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Ferroelectric behavior of bismuth titanate thin films grown via magnetron sputtering

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Abstract

Bismuth titanate ($Bi_xTi_yO_z$) thin films were grown using the r.f. magnetron sputtering technique on (100) silicon substrates. In the process, annealing was performed in both oxygen and dry air atmospheres at 600 °C for 30 and 120 min. The structure of the thin films was characterized through X-ray diffraction (XRD), and the ferroelectric response was determined with measurements of piezoelectric force microscopy (PFM). $Bi_4Ti_3O_{12}$ with a predominant orthorhombic phase was obtained in the annealed thin films. All the annealed films exhibited the characteristic hysteresis and butterfly loops of ferroelectric materials. Thermal annealing of $Bi_xTi_yO_z$ films in an atmosphere of air for 30 min resulted in the highest d_{33} value of 78 ± 14 pm/V, which decreased to 64 ± 26 pm/V for 120 min. On the other hand, annealing in an oxygen atmosphere produced $Bi_xTi_yO_z$ films with more uniform d_{33} values, 54 ± 3 pm/V and 42 ± 6 pm/V for 30 and 120 min, respectively. Ferroelectric coefficient values decreased with the increase of annealing time in an oxidant atmosphere, which can be explained by the vacancies present. These results are consistent with the experimental measurements carried out in other investigations.

Keywords: Ferroelectricity; Thin films; Bismuth titanate; PFM; Piezoelectric coefficient

1. Introduction

Lead-free materials have received considerable attention in recent decades due to interest in the reduction of environmental impact by using appropriate materials to replace harmful ones. For this purpose, bismuth-based film materials have been considered. For instance, bismuth layer-structured ferroelectric (BLSF) ceramics are compounds of great technological interest due to their application as a piezoelectric material with high Curie temperature (T_c), low temperature coefficients of dielectric constants, low aging rate, and strong anisotropic characters [1]. The most representative compound is bismuth titanate, $Bi_4Ti_3O_{12}$ (BIT), which belongs to the Aurivillius family, with a general formula of $(Bi_2O_2)[A_{m-1}(B)_mO_{3m+1}]$, which consists of $(Bi_2Ti_3O_{10})^{2-}$ layers sandwiched between bismuth oxide $(Bi_2O_2)^{2+}$ layers [2–4]. The BIT phase has been studied intensively due to its ferroelectric and piezoelectric properties over a wide temperature range close to 948 K which makes it suitable for applications in standard electronic devices (ceramic capacitors, piezoelectric transducers, sensors, etc.) or new ones such as nonvolatile ferroelectric random memories (NvFRAM) [5]. The ferroelectricity in BIT arises from a tetragonal structure (*I4/mmm*) turning into a lower symmetry structure such as an orthorhombic (B_2cb) [6] or monoclinic one (*Pc*) [7]. Although the monoclinic distortion in the lattice is reported to be too small to differentiate these two types [8], in terms of ferroelectricity, the space group *Pc* allows, besides the polarization component in the *a*(*b*) direction, another smaller contribution in *c*, while in

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B2cb only a polarization in *a*(*b*) appears. Experimentally, thermal processes in $Bi_xTi_yO_z$ thin films can induce dipole defects resulting from vacancies in the thin film, which could cause ferroelectric domain wall pinning [9–12] and also a common failure phenomenon called imprint [13,14], which can be noticed by a large voltage shift and deformation in the hysteresis loops [15,16]. So it is important to study the effects of vacancies in ferroelectric properties of thin films under different conditions of annealing parameters. Few studies have been carried out so far on the annealing atmosphere and determining the time for crystallization of the BIT ferroelectric phase. Simões et al. [17] have carried out the study of BIT thin films' growth through the polymeric precursor method and annealing in static air and oxygen atmospheres. A decrease in the ferroelectric behavior was evidenced in oxygen atmosphere as a consequence of the vacancies concentration, favoring the imprint phenomenon and reducing the piezoelectric coefficient dramatically. Nevertheless, reports about the effect of the annealing atmosphere on the ferroelectric properties of BIT films grown via the magnetron sputtering technique have not yet been discussed at the nanosize level.

Therefore, in this paper the effect of oxygen and dry air atmospheres during annealing treatment of BIT films on the structure and ferroelectric behavior is reported. The thin films were structurally characterized through X-ray diffraction (XRD), and their ferroelectric properties were analyzed using piezoresponse force microscopy (PFM).

2. Experimental details

BIT thin films were grown via an r.f. sputtering system, a model CIT-Alcatel HS 2000 with a balanced magnetron of 4 in. diameter, described in a previous paper [18], using a $Bi_4Ti_3O_{12}$ target of high purity (99.999%) and Si (100) wafer as a substrate. The substrates were ultrasonically cleaned in acetone solution for 15 min. The thin films were deposited at 350 °C for 45 min, with a target power supply of 150 W and 20 sccm argon flux. The thin films were crystallized through thermal annealing as a BIT phase that appears in the range 500–700 °C. A Linderberg Beng M furnace was employed in dry air (19.9% O_2) atmosphere and oxygen atmosphere with 20 sccm flux at 600 °C for 30 and 120 min. The crystalline structure of the films was characterized with a Rigaku X-ray difractometer (XRD) using incident geometry with Cu-Kα radiation ($\lambda = 1.5405$ Å) in the 2θ range 10–70° with steps of 0.02°. The ferroelectrical domain structure of the films was studied via contact resonance piezoresponse force microscopy (CR-PFM) and switching spectroscopy PFM in order to obtain the local hysteresis loops. Contact resonance condition is attained when the amplitude of oscillation (converse piezoelectric effect) of the tip-sample contact area is amplified by a factor of 10 to 100 times due to the resonant vibration of the cantilever in contact with the sample. The amplification factor is proportional to the Q-value of the corresponding resonance peak of the cantilever. The CR-PFM was carried out with Nanoscope IV Dimension 3100 equipment from Digital Instruments – Bruker with the addition of a high frequency lock-in amplifier SR844 Stanford Research Systems. An AC bias excitation at a resonance frequency of the AFM probe was applied to the conductive tip while the thin film's base was electrically grounded. CR-PFM measurements were performed with a modulation amplitude of 3 V at frequencies in the range from 352 to 378 kHz. The AFM probes used for these measurements were BudgetSensors Cr/Pt coated silicon probes with 450 μm length and 0.2 N/m spring constant as nominal parameters.

On the other hand, in the switching spectroscopy PFM (SS-PFM) measurements, a signal composed of DC squared pulses (half of the time with a DC bias and half with null bias) with a triangular envelope applied to the tip. This signal was superposed on an AC signal at a resonance frequency of the cantilever in order to produce the contact resonance effect. Modulation AC amplitude of 2 V and a frequency from 311 to 314 kHz, and a DC amplitude of the triangular envelope in the range from −21 to +21 V were used. The AFM probes used for these measurements were Rocky Mountain Nanotechnology RMN-12Pt400B platinum probes with 400 μm length and a 0.3 N/m spring constant as nominal parameters. The employed resonance frequencies are more sensitive for detecting the out-of-plane contribution; these are flexural modes of the cantilever. The local loops were obtained from the average of 20 loops in order to reduce the noise effect. In order to obtain d_{33} values in pm/V units, the PFM amplitude values (given in V) were compared with the vertical variations of height (given in pm) of the tip-sample surface as measured by the AFM system.

3. Results and discussion

Fig. 1 shows the XRD patterns of the annealed thin films at 600 °C. They exhibited a polycrystalline structure as a result of the numerous XRD peaks. The XRD peaks correspond to a mixture of standard crystal data of $Bi_4Ti_3O_{12}$ indexed as monoclinic (*Pc*), orthorhombic *(B2cb*), and tetragonal

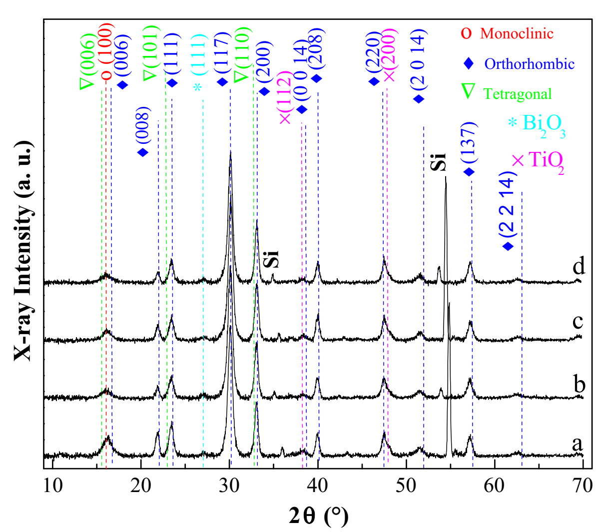

Fig. 1. XRD patterns of annealed $Bi_4Ti_3O_{12}$ thin films: (a) 30 min-air, (b) 30 min O_2, (c) 120 min-air and (d) 120 min-O_2.

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(*I4/mmm*), the orthorhombic structure being the main phase, but other minor phases of Bi_2O_3 and TiO_2 also appear. The films are highly textured in the (117) plane. The orientation α in the c direction in the (008) plane was estimated with the intensities relation [19]:

$$\alpha = \frac{I(008)}{I(008)+I(117)}$$

A thermal annealing via XRD in-situ as a function of annealing temperature in the range of 400–700 °C and in oxygen atmosphere was performed on the as-grown film in order to observe the evolution of phases in the formation of BIT. Fig. 2 shows these XRD patterns where the formation of $Bi_{12}TiO_{20}$ (B12) can be seen which appears at low temperatures (< 500 °C) during the crystallization of BIT [20]. The formation of BIT occurs in the range 500–700 °C. Also, other phases, such as Bi_2O_3 and TiO_2, are exhibited from the target composition. Moreover, the phase $Bi_2Ti_2O_7$ appears at high temperatures due to the volatilization of bismuth.

The annealing atmosphere was found to be very important for the crystalline structure of the BIT films. Although the α values were not particularly high, they showed a better orientation for the annealing in air atmosphere, diminishing from $0.162 \pm 0.052°$ to $0.091 \pm 0.010°$, and in oxygen atmosphere from $0.078 \pm 0.055°$ to $0.077 \pm 0.032°$ for 30 min and 120 min, respectively. So annealing in air atmosphere could favor an improved BIT crystallinity in c. Considering that the out-of-plane PFM signal is detected by the flexural deflection of the cantilever, the crystallographic alignment in c influences the ferroelectric behavior. Structurally, both films exhibited a thickness in the range of 200–250 nm.

PFM images of the ferroelectric behavior of BIT films through CR-PFM as a response to the bias voltage is shown in Fig. 3. These figures consisted of the topography as well as the PFM amplitude and the PFM phase images, which reveal information about the local electromechanical response and

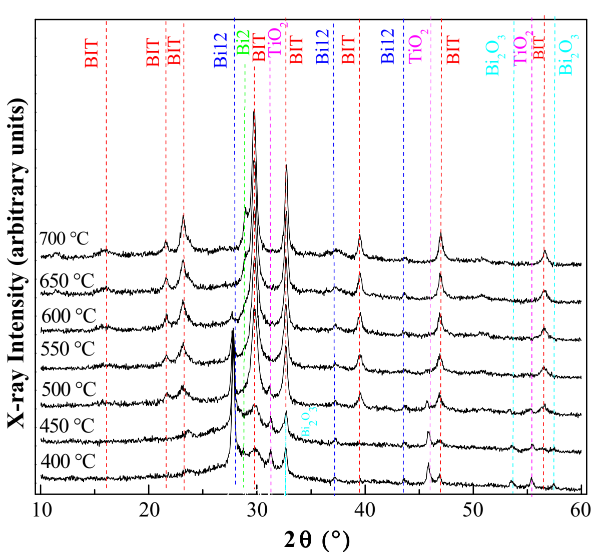

Fig. 2. XRD patterns of as grown films undergo thermal annealing in XRD in situ in the range of 400–700 °C. $Bi_xTi_yO_z$ phases: $Bi_4Ti_3O_{12}$ (BIT), $Bi_{12}TiO_{20}$ (Bi_{12}), and $Bi_2Ti_2O_7$ (Bi_7).

out-of-plane ferroelectric domain distribution [21]. The morphology of the thin films exhibited greater roughness in air atmosphere varying from 4.6 ± 2.6 nm to 13.7 ± 2.7 nm, while in oxygen it varied from 2.9 ± 0.1 nm to 5.1 ± 1.8 nm, for 30 and 120 min, respectively. Also, the grain size increased significantly in dry air at values from 11.8 nm to 17.5 nm, and in oxygen it was more uniform from 8.8 nm to 9.9 nm. The differences between the atmospheres can be explained by a lower nucleation rate in air atmosphere than in oxygen, which is more reactive.

In the PFM images, the high contrast difference in PFM amplitude and PFM phase for the annealed BIT films reveals the ferroelectric behavior of the samples. This is not a surprise, because as we observed from the XRD results, all the films exhibit the ferroelectric BIT phase. In the PFM phase images, dark and bright contrasts refer to the out-of-plane domain orientation as up and down directions (or vice versa), respectively. Intermediate contrast in the phase image with non-null amplitude could involve regions with in-plane domains, while the null piezoelectric amplitude response may emerge from the small non-ferroelectric regions of tetragonal and Bi_2O_3 and TiO_2 phases. The BIT thin films showed a remarkable influence of the annealing atmosphere. A higher diminution of ferroelectric behavior in oxygen atmosphere can be observed. This response can be affected by a lower crystallographic orientation in c, hampering its alignment in this axis as the AFM tip scans the surface. Also, point defects in the films can produce a decrease in ferroelectricity. The high temperature (600 °C) during the annealing may create considerable vacancies. BIT has a p-type conductivity for which the films annealed in oxygen atmosphere can increase the defects such as Bi or Ti vacancies [17]. But given that the Ti–O bonding is stronger than Bi–O, Bi and O vacancies are more probable. This phenomenon results in a rise of conductivity, because the oxygen ions can hop through the Bi ions. Also, annealing in oxygen atmosphere can yield disordered oxygen vacancies, enabling oxygen ions to emigrate and thus increase conductivity [17]. The mobile oxygen vacancies tend to be trapped in ferroelectric domain walls because they are energetically favorable positions acting as potential wells [9,22]. Finally, the domain wall-vacancy interaction freezes the domain wall leading to the suppressed switchable polarization [9].

PFM amplitude loops with the characteristic butterfly shapes and PFM phase loops are shown in Fig. 4. Butterfly loops come from the combination of piezoelectric and electrostatic effects [23]. The coercive bias for these measurements was in the range from 10 V to 12 V. The piezoelectric coefficient d_{33} was calculated from the slope of the linear region of the loop of the non-biased butterfly curves, exhibiting values of 82 pm/V and 73 pm/V for films annealed at air atmosphere, and 50 pm/V and 38 pm/V for films annealed in oxygen atmosphere for 30 min and 120 min, respectively. In order to determine an average of d_{33}, several measurements were obtained for each sample at steps of about 1.0 μm along a straight line. The average values and standard deviations are listed in Table 1. The piezoelectric coefficients measured for BIT films annealed in air

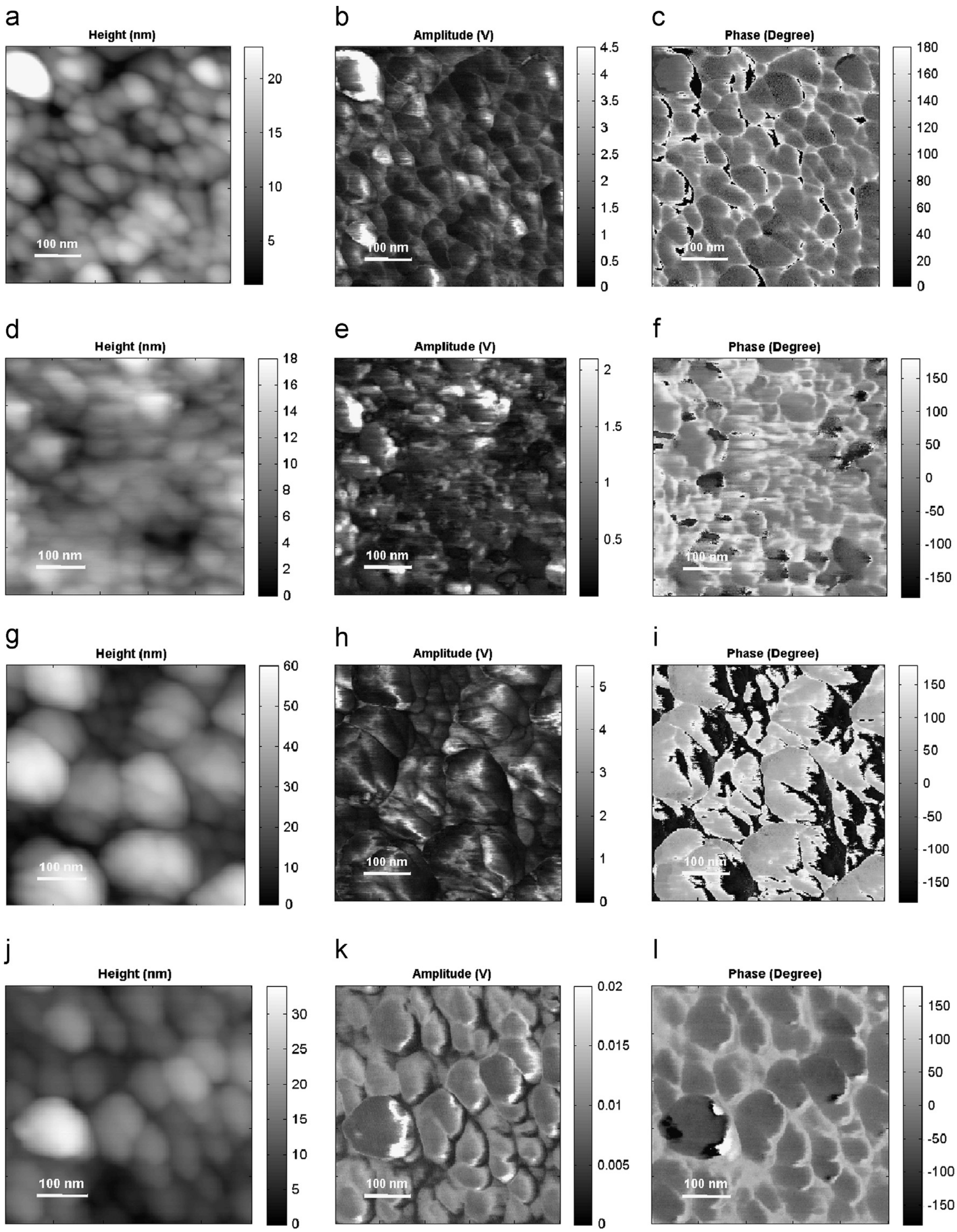

Fig. 3. CR-PFM images of $Bi_xTi_yO_z$ thin films annealed: in oxygen for 30 min and 120 min—(a and g) topography, (b and h) amplitude and (c and i) phase, respectively; in air for 30 min and 120 min—(d and j) topography, (e and k) amplitude and (f and l) phase, respectively.

showed the highest dispersion of values, which can be explained as a low quality oxidation process due to the mix of several gases and water vapor. Oxygen as an annealing atmosphere produces uniform ferroelectric properties in BIT films with low standard deviation but with lower d_{33} values. Standard deviation of d_{33} increases with the rise in annealing time for both annealing atmospheres. The annealing process improves the d_{33} values for the shorter annealing time. At longer times, the d_{33} values decrease in both air and oxygen atmosphere probably due to more concentration of defects that can enhance the current. The d_{33} values obtained in these studies are very promising since they are similar to the best values of ferroelectric materials as films reported in the literature. These ferroelectric materials include BIT (40 pm/V) [24], $Pb[Zr_xTi_{1-x}]O_3$ (PZT)

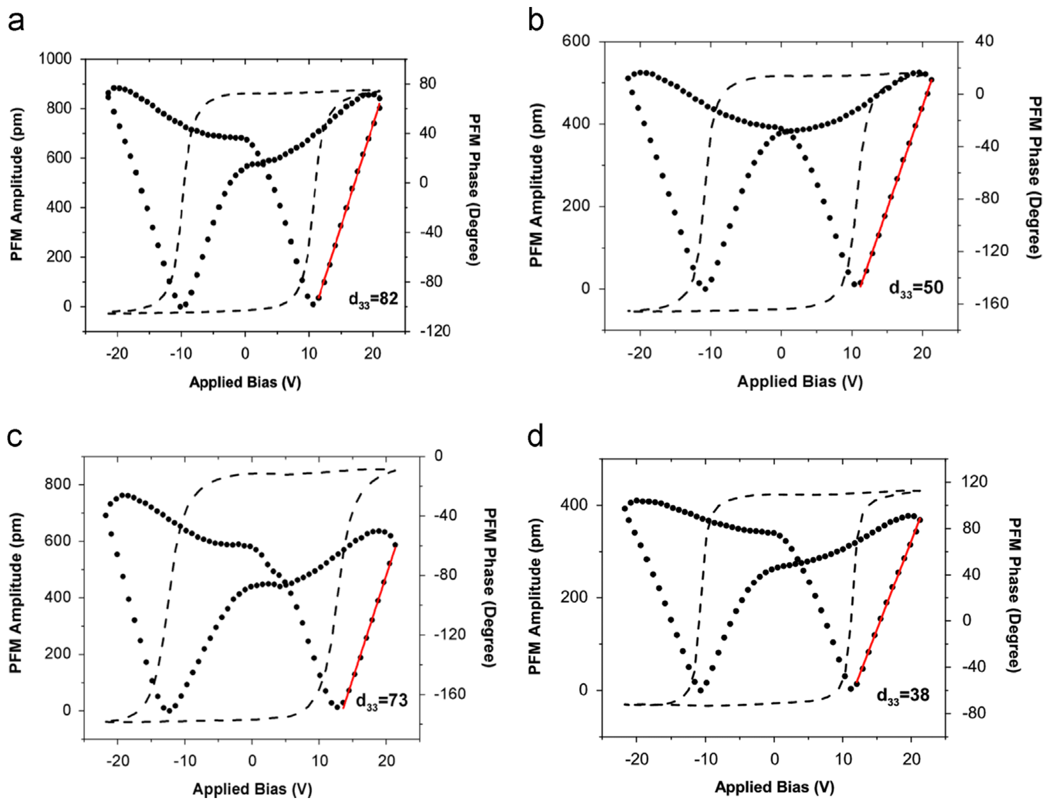

Fig. 4. Local PFM amplitude hysteresis loops of BIT annealed film: (a) 30 min, air; (b) 30 min, oxygen; (c) 120 min, air; and (d) 120 min, oxygen. The applied bias was from −21 V to 21 V.

Table 1
Average values and standard deviations of piezoelectric coefficient d_{33} of $Bi_xTi_yO_z$ films obtained from PFM amplitude hysteresis loops after thermal annealing in air and oxygen atmospheres for 30 and 120 min.

Sample	Time of annealing (min)	Average d_{33} (pm/V)	Standard deviation (pm/V)
$Bi_xTi_yO_z$ films annealed in	30	78	14
air atmosphere	120	64	26
$Bi_xTi_yO_z$ films annealed in	30	54	3
oxygen atmosphere	120	42	6

(40–110 pm/V) [25], $SrBi_2Ta_2O_9$ (17 pm/V) Nd-doped BIT (38 pm/V) [26], $Bi_{3.15}Eu_{0.85}Ti_3O_{12}$ (46.7 pm/V) [27], $Bi_{3.15}Dy_{0.85}Ti_3O_{12}$ (63 pm/V) [28] and $CaBi_4Ti_4O_{15}$ (30–560 pm/V) [29].

In the PFM phase loops (Fig. 4), a regular and stable dynamic can be observed in both atmospheres. Although the shape of the hysteresis loops is the expected one, they exhibited a slight shift along the applied bias voltage axis, which could suggest the existence of an imprint phenomenon. This can be attributed to the presence of freezing dipoles, due to vacancies, favoring a polarization state over another one.

These results suggest a better potential for BIT thin films annealed in oxidant atmosphere (controlled mix of a low proportion of oxygen with an inert gas) for NvRAM applications considering the higher d_{33} values and the regular, uniform, and stable ferroelectric properties.

4. Conclusions

The effect of oxygen and dry air atmosphere in the annealing process on the ferroelectric properties of $Bi_xTi_yO_z$ thin films was studied. The annealed films exhibited the $Bi_4Ti_3O_{12}$ phase and the presence of other minor phases (Bi_2O_2 and TiO_2). The structural and ferroelectric properties showed a strong influence of the annealing atmosphere. Thin films annealed in oxygen atmosphere show lower PFM response, which could indicate an increase of Bi and O vacancies that freeze the domain wall movement. Although the piezoelectric coefficient d_{33} was higher in air atmosphere, the values for samples annealed in oxygen showed less dispersion indicating that BIT thin films with uniform properties can be obtained in this atmosphere.

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