

# DETERMINATION OF 900 °C ISOTHERMAL SECTION OF Ni-Ti-Si SYSTEM<sup>①</sup>

Xu Honghui and Jin Zhanpeng

*Department of Materials Science and Engineering,  
Central South University of Technology, Changsha 410083, P. R. China*

**ABSTRACT** A partial isothermal section of the Ni-Ti-Si system at 900 °C was determined by means of diffusion triple technique, optical microscopy and electron probe microanalysis (EPMA) technique. Experimental results showed that there existed three ternary compounds  $\text{TiNiSi}_2$  ( $\tau_2$ ),  $\text{Ti}_{14}\text{Ni}_{49}\text{Si}_{37}$  ( $\tau_3$ ) and  $\text{TiNiSi}$  ( $E$ ). Ternary compounds  $\text{Ti}_{53}\text{Ni}_{37}\text{Si}_{10}$  ( $\chi$ ) and  $\text{Ti}_2\text{Ni}_3\text{Si}$  ( $\lambda$ ), which were reported to occur at 750 °C, were not found at 900 °C. The solubilities of Ni in compounds  $\text{TiSi}_2$ ,  $\text{TiSi}$  and  $\text{Ti}_5\text{Si}_3$  are more than 10% (mole fraction).

**Key words** phase diagram phase equilibrium ternary system nickel titanium silicon

## 1 INTRODUCTION

Among the following binary systems, Ni-Ti<sup>[1-2]</sup> and Si-Ti<sup>[2-3]</sup> are relatively simple and certain, while Ni-Si<sup>[4]</sup> is more complicated.

Little information for the ternary system Ni-Ti-Si is available. Only the isothermal section at 750 °C was comprehensively investigated by Markiv *et al*<sup>[5]</sup>, in which 6 ternary compounds,  $\text{TiNiSi}_2$  ( $\tau_2$ ),  $\text{Ti}_{14}\text{Ni}_{49}\text{Si}_{37}$  ( $\tau_3$ ),  $\text{TiNiSi}$  ( $E$ ),  $\text{Ti}_6\text{Ni}_{16}\text{Si}_7$  ( $T$ ),  $\text{Ti}_2\text{Ni}_3\text{Si}$  ( $\lambda$ ) and  $\text{Ti}_{53}\text{Ni}_{37}\text{Si}_{10}$  ( $\chi$ ) were involved. Later on, Williams *et al*<sup>[6]</sup> determined the isothermal section of the Ni-rich corner at 1000 °C, which included a ternary compound  $\text{Ti}_6\text{Ni}_{16}\text{Si}_7$  ( $T$ ). Takasugi *et al*<sup>[7]</sup> and Ochiai *et al*<sup>[8]</sup> also dealt with the phase equilibria of the Ni-rich corner. The purpose of the present work is to determine the isothermal section at 900 °C by means of diffusion triple technique<sup>[9]</sup>.

## 2 EXPERIMENTAL

6 mm × 6 mm × 12 mm bars of nickel (99.9%, mass fraction) and titanium (99.7%, mass fraction), and 6 mm × 12 mm × 12 mm

plates of silicon (99.99%, mass fraction) were used. Binary Ni-Ti diffusion couples were first prepared by diffusion welding at 900 °C for 48 h. One of the Ni-Ti couples and a Si block were ground, polished and cleaned, and then swathed together with tungsten wires to make a diffusion triple. Three well-prepared diffusion triples were sealed in a quartz capsule filled with high-purity argon, and annealed in a GK-2B type diffusion furnace at 900 °C for 348 h (the temperature was controlled within ±1 °C). Then the capsule was taken out from the furnace and cooled down at room temperature to prevent the triples from cracking. Finally the samples were ground and polished parallel to the diffusion direction for electron probe microanalysis (EPMA).

## 3 RESULTS AND DISCUSSION

Fig. 1 shows the electron micrographs of a Si-Ni-Ti triple annealed at 900 °C for 348 h. Table 1 lists the experimental data determined by EPMA.

In terms of the experimental data given in Table 1 and the data from literature<sup>[2-4,7]</sup>, the isothermal section of the Si-Ni-Ti system at 900 °C is constructed as Fig. 2.

① Received Dec. 2, 1996; accepted Feb. 27, 1996

**Table 1 Tie lines and tie triangles ( composition in mole fraction, %) determined by EPMA on Si-Ni-Ti triples**

TiNiSi <sub>2</sub> ( $\tau_2$ )		TiNiSi( $E$ )		Ti <sub>14</sub> Ni <sub>49</sub> Si <sub>37</sub> ( $\tau_3$ )		TiNiSi( $E$ )	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
43.22	27.24	36.23	30.64	35.81	47.28	32.59	37.63
43.93	29.72	35.10	33.14	34.74	47.90	31.07	37.68
41.85	31.55	33.75	35.12	33.50	49.84	29.57	37.93
TiNiSi <sub>2</sub> ( $\tau_2$ )		TiSi		TiNiSi <sub>2</sub> ( $\tau_2$ )		Ti <sub>14</sub> Ni <sub>49</sub> Si <sub>37</sub> ( $\tau_3$ )	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
47.54	23.25	49.30	14.03	47.12	33.33	41.14	42.98
45.28	24.72	48.21	12.88	42.27	35.57	38.48	44.84
44.15	26.05	47.13	14.12	41.67	35.11	37.05	46.10
TiNiSi( $E$ )		Ti <sub>5</sub> Si <sub>3</sub>		Ti <sub>2</sub> Ni		(Ti)	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
32.71	31.75	38.08	13.78	19.59	1.61	10.06	2.63
32.30	32.46	35.43	13.27	21.19	1.35	9.23	3.65
31.52	32.78	32.80	17.31	18.16	3.66	10.14	4.04
TiNiSi <sub>2</sub> ( $\tau_2$ )		TiSi <sub>2</sub>		Ni <sub>2</sub> Si		Ti <sub>14</sub> Ni <sub>49</sub> Si <sub>37</sub> ( $\tau_3$ )	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
55.01	20.19	62.60	9.14	34.28	58.90	35.70	48.58
49.30	23.31	59.68	11.10	32.82	61.96	35.08	49.65
Ti <sub>5</sub> Si <sub>3</sub>		Ti <sub>3</sub> Si		Ti <sub>5</sub> Si <sub>4</sub>		Ti <sub>5</sub> Si <sub>3</sub>	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
35.64	0.52	25.43	0.48	44.09	0.05	38.15	0.29
35.72	0.97	23.52	2.06	44.13	0.03	37.86	0.24
Ti <sub>2</sub> Ni		TiNi		Ti <sub>2</sub> Ni		(Ti)	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
0.45	33.26	0.86	48.04	0.33	31.93	1.03	9.67
1.33	32.87	0.97	48.34	1.04	31.55	1.47	8.52
TiNi		TiNi <sub>3</sub>		TiSi		TiSi <sub>2</sub>	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
0.95	52.6	1.32	72.29	50.68	0.16	65.84	0.13
1.11	51.92	2.06	71.78				
TiNi <sub>3</sub>		(Ni)		Ti <sub>5</sub> Si <sub>3</sub>		Ti <sub>2</sub> Ni	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
0.53	75.66	2.12	87.37	31.91	8.66	4.16	28.61
Ni <sub>3</sub> Si <sub>2</sub>		Ti <sub>14</sub> Ni <sub>49</sub> Si <sub>37</sub> ( $\tau_3$ )		Ti <sub>5</sub> Si <sub>4</sub>		TiSi	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
37.02	57.86	37.04	48.19	44.87	0.05	49.02	0.02
(Ni)		Ni <sub>3</sub> Si		TiNi		Ti <sub>2</sub> Ni	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
7.09	84.53	10.64	77.75	1.55	47.82	3.16	31.09
TiNi		TiNi <sub>3</sub>		TiNiSi( $E$ )		TiSi	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
2.23	51.25	4.65	68.91	26.04	39.58	43.87	25.70
TiNiSi <sub>2</sub> ( $\tau_2$ )		Ti <sub>14</sub> Ni <sub>49</sub> Si <sub>37</sub> ( $\tau_3$ )		TiNiSi( $E$ )		TiSi	
Si	Ni	Si	Ni	Si	Ni	Si	Ni
41.10	32.53	35.92	46.63	33.30	36.86	1.75	74.62
						6.52	82.75
						9.80	77.35

Notes: (1) (Ni) and (Ti) denote the solid solutions of Ni and Ti respectively.

(2) The formulae for the three ternary compounds  $\tau_2$ ,  $\tau_3$  and  $E$  are quoted from Ref. [5].



**Fig. 1** Electron micrograph in Si-Ti side(a) ;  
**Electron micrograph near three-phase juncture of  $Ti_2Ni$ ,  $TiNi$  and  $Ti_5Si_3$ (b) ;**  
**Electron micrograph near three-phase juncture of  $TiNi$ ,  $TiNi_3$  and  $TiNiSi$ (c)**

It is to be noted that the initial annealing time for triples was 624 h. During the annealing period, the reaction rate between Ni and Si was so higher than that between Ti and Si that the product volume in the Si-Ni side was too large to develop even and continuous phases, and the sequence of phase distribution was broken when the diffusion triples underwent such a long period of annealing. The massive phases produced in samples were dominated by  $TiNiSi_2$  ( $\tau_2$ ),  $Ti_{14}Ni_{49}Si_{37}$  ( $\tau_3$ ) and  $TiNiSi$  ( $E$ ). Hence, the annealing time was modified to 348 h. The first group of data were used as the supporting data for the second group.

Markiv *et al*<sup>[5]</sup> reported six stable ternary compounds, i. e.  $TiNiSi_2$  ( $\tau_2$ ),  $Ti_{14}Ni_{49}Si_{37}$  ( $\tau_3$ ),  $TiNiSi$  ( $E$ ),  $Ti_6Ni_{16}Si_7$  ( $T$ ),  $Ti_2Ni_3Si$  ( $\lambda$ ) and  $Ti_{53}Ni_{37}Si_{10}$  ( $\chi$ ) (the annealing condition of alloys was 750 °C, 1000 h). The present experimental information indicated that no new ternary compounds occurred, and two of the ternary compounds  $Ti_{53}Ni_{37}Si_{10}$  ( $\chi$ ) and  $Ti_2Ni_3Si$  ( $\lambda$ ) didn't form at 900 °C (referring to Figs. 1(b) and (c)). Three of the ternary compounds,  $TiNiSi_2$ ,  $Ti_{14}Ni_{49}Si_{37}$  and  $TiNiSi$  were found, and their phase scales were large enough for their determination. Unlike the experimental

results by Markiv *et al*,  $Ti_2Ni$ ,  $TiNi$  and  $Ti_5Si_3$  were in a three-phase equilibrium; so were  $TiNi$ ,  $TiNi_3$  and  $TiNiSi$ . The discrepancies between the experimental results of Markiv *et al* and ours could result from the temperature difference.

Williams *et al*<sup>[6]</sup> confirmed the existence of  $Ti_6Ni_{16}Si_7$  at 1 000 °C. Perhaps, under the condition of 900 °C, it was produced, but had no distinct phase boundaries with the surrounding phases, hence was not recognized.

The experimental results presented in Fig. 2 show that the phase areas of the ternary compounds  $TiNiSi_2$  ( $\tau_2$ ),  $Ti_{14}Ni_{49}Si_{37}$  ( $\tau_3$ ) and  $TiNiSi$  ( $E$ ) are substantially larger than those proposed by Ref. [5], and the solubilities of Ni in compounds  $TiSi_2$ ,  $TiSi$  and  $Ti_5Si_3$  are more than 10% (mole fraction) respectively. The phase zone of  $Ti_{14}Ni_{49}Si_{37}$  ( $\tau_3$ ) obviously extends parallel to the boundary binary system Ni-Si. It can be assumed that Ti and Ni reciprocally substitute each other in crystal lattice.

#### 4 SUMMARY

A partial isothermal section of the Ni-Ti-Si system at 900 °C was determined. Experimental

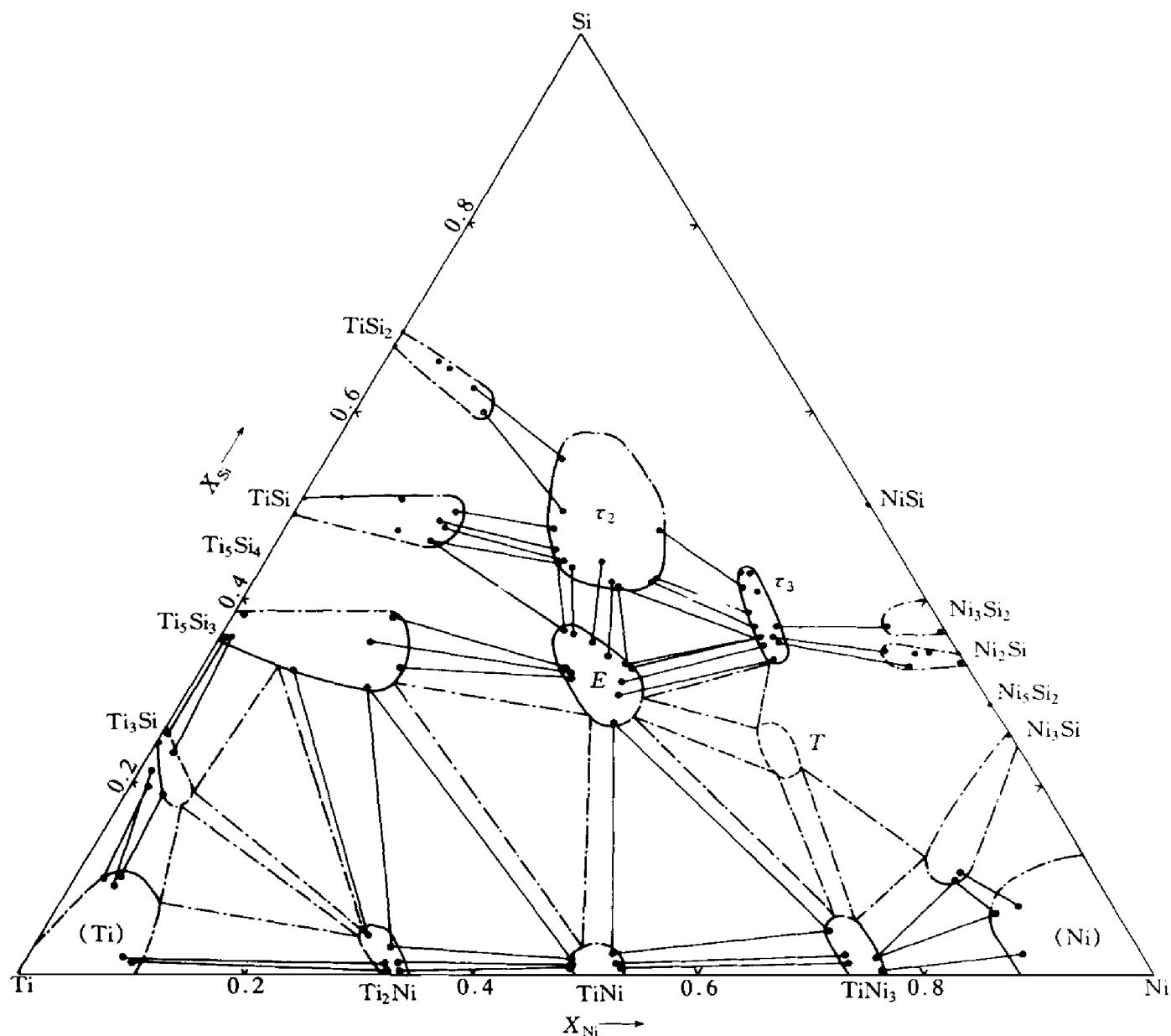


Fig. 2 Isothermal section of Ni-Ti-Si system at 900 °C

results showed that ternary compounds  $\text{Ti}_{57}\text{Ni}_{37}\text{Si}_{10}$  ( $\times$ ) and  $\text{Ti}_2\text{Ni}_3\text{Si}$  ( $\lambda$ ) didn't form, and whether or not they stably exist is to be confirmed. Three ternary compounds  $\text{TiNiSi}_2$  ( $\tau_1$ ),  $\text{Ti}_{14}\text{Ni}_{49}\text{Si}_{37}$  ( $\tau_3$ ) and  $\text{TiNiSi}$  ( $E$ ) were ascertained. The data on the ternary compound  $\text{Ti}_6\text{Ni}_{16}\text{Si}_7$  ( $T$ ) was lacking, and yet to be determined.

## REFERENCES

- 1 Liang H Y and Jin Z P. CALPHAD, 1993, 17(4): 415– 426.
- 2 Murray J L. Phase Diagrams of Binary Titanium Al-
- loys. OH: ASM International, Metal Park, 1987.
- 3 Seifeit Hans Jürgen, Lukas Hans Leo and Petzow Günter. Z Metallkd, 1996, 87(1): 2.
- 4 Nash P and Nash A. Bulletin of Alloy Phase Diagrams, 1987, 8(1): 6– 14.
- 5 Markiv B Ya, Gladyshevskii E I, Kripyakevich P I and Fedoruk T I. Izv Akad Nauk SSSR, Neorg Mater, 1966, 2(7): 1317– 1319.
- 6 Williams K J. J Inst Metals, 1971, 99, 310– 315.
- 7 Takasugi T, Shindo D, Izumi O and Hirabayashi M. Acta Metall Mater, 1990, 38(5): 739– 745.
- 8 Ochiai S, Oya Y and Suzuki T. Acta Metall Mater, 1984, 32(2): 289– 298.
- 9 Jin Z P. Scand J Metall, 1981, 10: 279– 287.

( Edited by peng Chaoqun )