X-RAY RIETVELD STRUCTURE REFINEMENT OF ErNiSb①

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ABSTRACT The crystal structure of compound ErNiSb has been refined by the Rietveld whole-pattern-fitting method from X-ray powder diffraction data. The compound ErNiSb is cubic, space group $F\bar{4}3m$ and the structure parameters and reliability factors were refined to be $a = 6.2683(1)$ Å, $V = 246.29$ Å^3, $Z = 4$, $D_x = 9.377$ g/cm^3, $R_B = 3.57\%$, $R_F = 3.64\%$, $R_p = 6.63\%$, $R_{WP} = 8.80\%$.

Key words X-ray powder diffraction rietveld analysis structure refinement

1 INTRODUCTION

The Rietveld method of whole-pattern-fitting structure refinement from powder diffraction pattern[1] has become a widely used method for its many important applications in the field of materials science research. In the Rietveld analysis, the structural and profile parameters can be adjusted to make the calculated intensity data fit the diffracted intensity data by least-squares method. The crystal structure of compound ErNiSb has been determined by X-ray powder diffraction method in our previous work[2], this compound is face centred cubic with space group $F\bar{4}3m$ and lattice parameter $a = 6.2693(1)$ Å. The atoms of Ni, Sb, Er are at specific positions: Ni at (0, 0, 0), Sb at (1/4, 1/4, 1/4) and Er at (3/4, 3/4, 3/4). As part of our ongoing research, this paper presents the results of the Rietveld structure refinement of compound ErNiSb.

2 RIETVELD STRUCTURE REFINEMENT

Details of the experimental X-ray powder diffraction data collection have been described in Ref. [2]. The scattered intensity data were taken in steps of 0.02° from 23.5° to 137.08° (2θ) with counting time of 2 s per step.

Crystal structure refinement of compound ErNiSb was carried out with the Rietveld whole-pattern-fitting method from X-ray powder diffraction data using computer program DBWS-9006PC[3]. The initial structural parameters are from Ref. [2], and scattering factors are taken from Ref. [4]. The refined polynomial was used to describe the background. The profile shape was modeled by Pearson Ⅶ profile function and corrected for asymmetry below 40° (2θ). The preferred orientation was taken into account using the Rietveld-Toraga model. The pattern was refined in the following manner. The first a few cycles of refinement included only the 2θ zero-point, the overall scale factor, three background coefficients, three half-width parameters. Once the best fit parameter values were obtained, lattice constants, profile shape mixing parameters, preferred orientation parameters, asymmetry parameter and overall isotropic temperature factors were refined. In total, 15 structural parameters and profile parameters including 3 background coefficients were refined.

3 RESULT AND DISUCSSION

Crystallographic data for compound ErNiSb as well as details of the Rietveld structure refinement are listed in Table 1.

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Table 1 Crystallographic data and details of the Rietveld structure refinement for compound ErNiSb

Parameters		Data
Space group		$F\bar{4}3m$
Cell Parameters a/ Å		6.2683(1)
Volume of unit cell/ Å^3		246.29
Number of formula unit Z		4
Calculated density/ g·cm^{-3}		9.377
2 θ range/ (°)		23.50~ 137.08
Step scan increment 2 θ/ (°)		0.02
Count time per step/ s		2
Number of refined parameters		15
Number of reflections		46
Reliability factors*	R_B	3.57%
	R_F	3.64%
	R_p	6.63%
	R_{WP}	8.80%

* Defined as follows:

$$R_P = \sum_i |Y_{oi} - Y_{ci}| / \sum_i Y_{ci};$$

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

$$R_B = \sum_i |I_{obc} - I_{cal}| / \sum_i I_{obc};$$

$$R_F = \sum_i |(I_{obc})^{1/2} - (I_{cal})^{1/2}| / \sum_i (I_{obc})^{1/2}.$$

The values of these R-factors were below 10% showing a good fit of peak profile and crystal structure had been obtained. R_p and R_{WP} are not directly comparable to the R value obtained in X-ray powder diffraction which proposed by Snyder[5], because they are sensitive to the background level, and both R_p and R_{WP} will be decreased as the background level is increased. According to Roderick *et al*[6], of all the reliability factors listed above, R_B represents the best measure of the validity of the crystal structure model in Rietveld analysis and can be compared with the R value as shown in Table 1. Fig. 1 is the ErNiSb unit cell which contains twelve atoms. Four Ni atoms, four Sb atoms and four Er atoms are located at 4 a, 4 c and 4 d positions, respectively. The interatomic distances between Ni and Sb or Ni and Er are both 2.7143 Å, which is smaller than the sum of radii of the metallic Ni and Sb or Ni and Er(R_{Ni} = 1.24 Å, R_{Sb} = 1.61 Å, R_{Er} = 1.76 Å). The reduced value is less than 4.8% and 9.5%, respectively.

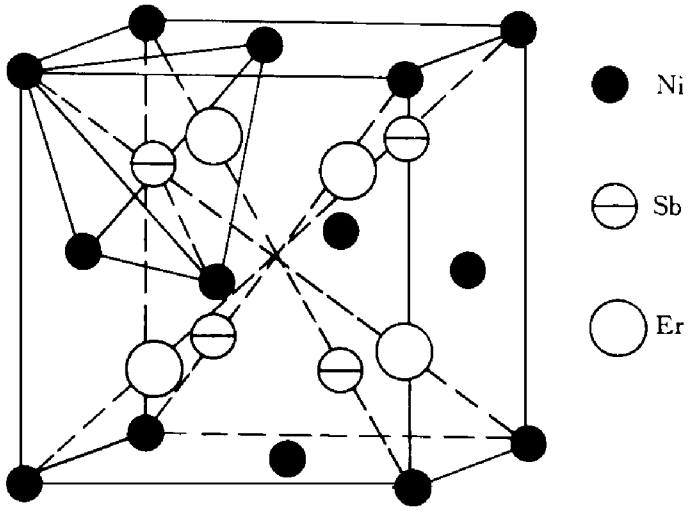

Fig. 1 Unit cell of ErNiSb

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