X-ray powder diffraction pattern and crystal structure of $AlCu_4Y$ ternary compound

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The ternary compound of $AlCu_4Y$ was synthesized by melting under argon atmosphere in the arc furnace. High-quality X-ray powder diffraction data of $AlCu_4Y$ have been collected using a Rigaku SmartLab X-ray powder diffractometer. The Rietveld refinement results of the X-ray diffraction pattern for the $AlCu_4Y$ compound showed that the $AlCu_4Y$ is the hexagonal structure, space-group *P6/mmm* (No. 191) with $a = 5.0658(1)$ Å, $c = 4.1569(1)$ Å, $V = 92.38$ Å^3, $Z = 1$, and the density is 6.66 g cm^{-3}, and the intensity ratio RIR is 1.96. © *2015 International Centre for Diffraction Data.* [doi:10.1017/S0885715615000627]

Key words: $AlCu_4Y$, X-ray powder diffraction, crystal structure, Rietveld refinement

I. INTRODUCTION

The phase diagram of the Al–Cu–Y ternary system has been studied deeply, and the ternary compounds of Al–Cu–Y system were studied extensively. At the same time, several ternary compounds have been reported, such as $Al_{7.6}Cu_{4.4}Y$, AlCuY, $Al_7Cu_2Y_3$, $Al_{8.43}Cu_{2.59}Y_3$, $Al_{5.4}Cu_{11.6}Y_2$, $Al_{1.05}Cu_{9.93}Y$, $Al_{4.745}Cu_{6.255}Y$ (Krachan *et al.*, 2003), Al_8Cu_4Y (Buschow *et al.*, 1976), Al_6Cu_6Y (Felner, 1980), $Al_{2.04}Cu_{2.96}Y$ (Kadir *et al.*, 2001), Al_7Cu_5Y (Drake *et al.*, 2010), but there were no reports about the $AlCu_4Y$ ternary compound. The related compounds with the similar crystal structures had been reported too, such as $AlCo_4RE$ (*RE* = Y, Dy, Gd, Nd, Pr, Sm, Tm, Tb, Ce, La), $AlNi_4RE$ (RE = Ce, Dy, Er, Gd, Ho, La, Nd, Pr, Sm Tb, Tm), and $AlCu_4RE$ (*RE* = Ce, Dy, Er, Gd, Ho, La, Nd, Pr, Sm, Tb, Tm, Yb) (ICSD Database, 2014.02). In this paper, we present the X-ray powder diffraction pattern and crystal structure with the Rietveld refinement method for the well-crystallized $AlCu_4Y$ compound.

II. EXPERIMENTAL

A. Synthesis

The sample of high-purity $AlCu_4Y$ compound was prepared by melting with stoichiometric elemental constituents with 99.99 wt% Al, 99.99 wt% Cu, and 99.9 wt% Y coming from China New Metal Materials Technology Co., Ltd. under high-purity argon atmosphere in a vacuum arc furnace. In the process of melting, titanium was melted firstly as an oxygen capture agent. In order to fuse together and uniformly of the composition elements, melting processes (three times in the experiment) have been executed many times, and the weight losses of the sample were <1 wt%. The sample of the $AlCu_4Y$ compound was enclosed in an evacuated quartz tube and annealed at the temperature of 1123 K for 30 days, and then cooled down to room temperature by the rate of 18 K h^{-1}. Finally, the compound was ground to powder with the particle size smaller than 10 μm in a steel mortar.

B. Data collection

The X-ray powder diffraction data of $AlCu_4Y$ ternary compound were collected at room temperature by the Rigaku SmartLab X-ray powder diffractometer, and using the Cu$K\alpha$ radiation equipped with a diffraction beam graphite monochromator. The diffractometer was operated at voltage 40 kV and current 150 mA, and the scan range of Bragg angle (2θ) was from 10° to 100° with the step size of 0.02° and the count-time of 2 s step^{-1}. In order to reduce the instrumental errors, the internal standard method with high-purity silicon has been used, and the X-ray powder diffraction data for the mixture of $AlCu_4Y$ and SRM Si were collected. The whole pattern of $AlCu_4Y$ was calibrated by linear fit with reference material SRM Si. The RIR value was calculated by the highest peak intensity ratio between $AlCu_4Y$ and corundum from the X-ray powder diffraction pattern that were collected by the mixture of 50 wt.% $AlCu_4Y$ and 50 wt.% corundum.

III. CONCLUSIONS

All peaks were indexed successfully in the experimental X-ray diffraction pattern of $AlCu_4Y$ by Jade 6.5 (Materials Data Inc., 2002) software with the hexagonal structure. The Smith–Snyder figure-of-merit F_N (Smith and Snyder, 1979) is $F_{28} = 169.8(0.0061, 33)$. The intensity ratio RIR value is 1.96. The X-ray powder diffraction data for $AlCu_4Y$ are listed in Table I. It was found that $AlCu_4Y$ and $AlCo_4Y$ (Ido *et al.*, 1990) have the similar crystal structure type (*P6/mmm*, No.191) by comparing the crystal structure information of $AlCu_4Y$ with that of $AlCo_4Y$. The cell parameters were

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TABLE I. X-ray powder diffraction data for $AlCu_4Y$ ($CuK\alpha_1$, with $\lambda = 1.5406$ Å)

No.	h	k	l	$2\theta_{cal}$	$2\theta_{obs}$	$\Delta 2\theta$[a]	I/I_0	d_{cal}	d_{obs}	Δd[b]
1	1	0	0	20.241	20.248	−0.007	4.3	4.3835	4.3821	0.0014
2	0	0	1	21.365	21.371	−0.006	8.3	4.1555	4.1543	0.0012
3	1	0	1	29.597	29.603	−0.006	29.7	3.0158	3.0151	0.0007
4	1	1	0	35.439	35.439	0	32.3	2.5308	2.5309	−0.0001
5	2	0	0	41.152	41.158	−0.006	26.4	2.1917	2.1914	0.0003
6	1	1	1	41.754	41.758	−0.004	100	2.1615	2.1613	0.0002
7	0	0	2	43.521	43.524	−0.003	33	2.0777	2.0776	0.0001
8	2	0	1	46.823	46.820	0.003	15.7	1.9386	1.9387	−0.0001
9	1	0	2	48.443	48.445	−0.002	1.5	1.8775	1.8774	0.0001
10	1	1	2	57.327	57.338	−0.011	13.6	1.6059	1.6056	0.0003
11	2	1	1	60.067	60.058	0.009	4.3	1.5390	1.5392	−0.0002
12	2	0	2	61.439	61.439	0	11.8	1.5079	1.5079	0
13	3	0	0	63.629	63.636	−0.007	4.7	1.4612	1.4611	0.0001
14	0	0	3	67.572	67.572	0	2.7	1.3852	1.3852	0
15	3	0	1	67.947	67.940	0.007	10.3	1.3784	1.3786	−0.0002
16	1	0	3	71.351	71.358	−0.007	2.2	1.3208	1.3207	0.0001
17	2	2	0	74.994	75.000	−0.006	7.3	1.2654	1.2653	0.0001
18	3	1	0	78.629	78.629	0	6.9	1.2158	1.2158	0
19	1	1	3	78.682	78.680	0.002	10.3	1.2151	1.2151	0
20	2	2	1	79.036	79.038	−0.002	1.4	1.2105	1.2105	0
21	3	0	2	80.253	80.244	0.009	3.2	1.1952	1.1953	−0.0001
22	2	0	3	82.271	82.278	−0.007	4.1	1.1709	1.1708	0.0001
23	3	1	1	82.621	82.618	0.003	1.9	1.1668	1.1669	−0.0001
24	4	0	0	89.319	89.321	−0.002	1.5	1.0959	1.0958	0.0001
25	2	2	2	90.915	90.908	0.007	7.6	1.0807	1.0808	−0.0001
26	2	1	3	92.911	92.919	−0.008	1.7	1.0627	1.0626	0.0001
27	4	0	1	93.258	93.259	−0.001	0.8	1.0596	1.0596	0
28	0	0	4	95.712	95.702	0.010	1.8	1.0390	1.0390	0

[a]$\Delta 2\theta = 2\theta_{cal} - 2\theta_{obs}$.
[b]$\Delta d = d_{cal} - d_{obs}$.

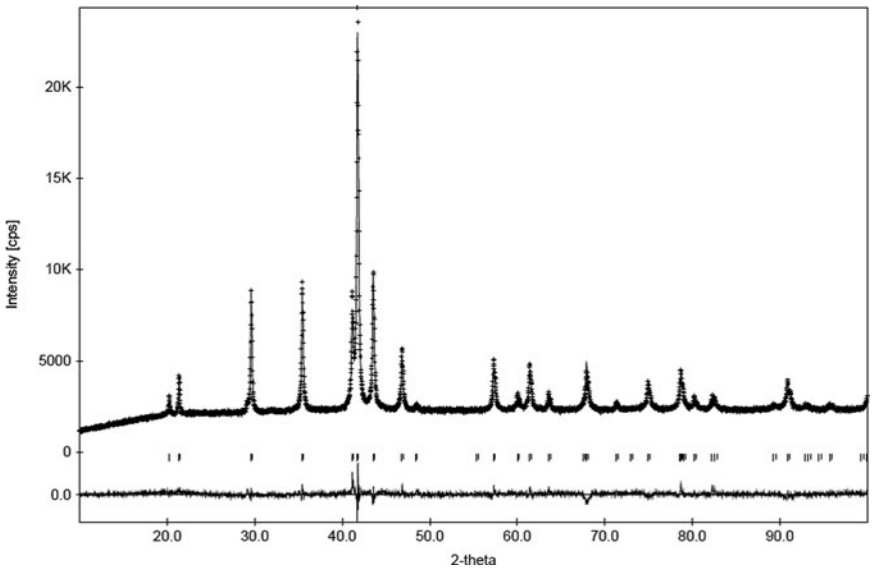


Figure 1. Observed, calculated, and residuals of X-ray powder diffraction pattern for $AlCu_4Y$ after refinement.

TABLE II. Atomic positions and occupancy of $AlCu_4Y$ with Rietveld refinement.

Atom	Position	X	Y	Z	SOF
Al	2*c*	0.3333	0.6667	0	0.0176(1)
Cu	2*c*	0.3333	0.6667	0	0.9824(1)
Al	3*g*	0.5	0	0.5	0.3187(1)
Cu	3*g*	0.5	0	0.5	0.6813(1)
Y	1*a*	0	0	0	1

obtained with $a = 5.0658(1)$ Å, $c = 4.1569(1)$ Å, $V = 92.38$ Å^3, $Z = 1$, and the density is 6.66 g cm^{-3} by Rietveld refinement of the X-ray powder diffraction pattern with DBWS9807 program (Young, 2000). The observed, calculated, and residuals of X-ray powder diffraction pattern for $AlCu_4Y$ after the Rietveld refinement are showed in Figure 1, and the atomic positions and occupancy of $AlCu_4Y$ after Rietveld refinement are listed in Table II with the goodness-of-fit parameters: $R_p = 2.57\%$, $R_{wp} = 3.34\%$, $R_B = 6.70\%$, $R_F = 5.27\%$, and $S = 1.69$.

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TABLE III. Distances between the selected atoms or atom groups for $AlCu_4Y$.

Atom group–atom group		Distance (Å)	Multiplicity	Atom group–atom group		Distance (Å)	Multiplicity
$Al_{0.0176}Cu_{0.9824}$	$Al_{0.3187}Cu_{0.6813}$	2.5413	4×	Y	$Al_{0.0176}Cu_{0.9824}$	2.9244	2×
	$Al_{0.3187}Cu_{0.6813}$	2.5415	2×		$Al_{0.0176}Cu_{0.9824}$	2.9249	4×
	$Al_{0.0176}Cu_{0.9824}$	2.9244	1×		$Al_{0.3187}Cu_{0.6813}$	3.2765	12×
	Y	2.9244	1×		Y	4.1569	2×
	$Al_{0.0176}Cu_{0.9824}$	2.9249	2×				
	Y	2.9249	2×				
$Al_{0.3187}Cu_{0.6813}$	$Al_{0.3187}Cu_{0.6813}$	2.5329	4×				
	$Al_{0.0176}Cu_{0.9824}$	2.5413	4×				
	Y	3.2765	4×				

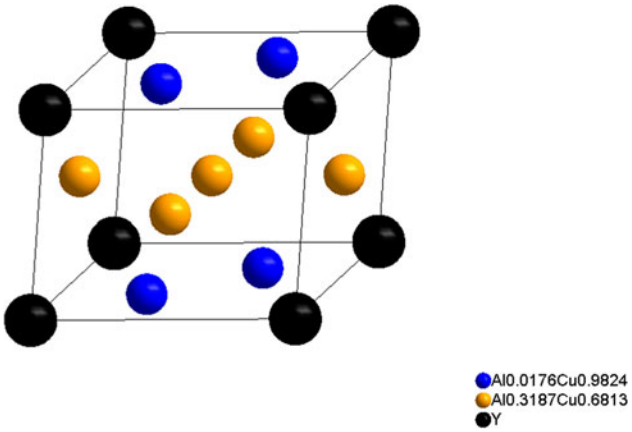


Figure 2. (Colour online) Unit-cell structure of $AlCu_4Y$.

The SOF of Al atom is 0.3187(1) in 3*g* positions, but only 0.0176(1) in 2*c* positions, and the total Al atoms in unit cell are 0.9913 that match with the chemical formula of $AlCu_4Y$. The Al atom prefers to occupy the 3*g* position because the atomic radius of Al is 1.43 Å that is bigger than the atomic radius of Cu with 1.28 Å, and the 3*g* position has larger gap than the 2*c* position. But if the 2*c* positions are all occupied by Cu atoms, then the goodness-of-fit parameters become worse with $R_p = 2.82\%$, $R_{wp} = 3.93\%$, $R_B = 9.42\%$, $R_F = 7.41\%$, and $S = 1.99$, and the total Al atoms are 0.9798 in unit cell. So it is appropriate that Al atoms occupy 2*c* positions with SOF 0.0176 and 3*g* positions with SOF 0.3187. The structure diagram of unit cell for $AlCu_4Y$ has been drawn and shown in Figure 2. The distances between the selected atoms or atom groups for $AlCu_4Y$ have been listed in Table III.

SUPPLEMENTARY MATERIAL

For supplementary material for this article, please visit http://dx.doi.org/10.1017/S0885715615000627.

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Buschow, K. H. J., van Vucht, J. H. N., and van den Hoogenhof, W. W. (**1976**). "Note on the crystal structure of the ternary rare earth – 3d transition metal compounds of the type RT_4Al_8," J. Less-Common Met. **50**, 145–150.

Drake, B. L., Capan, C., Young, C. J., Nambu, Y., Kuga, K., Xiong, Y. M., Karki, A. B., Nakatsuji, S., Adams, P. W., Young, D. P., and Chan, J. Y. (**2010**). "Crystal growth, structure and physical properties of Ln $(Cu, Al)_{12}$ (Ln = Y, Ce, Pr, Sm and Yb) and Ln $(Cu, Ga)_{12}$ (Ln = Y, Gd, Er and Yb)," J. Phys.: Condens. Matter **22**, 1–14.

Felner, I. (**1980**). "Crystal structures of ternary rare-earth-3d transition metal compounds of the RT_6Al_6 type," J. Less-Common Met. **72**, 241–249.

Ido, H., Konno, K., Cheng, S. F., Wallace, W. E., and Sankar, S. G. (**1990**). "Effect of Al-substitution on the magnetic properties of RCo_5 (R = rare earth)," J. Appl. Phys. **67**, 4638–4640.

Inorganic Crystal Structure Database (**2014**). *Fachinformationszentrum* (U.S. Department of Commerce on the behalf of the United States, Karlsruhe, Germany).

Kadir, K., Sakai, T., Uehara, I., and Eriksson, L. (**2001**). "YCu_3Al_2, an example of an AB_5 structure type," Acta Crystallogr. C: Cryst. Struct. Commun. **57**, 999–1000.

Krachan, T., Stel'makhovich, B. M., and Kuz'ma, Yu. B. (**2003**). "The Y–Cu–Al system," J. Alloys Compd. **349**, 134–139.

Materials Data Inc. (**2002**). *JADE Version 6. XRD Pattern Processing* (Materials Data Inc., Livermore, CA).

Smith, G. S., and Snyder, R. L. (**1979**). "FN: a criterion for rating powder diffraction patterns and evaluating the reliability of powder-pattern indexing," J. Appl. Crystallogr. **12**, 60–65.

Young, R. A., Larson, A. C., and Paiva-Santos, C. O. (**2000**). "*User's Guide to Program DBWS9807a for Rietveld Analysis of X-ray and Neutron Powder Diffraction Patterns with a PC and Various Other Computers*," (School of Physics, Georgia Institute of Technology, Atlanta, GA).

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