



Effects of Mn substitution on ferro- and piezoelectric properties of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ thin films

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ABSTRACT

The $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ (BSFO) and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ (BSFMO) ($x=0.01, 0.03, 0.05$) thin films were deposited on indium tin oxide/glass substrates via a metal organic deposition method. 1 at.% Mn doping leads to an evident reduction of the leakage current in BSFO film. More importantly, the $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.99}\text{Mn}_{0.01}\text{O}_3$ film exhibits the lowest coercive field ($E_c = 272$ kV/cm), the largest remanent polarization ($P_r = 53.6$ $\mu\text{C}/\text{cm}^2$) and the remanent out-of-plane piezoelectric coefficient ($d_{33} = 146$ pm/V). However, further increase of Mn doping content results in the deterioration of the charge retaining capability and the piezoelectric properties of the films. The negative influence of high Mn doping contents was discussed based on the structure change and the contribution of irreversible movement of non-180° domain walls in the aged films.

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1. Introduction

BiFeO_3 (BFO), as a promising lead-free candidate for the applications in data storage and piezoelectric sensors, has attracted enormous interest due to its superior ferroelectric and piezoelectric properties as well as the magnetoelectric coupling effect emerging at room temperature [1–6]. Recently, Fujino et al. have reported the discovery of a lead-free morphotropic phase boundary (MPB) between rhombohedral and pseudo-orthorhombic phases in Sm-doped BFO thin films [7]. More importantly, a large piezoelectric coefficient (~ 110 pm/V) was observed at the MPB composition, i.e., $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ (BSFO). They attributed the excellent piezoelectric properties of BSFO film to the presence of a low symmetry phase at the MPB. Thus, the polarization vector can be rotated readily within a suitable plane rather than be constrained to lie along a symmetry axis [8,9].

In our previous work, we found that high measuring frequency (10 kHz) was still required to obtain saturated P – E loops in Gd-doped BFO thin films especially for doping content higher than 10 at.% [10]. Similar phenomenon has been observed in the BFO thin films doped with La, Nd, Sm, Pr and Tb [11–16]. On the other hand, several groups have attempted to reduce the leakage current of BFO films by high-valence-ions substitution [17–19]. However, the frequencies for measuring the P – E loops still cannot be reduced to

below 10 kHz. The higher leakage current may result in the decrease of the effective electric field applied on the films and the domains are difficult to be fully switched, indicating that the coercive field (E_c), remnant polarization (P_r) and piezoelectric coefficient (d_{33}) measured in lanthanides-doped BFO films may not be intrinsic. Therefore, the leakage currents of lanthanides-doped BFO thin film should be further reduced from the basic research and application points of view.

It has been demonstrated that the insulating properties of the BFO films and lanthanides-doped BFO thin films can be improved by Mn substitution [20–24]. Most recently, we found that doping of Mn can damage the charge retaining capability of BFO films for doping contents higher than 3 at.%. This phenomenon was demonstrated to be related to the aging effects, which can be ascribed to the gradually stabilization of ferroelectric domains by the defect complexes formed between oxygen vacancies and low valence ions [25]. The piezoelectric properties will also be deteriorated due to the domain backswitching in the aged films. However, less attention has been paid on the negative influences of Mn doping on the long-term reliability and piezoelectric properties of both BFO and lanthanides-doped BFO thin films so far.

In this work, we investigated the effect of Mn substitution on the ferro- and piezoelectric properties of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ thin films prepared using metal organic decomposition. It was found that the Mn content of 1 at.% was enough to reduce the leakage current of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ thin film, which ensures that the intrinsic properties can be obtained. More importantly, $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.99}\text{Mn}_{0.01}\text{O}_3$ film exhibited the lowest E_c , the largest P_r and piezoelectric coefficient (d_{33}) among all films. Additionally, the negative influence of

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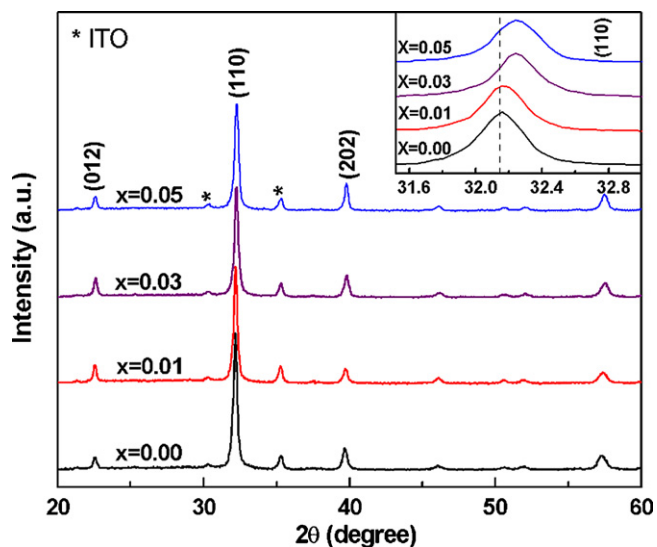


Fig. 1. X-ray diffraction (XRD) patterns of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.01, 0.03, 0.05$) thin films.

Mn doping content higher than 3 at.% on the ferro- and piezoelectric properties was demonstrated.

2. Experimental procedure

The $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ (BSFO) and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ (BSFMO) ($x = 0.01, 0.03, 0.05$) thin films were fabricated on ITO/glass substrates using a metal organic decomposition process. Similar to that reported in our previous work [10], the precursor solutions were prepared by dissolving bismuth nitrate, samarium nitrate, iron nitrate and manganese acetate in acetic acid and ethylene glycol. 2 at.% excess bismuth was added for repeatedly obtaining the results shown in this work. The films were deposited onto the substrates by spin coating and annealed layer by layer at 525°C for 1 min in N_2 following a pretreatment at 400°C . The film thickness is about 600 nm. Au top electrodes were deposited on the films using a sputtering system through a shadow mask with a diameter of $200\ \mu\text{m}$. We used X-ray diffractometer (D8, Bruker) to examine the structure of the films. The piezoelectric properties of the films were detected by an AFM working in piezoresponse mode. The detailed parameters adopted for piezoelectric measurements have been reported in our previous work [26]. A standard ferroelectric tester (Precision Pro, Radiant Technologies) was utilized to measure the leakage currents and ferroelectric properties.

3. Results and discussions

Fig. 1 shows the XRD patterns of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.01, 0.03, 0.05$) thin films deposited on ITO/glass substrates. It can be seen that all films exhibit phase-pure and polycrystalline structure although the sum of the doping content is on a high level (14–19 at.%). This can be ascribed to the uniformity of the precursor solution and oxide electrodes adopted, which can favor the nucleation and growth of BSFMO films. A small section of XRD pattern in the 2θ ranges of $31.5\text{--}33^\circ$ is also given to further clarify the effect of Mn doping on the structure of BSFO films (see the inset of Fig. 1). One can find that the diffraction peak of (110) shifts to the high angle of 2θ for BSFMO films with Mn doping contents of 3 and 5 at.%. On the other hand, such peak shift is not evident in $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.99}\text{Mn}_{0.01}\text{O}_3$ film, suggesting that 1 at.% Mn doping does not change the structure of BSFO film.

Fig. 2 shows the leakage currents for all films as a function of the electric field. It can be seen that, the leakage current of the $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ film with doping of 1 at.% Mn is lower than that of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ film by two orders of magnitude for the electric field higher than 100 kV/cm. However, further increase of Mn content from 1 at.% to 5 at.% results in the increase of the leakage current. Nevertheless, comparing to that of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ thin film, all the films doped with Mn exhibit lower leakage currents for

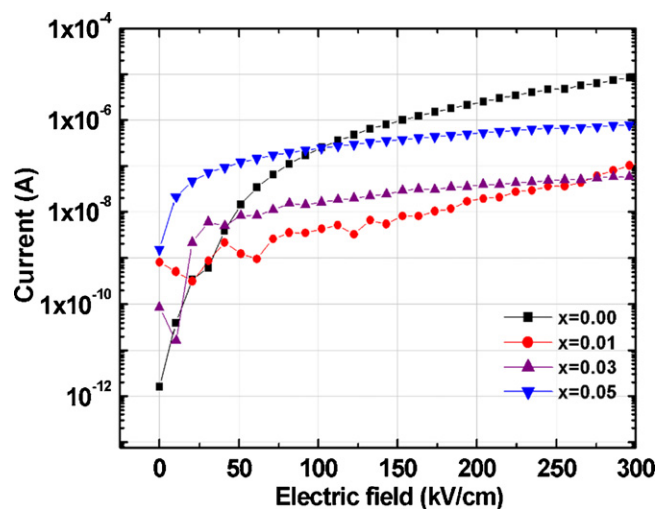


Fig. 2. Leakage currents as a function of electric field for $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.01, 0.03, 0.05$) thin films.

the electric fields higher than 100 kV/cm, which ensure that a lower frequency can be utilized in P - E loops measurements (see Fig. 3). This phenomenon should be related to the formation of defect complexes between $(\text{V}_{\text{O}^{2-}})^{\bullet\bullet}$ and Mn^{2+} . The electronic configuration of Mn is $[\text{Ar}]3d^54s^2$, which makes Mn apt to lose two electrons and reach a stable state with a half-filled 3d orbital. Furthermore, as N_2 atmosphere was adopted for annealing, the formation Mn^{2+} is more likely than Mn^{3+} . Thus, Mn^{2+} will inevitably cause the formation of oxygen vacancies for charge neutrality, i.e., the higher content of Mn doped, the more oxygen vacancies will be formed and more $(\text{Mn}_{\text{Fe}^{3+}}^{2+})' - (\text{V}_{\text{O}^{2-}})^{\bullet\bullet}$ will be produced. Notwithstanding $(\text{V}_{\text{O}^{2-}})^{\bullet\bullet}$ in the films can be restricted through the formation of $(\text{Mn}_{\text{Fe}^{3+}}^{2+})' - (\text{V}_{\text{O}^{2-}})^{\bullet\bullet}$, the released ones from $(\text{Mn}_{\text{Fe}^{3+}}^{2+})' - (\text{V}_{\text{O}^{2-}})^{\bullet\bullet}$ under the applied electric field still can act as donor-like trap centers for electrons. Obviously, the amount of the released $(\text{V}_{\text{O}^{2-}})^{\bullet\bullet}$, which is main charged defects responsible for the leakage current, will increase as the Mn doping increases. It is worth noting

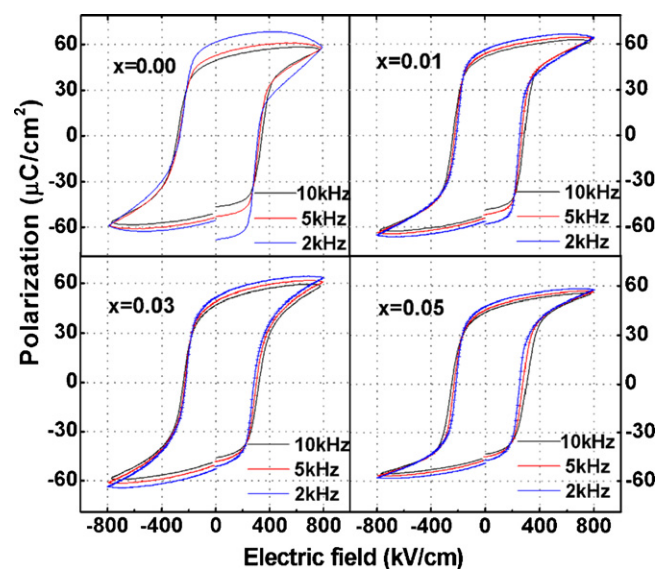


Fig. 3. P - E hysteresis loops of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.01, 0.03, 0.05$) thin films measured at various frequencies.

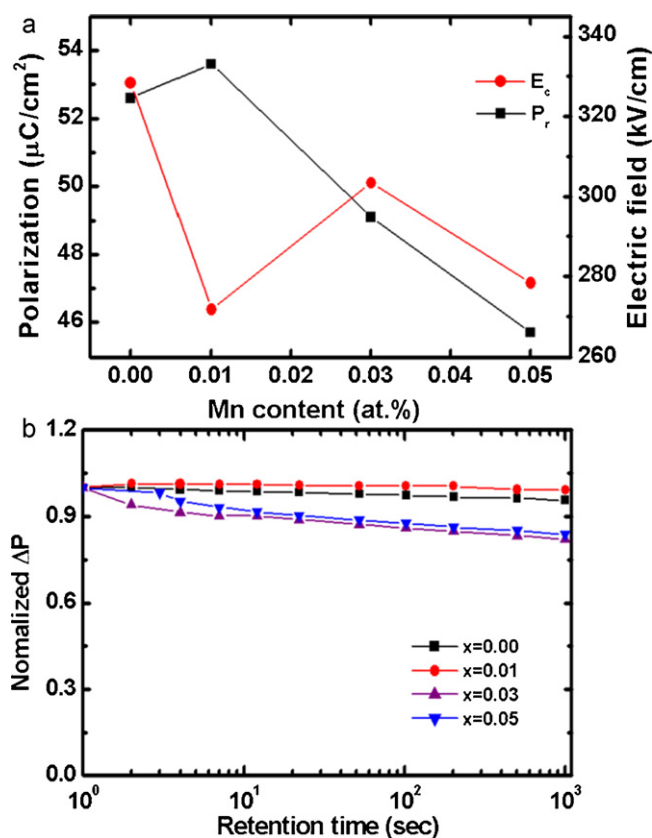


Fig. 4. (a) P_r and E_c as a function of Mn doping content at a measured frequency of 5 kHz. (b) The pulsed polarization (normalized ΔP) as a function of retention time for $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ ($x=0.01, 0.03, 0.05$) films.

that, the leakage current in BSFO film is higher than those in Mn-doped BSFO films, although the content of $(V_{O^{2-}})^{\bullet\bullet}$ in the former is lower than those in the latter films. This should be due to that the Mn^{2+} ions can restrain the formation of Fe^{2+} . Thus, the ionic conduction resulting from the electron transfer between Fe^{2+} and Fe^{3+} can be greatly reduced.

Fig. 3 plots the hysteresis loops for all the films ($\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ ($x=0.01, 0.03, 0.05$) thin films) measured at the frequencies of 10 kHz, 5 kHz and 2 kHz. Generally, for obtaining a saturated P - E hysteresis loop in BFO-based films, high frequency (>10 kHz) ac voltages should be used to eliminate the influence of leakage current. For the case of BSFO film, as the measured frequency decrease from 5 kHz to 2 kHz, a abrupt increase of P_r can be observed. Additionally, the square shape of P - E loop is deteriorated. This can be attributed to a large contribution from the leakage current, meaning that the P - E loops for BSFO film shown in Fig. 3 is not intrinsic. In contrast, the square shape of P - E loops in all Mn-doped films can be retained at the present measured frequency range. This is consistent with the lower leakage currents in the Mn-doped films than that in BSFO film. It is worth noting that, the doping of Mn can favor the decrease of E_c especially for $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.99}\text{Mn}_{0.01}\text{O}_3$ film, which exhibits a 17% reduction of E_c with respect to BSFO film. This should be due to the increase of the effective electric field induced by the lower leakage currents in BSFMO films.

For clarity, the values of E_c and P_r measured at 5 kHz as a function of Mn content is shown in Fig. 4a. Obviously, the value of P_r exhibits a monotonic decreasing trend and reduces by around 15% with the increase of Mn doping content from 1 at.% to 5 at.%. This phenomenon may result from one or more of the following factors: (i) the decrease of Curie temperature [27], (ii) the reduction of grain size with the increase of the doping content, and (iii) backswitching of the switched domains induced by aging. In contrast, the value of E_c shows a different trend from that of P_r with the increase of Mn doping content. This may be related to the competing mechanisms between the effective electric field and aging effect. The raise of effective electric field resulting from the lower leakage current can lead to the decrease of E_c . On the other hand, higher E_c has been frequently observed in the aging BiFeO_3 -based films because the applied electric field has to overcome the local fields associated with defect complexes. Therefore, the lowest E_c for $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.99}\text{Mn}_{0.01}\text{O}_3$ film should be due to the largest effective electric field resulting from the lowest leakage current as well as the least domain backswitching, which can be supported by the retention test shown in Fig. 4b. Evidently, the largest E_c for $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.97}\text{Mn}_{0.03}\text{O}_3$ film among all BSFMO films should be mainly caused by the most severe aging effect (again see Fig. 4b).

The piezoelectric and switching properties on micrometer scale were investigated as shown in Fig. 5a. The dark background region was formed by scanning with a -20 V bias voltage, while the inside $5\ \mu\text{m} \times 5\ \mu\text{m}$ square was polarized using 20 V . The frequency of the ac electric field adopted for the readout of the domain image is 10 kHz, which is far below the resonant frequency of the cantilever ($\sim 350\ \text{kHz}$) and fast enough not to interfere with the AFM system. Interestingly, the domain backswitching, which has been identified in retention test especially in $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.97}\text{Mn}_{0.03}\text{O}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.95}\text{Mn}_{0.05}\text{O}_3$ films, cannot be observed in all films. This phenomenon should be mainly due to the different testing parameters adopted in piezoelectric measurements using AFM and macroscopic retention test. The pulse width used to write in retention test is 0.04 ms. Thus, backswitching may happen in the domains that cannot be fully switched in such short time. In contrast, the polarizing time of each point using the AFM conducting tip is as long as 4 ms (corresponding to 1 Hz scanning frequency of AFM tip), which ensure that all domains can be switched completely, although aging occurs in Mn-doped BSFO films. Furthermore, the negative influence of the leakage path (such as 109° domain walls) in retention test induced by the larger top electrode adopted ($\sim 200\ \mu\text{m}$ in diameter) can be eliminated to a great extent in the piezoelectric measurement using the AFM system equipped with the conducting tip with a diameter less than 20 nm [28,29]. That is, the smaller effective electric field in the former test in comparison with that in the latter case should be an another origin responsible for the domain backswitching.

The dependence of the mean remanent d_{33} of the polarized region on Mn doping content calculated based on Fig. 5a is shown in Fig. 5b. It can be found that $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.99}\text{Mn}_{0.01}\text{O}_3$ film shows the largest piezoresponse around $146\ \text{pm}/\text{V}$, which is slightly larger than that ($\sim 140\ \text{pm}/\text{V}$) of BSFO film. Note that, compared to the remanent polarization, the remanent d_{33} exhibits a larger decreasing rate with the increase of Mn doping content from 1 to 5 at.% (the decreasing rates are 15% and 64% for the former and later cases, respectively). The abrupt decrease of the remanent d_{33} can be mainly ascribed to the structure change found in $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.97}\text{Mn}_{0.03}\text{O}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.95}\text{Mn}_{0.05}\text{O}_3$ films with respect to that of BSFO film (see the inset of Fig. 1), i.e., the structures of these two films should deviate from the morphotropic phase boundary of BSFO film. Additionally, the decrease of the contribution from irreversible movement of non- 180° domain walls in the aged BSFMO films with higher Mn contents can not be ruled out [30], since it has been demonstrated that the defect complexes in the aged films can act as the energy barriers that have to be overcome by the applied electric field during the movement of non- 180° domain walls.

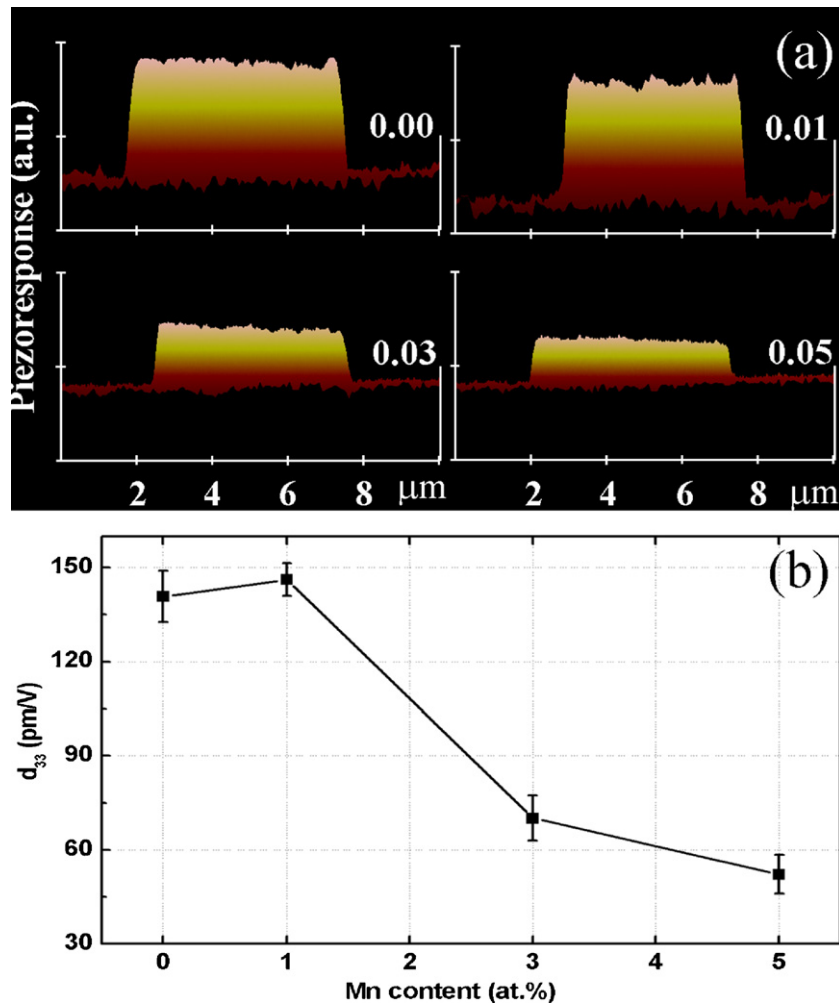


Fig. 5. (a) Section profiles of the piezoresponse images for the polarized $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ and $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.01, 0.03, 0.05$) films. (b) Remanent out-of-plane piezoelectric coefficient d_{33} as a function of Mn doping content for $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{1-x}\text{Mn}_x\text{O}_3$ ($x = 0.00, 0.01, 0.03, 0.05$) films.

4. Conclusions

In summary, the effect of Mn substitution on the ferro- and piezoelectric properties of $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{FeO}_3$ thin films was investigated. Compared to BSFO film, $\text{Bi}_{0.86}\text{Sm}_{0.14}\text{Fe}_{0.99}\text{Mn}_{0.01}\text{O}_3$ film exhibits the lower leakage current and E_c as well as the larger P_r and piezoelectric coefficient (d_{33}) demonstrating that 1 at.% Mn is necessary for obtaining the intrinsic ferro- and piezoelectric properties in the Sm doped BFO films. The reduced leakage currents in the Mn-doped BSFO films are believed to be related to the formation of Mn^{2+} , because Mn^{2+} is more stable than Mn^{3+} in the films annealed in N_2 according to its electronic configuration. The degradation of piezoelectric properties caused by further increase of Mn doping content was found to be more pronounced than that of ferroelectric properties. This phenomenon can be explained by the structure deviation from the MPB of BSFO film and less contribution from irreversible movement of non- 180° domain walls in aged BSFMO films doped with 3 and 5 at.% Mn.

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