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The Na_2O–SrO–B_2O_3 diagram in the B-rich part and the crystal structure of $NaSrB_5O_9$

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Abstract

The subsolidus phase relations in the B-rich part of the ternary system, Na_2O–SrO–B_2O_3, are investigated by the powder X-ray diffraction method. Four ternary compounds: $NaSrBO_3$, $NaSr_4B_3O_9$, $Na_3SrB_5O_{10}$ and $NaSrB_5O_9$ were found in it, the two lasts are new. $NaSrB_5O_9$ crystallizes in the monoclinic space group $P2_1/c$, with the lattice parameters $a = 6.4963(1)$ Å, $b = 13.9703(2)$ Å, $c = 8.0515(1)$ Å, $\beta = 106.900(1)°$. $Na_3SrB_5O_9$ is also monoclinic, space group $C2$, $a = 7.290(1)$ Å, $b = 13.442(2)$ Å, $c = 9.792(1)$ Å, $\beta = 109.60(1)$. $NaSrB_5O_9$ is isostructural with another pentaborate $NaCaB_5O_9$, and its structure was refined by Rietveld method based on the structural model of $NaCaB_5O_9$. The fundamental building units are $[B_5O_9]^{3-}$ anionic groups, forming complex thick anionic sheets, extending parallel to the ac plane. The Na and Sr atoms are all eight-coordinated with O atoms, forming trigonal dodecahedra. The $[NaO_8]$ polyhedra are distributed between the B–O sheets, while the $[SrO_8]$ polyhedra located in the sheets and connect with each other by edges to form infinite chains along the c-axis.
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Keywords: Diagram; Borates; X-ray diffraction

1. Introduction

Inorganic borates have long been a focus of research for their variety of structure type, transparency to a wide range of wavelengths, high laser damage tolerance, and high optical quality. Studies of alkali-metal and alkaline-earth-metal borates have produced a large family of compounds with outstanding physical properties [1,2], such as β-BaB_2O_4 [3], LiB_3O_5 [4], $Sr_2Be_2B_2O_7$[5] and $K_2Al_2B_2O_7$ [6]. Recently, photoluminescence is also found in many rare-earth ions doped alkaline-earth-metal borates [7–10]. Some have been used as useful phosphors, such as UV-emitting Eu^{2+}: SrB_4O_7 in lamps for medical applications and skin tanning [7]. Because of the similar radii and same valence with Sr^{2+} and Ba^{2+}, Eu^{2+} is easy to replace some sites of Sr and Ba atoms in crystal cell, and then photoluminescence can be found in the doped compounds. These

properties depend on the crystal structures of these borates with a variety of [BO] atomic groups. The various structures and properties inspirit us to explore more borates in the M_2O–$M'O$–B_2O_3 systems (M is alkali metal, and M' is alkaline-earth metal) to search for new functional materials. According to the previous research work [11–16], more ternary compounds exist in the B-rich part. Then the subsolidus phase relations in the B-rich part of the Na_2O–SrO–B_2O_3 system were investigated. Four new ternary compounds, $NaSrBO_3$, $NaSr_4(BO_3)_3$, $NaSrB_5O_9$, and $Na_3SrB_5O_{10}$ were synthesized successfully, and the powder XRD patterns were submitted for publication in the Powder Diffraction File (International Center for Diffraction Data) in 2004. The structures of $NaSrBO_3$ and $NaSr_4(BO_3)_3$ were determined from powder diffraction [11,12]. $Na_3SrB_5O_{10}$ has the same formula-type $A_3BC_5X_{10}$ with $Na_3CaB_5O_{10}$ [13], but crystallizes in a different space group. The structure of $NaSrB_5O_9$ was refined by Rietveld method based on the structural model of $NaCaB_5O_9$ [13], which is discussed in the following text. A comparison of

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the known ternary compounds in the B-rich part of the systems $Na_2O–SrO–B_2O_3$ and $Na_2O–CaO–B_2O_3$ is also presented here.

2. Experimental

2.1. Solid-state syntheses

Polycrystalline samples were prepared by sintering at high temperature through solid-state reactions. The mixtures of analycial-purity Na_2CO_3, $SrCO_3$, and H_3BO_3 were heated at 600 °C to decompose the carbonate and eliminate the water, and then elevated to sintering temperature between 700 and 850 °C for 72 h, depending on their compositions. In between sintering steps, the sample was cooled and then ground. Equilibrium was considered to have been reached when the X-ray pattern of a specimen showed no change upon successive heat treatments. The diffraction data were collected on an X-ray Rigaku diffractometer D/Max-2500 with CuKα radiation (40 kV, 140 mA) and an X-ray MAC Science diffractometer M21X with CuKα radiation (60 kV, 500 mA) at room temperature. Twenty-six samples with different compositions were prepared (Table 1) and the phase identification in the B-rich part of the diagram is shown in Fig. 1.
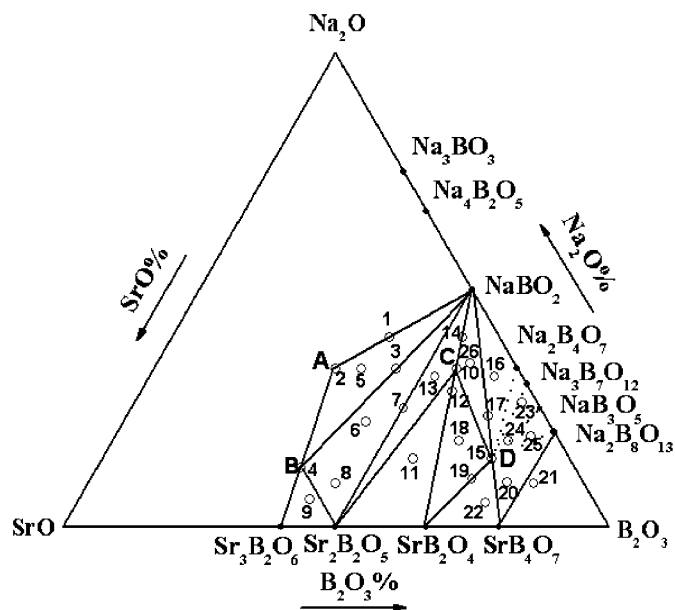
Table 1
List of phase identifications for various specimens in the B-rich part of $Na_2O–SrO–B_2O_3$[a] system

No.	$NaO_{0.5}$ (at%)	SrO (at%)	$BO_{1.5}$ (at%)	Phase identification
1	2	1	2	A + NaBO$_2$
2	1	1	1	A
3	3	2	4	B + NaBO$_2$
4	1	4	3	B
5	3.5	3	4	A + B + NaBO$_2$
6	2	3	4	B + NaBO$_2$ + Sr$_2$B$_2$O$_5$
7	2	2	4	NaBO$_2$ + Sr$_2$B$_2$O$_5$
8	1	5	5	B + NaBO$_2$ + Sr$_2$B$_2$O$_5$
9	0.6	4.5	5.5	B + Sr$_3$B$_2$O$_6$ + Sr$_2$B$_2$O$_5$
10	3	1	5	C
11	1	2	4	C + Sr$_2$B$_2$O$_5$ + SrB$_2$O$_4$
12	2	1	4	C + SrB$_2$O$_4$
13	2	1	3.3	C + NaBO$_2$ + Sr$_2$B$_2$O$_5$
14	6	1	8	C + NaBO$_2$
15	1	1	5	D
16	5	0.8	10	D + NaBO$_2$ + glass phase
17	6	2.6	17	D + NaBO$_2$
18	2	2	7	C + D + SrB$_2$O$_4$
19	1	2	7	D + SrB$_2$O$_4$
20	1.2	1.8	10	D + SrB$_4$O$_7$ + glass phase
21	1	1	9	SrB$_4$O$_7$ + glass phase
22	1	4	15	D + SrB$_2$O$_4$ + SrB$_4$O$_7$
23	6.6	0.7	18	D + glass phase
24	2	1	8	D + glass phase
25	2.5	0.6	10	D + glass phase
26	3	0.7	5	C + D + NaBO$_2$

B_2O_3 cannot be detected in X-ray diffraction because of their weak diffraction peaks.

[a]A: NaSrBO$_3$, B: NaSr$_4$(BO$_3$)$_3$, C: Na$_3$SrB$_5$O$_{10}$ and D: NaSrB$_5$O$_9$.

Fig. 1. Subsolidus phase relations in the B-rich part of the system $Na_2O–SrO–B_2O_3$.

2.2. Rietveld refinement

The data for $NaSrB_5O_9$ used for Rietveld refinement were collected over a 2θ range of 10–120° in the step scan mode with a step size of 0.02° and a measurement time of 1 s per step at room temperature. Additional technical details are given in Table 2. The diffraction pattern ($2\theta \leqslant 60°$) of the compound was indexed using DICVOL91 [17] by successive dichotomy method with Si as the internal standard. This gave out a monoclinic unit cell with $a = 6.4956(2)$ Å, $b = 13.9705(5)$ Å, $c = 8.0427(2)$ Å, $\beta = 106.907(2)°$. Systematic absence of $h0l$ with $l = 2n+1$, $0k0$ with $k = 2n+1$, and $00l$ with $l = 2n+1$ suggests that the possible space group is $P2_1/c$. According to the space group and the lattice parameters, it was found to be isostructural with $NaCaB_5O_9$. Then the structure was refined by the Rietveld method [18,19] with the Fullprof program [20] on the basis of the structural model of $NaCaB_5O_9$. The Na and Ca are mixed occupied in $NaCaB_5O_9$ because of the similar ionic radii of Na and Ca, which is similar to the situation in $NaCaBO_3$ [21]. However, the mixed occupancy had not been found in $NaSrB_5O_9$ after refined the occupancy of Na and Sr atoms. The position which Ca occupied 83% was taken as the position of Sr, and the other one which Na occupied 83% was considered to be the position of Na. In the final cycle of refinement a total of 83 parameters were refined (64 structural parameters and 19 profile parameters, including 5 background parameters and 5 peak shape parameters. The pseudo-Voigt function was used as peak shape function) and the finally agreement factors converged to $R_B = 5.19\%$, $R_p = 7.74\%$, $R_{wp} = 10.6\%$, and $R_{exp} = 3.11\%$. Lattice parameters were refined to be $a = 6.4963(1)$ Å,

Table 2
Crystallographic data, experimental details of X-ray powder diffraction, and Rietveld refinement data for $NaSrB_5O_9$

Chemical formula	$NaSrB_5O_9$
Formula weight	308.66
Crystal system	Monoclinic
Space group	$P2_1/c$ (no.14)
a (Å)	6.4963(1)
b (Å)	13.9703(2)
c (Å)	8.0515(1)
β (deg)	106.900(1)
Volume (Å^3)	699.16(2)
Z	4
d_c (g cm^{-3})	2.932
Diffractometer	MXP21VAHF/M21X, MAC Science
Radiation type	Cu$K\alpha$
Wavelength (Å)	1.5418
Profile range, 2θ (deg)	10–120
Step size, 2θ (deg)	0.02
Number of observation (N)	5500
Number of contributing reflections	2171(Kα1 + Kα2)
Number of structure parameters (P_1)	64
Number of profile parameters (P_2)	19
R_{Bragg} (%)	5.19
R_p (%)	7.74
R_{wp} (%)	10.6
R_{exp} (%)	3.11

Note: $R_p = \Sigma|y_{io}-y_{ic}|/\Sigma|y_{io}|$, $R_{wp} = [\Sigma w_i(y_{io}-y_{ic})^2/\Sigma w_i y_{io}^2]^{1/2}$, $R_{exp} = [(N-P_1-P_2)/\Sigma w_i y_{io}^2]^{1/2}$, $S = \Sigma[w_i(y_{io}-y_{ic})^2/(N-P_1-P_2)]^{1/2}$.
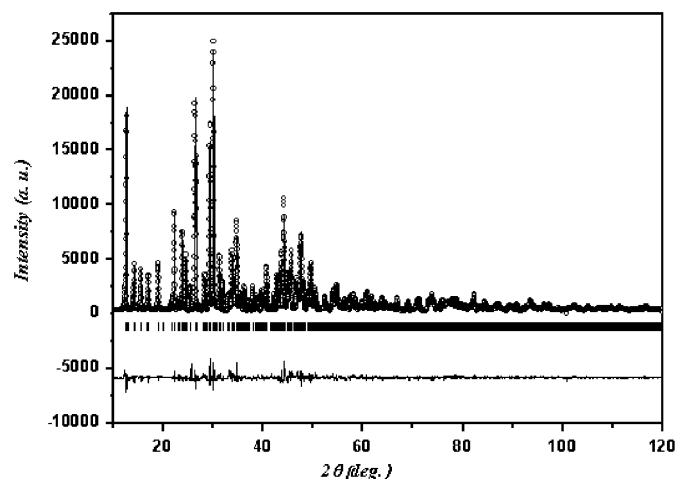

Fig. 2. Final Rietveld refinement plot of $NaSrB_5O_9$. Small circles (○) correspond to experimental values, and the continuous lines are the calculated pattern; vertical bars (|) indicate the positions of Bragg peaks. The bottom trace depicts the difference between the experimental and the calculated intensity values.

$b = 13.9703(2)$ Å, $c = 8.0515(1)$ Å, $\beta = 106.900(1)°$. The final refinement pattern is given in Fig. 2. The crystallographic data, fractional atomic coordinates and equivalent isotropic displacement parameters are reported in Tables 2 and 3; significant bond lengths and angles are listed in Table 4.

Table 3
Positional and atomic displacement parameters for $NaSrB_5O_9$

Atom	Site	x	y	z	U_{eq} (Å^2)
Sr	4e	0.1002(1)	0.2293(1)	0.5615(1)	0.0085(1)
Na	4e	0.4617(5)	0.5829(2)	0.6502(4)	0.0091(4)
O(1)	4e	0.8300(4)	0.6457(2)	0.6437(3)	0.0016(5)
O(2)	4e	0.1326(4)	0.5387(2)	0.7126(3)	0.0010(5)
O(3)	4e	0.4370(3)	0.4431(1)	0.8056(2)	0.0027(5)
O(4)	4e	0.0833(3)	0.3780(1)	0.7867(2)	0.0038(5)
O(5)	4e	0.7692(4)	0.4797(2)	0.6506(3)	0.0059(5)
O(6)	4e	0.7830(4)	0.3773(1)	0.8937(3)	0.0060(5)
O(7)	4e	0.7435(4)	0.3077(2)	0.5991(3)	0.0019(5)
O(8)	4e	0.5052(4)	0.1660(2)	0.5332(4)	0.0124(7)
O(9)	4e	0.4876(2)	0.2725(1)	0.7459(2)	0.0058(5)
B(1)	4e	0.9209(9)	0.5477(4)	0.6748(7)	0.0055(9)
B(2)	4e	0.2287(6)	0.4538(2)	0.7562(5)	0.0069(9)
B(3)	4e	0.8387(9)	0.3920(4)	0.7242(7)	0.0056(9)
B(4)	4e	0.5857(9)	0.2397(4)	0.6240(7)	0.020(1)
B(5)	4e	0.5624(7)	0.1385(3)	0.3720(5)	0.008(1)

3. Results and discussion

3.1. Subsolidus phase relations in the B-rich part

In the binary system $Na_2O-B_2O_3$, there are seven compounds with three of them polymorphic: $Na_2B_4O_7$ (hexamorphic) [22–24], NaB_3O_5 (dimorphic) [25,26] and $Na_2B_8O_{13}$ (dimorphic) [27–29]. Most of them were synthesized by fusing borax and boric acid, then only a glass phase can be obtained in our experimental conditions. It leads to some uncertain three phase sections which are indicated by dashed lines in Fig. 1. $Na_3B_7O_{12}$ is synthesized in a nitrogen atmosphere [30], which cannot be obtained in our experimental condition. Na_3BO_3 [31] and $Na_4B_2O_5$ [32] are not in the B-rich part, which are not studied in our experiment. So only $NaBO_2$ (ICDD-PDF 321046) can be confirmed existence. In the binary system $SrO-B_2O_3$, four compounds, with one of them dimorphic: SrB_2O_4 [33,34] are evidenced. The α-SrB_2O_4 [34] is a high pressure phase, which cannot be obtained under the present experimental conditions. So only SrB_2O_4 [33], $Sr_3B_2O_6$ [35], $Sr_2B_2O_5$ [36], and SrB_4O_7 [37] have been confirmed existence. In the binary system Na_2O-SrO, there is no compound reported up to now, and is not studied in this work. According to the results of the X-ray diffraction analysis, the subsolidus phase relations of the B-rich part in $Na_2O-SrO-B_2O_3$ system are shown in Fig. 1. There are 14 three-phase regions in this part under the present experimental conditions. No solid solution regions are found in all binary and ternary compounds, two-phase regions are joint-lines of the two compounds.

3.2. Description of the crystal structure of $NaSrB_5O_9$

Monoclinic $NaSrB_5O_9$ crystallizes in the space group $P2_1/c$. As illustrated in Fig. 3, the fundamental building unit of $NaSrB_5O_9$ is $[B_5O_9]^{3-}$ anionic group, which is composed of three $[BO_3]$ triangles (Δ) (give for clarity on

Table 4
Selected interatomic distances (Å) and angles (deg)

Sr–O1[i]	2.565(2)	Na–O1	2.563(4)
Sr–O1[ii]	2.534(2)	Na–O2	2.414(4)
Sr–O4	2.779(2)	Na–O3	2.350(4)
Sr–O4[iii]	2.649(2)	Na–O5	2.463(4)
Sr–O6[iii]	2.583(3)	Na–O5[ii]	2.601(4)
Sr–O7	2.658(3)	Na–O7[ii]	2.566(4)
Sr–O8	2.847(3)	Na–O8[i]	2.752(4)
Sr–O9	2.594(2)	Na–O9[i]	2.768(3)
B1–O1	1.483(6)	O1–B1–O2	117.52(4)
B1–O2	1.325(7)	O1–B1–O5	112.92(4)
B1–O5	1.342(6)	O2–B1–O5	129.46(5)
B2–O2	1.339(4)	O2–B2–O3	122.99(3)
B2–O3	1.303(4)	O2–B2–O4	113.87(3)
B2–O4	1.486(4)	O3–B2–O4	121.44(3)
B3–O4	1.533(6)	O4–B3–O5	115.70(4)
B3–O5	1.377(6)	O4–B3–O6	100.51(4)
B3–O6	1.523(6)	O4–B3–O7	107.08(4)
B3–O7	1.557(6)	O5–B3–O6	111.82(4)
		O5–B3–O7	112.04(4)
		O6–B3–O7	108.93(4)
B4–O7	1.454(6)	O7–B4–O8	129.23(5)
B4–O8	1.283(6)	O7–B4–O9	111.59(4)
B4–O9	1.393(6)	O8–B4–O9	118.10(4)
B5–O3[iii]	1.413(4)	O3[iii]–B5–O6[iii]	111.96(3)
B5–O6[iii]	1.410(5)	O3[iii]–B5–O8	105.84(3)
B5–O8	1.500(5)	O3[iii]–B5–O9[iii]	110.76(3)
B5–O9[iii]	1.589(4)	O6[iii]–B5–O8	115.58(4)
		O6[iii]–B5–O9[iii]	108.20(3)
		O8–B5–O9[iii]	104.22(3)

Note: symmetry codes: (i) $-x$, $y+1/2$, $-z+1/2$; (ii) $-x$, $-y$, $-z$; (iii) x, $-y+1/2$, $z+1/2$.

Fig. 4. Projection of the structure of $NaSrB_5O_9$ along [100].
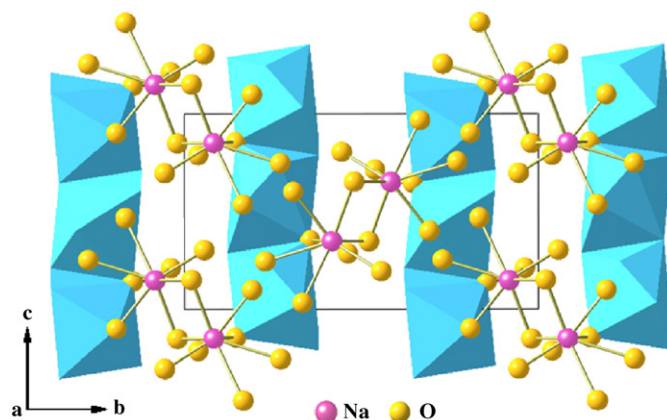
Fig. 5. Coordination environments of cations. The polyhedra are [SrO_8] trigonal dodecahedra.
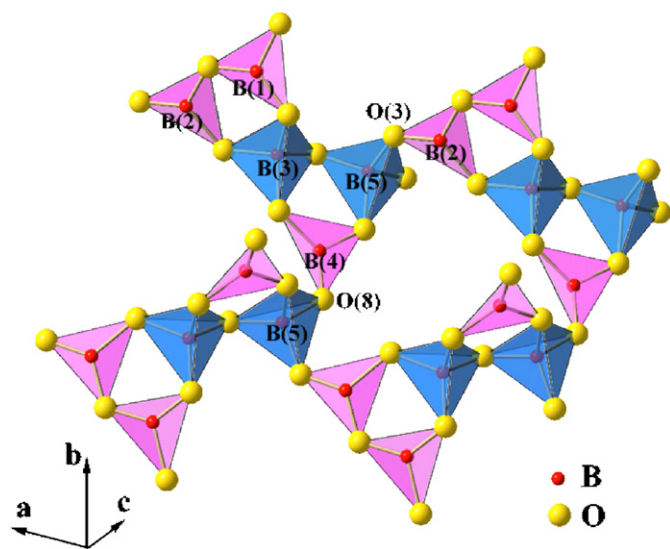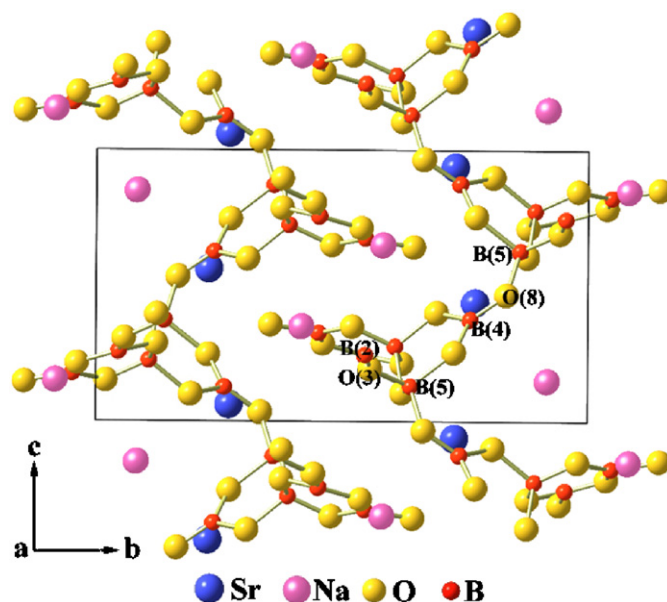

Fig. 3. Structure of B_5O_9 polyaniocs. Triangles are [BO_3] groups, and tetrahedra represent [BO_4] groups.

Fig. 3 the labels of the B atoms B(1), B(2) and B(4)) and two [BO_4] tetrahedra (T) (give for clarity on Fig. 3 the labels of the B atoms B(3) and B(5)). In the [B_5O_9] group,

one T(B(3)) in the middle, connects Δ(B(1)) + Δ(B(2)) and Δ(B(4)) + T(B(5)) on two sides, respectively. Then the $\Delta + \Delta$ are approximately perpendicular to the Δ on the other side. Those [B_5O_9]$^{3-}$ anionic groups connect with each other by sharing O(3) between B(2) and B(5), and sharing O(8) between B(4) and B(5), forming separated complex sheets (Fig. 4). The Na atoms are just distributed between the sheets, connect them by sharing O atoms with B atoms. Every Na atom is coordinated with eight O atoms to form trigonal dodecahedra, and every two trigonal dodecahedra are edge-sharing with each other, as illustrated in Fig. 5. The coordination surroundings of Sr atoms are shown in Figs. 5 and 6. The Sr atoms are surrounded by eight O atoms, forming trigonal dodecahedra. The [SrO_8] polyhedra are connected with each other by edges along the c-axis, forming infinite chains, while the [NaO_8] polyhedra distributed between the chains, sharing edges with them.
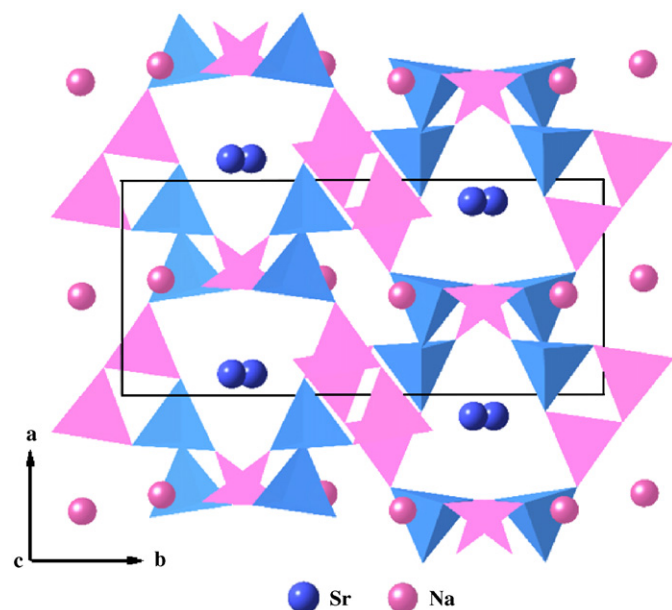
Fig. 6. Structure projection of $NaSrB_5O_9$ viewed along [001]. Triangles are $[BO_3]$ groups, and tetrahedral present $[BO_4]$ groups.

The positions of the Sr atoms are similar to that in SrB_4O_7 [7,37], which are almost enveloped by the [BO] network (Fig. 6). This kind of surroundings offers a possibility to stabilize a bivalent rare-earth ion without reducing atmosphere when the position of Sr^{2+} is replaced by some rare-earth ions, for example, Eu^{2+}. Then a photoluminescence can be expected in the doped compound. It is known that the Eu^{2+} doped borates will show various emissions, which can be correlated to the environment of the O atoms in the hosts. Materials with O atoms richly (more than two) coordinated by Ba or Sr atoms have longer Eu^{2+} emission wavelengths [10]. In $NaSrB_5O_9$, O(1) and O(4) are coordinated by two Sr atoms, and O(6), O(7), O(8), O(9) are only coordinated by one Sr atoms, while O(2), O(3) and O(5) has no Sr atoms surrounding. Such an environment is hopeful to get a shorter wavelength emission from a Eu^{2+} doped sample, such as blue or green emission.

3.3. Comparison of the known ternary compounds in the B-rich part of $Na_2O–SrO–B_2O_3$ and $Na_2O–CaO–B_2O_3$ systems

It is well known that the Sr and Ca atoms are easy to form isostructural compounds, such as the compounds in the binary system $CaO–B_2O_3$ [38–42] and $SrO–B_2O_3$ [33–37]. But it is not always the truth in the ternary compounds, even they have the same formula types, such as (i) $NaCaBO_3$ [21] and $NaSrBO_3$ [11], (ii) $NaCa_4(BO_3)_3$ [15] and $NaSr_4(BO_3)_3$ [12], (iii) $Na_3CaB_5O_{10}$ [13] and $Na_3SrB_5O_{10}$, and (iv) $NaCaB_5O_9$ [13] and $NaSrB_5O_9$. Only the last pair is isostructural. The first pair of orthoborates crystallizes in different crystal systems, $NaCaBO_3$ belongs

to the orthorhombic system, while $NaSrBO_3$ is monoclinic. The coordination methods of alkali and alkaline-earth cations are also different [11], although the fundamental building units are all $[BO_3]^{3-}$ groups. As to the second pair, $NaCa_4(BO_3)_3$ crystallizes in non-centrosymmetric space group *Ama*2, while $NaSr_4(BO_3)_3$ is in a centrosymmetric space group *Ia*−3*d*, which is a peculiar cubic borate. Even the structure of $Na_3SrB_5O_{10}$ has not been fully determined, a pure phase powder diffraction data can be indexed by DICVOL91, the systematic absence of $h+k = 2n+1$, $h01$ with $h = 2n+1$, $0k0$ with $k = 2n+1$, and 001 with $l = 2n+1$ indicates the possible space group is *C*2, *Cm* or *C*2/*m*. The details of the indexing result can be checked in the powder diffraction files from ICDD with number 56-0146. Since $Na_3CaB_5O_{10}$ crystallizes in *P*−1, the third pair of borates crystallizes in different space groups. As discussed above, alkali metal and alkaline-earth metal are very active, and are easy to form various crystal structures with boric acid, which bring us a large field to find new functional materials.

4. Conclusion

In this work, the subsolidus phase relations of the B-rich part of the $Na_2O–SrO–B_2O_3$ system were established from 26 samples by powder diffraction method. There are four ternary compounds in this part, including two new compounds, $NaSrB_5O_9$ and $Na_3SrB_5O_{10}$. The new borates were synthesized by solid-state reactions, and the crystal structure of $NaSrB_5O_9$ was refined from powder X-ray diffraction by Rietveld method, taking $NaCaB_5O_9$ as the structural model. It is composed of separated $[B_5O_9]^{3-}$ anionic sheets, with the $[NaO_8]$ polyhedra acting as the connections in between the sheets. The Sr atoms are located in the anionic sheets, and surrounded by [BO] groups, which offer a suitable place for it to be replaced by bivalent rare-earth ions without reducing atmosphere. $Na_3SrB_5O_{10}$ crystallizes in a different space group from $Na_3CaB_5O_{10}$. Because of the missing of diffraction peaks at high degree in both the powder diffraction and the single crystal diffraction, the structure of $Na_3SrB_5O_{10}$ has not been fully determined. Further experiment is under going to solve the problem.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jssc.2007.02.014.

References

[1] P. Becker, Adv. Mater. 10 (1998) 979–992.
[2] C.T. Chen, N. Ye, J. Lin, J. Jiang, W. Zeng, B. Wu, Adv. Mater. 11 (1999) 1071–1078.
[3] C.T. Chen, B. Wu, A. Jiang, G. You, Sci. China B 18 (1985) 235–243.
[4] C.T. Chen, Y. Wu, A. Jiang, B. Wu, G. You, R. Li, S. Lin, J. Opt. Soc. Am. B 6 (1989) 616–621.
[5] C.T. Chen, Y. Wang, B. Wu, K. Wu, W. Zeng, L. Yu, Nature 373 (1995) 322–324.
[6] Z. Hu, T. Higashiyama, M. Yoshimura, Y. Mori, T. Sasaki, Z. Kristallogr. 214 (1999) 433–434.
[7] Z.W. Pei, Q. Su, J. Alloys Compds. 198 (1993) 51–53.
[8] K.I. Schaffers, D.A. Keszler, Inorg. Chem. 33 (1994) 1201–1204.
[9] A. Diaz, D.A. Keszler, Mater. Res. Bull. 31 (1996) 147–151.
[10] A. Diaz, D.A. Keszler, Chem. Mater. 9 (1997) 2071–2077.
[11] L. Wu, X.L. Chen, Y. Zhang, Y.F. Kong, J.J. Xu, Y.P. Xu, J. Solid State Chem. 179 (2006) 1219–1224.
[12] L. Wu, X.L. Chen, H. Li, M. He, Y.P. Xu, X.Z. Li, Inorg. Chem. 44 (2005) 6409–6414.
[13] J. Fayos, R.A. Howie, F.P. Glasser, Acta Crystallogr. C 41 (1985) 1396–1398.
[14] L. Wu, X.L. Chen, Y.P. Xu, Y.P. Sun, Inorg. Chem. 45 (2006) 3042–3047.
[15] N. Penin, L. Seguin, M. Touboul, G. Nowogrocki, Inter. J. Inorg. Mater. 3 (2001) 1015–1023.
[16] Q. Huang, S. Lu, G. Dai, J. Liang, Acta Crystallogr. C 48 (1992) 1576–1578.
[17] A. Boultif, D. Louer, J. Appl. Crystallogr. 24 (1991) 987–993.
[18] H.M. Rietveld, Acta Crystallogr. 22 (1967) 151–152.
[19] H.M. Rietveld, J. Appl. Crystallogr. 2 (1969) 65–71.
[20] J. Rodriquez-Carvajal, M.T. Fernadez-Diaz, J.L. Martinez, J. Phys. Condens. Matter 3 (1991) 3215–3234.
[21] L. Wu, X.L. Chen, X.Z. Li, L. Dai, Y.P. Xu, M. Zhao, Acta Crystallogr. C 61 (2005) i32–i34.
[22] J. Krogh-Moe, Acta Crystallogr. B 30 (1974) 578–582.
[23] H. Li, J. Liang, J. Am. Ceram. Soc. 78 (1995) 470.
[24] A.S. Kanishcheva, A.V. Egorysheva, Yu.E. Gorbunova, Yu.F. Kargin, Yu.N. Mikhailov, V.M. Skorikov, Zh. Neorg. Khim. 49 (2004) 1006–1011.
[25] J. Krogh-Moe, Acta Crystallogr. B 30 (1974) 747–752.
[26] J. Krogh-Moe, Acta Crystallogr. B 28 (1972) 1571–1576.
[27] A. Hyman, A. Perloff, F. Mauer, S. Block, Acta Crystallogr. 22 (1967) 815–821.
[28] R.S. Bubnova, Yu.F. Shepelev, N.A. Sennova, S.K. Filatov, Z. Kristallogr. 217 (2002) 444–450.
[29] N. Penin, M. Touboul, G. Nowogrocki, J. Solid State Chem. 168 (2002) 316–321.
[30] N. Penin, M. Touboul, G. Nowogrocki, J. Alloys Compds. 363 (2004) 104–111.
[31] H. Koenig, R. Hoppe, Z. Anorg. Allg. Chem. 434 (1977) 225–232.
[32] H. Koenig, R. Hoppe, M. Jansen, Z. Anorg. Allg. Chem. 449 (1979) 91–101.
[33] J.B. Kim, K.S. Lee, I.H. Suh, J.H. Lee, J.R. Park, Y.H. Shin, Acta Crystallogr. C 52 (1996) 498–500.
[34] N.L. Ross, R.J. Angel, J. Solid State Chem. 90 (1991) 27–30.
[35] Z.F. Wei, X.L. Chen, F.M. Wang, W.C. Li, M. He, Y. Zhang, J. Alloys Compds. 327 (2001) 10–13.
[36] Q.S. Lin, W.D. Cheng, J.T. Chen, J.S. Huang, J. Solid State Chem. 144 (1999) 30–34.
[37] A. Perloff, S. Block, Acta Crystallogr. 20 (1966) 274–279.
[38] A. Vegas, F.H. Cano, S. Garcia-Blanco, Acta Crystallogr. B 31 (1975) 1416–1419.
[39] Q.S. Lin, W.D. Cheng, J.T. Chen, J.S. Huang, Acta Crystallogr. C 55 (1999) 4–6.
[40] A. Kirfel, Acta Crystallogr. B 43 (1987) 333–343.
[41] M. Marezio, J.P. Remeika, P.D. Dernier, Acta Crystallogr. B 25 (1969) 965–970.
[42] H. Huppertz, Z. Naturforsch. B 58 (2003) 257–265.