X-RAY POWDER DIFFRACTION STUDY FOR RE COMPOUND GdNiSn①

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ABSTRACT The compound GdNiSn has been studied by X-ray powder diffraction technique. The crystal structure and the X-ray diffraction data for this compound at room temperature are reported. The compound GdNiSn is orthorhombic with lattice parameters $a = 7.2044(1)$ Å, $b = 7.6895(6)$ Å, $c = 4.4772(4)$ Å, space group $Pna2_1$ and 4 formula units of GdNiSn in unit cell. The Smith and Snyder figure of index F_{30} for this compound is 35(0.015, 59).

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

1 INTRODUCTION

The Powder Diffraction File (PDF) compiled by Joint Committee on Powder Diffraction Standards-Intenational Center for Diffraction Data (JCPDS-ICDD) is a very useful tool for phase analysis. So far, in the compounds RENiSn (RE: rare earth) only compound CeNiSn has been reported in PDF. Skolozdra *et al*[1] studied the X-ray powder diffraction data of compound CeNiSn and showed it in PDF 38—897. The compound is orthorhombic, but its space group was not reported. Higashi *et al*[2] determined its space group by single crystal method. The space group is $Pn2_1a$ and the unit cell contains 4 formula units of CeNiSn. Ref. [3] indicates that the compounds RENiSn exhibit anisotropic, magnetic and transportation properties and have possible application in high technology. In this paper we studied the X-ray powder diffraction data of GdNiSn to show its PDF and its crystal structure.

2 EXPERIMENTAI DETAILS

The sample of GdNiSn were prepared by arc melting under pure argon from ingots of the pure elements: 99.999%Ni, 99.99%Sn and 99.9%Gd. The sample was remelted five times to ensure homogeneity.

The sample was enclosed in Ta foil and then sealed in an evacuated quartz tube at 10^{-3} Pa and annealed for 10 d at 1 050 ℃, then cooled at a rate of 10 ℃/h to room temperature. The compositions of the sample were confirmed by electron probe microanalysis (EPMA).

The sample is brittle. The ingot was ground in an agate mortar and pestle to a particle size of less than 10 μm. The powder was sealed in an evacuated glass tube and annealed at 400 ℃ for 2 d, then cooled at a rate of 10 ℃/h to room temperature. The treated alloy powder was used for the X-ray analysis. 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 for calibration of

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the diffraction intensity.

For the determination of d-data, the instrument was calibrated with high pure Si, supplied by Rigaku corporation, as external standard ($\triangle 2\theta = a_1 + a_2 \cdot \cos\theta + a_3 \cdot \sin 2\theta$). Standard software from Rigaku corporation was used for peak searching and $K_{\alpha 1}$, $K_{\alpha 2}$ separation for the low angle peaks.

The rear loading sample preparation technique, proposed by International Center for Diffraction Data, 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. The data are processed with D/max software.

3 RESULTS AND DISCUSSION

3.1 *X-Ray Powder Diffraction Data*

X-ray powder diffraction data for GdNiSn are given in Table 1. All diffraction patterns were indexed by TREOR program successfully according to orthorhombic system.

The lattice parameters of the compound was refined by a least-squared method using Lattice Constant Refinement Program:

$a = 7.2044(1)$ Å, $b = 7.6895(6)$ Å,

$c = 4.4772(4)$ Å.

The De Wolff figure of merit M_{20} and the Smith and Snyder figure F_{30} are 36 and 35(0.015, 59) respectively.

Ref. [1] showed that the compound CeNiSn is orthorhombic. The Smith and Snyder figure F_{23} for CeNiSn in Ref. [1] is 2(0.13994). Therefore, the data of GdNiSn in this paper is much better than that of CeNiSn in Ref. [1].

3.2 *Crystal Structure of* GdNiSn

From Table 1, it can be seen that the conditions limiting possible reflections (hkl: no condition, $0kl$: $k + l = 2n + 1$, $h0l$: $h = 2n + 1$, $0k0$: $k = 2n + 1$, $00l$: $l = 2n + 1$) is in good agreement with that of the space group $Pna2_1$ of CeNiSn. Therefore the space group $Pna2_1$ was chosen to calculate theoretical intensity. The LAZY PULVERIX program was used in calculating the theoretical diffraction intensity. The atom positions of Gd, Ni and Sn used in calculation are shown in Table 2. The calculation results are shown in Table 1.

An R-factor ($R = \Sigma | I_{cal} - I_{obs} | / \Sigma I_{cal}$) designed by Snyder[6] to assess the accuracy of relative intensities for this compound is 12.8%. According to Ref. [7], when the R-factor ranges from 5% to 15% for powder diffraction patterns, the inorganic phases are well-crytallized and have minimal preferred orientation. It means that the R-factor of low symmetrical phase GdNiSn is good value. The crystal structure data of GdNiSn are given in Table 3.

3.3 *The Interatomic Distances and Discussion*

The distances of the nearest atoms can be calculated from the atom positions in Table 2. Those between Gd and Ni, Gd and Sn, Ni and Sn and Gd and Gd are 2.96, 3.11, 2.41 and 3.69 Å, respectively. The radii of Gd, Ni and Sn are $r_{Gd} = 1.80$ Å, $r_{Ni} = 1.24$ Å and $r_{Sn} = 1.58$ Å, respectively, when their coordination numbers are 12. The sums of radii between two of them are longer than the distanes between representive atoms. It is reasonable according to the model of hard spheres because the atomic radii will reduce as its coordination number is less than 12[8]. The coordination numbers of Gd, Ni, Sn in this crystal structure are all less than 12.

4 CONCLUSIONS

(1) The RE compound GdNiSn at room temperature is orthorhombic with lattice parameter $a = 7.2044(1)$ Å, $b = 7.6895(6)$ Å, $c = 4.4772(4)$ Å, space group $Pna2_1$ and 4 formula units of GdNiSn in a unit cell.

(2) The X-ray powder diffraction data of GdNiSn was given in this paper, the figure is

Table 1 X-ray powder diffraction data of GdNiSn($2\theta < 90°$)

$2\theta_{obs}$/(°)	d/Å	hkl	I/I_0	$2\theta_{cal}$	$(I/I_0)_{cal}$	$2\theta_{obs}$(°)	d/Å	hkl	I/I_0	$2\theta_{cal}$	$(I/I_0)_{cal}$
16.875	5.250	110	2	16.850	1	57.486	1.6019	312	15	57.497	12
22.984	3.866	011	6	22.967	4			421	<1	60.351	3
23.133	3.842	020	1	23.115	3			232	<1	60.587	2
26.120	3.409	111	12	26.121	13	61.617	1.5040	340, 150	12	61.616	8
26.268	3.390	120	7	26.252	7			042	<1	63.764	1
31.841	2.808	201	9	31.860	9			113	<1	64.897	1
33.099	2.704	121	45	33.107	47			142	<1	65.216	1
33.975	2.637	211	100	33.976	100			151	<1	65.407	1
34.092	2.628	220	26	34.079	28	65.539	1.4232	341	26	65.550	16
37.189	2.416	130	12	37.203	10	65.883	1.4166	510	2	65.900	2
39.255	2.293	310	22	39.272	22	66.573	1.4035	402	4	66.586	4
40.237	2.239	002	34	40.254	35	66.768	1.3999	431	3	66.775	3
40.515	2.225	031	38	40.521	42	67.862	1.3800	412, 332	8	67.834	8
		131	<1	42.498	1	68.637	1.3663	123	5	68.652	5
43.258	2.090	230	4	43.288	3	69.156	1.3573	213	9	69.166	10
		311	<1	44.361	1	69.483	1.3517	242, 511	7	69.475	5
		022	<1	46.928	1			422	<1	71.513	1
		040	<1	47.244	2	73.192	1.2921	521	15	73.207	11
48.037	1.892	231	2	48.033	3	73.337	1.2899	033	7	73.349	6
48.659	1.870	122	4	48.697	3	76.196	1.2484	152, 342	9	76.198	8
		140	<1	49.004	2	78.721	1.2146	161	4	78.732	2
49.067	1.855	321	4	49.100	3	79.266	1.2076	260	5	79.280	2
50.625	1.8016	400	4	50.641	5	79.777	1.2012	600	2	79.811	2
52.134	1.7530	410, 330	8	52.113	8	80.122	1.1969	512	2	80.124	2
53.353	1.7158	141	3	53.358	5			143	<1	82.924	1
53.727	1.7047	222	14	53.740	14			601	<1	83.241	1
54.006	1.6966	240	5	54.026	5			413	<1	85.338	1
55.954	1.6420	132	3	55.965	5	86.957	1.1195	004	4	86.975	3
56.313	1.6324	411	6	56.296	5	89.303	1.0961	361	4	89.284	3
		420	<1	56.365	2						

Table 2 Structure parameters of GdNiSn

atom	site	x	y	z	BTEMP
Gd	4(a)	0.4850	0.1971	0.2535	0.5
Ni	4(a)	−0.2910	−0.3850	0.2681	0.8
Sn	4(a)	−0.1922	−0.0862	0.2353	0.6

Table 3 Crystal structure data for GdNiSn

Crystal System:	orthorhombic
Space group:	$Pna2_1$
Lattice parameter:	a = 7.2044(1) Å, b = 7.6895(6) Å, c = 4.4772(4) Å
Number formula unit:	z = 4
Volune of the unit cell:	v = 248.03 Å^3
Calculated density:	D_x = 8.962 g·cm^{-3}

F_{30} = 35(0, 015, 59).

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