Journal of Alloys and Compounds 479 (2009) 201–203

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

The isothermal section of the Pr–Ti–Si ternary system at 773 K

Yongzhong Zhan[a,*], Chunhui Li[a], Jingqi Liu[a], Yong Du[b], Honglou Mo[a]

[a] Key Laboratory of Nonferrous Metal Materials and New Processing Technology, Ministry of Education, Guangxi University, Nanning, Guangxi 530004, PR China
[b] State Key Laboratory of Powder Metallurgy, Central South University, Changsha, Hunan 410083, PR China

ARTICLE INFO

Article history:
Received 4 December 2008
Received in revised form 20 December 2008
Accepted 28 December 2008
Available online 10 January 2009

Keywords:
Metals and alloys
Phase diagrams
X-ray diffraction

ABSTRACT

The isothermal section of the Pr–Ti–Si ternary phase diagram at 773 K was investigated by powder X-ray diffraction (XRD) and differential thermal analysis (DTA). The binary compound Pr_3Si_4 is not observed at 773 K. There are nine binary compounds in this system, which are Ti_3Si, Ti_5Si_3, Ti_5Si_4, TiSi, $TiSi_2$, Pr_5Si_3, Pr_5Si_4, PrSi and $PrSi_2$. No ternary compounds were found in this work. The 773 K isothermal section of this ternary system consists of 12 single-phase regions, 21 two-phase regions and 10 three-phase regions. The compound Ti_5Si_3 has a homogeneity range extending from about 37 to 38 at.% Si. The existence of solid solubility of the other phases was not observed.

1. Introduction

An investigation of the Pr–Ti–Si ternary system is a part of a systematical study of the interaction of metals with titanium and silicon. Recently, we reported the phase equilibria of the La–Ti–Si [1], Zr–Ti–Si [2], Nb–Ti–Si [3] and Gd–Ti–Si [4] systems by means of experimental methods.

Ti–Si binary phase diagram [5] shows five intermediate phases, i.e. Ti_3Si, Ti_5Si_3, Ti_5Si_4, TiSi and $TiSi_2$ with high-melting points and tight homogeneity ranges. Morozkin [6] did not confirm that Ti_5Si_4 existed in the Dy–Ti–Si system at 1200 K. Pr–Si system has also been reported [7]. In Refs. [8–11], the intermetallic compound information of the crystal structure and crystallography in Pr–Si binary system was reported. The compounds existing in this system are Pr_5Si_3, Pr_5Si_4, PrSi, and $PrSi_2$. No intermetallic phase was found in Pr–Ti binary system. Structural data for the intermetallic compounds in the three binary systems are given in Table 1.

Up to now, no report on the isothermal section of the ternary Pr–Ti–Si system has been found. This study was carried out to construct the isothermal section of the Pr–Ti–Si phase diagram at 773 K, so as to establish the phase relationship and formation of the solid solutions in this system.

2. Experimental details

The purities of praseodymium, silicon and titanium used in this work are 99.58%, 99.99% and 99.99%, respectively. The alloys (each weighing 1.5 g) were prepared by arc-melting on a water-cooled copper crucible with a non-consumable tungsten electrode in high-pure argon atmosphere. The alloys were re-melted three times and turned around after melting for better homogeneity. For most alloys, the weight loss is less than 1% after melting.

Several typical ternary alloys with compositions that represent different parts of the isothermal section were examined through differential thermal analysis (DTA) to obtain the melting point. The homogenization temperature of the alloys were chosen on the basis of the binary phase diagrams of the Ti–Si, Ti–Pr and Pr–Si systems as well as the results of DTA. Then the samples were kept sealed in silica tubes in vacuum during homogenization. The alloys were homogenized at 1023 K for 480 h at first and then they were cooled down to 773 K and kept at this temperature for 240 h. Finally, all these annealed alloys were quenched in liquid nitrogen.

The quenched samples were pulverized, sealed in evacuated glass tube and annealed at 773 K for 4 days, followed by quenched into liquid nitrogen before X-ray diffraction (XRD) experiment. The XRD analysis was performed using a Rigaku D/Max 2500 V diffractometer with Cu Kα radiation, graphite monochromator, a voltage of 40 kV and a current of 200 mA. The materials data were analyzed by using JADE 5.0 software [13] and PCW (Powder Cell Windows software) [14]. A Powder Diffraction File (PDF release 2002) was used to determine the phase existence in each sample. DTA experiment was performed using a differential thermal analyzer (PerkinElmer). The samples were heated or cooled at a rate of 20 K/min during DTA.

3. Results and discussion

3.1. Intermetallic compounds

In this work, five binary compounds, i.e. Ti_3Si, Ti_5Si_3, Ti_5Si_4, TiSi and $TiSi_2$ have been confirmed in the Ti–Si binary system at 773 K. In the Pr–Ti binary system, no binary compounds were observed. The results obtained agree well with Ref. [5]. In the Pr–Si binary system, there are five binary compounds, i.e. Pr_5Si_3, Pr_5Si_4, PrSi, Pr_3Si_4 and $PrSi_2$ reported [7–11]. However, Pr_3Si_4 has not been observed at 773 K in this work, which agrees well with the results of Liu et al. [15]. In Fig. 1, the XRD pattern of the equilibrated binary alloy containing 42.86 at.% Pr and 57.14 at.% Si (Pr:Si = 3:4) indicates

* Corresponding author. Tel.: +86 771 3272311; fax: +86 771 3233530.
E-mail address: zyzmatres@yahoo.com.cn (Y. Zhan).

0925-8388/$ – see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2008.12.145

Table 1
Binary crystal structure data in the Pr–Ti–Si system.

Compound	Space group	Lattice parameters (nm)			Reference
		a	*b*	*c*	
Pr_5Si_3	*I*4/*mcm*	0.7814(5)	–	1.374(2)	[12]
Pr_5Si_4	$P4_12_12$	0.790		1.491	[12]
PrSi	*Pnma*	0.8240	0.3941	0.5920	[12]
$PrSi_2$	$I4_1/amd$	0.4205(5)	–	1.373(2)	[12]
Ti_3Si	$P4_2/n$	1.0196	–	0.5097	[6]
$TiSi_2$[a]	*Fddd*	0.8236(6)	0.4773(4)	0.8523(6)	[6]
$TiSi_2$[b]	*Cmcm*	0.361	1.377	0.365	[6]
Ti_5Si_3	$P6_3/mcm$	0.74610(3)	–	0.51508(1)	[6]
Ti_5Si_4	$P4_12_12$	0.7133	–	1.2977	[6]
TiSi[a]	*Pnma*	0.657	0.364	0.503	[6]
TiSi[b]	*Pmm*2	0.3618	0.6492	0.4973	[6]

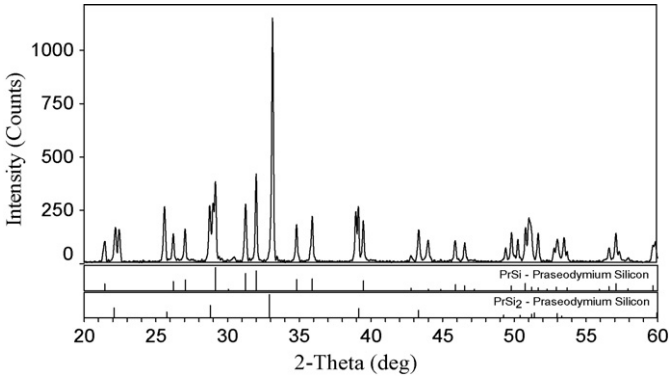

Fig. 1. XRD pattern of the equilibrated alloy (42.86 at.% Pr and 57.14 at.% Si) indicating the existence of $PrSi_2$ and PrSi.

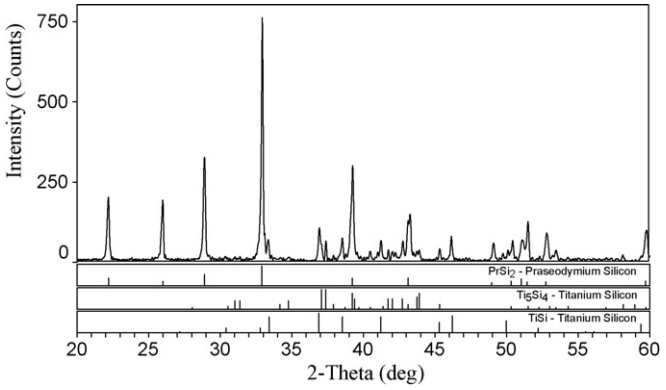

Fig. 3. XRD pattern of the equilibrated alloy (14 at.% Pr, 30 at.% Ti and 56 at.% Si) indicating the existence of Ti_5Si_4, TiSi and $PrSi_2$.

the existence of $PrSi_2$ and PrSi. Moreover, from the XRD analysis result of the equilibrated sample containing 36 at.% Pr, 14 at.% Ti and 50 at.% Si, it is clear that it consists of three phases, i.e. Ti_5Si_3, $PrSi_2$ and PrSi (as shown in Fig. 2). Both the above results indicate the nonexistence of Pr_3Si_4 phase in the Pr–Si system at 773 K.

In the previous works, no ternary compounds have been reported in this system. It is confirmed in this work that there was no ternary compound exists in the Pr–Ti–Si ternary system at 773 K.

3.2. Isothermal section

One hundred and fifty-two equilibrated samples have been prepared and analyzed to make clear the phase compositions. It is shown in Fig. 3 an example that the XRD pattern of the equilibrated sample containing 14 at.% Pr, 30 at.% Ti and 56 at.% Si consists of patterns of three phases, i.e. Ti_5Si_3, $PrSi_2$ and PrSi. Fig. 4 clearly shows that the equilibrated sample containing 12 at.% Pr, 28 at.% Ti and 60 at.% Si located in the three-phase region of $TiSi_2$, TiSi and $PrSi_2$. In Fig. 5, the XRD pattern of the equilibrated samples containing 14 at.% Pr, 46 at.% Ti and 40 at.% Si clearly indicates the existence of the three phases, i.e. Ti_5Si_3, PrSi and Pr_5Si_4.

The range of solid solubility for each phase at 773 K has been determined by XRD using the phase-disappearing method [16] and comparing the shift of the XRD pattern of the samples near the compositions of the binary phases. Three equilibrated samples ($Ti_{62}Si_{38}$, $Ti_{62.5}Si_{37.5}$ and $Ti_{63}Si_{37}$) have been analyzed by XRD, the results show that they all have only one phase with Mn_5Si_3 structure. It is obvious that they all belong to the extended region of Mn_5Si_3-type

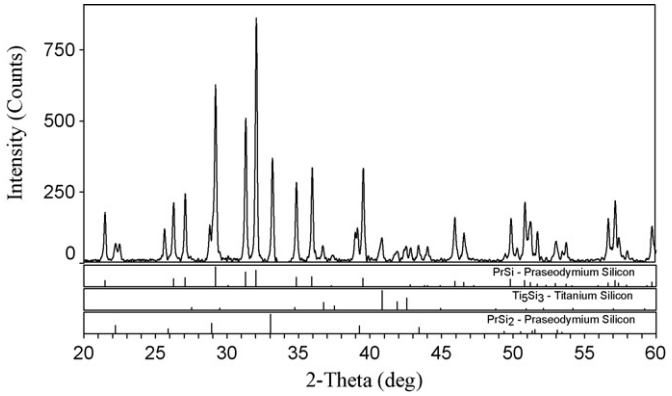

Fig. 2. XRD pattern of the equilibrated alloy (36 at.% Pr, 14 at.% Ti and 50 at.% Si) indicating the existence of Ti_5Si_3, $PrSi_2$ and PrSi.

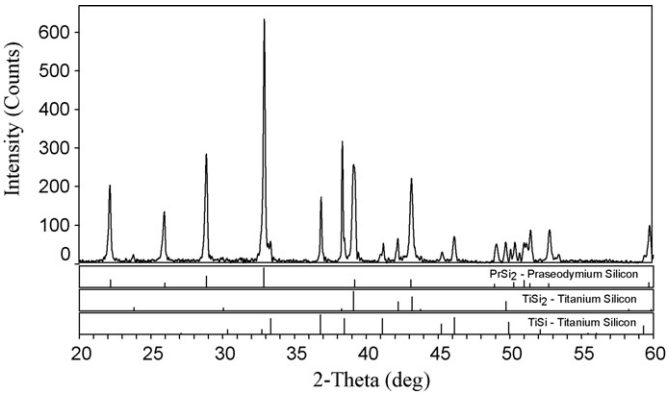

Fig. 4. XRD pattern of the equilibrated alloy (12 at.% Pr, 28 at.% Ti and 60 at.% Si) indicating the existence of $TiSi_2$, TiSi and $PrSi_2$.

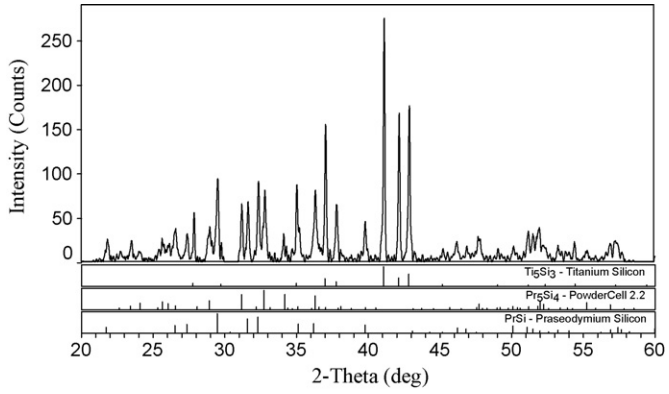

Fig. 5. XRD pattern of the equilibrated alloy (14 at.% Pr, 46 at.% Ti and 40 at.% Si) indicating the existence of Ti_5Si_3, PrSi and Pr_5Si_4.

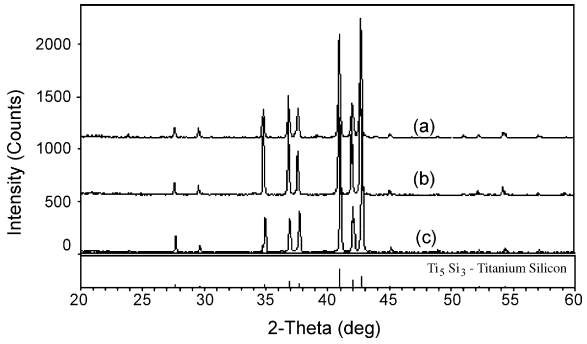

Fig. 6. The XRD patterns of some equilibrated Pr–Ti–Si ternary system samples: (a) 62 at.% Ti and 38 at.% Si; (b) 62.5 at.% Ti and 37.5 at.% Si%; and (c) 63 at.% Ti and 37 at.% Si.

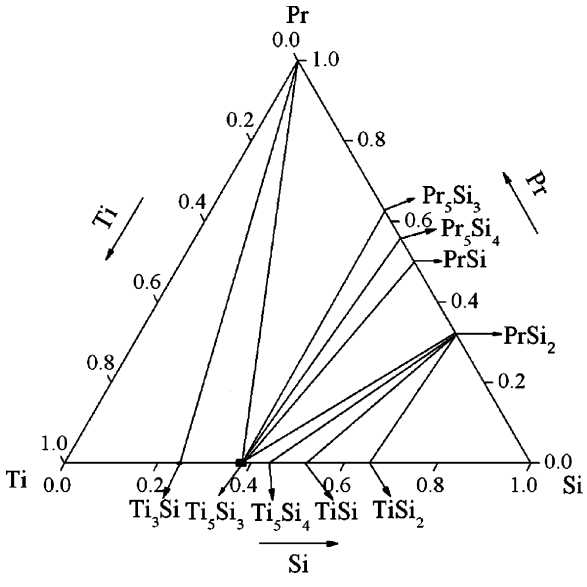

Fig. 7. The isothermal section of phase diagram of Pr–Ti–Si ternary system at 773 K.

Ti_5Si_3-based solid solution (shown in Fig. 6). The lattice parameters of the three alloys in the solid solution region of Ti_5Si_3 are listed in Table 2. However, another phase can be found in the samples when the chemical composition exceeds the range of 37–38 at.% Si, i.e.

Table 2
The lattice parameters of the alloys in the solid solution region of Ti_5Si_3.

Alloys	Space group	Lattice parameters (nm)		
		a	*b*	*c*
$Ti_{63}Si_{37}$	$P6_3/mcm$	0.74389(9)	–	0.51343(7)
$Ti_{62.5}Si_{37.5}$	$P6_3/mcm$	0.74597(5)	–	0.51541(4)
$Ti_{62}Si_{38}$	$P6_3/mcm$	0.74532(6)	–	0.51486(5)

Ti_3Si or Ti_5Si_4 was detected when Si content is lower than 37 at.% or higher than 38 at.%, respectively. Therefore, the above results show that the compound Ti_5Si_3 has a homogeneity range extending from about 37 to 38 at.% Si. The existence of solid solubility of the other phase was not observed.

Based on the experimental results, the phase relation of the ternary Pr–Ti–Si system at 773 K was determined. The isothermal section is shown in Fig. 7.

4. Conclusion

The phase equilibria of the ternary Pr–Ti–Si system at 773 K have been determined. The existence of the binary compound Ti_5Si_4 was confirmed. It is confirmed in this work that the binary compound Pr_3Si_4 does not exist in the Pr–Si binary system at 773 K. No ternary compound is found in the ternary system. The isothermal section consists of 12 single-phase regions, 21 two-phase regions and 10 three-phase regions. The compound Ti_5Si_3 has a homogeneity range extending from about 37 to 38 at.% Si. Solid solubility of the other phases was not detected.

Acknowledgements

The authors wish to express thanks to the financial support from the National Natural Science Foundation of China (50761003, 50831007), the Key Project of China Ministry of Education (207085) and the Opening Foundation of State Key Laboratory of Powder Metallurgy.

References

[1] Y. Zhan, Z. Yu, C. Li, Z. Sun, Y. Xu, Y. Wang, Y. Zhuang, J. Alloys Compd. 461 (2007) 128–131.
[2] Y. Zhan, J. Jiang, Z. Sun, J. Ma, C. Li, Z. Hu, J. Alloys Compd. (2008), doi:10.1016/j.jallcom.2008.06.156.
[3] Y. Zhan, Z. Sun, J. Jiang, J. Ma, X. Zhang, Y. Zhuang, J. Alloys Compd. (2008), doi:10.1016/j.jallcom.2008.01.009.
[4] Y. Zhan, J. Ma, Z. Sun, Z. Hu, Y. Du, J. Alloys Compd. (2008), doi:10.1016/j.jallcom.2008.07.098.
[5] T.B. Massalski, H. Okamoto, P.R. Subramanian, L. Kacprzak (Eds.), Binary Alloy Phase Diagrams, vol. 2, 2nd ed., The Materials Information Society, Materials Park, OH, 1990, pp. 3099–3101, 3105–3108, 3367–3371.
[6] A.V. Morozkin, J. Alloys Compd. 345 (2002) 155–157.
[7] L.A. Petrovoi, Diagramm' sostoyaniya Metallicheskikh system 31 (1987) 1 (in Russian).
[8] R.P. Elliott (Ed.), Constitution of Binary Alloys, 1965, pp. 745–746.
[9] M. Hansen (Ed.), Constitution of Binary Alloys, 2nd ed., 1958, p. 1134.
[10] F.A. Shunk (Ed.), Constitution of Binary Alloys, 1960, p. 620.
[11] K.A. Gschneidner, L. Eyring, Handbook on the Physics and Chemistry of Rare Earth, vol. 7, 1984, pp. 155–156.
[12] P. Villars, Pearson's Handbook of Crystallographic Data, Materials Park, OH, 1997, pp. 2695–2696.
[13] Materials Data Release 5, XRD Processing, Materials Data Inc. (MDI), 1999.
[14] Powder Cell for Windows, Version 2.4, 2000.
[15] J. Liu, X. Wei, J. Alloys Compd. 289 (1999) 178–180.
[16] Y. Zhan, Y. Du, Y. Zhuang, in: J. Zhao (Ed.), Methods for Phase Diagram Determination, Elsevier Science, Amsterdam, 2007, pp. 118–122.