X-RAY POWDER DIFFRACTION DATA AND STRUCTURE REFINEMENT OF COMPOUND $ErNi_2Si_2$ ①

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ABSTRACT The X-ray powder diffraction data for $ErNi_2Si_2$ are given and the crystal structure has been refined by the Rietveld whole-pattern-fitting method. The compound $ErNi_2Si_2$ crystallizes with the $ThCr_2Si_2$ type structure (tetragonal, space group I4/mmm, $Z=2$). The refined lattice parameters are $a=3.9321(1)$ Å, $c=9.5237(1)$ Å and the structure was refined to $R_p=13.09\%$, $R_{wp}=16.69\%$. The figure of merit F_N for the powder data is $F_{30}=108.2\ (0.0063, 44)$.

Key words $ErNi_2Si_2$ X-ray powder diffraction Rietveld structure refinement

1 INTRODUCTION

The rare earth compounds RET_2Si_2, where RE is a rare earth element and T is a transition metal element, have been studied with great interest for a few decades[1]. Most of these compounds with $ThCr_2Si_2$ type structure (space group I4/mmm, standardized atom coordinates Th in 2a: 0, 0, 0; Cr in 4d: 0, 1/2, 1/4 and 1/2, 0, 1/4; Si in 4e: 0, 0, Z and 0, 0, $-Z$) have a variety of magnetic properties[2, 3]. The crystal structure and X-ray powder diffraction data of a single phase are the basis of materials science study. Since there are no reports on X-ray powder diffraction data for $ErNi_2Si_2$ in the Powder Diffraction File (PDF), in this paper, we report the X-ray powder diffraction data and the Rietveld structure refinement of $ErNi_2Si_2$.

2 EXPERIMENTAL

The sample of compound $ErNi_2Si_2$ was prepared by arc melting starting materials of at least 99.9% purity. The sample was turned upside down and remelted five times to ensure homogeneity. The mass loss of the sample during arc melting was less than 0.5%. To achieve equilibrium, the sample was wrapped with Ta foil, sealed into an evacuated quartz tube and annealed for 10 days at 1 000 ℃, then cooled at a rate of 10 ℃/h to room temperature. The composition of the sample was confirmed by electron probe microanalysis (EPMA). The powder sample was obtained by pestling the annealed alloy to a particle size of less than 10 μm in an agate mortar. The powder was sealed in an evacuated glass tube and annealed at 500 ℃ for 5 days, then cooled at a rate of 10 ℃/h to room temperature.

Powder was loaded into aluminum cavity specimen holder by the "Rear loading sample preparation technique" proposed by Schreiner[4]. The X-ray powder diffraction experiment was performed for $ErNi_2Si_2$ on the Rigaku D/max-RC diffractometer equipped with a graphite monochromater using CuK_α radiation ($\lambda=1.54060$ Å). Data were collected in the step-scan mode with a step size of 0.02°(2θ) and a counting time of 2s per step over the range of 15 ~ 140°(2θ). Powder data were collected at room temperature (25 ± 1 ℃). High purity Si powder was used as an internal standard for the

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determination of d value. Some of the experiment details are the same as that in Ref. [5]. The structure refinement of the compound $ErNi_2Si_2$ was carried out by the Rietveld whole-pattern-fitting method[6] using the program DBWS-9411PC[7].

3 RESULTS AND DISCUSSION

3.1 The X-ray powder diffraction data of $ErNi_2Si_2$

The XRD patterns of the sample consist of 88.51% (mass) $ErNi_2Si_2$ phase and 11.49% (mass) Er_2O_3 phase. The diffraction pattern was successfully indexed with a tetragonal lattice using the program TREOR[8] after excluding the peaks of Er_2O_3. X-ray powder diffraction data for $ErNi_2Si_2$ are listed in Table 1. The Smith and Snyder figure of merit[9] F_{30} is 108.2 (0.0063, 44), showing the powder pattern indexing is highly reliable.

Table 1 X-ray powder diffraction data for $ErNi_2Si_2$

$2\theta_{obs}$/(°)	I/I_{0obs}	d_{obs}/Å	hkl	d_{cal}/Å	$\Delta 2\theta$/(°)	$2\theta_{obs}$/(°)	I/I_{0obs}	d_{obs}/Å	hkl	d_{cal}/Å	$\Delta 2\theta$/(°)
18.605	2	4.765	002	4.763	−0.008	91.009	5	1.0800	305	1.0799	0.002
24.463	45	3.636	101	3.635	−0.006	91.402	6	1.0763	217	1.0763	0.002
32.175	9	2.780	110	2.781	0.009				226	1.0460	
36.335	77	2.471	103	2.471	0.001	96.620	6	1.0315	323	1.0315	−0.003
37.424	100	2.401	112	2.401	0.005				109	1.0221	
37.737	24	2.382	004	2.382	−0.004	98.267	10	1.0186	208	1.0185	−0.002
46.131	35	1.9661	200	1.9662	0.001	103.158	5	0.98319	400	0.98310	−0.014
			202	1.8174		103.772	7	0.97905	316	0.97901	−0.006
			114	1.8088					402	0.96275	
52.985	10	1.7295	211	1.7294	−0.005				0010	0.95263	
53.393	27	1.7146	105	1.7146	0.003	108.534	1	0.94894	411	0.94900	0.010
			006	1.5877		108.920	5	0.94665	325	0.94655	−0.018
60.093	27	1.5384	213	1.5384	0.001				307	0.94407	
61.068	18	1.5162	204	1.5162	0.003				330	0.92686	
67.287	9	1.3904	220	1.3903	−0.004	114.980	3	0.91344	413	0.91343	−0.001
67.929	19	1.3788	116	1.3788	0.002				332	0.90981	
			222	1.3346					404	0.90872	
72.783	3	1.2983	301	1.2986	0.015				219	0.90684	
73.178	18	1.2923	215	1.2923	0.000	116.792	6	0.90444	228	0.90443	−0.002
73.604	5	1.2859	107	1.2860	0.016	117.449	5	0.90127	1110	0.90124	−0.006
			310	1.2435		122.326	6	0.87934	420	0.87931	−0.008
			206	1.2352					422	0.86468	
78.950	5	1.2117	303	1.2116	−0.002				334	0.86376	
79.626	22	1.2031	312	1.2032	0.012				318	0.86006	
			224	1.2007					2010	0.85726	
80.622	6	1.1907	008	1.1908	0.012	129.172	5	0.85283	415	0.85286	0.010
			314	1.1023					327	0.85104	
89.453	2	1.0946	118	1.0946	0.007	131.227	3	0.84576	1011	0.84579	0.010
90.630	2	1.0834	321	1.0836	0.015						

3.2 Structure refinement of $ErNi_2Si_2$

The Rietveld method was used to refine the crystal structure of $ErNi_2Si_2$. The $ErNi_2Si_2$ phase and an impurity phase of Er_2O_3 were included in the refinement. The profile function used for describing the peak shape was a Pearson Ⅶ type, the background was modeled as a refined polynomial. The tetragonal $ThCr_2Si_2$ structure[1] was used as a trial structure, and scattering factors were taken from International Tables for X-ray Crystallography[10].

A total of 37 parameters, including 3 polynomial background and 20 structural parameters for $ErNi_2Si_2$ and 14 parameters for Er_2O_3 and for the global parameters of zero point, scale factor, preferred orientation and peak shape were refined.

The observed, calculated and difference diffraction patterns for the Rietveld refinement of $ErNi_2Si_2$ are displayed in Fig. 1.

The refined structural parameters and R factors of $ErNi_2Si_2$ are given in Table 2. The refined lattice parameters are a= 3.932 1(1) Å and c= 9.523 7(1) Å. These results confirm that the compound $ErNi_2Si_2$ belongs to the tetragonal $ThCr_2Si_2$ type structure with space group I4/mmm. Each unit cell contains two chemical formula units of $ErNi_2Si_2$. The pattern R factor R_p and the weighted pattern R factor R_{wp} obtained with the Rietveld refinernent for $ErNi_2Si_2$ are 13.09% and 16.69%, respectively. Fig. 2 is a representation of the crystal structure of $ErNi_2Si_2$ in three dimensional view and the interatomic distances for $ErNi_2Si_2$ are given in Table 3.

Table 2 Refined atomic position parameters of $ErNi_2Si_2$

Atom	Site	x	y	z	N
Er	2a	0	0	0	2.0
Ni	4d	0	0.5	0.25	4.0
Si	4e	0	0	0.378 3(4)	4.0

($ErNi_2Si_2$: I/4mmm, No. 139; a= 3.932 1(1) Å, c= 9.523 7(1) Å, Z= 2; R_{wp}= 16.69%, R_p= 13.09%)

4 CONCLUSION

The X-ray powder diffraction data of $ErNi_2Si_2$ compound have been investigated by

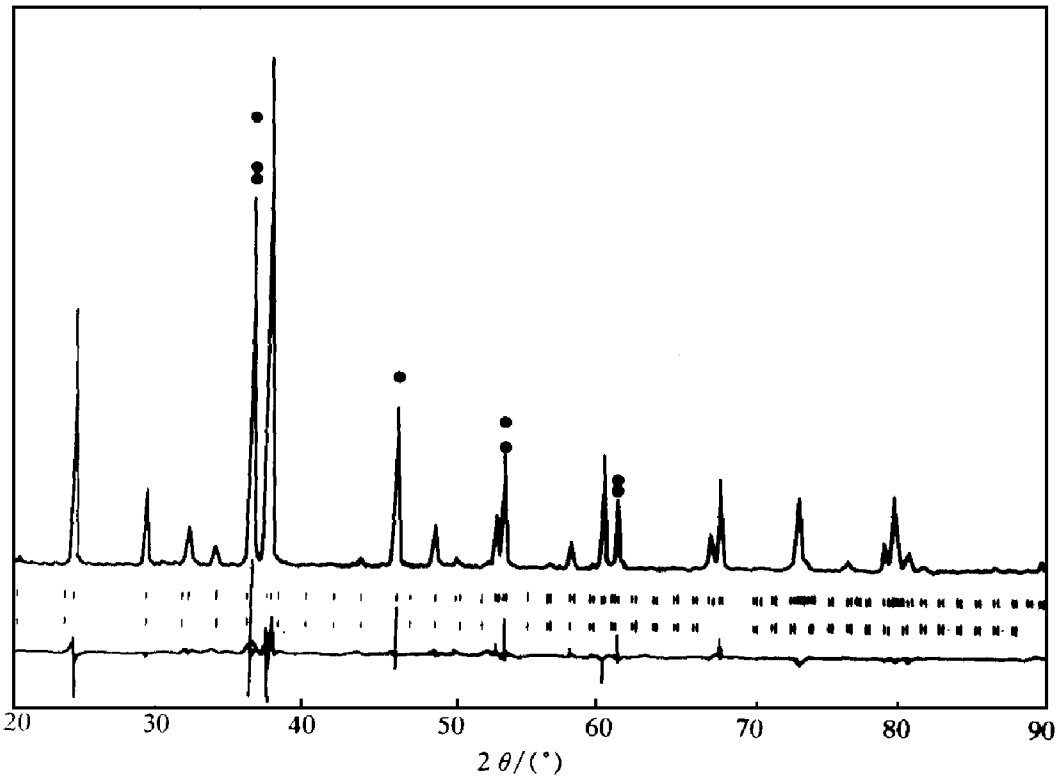

Fig. 1 The observed, calculated and difference diffraction patterns of $ErNi_2Si_2$

Table 3 Interatomic distances for $ErNi_2Si_2$(Å)

Atoms	Distance/ Å
Er-Er	3.9321
Er-Ni	3.0877
Er-Si	3.0123
Ni-Si	2.3148
Si-Si	2.3181

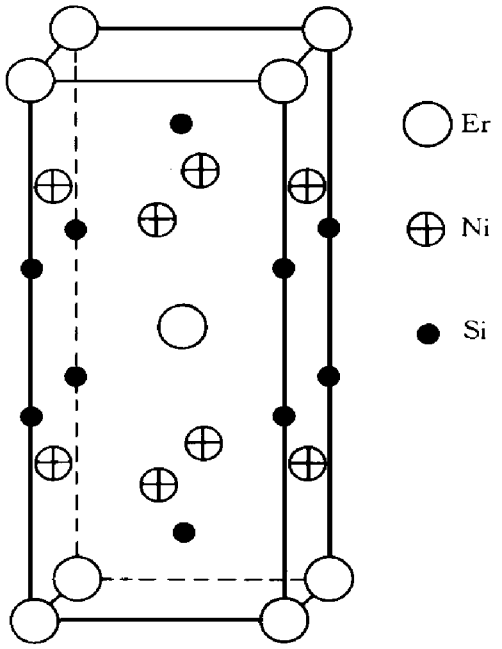

Fig. 2 The unit cell of $ErNi_2Si_2$

experimental methods and the crystal structure has been refined by Rietveld method. The compound has the tetragonal $ThCr_2Si_2$ type structure, space group I4/mmm (No. 139), a = 3.9321(1) Å, c = 9.5237(1) Å, V = 147.249 (3) Å^3, Z = 2 and D_x = 7.889g/cm^3. The refined results show that 2 Er, 4 Ni and 4 Si atoms occupy the 2a, 4d and 4e positions, respectively. The figure of merit F_N for the powder data is F_{30}= 108.2 (0.0063, 44).

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