Chloride flux growth of crystalline strontium niobates and nitridation to perovskite SrNbO$_2$N

Yue Li$^{a,b,1}$, Feng-Qiang Xiong$^{b,1}$, Ruiling Ou$^{a,b}$, Lipeng Wan$^{a,b}$, Congling Yin$^{c}$, Ruxin Che$^{a,⁎}$, Minghui Yang$^{b,⁎}$

$^a$ College of Materials Science and Engineering, College of Environmental and Chemical Engineering, Dalian Jiaotong University, Dalian 116028, China
$^b$ Dalian National Laboratory for Clean Energy, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, Dalian 116023, China
$^c$ School of Materials Science and Engineering, Jiangxi University of Science and Technology, Ganzhou 341000, China

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ABSTRACT

Highly crystalline (110) layered perovskite Sr$_2$Nb$_2$O$_7$, (111) layered perovskite Sr$_5$Nb$_4$O$_{15}$, and complex perovskite Sr$_2$Nb$_2$O$_9$ were prepared by NaCl-KCl flux growth method from SrCO$_3$ and Nb$_2$O$_5$. This flux synthesis achieves single strontium niobate phase in contrast to mixed niobates from the solid state reaction with the same heating parameters. A little excess of Sr source was found to be required for the synthesis of Sr$_5$Nb$_4$O$_{15}$ and Sr$_2$Nb$_2$O$_7$ at elevated temperature due to slight evaporation. The three strontium niobates were converted to perovskite SrNbO$_2$N via thermal ammonolysis under NH$_3$ flow at 900 °C. Post-wash treatment was performed to remove the byproduct SrO. This makes additional nanopores in SrNbO$_2$N in the cases of Sr$_5$Nb$_4$O$_{15}$ and Sr$_2$Nb$_2$O$_7$, and results in increasing surface areas of SrNbO$_2$N with Sr:Nb ratios in the precursors (from 9.9 to 19.8 and 35.5 m$^2$/g). On the other hand, the UV–Vis diffusion reflectance spectra reveal decreasing light absorption by defects in SrNbO$_2$N prepared from precursor with higher Sr:Nb ratio. SrNbO$_2$N prepared from Sr$_2$Nb$_2$O$_7$ would be advantageous for applications that require high surface area and low defect density of the material.

1. Introduction

Oxides and oxynitrides with perovskite related crystal structures are significantly attractive for research due to their various unique properties such as dielectric [1–3], magnetic [4], optical properties [2,5], ionic conductivity [6], photocatalytic [7–9], and so on. Combination of Sr(II) and Nb(V) does not fit the standard perovskite ABO$_3$, but they are made-up of compounds with perovkite derivative structures. Hence, Fig. 1 shows the structures of Sr$_2$Nb$_2$O$_7$, Sr$_5$Nb$_4$O$_{15}$, Sr$_2$Nb$_2$O$_9$, and SrNbO$_2$N. Sr$_2$Nb$_2$O$_7$ is a (110) layered perovskite [7], which has an orthorhombic lattice with space group Cmc21 and lattice constants: $a$=3.9577 Å, $b$=26.787 Å, $c$=5.7014 Å [10]. Sr$_5$Nb$_4$O$_{15}$ is a (111) layered perovskite [8]; it has a hexagonal lattice with space group P-$\overline{3}$m1 and lattice constants: $a$=5.6565 Å, $c$=11.4586 Å [11]. Sr$_2$Nb$_2$O$_9$ is equivalent to Sr(Sr$_{1/3}$Nb$_{2/3}$)O$_3$, which can be referred to as a complex perovskite with Sr on both A sites and 1/3 B sites, and it has a cubic lattice with space group Pm-3m and $a$=8.2682 Å [1]. SrNbO$_2$N is an oxynitride perovskite, which has a cubic lattice with space group Pm-3m and $a$=4.044 Å [12]. The layered perovskites Sr$_2$Nb$_2$O$_7$ and Sr$_5$Nb$_4$O$_{15}$ are both highly efficient photocatalysts under UV light [7,8]. On the other hand, the substitution of O$^2$− with less electronegative and more polarizable N$^3$− raises the valence band maximum and decreases the band gap remarkably, so N-doped Sr$_2$Nb$_2$O$_7$ and N-doped Sr$_5$Nb$_4$O$_{15}$ are functional for visible-light photocatalysis [13,14]. Furthermore, the stoichiometric replacement product SrNbO$_2$N is an emerging photocatalyst for water splitting which may respond to wide range of sun light (band edge ~680 nm) [15,16]. Sr$_2$Nb$_2$O$_7$ has also been investigated for its thermoelectric, high-temperature ferroelectric and piezoelectric applications [17–19]. It may be interesting idea to note that characters such as high crystallinity and low defect density are crucial for the performance of these materials in the application fields. For instance, low crystallinity and high defect density decrease photocatalytic activity by accelerating recombination of photogenerated electrons and holes [20].

Sr$_2$Nb$_2$O$_7$, Sr$_5$Nb$_4$O$_{15}$ and Sr$_2$Nb$_2$O$_9$ are generally prepared via solid-state reaction [1,7,21], and polymerizable complex synthesis [8,22]. Up to now, only the flux synthesis of Sr$_2$Nb$_2$O$_7$ has been reported among them [23]. Generally, the solid state reaction method...
gives good crystallinity, but requires high temperature and long holding time for a complete conversion; while polymerizable complex synthesis is performed at a moderate temperature, but the crystallinity is low unless calcined at high temperature. In contrast, flux synthesis at lower temperatures than solid state reaction is still advantageous for high crystallinity and tunable distinct facets of the product [24–26]. Therefore, developing flux synthesis for the series of strontium niobates is desirable for the applications purposes. In addition, it would be illuminative for flux synthesis of similar oxides with two metals.

Conventionally, SrNbO$_2$N is prepared via thermal ammonolysis of equal-cation-stoichiometry Sr$_2$Nb$_2$O$_7$ in NH$_3$ atmosphere [27,28]. However, Nb(V) is readily reduced at high temperature, either via reaction with H$_2$ from NH$_3$ splitting or decomposition of Nb-N bond which in turn evolves N$_2$, and this results in defects in conventional SrNbO$_2$N. Interestingly, SrNbO$_2$N prepared from Sr$_5$Nb$_4$O$_{15}$ exhibits lower defect absorption at wavelengths beyond the band edge, smaller particle size and higher specific surface area than that prepared from Sr$_2$Nb$_2$O$_7$ [16]. Similar phenomena have also been found in LaTiO$_2$N prepared from La$_2$TiO$_5$ compared to that prepared from La$_2$Ti$_2$O$_7$ [27,28]. From previous reports, we notice that neither insight into the mechanism for the low defect density has been proposed, nor the improvement in these preparations has been discussed as a strategy.

Herein, we report the growth of pure Sr$_2$Nb$_2$O$_7$, Sr$_5$Nb$_4$O$_{15}$ and Sr$_4$Nb$_2$O$_9$ crystals by the chloride flux method, and their subsequent transformation to SrNbO$_2$N via nitridation under NH$_3$ flow. A little excess of Sr source and elevated temperatures were found to be required for the synthesis of Sr$_5$Nb$_4$O$_{15}$ and Sr$_4$Nb$_2$O$_9$. The effects of excess Sr/Nb stoichiometry on the preparation of SrNbO$_2$N are discussed in detail with the mechanism. The light absorption by defects of SrNbO$_2$N samples are in decreasing order of the precursors: Sr$_2$Nb$_2$O$_7$, Sr$_5$Nb$_4$O$_{15}$ and Sr$_4$Nb$_2$O$_9$, while the specific surface areas of SrNbO$_2$N samples are in increasing order. SrNbO$_2$N prepared from Sr$_2$Nb$_2$O$_7$ is almost free of light-absorbing defects, and it also has a high surface area (35.5 m$^2$/g).

2. Experimental section

2.1. Growth of Sr$_2$Nb$_2$O$_7$, Sr$_5$Nb$_4$O$_{15}$ and Sr$_4$Nb$_2$O$_9$ crystals

Strontium niobate crystals were grown via flux (molten-salt) method. Typically, SrCO$_3$ (Aladdin, 99.95%), Nb$_2$O$_5$ (Sinopharm Chemical, 99.99%), NaCl and KCl (Aladdin, 99.99%) were mixed in a molar ratio of Sr:Nb:Na:K=x:1:4:4, where x is a ratio modified from the stoichiometry of the strontium niobate. Here, NaCl and KCl were used as flux [30]. The mixture was heated in air to 650 °C at a heating rate of 20 °C/min, further to 750 °C at a heating rate of 2 °C/min, and held for 60 min. This slow heating procedure around the melting point of the mixed flux (680 °C) and decomposing temperature of SrCO$_3$ (720–750 °C [23]) allows a mild evolution of CO$_2$. Hence this process creates an opportunity of avoiding tempestuous boiling. Then, the mixture was heated to the aimed temperature at heating rate of 5 °C/min and held for 5 h, followed by cooling to 650 °C at a cooling rate of 5 °C/min, and finally naturally cooled to room temperature. The products were thoroughly washed with water to remove the flux, and dried at 60 °C. The ratio x and the aimed temperature were found to be 1, 900 °C, (1.275, 1000 °C) and (2.2, 1100 °C) for pure Sr$_2$Nb$_2$O$_7$, Sr$_5$Nb$_4$O$_{15}$ and Sr$_4$Nb$_2$O$_9$, respectively.

2.2. Preparation of SrNbO$_2$N

The pure strontium niobates were nitrided under a 250 sccm NH$_3$ (99.999%) flow at 900 °C for 15 h, followed by natural cooling. For Sr$_2$Nb$_2$O$_7$, the nitriding period of 15 h was found to be insufficient for a complete conversion, so a 30 h nitridation was used for complete conversion. The obtained products were washed via adding diluted HCl under stirring until the pH was 3. This was followed by filtering, washing with water and the sample was then dried at 60 °C. By washing with diluted HCl under controlled pH, SrO generated during thermal ammonolysis was dissolved while SrNbO$_2$N phase remained, and nanopores were generated. The final products are labeled as SrNbO$_2$N-f.227, SrNbO$_2$N-f.5415 and SrNbO$_2$N-f.429 corresponding to the precursors Sr$_2$Nb$_2$O$_7$, Sr$_5$Nb$_4$O$_{15}$ and Sr$_4$Nb$_2$O$_9$, respectively.

2.3. Characterizations

The crystal structure was analyzed using X-ray diffraction (XRD, Rigaku Miniflex 600 X-ray diffractometer, Cu Kα; scanning rate, 5°/min). Crystal structures and the lattice parameters were calculated with the use of the Rietveld method by the software package Bruker TOPAS. The morphology was characterized by scanning electron microscopy (SEM, JSM-7800F, Japan). The specific surface area and pore size distribution were determined with a N$_2$ physisorption technique (Autosorb iQ2) using the Brunauer-Emmett-Teller model and Barret-Joyaer-Halenda method, respectively. UV–Vis diffuse reflectance spectra was recorded on a spectrophotometer (Hitachi U-3900 spectrometer) using a BaSO$_4$ standard as the reference, and the spectra were converted from reflectance to the Kubelka-Munk function.

3. Result and discussion

3.1. Flux synthesis of Sr$_2$Nb$_2$O$_7$, Sr$_5$Nb$_4$O$_{15}$ and Sr$_4$Nb$_2$O$_9$ crystals

The flux synthesis of Sr$_2$Nb$_2$O$_7$ was compared with the solid state reaction method using the same heating procedure. As shown by the XRD patterns in Fig. 2, SrNbO$_2$N (ICCD PDF#28-1243) and Sr$_5$Nb$_4$O$_{15}$ (ICCD PDF#48-0421) are included in the products from solid state
reaction of stoichiometric SrCO₃ and Nb₂O₅ at 900 °C for 5 h besides Sr₂Nb₂O₇ (ICCD PDF#52-0321). In contrast, pure and highly crystalline Sr₂Nb₂O₇ is obtained from the chloride flux synthesis. It may be worthwhile to note that decomposition of SrCO₃ gives rise to SrO formation at temperatures higher than 750 °C [23], and SrO is dissolvable in the mixed chloride molten salt while Nb₂O₅ is insoluble in alkali chlorides [23]. Therefore, the diffusion of SrO is facilitated in the flux synthesis, which leads to a uniform reaction. In contrast, the diffusion of reactants is slow in solid state reaction, and the local ratio of the reactants is not uniform; this gives rise to other products with different Sr/Nb ratios unless the reaction temperature is elevated.

Furthermore, the flux synthesis of Sr₅Nb₄O₁₅ was firstly tried using the same parameters used for the synthesis of Sr₂Nb₂O₇ but different Sr/Nb ratio. Eventually the XRD pattern indicates the present of trace Sr₂Nb₂O₇ in the product at 900 °C with stoichiometric feed ratio (Sr/Nb=1.25, Fig. 3a), which suggests slight evaporation of Sr source during the synthesis. Even when a Sr-excess feed ratio (Sr/Nb=1.275) is used, Sr₂Nb₂O₇ together with SrCO₃ (ICCD PDF#05–0418) remains in the product (Fig. 3b). This may result from the recombination of SrO and CO₂ during cooling. Moreover, it is indicated that the reaction temperature at 900 °C for 5 h is insufficient for the flux synthesis of Sr₅Nb₄O₁₅. In an optimized step, a reaction temperature of 1000 °C for 5 h was used, and pure and highly crystalline Sr₅Nb₄O₁₅ was obtained with feed ratio Sr/Nb=1.275 (Fig. 3c).

Similarly, the flux synthesis of Sr₄Nb₂O₉ was performed at 1000 °C for 5 h at first. As shown by the XRD patterns in Fig. 4a, there is appearance of Sr₅Nb₄O₁₅ peaks together with Sr₄Nb₂O₉ peaks in the product with stoichiometric feed ratio (Sr/Nb=2). Furthermore both Sr₅Nb₄O₁₅ and SrCO₃ peaks appear when the feed ratio is increased (Sr/Nb=2.1, Fig. 4b). These indicate slight evaporation of Sr source and an incomplete reaction at 1000 °C for 5 h. When the synthesis is carried out at 1100 °C, obviously less amount of Sr₅Nb₄O₁₅ was obtained compared to synthesis at 1000 °C, and SrCO₃ was not observed in the product with reactants feeding ratio Sr/Nb=2.1 (Fig. 4c). Moreover, pure and highly crystalline Sr₄Nb₂O₉ was obtained with feeding ratio Sr/Nb=2.2 (Fig. 4d).

It is important to note that as SrO is dissolvable in the mixed chloride molten salt and Nb₂O₅ is insoluble [23], the flux facilitates the diffusion of SrO and thus the reaction with Nb₂O₅. Therefore, it is reasonable to postulate that Sr₂Nb₂O₇, Sr₅Nb₄O₁₅ and Sr₄Nb₂O₉ are formed in series along with the arrival of SrO. This is consistent with the phenomenon that Sr₂Nb₂O₇ and Sr₅Nb₄O₁₅ form in the synthesis of Sr₄Nb₂O₉ and Sr₅Nb₄O₁₅ respectively due to incomplete reaction. The synthesis temperature for the pure phase Sr₅Nb₄O₁₅ < Sr₅Nb₄O₁₅ < Sr₅Nb₄O₁₅, which indicates that the reactivity of reactant SrO decreases as the amount of Nb₂O₅ increases.

The morphology of the pure strontium niobates is shown in Fig. 5, which indicates that Sr₅Nb₄O₁₅ crystals prepared through flux synthesis are sheet-like with sharp facets, and interestingly some of the sheets assemble in form of pyramids (Fig. 5a). The sizes of the sheets are 200–500 nm and the thicknesses are 50–100 nm. It is obvious from SEM images in Fig. 5b that Sr₅Nb₄O₁₅ crystals are also sheet-like with sharp
facets, and the sizes range from 5 to 30 µm. Furthermore the size of Sr2Nb2O9 crystals are in the tens micrometers regime (Fig. 5c). The elevation of reaction temperature contributes to the crystal size increments of both Sr5Nb4O15 and Sr4Nb2O9.

3.2. SrNbO2N samples derived from pure Sr2Nb2O7, Sr5Nb4O15 and Sr4Nb2O9

As shown by the XRD patterns in Fig. 6, all the three strontium niobates are successfully converted to perovskite SrNbO2N (ICCD PDF#39-0675) after nitridation. It is obvious from the result that SrO (ICCD PDF#48-1477) phase appears in the product from Sr4Nb2O9 (Fig. 6c). Interestingly, the SrO phase is not detected in the XRD pattern of the product from another Sr-excess precursor Sr5Nb4O15 (Fig. 6b). This may result from high dispersion and small size of SrO, which is in line with the presence of SrCO3 phase in the product after washing with water without excluding CO2. In view of this fact, the products were washed with diluted HCl to remove SrO. As shown in Fig. 7, pure SrNbO2N is the product of all the three samples after the acidic treatment, and the calculated lattice parameters are closed to each other (a=4.042 Å, 4.042 Å and 4.038 Å for SrNbO2N-f.227, SrNbO2N-f.5415 and SrNbO2N-f.429, respectively). The particle sizes of SrNbO2N-f.227, SrNbO2N-f.5415 and SrNbO2N-f.429 are about 78 nm, 39 nm and 20 nm respectively estimated from broadening of XRD peaks by using the Debye-Scherrer equation.

SEM images of SrNbO2N-f.227, SrNbO2N-f.5415 and SrNbO2N-f.429 are shown in Fig. 8. All three samples are porous; the particle sizes are in the order of SrNbO2N-f.227 > SrNbO2N-f.5415 > SrNbO2N-f.429, which is consistent with the XRD results. This could results from the homogenously excess SrO in the precursor Sr3Nb15O40 and Sr5Nb2O15. It is of good interest to note that during the transformation of Sr5Nb4O15 and Sr4Nb2O9 to SrNbO2N, SrO would be generated among SrNbO2N domains, which subsequently restrict their growth. The surface area and pore size distribution were analyzed using the N2 physisorption technique. As shown in Fig. 9, hysteresis loops are observed in the isotherms of SrNbO2N samples, which indicates mesoporous structures [31]; the mesopore size distribution of the three samples are all major in 2–10 nm, and specific pore volumes are in the order of SrNbO2N-f.227 < SrNbO2N-f.5415 < SrNbO2N-f.429. The specific surface area are 9.9 m2 g−1, 19.8 m2 g−1 and 35.5 m2 g−1 for SrNbO2N-f.227, SrNbO2N-f.5415 and SrNbO2N-f.429, respectively. In SrNbO2N-f.227, the pores arise from the substitution of 3 oxygen atoms by 2 nitrogen atoms during nitridation. Comparing with SrNbO2N-f.227, additional pores in SrNbO2N-f.5415 and SrNbO2N-f.429 arise from the removal of excess SrO. Therefore, the decrease in particle size of SrNbO2N and the increase in pore amount induced by homogenously excess SrO in the precursors result in an increasing specific surface area in the order of SrNbO2N-f.227, SrNbO2N-f.5415 and SrNbO2N-f.429.

The optical characters of semiconductor SrNbO2N are investigated using UV/Vis diffuse reflectance spectroscopy. As shown in Fig. 10, the absorption band edges of SrNbO2N samples are ~680 nm, which corresponds to the band gap ~1.8 eV, and the band edges have redshifts of ~350 nm comparing with the precursor oxides. This arises from the substitution of O2− with less electronegative and more polarizable N3− which raises the valence band to maximum. The spectrum of SrNbO2N-f.227 has an obvious flat edge among all the wavelengths longer than the band edge, which has been assigned to the

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Fig. 5. SEM images of pure strontium niobates, (a) Sr2Nb2O7 synthesized at 900 °C with feed ratio of Sr:Nb=1:1, (b) Sr3Nb4O15 synthesized at 1000 °C with feed ratio of Sr:Nb=1.275:1 and (c) Sr4Nb2O9 synthesized at 1100 °C with feed ratio of Sr:Nb=2.2:1.

Fig. 6. XRD patterns of products from nitridation of (a) Sr2Nb2O7, (b) Sr5Nb4O15 and (c) Sr4Nb2O9.

Fig. 7. XRD patterns of products from nitridation and acid treatment using precursors (a) Sr2Nb2O7, (b) Sr5Nb4O15, and (c) Sr4Nb2O9.

Fig. 8. SEM images of SrNbO2N-f.227, SrNbO2N-f.5415 and SrNbO2N-f.429.
absorption by defects, such as reduced Nb species [28,32]. The spectrum of SrNbO2N-f.5415 shows much lower defect absorption than SrNbO2N-f.227, similarly as reported by Urabe et al., which indicates the presence of low density of defect states in SrNbO2N-f.5415 [16]. And the spectrum of SrNbO2N-f.429 exhibits even lower defect absorption than SrNbO2N-f.5415, which is nearly free of defect absorption centers. These results suggest that excess SrO in precursors Sr5Nb4O15 and Sr4Nb2O9 inhibits the formation of a defect absorption center during the process of nitridation.

The Sr:Nb ratio of the precursors increases successively in the sequence of Sr2Nb2O7, Sr5Nb4O15 and Sr4Nb2O9. As Sr is much more electropositive than Nb, it is postulated that the electron donation effect of Sr in Sr(II)-O bonds to Nb(V)-O bonds decreases the effective positive charge of Nb(V) though the Nb(V) atoms are all hexacoordinated in these compounds. Hence the formation of low-valent Nb together with related defects is inhibited during nitridation of the precursors in the order of Sr2Nb2O7 < Sr5Nb4O15 < Sr4Nb2O9. Since the unit cells of Sr2Nb2O7 and Sr5Nb4O15 are composed of fixed atoms while Sr4Nb2O9 contains mixed atom sites, the effective positive charges of Nb(V) in Sr2Nb2O7 and Sr5Nb4O15 were computed via Mulliken atomic population analysis (See Supplementary Material, Tables S1 and S2). The Mulliken charges of Nb(V) atoms in Sr5Nb4O15 (+1.10, +1.12) are lower than those in Sr5Nb4O15 (+1.20, +1.21). This agrees with the above postulation. XPS of Nb 3d for SrNbO2N samples prepared from the normal precursor Sr5Nb4O15 and Sr-excess precursor Sr5Nb4O15 are shown in Fig. S2. There are two pairs of Nb 3d peaks in each sample. The Nb 3d5/2 peaks at 206.9 eV and 205.6 eV could be assigned to Nb(V) and Nb(IV), respectively [33]. The ratio of Nb(VI) in total Nb near the surface calculated from XPS for SrNbO2N-f.5415 (34%) is lower than that for SrNbO2N-f.227 (52%). This confirms that the reduction of Nb(V) on reaction interface for thermal ammonolysis of Sr5Nb4O15 is inhibited somewhat comparing with that of Sr5Nb4O7.
Analogously, in the preparation of a mixed metal nitride containing a reducible metal (commonly labeled as B) and a highly electropositive metal (commonly labeled as A), such as alkaline earth or rare earth element, the excess oxide A of the precursor would help to stabilize the high valence state of B. This makes a strategy to inhibit the reduction of high-valent state metal in the preparation of such kind of mixed metal nitrides.

4. Conclusions

In summary, pure and highly crystalline \( \text{Sr}_2\text{Nb}_2\text{O}_7 \), \( \text{Sr}_5\text{Nb}_4\text{O}_{15} \) and \( \text{Sr}_2\text{Nb}_2\text{O}_8 \) with sharp facets are successfully prepared from mixed chloride flux synthesis. The temperature for the synthesis of pure phase increases in the order of \( \text{Sr}_2\text{Nb}_2\text{O}_7, \text{Sr}_5\text{Nb}_4\text{O}_{15} \), and \( \text{Sr}_2\text{Nb}_2\text{O}_8 \), as a result of the decrease in the reactivity of \( \text{SrO} \) as the stoichiometric three strontium niobates, the homogenously excess \( \text{SrO} \) in the precursor oxides \( \text{Sr}_2\text{Nb}_2\text{O}_7 \) and \( \text{Sr}_5\text{Nb}_4\text{O}_{15} \) restricts the particle size of \( \text{SrNbO}_2\text{N} \) and makes additional nanoparticles in \( \text{SrNbO}_2\text{N} \) except for those arising from N substitution for O. These effects make the accessibility of the porous perovskite-type nitride with tunable porosity, and lead to an increasing surface area of \( \text{SrNbO}_2\text{N} \) with tunable ratio. Moreover, the homogenously excess \( \text{SrO} \) can decrease the effective positive charge of \( \text{Nb}(V) \), which inhibits the reduction of \( \text{Nb}(V) \) during nitridation, and thus has fewer low-valent Nb defects in \( \text{SrNbO}_2\text{N} \) prepared from precursor with higher \( \text{Sr}:\text{Nb} \) ratio. \( \text{SrNbO}_2\text{N} \) prepared from \( \text{Sr}_2\text{Nb}_2\text{O}_8 \) has a surface area as high as \( 35.5 \, \text{m}^2 \, \text{g}^{-1} \), and is nearly free of defect absorption centers, which would be advantageous for applications that require high surface area (such as electrode and catalytic materials) and low defect density (such as dielectrics, photocatalytic materials, and photocatalysts).

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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.ceramint.2017.03.071.

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