CRYSTAL STRUCTURE AND X-RAY POWDER DIFFRACTION DATA FOR RE COMPOUND HoNiSb①

Zeng, Lingmin Li, Jungqin Zhang, Liping Zhuang, Yinghong Hao, Jianmin*
Institute of Materials Science, Guangxi University, Nanning 530004
** Tianjin Electronic Materials Research Institute*

ABSTRACT The compound HoNiSb has been studied by means of X-ray powder diffraction. The crystal structure and the X-ray powder diffraction data for this alloy phase are presented. The compound HoNiSb is cubic (space group $F\bar{4}3m$, $Z = 4$). At room temperature, the lattice constant is $a = 6.2873(4)$ Å. There are twelve atoms in unit cell. Four Ni atoms, four Sb atoms and four Ho atoms occupy 4(a), 4(c) and 4(d) positions respectively.

Key words RE compound HoNiSb X-ray diffraction data crystal structure

1 INTRODUCTION

Rare earth and its compounds have a lot of unique properties. For the identification of phases and the development of new materials, it is essential to carry out systematic investigations of crystal structure and X-ray powder diffraction data for rare earth compounds.

So far among ReNiSb compounds, the diffraction data of NdNiSb and DyNiSb can be found in Powder Diffraction File compiled and published by the International Centre for Diffraction Data[1], but the data of HoNiSb were not reported at all. So the purpose of this work was to study its crystal structure and X-ray powder diffraction data by X-ray powder diffraction.

2 EXPERIMENTAL DETAILS

Alloy buttons were prepared by electric arc furnace melting in pure argon, starting from the nominal compositions using 99.999% Ni, 99.99%Sb and 99.9%Ho. To ensure homogeneity, the sample was remelted five times. In order to compensate weight losses by vaporization of Sb, 8% extra amount of Sb was put in beforehand.

The sample was enclosed in Ta foil and then sealed in an evacuated quartz tube and annealed for 10 d at 1 050 ℃, then cooled at a rate of 10 ℃/h to room temperature. Electron probe microanalysis (EPMA) of the sample was carried out and the composition of the sample were confirmed.

The sample is brittle. The ingot was ground in an agate mortar and pestle to a particle size of less than 10 μm. The treated alloy powder was used for the X-ray analysis and the density measurements. Powder diffraction data were collected with a Rigaku D/max-RC diffractometer using a Cu target ($\lambda = 1.54060$ Å) operated at 50 kV and 180 mA. The instrument was equipped with a diffracted beam graphite monochromator, 1° divergence slit, 0.15 mm receiving slit and scintillation counter. The NIST SRM 1976 provided by ICDD was used in calibration of powder X-ray equipment for diffraction intensity.

For the determination of d-data, the instrument was calibrated with high pure Si, supplied by Rigaku corporation, as external standard ($\Delta 2\theta = a_1 + a_2 \cdot \cos\theta + a_3 \cdot \sin 2\theta$). Standard software from Rigaku corporation

① Supported by a Grant-in-Aid from the International Centre for Diffraction Data and the Natural Science Foundation of Guangxi; Received Mar. 6, 1995, accepted May 23, 1995

was used for peak searching, $K_{\alpha 1}$, $K_{\alpha 2}$ separation for the peaks less than 58°2θ.

The rear loading sample preparation technique, proposed by Schreiner[2], was adopted to reduce the effects of preferred orientation for measuring intensity.

The scan range was 10 to 145°2θ with a step size of 0.02° and a count time of 2 s per step. Powder data were collected at room temperature (25 ± 1 ℃). Intensity was measured by integrated areas from step-scan data.

3 RESULTS AND DISCUSSION

3.1 *X-ray Powder Diffraction Data*

X-ray powder diffraction data for HoNiSb are given in Table 1. All of the diffraction patterns were indexed by TREOR program successfully according to cubic structure.

Table 1 X-ray Powder Diffraction Data for HoNiSb

2θ/(°)	I_{obs}	d_{obs}	hkl	d_{cal}	I_{cal}
24.472	9	3.635	111	3.6300	9.7
28.332	55	3.148	200	3.1437	53.9
40.528	100	2.224	220	2.2229	100
47.916	4	1.897	311	1.8957	4.0
50.208	13	1.816	222	1.8150	14.0
58.678	18	1.572	400	1.5718	15.2
			331	1.4424	1.4
66.434	17	1.4061	420	1.4059	17.0
73.756	27	1.2836	422	1.2834	27.8
			511	1.2100	0.7
			333		0.2
87.735	10	1.1116	440	1.1115	8.3
			531	1.0628	0.8
94.618	7	1.0480	442	1.0479	6.5
			600		1.6
101.593	15	0.9941	620	0.9941	12.6
			533	0.9588	0.3
108.747	7	0.9477	622	0.9479	5.6
116.209	5	0.9073	444	0.9075	3.9
			551	0.8804	0.3
			711		0.3
124.137	5	0.8719	640	0.8719	6.0
132.994	28	0.8400	642	0.8402	27.0
			553	0.8185	0.4
			731		0.8

The lattice parameter of the compound was refined by a least-squares method using Lattice Constant Refinement program, a = 6.2873(4) Å. The De Wolff figure of merit M_{15}[3] and the Smith and Snyder figure F_{15}[4] are 135.94 and 32.08 respectively.

3.2 *The Crystal Structure of* HoNiSb

It is obvious from that the h,k,l are all even or all odd that the compound belongs to face centred cubic. Because the diffraction pattern of HoNiSb is completely similar to that of DyNiSb, the space group $F\bar{4}3m$ was chosen to calculate theoretical intensity. From experimental value of density D_{EXP} = 9.11 cm^{-3}, we can obtain Z = 3.95. Considering the measuring error, we can confirm that there are four formula units in each unit cell, i.e. there are four Ho atoms, four Ni atoms and four Sb atoms in one unit cell.

The Lazy Pulver Ⅸ program was used in calculating the theoretical diffraction intensity. The atom positions and temperature factor used in calculation are shown in Table 2. Because individual atom thermal parameters were not available, isotropic thermal parameters were estimated as 0.5 for all elements.

Table 2 The atoms structure parameter, occupy rate and temperature factor for HoNiSb

Atom	Wyckoff notation	coordinate +(0,0,0; $\frac{1}{2},\frac{1}{2},0;\frac{1}{2},0,\frac{1}{2};0,\frac{1}{2},\frac{1}{2}$)	occupy rate	B
4Ni	4a	0,0,0	1	0.5
4Sb	4c	$\frac{1}{4},\frac{1}{4},\frac{1}{4}$	1	0.5
4Ho	4d	$\frac{3}{4},\frac{3}{4},\frac{3}{4}$	1	0.5

An R-factor designed by Snyder[5] to assess the accuracy of relative intensities, $R = \sum |I_{cal} - I_{obs}| / \sum I_{cal}$, was adopted. The value of this R-factor was 6.6% showing the

calculated intensities were in agreement with the observed intensities. The unit cell of HoNiSb was shown in Fig. 1, and its crystal structure data were summarized in Table 3.

Table 3 Crystal structure data for HoNiSb

Crystal system	cubic
Space group	$F\bar{4}3m$
Cell parameter a/Å	6.287 3(4)
Number of formula unit Z	4
Volume of the unit cell/Å^3	248.54
formula weight	245.37
calculated density/g·cm^{-3}	9.231
experimental density/g·cm^{-3}	9.11

3.3 *The InteratomicDistances and Discussion*

Fig. 1 shows that the four Ni atoms, four Sb atoms and four Ho atoms located at 000+face-centering translations, $\frac{1}{4}\frac{1}{4}\frac{1}{4}$+face-centering translations and $\frac{3}{4}\frac{3}{4}\frac{3}{4}$+face-centering translations respectively, i. e. Sb and Ho atoms occupy the centre of tetrahedral sites of the *FCC* structure. So, the nearest atoms around the Sb or Ho are four Ni atoms and the nearest interatomic distances between Ni and Sb or Ni and Ho are all 2.722 5 Å which is smaller than the sum of metallic Ni and Ho or Ni and Sb radii (R_{Ho} = 1.76 Å, R_{Ni} = 1.24 Å, R_{Sb} = 1.61 Å). The reduced value is not more than 9.3% and 4.5% respectively. That means both Ho and Sb atoms contract a little.

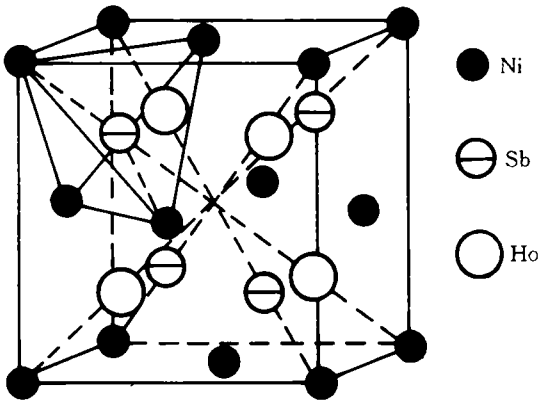

Fig. 1 Unit cell of HoNiSb

REFERENCES

1 International Centre for Diffraction Data, Powder Diffraction File, Alphabetical Indexes sets 1—43, 1993.
2 Schreiner WN. Guidelines for Grant-in-Aid proposals.
3 de Wolff PM. J Appl Cryst, 1968, 1: 108.
4 Smith, GS, Snyder, RL. J Appl Cryst, 1979, 12: 60.
5 Snyder, RL. In: Advances in X-Ray Analysis, 1983, 26: 1—6.