X-RAY POWDER DIFFRACTION DATA AND STRUCTURAL REFINEMENT OF $Er_3Co_6Sn_5$ ①

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ABSTRACT The compound $Er_3Co_6Sn_5$ has been studied by means of X-ray powder diffraction technique and refined by Rietveld method. The compound $Er_3Co_6Sn_5$ has an orthorhombic $Dy_3Co_6Sn_5$-type structure, space group Immm (No.71), $Z = 2$ and $D_x = 9.465\ g/cm^3$. At room temperature, the lattice constants are $a = 4.2966(4)$ Å, $b = 12.2897(9)$ Å, $c = 9.6271(6)$ Å. The Rietveld structural refinement was performed and led to $R_{wp} = 0.196$ and $R_p = 0.153$. The figure of merit F_N for the X-ray powder diffraction data is $F_{30} = 34.7\ (0.0090, 96)$. The present structural refinement supports that Er(1), Er(2), Co(1), Co(2), Sn(1) and Sn(2) atoms occupy the 2 *a*, 4 *g*, 4 *j*, 8 *l*, 2 *c* and 8 *l* positions, respectively.

Key words $Er_3Co_6Sn_5$ X-ray powder diffraction Rietveld structural refinement

1 INTRODUCTION

Rare metals and their compounds have outstanding properties and are paid increasing attention to. Numerous investigations in the ternary system rare earth-cobalt-tin have been made and novel compounds were discovered[1,2]. Among them, $Er_3Co_6Sn_5$ was reported that crystallizes in the orthorhombic structure, a new ternary ordered version of La_3Al_{11}-type, with space group Immm (No. 71)[1]. But the X-ray powder diffraction data for $Er_3Co_6Sn_5$ was not reported at all. No structural refinement for this compound was found in literature. So the purpose of this work is to study its X-ray powder diffraction data and refine its crystal structure.

2 EXPERIMENTAL DETAILS

The alloy $Er_3Co_6Sn_5$ was prepared by arc melting under pure argon and the raw materials are of at least 99.9% purity. The sample was turned over and remelted five times to ensure good homogeneity. The alloy button was enclosed in an evacuated quartz tube and annealed at 1 000 ℃ for 10 d, then cooled down at a rate of 10 ℃/h to room temperature. Electron probe microanalysis (EPMA) of the sample was carried out to confirm the composition of the sample.

The alloy was brittle. The ingot was ground in an agate mortar and pestle to a particle size of less than 10 μm. Powder diffraction data were collected with a Rigaku D/max-RC diffractometer using a Cu target ($\lambda = 1.54060$ Å). The scan range was 10° to 120° (2θ) with a step size of 0.02° and a count time of 2 s per step. A portion of powder sample was mixed with high purity Si powders as the internal standard for the determination of *d*-values. For relative intensity determination, another portion of the powder

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sample without internal standard was loaded into aluminum cavity specimen holder by the "Rear Loading" method[3]. The NIST SRM 1976 was used to calibrate the powder X-ray equipment for diffraction intensity. Some experimental details are the same as those in Ref.[4]. The crystal structural refinement of compound $Er_3Co_6Sn_5$ has been carried out by Rietveld method from X-ray powder diffraction data.

3 RESULTS AND DISCUSSION

Table 1 shows the X-ray powder diffraction data for $Er_3Co_6Sn_5$.

All of the X-ray diffraction patterns were indexed successfully on the basis of an orthorhombic structure by using TEROR program[5]. The lattice parameters obtained by the least-squared method are $a = 4.2966(4)$ Å, $b = 12.2897(9)$ Å, $c = 9.6271(6)$ Å. The space group for the orthorhombic structure is Immm (No. 71). There are two formula units of $Er_3Co_6Sn_5$ in each unit cell. The Smith and Snyder figure of merit[6] F_N is $F_{30} = 34.7(0.0090, 96)$. The index result is given in Table 1.

The Rietveld's powder diffraction profile-fitting technique[7,8] was used to analyze the X-ray powder diffraction patterns of the compound $Er_3Co_6Sn_5$. The lattice constants obtained in this work and the atomic position parameters of $Sm_3Co_6Sn_5$[1] were taken as starting values to refine the structural parameters of $Er_3Co_6Sn_5$. The atomic scattering factors were selected from the *International Tables for X-ray Crystallography IV (1974)*[9]. The profile shape model was a Pearson VII type. The crystal structure was successfully refined by using the program DBWS-9006PC[10]. The results of the Rietveld structural refinement are presented in Table 2, the refined atomic parameters are listed in Table 3. The refined lattice constants are $a = 4.2958(1)$ Å, $b = 12.2902(2)$ Å, $c = 9.6267(1)$ Å which are in good agreement with that obtained by a least-squared method. The pattern *R*-factor R_p and the weighted pattern *R*-factor R_{wp} are $R_p = 0.153$ and $R_{wp} = 0.196$, respectively.

Table 1 X-ray powder diffraction data for $Er_3Co_6Sn_5$

$2\theta_{exp}$/(°)	I/I_0	d_{exp}/Å	*hkl*
11.665	2	7.58	011
14.414	2	6.140	202
8.747	8	3.103	112
30.116	56	2.965	130
34.618	21	2.589	042
34.854	38	2.572	103
35.510	100	2.526	132
37.901	31	2.372	123
40.212	7	2.2408	024
42.024	32	2.1483	200
42.313	32	2.1343	150
43.702	14	2.0696	114
44.156	22	2.0494	060
46.006	6	1.9712	143
46.520	25	1.9506	152
48.259	6	1.8843	062
55.517	6	1.6539	242
57.445	15	1.6029	204
59.557	3	1.5510	224
60.021	7	1.5401	172
62.606	10	1.4826	260
64.448	7	1.4446	253
66.160	22	1.4113	136
69.471	5	1.3519	330
71.471	11	1.3189	183
72.179	6	1.3077	303
72.578	11	1.3015	190
73.628	5	1.2855	206
74.051	5	1.2792	323
75.143	8	1.2633	066
75.614	5	1.2566	093
87.415	4	1.1148	138
90.081	7	1.0886	266
90.526	5	1.0844	293
96.363	3	1.0336	336
99.321	4	1.0106	196
101.347	2	0.9958	383
102.415	4	0.9883	433
104.350	2	0.9752	2104

Table 2 Crystallographic data and Rietveld structural refinement for $Er_3Co_6Sn_5$

Space group	Immm (No.71)
Cell Parameters/ Å	a = 4.2966(4), b = 12.2897(9), c = 9.6271(6)
Volume of unit cell/ Å^3	508.35
Number of formula unit Z	2
Radiation	$CuK_{\alpha 1}$
2θ/(°)	10.00 ~ 120.00
Step scan increment $\Delta 2\theta$/(°)	0.02
Count time/(s·step^{-1})	2
Number of refined parameters	21
Number of reflections	482
Reliability factors(R-factor)	R_P^* = 0.153, R_{WP}^* = 0.196

* $R_P = \sum_i |Y_{oi} - Y_{ci}| / \sum_i Y_{oi}$;
$R_{WP} = [\sum_i W_i (Y_{oi} - Y_{ci})^2 / \sum_i W_i Y_{oi}^2]^{1/2}$

To make a comparison, pseudo-Voigt (pV) and Modified Thompson-Cox-Hastings pseudo-Voigt (Mod-TCH pV) profile functions were used in this refinement, too. The pattern R-factors from pV and Mod TCH pV profile functions are R_p = 0.157 and R_p = 0.155, respectively.

Table 3 Refined atomic parameters for $Er_3Co_6Sn_5$

Atom	Site	x	y	z	N
Er(1)	2 a	0	0	0	2
Er(2)	4 g	0	0.3151(3)	0	4
Co(1)	4 j	0	1/2	0.1959(9)	4
Co(2)	8 l	0	0.1078(5)	0.2742(7)	8
Sn(1)	2 c	0	0	1/2	2
Sn(2)	8 l	0	0.3237(3)	0.3416(4)	8

The present structural refinement supports that the atomic positions of $Er_3Co_6Sn_5$ are similar to those of $Sm_3Co_6Sn_5$. In the structure of $Er_3Co_6Sn_5$, Er(1) and Er(2) atoms occupy the 2 a and 4 g positions, Co(1) and Co(2) atoms occupy the 4 j and 8 l positions, Sn(1) and Sn (2) atoms are at the 2 c and 8 l positions, respectively. Fig.1 shows the difference between the experimental and calculated patterns of X-ray diffraction patterns of $Er_3Co_6Sn_5$.

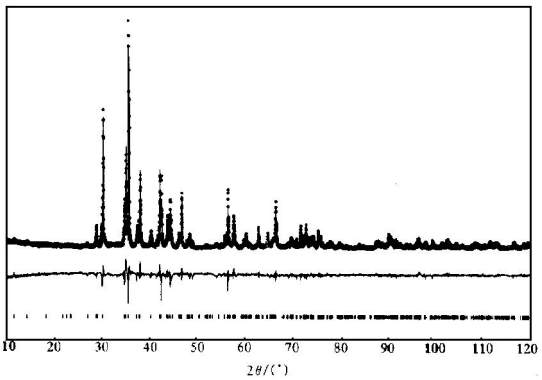

Fig.1 X-ray diffraction patterns for $Er_3Co_6Sn_5$
● —Experimental pattern;
Solid line —Calculated pattern;
| —Possible positions of Bragg reflections;
Middle curves —Difference between experimental and calculated patterns

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