

Phase relationships in the Pr–Fe–Sb system at 773 K

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Abstract

The isothermal section of the phase diagram of the Pr–Fe–Sb ternary system at 773 K has been investigated mainly by X-ray powder diffraction with the aid of metallographic analysis. Four ternary compounds Fe₄PrSb₁₂, FePrSb₂, Fe₁₃Pr₆Sb and FePrSb₃ have been confirmed and a new ternary compound PrFe₂Sb₂ was found in this system at 773 K. The isothermal section consists of 16 single-phase regions, 33 two-phase regions and 19 three-phase regions.

The homogeneity range of ϵ (FeSb) phase extended from about 44 at.% Sb to 46 at.% Sb. The solubilities for the other single-phase regions were not observed. The structure type for PrFe₂Sb₂ is Ga₂S₃-type (sp. gr. Cc (no. 9), $a = 0.60719$ nm, $b = 0.60867$ nm, $c = 1.33051$ nm, $\beta = 103.1^\circ$).

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1. Introduction

The thermoelectric material is a kind of function materials which transfer the thermal energy into electric energy or transfer the electric energy into thermal energy directly by the thermoelectric effect of materials. The applications of thermoelectric materials in electricity generators or refrigeration devices have many advantages: small, cheap, lightweight, and quiet. The transfer efficiency is proportional to the value of figure of merit ZT and $ZT = \sigma S^2 / \kappa$ (S is the Seebeck coefficient, σ the electrical conductivity, κ the thermal conductivity of the material and T is the temperature (K)) [1,2]. Recently, filled skutterudites have been identified as potential thermoelectric materials due to their reduced thermal conductivity and then more favorable results in large ZT [3,4]. The filled skutterudites correspond to the formula RT_4Pn_{12} , where R is Ba, Ca, Sr or rare earth atom (La, Ce, Pr, Nd, Sm, Eu, Yb), T is a transition metal (Fe, Ru, Co, Rh, Ir, Os,

...) and Pn is a pnictogen (P, As, Sb, ...). The Pr-filled skutterudite compound Fe₄PrSb₁₂ exists in the Pr–Fe–Sb ternary system. The investigation of the Pr–Fe–Sb ternary system can provide the information for preparation of high quality Pr-filled skutterudite compound and the information of the new phase in this system. In this paper we studied phase relationships in the Pr–Fe–Sb system at 773 K.

The phase diagrams of the Fe–Sb, Pr–Fe and Pr–Sb binary systems are reported [5]. Two intermetallic compounds FeSb and FeSb₂ exist in the Fe–Sb binary system. The FeSb intermetallic compound has a homogeneity range from about 44 at.% Sb to 46 at.% Sb at 773 K. Only one intermetallic compound Fe₁₇Pr₂ was found in the Pr–Fe binary system. Five intermetallic compounds Pr₂Sb, Pr₅Sb₃, Pr₄Sb₃, α PrSb and PrSb₂ were reported in Pr–Sb binary system. Three compounds Fe₄PrSb₁₂ [6], FePrSb₂ [7] and Fe₁₃Pr₆Sb [8] were early identified. The phase relation in the Pr–Fe–Sb system at 600 °C was reported, which showed the existence of another two compounds FePrSb₃ and Fe₂Pr₅Sb [9] just before our work finished. However, there are some disagreements between our work and those reported in Ref. [9]. We would like to report it in this paper.

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2. Experimental details

The purities of praseodymium, iron and antimony used in this work are 99.9%, 99.9% and 99.99%, respectively. The alloy buttons (each weighing 2 g) were prepared by arc melting using a non-consumable tungsten electrode and a water-cooled copper tray under an atmosphere of purified argon. All samples (total 73) were sealed in evacuated quartz tubes for homogenization heat treatment. The homogenization temperatures were chosen on the basis of the binary phase diagrams of the Pr–Sb, Pr–Fe and Fe–Sb systems. All samples were treated at 873 K for 250 h and then cooled to 773 K at a rate of 10 K/h. They were kept at 773 K for 300 h and finally quenched into liquid nitrogen.

The samples were powdered for X-ray diffraction (XRD) analysis. For the non-brittle samples, they were sealed in evacuated glass tube, annealed at 773 K for 4 days and then quenched into liquid nitrogen before XRD experiment. The powder of the brittle samples was directly used for X-ray diffraction analysis after grinding. The X-ray diffraction analysis was performed with powder using a Rigaku D/Max 2500 V diffractometer with a copper target, graphite monochromator, a voltage of 40 kV and a current of 200 mA. The XRD Data were analyzed using JADE 5.0 software [10], PCW (powder Cell Windows software) 2.4 [11], Pearson’s Handbook of crystallographic Data [12] and the Powder Diffraction File (PDF release 2002).

3. Results and discussion

3.1. The isothermal section of the Pr–Fe–Sb ternary system at 773 K

Experimental results show that the existence of five intermetallic compounds Pr_2Sb , Pr_5Sb_3 , Pr_4Sb_3 , αPrSb and PrSb_2 in the Pr–Sb binary system, two intermetallic compounds FeSb and FeSb_2 in the Fe–Sb binary system, and one intermetallic compound $\text{Fe}_{17}\text{Pr}_2$ in Pr–Fe binary system was confirmed, at 773 K, which is in a good agreement with those reported in Ref. [5]. We obtained five ternary compounds in the Pr–Fe–Sb system: the early known antimonides $\text{Fe}_4\text{PrSb}_{12}$, FePrSb_2 , $\text{Fe}_{13}\text{Pr}_6\text{Sb}$, FePrSb_3 and a new compound with the PrFe_2Sb_2 stoichiometry. Details of crystallographic data of the initial components and the compounds in the Pr–Fe–Sb ternary system are given in Table 1.

By comparing and analyzing the X-ray diffraction patterns of the samples, and identifying the existence of phases in each

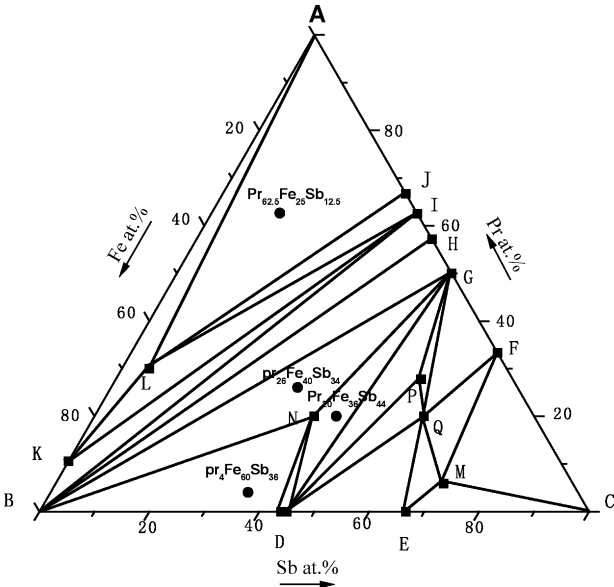


Fig. 1. The isothermal section of phase diagram of Pr–Fe–Sb ternary system at 773 K.

sample, we constructed the isothermal section of phase diagram of the Pr–Fe–Sb system at 773 K, as shown in Fig. 1, which consists of 16 single-phase regions, 33 two-phase regions and 19 three-phase regions.

The homogeneity range of ϵ (FeSb) phase is from about 44 at.% Sb to 46 at.% Sb, and the homogeneity range of the other phases was not observed.

3.2. Phase analysis

Chykhrij et al. [9] found the existence of the compound $\text{Pr}_5\text{Fe}_2\text{Sb}$ in Pr–Fe–Sb system at 873 K. However, we could not obtain this compound at 773 K. The X-ray diffraction pattern (XRDP) for the sample with composition of $\text{Pr}_5\text{Fe}_2\text{Sb}$ ($\text{Pr}_{62.5}\text{Fe}_{25}\text{Sb}_{12.5}$) shown in Fig. 2, consists of Pr, $\text{Fe}_{13}\text{Pr}_6\text{Sb}$

Table 1
Crystallographic data of the initial components and compounds in the Pr–Fe–Sb ternary system

Phase	Space group	Structure type	Lattice parameters (nm)				Reference
			<i>a</i>	<i>b</i>	<i>c</i>	$\beta(^{\circ})$	
A(Pr)	<i>P6₃/mmc</i>	La	0.36715	0.36715	1.1830		[12]
B(Fe)	<i>Im$\bar{3}$m</i>	W	0.29315	0.29315	0.29315		[12]
C(Sb)	<i>R$\bar{3}$m</i>	As	0.43084	0.43084	1.1274		[12]
D(FeSb)	<i>P6₃/mmc</i>	AsNi	0.407	0.407	0.513		[12]
E(FeSb ₂)	<i>Pnn2</i>	FeSb ₂	0.58328(5)	0.65376(5)	0.31973(3)		[12]
F(PrSb ₂)	<i>Cmca</i>	Sb ₂ Sm	0.626	0.612	1.816		[12]
G(PrSb)	<i>Fm$\bar{3}$m</i>	ClNa	0.6375	0.6375	0.6375		[12]
H(Pr ₄ Sb ₃)	<i>I4$\bar{3}$d</i>	Pa ₄ Th ₃	0.9458(1)	0.9458(1)	0.9458(1)		[12]
I(Pr ₅ Sb ₃)	<i>P6₃/mcm</i>	Mn ₅ Si ₃	0.923	0.923	0.651		[12]
J(Pr ₂ Sb)	<i>I4/mmm</i>	La ₂ Sb	0.455	0.455	1.782		[12]
K(Fe ₁₇ Pr ₂)	<i>R$\bar{3}$m</i>	Th ₂ Zn ₁₇	0.8585(5)	0.8585(5)	1.2464(8)		[12]
L(Fe ₁₃ Pr ₆ Sb)	<i>I4/mcm</i>	Co ₁₁ Ga ₃ La ₆	0.8108(1)	0.8108(1)	2.3303(7)		[12]
M(Fe ₄ PrSb ₁₂)	<i>Im$\bar{3}$</i>	Fe ₄ LaSb ₁₂	0.91351(2)	0.91351(2)	0.91351(2)		[12]
N(PrFe ₂ Sb ₂)	<i>Cc</i>	Ga ₂ S ₃	0.60719	0.60867	1.33051	103.1	This work
P(FePrSb ₂)	<i>P4/nmm</i>	CuSi ₂ Zr	0.43616(10)	0.43616(10)	0.97552(29)		[12]
Q(FePrSb ₃)	<i>Pbcm</i>	CeNiB ₃	1.251(8)	0.606(4)	1.848(9)		[9]

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