



Effect of bottom electrode and resistive layer on the dielectric and ferroelectric properties of sol–gel derived BiFeO₃ thin films

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ABSTRACT

The effect of RuO₂ electrodes and La₂O₃ resistive layer were analyzed on the electrical and ferroelectric properties of BiFeO₃ (BFO) films deposited on Pt/Ti/SiO₂/Si substrates using a chemical solution method. XRD patterns revealed that BFO films with RuO₂ and La₂O₃ layers appeared preferred (0 1 2) orientation. The surfaces of the BFO films with the La₂O₃ layer appeared uniform with dense grain clusters. The utilization of La₂O₃ and RuO₂ layers reduced the diffusion of Ti and Si into the BFO films. Leakage mechanism of the films found specific to applied electric field. At lower applied electric field space-charge-limited current found dominant and at an applied electric field >80 kV/cm surface-limited conduction (Fowler–Nordheim type) found the dominant. The BFO films grown with La₂O₃ resistive layers also contained lower concentrations of Fe²⁺, which resulted enhancement of dielectric constant (ϵ), remnant polarization and lower leakage current. Among the studied films RuO₂/BFO/La₂O₃/BFO/RuO₂/Pt/Ti/SiO₂/Si exhibited the best with value of relative permittivity (ϵ_r) 208.8, leakage current 5.503×10^{-6} kV/cm², remnant polarization ($2P_r$) 3.80 μ C/cm², and coercive field ($2E_c$) 78.22 kV/cm.

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1. Introduction

In recent years multiferroic BiFeO₃ (BFO) is being studied extensively. BFO is antiferromagnetic with the Néel temperature ($T_N \approx 370^\circ\text{C}$) and ferroelectric with the Curie temperature ($T_C \approx 830^\circ\text{C}$) and probably be an excellent candidate for multiferroic applications [1–3]. However, the practical device realization of BFO has been hindered due to high leakage current, which mainly results from the presence of defects [4–6]. To reduce leakage current in BFO film, researchers considered ion substitutions in the Bi and/or Fe sites as well as by employing conducting oxide electrodes [7–12] and preparation of high-quality samples [13] to improve the electrical properties of BFO. The conducting oxide layers generally considered prevent inter-diffusion between the metallic electrodes and the BFO. Among the oxide electrodes, RuO₂ appears as stable conductive oxide with bulk resistivity $\leq 100 \mu\Omega \text{ cm}$, large work function, high thermal and chemical stability and also acts as a good oxygen diffusion barrier [14–18]. Additional resistive inter layer like thin La₂O₃ film can be introduced for further possible reduction of the leakage currents as La₂O₃ films are reported to be mechanically stable and have high electrical breakdown field strength [19–21]. In this work, we explore the use of RuO₂ as top and bottom electrodes and

La₂O₃ as resistive layer and study their systematic effects on the structure, morphology, and electrical properties of the BFO films.

2. Experimental details

RuO₂ electrodes were deposited on Pt/Ti/SiO₂/Si substrates by d.c. sputtering in an atmosphere of Ar:O₂ (1:3) at a power of 200 W for 15 min. The sputtering parameters used were described elsewhere [22]. The Ru films were annealed within the temperature range of 450–700 °C in air ambient. Thickness of the RuO₂ thin films was ~ 65 nm. BFO thin films were deposited on the RuO₂ coated Pt/Ti/SiO₂/Si substrates by a chemical solution deposition method. BFO solution was prepared by dissolving bismuth nitrate and iron nitrate in 2-methoxyethanol. The precursor solution was spin-coated at 4000 rpm for 20 s and dried at 240 °C. This process was repeated several times to achieve a film thickness ~ 550 nm. The film was annealed in a rapid thermal annealing (RTA) furnace at 550 °C in N₂ ambient. Using r.f. sputtering La₂O₃ layer of ~ 20 nm was developed on the BFO film and was annealed at 550 °C prior to further deposition of BFO.

The crystalline quality of the RuO₂, BFO, BFO–La₂O₃ films were assessed by XRD using the General Area Detector System (GADDS, D8-ADVANCE, Bruker), and the microstructures were studied using a field-emission scanning electron microscopy (FESEM, JSM-6700F, JEOL Ltd.). For electrical measurements, RuO₂ dot electrodes of 0.2 mm in diameter were sputter-deposited on the top surface of the BFO films through a shadow mask. The resistivity of the RuO₂ films was determined by the four-point probe measurement (MCP-T360). Dielectric measurements were carried out using an impedance analyzer (HP 4194A). Polarization–electric field (P – E) hysteresis loops were characterized by a ferroelectric tester (Radiant Inc.). Current–voltage (I – V) measurements were carried out using a Keithley 5467A electrometer. A positive bias voltage was applied to the top RuO₂ electrode. X-ray Photoelectron Spectroscopy (XPS, ESCALab 250i-XL) and the Secondary Ion Mass Spectrometry (ION-TOF SIMS IV) were used to analyze the chemical composition and the depth profiling of the stacked films. The synthesized films are defined as fol-

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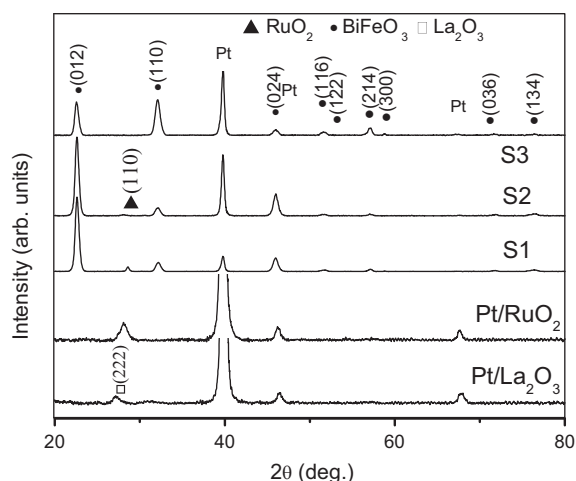


Fig. 1. XRD diffraction patterns of S1, S2, S3, RuO₂ and La₂O₃ thin films.

lows: RuO₂/BFO/La₂O₃/BFO/RuO₂/Pt/Ti/SiO₂/Si: S1, RuO₂/BFO/ RuO₂/Pt/Ti/SiO₂/Si: S2, and RuO₂/BFO/Pt/Ti/SiO₂/Si: S3.

3. Results and discussion

Fig. 1 depicts the XRD patterns of the annealed RuO₂, La₂O₃, S1, S2 and S3 films. It is observed that the RuO₂ films appear (1 1 0) preferred orientation with polycrystalline nature. The peaks are indexed according to the JCPDS card no. 40-1290. Electrical conductivity studies of the RuO₂ thin films annealed at 500–700 °C are carried out. We observe increase of conductivity for the films annealed at ≥600 °C, which attributes the increase of grain size as well as the inter layer diffusion. The conductivity of the RuO₂ films annealed at 550 °C is 9632 S cm⁻¹, which is comparable to earlier reported values [23]. For further BFO thin film depositions we use the RuO₂ films annealed at 550 °C. It is also observed that the S1, S2 and S3 films appear phase pure polycrystalline nature. The peaks are indexed according to the JCPDS card no. 82-1254. The average crystalline size of the BFO films grown with and without RuO₂ and La₂O₃ are determined using Scherrer's formula [24]. Table 1 summarizes crystalline size and dependence of preferred orientation of films. The (0 1 2)-orientation ratio is calculated using Eq. (1) [25]:

Table 1
Crystalline size and dependence of preferred orientation of films.

Sample	Preferred (0 1 2) orientation (%)	Av. crystalline size (nm)
S1 (with RuO ₂ and La ₂ O ₃)	82.06	114.42
S2 (with RuO ₂ , no La ₂ O ₃)	83.63	120.00
S3 (with Pt, no La ₂ O ₃)	44.49	91.66

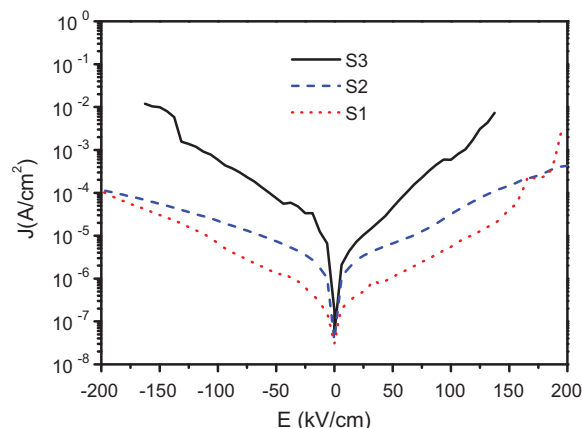


Fig. 3. Room temperature leakage properties (log *J* vs *E* plots) of the S1, S2 and S3 films.

$$\text{Orientation ratio} = \frac{I(012)_{\text{BFO}}}{I(012)_{\text{BFO}} + I(110)_{\text{BFO}} + I(116)_{\text{BFO}}} \times 100 \quad (1)$$

where *I*(0 1 2), *I*(1 1 0), and *I*(1 1 6) refer the peak intensity of the (0 1 2), (1 1 0) and (1 1 6) orientations of the BFO films. It is observed that in the S1 and S2 films the (0 1 2) peak shows the highest intensity among the other peaks, which indicates that RuO₂ layer promotes the growth in the (0 1 2) plane. The preferred orientation in the BFO film is retained when the La₂O₃ layer is applied between the BFO films (S1). The XRD pattern of sputter-deposited La₂O₃ on Pt shows reflections belonging to cubic La₂O₃, indexed according to JCPDS card no. 22-0369. The weak peak intensities are a consequence of the thin (~40 nm) La₂O₃ layer [26]. The presence of La₂O₃ formed on BFO thin films before the stacking of another layer takes place is confirmed by XPS results, which shows that the position of the La-3d_{5/2} peak is at 834.9 eV, corresponding to the exact position

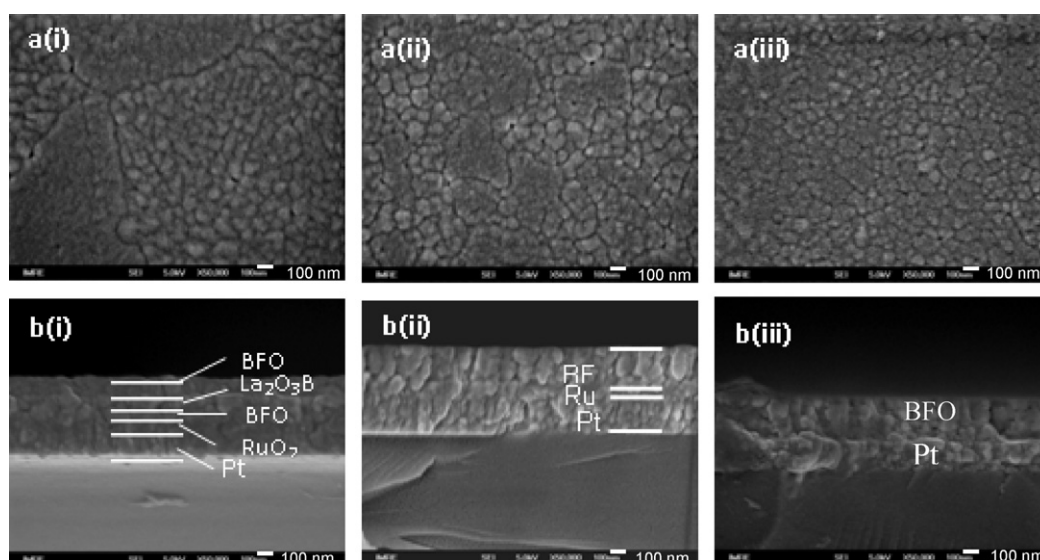


Fig. 2. SEM (a) surface morphology and (b) thickness of (i) S1, (ii) S2 and (iii) S3 films.

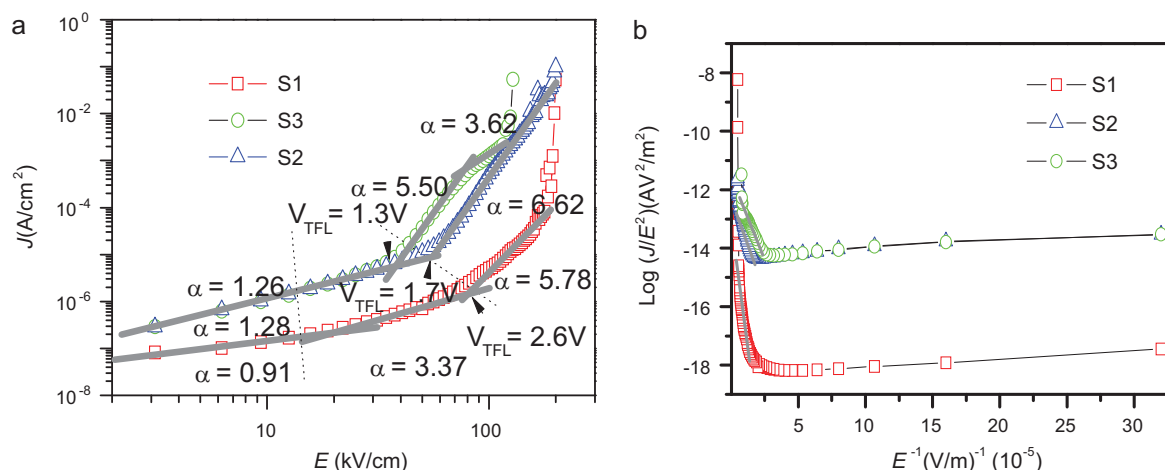


Fig. 4. (a) $\log(J)$ vs $\log(E)$ plots at the positive bias of the films measured at room temperature. The exponent, α , in $J \propto E^\alpha$, which is also the slope of the curve for each region in the films is shown, and (b) $\log(J/E^2)$ vs $1/E$ plot at the positive bias of the films measured at room temperature.

of the La-3d_{5/2} peak in La₂O₃ [27,28]. We can imply that the La₂O₃ layer formed between BFO films is pure.

The surface morphologies and thicknesses of the samples are displayed in Fig. 2(a, b). It is observed that the thicknesses of all the films are very well-defined. The microstructures of S1, S2 and S3 films are equally dense. From Table 1, we observe largest grain size in S2, followed by S1 and S3 films. It again indicates that RuO₂ promotes the growth of grains. The addition of La₂O₃ may reduce the grain size slightly but generally, there is an observable difference to the grain size of the film without La₂O₃ and RuO₂ (S3). It is also observed that morphology of S1 consists of many clusters of grains growing together while grains are grown uniformly in S2 and S3 films. The difference in morphology is most likely stemmed from the La₂O₃ layer in S1. The microstructure of S1 may well resemble the rosette structure observed in Zr-rich PZT films [29–31]. Since the rosette structures are usually formed from a low nucleation rate, this may suggest that the clusters of grains found in the films are a result of different nucleation rates [30]. The La₂O₃ layer may slow down the nucleation rate of the BFO films.

Fig. 3 shows the leakage current dependence on electric field ($\log J$ vs E) plots of the S1, S2, and S3 films. The leakage current is the lowest for S1 and is the highest for S3 films. The barrier

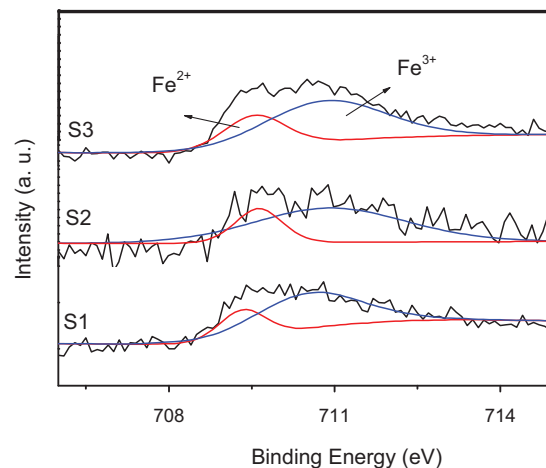


Fig. 5. XPS spectra of Fe 2p lines for S1, S2 and S3 after peak fitting.

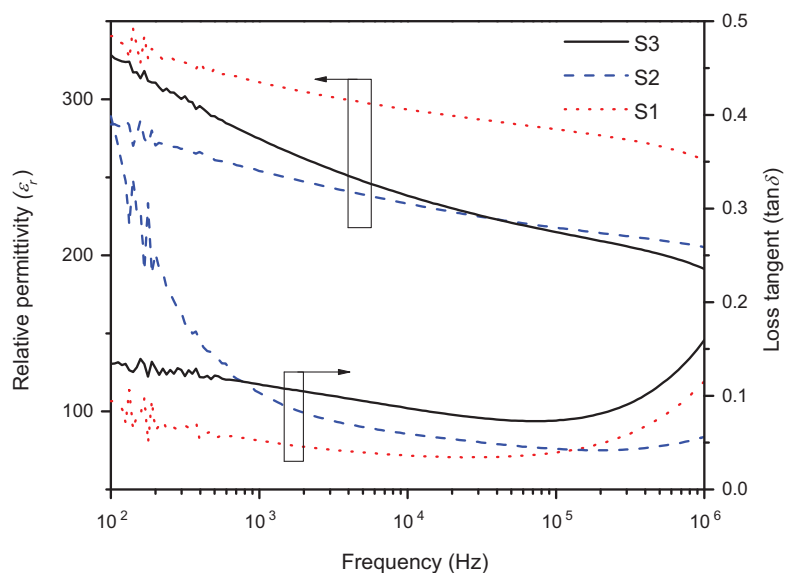


Fig. 6. Frequency dependence of relative permittivity (ϵ_r) and loss tangent ($\tan \delta$) for the S1, S2, and S3 films measured at room temperature.

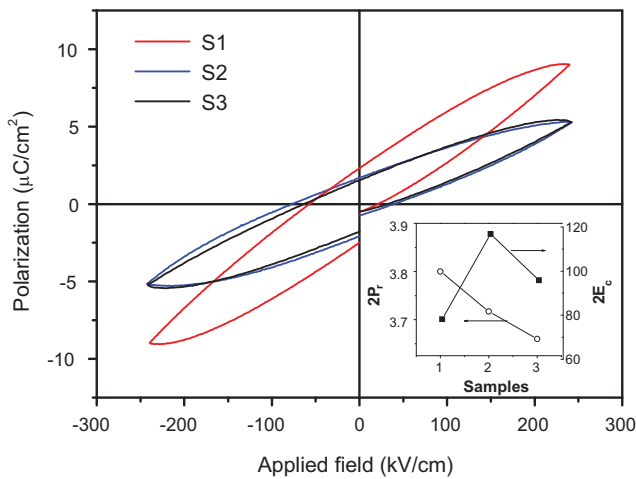


Fig. 7. P - E hysteresis loops of S1, S2 and S3 measured at room temperature. Insert shows the change in $2P_r$ and $2E_c$ of the same films.

layers result in lower leakage current, possibly due to the inhibition of defects such as oxygen vacancies [32]. The fluctuation in valency of Fe^{2+} and Fe^{3+} in BFO films also affects the leakage current properties in BFO thin films [8,31]. It is essential to understand the influence of La_2O_3 and RuO_2 on the electrical properties of BFO films and the origin of leakage current mechanism. The leakage current is mostly dominated by the surface-limited processes (thermionic Schottky and quantum mechanical Fowler-Nordheim currents) and the bulk-limited processes (Poole-Frenkel and Space-Charge-Limited Currents). From the $\log J$ vs $\log E$ plot in Fig. 4(a), it is observed that at low electric field (<20 kV/cm) the curve follows Ohmic behavior with the slope (α) value ~ 1 in each film. At higher electric field (20–100 kV/cm), the curve in each film increases with α value > 1 . This type of observation in the J - E behaviors of the BFO films resembles the Space Charge Limited Current (SCLC) type of conduction. At low electric field, Ohmic conduction comes from the injection of electrons from the electrode or shallow traps near the conduction band of the film into the conduction band. It should be pointed out that the electrons are injected from the bottom elec-

trode into BFO when it is positive bias. As electric field increases, the amount of injected electrons in the insulator increases, which becomes more than the thermally stimulated density of electrons, which dominate SCLC conduction. However, the slopes obtained for the films are higher than 2. The curves change according to the modified Child's law where $J \propto E^\alpha$, $\alpha > 1$. It is also noted that there are three different slope gradient regions for S1 and S3, and two slope gradient regions for S2 films. Each slope signifies different electron trap levels. According to Lampert [33], the voltage dependence of a film having different trap levels distributed in the band gap region is expressed in Eq. (2)

$$J = e\mu N_c \left(\frac{\varepsilon}{eN_c k T_f} \right) \frac{V^{l+1}}{d^{2l+1}} \quad (2)$$

where N_c is the effective density of states in the conduction band, μ is the charge carrier mobility, d is the film thickness, k is the Boltzmann constant and T_f is the temperature characterizing the trap distribution. For $l > 1$, or a slope of more than 2, there are deep electron trap levels in the films. The slope of ~ 3 for S1 in the middle range is probably due to the density of the trap levels. The slope of ~ 6 at higher electric field for all three samples is a result of completely filled deep traps and the double injection of charges into the bulk film. Finally, the voltage for filling up the deep electron traps is known as the trap filled limited voltage V_{TFL} , which is the transition voltage from Ohmic to modified Child's law. We found that V_{TFL} changes according to $S1 > S2 > S3$. This indicates that deeper traps are found in S1 compare to S2 and S3. This also implies that La_2O_3 layer between the BFO films creates deeper traps in the film. SCLC mechanism has been reported to be the main leakage mechanism in BFO films [34–38].

In addition to SCLC mechanism, Poole-Frenkel (PF) conduction, which are associated with field-enhanced thermal excitation such as the release of trapped electrons or holes. Schottky emission (SE) is another leakage mechanism, which represents the field-enhanced thermionic current limited by the electrode. Therefore, it is necessary to determine whether PF or SE is a dominant leakage mechanism in the films. Both PF and SE can be expressed as Eq. (3)

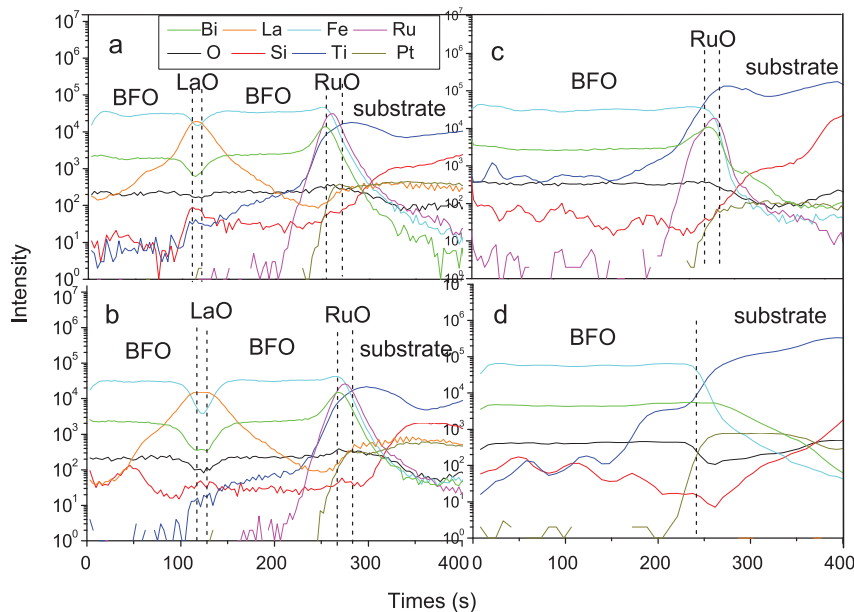


Fig. 8. SIMS depth profiles of (a) S1 before annealing, (b) S1 after annealing, (c) S2 and (d) S3 after annealing.

[39–42]:

$$\frac{J}{E} = J_0 \exp \left[\frac{-(\phi_t - e\sqrt{eE/\alpha\pi\epsilon_0\epsilon_{\text{opt}}})}{kT} \right] \quad (3)$$

where J_0 is a constant, ϕ_t is the trap ionization energy, e is the electron charge, ϵ_{opt} is the optical (electronic) dielectric constant of the film, ϵ_0 is the dielectric constant of vacuum, k is the Boltzmann's constant, T is the temperature and coefficient $\alpha = 1$ for PF and 4 for SE. The conduction current is governed by PF emission or SE if the $\ln(J/E)$ vs $E^{1/2}$ plot is linear with a slope of $e\sqrt{e/(\pi\epsilon_0\epsilon_{\text{opt}})}/kT$ or $e\sqrt{e/(4\pi\epsilon_0\epsilon_{\text{opt}})}/kT$, respectively. ϵ_{opt} can be calculated from the slope, which can be used to calculate the optical refractive index (n) through the relation of $n = \sqrt{\epsilon_{\text{opt}}}$. We calculate the n values for each of the slopes in the films based on the above equations. However, the n values obtained do not match with that of the BFO thin films obtained by spectroscopic ellipsometry measurement, which is about 2.5 [34,43]. This clearly indicates that PF emission or Schottky emission is not the main leakage mechanism in the films with and without the resistive layer.

Fig. 4(b) shows the $\log(J/E^2)$ vs $1/E$ plots of the films at the positive bias. We observed linear negative slopes at high electric fields (>80 kV/cm). Therefore, at higher electric fields (>80 kV/cm), Fowler–Nordheim, a surface-limit conduction is the dominant conduction mechanism in all the films. The effect of injecting charge carriers from the electrodes into an insulator layer usually occurs at high electric fields in BFO, PZT, and BST films [34,38,44].

X-ray photoelectron spectroscopy (XPS) of the S1, S2, and S3 films are shown in Fig. 5. The Fe 2p_{3/2} peaks are fitted into two peaks at about 709.7 and 711.2 eV, which correspond to the Fe²⁺ and Fe³⁺ states, respectively. The areas encapsulated by the curves give the concentration ratio of Fe²⁺ and Fe³⁺. The ratios of Fe²⁺ to Fe³⁺ of S1, S2, and S3 films are 22:78, 26:74, and 59:41, respectively. The highest amount of Fe³⁺ in S1 among the three films suggests that the amount of oxygen vacancies in S1 films is the lowest, which accounts for its improved leakage current properties.

Fig. 6 plots the frequency dependence of the relative permittivity (ϵ_r) and loss tangent ($\tan\delta$) for the S1, S2, and S3 films, which shows highest ϵ_r and lowest $\tan\delta$ values as 280.8 and 0.0395 respectively at 10 kHz for the S1 films. This improvement supports the contribution of La₂O₃ and RuO₂ layers by preventing the diffusion of the substrate elements into the BFO, as in the case of a RuO₂ barrier between PZT and a metal foil [45]. The ϵ_r and leakage current density of the La₂O₃ thin film on Pt/Ti/SiO₂/Si substrate is ~ 17.4 at 10 kHz and 6.06×10^{-7} A/cm² at 500 kV/cm, respectively. The leakage current of the current La₂O₃ film is an order higher than those obtained by atomic layer deposition and electron beam evaporation ($\sim 10^{-8}$ A/cm²) [28]. This difference could be a result of the thinner La₂O₃ films formed by the latter, which range from 3.5 to 5 nm. La₂O₃ has large band gap (~ 5.5 eV), low leakage current, and high ϵ_r value make it suitable as effective sink/buffer layer for lowering of leakage current in BFO films by inhibiting the conduction of mobile charges. Therefore, the leakage current and dielectric properties of S1 and S2 films are improved.

Fig. 7 presents the P – E hysteresis loops of the samples. Comparatively better ferroelectric properties with $2P_r$ and $2E_c$ values of 3.80 $\mu\text{C}/\text{cm}^2$ and 78.22 kV/cm respectively obtained for the film S1. Inset compares the $2P_r$ and $2E_c$ values of the studied films. The possible reason for this low value is not clear at this time as both low and high values are reported in the literature [46–49]. It is also known that the measurement frequency affects the shape of the hysteresis and the polarization values too. The poorer electrical properties in S3 film is due to the obvious Si and Ti diffusion from the substrates into the BFO layers in all the samples are analyzed using SIMS as shown in Fig. 8. There is no diffusion of Ru and La into

BFO layers in S1, S2, and S3 films observed after annealing. This confirms absence of intermediate phase formation between RuO₂ and BFO films [50,51].

4. Conclusions

We employ RuO₂ and La₂O₃ as oxide electrodes and resistive layers to synthesize BFO films on Pt/Ti/SiO₂/Si substrates using a chemical solution deposition method. The BFO films with RuO₂ and La₂O₃ layers show preferred (0 1 2)-orientation growth with dense morphology. RuO₂ and La₂O₃ resistive layers improve the value of ϵ_r and P_r and reduce leakage currents of the BFO films. It is found that diffusions of Ti and Si from the substrate into the BFO films are impeded by the RuO₂ barrier layer. XPS analysis also shows that the BFO films grown with the barrier layers contain smaller amount of Fe²⁺ than those without the barrier layers, which cause lower oxygen vacancies in the former and result improved leakage current properties. Space-charge-limited current dominates in all the films at electric field below ≤ 80 kV/cm and surface-limit conduction current dominates at higher electric field >80 kV/cm.

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