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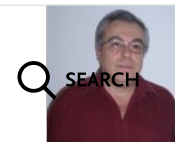
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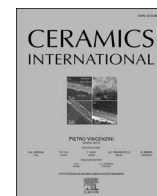
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Structural transformation in Mn-substituted $\text{Sr}_2\text{Bi}_2\text{Ta}_2\text{TiO}_{12}$ Aurivillius phase synthesized by hydrothermal method: A comparative study and dielectric properties

Ilona Bella^a, Tio Putra Wendari^a, Novesar Jamarun^a, Nandang Mufti^b, Zulhadjri^{a,*}

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ABSTRACT

In this study, a hydrothermal method was applied to synthesize the three-layer Aurivillius phase $\text{Sr}_2\text{Bi}_2\text{Ta}_2\text{TiO}_{12}$ (SBTTO) and Mn-substituted $\text{Sr}_{1.5}\text{Bi}_{2.5}\text{Ta}_2\text{Ti}_{0.5}\text{Mn}_{0.5}\text{O}_{12}$ (SBTMO), with the use of NaOH as a mineralizer. The crystal structure, morphology, dielectric properties, and the correlation between the structural transformation and dielectric properties were investigated. The XRD data reveal that the SBTTO sample adopts a tetragonal crystal structure with the $I4/mmm$ space group and is then transformed into an orthorhombic structure with the $B2cb$ space group for SBTMO. The morphology of both samples was observed by SEM, which showed anisotropic plate-like grains. With the Mn substitution, the ferroelectric transition temperature (T_c) significantly increases as the influence of the $6s^2$ lone pair of Bi^{3+} increases, and this in turn further induces the relaxor-ferroelectric behavior. Consequently, the increase in T_c confirms the structural transformation from the paraelectric-tetragonal to the ferroelectric-orthorhombic phase.

1. Introductions

Ferroelectric material has the ability to change the value of electric polarization but remain in a switched state, even when the field is removed. These ferroelectric oxide materials are widely applied in electronic devices such as data storage (Fe-RAMs), energy storage, sensors, actuators, transducers and capacitors [1–4]. Among them, Aurivillius phases often exhibit excellent ferroelectric properties in nature, attributable to their structural properties. The Aurivillius phase represents the bismuth layered oxides with the general formula $[\text{Bi}_2\text{O}_2]^{2+}[\text{A}_{m-1}\text{B}_m\text{O}_{3m+1}]^{2-}$. The structure is constructed from perovskite-like layers $[\text{A}_{m-1}\text{B}_m\text{O}_{3m+1}]^{2-}$ and bismuth oxide layers $[\text{Bi}_2\text{O}_2]^{2+}$, stacked along the crystallographic c -axis, where m denotes the number of perovskite layers. The A -site cation in the perovskite-layers is occupied by mono-, di-, or trivalent cations (Na^+ , K^+ , Ca^{2+} , Sr^{2+} , Ba^{2+} , etc.) and the B -site cation is the transition metal with a high valency (Ti^{4+} , Nb^{5+} , Ta^{5+} , etc.) [5]. The crystal structure of the three-layer Aurivillius phase is depicted in Fig. 1a [6,7]. The perovskite-layer structure can accommodate a wide range of A - and B -site cation substitutions, allowing for control of the structure as well as of its physical properties. The Bi_2O_2 insulator layers and the distorted

BO_6 octahedra in the perovskite layers play an essential role in terms of the ferroelectricity in the Aurivillius phases [8].

The majority of studies concentrated on the three-layer Aurivillius phases are focused on $\text{Bi}_4\text{Ti}_3\text{O}_{12}$, which adopts a non-centrosymmetric crystal structure with the $B2cb$ space group, and exhibits a high dielectric constant with a high ferroelectric transition temperature (T_c) of 670 °C [9]. This pronounced ferroelectricity in $\text{Bi}_4\text{Ti}_3\text{O}_{12}$ is attributable to the effect of $6s^2$ lone pair electrons of Bi-rich content in the perovskite layers, which induce a highly distorted octahedral structure, as seen in Fig. 1b [10]. However, another member of the three-layer Aurivillius family with the general formula of $\text{A}_2\text{Bi}_2\text{B}_2\text{TiO}_{12}$ ($A = \text{Ca}^{2+}$, Ba^{2+} , Sr^{2+} ; $B = \text{Nb}^{5+}$, Ta^{5+}) is reported to be paraelectric in nature since it possesses the $I4/mmm$ tetragonal crystal structure (see Fig. 1) [11]. To date, research focused on the $\text{Sr}_2\text{Bi}_2\text{Ta}_2\text{TiO}_{12}$ compound is lacking.

In order to improve the ferroelectricity of the Aurivillius phase, the substitution of the smaller A -site cations with a d^0 shell is a feasible way to induce the distortion of BO_6 octahedra [12]. On the other hand, the substitution of B -site cations with the first-row transition cations with a half-filled d -shell (d^n) is well-known to promote magnetization and in turn exhibits the needed multiferroic properties [13,14]. Besides, the

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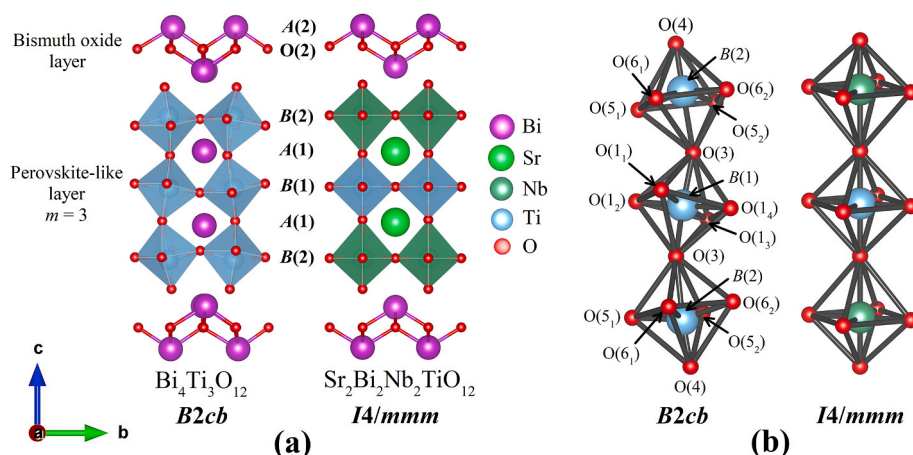


Fig. 1. (a) Crystal structure models of three-layer Aurivillius phases with $B2cb$ and $I4/mmm$ space group viewed along the ac -plane. (b) View of linked BO_6 octahedra projected along the c -axis. Atomic coordinates were taken from Refs. 6 and 7.

substitution of B -site cations with various ionic radii potentially enhances the structural distortion in BO_6 octahedra, thus improving its ferroelectricity [15,16]. The substitution of the Mn^{3+} cation (d^4) for the higher valence state cation (Nb^{5+} or Ti^{4+}) in the B -site cation was reported to improve the dielectric properties since it is compensated by the increase in Bi^{3+} contents to maintain the charge neutrality [8,17]. Therefore, the substitution of Mn^{3+} for Ti^{4+} in the $Sr_2Bi_2Ta_2TiO_{12}$ is expected to improve the ferroelectricity and raise the magnetization, which is especially useful for data storage applications.

The synthesis of the multiferroic Aurivillius phase is challenging since the different characters of d^0 and d^n ions with the various ionic radii possibly destroy the structure when partial substitutions are performed [18]. A conventional solid-state method is the most popular method applied in the preparation of the Aurivillius phase. However, the use of high-temperature sintering often results in the volatilization of Bi^{3+} and oxidation of the Mn^{3+} cation, leading to the formation of the impurity phase [13,19]. Therefore, the synthesis using a liquid-phase reaction medium such as the hydrothermal method likely favors the synthesis of the multiferroic Aurivillius phase at a low temperature. Hydrothermal synthesis involves the use of solvents with a temperature and pressure above the boiling point [4]. The oxide precursors increase the solubility of the solid and in turn accelerate the reaction rate. The use of hydrothermal synthesis can have many advantages such as high-pressure synthesis, lower-temperature synthesis, faster ionic diffusion, and well-controlled crystal growth and morphology [20]. Moreover, it is well-known that the compositional homogeneity and morphology significantly impact the physical properties [21,22].

In this work, the hydrothermal method was employed to report the synthesis of the Mn-substituted $Sr_2Bi_2Ta_2TiO_{12}$ phases with the chemical formula $Sr_{2-x}Bi_{2+x}Ta_2Ti_{1-x}Mn_xO_{12}$ ($x = 0$ and 0.5). The substitution of Mn^{3+} for Ti^{4+} was also compensated by the substitution of Bi^{3+} for Sr^{2+} to maintain overall charge neutrality. Therefore, we synthesized compounds of composition $Sr_2Bi_2Ta_2TiO_{12}$ and $Sr_{1.5}Bi_{2.5}Ta_2Ti_{0.5}Mn_{0.5}O_{12}$, abbreviated as SBTTO and SBTMO, respectively. We investigate the relationships between the structure and its dielectric properties. The structure, morphology, and dielectric properties of both compounds were all considered.

1.1. Experimental procedures

The samples of $Sr_2Bi_2Ta_2TiO_{12}$ and $Sr_{1.5}Bi_{2.5}Ta_2Ti_{0.5}Mn_{0.5}O_{12}$ were prepared by the hydrothermal method, with $Sr(NO_3)_2$ (Merck, 99.9%), Bi_2O_3 , TiO_2 , Ta_2O_5 , Mn_2O_3 (Aldrich, $\geq 99.9\%$) as precursors. Stoichiometric amounts of precursors were weighed and added to 60 mL of NaOH 3 M. The solution was stirred and then transferred into a 100 mL Teflon vessel before being placed into a stainless-steel autoclave. The

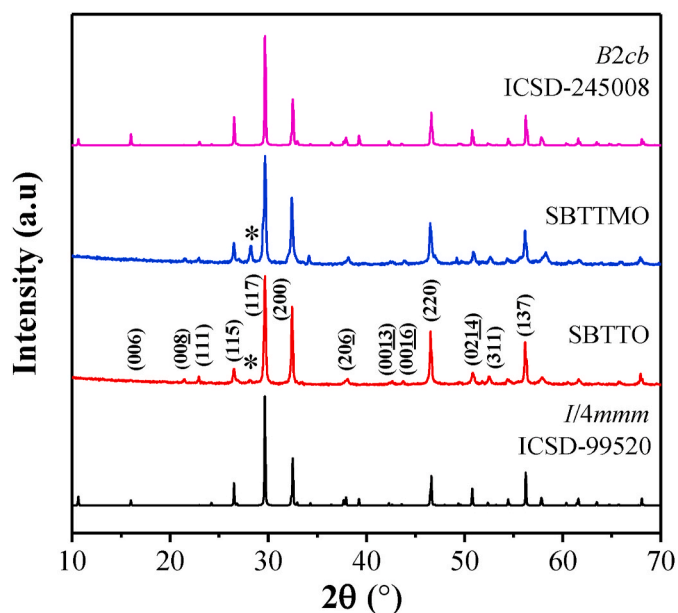


Fig. 2. X-ray diffraction patterns of SBTTO and SBTMO samples indexed according to the standard three-layer Aurivillius phase with $I4/mmm$ and $B2cb$ space groups. Both samples exhibit a peak from an unidentified impurity phase.

samples were heated at a temperature of 240 °C for 120 h. The precipitates were filtered and then washed with deionized water to remove the base residue until pH 7 was recorded. The powder products were heated at 110 °C for 6 h, and calcined at 550 °C for 5 h and 900 °C for 4 h. XRD analysis was performed to investigate the formation of phase oxide and structural evolution using an X'Pert3 Powder PANalytical with Cu $K\alpha$ radiation. The structures of the unit cells were determined by the Le Bail refinement technique, using the RIETICA program [23]. FTIR spectroscopy was carried out using a PerkinElmer 1600 FTIR spectrophotometer at room temperature. The surface morphology of the products was characterized by scanning electron microscopy (SEM; FEI INSPECT S50). The powders were then pressed into pellets and heated at 900 °C for 5 h to form a ceramic. The ceramic pellets were coated with silver paste (Aldrich, 99%) as electrodes. The temperature and frequency dependence of the dielectric properties were measured by using an LCR meter (BK Precision 891) with an amplitude of 1 V.

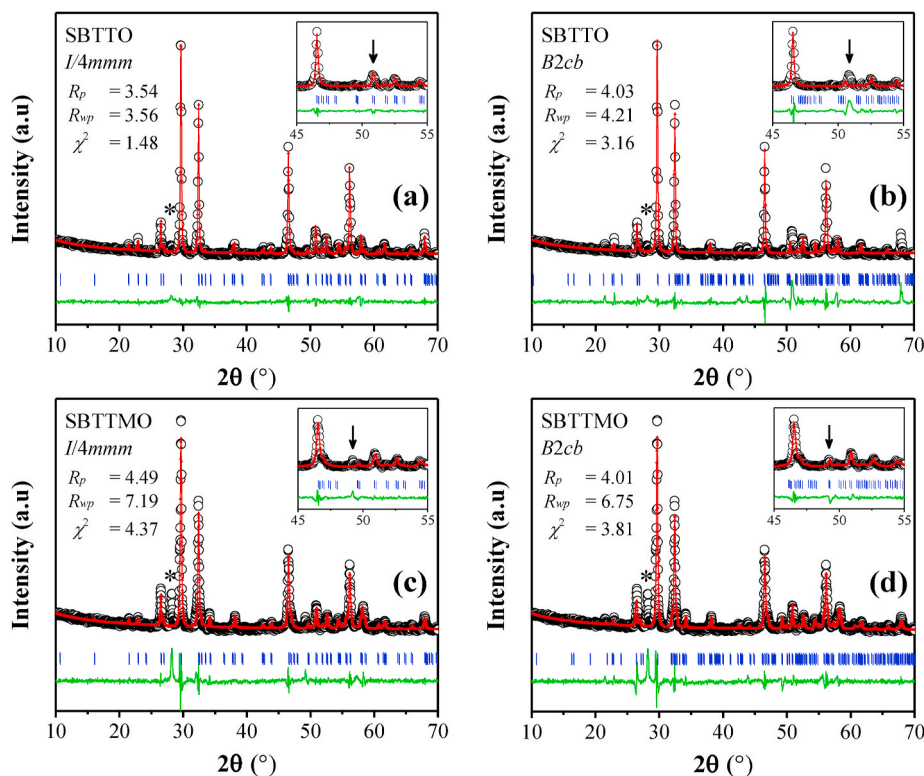


Fig. 3. Le Bail refinement fits of Aurivillius (a–b) SBTTO and (c–d) SBTtMO samples using the $I4/mmm$ and $B2cb$ space groups: experimental data (black circles), calculated data (red line), and difference data (green line). The blue tick marks indicate the positions of allowed Bragg reflections in each space group. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

2. Results and discussion

Fig. 2 depicts the X-ray diffraction patterns of Aurivillius SBTTO and SBTtMO samples synthesized using the hydrothermal method. The XRD patterns are compared to the standard diffraction data of the three-layer Aurivillius phase with the $I4/mmm$ (ICSD-99520) and $B2cb$ space groups (ICSD-245008). Since we investigate the effect of composition on the structure, we compare the samples using both space groups, which is commonly reported for the three-layer Aurivillius phase at room temperature [24]. This shows that XRD patterns match with the standard data, confirming the formation of the three-layer Aurivillius phase, while an unidentified impurity can be found at $2\theta = 28.17^\circ$ for SBTTO and $2\theta = 29.23^\circ$ for SBTtMO. The most intense diffraction peak (1 1 5) for all samples also reflects the formation of the three-layer Aurivillius phase ($m = 3$). This is in agreement with the fact that the most intense reflection is (1 1 $\underline{2m+1}$) [25]. It was difficult to observe a difference in the peak to indicate the structural transformation.

To investigate the structure of the phase, the Le Bail refinement of the XRD data was performed with the RIETICA program to determine the structural phase and the lattice parameters [23]. The initial parameters for refinement were taken from the space group $I4/mmm$ ($a = b = 3.8925 \text{ \AA}$, $c = 33.1876 \text{ \AA}$) (ICSD-99520) and $B2cb$ ($a = 5.5051 \text{ \AA}$, $b = 5.5057 \text{ \AA}$, $c = 33.1947 \text{ \AA}$) (ICSD-245008). The parameters were automatically refined to obtain the optimal Le Bail profile and the value of the reliability factors (R_p , R_{wp} , χ^2), which are critical parameters for calculation accuracy, as shown in Fig. 3. In Fig. 3a, the XRD peaks of SBTTO were fitted with the $I4/mmm$ space group. However, for the $B2cb$ space group, the peak at $2\theta = 50.8^\circ$ was not fitted, as shown in Fig. 3b. Moreover, the reliability factors also indicate the optimal value for the $I4/mmm$ space group. These results suggest that the SBTTO sample has a tetragonal structure.

Subsequently, we also refined the overall structure of SBTtMO using both space groups, as depicted in Fig. 3c–d. The refinement results

Table 1

Refined lattice parameters of the Aurivillius SBTTO and SBTtMO samples.

	SBTTO	SBTtMO
Space group	$I4/mmm$	$B2cb$
Crystal class	Tetragonal	Orthorhombic
a (Å)	3.8990(6)	5.5198(1)
b (Å)	3.8990(6)	5.5016(6)
c (Å)	33.1165(8)	33.1180(3)
V (Å ³)	503.461(8)	1005.735(7)
$(a-b)/(a+b)$	0	0.00165
Z	2	4
R_p	3.548	4.013
R_{wp}	3.560	6.715
χ^2	1.485	3.807

reveal that the good fits of SBTtMO were obtained for the $B2cb$ space group. Meanwhile, for the $I4/mmm$ space group, the peak at $2\theta = 49.1^\circ$ could not be indexed by Bragg reflections. The reliability factors of the $B2cb$ space group are also observed to be smaller than $I4/mmm$, which suggests the orthorhombic $B2cb$ structure to be adopted in the SBTtMO sample at room temperature. The XRD data imply that the compositional-induced structural transformation is from tetragonal to orthorhombic in SBTtMO.

It was reported that the lone-pair effect of $6s^2$ -electrons of Bi^{3+} ions in the perovskite layers is essential for the highly distorted BO_6 structure in the Aurivillius phases [8]. According to the nominal formula of $\text{Sr}_{2-x}\text{Bi}_{2+x}\text{Ta}_2\text{Ti}_{1-x}\text{Mn}_x\text{O}_{12}$, the substitution of Mn^{3+} for Ti^{4+} in SBTtMO increases the proportion of Bi^{3+} on the A-site, leading to a higher degree of structural distortion such as rotations and tilts of BO_6 octahedra, as demonstrated in Fig. 1b [26]. This distortion results in the break of inversion of the parent tetragonal structure, consequently giving rise to an orthorhombic structure [27].

Table 1 shows the refined lattice parameters obtained from the XRD

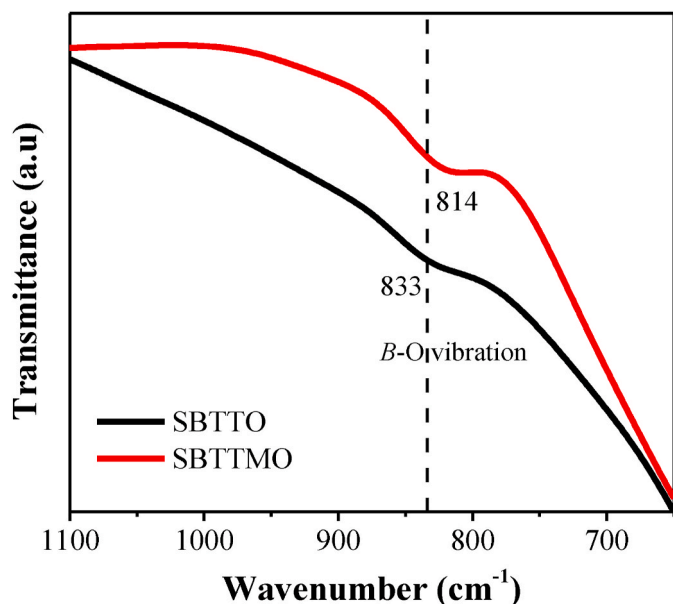


Fig. 4. FTIR spectra of Aurivillius SBTTO and SBTtMO samples at room temperature.

fit based on each space group. An increase in the degree of orthorhombic distortion in the SBTtMO sample leads to the difference between the a and b lattice parameters. This can also be expressed in terms of the orthorhombicity ratio $(a-b)/(a+b)$ in Table 1. In the Aurivillius phases with $B2cb$ symmetry, the atomic displacement from the center of position in the b -axis gives rise to ferroelectricity [24]. This structural transformation can also be expected to increase the ferroelectric properties of the SBTtMO sample, as discussed hereafter.

Fig. 4 shows the IR spectra of the SBTTO and SBTtMO samples at room temperature from 650 to 1100 cm^{-1} . Both samples exhibit a phonon mode of $\sim 833 \text{ cm}^{-1}$, ascribed to the B -O symmetric stretching mode of BO_6 octahedra [8]. The vibration mode of 833 cm^{-1} in SBTTO shifted toward the lower wavenumber of 814 cm^{-1} for SBTtMO. This is

in agreement with Hooke's law since the bond strength of Mn-O (362 kJ/mol) is lower than that of Ti-O (666.5 kJ/mol) [8,28]. This result reveals that the Mn^{3+} cations incorporate into the BO_6 octahedra layers.

The grain morphologies of the SBTTO and SBTtMO samples observed using SEM are shown in Fig. 5a–b. The grain morphology is anisotropic and plate-like in nature and spreads evenly across all samples, which is a typical grain growth for the Aurivillius phases. It was found that the uniform grain size distribution can be obtained when using the hydrothermal method, unlike the high-temperature method [22,29]. The particle size distribution of SBTTO and SBTtMO was determined by ImageJ software, which is shown in Fig. 5c–d. The particle size of SBTTO is in the range of $0.21\text{--}1.28 \mu\text{m}$, and is reduced in size with Mn^{3+} substitution to the range of $0.12\text{--}0.8 \mu\text{m}$ for SBTtMO. Moreover, the particle agglomeration is decreased and the particle shape becomes more uniform for the samples containing Mn^{3+} [18].

Fig. 6 depicts the temperature dependence of the dielectric constant (ϵ) and dielectric loss ($\tan \delta$) at a frequency range of 50 kHz–300 kHz. The dielectric properties of the samples were investigated for high frequencies, since this best reflects the intrinsic polarizability being correlated to the structural properties of the sample [17]. The magnitude of ϵ for SBTTO initially decreases with an increase in temperature, and then significantly increases above 350°C , indicating that the sample becomes more conductive. This conductive behavior is also observed by the significant increase in the dielectric loss at this temperature. There is no ferroelectric-paraelectric transition peak (T_c) observed at this measured temperature, and the dielectric constant increases as the temperature decreases, suggesting that T_c is below room temperature. The T_c below RT was also previously observed in $\text{Sr}_2\text{Bi}_2\text{TiNb}_2\text{O}_{12}$, where the T_c of -136°C is relevant [11]. This result confirms that the SBTTO exhibits paraelectric behavior in nature, which is in agreement with the centrosymmetric tetragonal structure of this sample.

For SBTtMO, the dielectric constant exhibits the single broad peak shown in Fig. 6b, corresponding to a phase transition from the ferroelectric to the paraelectric phase (T_c). The T_c peak of the SBTtMO sample indicates the predominance of the ferroelectric phase, according to a non-centrosymmetric orthorhombic structure with the $B2cb$ space group. Furthermore, the T_c peak is between 300°C and 350°C , strongly depending on frequency (ΔT relaxation); this can be attributed to the contribution of the relaxor-ferroelectric behavior [30]. The dielectric

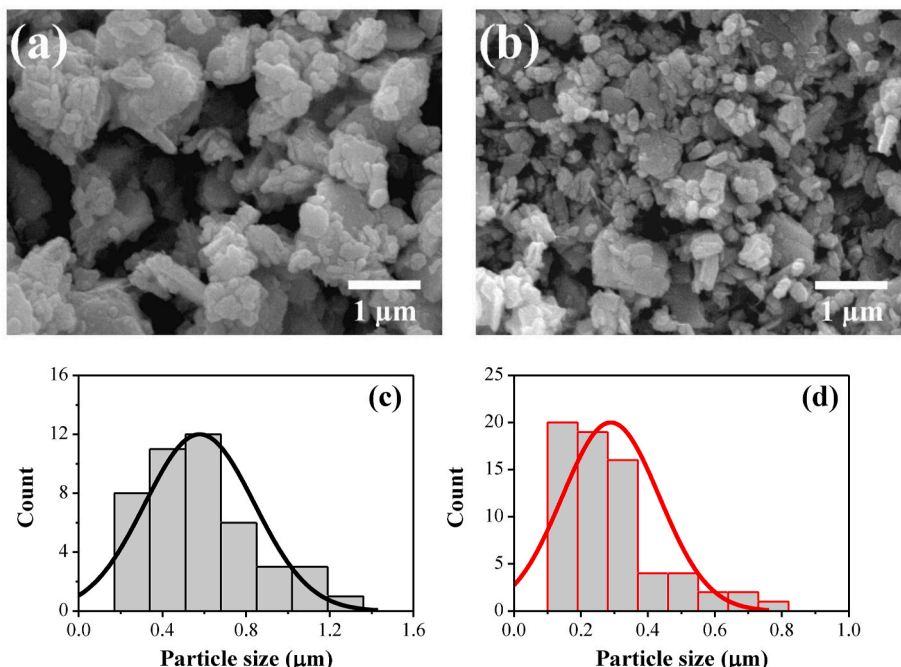


Fig. 5. SEM micrographs of Aurivillius (a) SBTTO and (b) SBTtMO samples. Particle size distribution of Aurivillius (c) SBTTO and (d) SBTtMO samples.

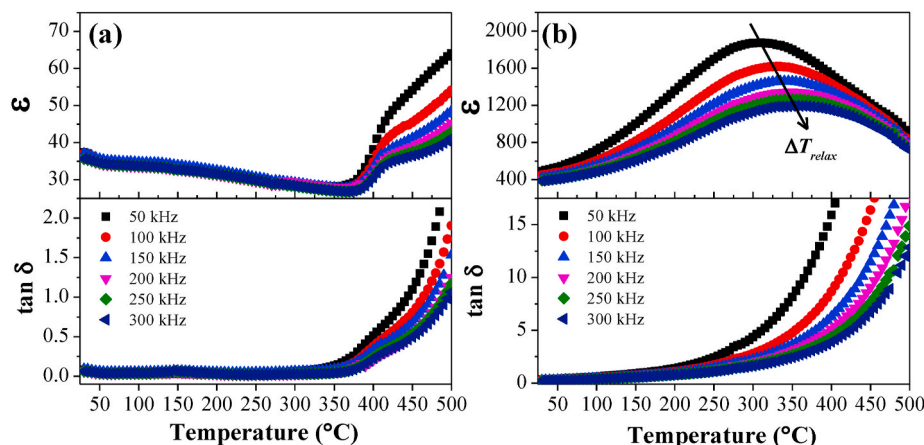


Fig. 6. Dielectric constant (ϵ) and dielectric loss ($\tan \delta$) of Aurivillius (a) SBTTO and (b) SBTTMO samples as a function of temperature at various frequencies.

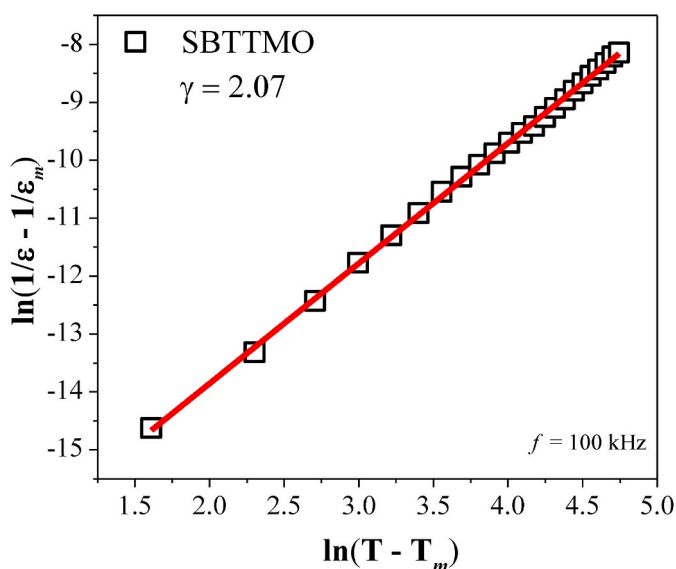


Fig. 7. A modified Curie-Weiss fitted line to quantify the degree of relaxor behavior for the SBTTMO sample.

loss increases with temperature, indicating the increased conductivity by thermal effects. Furthermore, the relaxor behavior in the SBTTMO sample was evaluated by the degree of diffuseness (γ) using a modified Curie-Weiss law [11]:

$$1/\epsilon_r - 1/\epsilon_m = (T - T_m)^\gamma / C$$

The γ value was obtained from the linear fitting to plots of $\ln(1/\epsilon_r - 1/\epsilon_m)$ versus $\ln(T - T_m)$, as shown in Fig. 7. This value is in the range of 1–2, where $\gamma = 1$ represents a normal ferroelectric behavior, while $\gamma = 2$ represents a relaxor-ferroelectrics behavior. The fitted value of γ is 2.07, which exhibits the pronounced relaxor behavior in SBTTMO with $\Delta(T_m(300 \text{ kHz}) - T_m(50 \text{ kHz}))$ of 50 °C. This is strongly induced by the increased compositional disorder of both A-site cation ($\text{Sr}^{2+}/\text{Bi}^{3+}$) and B-site cations ($\text{Ta}^{5+}/\text{Ti}^{4+}/\text{Mn}^{3+}$) [8,30]. This cation disorder causes the breaking of the long-range ferroelectric order into the polar nanoregion domain,

and is thus reckoned to have a relaxor ferroelectric behavior.

Compared to the SBTTO, both the T_c and the magnitude of ϵ at 100 kHz significantly increase for SBTTMO, as shown in Table 2. The increased dielectric properties are attributed to an increase in the structural distortion, as discussed in the refinement results. This increase in the dielectric properties is also consistent with the lower Goldschmidt tolerance factor (t) of the perovskite layers of SBTTMO (0.950), compared to SBTTO (0.991), which is also related to the increase in T_c [31]. Therefore, the significant increase in T_c reveals the structural transformation from the paraelectric-tetragonal to the ferroelectric-orthorhombic phase ($I4/mmm \rightarrow B2cb$), which is in agreement with the XRD study. It has been established that a non-centrosymmetric crystal structure gives rise to an electrical dipole moment, which may favor an increase of T_c . It is also found that the dielectric loss at room temperature significantly increases since the introduction of Mn^{3+} cation with unpaired electrons (d^4) in SBTTMO increases the charge transport [14].

3. Conclusion

The three-layer Aurivillius $\text{Sr}_2\text{Bi}_2\text{Ta}_2\text{TiO}_{12}$ (SBTTO) and $\text{Sr}_{1.5}\text{Bi}_{2.5}\text{Ta}_2\text{Ti}_{0.5}\text{Mn}_{0.5}\text{O}_{12}$ (SBTTMO) were synthesized by the hydrothermal method using NaOH 3 M as a mineralizer. The XRD data indicate the formation of the three-layer Aurivillius phase with an unidentified impurity phase in both samples. The Le Bail refinement from the XRD data confirms a structure transformation from tetragonal symmetry ($I4/mmm$) to an orthorhombic symmetry ($B2cb$), with the substitution of Mn^{3+} for Ti^{4+} . An anisotropic plate-like grain morphology with agglomeration was observed across all samples. The ferroelectric transition temperature (T_c) is below room temperature for SBTTO but significantly increases to 335 °C for SBTTMO at 100 kHz; this is attributed to the increase in the structural distortion and a non-centrosymmetric crystal structure. Furthermore, the SBTTMO sample exhibits a pronounced relaxor-ferroelectric behavior driven by the increased disorder of the A-site cation ($\text{Sr}^{2+}/\text{Bi}^{3+}$) in the perovskite layer, as well as the B-site cations ($\text{Ta}^{5+}/\text{Ti}^{4+}/\text{Mn}^{3+}$).

Declaration of competing interest

The authors declare that they have no known competing financial

Table 2

Dielectric properties of the Aurivillius SBTTO and SBTTMO samples measured at 100 kHz.

Sample	ϵ_{RT}	$\tan \delta$ (RT)	T_m (°C)	ϵ_m	$\tan \delta$ (T_m)	Tolerance factor (t)	γ	ΔT_{relax} (°C)
SBTTO	37.03	0.072	< RT	–	–	0.991	–	–
SBTTMO	447.69	0.291	335	1618.22	4.027	0.950	2.07	50

interests or personal relationships that could have appeared to influence the work reported in this paper.

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