XRPD and Rietveld refinement for Al_5NdNi_2 compound

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A new ternary compound Al_5NdNi_2 was prepared by melting a stoichiometric mixture of aluminum, neodymium, and nickel in an arc furnace and annealing in vacuum. The crystal structure of Al_5NdNi_2 was studied by X-ray powder diffraction technique and Rietveld analysis. All diffraction lines of Al_5NdNi_2 were indexed, and the lattice parameters were refined with an orthorhombic structure type of space group *Immm* (No.71) using Rietveld analysis program DBWS-9807. The lattice parameters are presented, $a = 7.0508(1)$ Å, $b = 9.5690(1)$ Å, $c = 3.9792(1)$ Å, $V = 268.47$ Å^3, $Z = 2$, $\rho = 4.91$ g cm^{-3}, and $RIR = 1.23$. © *2018 International Centre for Diffraction Data*.
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Key words: Al_5NdNi_2, X-ray powder diffraction, Rietveld refinement

I. INTRODUCTION

A large number of crystal structures of compounds in the Al–Nd–Ni ternary system have been reported in the ICSD (Inorganic Crystal Structure Database, 2015), such as AlNdNi, $AlNdNi_4$, $AlNd_2Ni_2$, $AlNd_3Ni_8$, Al_3NdNi_2, Al_4NdNi, etc. To our knowledge, there is no report about Al_5NdNi_2 compound. In this paper, the experimental X-ray powder diffraction (XRPD) pattern is presented, and the crystal structure of Al_5NdNi_2 is studied by the XRPD technique and Rietveld analysis.

II. EXPERIMENTAL

A. Al_5NdNi_2 preparation

The raw materials of pure metal with 99.99 wt.% aluminum, 99.99 wt.% neodymium, and 99.99 wt.% nickel were supplied by China New Metal Materials Technology Co., Ltd. The compound of Al_5NdNi_2 was prepared by melting the stoichiometric composition under argon atmosphere in an electric arc furnace. The total mass of the Al_5NdNi_2 sample is 2 g with the composition proportion of 34.02 wt.% Al, 36.38 wt.% Nd, and 29.60 wt.% Ni. In order to capture the residual oxygen, a titanium ingot was melted first before the alloy sample melting. In order to ensure that these elements fused together completely and the composition distributed uniformly, the sample was melted at least three times while being turned over in the gap. The melting processes were considered to be successful when the weight losses of the sample were <1 wt.%. Then, the sample ingot was enclosed in an evacuated quartz glass tube and annealed at the temperature of 1103 K for 1 month, and then cooled down at the rate of 0.2 K min^{-1} to ambient temperature. A sample was prepared for X-ray diffraction (XRD) testing by grinding Al_5NdNi_2 granules in a steel mortar.

B. Data collection and analyses

The XRPD data of Al_5NdNi_2 ternary compound were collected at the room temperature using the Rigaku Smart Lab (9) powder diffractometer which was equipped with a copper rotating anode powered with a voltage of 40 kV and current of 150 mA, and a diffracted-beam graphite monochromator. The goniometer radius is 300 mm, and the diffractometer was operated with the incident slit 1/2° and the receiving slit 0.3 mm. The scan range of diffraction angle (2θ) was from 10° to 100° with stepping-scanning mode, step size 0.02°, and 2.5 seconds per step. In order to calibrate systematic errors of 2θ locations in the experimental data, the internal standard method was employed, and the XRPD data for Al_5NdNi_2 mixed with high purity silicon as the internal standard material was collected.

The observed values of 2θ of the diffraction lines were chosen by the peak searching function of Jade 6.5 XRD pattern processing software (Materials Data Inc., 2002) based on the Savitzky–Golay 2nd derivatives combined with the counting statistics of intensity data. Structure refinement of Al_5NdNi_2 was performed by the Rietveld method using the

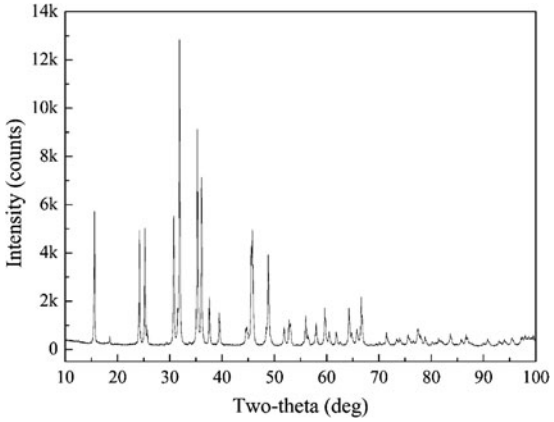

Figure 1. Experimental XRPD pattern of Al_5NdNi_2.

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TABLE I. Calculated and observed values of XRPD data for Al_5NdNi_2 (Cu $K\alpha_1$, with $\lambda = 1.5406$ Å).

No.	h	k	l	$2\theta_{cal}$	$2\theta_{obs}$	$\Delta 2\theta$[a]	I/I_o	d_{cal}	d_{obs}	Δd[b]
1	1	1	0	15.601	15.600	0.001	37.8	5.6753	5.6756	−0.0003
2	0	2	0	18.531	18.530	0.001	2.2	4.784	4.7844	−0.0004
3	0	1	1	24.210	24.211	−0.001	36.1	3.6731	3.673	0.0001
4	2	0	0	25.247	25.241	0.006	36.4	3.5247	3.5255	−0.0008
5	1	0	1	25.693	25.693	0	4.4	3.4644	3.4644	0.0000
6	1	3	0	30.744	30.748	−0.004	39.5	2.9058	2.9054	0.0004
7	2	2	0	31.501	31.507	−0.006	9.9	2.8377	2.8372	0.0005
8	1	2	1	31.867	31.871	−0.004	100	2.8059	2.8056	0.0003
9	2	1	1	35.261	35.261	0	72.3	2.5432	2.5432	0.0000
10	0	3	1	36.065	36.062	0.003	56.7	2.4883	2.4885	−0.0002
11	0	4	0	37.571	37.575	−0.004	16.4	2.392	2.3917	0.0003
12	3	1	0	39.455	39.460	−0.005	10.6	2.282	2.2817	0.0003
13	2	3	1	44.535	44.539	−0.004	4.4	2.0328	2.0326	0.0002
14	3	0	1	44.758	44.752	0.006	4.4	2.0232	2.0234	−0.0002
15	0	0	2	45.570	45.570	0	33.5	1.989	1.989	0.0000
16	2	4	0	45.807	45.808	−0.001	36.6	1.9793	1.9792	0.0001
17	1	4	1	46.074	46.074	0	4.5	1.9684	1.9684	0.0000
18	3	3	0	48.055	48.050	0.005	0.2	1.8918	1.8919	−0.0001
19	1	1	2	48.456	48.460	−0.004	5.6	1.877	1.8769	0.0001
20	3	2	1	48.834	48.840	−0.006	31.5	1.8634	1.8632	0.0002
21	1	5	0	49.303	49.301	0.002	3	1.8468	1.8468	0.0000
22	0	2	2	49.595	49.595	0	0.2	1.8366	1.8366	0.0000
23	4	0	0	51.835	51.837	−0.002	5.6	1.7623	1.7623	0.0000
24	2	0	2	52.806	52.814	−0.008	8.9	1.7322	1.732	0.0002
25	0	5	1	53.062	53.072	−0.01	6.8	1.7244	1.7242	0.0002
26	4	2	0	55.523	55.515	0.008	0.2	1.6537	1.6539	−0.0002
27	1	3	2	55.979	55.987	−0.008	10.7	1.6413	1.6411	0.0002
28	2	2	2	56.450	56.450	0	3	1.6287	1.6287	0.0000
29	0	6	0	57.768	57.763	0.005	3.2	1.5947	1.5948	−0.0001
30	4	1	1	57.997	57.991	0.006	7.3	1.5889	1.589	−0.0001
31	2	5	1	59.640	59.651	−0.011	12.9	1.549	1.5487	0.0003
32	3	4	1	59.822	59.826	−0.004	3.3	1.5447	1.5446	0.0001
33	0	4	2	60.486	60.495	−0.009	4.1	1.5293	1.5291	0.0002
34	3	1	2	61.826	61.823	0.003	4.8	1.4994	1.4994	0.0000
35	3	5	0	62.547	62.541	0.006	1	1.4838	1.4839	−0.0001
36	2	6	0	64.034	64.026	0.008	1.8	1.4529	1.4531	−0.0002
37	1	6	1	64.248	64.243	0.005	13.2	1.4486	1.4487	−0.0001
38	4	3	1	64.769	64.779	−0.010	3.5	1.4382	1.438	0.0002
39	4	4	0	65.762	65.772	−0.010	4.8	1.4188	1.4186	0.0002
40	2	4	2	66.602	66.611	−0.009	16.7	1.403	1.4028	0.0002
41	5	1	0	67.043	67.037	0.006	0.7	1.3948	1.3949	−0.0001
42	3	3	2	68.380	68.375	0.005	0.5	1.3708	1.3708	0.0000
43	1	5	2	69.384	69.383	0.001	0.7	1.3533	1.3534	−0.0001
44	1	7	0	70.065	70.078	−0.013	1.4	1.3419	1.3416	0.0003
45	5	0	1	70.852	70.851	0.001	0.7	1.3289	1.3289	0.0000
46	4	0	2	71.460	71.460	0	4.7	1.319	1.3191	−0.0001
47	0	1	3	71.813	71.812	0.001	0.8	1.3134	1.3134	0.0000
48	0	7	1	73.151	73.155	−0.004	0.4	1.2927	1.2926	0.0001
49	5	3	0	73.361	73.362	−0.001	2	1.2895	1.2895	0.0000
50	5	2	1	73.969	73.969	0	2.5	1.2804	1.2804	0.0000
51	4	2	2	74.567	74.568	−0.001	0.6	1.2716	1.2716	0.0000
52	1	2	3	75.561	75.558	0.003	3.6	1.2573	1.2574	−0.0001
53	3	6	1	75.910	75.910	0	1.5	1.2524	1.2524	0.0000
54	0	6	2	76.503	76.502	0.001	1.1	1.2442	1.2442	0.0000
55	4	5	1	77.357	77.356	0.001	3.7	1.2325	1.2326	−0.0001
56	2	1	3	77.491	77.490	0.001	5.2	1.2308	1.2308	0.0000
57	0	3	3	77.970	77.972	−0.002	2.9	1.2244	1.2244	0.0000
58	2	7	1	78.794	78.799	−0.005	3	1.2136	1.2136	0.0000
59	0	8	0	80.189	80.186	0.003	1.2	1.196	1.196	0.0000
60	3	5	2	80.732	80.731	0.001	0.8	1.1893	1.1893	0.0000
61	4	6	0	81.299	81.299	0	0.6	1.1824	1.1824	0.0000
62	3	7	0	81.378	81.379	−0.001	2.5	1.1815	1.1815	0.0000
63	6	0	0	81.933	81.934	−0.001	1.2	1.1749	1.1749	0.0000
64	2	6	2	82.075	82.079	−0.004	1	1.1732	1.1732	0.0000
65	5	4	1	83.072	83.078	−0.006	0.5	1.1616	1.1616	0.0000
66	2	3	3	83.517	83.517	0	2.1	1.1566	1.1566	0.0000

Continued

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TABLE I. Continued

No.	h	k	l	$2\theta_{cal}$	$2\theta_{obs}$	$\Delta 2\theta$[a]	I/I_o	d_{cal}	d_{obs}	Δd[b]
67	4	4	2	83.652	83.651	0.001	4.1	1.1551	1.1551	0.0000
68	3	0	3	83.675	83.678	−0.003	3.8	1.1548	1.1548	0.0000
69	1	4	3	84.618	84.618	0	0.3	1.1443	1.1443	0.0000
70	5	1	2	84.832	84.832	0	0.4	1.142	1.142	0.0000
71	6	2	0	84.924	84.921	0.003	0.3	1.141	1.141	0.0000
72	5	5	0	85.472	85.473	−0.001	0.5	1.1351	1.1351	0.0000
73	2	8	0	85.705	85.709	−0.004	1.3	1.1326	1.1325	0.0001
74	1	8	1	85.898	85.899	−0.001	0.8	1.1305	1.1305	0.0000
75	3	2	3	86.657	86.659	−0.002	3.6	1.1226	1.1225	0.0001
76	6	1	1	86.998	87.001	−0.003	1.2	1.119	1.119	0.0000
77	1	7	2	87.650	87.655	−0.005	0.5	1.1124	1.1123	0.0001
78	0	5	3	89.941	89.939	0.002	1	1.0899	1.0899	0.0000
79	5	3	2	90.780	90.783	−0.003	2.3	1.082	1.082	0.0000
80	6	3	1	92.942	92.944	−0.002	1.5	1.0624	1.0624	0.0000
81	6	4	0	93.846	93.842	0.004	1.2	1.0545	1.0546	−0.0001
82	4	1	3	94.011	94.008	0.003	2	1.0531	1.0532	−0.0001
83	1	9	0	94.234	94.235	−0.001	0.8	1.0512	1.0512	0.0000
84	4	7	1	95.292	95.294	−0.002	1.7	1.0423	1.0423	0.0000
85	2	5	3	95.422	95.420	0.002	2.5	1.0413	1.0413	0.0000
86	3	4	3	95.580	95.581	−0.001	1.1	1.04	1.0399	0.0001
87	3	8	1	96.863	96.860	0.003	0.1	1.0296	1.0296	0.0000
88	0	9	1	97.178	97.175	0.003	1.6	1.0271	1.0271	0.0000
89	0	8	2	97.444	97.443	0.001	1.3	1.025	1.025	0.0000
90	5	6	1	97.969	97.966	0.003	2.3	1.0209	1.0209	0.0000
91	4	6	2	98.551	98.547	0.004	0.7	1.0164	1.0164	0.0000
92	3	7	2	98.629	98.630	−0.001	1.6	1.0158	1.0158	0.0000
93	6	0	2	99.186	99.185	0.001	1.4	1.0116	1.0116	0.0000
94	1	6	3	99.523	99.522	0.001	1.7	1.0091	1.0091	0.0000

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

DBWS-9807 program (Young *et al*., 2000). In order to obtain the reference intensity ratio (RIR) value, the XRPD data of a mixture of 50 wt.% Al_5NdNi_2 and 50 wt.% NIST SRM 676a alumina was collected (Cline *et al*., 2011).

III. RESULTS AND DISCUSSION

The experimental XRPD pattern of the Al_5NdNi_2 alloy is shown in Figure 1. All diffraction lines in the pattern were indexed successfully with an orthorhombic structure of

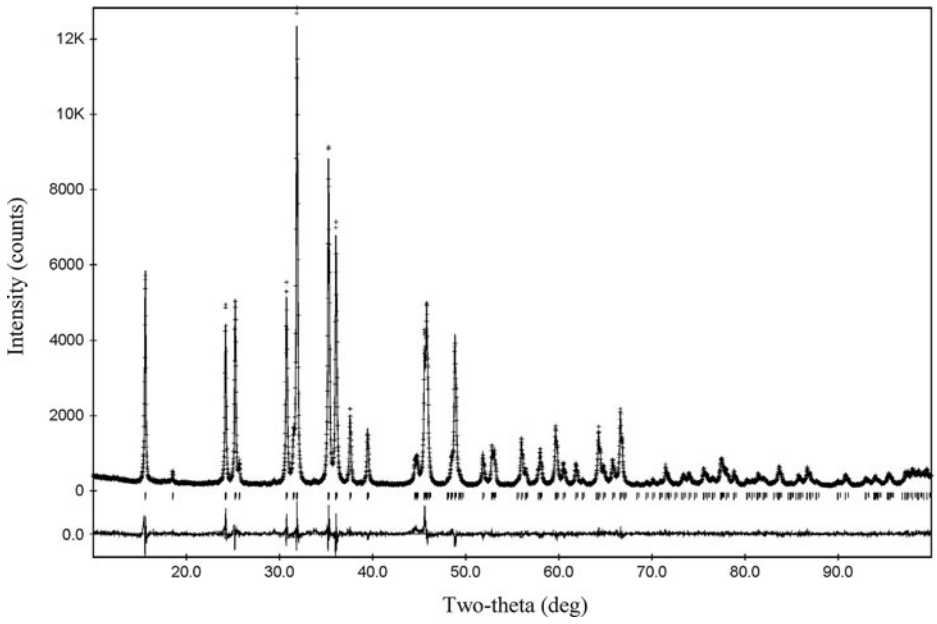

Figure 2. Observed, calculated, and residuals of XRPD pattern for Al_5NdNi_2 with Rietveld refinement.

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TABLE II. Atomic positions and occupancy of Al_5NdNi_2 with Rietveld refinement.

Atom	Position	x	y	z	SOF	B_{eq} (Å^2)
Al	2b	0	0.5	0.5	1	0.25 (8)
Al	8n	0.1876	0.3413	0	1	0.33 (9)
Nd	2a	0	0	0	1	0.33 (4)
Ni	4h	0	0.2603	0.5	1	0.16 (7)

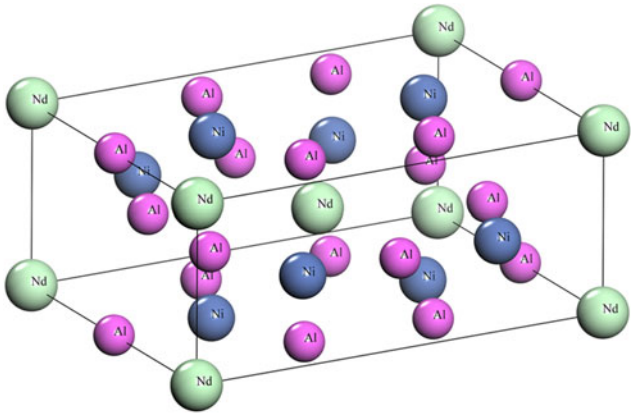

Figure 3. (Color online) Crystal structure of the Al_5NdNi_2.

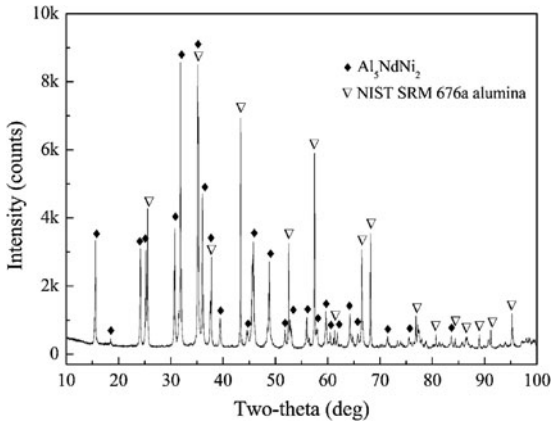

Figure 4. XRPD data of a mixture of Al_5NdNi_2 and corundum in equal proportions by weight.

space group *Immm* (No.71) using Jade 6.5. The lattice parameters were determined to be $a = 7.0493$ (1) Å, $b = 9.5680$ (1) Å, $c = 3.9780$ (1) Å, $V = 268.30$ Å^3, $\rho = 4.91$ g cm^{-3}, and $Z = 2$ by cell refinement from the list of peaks. Internal theta calibration was executed before locating the peaks. The F_{30} (Smith–Snyder figure-of-merit) is 307.6(0.0030, 31) (Smith and Snyder, 1979). It was found that Al_5NdNi_2 and Al_5CeNi_2 share the same structure type as Al_5NdNi_2 by comparing crystal structure information with Al_5CeNi_2 from the report (Isikawa *et al.*, 1994). The calculated and observed values of XRPD data for Al_5NdNi_2 are listed in Table I.

Rietveld refinement of Al_5NdNi_2 was carried out with the DBWS-9807 program. The best results of Rietveld refinement for Al_5NdNi_2 were obtained when the 2b and 8n sites were only occupied by Al atoms, 2a sites were occupied by Nd atoms, and 4h sites were occupied by Ni atoms. The lattice parameters, refined by Rietveld refinement method, were $a = 7.0508(1)$ Å, $b = 9.5690(1)$ Å, $c = 3.9792(1)$ Å, $V = 268.47$ Å^3, $\rho = 4.91$ g cm^{-3}, and $Z = 2$. The R factors were $R_p = 6.02\%$, $R_{wp} = 7.83\%$, $R_{exp} = 4.62\%$, $S = 1.69$, $R_B = 3.51$, and $R_F = 2.50$. The observed, calculated and residuals of XRPD pattern for Al_5NdNi_2 after Rietveld refinement are shown in Figure 2, and Table II shows the atomic sites and occupancy of Al_5NdNi_2 after refinement. The structure diagram of Al_5NdNi_2 is shown in Figure 3.

RIR is the ratio of the intensity of the strongest analyte line to the intensity of the (113) line of corundum when the analyte is mixed 50 : 50 by weight with corundum (Schreiner, 1995). The XRPD data of a mixture of Al_5NdNi_2 and corundum in equal proportions by weight were shown in Figure 4. The corundum (113) line of 2θ 43.34° is the strongest peak for corundum, and the Al_5NdNi_2 (121) line of 2θ 31.87° is the strongest peak for Al_5NdNi_2. The peak height of these two non-overlapped peaks were determined by Jade 6.5, which were used to experimentally measure the RIR, and the RIR value is 1.23.

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SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S088571561800026X

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