X-ray powder diffraction data for the $Al_3Ho_2Si_2$ ternary compound

Zhao Lu,[1] Ming Qin,[1,a)] Liuqing Liang,[1] Shuhui Liu,[1] Caiming Huang,[1] Peiling Qing,[1] and Lingmin Zeng[2]
[1]*Department of physics and communication engineering, Baise University, Baise, Guangxi 533000, China*
[2]*Institute of Materials Science, Guangxi University, Nanning, Guangxi 530004, China*

(Received 9 April 2013; accepted 5 May 2013)

Crystal and X-ray powder diffraction data are presented for the $Al_3Ho_2Si_2$ ternary compound. The powder pattern was indexed and refined on a monoclinic cell with the $Al_3Y_2Si_2$ structure type with space group $C12/m1$, $a = 10.1096(2)$ Å, $b = 4.020(6)$ Å, $c = 6.5734(6)$ Å, $\beta = 100.848(2)°$, $V = 262.37$ Å^3, $Z = 2\rho x = 5.910$ g cm^{-3}, $F30 = 142.8(0.006, 35)$, and RIR = 0.91. © *2013 International Centre for Diffraction Data*. [doi:10.1017/S088571561300050X]

Key words: $Al_3Ho_2Si_2$, powder X-ray diffraction

I. INTRODUCTION

Silicides are both scientifically and technologically important, and have been extensively studied (Szytuda and Leciejewicz, 1994). The crystal structure of the $Al_3Ho_2Si_2$ compound was firstly studied by neutron diffraction (Chen *et al.*, 1999). The compound is monoclinic, space group $C12/m1$, $a = 10.083(3)$, $b = 4.0175(9)$, $c = 6.566(1)$ Å, $\beta = 100.73(2)°$, and $Z = 2$. These results agree with those obtained from single-crystal X-ray diffraction data by Bobev *et al.* (2005).

Until now, experimental X-ray powder diffraction pattern for $Al_3Ho_2Si_2$ has not been reported in the PDF (ICDD, 2011). In this paper, we present high-quality powder X-ray diffraction data for well-crystallized $Al_3Ho_2Si_2$.

II. EXPERIMENTAL

A. Synthesis

Polycrystalline specimen of $Al_3Ho_2Si_2$ with a total weight of 2 g was prepared by arc melting the stoichiometric amounts of elemental constituents (99.9 wt% Al, 99.9 wt% Ho, and 99.999 wt% Si) under high purity argon atmosphere. To ensure the homogeneity of the sample, it was melted several times. The weight losses were less than 1 wt%. After melting, the sample was enclosed in an evacuated quartz tube and annealed at 1073 K for 720 h, and finally cooled down to room temperature at a rate of 10 K h^{-1}. The annealed ingot was ground in an agate mortar, pestled to particle sizes of not larger than 10 μm, and then annealed in an evacuated quartz tube at 573 K for 48 h to remove residual stresses.

B. Data collection

Powder X-ray diffraction data were collected at room temperature on a Rigaku D/max 2006 diffractometer with Cu$K\alpha$ radiation and a diffracted-beam graphite monochromator. The diffractometer was operated at 40 kV and 150 mA; the 2θ scan range was from 20 to 100° with a step size of 0.02° and a counting time of 2 s per step. After data collection, the stabilities of both the X-ray source and the specimen were checked by recording again the diffraction lines at low angles. Two sets of XRD data were collected, one with SRM 640 Si added as an internal standard to correct for possible systematical errors in the observed peak positions and the other without SRM 640 Si. The XRD pattern recorded from the specimen added with Si internal standard was used for indexing and for determining space group and unit-cell information, while the XRD pattern without Si internal standard was used for determining diffraction intensities. The $2\theta_{obs}$ values of the peaks were determined by the Savitzky–Golay second derivative using JADE 6.0 (2002) XRD Pattern Processing software of Materials Data, Inc. after smoothing the patterns, fitting and removing the background, and stripping the Cu$K\alpha_2$ peaks. Values of unit-cell parameters were then obtained by the least-squares method using JADE 6.0.

III. RESULTS

The powder diffraction pattern of $Al_3Ho_2Si_2$ (Figure 1) were well indexed with the program JADE 6.0. The compound crystallizes in the C-centered monoclinic with the $Al_3Y_2Si_2$ structure type (space group $C12/m1$), the lattice parameters $a = 10.1096(2)$ Å, $b = 4.020(6)$ Å, $c = 6.5734(6)$ Å, $\beta = 100.848(2)°$, $V = 262.37$ Å^3, $Z = 2$, and $\rho_x = 5.910$ g cm^{-3}. The figure of merit for indexing F_N (Smith and Snyder, 1979) is $F_{30} = 142.8(0.006, 35)$. The value of RIR (RIR = 0.91) was obtained from the value of the ratio of the strongest line in the pattern to the strongest line of corundum in a 50–50 wt% mixture of the two compounds. The observed and the calculated X-ray powder diffraction data for $Al_3Ho_2Si_2$ are listed in Table I.

a)Author to whom correspondence should be addressed. Electronic mail: qm6327@sohu.com

https://doi.org/10.1017/S088571561300050X Published online by Cambridge University Press

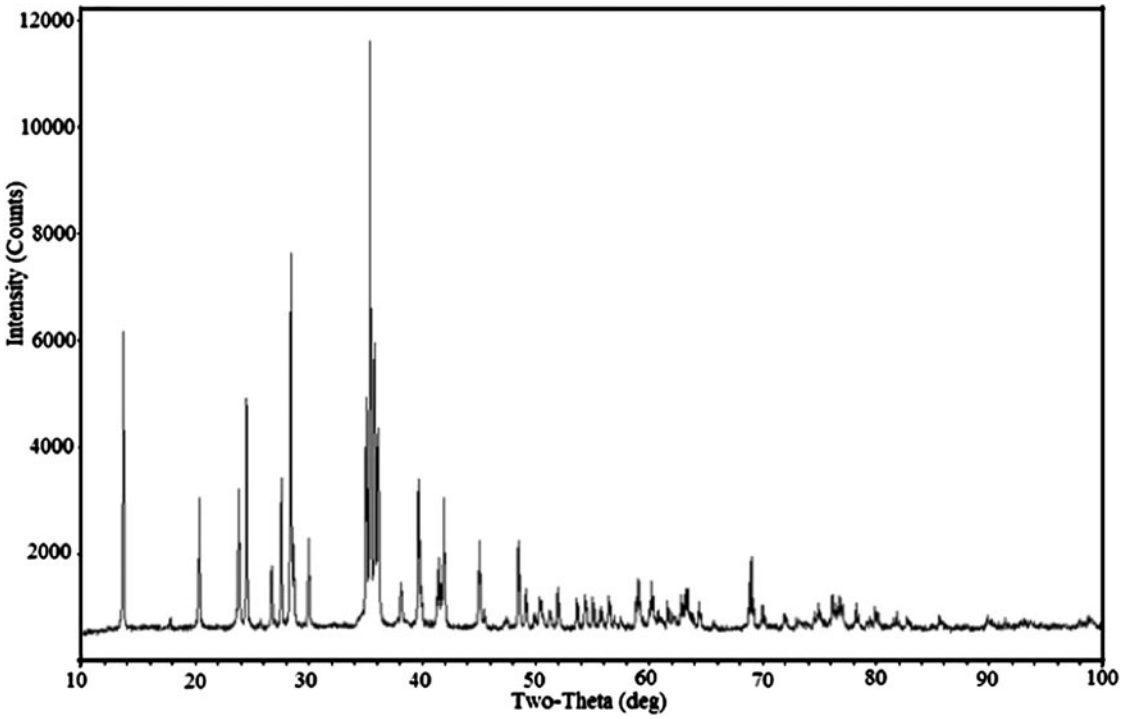


Figure 1. The powder X-ray diffraction pattern of $Al_3Ho_2Si_2$.

TABLE I. Powder diffraction data for $Al_3Ho_2Si_2$ (Cu$K\alpha_1$, with $\lambda = 1.5406$ Å).

No	$2\theta_{obs.}$	h	k	l	$2\theta_{cal.}$	$\Delta 2\theta$	I/I_0	$d_{obs.}$	$d_{cal.}$
1	13.695	0	0	1	13.705	−0.010	22.7	6.4604	6.4559
2	17.841	2	0	0	17.852	−0.011	0.8	4.9675	4.9645
3	20.381	−2	0	1	20.394	−0.013	22.5	4.3538	4.3510
4	23.856	1	1	0	23.861	−0.005	23.4	3.7270	3.7262
5	24.562	2	0	1	24.572	−0.010	39.1	3.6213	3.620
6	26.738	−1	1	1	26.743	−0.005	10.5	3.3313	3.3307
7	27.601	0	0	2	27.611	−0.010	25.4	3.2292	3.2280
8	28.445	1	1	1	28.467	−0.022	64.5	3.1352	3.1328
9	30.003	−2	0	2	30.021	−0.018	15.3	2.9759	2.9741
10	35.095	3	1	0	35.091	0.004	35.8	2.5549	2.5551
11	35.443	−1	1	2	35.454	−0.011	100.0	2.5306	2.5298
12	35.840	−3	1	1	35.838	0.002	47.5	2.5035	2.5035
13	35.898	2	0	2	35.895	0.003	34.5	2.4996	2.4997
14	36.162	4	0	0	36.157	0.005	32.1	2.4819	2.4822
15	36.224	−4	0	1	36.215	0.009	34.1	2.4778	2.4784
16	38.123	1	1	2	38.121	0.002	7.4	2.3586	2.3587
17	39.757	3	1	1	39.748	0.009	25.3	2.2654	2.2658
18	41.321	4	0	1	41.318	0.003	4.5	2.1832	2.1833
19	41.475	−4	0	2	41.473	0.002	11.6	2.1754	2.1755
20	41.742	−3	1	2	41.741	0.001	4.6	2.1621	2.1622
21	41.940	0	0	3	41.947	−0.007	22.3	2.1524	2.1520
22	45.059	0	2	0	45.067	−0.008	15.2	2.0103	2.0100
23	47.214	−1	1	3	47.219	−0.005	1.0	1.9235	1.9233
24	47.321	0	2	1	47.327	−0.006	1.1	1.9194	1.9191
25	48.501	3	1	2	48.506	−0.005	14.9	1.8754	1.8752
26	49.178	2	0	3	49.171	0.007	6.7	1.8512	1.8514
27	49.939	−2	2	1	49.939	0.000	1.8	1.8247	1.8247
28	50.397	1	1	3	50.403	−0.006	5.6	1.8092	1.8090
29	50.594	−4	0	3	50.596	−0.002	4.0	1.8026	1.8026
30	51.319	−3	1	3	51.339	−0.020	2.0	1.7788	1.7782
31	51.998	2	2	1	51.996	0.002	7.0	1.7572	1.7573
32	53.677	0	2	2	53.673	0.004	4.9	1.7061	1.7063
33	54.420	−5	1	2	54.448	−0.028	5.6	1.6846	1.6838

Continued

https://doi.org/10.1017/S088571561300050X Published online by Cambridge University Press

TABLE I. Continued

No	$2\theta_{obs.}$	h	k	l	$2\theta_{cal.}$	$\Delta 2\theta$	I/I_0	$d_{obs.}$	$d_{cal.}$
34	55.097	−2	2	2	55.102	−0.005	4.9	1.6655	1.6653
35	55.777	5	1	1	55.783	−0.006	3.2	1.6468	1.6466
36	56.494	−2	0	4	56.492	0.002	3.9	1.6276	1.6276
37	57.008	0	0	4	57.012	−0.004	1.8	1.6141	1.614
38	57.561	−6	0	2	57.561	0.000	1.6	1.5999	1.5999
39	58.918	2	2	2	58.911	0.007	4.6	1.5663	1.5664
40	59.101	4	2	0	59.090	0.011	8.1	1.5618	1.5621
41	60.055	3	1	3	60.048	0.007	2.3	1.5393	1.5394
42	60.240	6	0	1	60.239	0.001	7.3	1.5350	1.535
43	60.874	−1	1	4	60.865	0.009	1.8	1.5205	1.5207
44	61.704	−5	1	3	61.710	−0.006	4.1	1.5021	1.5019
45	62.122	4	0	3	62.125	−0.003	1.9	1.4929	1.4929
46	62.898	−4	2	2	62.90	−0.002	3.8	1.4764	1.4763
47	63.258	0	2	3	63.254	0.004	6.1	1.4688	1.4689
48	63.480	−3	1	4	63.492	−0.012	6.3	1.4642	1.464
49	64.462	1	1	4	64.462	0.000	4.2	1.4443	1.4443
50	68.901	2	2	3	68.895	0.006	7.9	1.3617	1.3618
51	69.088	−7	1	1	69.080	0.008	12.3	1.3584	1.3586
52	70.057	−4	2	3	70.058	−0.001	3.8	1.3420	1.342
53	71.963	−5	1	4	71.972	−0.009	2.5	1.3111	1.3109
54	73.040	1	3	1	73.043	−0.003	1.7	1.2944	1.2943
55	73.362	−6	2	1	73.364	−0.002	1.3	1.2895	1.2894
56	73.801	3	1	4	73.795	0.006	1.0	1.2829	1.283
57	74.640	5	1	3	74.641	−0.001	1.6	1.2705	1.2705
58	75.035	−2	2	4	75.028	0.007	2.6	1.2648	1.2649
59	76.242	−1	1	5	76.255	−0.013	4.9	1.2478	1.2476
60	76.719	8	0	0	76.725	−0.006	2.4	1.2412	1.2411
61	76.862	−8	0	2	76.865	−0.003	6.0	1.2393	1.2392
62	77.099	−3	3	1	77.101	−0.002	3.6	1.2360	1.236
63	78.302	6	2	1	78.307	−0.005	4.1	1.220	1.22
64	79.980	4	2	3	79.990	−0.010	3.5	1.1986	1.1985
65	82.803	7	1	2	82.801	0.002	2.2	1.1647	1.1648
66	85.631	−7	1	4	85.622	0.009	1.6	1.1334	1.1335
67	89.886	3	1	5	89.890	−0.004	2.0	1.0904	1.0904
68	90.341	−5	3	2	90.344	−0.003	1.0	1.0861	1.0861
69	91.426	0	0	6	91.431	−0.005	1.6	1.076	1.076
70	93.218	6	0	4	93.217	0.001	0.5	1.060	1.06
71	96.523	−5	3	3	96.518	0.005	0.7	1.0323	1.0323
72	98.118	−3	3	4	98.113	0.005	1.0	1.0197	1.0198
73	98.764	1	1	6	98.757	0.007	1.6	1.0148	1.0148
74	99.021	2	0	6	99.018	0.003	1.9	1.0128	1.0129

ACKNOWLEDGEMENT

This work was supported by the Natural Science Foundation of Guangxi (No. 2011GXNSFA018034).

Bobev, S., Tobash, P. H., Fritsch, V., Thompson, J. D., Hundley, M. F., Sarrao, J. L., and Fisk, Z. (**2005**). "Ternary rare-earth alumo-silicides–single-crystal growth from Al flux, structural and physical properties," J. Solid State Chem. **178**, 2091–2103.

Chen, X.-Z., Sieve, B., Henning, R., Schultz, A. J., Brazis, P., Kannewurf, C. R., Cowen, J. A., Crosby, R., and Kanatzidis, M. G. (**1999**). "$Ln_2Al_3Si_2$ (Ln = Ho, Er, Tm): new silicides from molten aluminium – determination of the Al/Si distribution with neutron crystallography and metamagnetic transitions," Angew. Chem. Int. ed. **38**, 693–696.

ICDD (**2011**). PDF-2 2011 (Database), edited by S. Kabekkodu (International Centre for Diffraction Data, Newtown Square, PA, USA).

JADE Version 6.0 (**2002**). "XRD pattern processing," Materials Data Inc., Livermore, CA.

Smith, G. S. and Snyder, R. L. (**1979**). "$F_{N:}$ a criterion for rating powder diffraction patterns and evaluating the reliability of powder pattern indexing," J. Appl. Cryst. **12**, 60.

Szytuda, A. and Leciejewicz, J. (**1994**). in CRC Handbook of Crystal Structures and Magnetic Properties of Rare Earth Intermetallics (CRC Press, Boca Raton, FL), p. 110.

https://doi.org/10.1017/S088571561300050X Published online by Cambridge University Press