

A probable new phase $H(Ni_2Ti_9Si_9)$ in Ti-Ni-Si ternary system at 1100 °C *

HU Xiaohua, CHEN Guoliang, NI Kequan

State Key Lab for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China

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Abstract: A probable new phase was detected in Ni-Ti-Si ternary system at 1100 °C. Its composition is around Ni:Ti:Si = 10:45:45 (atom fraction) according to the results of EPMA (Electron Probe Microanalysis) of 4 alloys containing the phase. The SEM microstructure difference of the 4 alloys can give some proof of its existence.

Key words: new phase, $H(Ni_2Ti_9Si_9)$ phase, microstructure, chemical composition

Six ternary phase (G, G', G'', F, V, E) was reported by J.H. Westbrook, et al^[1] in his Ni-Ti-Si 1000 °C isotherm about 40 years ago. The F phase whose composition is around NiTi was never reported latter, and the compositions and crystal structures of the other 5 were examined by latter investigators^[3-6]. Latter work on Ti-Si binary system proved the existence of Ti_5Si_4 up to 1570 °C^[2] which was not included in Westbrook's diagram^[1] and is in agreement with our work.

In our work of constructing Ti-Ni-Si 1100 °C isothermal section, a probable phase with the composition of $Ni_2Ti_9Si_9$ is found. This phase has small composition range and is around TiSi, Ti_5Si_4 , E, V. For convenience, it is designated as H in our discussion.

1 Experiment

Alloys were melted by vacuum ($\sim 10^{-3}$ Pa) arc furnace. The purity of the raw materials (mass fraction) is 99.99% Si, 99.9% Ti, and 99.99% Ni. The ingots were reverted and re-melted 3 times to ensure the homogeneity of the compositions. Every button was designed to be 15 g. The weight loss after being melted is less than 1% for all alloys. The nominal chemical compositions of alloys which contain this phase are shown in Table 1.

All the alloy buttons were sealed in quartz tubes that were evacuated to $10^{-3}\sim 10^{-2}$ Pa and filled with 4×10^3 Pa Ar gas to prevent them from oxidation and volatilization. Then they were annealed at 1100 °C for 72 h and water quenched. 7 mm high metallographic and EPMA (Electron Probe Microanalysis) specimens were cut

from the center of button. XRD powder specimens were prepared by grinding some of the rest part around the center. All observations were made at the center part of specimens, in order to eliminate the possible contamination from the surface section.

Table 1 The chemical compositions of alloys containing phase H (atom fraction) %

No.	Ti	Ni	Si	Phases
1	35	24	41	E, H
2	48	4	48	TiSi, Ti_5Si_4 , H
3	52	2	46	TiSi, Ti_5Si_4 , H
4	46	10	44	Ti_5Si_4 , E, H

Oxidation method was proved to be efficient in this system by J.H. Westbrook, et al^[1] and us to show metallographic microstructures. After grinding and polishing, all metallographic specimens were heated at 500 °C in air for about 2~10 min. Cambridge Steroscan 250 KII SEM was used to observe and take pictures of as-treated specimens. Microspec WDX (EPMA) was used to determine the composition of phases, where the standard specimens used were pure Ni, pure Ti and $TiSi_2$ compound.

XRD analysis was conducted at PHILIPS APD-10 X-ray diffractometer with 40 kV and 35 mA respectively. The wave-length $K\alpha_1$ of X-ray is 0.154 056 nm and the range of angle 2θ is $10^\circ\sim 110^\circ$.

2 Results

The phase identifications and electron probe microana-

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lysis results of the 4 alloys are listed in Table 2, and the SEM microstructures are shown in Fig. 1. The comparison of the partial isotherms between 1 000 °C⁽¹⁾ and 1 100 °C is shown in Fig. 2.

Table 2 Electron-probe microanalysis of specimens (atom fraction) %

Alloy number	Phases	Ni	Ti	Si
45	H	11.53	43.16	45.31
	E	28.09	34.41	37.49
	TiSi	0.23	49.69	50.08
24	Ti ₃ Si ₄	0.33	54.73	44.94
	H	10.19	44.72	45.08
	TiSi	0.22	50.22	49.56
26	Ti ₃ Si ₄	0.38	55.03	44.59
	H	9.99	45.63	44.38
	Ti ₃ Si ₄	0.65	54.49	44.87
28	E	27.34	35.04	37.63
	H	10.43	45.23	43.33

With the possible existence of this phase, the three-phase field (Ti₃Si₄ + TiSi + E) at 1 000 °C⁽¹⁾ (or more reasonably (Ti₃Si₄ + TiSi + E) from above) (as shown in Fig. 2(b)) will be precluded. As in Fig. 2(a), the alloy No. 24 lies near two-phase linkage line of H and TiSi, and the amount of Ti₃Si₄ phase is very small. The SEM microstructure picture Fig. 1(a) proves the above deduction to be true where there is considerable amount of TiSi and H phase. However, if H is looked as phase E, the amount should be very small. The alloy No. 26 as in Fig. 1(b) has a smaller H phase, as is consistent with Fig. 2(a). The alloy No. 28 lies in the E+H+Ti₃Si₄ phase field and is very close to H phase (as shown in Fig. 2(a)), and H becomes the main phase as shown in Fig. 1(c). It is proved by the microstructure of the two-phase alloy No. 45 (as shown in Fig. 1(d)) of the coexistence of phases E and H. Table 2 shows that the phase homogeneity range is small, for the H phase compositions of the 4 alloys are very close to Ni₂Ti₃Si₅.

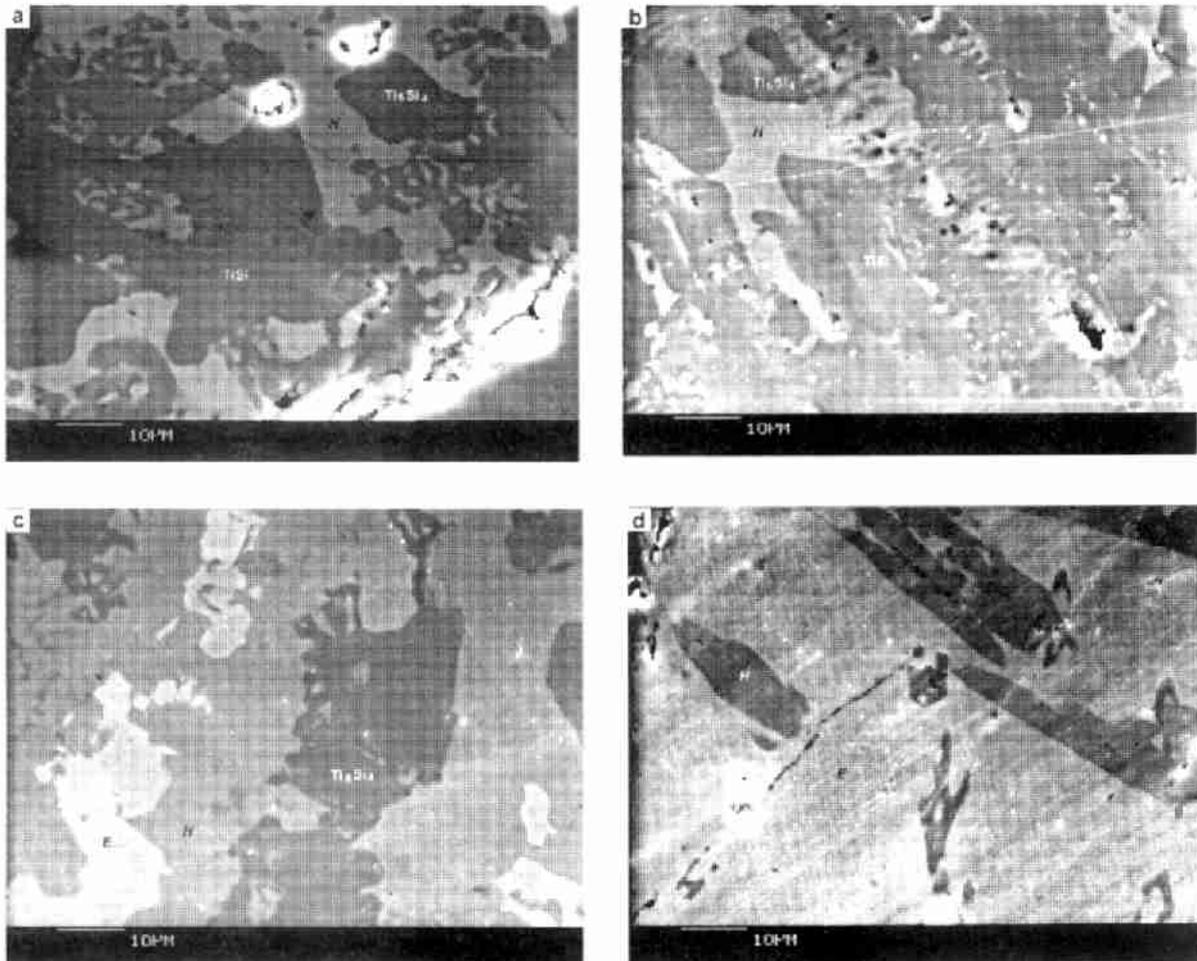


Fig.1 SEM picture of alloys with phase H ((a) No.24; (b) No.26; (c) No.28; (d) No.45)

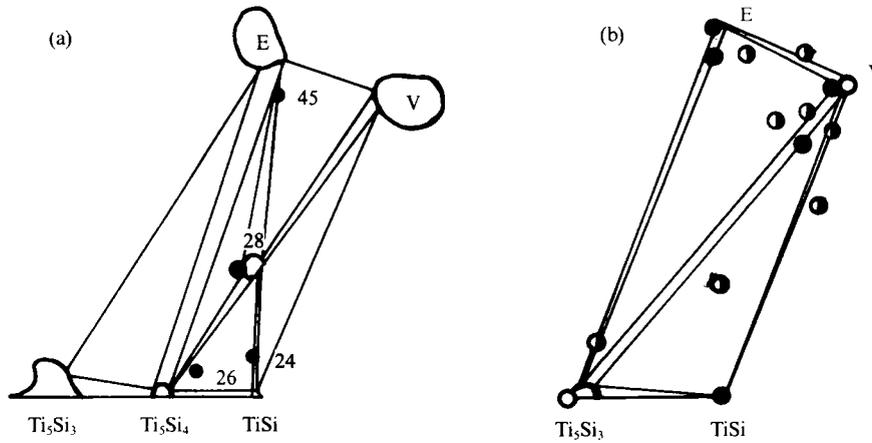


Fig.2 The comparison of partial isothermal section at 1000 °C⁽¹⁾ 1100 °C
 (a) 1100 °C isothermal section; (b) 1000 °C isothermal section

3 Conclusions

- (1) The phase H (Ni₂Ti₉Si₉) which was not reported before probably exists in Ni-Ti-Si system at 1100 °C;
- (2) The compositions of this phase are around Ni:Ti:Si =10:45:45 (atom fraction) and its composition range is small;
- (3) The existence of H has precluded the Ti₅Si₄-TiSi-E three-phase field and make the phase relations at this region more complicated.

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