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Lanthanum rhodium antimonide, $La_{0.1}Rh_8Sb_{24}$

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Abstract

The structure of lanthanum rhodium antimonide, $La_{0.1}Rh_8Sb_{24}$, has been determined from single-crystal X-ray data. It crystallizes in the cubic space group $Im\bar{3}$ (No.204), with a=9.2213(15) Å, V=784.1(2) Å^3 and the $LaFe_4P_{12}$ structure type. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Rare earth compounds; Transition metal compound; X-ray diffraction; Structure determination

1. Introduction

In recent decades, ternary RE–M–Sb systems (where RE is a rare-earth metal and M is a transition metal or a main-group metal) have attracted wide interest [1] because of their unusual physical properties. Several phases such as REM_2Sb_2 [2], RE_3MSb_5 [3], $REMSb_3$ [4], $REMSb_2$ [5], REMSb [6] and REM_4Sb_{12} [7] have been reported. In this paper, we report the structure of $La_{0.1}Rh_8Sb_{24}$.

2. Experimental

2.1. Syntheses

A single crystal of $La_{0.1}Rh_8Sb_{24}$ was obtained from a ternary sample of $LaRh_4Sb_{12}$ by the recrystallization of a cold-pressed mixture of lanthanum (Alfa, −100 mesh, 99.9%), Rhodium (Alfa, −325 mesh, 99.95%) and antimony (Alfa, −100 mesh, 99.99%) in the presence of iodine at 750°C for 15 days. Two kinds of single crystal were found in the sample. One is cube-like and another is needle-like.

2.2. Structure determinations

Both single crystals were glued onto glass fibers for the structure determination. Before data collection the quality of the crystals was checked by Laue photographs. Data collection was carried out on a Rigaku AFC6R with graphite monochromated Mo Kα radiation and a 12-kW rotating anode. Initial cell parameters of $La_{0.1}Rh_8Sb_{24}$ were obtained from 25 carefully centered reflections in the range $10° \leqslant 2\theta \leqslant 25°$. The structure belongs to the cubic system. Three standard reflections monitored every 150 reflections showed no significant variation in intensity throughout the data collection. Further details of data collection are given in Table 1. The data collected were corrected for Lorentz and polarization effects as well as for absorption effects. The Laue class $m\bar{3}$ and the reflection

Table 1
Crystal data and structure refinement for $La_{0.1}Rh_8Sb_{24}$

Formula	$La_{0.1}Rh_8Sb_{24}$
Formula mass	3759.17
Space group	$Im\bar{3}$
a (Å)	9.2213(15)
b (Å)	9.2213(15)
c (Å)	9.2213(15)
V (Å^3)	784.1(2)
Z	1
T (K)	293(2)
D (×g cm^{-3})	7.961
Diffractometer	Rigaku AFC6R
Crystal dimensions (mm)	0.21×0.06×0.05
Radiation (monochromated In incident beam)	Mo Kα (0.71069 Å)
Data collected	$0 \leqslant h \leqslant 12$, $0 \leqslant k \leqslant 12$, $0 \leqslant l \leqslant 12$
No. of data collected	675
No. of reflections	227
No. of refined parameter	12
$R[F^2 > 2\theta(F^2)]$	0.0189
w$R(F^2)$	0.0314
S	1.157

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Table 2
Atomic coordinates and equivalent isotropic displacement parameters (Å^2) for $La_{0.1}Rh_8Sb_{24}$: $U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \boldsymbol{a}_i \boldsymbol{a}_j$

Atom	Wyckoff position	*x*	*y*	*z*	Occ.	U_{eq}
Sb	24*g*	0.15437(2)	0.34000(3)	0.00000	1	0.00680(13)
Rh	8*c*	1/4	1/4	1/4	1	0.00483(14)
La	2*a*	0.00000	0.00000	0.00000	0.05	0.025(8)

conditions (*hkl*, $h + k + l = 2n$; 0*kl*, $k + l = 2n$; *hhl*, $l = 2n$; *h*00, $h = 2n$) pointed to the possible space groups $Im\bar{3}$, $I2_13$ and *I*23. Final cell parameters for $La_{0.1}Rh_8Sb_{24}$ were obtained from least-squares analysis of 227 reflections. The structure of $La_{0.1}Rh_8Sb_{24}$ was solved using the SHELX97 [8] program package. Based on the similarity of the powder patterns of $La_{0.1}Rh_8Sb_{24}$ with those of $LaFe_4P_{12}$ [9], the space group $Im\bar{3}$ was chosen and examining the initial atomic positions obtained by direct methods confirms that these compounds are isostructural. Final refinement of the structures of both samples gives almost the same results (cubic-shaped single crystal: $a =$ 9.223(1) Å, $R = 0.027$, w$R = 0.0394$, GOF = 1.178, the difference in electron density $\Delta\rho_{max} = 1.557$ e Å^{-3}, $\Delta\rho_{min} = -1.273$ e Å^{-3}). The more accurate results presented here were obtained from the needle-like crystal.

3. Results and discussion

The final results for the atomic position, occupancy and the anisotropic displacement parameter are given in Table 2. Selected interatomic distances in the compound are listed in Table 3. Fig. 1 shows a projection of this structure-type along [100]. Each Rh atom is surrounded by six Sb atoms at distance of 2.6042(4) Å and each La atom is surrounded by 12 Sb atoms at distance of 3.4430(6) Å. The La atom positions (2*a*) are only partially occupied. If the positions are assumed to be vacant, then the final *R* values were $R_1 = 0.0217$ and w$R_2 = 0.0402$ with $\Delta\rho_{max} =$ 3.56 e Å^{-3} instead of $R_1 = 0.0189$ and w$R_2 = 0.0314$ with $\Delta\rho_{max} = 1.47$ e Å^{-3}. The Hamilton test [10] yields a probability of significance for the fractionally occupied model of greater than 0.99995.

Table 3
Selected interatomic distances (Å) for $La_{0.1}Rh_8Sb_{24}$

La–Sb	3.4430(6)	Rh–Sb	2.6042(4)
Sb–Sb	2.8459(6)	Sb–Sb	2.9509(7)
Sb–Sb	3.5130(3)		

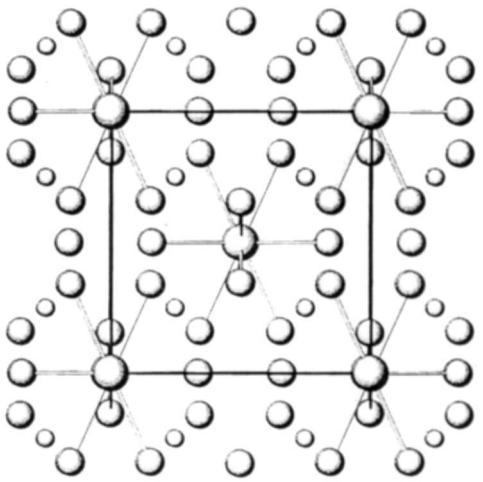

Fig. 1. Projection of cubic $La_{0.1}Rh_8Sb_{24}$ along [100]; large circles, La; medium circles, Sb; small circles, Rh.

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