



# Sr<sub>4</sub>AlNbO<sub>8</sub>: A new crystal structure type determined from powder X-ray data

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## ABSTRACT

Sr<sub>4</sub>AlNbO<sub>8</sub> was synthesized at 1500 °C in air. The crystal structure was initially determined from powder X-ray diffraction data, and later refined with combined X-ray and neutron diffraction data (*P*2<sub>1</sub>/*c*; *a* = 7.17592(2) Å, *b* = 5.80261(2) Å, *c* = 19.7408(1) Å; β = 97.5470(1)°, *V* = 814.869(3) Å<sup>3</sup>, *Z* = 4, *R*<sub>p</sub>/*R*<sub>wp</sub> = 10.04%/13.18% for X-ray data, 4.40%/5.67% for neutron data, and 7.71%/10.74% in total with χ<sup>2</sup> of 3.76, 23 °C). The crystal structure is a new structure type and may be described as a three-dimensional polyhedral network resulting from the corner-sharing of NbO<sub>6</sub> and Sr1O<sub>6</sub> octahedra and AlO<sub>4</sub> tetrahedra. Also, the other strontium atoms (Sr2, Sr3, and Sr4) occupy the larger cavities surrounded by oxygen atoms to form nine, eight, and 11 coordination, respectively. Considering that Sr, Al, and Nb atoms are crystallographically distinct in terms of interatomic distances and polyhedral coordination, Sr<sub>4</sub>AlNbO<sub>8</sub> can be regarded as a stoichiometric compound.

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## 1. Introduction

Sr<sub>4</sub>AlNbO<sub>8</sub> was first identified in studies on subsolidus phase relations and dielectric properties in the SrO–Al<sub>2</sub>O<sub>3</sub>–Nb<sub>2</sub>O<sub>5</sub> system by J.Y. Chan et al. Monoclinic lattice parameters and space group were determined by powder X-ray and electron diffraction experiments (*P*2<sub>1</sub>/*c*; *a* = 7.1728(2) Å, *b* = 5.8024(2) Å, *c* = 19.733(1) Å; β = 97.332(3)°) [1]. However, the structure was not determined because the single crystal growth was unsuccessful. Due to the lack of the crystal structure, even the composition Sr<sub>4</sub>AlNbO<sub>8</sub> has not been confirmed to date, which would have made a further investigation difficult such as tuning of the compositions for an improved dielectric property.

Structure determination from powder diffraction data has developed rapidly since 1990s [2]. We have established our own structure solving procedure, and in this work, the crystal structure of Sr<sub>4</sub>AlNbO<sub>8</sub> was successfully determined using powder X-ray diffraction data, which was then refined with combined X-ray and neutron diffraction data. The present report describes the previously undetermined crystal structure of Sr<sub>4</sub>AlNbO<sub>8</sub>.

## 2. Experimental

Sr<sub>4</sub>AlNbO<sub>8</sub> was synthesized by a solid-state reaction from a nominal mixture of high-purity SrCO<sub>3</sub> (99.999%, Strem), Al<sub>2</sub>O<sub>3</sub>

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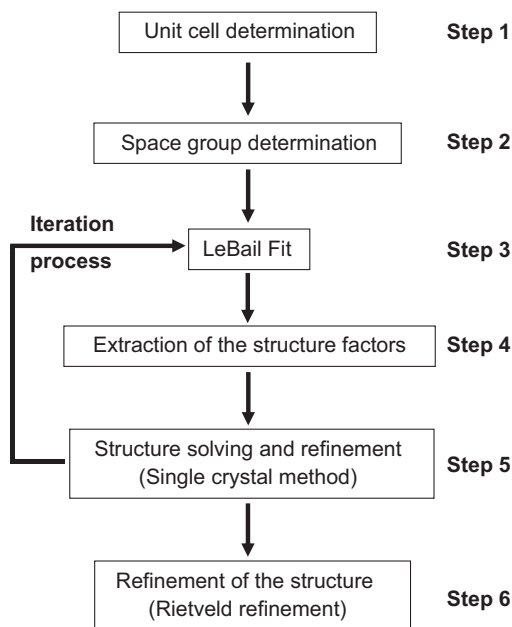
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(99.99%, Aldrich), and Nb<sub>2</sub>O<sub>5</sub> (99.99%, Acros) via ball-milling in ethanol for 24 h. The mixture was dried, pressed into pellets, heated in air at 950 °C for 48 h, and again at 1500 °C for 48 h with intermediate grinding and pressing; the yield was about 93% by weight. The minor impurity phases were Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub> [3], Sr<sub>3</sub>Al<sub>2</sub>O<sub>6</sub> [4], Sr<sub>6–2x</sub>Nb<sub>2+2x</sub>O<sub>11+3x</sub> (*x* ≈ 0.23) [5], and Sr<sub>2</sub>AlNbO<sub>6</sub> [1].

The powder X-ray diffraction (XRD) data were recorded at room temperature using synchrotron radiation (λ = 1.543 Å) from 8C2 powder beam line of the Pohang Light Source (PLS) in Korea with a six multi-detector system over an angular range of 8° ≤ 2θ ≤ 130° with a step of 0.005° and a total measurement time of 12 h. Neutron powder diffraction measurement was performed at room temperature in air using a HANARO HRPD equipment with a 32-He-3 multi-detector system and a Ge (331) monochromator operated by the Korea Atomic Energy Institute, Daejeon, Korea. The data were collected with a wavelength of 1.8371 Å over the 2θ range of 10–150° with a step of 0.05° and a total measurement time of 3 h. The sample amounts used were ~10 g.

The structure determination from the powder XRD data was performed using a combination of the powder profile refinement program GSAS [6] and the single crystal structure refinement program CRYSTALS [7]. For a three-dimensional view of the Fourier density maps, MCE was used [8]. The flow diagram of the procedure is presented in Fig. 1.

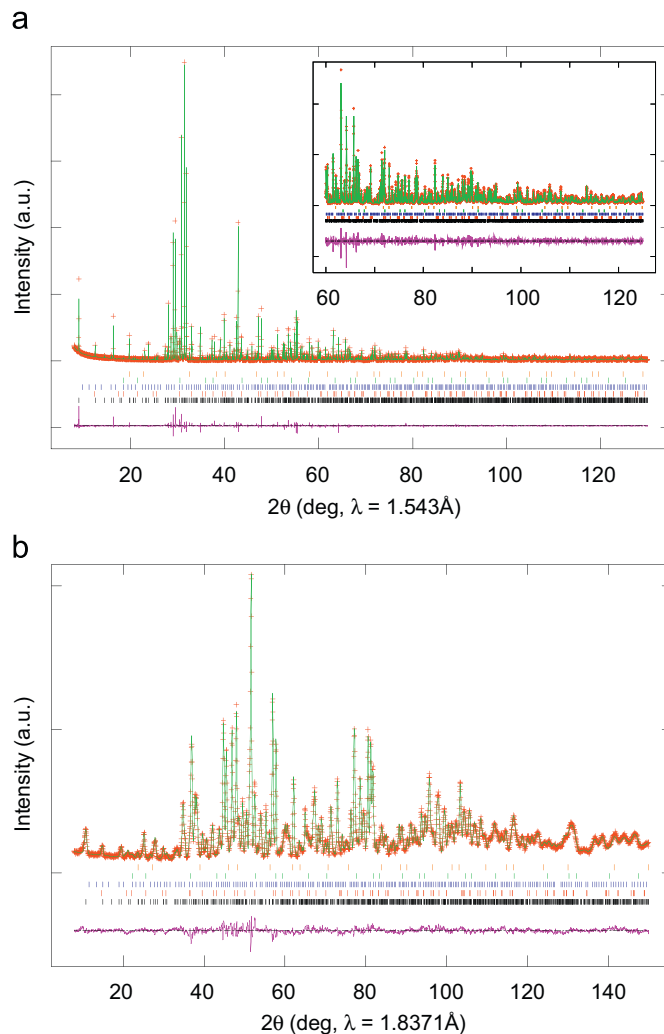
Obtaining the structure began by LeBail fitting of powder X-ray data for the Sr<sub>4</sub>AlNbO<sub>8</sub> phase (step 3 in Fig. 1) with the space group and the initial unit cell parameters known from the previous work [1], while Rietveld fitting was carried out for the



**Fig. 1.** Flow diagram of the procedure for structure determination from powder diffraction data.

impurity phases. The atomic parameters for the impurities were adopted from values found in the literature [1,3–5]. At the beginning, a structural model with only a dummy atom at an arbitrary position in the unit cell was used. At this step, the peak profile coefficients and unit cell parameters were refined. Structure factors were extracted from the powder data (step 4), and used as input data in the next step. The data were treated as if they represented single crystal data. Then, direct methods were used for the initial solution of the structure using SHELXS [9] run in CRYSTALS, which yielded several metal positions. The remainder of the refinement was similar to a routine single crystal method (step 5). However, not all the atoms could be identified at once. The partial model at this stage replaced the initial dummy-atom model, and was used for a LeBail fit in GSAS (step 3 again). Then, improved structure factors were extracted (step 4 again), which were used for the improved data in the next step 3 to step 5 was iterated until a complete and satisfactory structural model was obtained.

Finally, a combined Rietveld refinement of X-ray and neutron diffraction data was employed to complete the structure determination (step 6). Neutron diffraction data would be very helpful, especially when used as a combination with X-ray data, to confirm and refine the structure because neutron scattering length for oxygen atom ( $b_{c,O} = 5.803$ ) is higher than Al ( $b_{c,Al} = 3.449$ ) and even closer to heavier atoms of Sr and Nb ( $b_{c,Sr} = 7.02$ ,  $b_{c,Nb} = 7.054$ ). The refinement parameters were scale factors, background, unit cell parameters, peak profile coefficients, atomic coordinates, and isotropic thermal parameters. For the impurity phases, the atomic parameters were not refined—only the scale factors and peak profile functions were refined. Before the impurity phases were included in the refinement, the residual factors were  $R_p/R_{wp} = 11.95\%/18.09\%$  for X-ray data,  $5.41\%/7.86\%$  for neutron data, and  $9.25\%/14.76\%$  in total with  $\chi^2$  of 7.02. Including all impurities, the final residuals were reduced to  $R_p/R_{wp} = 10.04\%/13.18\%$  for X-ray data,  $4.40\%/5.67\%$  for neutron data, and  $7.71\%/10.74\%$  in total with  $\chi^2$  of 3.76. It should be mentioned that, although the residual values were significantly reduced by taking account of the impurities, they did not have an effect on refinement of the  $Sr_4AlNbO_8$  structure because the main peaks for the impurity phases were relatively weak and did not



**Fig. 2.** Combined (a) X-ray and (b) neutron Rietveld refinement profiles for  $Sr_4AlNbO_8$  including the minor impurity phases, recorded at room temperature. The cross line marks experimental points and the solid lines are the calculated profiles. The lower traces show the difference curves, and the ticks denote expected peak positions for  $Sr_4AlNbO_8$ ,  $Sr_4Nb_2O_9$ ,  $Sr_3Al_2O_6$ ,  $Sr_{6-2x}Nb_{2+2x}O_{11+3x}$  ( $x \approx 0.23$ ) and  $Sr_2AlNbO_6$ , respectively, in order from the bottom. The inset shows the high angle data in detail.

overlap significantly with the  $Sr_4AlNbO_8$  peaks as shown in the Supporting Information. The final profile fits are shown in Fig. 2, and powder refinement results are given in Table 1. The refined atom parameters and isotropic temperature factors are given in Table 2, and the important bond distances and angles are given in Table 3.

### 3. Results and discussion

#### 3.1. Synthesis

$Sr_4AlNbO_8$  was reported unstable above  $1525^\circ\text{C}$  with a solidus temperature of  $\sim 1575^\circ\text{C}$  [1]. In order to obtain a single phase, several different syntheses were attempted by varying the temperatures from  $1200$  to  $1500^\circ\text{C}$ , the duration times from  $10$  to  $72$  h, and Al/Nb ratios from  $0.8$  to  $1.2$ . However, a single phase was not obtained. It seems that the impurity phases are thermodynamically or kinetically stable enough to compete with  $Sr_4AlNbO_8$  under our synthetic condition. The weight percentages of each component were subtly varied depending on the synthetic condition. However, a  $1:1$  Al/Nb ratio resulted in the highest yield

**Table 1**

Crystal data and structure refinement for Sr<sub>4</sub>AlNbO<sub>8</sub> from the combined powder X-ray and neutron diffraction data

Chemical formula	Sr <sub>4</sub> AlNbO <sub>8</sub>
Formula weight	598.36
Crystal system, space group, Z	P2 <sub>1</sub> /c, 4 (no. 14)
Lattice constants (Å, deg), V (Å <sup>3</sup> )	a = 7.17592(2), b = 5.80261(2), c = 19.7408(1), β = 97.5470(1), V = 814.869(3)
d <sub>calc</sub> (g/cm <sup>3</sup> )	4.877
Temperature (K)	296
Number of reflections (X-ray/neutron) <sup>a</sup>	1381/993
R <sub>p</sub> /R <sub>wp</sub> /R <sub>exp</sub> /R <sub>B</sub> (X-ray) (%) <sup>b</sup>	10.04/13.18/6.87/4.73
R <sub>p</sub> /R <sub>wp</sub> /R <sub>exp</sub> /R <sub>B</sub> (neutron) (%) <sup>b</sup>	4.40/5.67/2.81/4.93
R <sub>p</sub> /R <sub>wp</sub> (total) (%) <sup>b</sup>	7.71/10.74
Goodness of fit (total)	2.08
Reduced χ <sup>2</sup>	3.76
Total refined parameters	106

<sup>a</sup> The number of reflections for the minor phases other than Sr<sub>4</sub>AlNbO<sub>8</sub> are not counted.

<sup>b</sup> R<sub>p</sub> = 100∑|I<sub>o</sub> - I<sub>c</sub>|/∑I<sub>o</sub>; R<sub>wp</sub> = 100(∑w|I<sub>o</sub> - I<sub>c</sub>|<sup>2</sup>/∑w|I<sub>o</sub>|<sup>2</sup>)<sup>1/2</sup>; χ<sup>2</sup> = 100∑w|I<sub>o</sub> - I<sub>c</sub>|<sup>2</sup>/(N<sub>obs</sub> - N<sub>var</sub>); R<sub>exp</sub> = R<sub>wp</sub>/|χ|.

**Table 2**

Atomic coordinates and isotropic displacement (Å<sup>2</sup> × 10<sup>2</sup>) for Sr<sub>4</sub>AlNbO<sub>8</sub> at room temperature (SG = P2<sub>1</sub>/c, no. 14, Z = 4, a = 7.17592(2) Å, b = 5.80261(2) Å, c = 19.7408(1) Å, β = 97.5470(1)°)

Atom <sup>a</sup>	x	y	z	U <sub>iso</sub>
Sr1	-0.02650(14)	0.75111(26)	0.09694(5)	1.38(3)
Sr2	0.49650(14)	0.74925(26)	0.13494(5)	1.33(3)
Sr3	0.71113(14)	0.27918(24)	0.04621(5)	1.46(3)
Sr4	0.14160(15)	0.27571(27)	0.21000(6)	1.92(3)
Al	0.2663(4)	0.2126(7)	0.05751(16)	1.41(9)
Nb	0.66179(13)	0.25760(24)	0.21096(5)	1.04(3)
O1	0.5001(8)	0.4931(8)	0.25525(26)	1.4(1)
O2	0.7942(6)	0.5050(7)	0.17660(21)	0.9(1)
O3	0.8233(6)	0.2277(9)	0.29435(20)	2.6(1)
O4	0.7895(6)	0.0312(8)	0.16304(21)	1.4(1)
O5	0.4495(5)	0.2848(8)	0.12551(18)	1.5(1)
O6	0.0620(6)	0.3419(7)	0.07830(22)	2.2(1)
O7	0.3407(5)	0.2989(7)	-0.02140(18)	1.2(1)
O8	0.2460(6)	-0.0853(6)	0.05784(20)	1.2(1)

<sup>a</sup> All atoms are in the (4e) site with a full occupancy.

of the main phase. Although any sample would have resulted in the same crystal structure of Sr<sub>4</sub>AlNbO<sub>8</sub>, the sample used for the data collection was synthesized at 1500 °C for 48 h. The weight percentages of each component in the sample were 92.9% (Sr<sub>4</sub>AlNbO<sub>8</sub>), 4.4% (Sr<sub>4</sub>Nb<sub>2</sub>O<sub>9</sub>), 1.3% (Sr<sub>3</sub>Al<sub>2</sub>O<sub>6</sub>), 0.8% (Sr<sub>6-2x</sub>Nb<sub>2+2x</sub>O<sub>11+3x</sub>, x ≈ 0.23), and 0.6% (Sr<sub>2</sub>AlNbO<sub>6</sub>), respectively, according to the Rietveld refinement using TOPAS software package [10] (Supporting Information), which was performed after the Sr<sub>4</sub>AlNbO<sub>8</sub> structure was completely solved and refined.

There may be a method for synthesizing Sr<sub>4</sub>AlNbO<sub>8</sub> as a single phase, but our main interest was to determine the crystal structure of Sr<sub>4</sub>AlNbO<sub>8</sub> itself, and fortunately, the small amounts of impurity phases did not disturb the structure determination at all.

### 3.2. Crystal structure

Sr<sub>4</sub>AlNbO<sub>8</sub> crystallizes in a new structure type with four formula units in the space group P2<sub>1</sub>/c of the monoclinic system. Since there are no metal–metal or O–O bonds, and the compound was prepared in air, the formal oxidation states of Sr, Al, Nb, and O

**Table 3**

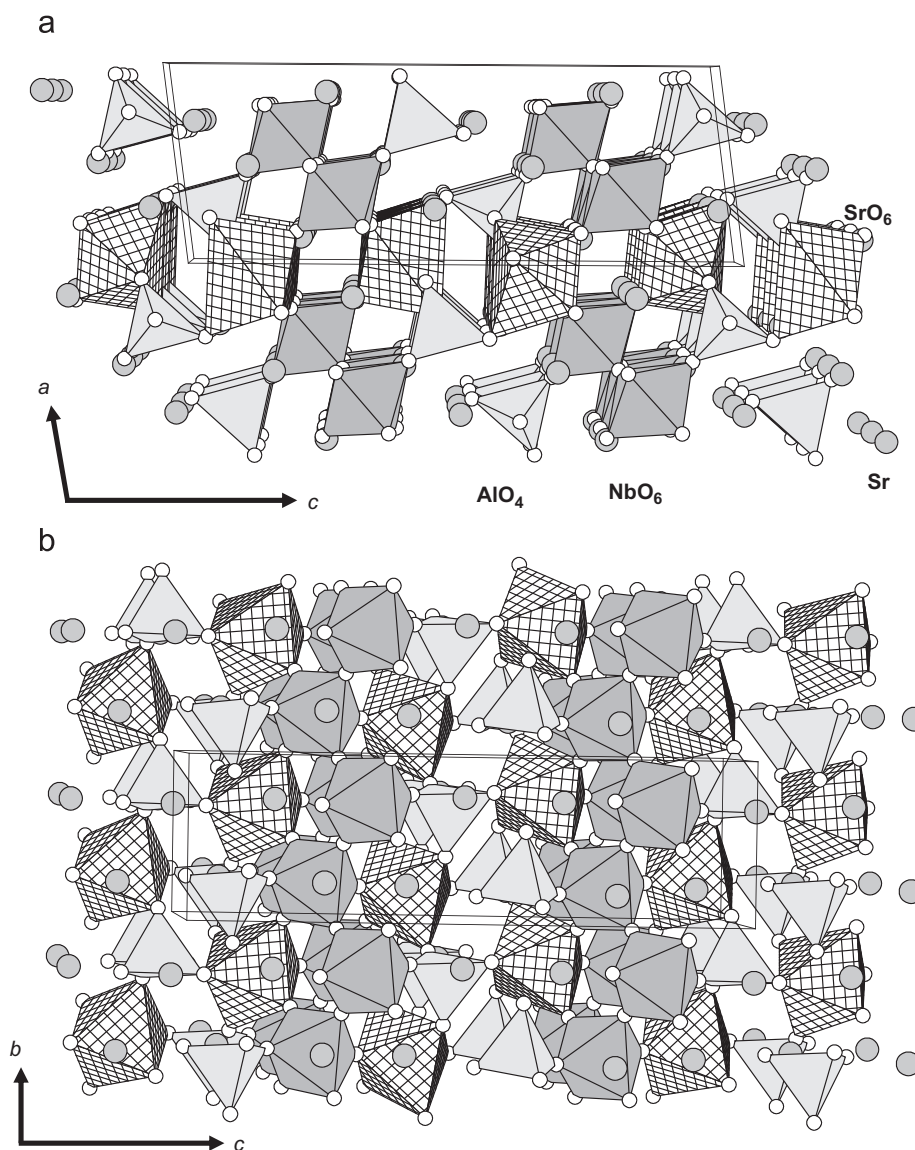
Selected interatomic distances (Å) and angles (deg) in Sr<sub>4</sub>AlNbO<sub>8</sub> at room temperature

Sr1–O2	2.588(4)	Nb–O1	2.060(5)	Al–O5	1.801(5)
Sr1–O3	2.435(4)	Nb–O1	2.087(5)	Al–O6	1.743(5)
Sr1–O4	2.557(4)	Nb–O2	1.895(4)	Al–O7	1.783(5)
Sr1–O6	2.498(4)	Nb–O3	1.892(4)	Al–O8	1.735(5)
Sr1–O7	2.551(4)	Nb–O4	1.920(4)		
Sr1–O8	2.391(4)	Nb–O5	2.125(4)	Sr4–O1	2.899(6)
				Sr4–O1	3.051(5)
Sr2–O1	2.799(5)	Sr3–O2	2.880(4)	Sr4–O2	2.826(4)
Sr2–O1	2.586(5)	Sr3–O4	2.714(4)	Sr4–O2	2.724(4)
Sr2–O2	2.605(4)	Sr3–O5	2.598(4)	Sr4–O3	2.638(6)
Sr2–O3	2.841(4)	Sr3–O6	2.540(4)	Sr4–O3	3.192(6)
Sr2–O4	2.664(4)	Sr3–O7	2.816(4)	Sr4–O3	2.638(6)
Sr2–O5	2.719(5)	Sr3–O7	3.403(4)	Sr4–O4	2.940(4)
Sr2–O5	3.129(5)	Sr3–O7	2.515(4)	Sr4–O4	2.898(4)
Sr2–O7	2.675(4)	Sr3–O8	2.397(4)	Sr4–O5	2.938(4)
Sr2–O8	2.398(4)			Sr4–O6	2.616(4)
O5–Al–O6	106.2(3)	O1–Nb–O1	89.1(1)	O2–Sr1–O7	79.67(13)
O5–Al–O7	108.2(2)	O1–Nb–O2	89.2(2)	O3–Sr1–O4	82.84(15)
O5–Al–O8	106.4(3)	O1–Nb–O3	90.4(2)	O3–Sr1–O8	83.46(13)
O6–Al–O7	116.3(3)	O1–Nb–O5	86.4(2)	O3–Sr1–O6	86.70(16)
O6–Al–O8	110.8(3)	O2–Nb–O5	90.0(2)	O4–Sr1–O7	83.95(13)
O7–Al–O8	108.6(3)	O4–Nb–O5	89.9(2)	O6–Sr1–O7	91.60(13)
		O1–Nb–O2	176.2(2)	O6–Sr1–O8	95.47(15)
O1–Nb–O5	84.4(2)	O1–Nb–O4	174.0(2)	O4–Sr1–O8	114.30(15)
O1–Nb–O3	87.6(2)	O3–Nb–O5	172.1(2)	O7–Sr1–O8	123.63(13)
O1–Nb–O4	88.6(2)			O2–Sr1–O8	155.24(14)
O2–Nb–O4	92.7(2)	O2–Sr1–O3	73.85(14)	O3–Sr1–O7	152.86(13)
O3–Nb–O4	95.1(2)	O2–Sr1–O4	73.11(13)	O4–Sr1–O6	146.89(15)
O2–Nb–O3	95.9(2)	O2–Sr1–O6	73.81(14)		

may be assigned as +2, +3, +5, and –2, respectively. The crystal structure is illustrated in Fig. 3, where two different views are presented for better visualization.

The structure may be described as a three-dimensional polyhedral network resulting from the corner-sharing of NbO<sub>6</sub> and Sr1O<sub>6</sub> octahedra and AlO<sub>4</sub> tetrahedra; the other strontium atoms (Sr2, Sr3, and Sr4) occupy the larger cavities surrounded by oxygen atoms. AlO<sub>4</sub> forms a relatively regular tetrahedron: the average distance of d(Al–O) and angle of (O–Al–O) are 1.766(0.034) Å and 109.4(3.8)°, respectively, where the numbers in parentheses refer to the estimated standard deviations (esd). NbO<sub>6</sub> also forms a relatively regular octahedron: the average values of d(Nb–O) and (O<sub>a</sub>–Nb–O<sub>b</sub>) are 2.00(0.11) Å and 89.9(3.3)°, respectively, where O<sub>a</sub> and O<sub>b</sub> are neighboring oxygen atoms of the octahedron. The average values of d(Sr1–O) and (O<sub>a</sub>–Sr1–O<sub>b</sub>) for Sr1O<sub>6</sub> are 2.503(0.077) Å and 89(16)°, respectively, in which the high esd value of the angles indicated the very distorted octahedron. Sr2, Sr3, and Sr4 have a nine, eight, or eleven-coordination with oxygen atoms, respectively, to form irregular polyhedra. It should be noted that the charge neutrality condition is satisfied with the stoichiometric composition of Sr<sub>4</sub>AlNbO<sub>8</sub>; also, the Al, Nb, and Sr atoms in this structure are crystallographically distinct in terms of interatomic distances and coordination polyhedra. Thus, it is concluded that Sr<sub>4</sub>AlNbO<sub>8</sub> can be regarded as a stoichiometric compound, even though the possibility of cation mixing and/or defects in small amounts cannot be excluded.

The AlO<sub>4</sub> tetrahedron is engaged in corner-sharing with one NbO<sub>6</sub> and three Sr1O<sub>6</sub> octahedra. The NbO<sub>6</sub> octahedron is corner-sharing with one AlO<sub>4</sub> tetrahedron, two NbO<sub>6</sub> octahedra, and three Sr1O<sub>6</sub> octahedra, while the Sr1O<sub>6</sub> octahedron is engaged in corner-sharing with three AlO<sub>4</sub> tetrahedra and three NbO<sub>6</sub> octahedra. It is also noted that the NbO<sub>6</sub> octahedra are connected to one another such that they form zigzag one-dimensional arrangements along the b-axis, as shown in Fig. 3.



**Fig. 3.** (a) (010) view, and (b) (100) view of crystal structure of  $\text{Sr}_4\text{AlNbO}_8$ .  $\text{NbO}_6$ ,  $\text{AlO}_4$  and  $\text{SrO}_6$  are shown as dark gray octahedra, gray tetrahedra and hatched octahedra, respectively.  $\text{Sr}_2$ ,  $\text{Sr}_3$  and  $\text{Sr}_4$  are shown as gray spheres. The unit cell is outlined.

Selected metrical data for the  $\text{Sr}_4\text{AlNbO}_8$  structure are listed in Table 3. The Al–O distances of 1.735(5)–1.801(5) Å are comparable to those of 1.743–1.790 Å found in the Al–O tetrahedron of  $\text{Sr}_3\text{Al}_2\text{O}_6$  [4]. The Nb–O distances of 1.892(4)–2.125(4) Å are in the range 1.810–2.282 Å in Nb–O octahedra of  $\text{Sr}_3\text{Nb}_5\text{O}_{17}$  [11]. Sr–O distances of 2.391(4)–2.588(4) Å in the  $\text{SrO}_6$  octahedron are also comparable to those of 2.455–2.502 Å in the Sr–O octahedra of  $\text{Sr}_3\text{Al}_2\text{O}_6$  [4].

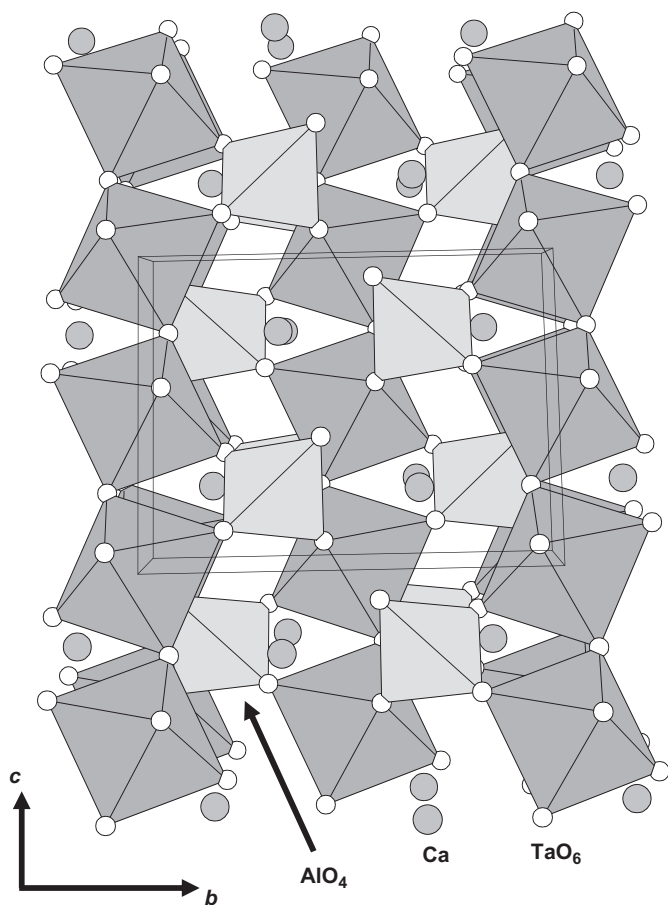
The bond valence sums [12] calculated using the software Valence [13] for the  $\text{Sr}_4\text{AlNbO}_8$  crystal structure are 2.16 (for  $\text{Sr}_1$ ), 2.03 ( $\text{Sr}_2$ ), 1.92 ( $\text{Sr}_3$ ), 1.53 ( $\text{Sr}_4$ ), 2.91 (Al), 4.99 (Nb), 1.95 (O1), 2.08 (O2), 2.02 (O3), 1.95 (O4), 2.03 (O5), 1.71 (O6), 1.75 (O7), and 2.20 (O8) v.u., respectively. These values generally match the expected charges of the ions reasonably. The lower valence sum of 1.53 for  $\text{Sr}_4$  indicates that the  $\text{Sr}_4$  atom has a longer average Sr–O distance than expected, and occupies a relatively larger cavity than the other strontium atoms.

The crystal structure of  $\text{Sr}_4\text{AlNbO}_8$  is a new type in terms of atomic ratios of (1:2:3:8) for (tetrahedral:octahedral:higher-coordinated metal:oxygen) and its unique polyhedral network, which was not found in existing compounds, to our knowledge.

Even though the unit cell parameters of  $\text{Sr}_4\text{AlNbO}_8$  could be related to the unit cell parameter for an ideal perovskite  $a_c$  ( $\sim 4$  Å):  $a = \sqrt{3}a_c$ ,  $b = \sqrt{2}a_c$ ,  $c = 2\sqrt{6}a_c$ , and  $\beta \approx 97^\circ$  according to Chan et al. [1], it was also unsuccessful to find any noticeable relationship between the two structures. However, it is worthwhile to note the structure of  $\text{CaAlTaO}_5$  ( $C2/c$ ,  $a = 6.6760(3)$ ,  $b = 8.9546(3)$ ,  $c = 7.3494(3)$  Å,  $\beta = 114.098(3)^\circ$ ) [14] as shown in Fig. 4, in which the structure consists of corner-sharing of  $\text{TaO}_6$  octahedra and  $\text{AlO}_4$  tetrahedra, and Ca atoms in seven-coordinated cavities surrounded by oxygen atoms. It has a polyhedral network similar to  $\text{Sr}_4\text{AlNbO}_8$  in the sense that it also consists of zigzag chains of corner-sharing  $\text{TaO}_6$  octahedra parallel to  $c$ -axis. The obvious difference would be that the  $\text{TaO}_6$  zigzag chains in  $\text{CaAlTaO}_5$  are linked by  $\text{AlO}_4$  tetrahedra only, but the  $\text{NbO}_6$  zigzag chains in  $\text{Sr}_4\text{AlNbO}_8$  are linked by a combination of  $\text{AlO}_4$  tetrahedra and  $\text{SrO}_6$  octahedra.

#### 4. Conclusions

$\text{Sr}_4\text{AlNbO}_8$  was synthesized, and the previously undetermined crystal structure was solved and refined with powder X-ray and



**Fig. 4.** (100) view of crystal structure of  $\text{CaAlTaO}_5$ .  $\text{TaO}_6$  and  $\text{AlO}_4$  are shown as dark gray octahedra and gray tetrahedra, respectively. Ca atoms are shown as gray spheres. The unit cell is outlined.

neutron data. It adopts a new structure type, consisting of corner-sharing of  $\text{NbO}_6$  and  $\text{SrIO}_6$  octahedra,  $\text{AlO}_4$  tetrahedra and higher-coordinated Sr atoms, to form a three-dimensional polyhedral network. This work would provide an important basis for understanding the properties of the material as well as promoting a further investigation such as substitution and/or tuning of the compositions for an improved property.

### Supporting information

X-ray Rietveld refinement profiles for  $\text{Sr}_4\text{AlNbO}_8$ , using the TOPAS software package, where the amounts of the minor

impurity phases are graphically shown. Further details of the crystal structure investigations can be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany (fax: +49 7247 808 666; e-mail: [crysdta@fiz-karlsruhe.de](mailto:crysdta@fiz-karlsruhe.de)) on quoting the depository number CSD 419363 for  $\text{Sr}_4\text{AlNbO}_8$ .

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### Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.jssc.2008.07.009](https://doi.org/10.1016/j.jssc.2008.07.009).

### References

- [1] J.Y. Chan, I. Levin, T.A. Vanderah, R.G. Geyer, R.S. Roth, *Int. J. Inorg. Mater.* 2 (2000) 107–114.
- [2] W.I.F. David, K. Shankland, L.B. McCusker, C. Baerlocher, *Structure Determination from Powder Diffraction Data*, Oxford University Press Inc., New York, 2002.
- [3] M. Weiden, A. Grauel, J. Norwig, S. Horn, F. Steglich, *J. Alloys Compd.* 218 (1995) 13–16  
ICSD (Inorganic Crystal Structure Database) #79217, ver. 2005-1, Karlsruhe, Germany.
- [4] B.C. Chakoumakos, G.A. Lager, J.A. Fernandez-Baca, *Acta Crystallogr. C* 48 (1992) 414–419  
ICSD (Inorganic Crystal Structure Database) #71860, v. 2005-1, Karlsruhe, Germany.
- [5] M.-R. Li, S.-T. Hong, *Chem. Mater.* 20 (2008) 2736–2741.
- [6] A.C. Larson, R.B. Von Dreele, *General Structure Analysis System (GSAS)*, Los Alamos National Laboratory Report LAUR 86-748, 2000.
- [7] P.W. Betteridge, J.R. Carruthers, R.I. Cooper, C.K. Prout, D.J. Watkin, *J. Appl. Crystallogr.* 36 (2003) 1487.
- [8] M. Husak, B. Kratochvil, *J. Appl. Crystallogr.* 36 (2003) 1104.
- [9] G.M. Sheldrick, *SHELXS-86*, Universität Göttingen, Germany, 1986.
- [10] R.W. Cheary, A. Coelho, *J. Appl. Crystallogr.* 25 (1992) 109–121  
Bruker AXS, TOPAS 3, Karlsruhe, Germany, 2000.
- [11] H.W. Schmalle, T. Williams, A. Reller, *Acta Crystallogr. C* 51 (1995) 1243–1246  
ICSD (Inorganic Crystal Structure Database) #79699, v. 2005-1, Karlsruhe, Germany.
- [12] I.D. Brown, D. Altermatt, *Acta Crystallogr. B* 41 (1985) 244–247.
- [13] N.E. Brese, M. O’Keeffe, *Acta Crystallogr. B* 47 (1991) 192–197.
- [14] M. Sales, G. Eguia, P. Quintana, L.M. Torres-Martinez, A.R. West, *J. Solid State Chem.* 143 (1999) 62–68.