi alloys

Alloy	Phase	Chemical compositions (at.%)			
		Nb	Si	Ti	Ni
NST-0Ni	Nbss	69.39	3.06	27.55	—
	Nb ₃ Si	59.24	25.63	15.13	—
	β -Nb ₅ Si ₃	48.11	36.33	15.56	—
NST-1Ni	Nbss	73.56	2.11	23.54	0.79
	Nb ₃ Si	57.34	24.65	16.49	1.52
	α -Nb ₅ Si ₃	48.42	34.43	15.53	1.62
	Ti ₂ Ni	35.23	1.63	43.86	19.28

NST-2Ni	Nbss	70.62	2.39	25.73	1.26
	Nb ₃ Si	56.14	24.81	17.08	1.97
	α -Nb ₅ Si ₃	48.66	34.12	15.11	2.11
	Ti ₂ Ni	38.15	2.16	40.46	19.23
NST-3Ni	Nbss	70.38	2.92	25.01	1.69
	Nb ₃ Si	57.32	21.94	17.61	3.13
	α -Nb ₅ Si ₃	47.08	34.11	15.03	3.78
	Ti ₂ Ni	32.78	2.02	44.11	21.09
NST-4Ni	Nbss	70.88	2.67	24.64	1.81
	Nb ₃ Si	51.98	23.74	19.99	4.29
	α -Nb ₅ Si ₃	48.05	31.16	15.87	4.92
	Ti ₂ Ni	35.85	3.12	40.47	20.56

NST-3Ni alloy with addition of 3at.% Ni is also mainly consisted of Nbss phase, Nb₃Si phase, α -Nb₅Si₃ phase and a small amount of black Ti₂Ni phase. However, the microstructure morphology is significantly different from NST-0Ni alloy, NST-1Ni alloy and NST-2Ni alloy. The amount of α -Nb₅Si₃ phase is obviously increased and the distribution of α -Nb₅Si₃ phase is much more continuous. Nbss phase is surrounded by α -Nb₅Si₃ phase, and fine eutectic structure consisted of Nbss phase and α -Nb₅Si₃ phase is formed in the alloy. Content of element Ni in α -Nb₅Si₃ phase of NST-3Ni alloy is the highest of 3.78at.%, that in Nb₃Si phase is 3.13at.%, and that in Nbss is the lowest of 1.69at.%.

Microstructure of NST-4Ni alloy with addition of 4at.% Ni is similar to that of NST-3Ni alloy, and Nbss phase is also surrounded by α -Nb₅Si₃ phase. However, fine eutectic structure consisted of Nbss phase and α -Nb₅Si₃ phase is formed in NST-4Ni alloy, and the amount of fine eutectic structure is obviously increased. The amount of Nb₃Si phase is reduced and its distribution is not continuous at all. Content of element Ni in α -Nb₅Si₃ phase of NST-3Ni alloy is the highest of 4.92at.%, that in Nb₃Si phase is 4.29at.%, and that in Nbss is the lowest of 1.81at.%.

Above all, addition of Ni into Nb-22Si-20Ti alloy can lead to phase transformation from β -Nb₅Si₃ phase to α -Nb₅Si₃ phase and formation of Ti₂Ni phase. Furthermore, element Ni first solubilizes in silicide, and the content of Ni in α -Nb₅Si₃ is the highest, that in Nbss phase is the lowest. Fine eutectic structure consisted of Nbss phase and α -Nb₅Si₃ phase is formed in NST-3Ni alloy and NST-4Ni alloy.

3.3 Mechanical properties

3.3.1 Microhardness

Microhardness of silicide phase and Nbss phase in Nb-22Si-20Ti-xNi ($x=0, 1, 2, 3, 4$ at.%) alloys with

different addition of Ni are listed in Table 2. Results show that microhardness of silicide phase and Nbss phase increase with increases with increasing of Ni, and that of silicide phase is further bigger than that of Nbss phase in different alloys. Microhardness of silicide phase in NST-0Ni alloy is 1298.5 HV, and microhardness of Nbss phase in NST-0Ni alloy is 645.5 HV. By addition of 4at.%Ni into Nb-22Si-20Ti alloy, microhardness of silicide phase in NST-4Ni alloy is increased to 1450.1HV, which is increased by 10.5% compared to that in NST-0Ni alloy. Meanwhile, microhardness of Nbss phase in NST-4Ni alloy is increased to 718.3HV, which is increased by 11.2% compared to that in NST-0Ni alloy.

Table 2 Microhardness of Nb-22Si-20Ti-xNi alloys

Alloy	Silicides	Nbss
NST-0Ni	1298.5	645.5
NST-1Ni	1321.9	664.3
NST-2Ni	1380.7	684.8
NST-3Ni	1407.7	703.8
NST-4Ni	1450.1	718.3

Research by Sankar et al ^[27] showed that addition of 3at.%Ni into Nb-16Si alloy can lead to microhardness increase of Nb₃Si phase from 924 HV to 1578 HV and that of Nbss phase from 454 HV to 582 HV. Sankar et al found that content of Ni in Nb₃Si phase and Nbss phase are increased from 0 to 6.4at.% and 1.1at.% respectively when addition of Ni into Nb-16Si alloy is increased from 0 to 3at.%Ni, which can provide strong solid solution strengthening effect, and consequently lead to microhardness increase of Nb₃Si phase and Nbss phase. As for Nb-22Si-20Ti alloy, content of Ni in Nbss phase is obviously increased, while that of other elements is not obviously changed according to EDS results in Table 1, which shows that addition increase of Ni into Nb-22Si-20Ti alloy can increase content of Ni dissolved

in Nbss phase and improve the solid solution strengthening effect of alloys. Furthermore, element Ni dissolved in silicide phase is higher than that in Nbss phase, therefore that the solid solution strengthening effect caused by addition of Ni in silicide phase is much stronger.

3.3.2 Compression properties

Fig.3 shows compressive results for Nb-22Si-20Ti-xNi alloys with different addition of Ni which were conducted at room temperature. Results show that compressive strength of the alloys exhibits an increase trend with increasing of Ni added into Nb-22Si-20Ti-xNi alloys, and the fracture strain first decrease then increase with increasing of Ni added into Nb-22Si-20Ti-xNi alloys. Compressive strength of NST-0Ni alloy is 1972.5 MPa and the fracture strain is 7.5%, and that for NST-1Ni alloy is increased to 2085.1 MPa which is increased by 5.7% compared to that for NST-0Ni alloy, and the fracture strain is reduced to 7.4%. Compressive strength of NST-2Ni alloy is increased to the highest of 2295.9 MPa which is increased by 16.4% compared to that for NST-0Ni alloy, and the fracture strain is reduced to the lowest of 7.2%. Compressive strength of NST-3Ni alloy is slightly decreased to 2244.8 MPa compared to that for NST-2Ni alloy, and the fracture strain is 7.3%. Compressive strength of NST-4Ni alloy is slightly decreased to 2156.6 MPa compared to that for NST-2Ni alloy, and the fracture strain is slight increased to 7.5%. Wang et al [28] reported that compressive strength of Nb-16Si-24Ti alloy was 1912 MPa and that of Nb-16Si-24Ti-0.5Ru was up to 2012 MPa. However, compressive strength of alloys in the present study is slightly higher than that of the above alloys.

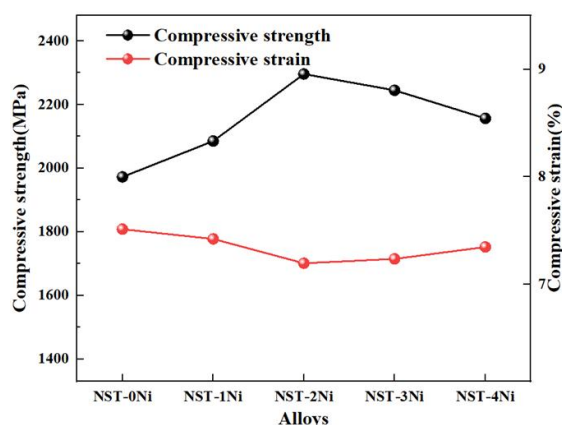


Fig. 3 Compression strength and compressive strain of alloys with different Ni contents

Addition of Ni into Nb-22Si-20Ti alloy can improve

compressive strength, but reduce fracture strain of alloys. When addition of Ni into Nb-22Si-20Ti alloy is increased from 0 to 2at.%, the compressive strength of alloy is increased for solid solution strengthening effect caused by addition of Ni, but the fracture strain is decreased. When addition of Ni into Nb-22Si-20Ti alloy is increased to 3at.% and 4at.%, compressive strength of NST-3Ni and NST-4Ni alloy is reduced compared with that of NST-2Ni alloy. The reduction of compressive strength for NST-3Ni and NST-4Ni alloy is attributed to the brittle Ti_2Ni phases with low strength [29] which are distributed along the grain boundaries. And the cracks always initiate at the interface between Ti_2Ni phases and Nb_3Si phase or $\alpha-Nb_5Si_3$ phase, and therefore reduce strength of NST-3Ni and NST-4Ni alloy.

3.3.3 Fracture morphology

Fig.4 shows fracture morphology of Nb-22Si-20Ti-xNi alloys with different addition of Ni after compressive experiments at room temperature. Fracture surface of NST-0Ni alloy without any addition of element Ni exhibit smooth cleavage planes, tearing edges and some dimples, some river-like patterns can also be observed on the fracture surface (shown in Fig.4(a)). Cleavage planes on fracture surface of NST-1Ni alloy increase, while tearing edges and dimples on fracture surface of NST-1Ni alloy decrease (shown in Fig.4(b)). Cleavage planes on fracture surface of NST-2Ni alloy continue to increase, while tearing edges and dimples on fracture surface of NST-2Ni alloy decrease and the fracture surface is getting much flatter (shown in Fig.4(c)). Comparing with fracture surface of NST-2Ni alloy, cleavage planes on fracture surface of NST-3Ni alloy decrease, and tearing edges and dimples on fracture surface of NST-3Ni alloy increase and the fracture surface is getting much coarser (shown in Fig.4(d)). Cleavage planes on fracture surface of NST-4Ni alloy further decrease, and tearing edges and dimples on fracture surface of NST-4Ni alloy further increase and the fracture surface is the coarsest of all (shown in Fig.4(e)). Fracture surface of Nb-22Si-20Ti-xNi alloys with different addition of Ni are mainly consisted of massive cleavage planes and tearing edges, which indicates that the fracture mode of the alloys are quasi cleavage fracture. The area of cleavage planes on fracture surface of the alloys first increase then decrease with increasing of Ni content, while tearing edges and dimples on fracture surface of the alloys first decrease then increase with increasing of Ni content.

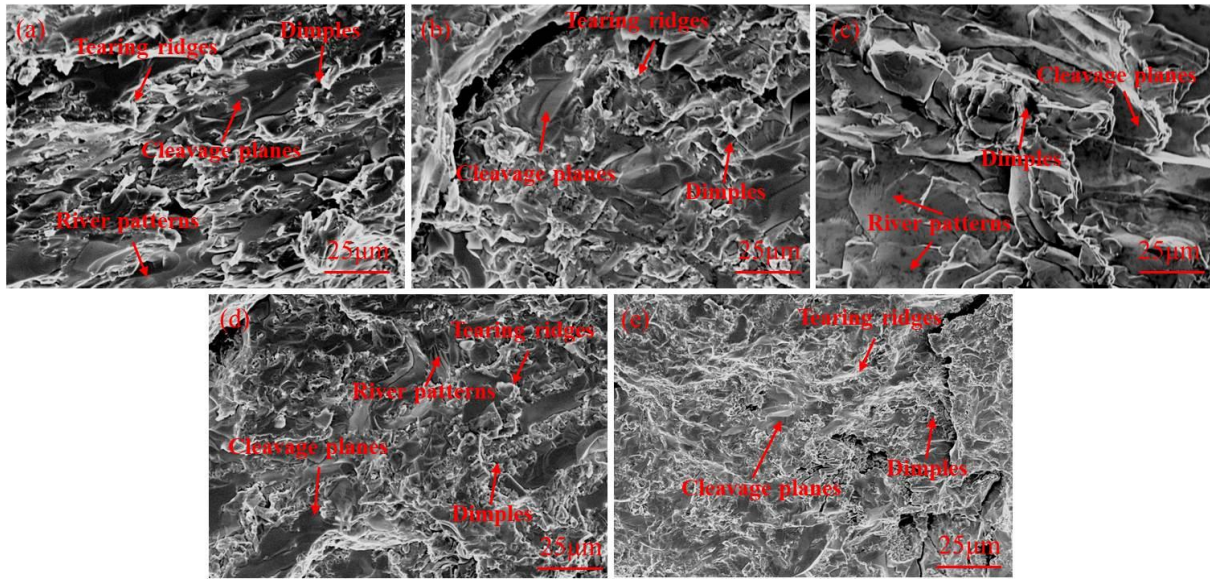


Fig. 4 Fracture morphology of alloys

(a) NST-0Ni; (b) NST-1Ni; (c) NST-2Ni; (d) NST-3Ni; (e) NST-4Ni

Fracture of silicide will form smooth cleavage planes during compressive tests of alloys, and some of the cleavage planes can exhibit river-like patterns which indicate that silicide can limit plasticity of alloys. Tearing edges on fracture surface of alloys indicate that Nbss solid solution can provide certain plastic deformation during compressive tests of alloys. Fracture strain of NST-1Ni alloy and NST-2Ni alloy is lower than that of NST-0Ni alloy for the solid solution strengthening effect, and therefore the area of cleavage planes on fracture surface of NST-2Ni alloy is the biggest and the fracture surface is the smoothest. Consequently, the fracture strain of NST-2Ni alloy is the smallest. As for NST-3Ni alloy and NST-4Ni alloy, fine eutectic structure consisted of Nbss phase and α -Nb₅Si₃ phase is formed in the alloys, and tearing edges and dimples on fracture surface of the alloys increases which can improve plasticity of alloys. Therefore, fracture strain of NST-3Ni alloy and NST-4Ni alloy is much higher than that of NST-2Ni alloy. However, the increase of fracture strain caused by formation of eutectic structure is not big enough to compensate for the decrease of fracture strain caused by the solid solution strengthening effect. Consequently, the fracture strain of NST-3Ni alloy and NST-4Ni alloy is still smaller than that of NST-0Ni alloy.

4 Conclusions

(1) Nb-22Si-20Ti alloy without any addition of Ni is consists of Nbss phase, β -Nb₅Si₃ phase and Nb₃Si phase. Addition of Ni can lead phase transformation from primary β -Nb₅Si₃ phase to α -Nb₅Si₃ and formation of Ti₂Ni phase in the alloys.

(2) Addition of Ni into Nb-22Si-20Ti alloy can improve microhardness of phases in the alloys. Microhardness of silicide increases from 1298.5 HV to 1450.1 HV and that of Nbss increases from 645.5 HV to 718.3 HV by increasing addition of Ni from 0 to 4at.%.

(3) Compressive strength of Nb-22Si-20Ti-xNi alloys first increase then decrease, while fracture strain decreases with increasing of Ni. Compressive strength of NST-2Ni alloy reaches the highest of 2295.9 MPa, which is improved by 16.4% compared with that of NST-0Ni alloy.

(4) Solid solution strengthening effect caused by addition of Ni can improve microhardness and compressive strength for Nb-22Si-20Ti-xNi alloys, but decrease fracture strain of alloys. In addition, formation of brittle and low strength Ti₂Ni phase will lead to decrease of strength.

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Conflicts of interest:

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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