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Influence of substitution of Nd³⁺ for Bi³⁺ on structure and piezoelectric properties of SrBi_{2- χ}Nd_{χ}Nb₂O₉ (χ =0, 0.1, 0.2 and 0.4)

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Abstract: SrBi_{2- χ}Nd_{χ}Nb₂O₉ (χ =0, 0.1, 0.2 and 0.4) bismuth layer-structured ferroelectric ceramics were prepared by the solid-state reaction sintering method. The accurate position of Nd element in SrBi_{2- χ}Nd_{χ}Nb₂O₉ ceramics was determined by the X-ray Rietveld method and Synchrotron radiation X-ray absorption fine structure (XAFS) technology. The partial substitution of Nd³⁺ for Bi³⁺ leads to the decrease in the distortion of NbO₆ octahedron for SrBi_{2- χ}Nd_{χ}Nb₂O₉ ceramics and also lowers the piezoelectric properties of SrBi_{2- χ}Nd_{χ}Nb₂O₉ ceramics. Meanwhile, the temperature coefficient of resonant frequency (TCF) decreases when Nd element partially replaces Bi element in SrBi_{2- χ}Nd_{χ}Nb₂O₉ ceramics.

Key words: bismuth layer-structured ferroelectrics; X-ray Rietveld method; synchrotron radiation XAFS; piezoelectric properties

1 Introduction

Bismuth layer-structured ferroelectrics (BLSFs) have attracted much interest for their fatigue-free properties in nonvolatile ferroelectric thin film random access memory and high temperature piezoelectric devices. BLSFs can be described by the general formula $(Bi_2O_2)^{2+}(A_{m-1}B_mO_{3m-1})^{2-}$ (*m*=1, 2, 3, 4, 5, 6). The crystal structure of these compounds consists of fluorite-like $(Bi_2O_2)^{2+}$ layers interleaved with (*m*-1) perovskite-like blocks $(A_{m-1}B_mO_{3m-1})^{2-}[1]$. SrBi₂Nb₂O₉ (SBN) is a member of BLSFs family (i.e. *m*=2, A=Sr²⁺, B=Nb⁵⁺), which is composed of Bi₂O₂ layers and perovskite-like SrNb₂O₇ units with double NbO₆ octahedral layers.

BLSFs have been widely studied because of their excellent fatigue-free properties[2–4]. In previous studies, we have found that Pr^{3+} , La^{3+} , Nd^{3+} and Sm^{3+} elements can be introduced into Bi site for SBN ceramics and a relaxor behavior is reduced by the substitution of these lanthanide (Ln) elements for Bi ions[5–8]. However, there are few detailed studies about the

accurate position of the dopant element like Nd³⁺ for these Ln-doped SBN ceramics[9]. On the other hand, SBN is suitable for fine tolerance resonator applications due to its low temperature coefficient of resonant frequency (TCF) value, low electromechanical coupling coefficient (K_n) , and high mechanical quality factor (Q_m) . In addition, the substitution of Nd³⁺ for Bi³⁺ is most likely to further lower TCF value like the case of Sm-doped SBN[8]. Therefore, in the present work, the accurate position of Nd dopant was investigated by means of the X-ray Rietveld refinement method and XAFS technique and the effects of the Nd dopant on the piezoelectric structure and properties of $SrBi_{2-\gamma}Nd_{\gamma}Nb_{2}O_{9}$ ($\chi=0, 0.1, 0.2$ and 0.4) were also studied in detail.

2 Experimental

The conventional solid-state reaction sintering method was used to prepare $SrBi_{2-\chi}Nd_{\chi}Nb_{2}O_{9}$ ($\chi=0, 0.1, 0.2$ and 0.4) ceramics. Details about the preparation procedure were described elsewhere[7]. The ceramic

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samples for piezoelectric measurement were poled in a silicon oil bath at 180 $^{\circ}$ C under the electric field of about 8–10 kV/mm for 5–15 min.

Phase identification of samples was carried out by X-ray diffractometer (model: Dmax2550 V, Rigaku, Tokyo, Japan) with monochromic Cu K_a radiation. Intensities were measured in steps of 0.02° for 1 s in the range of 10°-120° at room temperature. 2θ Crystallographic and structural parameters were refined by the Rietveld method using the TOPAS software. XAFS measurements at Nd-L₃ absorption edge were performed using synchrotron radiation at the National Synchrotron Radiation Laboratory of China. The P-E hysteresis loops were measured by a TF analyzer (Model 2000, aixACCT Systems, Germany) at the temperature of 150 °C with a frequency of 1 Hz. The piezoelectric measurements were performed for 48 h after the poling. The piezoelectric constants, d_{33} , were measured using a quasi-static d₃₃ meter (Model ZJ-2, Institute of Acoustics, Chinese Academy of Science). The planar coupling factor $K_{\rm p}$ and the mechanical quality factor $Q_{\rm m}$ were determined using the resonance-antiresonance method. Resonance and anti-resonance frequencies in the temperature range from -20 to 140 °C were measured using precision impedance analyzer (Agilent 4294A, USA) combined with a temperature-controlling oven (VT7004, Germany). TCF was calculated using

$$TCF = \Delta f_r / (f_{r, 20 \ C} \times 100) \tag{1}$$

where Δf_r is the variation of the resonance frequency within the temperature range from -20 to 80 °C, and $f_{r, 20 °C}$ is the resonance frequency at 20 °C.

3 Results and discussion

3.1 Analysis of X-ray Rietveld refinement and XAFS measurement for SrBi_{2-y}Nd_yNb₂O₉

 $SrBi_{2-\chi}Nd_{\chi}Nb_{2}O_{9}$ ($\chi=0, 0.2$ and 0.4) ceramics powders were carried out by X-ray Rietveld refinement. Fig.1 shows the fitting result of X-ray Rietveld refinement of SrBi_{1.8}Nd_{0.2}Nb₂O₉ (i.e. χ =0.2) ceramics powder and Table 1 gives the Rietveld refined structural parameters for this sample. Some previous studies about SBN ceramics powders have been reported that the exchange of Sr²⁺ and Bi³⁺ occurs in SBN[10-12]. So, in our refinement for SrBi2-yNdyNb2O9, we assumed that some exchange of Sr²⁺ and Bi³⁺ existed for these three samples. The refinement was performed in the orthorhombic space group $A2_1am$. The results of refinement for these samples are presented in Table 1 and Table 2. It can be seen from Table 2 that the refinement parameters for these samples including three reliability factors (R_{wp} , R_{exp} and R_p) are reasonable values, which represent the reliability of the refined structural parameters. Table 1 shows that the exchange amount of Sr^{2+} and Bi^{3+} for $\chi=0.2$ sample is about 16.2%. Similarly, this exchange of Sr^{2+} and Bi^{3+} also happens for $\chi=0$ and 0.4 samples and the exchange amount is 16.7% and 15.1%, respectively, according to their refinement. On the other hand, considering that the radius of $Nd^{3+}(0.111)$ nm, coordinate number 8) is closer to Bi3+ (0.117 nm, coordinate number 8) and larger than Sr^{2+} (0.126 nm, coordinate number 8) and both Nd and Bi ions are trivalent, the refinement was performed based on the assumption that Nd³⁺ occupies the Bi-site in Bi₂O₂ layers for the Nd-doped SBN samples (χ =0.2 and 0.4). The



Fig.1 Fitting result of X-ray Rietveld refinement of $SrBi_{1.8}Nd_{0.2}Nb_2O_9$ (i.e. $\chi=0.2$) ceramics powder: (a) Experimental data spectrum; (b) Difference between data points and fitted line; (c) Position of diffraction peaks

SUN Lin, et al/Trans. Nonferrous Met. Soc. China 19(2009) 1459-1463

Site	$N_{ m p}$	x	У	Ζ	Atom	Occupancy	$B_{\rm eq}$
01	4	0.952 8(63)	0.784 200(66)	0	O^{-2}	1	0.670(19)
O2	8	0.429 7(42)	0.797 300(43)	0.3391 000(43)	O^{-2}	1	0.670(19)
O3	8	0.262 0(11)	0.996 000(25)	0.2422 000(59)	O^{-2}	1	0.670(19)
O4	8	0.762 8(82)	0.952 200(56)	0.0834 400(55)	O^{-2}	1	0.670(19)
05	8	0.761 2(75)	0.057 200(52)	0.5719 400(56)	O^{-2}	1	0.670(19)
Sr1	4	0	0.245 500(11)	0	Sr^{+2}	0.837 9(41)	0.890(68)
					Bi ⁺³	0.162 1(41)	0.890(68)
Bi2	8	0.498 1(22)	0.733 83(33)	0.201 505(41)	Sr^{+2}	0.081 0(21)	1.351(32)
					Bi ⁺³	0.819 0(21)	1.351(32)
					Nd ⁺³	0.1	1.351(32)
Nb1	8	0.508 4(21)	0.749 85(85)	0.413 926(56)	Nb ⁺⁵	1	0.375(44)

Table 1 Rietveld refined structural parameters for $SrBi_{1.8}Nd_{0.2}Nb_2O_9$ (i.e. $\chi=0.2$)

Table 2 Rietveld refined lattice constants for $SrBi_{2-\gamma}Nd_{\gamma}Nb_{2}O_{9}$ ($\chi=0, 0.2$ and 0.4)

Sample	a/Å	b/Å	$c/\text{\AA}$	$R_{\rm exp}$ /%	<i>R</i> _{wp} /%	<i>R</i> _p /%	Tilting angle, $\alpha/(^{\circ})$
$\chi = 0$	5.515 06(4)	5.513 45(4)	25.098 50(4)	3.57	8.61	6.61	9.72
$\chi = 0.2$	5.510 60(7)	5.510 59(7)	25.084 53(4)	3.97	7.94	5.91	9.45
$\chi = 0.4$	5.509 40(1)	5.509 50(1)	25.107 14(5)	3.36	6.99	5.28	5.48

reasonable structure parameters obtained by Rietveld refinement reflect that Nd^{3+} replaces Bi^{3+} for $SrBi_{2-\chi}Nd_{\chi}Nb_{2}O_{9}$ samples.

Table 2 lists the lattice constant of $\text{SrBi}_{2-\chi}\text{Nd}_{\chi}\text{Nb}_2\text{O}_9$ (χ =0, 0.2 and 0.4) samples and their titling angle for NbO₆ octahedron, which was obtained by the Rietveld refinement. Compared with those of SBN (i.e, χ =0), the lattice constants *a* and *b* decrease by 0.1% and 0.07%, respectively, and the lattice constant *c* increases by 0.03%. Thus, the structural change mainly occurs in *a*-*b* plane when Nd³⁺ partially substitutes for Bi³⁺ in the Bi₂O₂ layer for Nd-doped SBN samples. It should be noted that the titling angle for NbO₆ octahedron decreases from 9.72° for χ =0 to 9.45° for χ =0.2 and 5.48° for χ =0.4 sample with the increase in the Nd content for Nd-doped SBN samples. The decrease in the titling angle is related with the 6s² lone-pair electrons of Bi ions in Bi₂O₂ layers[8].

In order to further determine whether the doped Nd³⁺ indeed substitutes for Bi³⁺ in Bi₂O₂ layers or not, synchrotron radiation XAFS technique is used to probe the local environment of doped Nd³⁺ in SBN ceramics. Fig.2 shows the extended-XAFS spectra of Nd-L₃ absorption edge in SrBi_{2- χ}Nd_{χ}Nb₂O₉ (χ =0.2 and 0.4) samples. Since Nd-L₃ absorption edge lies in the 6 208 eV position and Nd-L₂ absorption edge is close to Nd-L₃, the range of selected energy varies from 6 100 to 6 700 eV. As illustrated in Fig.2, some vibration peaks appear near the high energy side of Nd-L₃ edge, which just is attributed to the extended X-ray absorption fine structure. In addition, It can be seen from Fig.2 that the energy position of Nd-L₃ absorption edge is the same and three little vibration peaks behind Nd-L3 edge have the similar shape for $\chi=0.2$ and $\chi=0.4$ samples. These observations reflect that the Nd atoms in $\gamma=0.2$ and $\gamma=0.4$ samples have the same electrovalent structure and the similar local coordination structure[13-14]. Fig.3 gives Fourier transform magnitudes of the Nd-L3 edge extended-XAFS spectrum of SrBi_{2- χ}Nd_{χ}Nb₂O₉ (χ =0.2 and 0.4) samples. The strongest peak in Fig.3 represents the distance between Nd atom and the nearest coordination atom O (i.e. the first coordination shell peak). It is obvious that the first coordination shell peak of $\chi = 0.2$ sample (near 2.2 Å) is the same as that of $\chi = 0.4$ sample and both the second coordination shell peaks of $\chi = 0.2$ and 0.4 samples appear at about 3.4 Å, which further indicates that the local structures around Nd atoms in $\chi=0.2$ and χ =0.4 samples are very similar. Furthermore, it was ever reported by WACHSMUTH et al[15] that the first and second coordination shell peaks for the Bi atom of Ba_{0.6}Sr_{0.4}Bi₂Nb₂O₉ were near 2.20 and 3.61 Å using synchrotron radiation extended-XAFS technology. More importantly, the configurations of the first and second coordination shell peaks for Nd-L₃ edge of $SrBi_{2-\chi}Nd_{\chi}Nb_{2}O_{9}$ ($\chi = 0.2$ and 0.4) samples are very similar to those of Bi-L₃ edge of Ba_{0.6}Sr_{0.4}Bi₂Nb₂O₉. These results above further indicate that Nd³⁺ really occupies Bi-site in Bi2O2 layers for SrBi2-yNdyNb2O9 samples. Therefore, the combination of X-ray Rietveld refinement and synchrotron radiation extended-XAFS

technology strongly confirm that the doped Nd^{3+} successfully substitutes for Bi^{3+} in $SrBi_{2-\chi}Nd_{\chi}Nb_{2}O_{9}$ samples.



Fig.2 Extended-XAFS spectra of Nd-L₃ edge in $SrBi_{2-\gamma}Nd_{\gamma}Nb_{2}O_{9}$ ($\gamma = 0.2$ and 0.4) samples



Fig.3 Fourier transform magnitudes of Nd-L₃ edge extended-XAFS spectrum of $\text{SrBi}_{2-\chi}\text{Nd}_{\chi}\text{Nb}_2\text{O}_9(\chi=0.2 \text{ and } 0.4)$ samples (*R*: interatomic distance between Nd atoms and other atoms)

3.2 Ferroelectric and piezoelectric properties

Fig.4 shows the P - E hysteresis loops of SrBi_{2- χ}Nd_{χ}Nb₂O₉ (χ =0, 0.1, 0.2 and 0.4) ceramics measured at 150 °C. The P-E hysteresis loops of all samples approach saturation under the strong external electric field. With the increase in Nd content, both the coercive field ($2E_c$) and the remanent polarization ($2P_r$) gradually decrease. For instance, $2E_c$ decreases from 91.31 kV/cm for χ =0 to 70 kV/cm for χ =0.2 and $2P_r$ also decreases from 15.6 μ C/cm² for χ =0 to 11.90 μ C/cm² for χ =0.2. It may be related to the decrease in NbO₆ structural distortion due to the increase in Nd content for SrBi_{2- χ}Nd_{χ}Nb₂O₉ (χ =0, 0.1, 0.2) ceramics. As for χ =0.4 sample, its P-E hysteresis loop is not characteristic of the ferroelectric hysteresis loop because of its low transition temperature (T_m =75 °C) and it is observed that

both $2P_r$ and $2E_c$ of χ =0.4 sample approach zero. These results indicate that the partial substitution of Nd³⁺ for Bi³⁺ leads to the deterioration of the ferroelectric properties of Nd-doped SBN ceramics.



Fig.4 Hysteresis loops of $\text{SrBi}_{2-\chi}\text{Nd}_{\chi}\text{Nb}_2\text{O}_9$ ($\chi=0, 0.1, 0.2$ and 0.4) samples

Table 3 shows the piezoelectric characteristics of $SrBi_{2-\gamma}Nd_{\gamma}Nb_{2}O_{9}$ ($\gamma=0, 0.1$ and 0.2) ceramics. As illustrated in Table 3, Nd-doped SBN ceramics have low dielectric constant $\varepsilon_{\rm r}$, low piezoelectric constant d_{33} , low electromechanical coupling coefficients K_p and high mechanical quality factor Q_m compared with lead zirconate titanate (PZT) ceramics. Furthermore, it should be noted that d_{33} decreases from 11 pC/N for $\chi=0$ to 6 pC/N for χ =0.2 when Nd content (χ) increases gradually in Nd-doped SBN ceramics. This result is consistent with the decrease in $2P_r$ due to the substitution of Nd³⁺ for Bi³⁺ in Bi₂O₂ layers. SBN is expected as the candidate for the fine-tolerance resonator application because it has low ε_r , low K_p , high Q_m and low TCF value[16]. However, the TCF value of SBN is not low enough to satisfy the requirement of fine-tolerance resonator application. Thus, the doping-element method was used to decrease the TCF value. For example, there were some research papers which reported that the substitution of Ba^{2+} for Sr^{2+} induced the dielectric relaxor behavior and deceased TCF values for (Sr_{1-y}Ba_y)Bi₂Nb₂O₉ ceramics [16–17]. Considering that the substitution of Nd^{3+} for Bi³⁺ also induces the dielectric relaxor behavior for SrBi_{2-x}Nd_xNb₂O₉ ceramics[7], it is naturally expected this substitution can lead to lower TCF values. Fig.5 gives the temperature dependence of resonance frequency of $SrBi_{2-\chi}Nd_{\chi}Nb_{2}O_{9}$ ($\chi=0, 0.1$ and 0.2) ceramics in the range from −20 to 140 °C. It can be apparently seen from Fig.5 that TCF values of $\chi=0.1$ and 0.2 samples are lower than that of $\chi=0$ sample. In other words, the substitution of Nd³⁺ for Bi³⁺ decreases the TCF values of SBN ceramics. The reason for it is possibly related to the dielectric relaxor behavior of Nd-doped SBN ceramics[17].

Table 3 Piezoelectric characteristics of $SrBi_{2-\chi}Nd_{\chi}Nb_{2}O_{9}$ ($\chi=0$, 0.1 and 0.2) ceramics

SrBi _{2-x} Nd _x NbO ₉ sample	ε _r (at 1 kHz, RT)	$\frac{d_{33}}{(\text{pC}\cdot\text{N}^{-1})}$	<i>K</i> _p /%	$Q_{\rm m}$
χ=0	145	11	4.5	3 250
<i>χ</i> =0.1	152	7	5	3 960
χ=0.2	155	6	4	3 390



Fig.5 Temperature dependence of resonance frequency of $SrBi_{2-\gamma}Nd_{\gamma}Nb_{2}O_{9}$ ($\chi=0, 0.1$ and 0.2) ceramics

4 Conclusions

1) SrBi_{2-x}Nd_xNb₂O₉ (χ =0, 0.1, 0.2 and 0.4) ceramics were synthesized by the conventional solid-state reaction sintering method. The substitution of Nd³⁺ for Bi³⁺ in the Bi₂O₂ layers for SrBi_{2-x}Nd_xNb₂O₉ ceramics were determined by the combination of X-ray Rietveld refinement and synchrotron radiation XAFS technique. X-ray Rietveld refinement also indicates that the substitution of Nd³⁺ for Bi³⁺ leads to the decrease in the structural distortion of NbO₆ octahedron, which is described by title angle.

2) The substitution of Nd³⁺ for Bi³⁺ decreases the remnant polarization and piezoelectric constant, whereas it decreases the temperature coefficient of resonance frequency values of SBN ceramics. $SrBi_{2-\chi}Nd_{\chi}Nb_{2}O_{9}$ ceramics can be regarded as one of candidates for fine-tolerance resonator applications.

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