Effect of substrate temperature on the structural and electrical properties of La and Mn co-doped BiFeO₃ thin films

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The effect of substrate temperature on the structural and electrical properties of multiferroic Bi₉₀Laₐ₁₀Fe₀₉₅Mn₀₅O₃ (BLFMO) thin films deposited on Pt (111)/Ti/SiO₂/Si substrate using pulsed laser deposition (PLD) has been investigated. Films with substrate temperature ranging from 450 °C to 650 °C have been deposited. The grain size and roughness are found to increase with substrate temperature. The film deposited at 575 °C exhibits maximum remnant polarization around 39 μC/cm² and a coercive field of 400 kV/cm. The cyclic fatigue study of the sample shows only 4% loss after 10⁸ cycles. Complex impedance study of the BLFMO thin films demonstrates electrical homogeneity of the sample. AC conductivity data has been fitted using Jonscher single power law, and value of n found to be ~1 indicating translational hopping conduction mechanism. The lowest leakage current found is 4.37 × 10⁻⁷ A/cm² at 575 °C. The leakage current mechanism is found to be dominated by space charge limited current and Fowler-Nordheim conduction mechanism.

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1. Introduction
The last decade has seen enormous research on multiferroic materials due to their versatile functionalities. Many multiferroic materials have been studied to explore fascinating physics associated with it. Bismuth ferrite (BiFeO₃) is one of the room temperature multiferroic material, which is widely investigated due to excellent ferroelectric properties [1–5]. BiFeO₃ (BFO) exhibits rhombohedral distorted perovskite crystal structure with R₃c space group. BFO has high Curie temperature (Tc = 850 °C) and Neel temperature (TN = 377 °C) with large remnant polarization around 100 μC/cm² in thin films [6,7]. These properties make BFO a suitable candidate for high-temperature applications.

BFO thin films can be used for various applications such as spintronics, memories, sensors, microelectromechanical devices [2,8–10]. BFO thin films show scattered physical properties under different processing conditions [11–14] and are stable only in a narrow region of temperature and oxygen pressure [15,16]. Higher leakage current due to low resistive behavior originating from Bi volatility and Fe charge fluctuation limit the use of BFO in technological applications. To achieve excellent electrical properties, it is necessary to optimize BFO thin films with respect to deposition parameters, namely substrate temperature and oxygen pressure. Further, the thin films should be free from oxygen vacancies and defects. To improve material stability and performance, various dopants such as La on Bi site and Mn on Fe site are substituted, and electrical properties of thin films have been investigated [17–22]. Simultaneous A and B-site substitution have also been carried out to enhance the physical and electrical properties [23–26]. Many researchers have investigated the effect of La and Mn co-doped BFO thin films on the physical properties by using different substrates, substrate orientation, and the buffer layer [27–35]. However, the effect of substrate temperature on the physical and electrical properties is not yet reported. Mn will act as an acceptor ion that suppresses the formation of Fe²⁺ ion while La doping on Bi site suppress the formation of oxygen vacancies. Therefore, co-doping by La and Mn will improve the electrical properties of BFO thin films.

The present paper aims to investigate the effect of substrate temperature on the structural and electrical properties of BLFMO thin films deposited by pulsed laser deposition. The substrate temperature is a crucial factor that influences the various properties of thin films such as crystallinity, uniformity, and electrical properties [36–38]. Further, the dielectric and ferroelectric properties of the BLFMO films are evaluated and correlated to the microstructure of the films. Leakage current characteristics of thin films are dependent on deposition conditions, composition, and the microstructure. Understanding the current
transport mechanism is crucial to improving the device quality. Here, we have explored the leakage current mechanism of BLFMO thin film to gain insight into the conduction mechanism.

2. Experimental

BLFMO thin films were deposited on Pt (111)/TiO2/SiO2/Si substrate by pulsed laser deposition (PLD) system (EXcel Instruments, Mumbai, India). The laser source having wavelength 248 nm and pulse duration 25 ns was used for deposition (COMPexPro 102 KrF laser, Lambda Physik, Germany). Targets for PLD were synthesized by PVA sol-gel method [39] followed by microwave sintering to achieve density $\geq 93\%$. BLFMO thin films were deposited at different substrate temperature such as 450, 500, 550, 575, 600 and 650 °C. Oxygen gas pressure, laser fluence and the substrate to target distance were kept constant at 0.7 Pa, 1.5 J/cm² and 4.5 cm respectively. The phase purity of the samples was examined using X-ray diffractometer (PANalytical X’pert PRO) in the scanning range from 20° to 60° at a scan rate of 0.01°/30 s. Surface morphology of the thin films was studied using atomic force microscopy (Veeco Nanoscope IV). The thickness of the films is ~250 nm. The electrical characterization was performed by using Metal-Insulator-Metal (MIM) structure with the top gold electrode having thickness ~100 nm. Novocontrol’s high-resolution “Alpha A” analyzer was used for the measurement of relative permittivity, loss tangent and ac conductivity ($\sigma_{ac}$). Polarization-Electric field loops were measured using TF 2000 loop tracer (aixACCT GmbH). The I-V characteristics of BLFMO thin films were measured by using Keithley 2635B source meter. The Time-of-Flight SIMS (TOF-SIMS) data was acquired using a PHI TRIFT V nanoTOF™ instrument from ULVAC-Physical Electronics, MN, USA. 7 keV Cs⁺ ion beam was scanned in a raster size of 200 $\mu$m × 200 $\mu$m in a dual beam mode for sputtering. The crater depth was measured by Dektak XT from Bruker.

3. Results and discussion

The X-ray diffraction pattern of BLFMO thin films grown at different substrate temperatures is depicted in Fig. 1. All films are polycrystalline in nature, and BLFMO is indexed in the pseudocubic unit cell. The purity phase marked by * in the XRD pattern is identified as Bi2O3 and matches with the JCPDF file. 01-074-1373. BLFMO thin films deposited at all substrate temperature have random orientation in nature. The splitting of the (110) peak is not observed in all XRD patterns of thin films. This is due to the decreasing of the monoclinic tilting present in the pure BFO lattice [40].

The surface morphology of all samples have been studied by atomic force microscopy, and the 2D images are represented in Fig. 2. The surface of the films appears crack free, fine-grained and uniformly distributed. The films deposited at 650 °C shows clusters of grains on the film surface. This is because higher energy/temperature on the film surface leads to a reaction between different species and results in rough surface.

The variation of grain size and roughness as a function of substrate temperature is depicted in Fig. 3. It is observed that average grain size increases from 45 nm to 160 nm and the surface roughness of sample increases from 2.5 nm to 9.4 nm as the deposition temperature
increases from 450 °C to 650 °C. This is mainly because, at low substrate temperature, the film growing species does not have enough surface mobility to find out the minimum energy sites and get adsorbed immediately when they arrive on the substrate. Whereas at high temperature, the surface mobility of adsorbed species increases and thus leads to an increase in grain size.

The relative permittivity and tan δ for BLFMO thin films deposited at different temperatures are measured and represented in Fig. 4(a, b). The relative permittivity and loss tangent decreases below 1 kHz. The increase in the relative permittivity at a lower frequency is due to space charges and defects present in the sample. It can be observed that for the substrate temperature below 575 °C, the samples are having large number of space charges and defects as the permittivity increases very rapidly below 1 kHz. This is fairly understandable as the lower temperature does not provide enough surface mobility to the growing species and leads to defects in the film. However, as the frequency increases above 1 kHz, the space charges and defects do not respond to varying electric field and thus the dielectric constant saturates. The relative permittivity decreases from 243 to 131 while tan δ changes from 0.29 to 0.06 at 1 kHz frequency as substrate temperature increases from 450 °C to 650 °C. As observed from Fig. 4(c) that the samples deposited at 575 °C and 650 °C shows almost the similar behavior but when measured other electrical properties, samples deposit at 650 °C could not meet the desired result which is explained in later sections. It is also observed that for BLFMO thin films, both εr' and tan δ decreases with increasing frequency with the absence of a peak in loss tangent. According to Jonscher et al., this kind of behavior is due to hopping conductivity [41]. The loss tangent decreases with substrate temperature except at 550 °C where it increases sharply to 1.26.

The relative permittivity of BLFMO thin films decreases as the substrate temperature increases as shown in Fig. 4(c) which is contrary to the reported literature [36,42–44]. There are several factors responsible for such behavior. As the oxygen vacancies are the primary cause of conduction and generates at higher temperatures due to the volatility of Bismuth or the transformation from Fe3+ to Fe2+ may lead to such behavior. Further, with an increase in substrate temperature, grain size increases with a decrease in grain boundaries. This increases intergranular contacts and increases the conductivity. Another plausible reason for the decrease in the relative permittivity is the formation of a dead layer at the interfaces as reported by Lee et al. for BFO thin films [45].

Fig. 3. Variation of grain size and surface roughness for BLFMO samples deposited at different substrate temperature.

Fig. 4. Variation of (a) relative permittivity (εr') and (b) loss tangent (tan δ) with frequency deposited at different temperatures, (c) variation of relative permittivity and tan δ at 1 kHz with substrate temperature.
There is possibility of Bi-Pt alloys formation at substrate and film inter-
face [8,46]. As reported by Okamoto et al. Bi will react to Pt and forms
Bi2Pt and different alloys due to the low vapor pressure of Bismuth
[47]. They have also provided the phase diagram of Bi and Pt and it is
clear that different phases of Bi and Pt are formed in between the tem-
perature 269 °C to 660 °C. The reactivity is higher at higher temperature;
thus it favors the formation of dead layer. Therefore, to investigate inter-
face quality, a SIMS analysis of the BLFMO/Pt interface is carried out, and
the composition pro
fi
le is depicted in Fig. 5. The SIMS depth pro-
fi
le of the BLFMO/Pt thin film deposited at 575 °C exhibits considerable out-
diffusion of Pt ions with the BLFMO film. The depth profiles also reveal
that each component of BLFMO films exhibits a uniform distribution ex-
cept at the interface between the BLFMO and the Pt electrode. The composi-
tional profiles near the BLFMO/Pt interface are not sharp at the interface suggesting diffusion of Pt at BLFMO/Pt interface and thus
forming a dead layer at the interface. The formation of dead layer at the
interface will reduce overall capacitance of the sample and hence relative permittivity decreases as the growth temperature increases.

The ferroelectric hysteresis curves for BLFMO thin films deposited at
various substrate temperatures are exhibited in Fig. 6. The samples de-
posited at 450 °C and 650 °C exhibit very poor ferroelectric properties.
As the substrate temperature increases up to 575 °C, both remnant
polarization and coercive field increase and thereafter decrease. Further,
the high-quality P-E loop is observed at 575 °C with the highest polariza-
tion of 39 μC/cm² at 400 kV/cm.

It is observed that the remnant polarization in the optimized sample
deposited at 575 °C is lower than reported by Wang et al. [33]. The fer-
roelectric polarization is the contribution of ferroelectric domain
switching, electrical conductivity and linear resistive components [48].
In our case, the dead layer at the interface reduces the overall capaci-
tance of the sample and hence the ferroelectric polarization decreases.
The variation of remnant polarization and coercive field with substrate
temperature is depicted in Fig. 7 (a). At higher temperature, higher oxy-
gen vacancy concentration results in more Fe²⁺ ions [49]. These oxy-
gen vacancies create domain wall pinning and thus decrease the remnant polarization. To check the reliability of the BLFMO sample for memory applications, fatigue test has been carried out till 10⁸ cycles on the optimized sample namely deposited at 575 °C. It can be depicted from Fig. 7 (b) that only 4% of the remnant polarization decreases till 10⁸ cycles. The good fatigue endurance is due to reduction of leakage current by La and Mn doping.

Impedance spectroscopy is a versatile tool to analyze the transport
mechanism behavior between grain and grain boundaries in the sam-
ple. To decipher the contributions of grain and grain boundaries in
BLFMO thin films, we have used impedance spectroscopy. Complex im-
pedance plots (Z’ vs. Z”) for BLFMO thin films grown at different sub-
strate temperatures are exhibited in Fig. 8. The dotted line represents measured data while the solid line shows the fitted data by the equiva-
 lent RC circuit. For electrical circuits having one parallel series compo-
nents, impedance can be expressed by the mathematical equations
given by equation,

\[ Z' = \frac{R_g}{1 + (\omega R_g C_g)^2} \]
\[ Z'' = R_g \left[ \frac{\omega R_g C_g}{1 + (\omega R_g C_g)^2} \right] \]

where, \( \omega \) is the angular frequency. Rg and Cg are grain resistance and
grain capacitance respectively.

Complex impedance data of BLFMO thin films deposited with differ-
ent substrate temperature is fitted using the Eq. (1). The presence of
only one semicircle in the complex impedance plot indicates the homo-
geneous electrical microstructure i.e. grain and grain boundaries have
same electrical properties. This rules out the effects of grain boundaries

\[ E (kV/cm) \]
\[ P (\mu C/cm²) \]

**Fig. 6.** Polarization-Electric field loops for BLFMO thin films deposited on Pt (111)/Ti/SiO₂/Si substrate at different substrate temperatures.
and electrode thin film interface. Fig. 9 (a) depicts the variation of $R_g$ and $C_g$ values obtained by fitting the complex impedance plots. The resistance ($R_g$) of the thin films increases with increase in substrate temperature with a sudden dip at 550 °C. The extracted capacitance ($C_g$) values are of the order of nano Farad and decrease from 1.11 to 0.5 nF as the substrate temperature increases. The equivalent circuit is for understanding the variation of impedance is represented in Fig. 9 (b).

The electrical conductivity of a dielectric material is a combination of contributions from lattice energy, intrinsic charge carrier’s density, impurities, and defects. BLFMO thin films may contribute to electrical conduction via ionic conduction (intrinsic and extrinsic), electronic conduction (electron charge hopping) and space charge conduction (electrode effect). The relation between conductivity and charge carriers can be given by Jonscher power law [50]

$$\sigma_{ac} = \sigma_{dc} + A\omega^n; (0 \leq n \leq 1)$$

where $\sigma_{dc}$ is the extrapolated dc conductivity, $A$ is material-specific constant, and $n$ is temperature and frequency dependent exponent factor. The electrical conductivity is measured with respect to frequency for various samples, and then the curves are fitted using Jonscher law are shown in Fig. 10 (a).

In BLFMO thin films, ac conductivity has a strong dependence on frequency. AC conductivity decreases with a decrease in frequency and becomes static at a low-frequency region called as a plateau region. The value of $n$ can reveal information regarding different conduction mechanisms that may exist in the thin films. In BLFMO thin films, it is observed that $n < 1$ (Fig. 10 (b)), and is attributed to conduction due to the translational hopping of the charge carriers. The dc conductivity after fitting by the Eq. (2) is plotted in Fig. 10 (b) and is found to increase by an order of magnitude with substrate temperature. In BLFMO thin films the dc conductivity first increases then decreases at 575 °C. All the experimental parameters are summarized in Table 1.
It is observed that, as the substrate temperature increases the thin films roughness increases. This may increase the leakage current [51]. The films deposited at 575 °C have smaller grain size as compared to the films deposited at a higher substrate temperature. Smaller grains are effective in blocking the leakage current paths. This reduces the leakage current in BLFMO thin films [52]. The leakage current density of BLFMO thin films deposited at various substrate temperatures is represented in Fig. 11 (a). Below 575 °C, the BLFMO thin films show higher leakage current and this may be due to the presence of vacancies/defects due to Bi/Fe ion reduction as the optimized growth windows of BFO thin films is very small [15,16]. The leakage current density first decreases to $4.37 \times 10^{-6}$ A/cm$^2$ at 575 °C and subsequently above this temperature, it increases again (Fig. 11 (b)). The leakage current density recorded in our sample is improved and lower than that of the reported in literature [28,30,33].

To find out the leakage current mechanism present in the sample, the J-E curves were fitted to various models. The leakage current mechanism can be broadly divided into two categories, such as bulk limited conduction namely space-charge-limited-conduction (SCLC) and Poole-Frenkel (PF) emission. The Schottky emission and Fowler-Nordheim (FN) tunneling are categorized into interface limited conduction mechanism.

The current density of the SCLC is represented as,

$$J_{SCLC} = \frac{\mu \varepsilon_r \varepsilon_0 E^2}{8d}$$

(3)

where, J is the current density, E is the electric field, $\mu$ is the carrier mobility, $\varepsilon_r$ is the relative dielectric permittivity, $\varepsilon_0$ is the permittivity of free space, and d is the film thickness.

Fig. 11 (c) depicts the log (J) vs. log (E) plots at the low electric field which represents the SCLC mechanism. The slopes are in the vicinity of 1 at low electric fields, indicating Ohmic conduction behavior. The slopes for 450 °C is equal to 3.3 and 650 °C (\sim 2.1 kV/cm) is equal to 18.63 respectively. With increasing electric field, the electrons get injected into the film and their density increases than that of thermally stimulated electrons. This leads to higher conduction in the sample. These slopes also do not satisfy modified Childs law ($J \propto E^2$), according to which the slope should be in the order of 2. So the current mechanism cannot be fully explained by SCLC mechanism. Therefore, in order to identify the leakage current mechanisms in the high electric field region, bulk limited Poole–Frenkel and interface limited Schottky emission and Fowler-Nordheim (FN) tunneling are considered for evaluation of the leakage behavior.

Poole-Frenkel emission which is described by the following equation,

$$J_{WF} = B E \varepsilon_0 \sqrt{\varepsilon_r \varepsilon_0 \varepsilon_{opt} K_B T}$$

(4)

where $\varepsilon_{opt}$ is the optical dielectric permittivity, B is the constant and $E_t$ is the trap ionization energy. $K_B$ is the Boltzmann constant, $q$ is the electronic charge and E is the electric field at the metal/insulator interface.

Fig. 9.

(a) Resistance and capacitance values estimated after fitting equivalent circuit, (b) equivalent circuit used for the fitting of experimental data.

Fig. 10.

(a) Variation of ac conductivity with frequency for BLFMO thin films deposited at different substrate temperatures, (b) dc conductivity and $n$ as a function of substrate temperature for BLFMO thin films.
move freely into the crystal and hence does not require large thermal fluctuations.

Interface limited Schottky emission arises from thermally or field activated electrons passing through Schottky barrier, which is a contact barrier between metal and semiconductor or insulator resulting from different Fermi levels between them. The equation for leakage current density for Schottky emission is given by,

\[ J_s = A T^2 e^{-\frac{\phi}{kT}} \]

where,

\[ A = \frac{4\pi q m^* k^2}{h^3} \]

where, \( J_s \) is the current density, \( h \) is the Planck’s constant, \( \phi \) is the barrier height at the cathode, \( A \) is the Richardson constant and \( m^* \) is the effective electron mass.

The dominating leakage current mechanism could be identified by plotting \( \ln(J/E) \) vs. \( E^{1/2} \) and \( \ln(J) \) versus \( E^{1/2} \) for Poole-Frenkel and interface limited Schottky emission (graph not shown here). If the Schottky and PF emission occur in the samples, the \( \ln(J) \) versus \( E^{1/2} \) and \( \ln(J/E) \) versus \( E^{1/2} \) relationships should be linear with a slope of \( \sqrt{q^3/(kT^2)}\varepsilon_{\text{opt}} \) and \( \sqrt{q^3/(kT^2)}\varepsilon_{\text{opt}} \) respectively. Using this slope \( \varepsilon_{\text{opt}} \) can be derived to determine whether these two emissions occur. The refractive index of BFO is 2.5, so the optical permittivity of \( \varepsilon_{\text{opt}} = 2.5^2 = 6.25 \) is expected [53]. The \( \varepsilon_{\text{opt}} \) values observed from both PF and Schottky emission are found to be in the range of 0.2 < \( \varepsilon_{\text{opt}} \) < 34.5 (except ~6.25). Therefore, in BLFMO thin films, both the PF and Schottky emission are ruled out because \( \varepsilon_{\text{opt}} \) deviates largely from the theoretical value.

Further, interface limited FN tunneling is considered to find out contribution to leakage current at high electric field which may be described by the following equation,

\[ J_{FN} = C E^2 e^{-\frac{\phi}{kT}} \]

where \( C \) is the constant and \( \phi \) is the potential barrier height.

To find out the contribution from FN tunneling \( \ln(J/E^2) \) vs. \( E^{-1} \) is plotted as shown in Fig. 11 (d). For FN tunneling to dominate, the \( \ln(J/E^2) \) vs. \( E^{-1} \) slope should be linear with a negative slope [54]. All the BLFMO thin films shows a negative slope of \(-100\) except for the sample

<table>
<thead>
<tr>
<th>Substrate temp (°C)</th>
<th>( \varepsilon_r' ) (at 1 kHz)</th>
<th>tan ( \delta ) (at 1 kHz)</th>
<th>( \sigma_{ac} ) at 1 kHz (S/cm)</th>
<th>( \rho_{dc} ) (Ω·cm)</th>
<th>( P_r ) (µC/cm²)</th>
<th>( E_c ) (kV/cm)</th>
<th>( J @ 200 \text{ kV/cm} ) (A/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>450</td>
<td>243</td>
<td>0.29</td>
<td>4.37 × 10⁻⁸</td>
<td>4.95 × 10⁷</td>
<td>0.8</td>
<td>57.57</td>
<td>1.01 × 10⁻³</td>
</tr>
<tr>
<td>500</td>
<td>221</td>
<td>0.25</td>
<td>3.46 × 10⁻⁸</td>
<td>7.89 × 10⁷</td>
<td>4.96</td>
<td>272.79</td>
<td>1.90 × 10⁻³</td>
</tr>
<tr>
<td>550</td>
<td>214</td>
<td>1.3</td>
<td>1.82 × 10⁻⁷</td>
<td>6.09 × 10⁸</td>
<td>34.81</td>
<td>286.57</td>
<td>4.40 × 10⁻⁵</td>
</tr>
<tr>
<td>575</td>
<td>140</td>
<td>0.06</td>
<td>5.81 × 10⁻⁸</td>
<td>2.50 × 10⁹</td>
<td>38.51</td>
<td>400</td>
<td>4.37 × 10⁻⁵</td>
</tr>
<tr>
<td>600</td>
<td>194</td>
<td>0.12</td>
<td>1.51 × 10⁻⁸</td>
<td>4.09 × 10⁹</td>
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<td>275.96</td>
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<tr>
<td>650</td>
<td>131</td>
<td>0.06</td>
<td>5.50 × 10⁻⁹</td>
<td>2.35 × 10⁹</td>
<td>1.68</td>
<td>52.7</td>
<td>4.40 × 10⁻⁴</td>
</tr>
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</table>

Fig. 11. (a) Leakage current density as a function of electric field for BLFMO deposited at various substrate temperatures, (b) Variation of Leakage current density (J) with respect to substrate temperature at 200 kV/cm. Leakage current density fitted by (c) space charge limited conduction (SCLC) mechanism, (d) Fowler-Nordheim (FN) tunneling conduction mechanism for BLFMO thin films.
deposited at 450 °C where the slope is — 57. The different slopes and onset voltages indicate that injection of charge carriers into the films at a different rate and different excitation voltages. The thin films deposited at 450 °C exhibits straight line with onset voltages of 90 kV/cm. The film deposited at 650 °C shows fast conduction with onset voltage of 125 kV/cm. Therefore, it can be concluded that SCLC and FN tunneling conduction mechanisms dominate the leakage current behavior in BLFMO thin films.

4. Conclusions
La and Mn co-substituted BFO thin films have been grown using PLD and subsequently electrical properties are studied as a function of substrate temperature. The permittivity decreases as the substrate temperature increases, this may be either due to the presence of secondary/impurity phase or due to the formation of dead layer at the BLFMO/Pt interface. AC conductivity data has been fitted using Jonscher single power law, and value of n found to be < 1 indicating translational hopping conduction mechanism which also explain the absence of a peak in loss tangent while ε‘ and tan δ decreases as a function of frequency. Ferroelectric polarization increases till 575 °C and thereafter decreases as a function of deposition temperature except at 550 °C. At higher temperature increases, this may be either due to the presence of secondary/impurity phase or due to the formation of dead layer at the BLFMO/Pt interface. AC conductivity of the deposited BLFMO thin films deposited at 450 °C exhibits straight line with onset voltage of 90 kV/cm. The film deposited at 650 °C shows fast conduction with onset voltage of 125 kV/cm. Therefore, it can be concluded that SCLC and FN tunneling conduction mechanisms dominate the leakage current behavior in BLFMO thin films.

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