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The isothermal section of the Dy–Fe–Sb ternary system at 773 K

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Abstract

The isothermal section of the Dy–Fe–Sb ternary system at 773 K has been investigated mainly by X-ray powder diffraction in this work. The existence of eight binary compounds and one ternary compound were conformed in this system. The section consists of twelve single-phase regions, twenty-two two-phase regions and eleven three-phase regions. The compound Dy_4Sb_3 was not stable at 773 K. The homogeneity range of ε (FeSb) phase extended from about 44 at.% Sb to 46 at.% Sb and the maximum solid solubility of Sb in Fe phase is about 3 at.% Sb at 773 K.

Keywords: Rare earth alloys and compounds; Phase diagrams; X-ray diffraction

1. Introduction

The rare earth intermetallic compounds have been studied because of their excellent properties, for example, the compounds $Tb_{1-X}Dy_XFe_2$, $Tb_{0.3}Dy_{0.7}(Fe_{1-X}V_X)_2$, $Tb_{0.3}Dy_{0.7}(Fe_{1-X}Be_X)_2$ and $Tb_{0.36}Dy_{0.64}(Fe_{1-Y}Co_Y)_2$ with C15-type phase structure have giant magnetostrictive property [1–5]. The phase relationships of the RE–Fe–Sb (RE = La, Pr, Nd, Ho, Gd, Er and Yb) ternary systems were reported previously [6–10]. Up to the present, the phase diagram of the Dy–Fe–Sb ternary system has not been reported. The existence of compound Dy_4Sb_3 was disputed in Dy–Fe–Sb ternary system. In order to determine the existence of the phases and the phase relationships in the Dy–Fe–Sb system, to provide useful information for searching new materials, in this paper, we studied phase relationships in the Dy–Fe–Sb system at 773 K.

Ref. [11] reported the binary diagrams of the Dy–Fe, Dy–Sb and Fe–Sb systems. At 773 K, there are four intermetallic compounds Fe_2Dy, Fe_3Dy, $Fe_{23}Dy_6$ and $Fe_{17}Dy_2$ in the Dy–Fe binary system, three intermetallic compounds DySb, Dy_4Sb_3 and Dy_5Sb_3 in the Dy–Sb binary system, two intermetallic compounds FeSb and $FeSb_2$ in the Fe–Sb binary system. One ternary compound $FeDy_6Sb_2$ was reported in Ref. [12].

2. Experimental details

The purities of dysprosium, iron and antimony used in this work are 99.9%, 99.9% and 99.99%, respectively. The alloy buttons (each 2 g) were prepared by arc melting using a non-consumable tungsten electrode and a water-cooled copper tray under an atmosphere of pure argon. All samples (total 112) 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 Dy–Sb, Dy–Fe and Fe–Sb systems. The samples in the Sb-rich region were annealed at 853 K for 25 days. The other samples were annealed at 993 K for 25 days. Subsequently, the samples were cooled to 773 K at a rate of 9 K/h and kept at 773 K for 10 days (some samples were re-annealed were kept at 773 K another 10 days if necessary), then quenched into an ice water mixture.

The samples were powdered for X-ray diffraction (XRD) analysis. The non-brittle powder, were sealed in evacuated glass tube, annealed at 773 K for 4 days and then quenched into liquid nitrogen before XRD experiment. The brittle powder samples were directly used for XRD analysis after grinding. The X-ray

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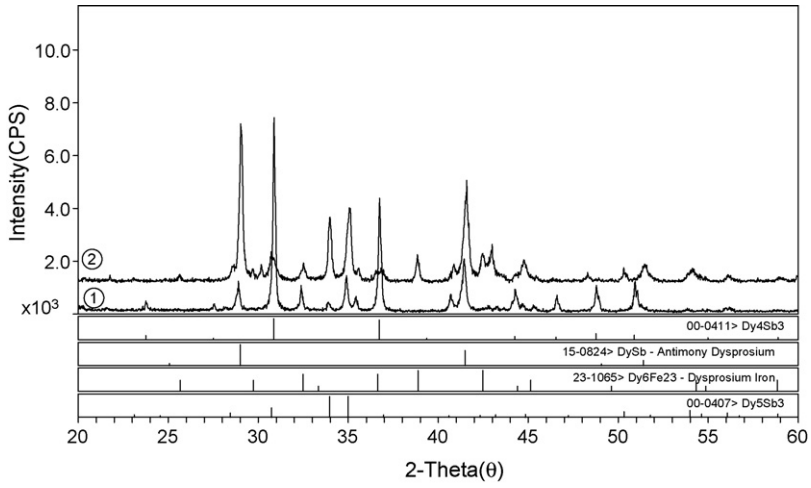

Fig. 1. XRDP of sample with composition $Fe_{54}Dy_{31}Sb_{15}$ under different annealed times: (①) XRDP of the samples which were only annealed at 773 K for 10 days: $Dy_4Sb_3 + Dy_5Sb_3 + DySb + Fe_{23}Dy_6$; (②) XRDP of the samples after annealing at 773 K for 10 days again: $Dy_5Sb_3 + DySb + Fe_{23}Dy_6$.

diffraction analysis was performed 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 softwares JADE 5.0 [13] and Powder Cell Windows (PCW 2.4) [14], Pearson's Handbook of crystallographic Data [15] and the Powder Diffraction File (PDF release 2002).

3. Results and discussion

3.1. Phase analysis

In this work, four binary compounds Fe_2Dy, Fe_3Dy, $Fe_{23}Dy_6$ and $Fe_{17}Dy_2$ were confirmed at 773 K in the Dy–Fe system. Two binary compounds FeSb and $FeSb_2$ have been confirmed in the Fe–Sb system at 773 K. The FeSb intermetallic compound has a homogeneity range from about 44 at.% Sb to 46 at.% Sb and the maximum solid solubility of Sb in Fe phase is about 3 at.% Sb at 773 K. The results were estimated by the disappearing method of the X-ray powder diffraction and were in good agreement with that reported in Ref. [11].

In the Dy–Sb system at 773 K, we confirmed the existence of compounds DySb and Dy_5Sb_3. Ref. [11] point out that the compound Dy_4Sb_3 is stable in the Dy–Sb system from 473 K to 2053 K. However Refs. [16–18] reported that compound Dy_4Sb_3 was not observed at 673 K, 1070 K or 1100 K. The X-ray diffraction patterns (XRDPs) for the samples near the composition of Dy_4Sb_3 showed that the compound Dy_4Sb_3 is present in some samples but absent in the others annealed at 773 K for 10 days. The intensities of XRDP of compound Dy_4Sb_3 were weaken markedly or disappeared in the same samples re-annealed at 773 K for another 10 days. The XRDP of the samples with composition of $Fe_{54}Dy_{31}Sb_{15}$ annealed at 773 K for different time are shown in Fig. 1. The XRDP for the samples annealed at 773 K for 10 days consists of the patterns of the Dy_4Sb_3, Dy_5Sb_3, DySb and $Fe_{23}Dy_6$ four phases. The XRDP for the same sample annealed at 773 K for another 10 days consists of the patterns of the Dy_5Sb_3, DySb and $Fe_{23}Dy_6$ three phases. It indicated that Dy_4Sb_3 may be decomposed into Dy_5Sb_3 and DySb for the annealing time long enough at 773 K. The XRDP of the sample with composition $Fe_{35}Dy_{41}Sb_{24}$ annealed at 773 K for 10 days, shown in Fig. 2, consists of Dy_5Sb_3, DySb and $Fe_{23}Dy_6$ three phases. These results indicated that the compound Dy_4Sb_3 should not be stable at 773 K. The decomposition of Dy_4Sb_3 was

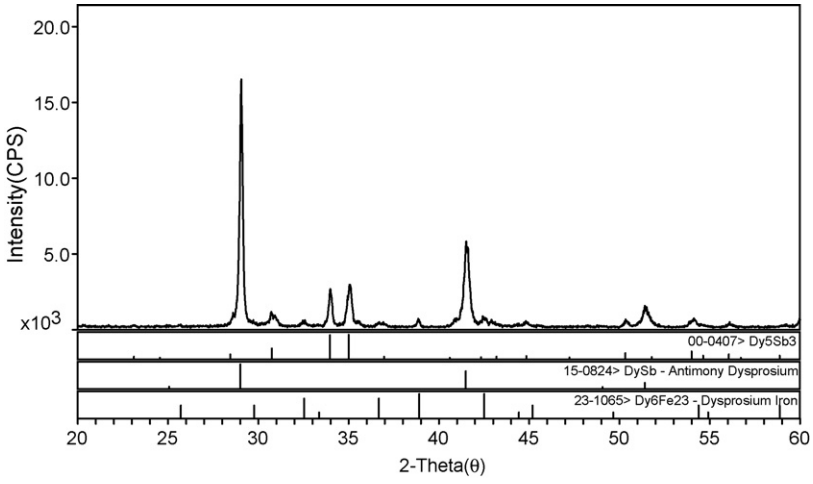

Fig. 2. XRDP of the sample with composition $Fe_{35}Dy_{41}Sb_{24}$: $Dy_5Sb_3 + DySb + Fe_{23}Dy_6$.

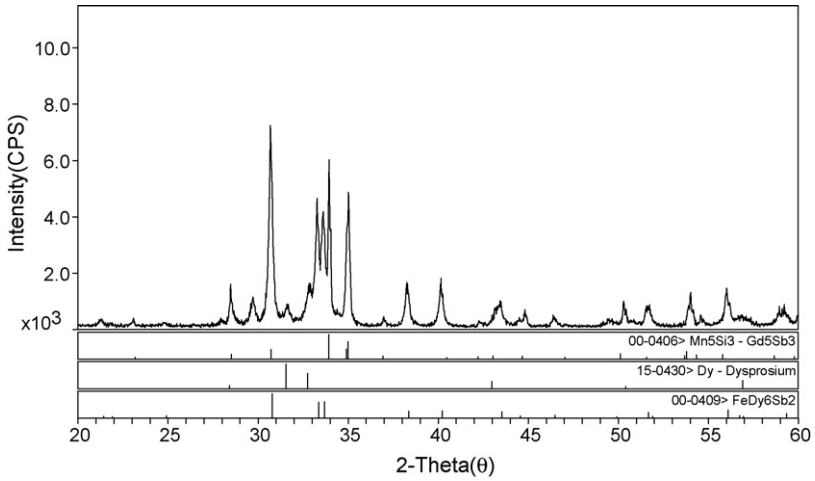

Fig. 3. XRDP of sample with composition $Fe_6Dy_{74}Sb_{20}$: Dy_5Sb_3 + Dy + $FeDy_6Sb_2$.

completely at 773 K for long enough time. The result is in good agreement with that reported in Refs. [16–18].

Ternary compound $FeDy_6Sb_2$ was confirmed in the Dy–Fe–Sb system at 773 K. The XRDP of the compound $FeDy_6Sb_2$ was observed in the XRDPs of samples with composition $Fe_6Dy_{74}Sb_{20}$ (Fig. 3, fourth phase in Fig. 3 is Dy_2O_3) and $Fe_{11}Dy_{65}Sb_{24}$ (Fig. 4). The crystallographic data of the initial components and compounds in the Dy–Fe–Sb ternary system is given in Table 1.

3.2. The isothermal section of the Dy–Fe–Sb ternary system at 773 K

By comparing and analyzing the X-ray diffraction patterns of all samples, and identifying the existence of phases in each sample, we constructed the isothermal section of the phase diagram of the Dy–Fe–Sb ternary system at 773 K. The isothermal section, shown in Fig. 5, consists of twelve single-phase regions, twenty-two two-phase regions and eleven three-phase regions. The twelve single-phase regions are: A (Dy), B (Fe_2Dy), C (Fe_3Dy), D ($Fe_{23}Dy_6$), E ($Fe_{17}Dy_2$), F (Fe), G (FeSb), H

Table 1
Crystallographic data of the initial components and compounds in the Dy–Fe–Sb system of 773 K

Phase	Space group	Structure type	attice parameters (nm)			References
			a	*b*	*c*	
αFe	$Im\bar{3}m$	W	0.29315	–	–	[15]
$Fe_{17}Dy_2$	$P6_3/mmc$	$Ni_{17}Th_2$	0.8453	–	0.8287	[15]
$Fe_{23}Dy_6$	$Fm\bar{3}m$	$Mn_{23}Th_6$	1.210	–	–	[15]
Fe_3Dy	$R\bar{3}m$	Be_3Nb	0.5125	–	2.4578	[15]
Fe_2Dy	$Fd3m$	Cu_2Mg	0.7328	–	–	[15]
αDy	$P6_3/mmc$	Mg	0.3458	–	0.5466	[15]
αDySb	$Fm3m$	NaCl	0.6154	–	–	[15]
Dy_5Sb_3	$P6_3/mcm$	Mn_5Si_3	0.8882	–	0.6270	[15]
Sb	$R\bar{3}m$	α-As	0.43084	–	1.1274	[15]
FeSb	$P6_3/mmc$	NiAs	0.407	–	0.513	[15]
$FeSb_2$	$Pnn2$	FeS_2	0.58328	0.65376	0.31973	[15]
$FeDy_6Sb_2$	$P\bar{6}2M$	$CuSi_2Zr$	0.81449	0.41641	0.23923	[12]

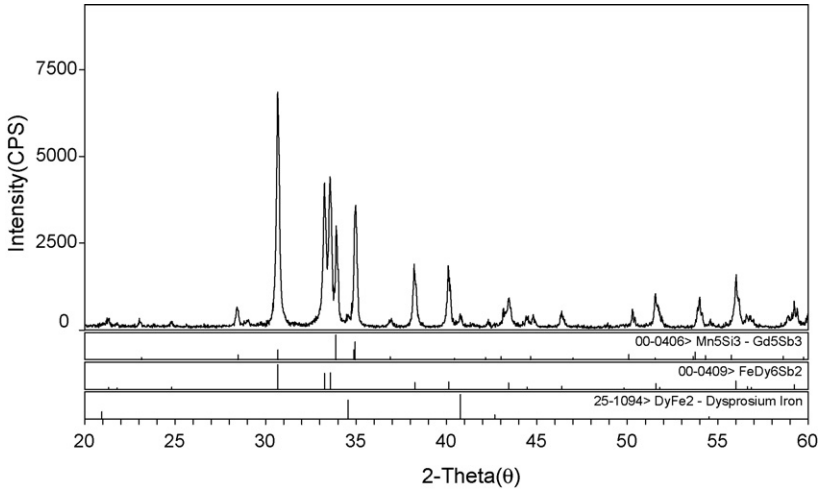

Fig. 4. XRDP of sample with composition $Fe_{11}Dy_{65}Sb_{24}$: Dy_5Sb_3 + Fe_2Dy + $FeDy_6Sb_2$.

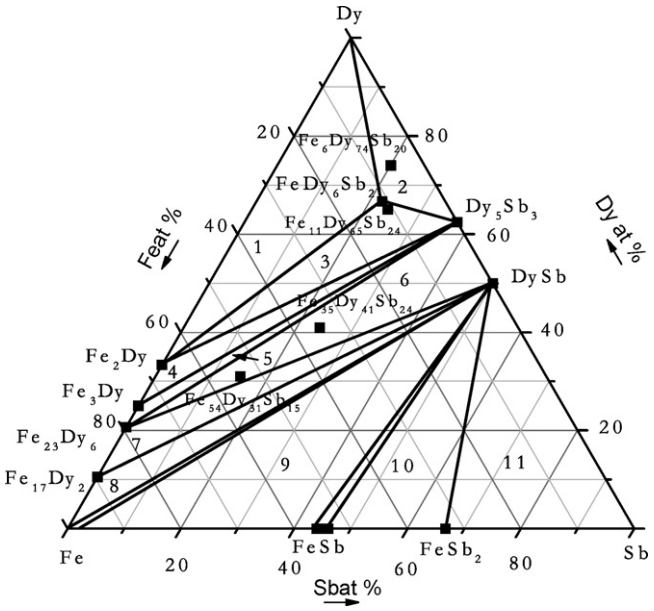

Fig. 5. The isothermal section of Dy–Fe–Sb ternary system at 773 K.

Table 2
Details of the three-phase regions in the Dy–Fe–Sb system of 773 K

Phase regions	Phase composition
1	$Dy + FeDy_6Sb_2 + Fe_2Dy$
2	$Dy + FeDy_6Sb_2 + Dy_5Sb_3$
3	$FeDy_6Sb_2 + Dy_5Sb_3 + Fe_2Dy$
4	$Fe_2Dy + Dy_5Sb_3 + Fe_3Dy$
5	$Fe_3Dy + Dy_5Sb_3 + Fe_{23}Dy_6$
6	$DySb + Fe_{23}Dy_6 + Dy_5Sb_3$
7	$Fe_{17}Dy_2 + Fe_{23}Dy_6 + DySb$
8	$DySb + Fe_{17}Dy_2 + Fe$
9	$DySb + Fe + FeSb$
10	$DySb + FeSb + FeSb_2$
11	$DySb + FeSb_2 + Sb$

($FeSb_2$), I (Sb), J (DySb), K (Dy_5Sb_3) and L ($FeDy_6Sb_2$). Twenty-two two-phase regions are: A + B, B + C, C + D, D + E, E + F, F + G, G + H, H + I, I + J, J + K, K + A, A + L, L + B, L + K, B + K, K + C, K + D, D + J, J + E, J + F, J + G and J + H. Eleven three-phase regions are: A + B + L, A + L + K, L + B + K, B + C + K, C + D + K, K + D + J, D + E + J, E + F + J, F + G + J, G + H + J and H + I + J. Details of three-phase regions are given in Table 2.

4. Conclusion

We have investigated and constructed the isothermal section of the Dy–Fe–Sb ternary system at 773 K. Eight binary compounds Fe_2Dy, Fe_3Dy, $Fe_{23}Dy_6$, $Fe_{17}Dy_2$, FeSb, $FeSb_2$ DySb and Dy_5Sb_3 and one ternary compound $FeDy_6Sb_2$ have been confirmed. The compound Dy_4Sb_3 was not stable at 773 K. The homogeneity range of FeSb is 44 at.% Sb–46 at.% Sb and the maximum solid solubility of Sb in Fe phase is about 3 at.% Sb at 773 K. The solid solubilities for the other single-phase regions were not observed. The isothermal section of the Dy–Fe–Sb ternary system at 773 K consists of twelve single-phase regions, twenty-two two-phase regions and eleven three-phase regions.

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