**Room Temperature Weak Multiferroism and Magnetodielectric Effect in Highly
Oriented $(Y_{0.9}Bi_{0.1})(Fe_{0.5}Cr_{0.5})O_3$ Thin Films**

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Abstract

High quality (100) oriented thin films of $(Y_{0.9}Bi_{0.1})(Fe_{0.5}Cr_{0.5})O_3$ [YBFCO] were deposited on (100) LAO substrates with conducting LNO buffer layer as bottom electrode. YBFCO thin film was found to demonstrate interesting room temperature multiferroic properties where the origin of weak ferroelectricity could be mainly attributed to disordered transition metal ions of unlike spins, and magnetic behaviour was ascribed to uncompensated weak ferromagnetism arises due to magnetic cluster state in disordered system. Dielectric anomaly near magnetization reversal temperature (T_{irr}) revealed magnetodielectric effect in YBFCO film. In addition, leakage current behaviour of the film was explained by trap-controlled space-charge-limited current conduction (SCLC) mechanism.

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1. Introduction

Room temperature multiferroic materials have attracted tremendous research interest due to their potential applications in sensors, spintronics, and non-volatile memories [1,2]. The single phase multiferroic materials are rare because of mutual exclusion between ferroelectricity and ferromagnetism at room temperature. Over the past few years, intensive research has been carried out to find alternative ways to combine ferroelectricity and magnetism in a single phase of material. Very recently, isostructural rare-earth ferrites (RFeO_3 , where R = rare earth) and rare-earth chromites (RCrO_3) have been widely investigated as promising single phase multiferroics [3,4]. RFeO_3 and RCrO_3 have a centrosymmetric orthorhombic perovskite structure (space group $Pnma$) and exhibit a canted antiferromagnetism or weak ferromagnetism below Néel temperature (T_N) due to antisymmetric Dzyaloshinskii-Moriya (DM) interaction [3,5]. A recent report on YFeO_3 (YFO) ceramics, showed room temperature multiferroism with weak ferroelectricity and weak ferromagnetism below $T_N \sim 650\text{K}$ [3]. Whereas in YCrO_3 (YCO), weak ferromagnetism was observed below $T_N \sim 142\text{K}$, and the polarization ordering can be expected below $T_C \sim 470\text{K}$ [6,7]. Both of these perovskite oxide families showed magnetically driven ferroelectricity, although, the magnitude of observed polarization was found to be very less ($\sim \text{nC/cm}^2$) [3,8]. Taking the advantages of both these compounds to tune the ferroics ordering temperatures and tailoring the multiferroic properties, solid solutions of the type $\text{YFe}_{1-x}\text{Cr}_x\text{O}_3$ were studied by several research groups [8-10]. One such solid solution $\text{YFe}_{0.5}\text{Cr}_{0.5}\text{O}_3$ (YFCO) with 1:1 Fe:Cr composition gained considerable attention as potential application in spintronics due to the presence of bipolar switching of magnetization under low magnetic fields [10,11]. The weak ferromagnet YFCO showed interesting magnetic properties, such as first order spin-reorientation and temperature as well as magnetic field induced magnetization reversal [5,8,9]. Like end members, YFCO also

showed centrosymmetric orthorhombic structure ($Pnma$) and exhibit canted antiferromagnetism or weak ferromagnetism below Néel temperature (T_N). Few reports on ceramic YFCO demonstrated weak ferroelectric behaviour below the magnetic ordering temperature ($T_N = 260$ K) and magnetodielectric effect around T_N [8,10,11]. Based on the recent report by Rajeswaran et al., the ferroelectricity in YFCO was found to be two orders higher than YFO where the origin of ferroelectricity ascribed to disordered transition metal ions (i.e. Fe^{3+} and Cr^{3+}) of nonequivalent spins [8]. Magnetization reversal in YFCO was also reported, which can be attributed to the competition between the single ion magnetic anisotropy and the antisymmetric (DM) interaction [9]. Furthermore, YFCO showed very unusual zero magnetization at compensation temperature (T_{comp}) ~ 250 K, above which the weak uncompensated magnetic moment due to the random distribution of magnetic clusters of Cr and Fe was found to remains up to higher temperature ~ 550 K [12]. Recently, Bismuth (Bi) ion doped orthorhombic perovskite system $(La_{1-x/2}Bi_{x/2})(Fe_{0.5}Cr_{0.5})O_3$ has been reported by Vijayanandhini et al., which showed higher magnetic transition temperature (~ 450 K) as compared to undoped $LaFe_{0.5}Cr_{0.5}O_3$ (LFCO) (~ 270 K) [13]. The compensation temperature was found to decrease with Bi-ion concentration, whereas the effective paramagnetic moment and coercivity increased with Bi concentration [13]. Although, the authors studied detailed magnetic behaviour of Bi doped LFCO system, but the ferroelectric properties were not discussed, which could be further interesting because of $6s^2$ lone pair effect of Bi-ion, as observed in case of $BiMO_3$ ($M = Fe$ and Mn) multiferroics [14,15]. Despite of considerable number of reports on mixed oxides of orthoferrite and orthochromites ceramics, thin film studies on this material system are lacking in literature.

In this work, we fabricated YFO and YCO perovskites based mixed oxide thin films, where the Yttrium (Y) was replaced by specific amount of Bi-ion to take the advantage of its lone pair activity. Highly (100) oriented $(Y_{0.9}Bi_{0.1})(Fe_{0.5}Cr_{0.5}O_3)$ (YBFCO) thin films were

deposited by pulsed laser deposition technique on LaAlO_3 (LAO) (100) substrates containing conducting LaNiO_3 (LNO) buffer layer as bottom electrode. The room temperature multiferroic properties of YBFCO films were investigated. Piezoresponse force microscopic (PFM) studies were deployed to confirm the ferroelectric behavior of the film. DC magnetization measurements were carried out to explore the magnetic behavior of the film. Temperature dependent dielectric measurements were performed to study the dielectric response of the film. In addition, the electrical transport properties of YBFCO film were also discussed.

2. Experimental details

LNO and YBFCO films were deposited on commercially available (100) LAO substrate using pulsed laser deposition technique. A KrF pulsed excimer laser (248 nm wavelength) operating at a fluence of $\sim 1.8 \text{ J/cm}^2$ and repetition rate of 10Hz was used for ablation of single phase ceramic targets of LNO and YBFCO prepared by solid state reaction method. Firstly, the deposition chamber was evacuated to a base pressure of $\sim 10^{-6}$ torr, and $\sim 100 \text{ nm}$ thick oriented layer of LNO was deposited on the LAO (100) substrate at the fixed temperature of 650°C and in oxygen ambient at a partial pressure of $\sim 150 \text{ mTorr}$. In the second step of deposition, the YBFCO film of expected thickness $\sim 360 \text{ nm}$ was deposited on LNO coated LAO (100) substrates at the fixed temperature of 720°C and at an oxygen partial pressure of $\sim 200 \text{ mTorr}$. After deposition, YBFCO film was annealed at 750°C for 20 min in excess oxygen pressure of 400 mTorr to suppress the oxygen vacancy and/or enhance the crystal structure quality. To fabricate metal-insulator-metal (MIM) capacitor structure the Pt top electrodes of thickness $\sim 60 \text{ nm}$ and the diameter of $\sim 200 \mu\text{m}$ were deposited at room temperature by dc-magnetron sputtering. The structural properties of the films were investigated by recording x-ray diffraction pattern of the films using Rigaku Ultima *III*- x-ray diffractometer. The surface morphology and the thickness of the films were investigated by

atomic force microscope using multimode Nanoscope V (Veeco-AFM contact mode instrument) and field emission scanning electron microscope (FE-SEM, JEOL JSM-7500F), respectively. The energy dispersive x-ray (EDX) microanalysis was carried out in an average mode to confirm the composition of the film as well as target pellets. Ferroelectric properties were characterized using a RT6000 loop tester (Radiant Technologies) and piezoresponse force microscopy (PFM) using Co/Cr coated tip in multimode Nanoscope V. Macroscopic magnetic hysteresis loops of the YBFCO film were recorded with a vibrating sample magnetometer (VSM, Lakeshore 7400) at room temperature. Superconducting quantum interference device (SQUID, MPMS XL-7, Quantum Design) magnetometer was used to study the magnetization versus temperature behaviour of bulk YBFCO sample in field-cooled (FC) and zero field-cooled (ZFC) protocols. The temperature dependence of dielectric permittivity and dielectric loss were measured from 80-350K in the frequency range of 500 to 100 kHz using LCR meter (Agilent E4980A) interfaced with programmable Joule Thompson thermal stage system (MMR technology, model# K-20). The DC leakage current measurements were studied through current-voltage (I-V) measurements in top-bottom configuration using a Keithley-2401 source-measure unit.

3. Results and discussion

Figure 1(a) showed the room temperature X-ray diffraction patterns of LNO/LAO and YBFCO/LNO/LAO structures. In the 2θ range ($20-80^\circ$), one can see the respective peaks corresponding to (100), (200), and (300) planes of LNO and LAO [16]. The rest of the two peaks with less intensities are indexed as (200) and (400) planes of YBFCO corresponding to orthorhombic structure with Pnma space group (JCPDS#34-0365), which indicates highly a-axis oriented nature of YBFCO film. From EDX microanalysis of YBFCO film, the compositions observed were found to be in reasonable agreement with the nominal ones,

therefore confirmed the stoichiometric growth of the film (results were not shown). Further, the lattice strain in the film was calculated using the equation:

$$\varepsilon = \frac{a_{bulk} - a_{film}}{a_{bulk}} \times 100 \quad (1)$$

where, ε is the lattice strain, a_{bulk} is the measured lattice constant of the bulk YBFCO, and a_{film} is the lattice constant of the film measured from XRD-pattern. It is found that the film has 1.8% in-plane strain. The in-plane strain could be thermally generated, because the lattice mismatch between LNO buffer layer and film was found to be insignificant (less than 0.3%). The AFM measurements were employed to study the surface morphology of the film over $2 \mu\text{m} \times 2 \mu\text{m} \times 30 \text{ nm}$ Z-scale area, as shown in the inset of Fig. 1(a), showing nearly smooth and homogenous surface of the film with the root mean square (rms) roughness of $\sim 2.5 \text{ nm}$. Cross-sectional SEM image of YBFCO/LNO/LAO structure was depicted in Fig. 1(b). From the cross-sectional view, one can distinguish the LAO substrate, LNO, and YBFCO layers where the thickness of the YBFCO film and LNO buffer layer were confirmed to be ~ 350 and $\sim 120 \text{ nm}$, respectively [see Fig. 1(b)]. Figure 1(c) showed the schematic diagram of Pt/YBFCO/LNO metal-insulator-metal capacitor structure used for ferroelectric and transport measurements.

The room temperature ferroelectric properties of the (100) oriented YBFCO films were evaluated. Figure 2(a) showed P-E hysteresis loops measured on Pt/YBFCO/LNO capacitor structure at different applied voltages at room temperature. The remanent polarization (P_r) value of $\sim 1.8 \mu\text{C}/\text{cm}^2$ at a maximum applied field of $\sim 575 \text{ kV}/\text{cm}$ was found, which suggests the weak ferroelectricity in YBFCO thin film. Butterfly nature of the C-V curve confirms the room temperature ferroelectric nature of the film, as shown in Fig. 2(b). To further corroborate the ferroelectric nature of YBFCO thin film, the PFM studies were carried out. Figure 2(c) shows the polarization reversal in YBFCO film with square pattern of the

two opposite polarization domains which were confirmed by the different colour contrast while applying DC tip bias of ± 12 V. The room temperature out-of-plane PFM (OP-PFM) phase hysteresis loop and amplitude signals versus voltage curves again confirm the ferroelectric nature of YBFCO film, as shown in Fig. 2(d). However, it can be noticed that the hysteresis loop shows an offset due to the stress-induced changes in the imprint and switching behaviour in Pt/YBFCO/LNO capacitor [17]. One possible reason behind the polar ordering in YBFCO thin films could be the cation disorder at the B-site by incorporation of two different magnetic ion [Cr^{3+} ($S = 3/2$) and Fe^{3+} ($S = 5/2$)] of nonequivalent spin [8]. The canted nonequivalent spins of Fe and Cr-ion arise by Dzyaloshinskii-Moriya (D-M) interaction produces canted antiferromagnetism or weak ferromagnetism in these mixed oxides [8]. In such canted antiferromagnetic oxides, the counterclockwise and clockwise spin helical arrangements are formed causing displacement of the oxygen atoms due to inverse D-M interaction which in turn induce local polarization. Such induced local polarization would cancel out due to the alternating arrangement of the spin pairs in case of equivalent B-site cations. If there is a random arrangement of different cations at the B-site than the oxygen displacements need not to be equivalent and thus can cause a net induced polarization. The observed polarization of oriented YBFCO thin film was found one order higher as compared to the bulk YFCO and YFO [3,8,18]. In spite of the cation disorder at the B-site, the role of the $6s^2$ lone pairs of Bi^{3+} cation could be a possible reason behind higher polarization observed in YBFCO films as compared to the YFCO ceramics [15]. Therefore, effect of Bi lone pair could be another possibility to justify the improved ferroelectric behaviour of YBFCO film.

After confirming the ferroelectricity, we carried out the room temperature magnetization measurements on YBFCO film. Figure 3(a) showed the magnetization versus field (M-H) curve measured at room temperature with the magnetic field perpendicular to the

film plane. The existence of a clear loop opening in M-H curve without the saturation of magnetization can be attributed to the canted antiferromagnetic (CAFM) or weak ferromagnetic ordering, as observed in YFO [3]. Further, the observed values of the coercive field ~ 550 Oe and the remanent magnetization ~ 2.5 emu/cc suggest a weak ferromagnetic behaviour of YBFCO film. This magnetic behaviour of the film can be explained by considering the random distribution of canted nonequivalent spins of Fe^{3+} and Cr^{3+} in the octahedral sites which leads to a distribution of the weak ferromagnetic components inside the material [12,19]. However, such magnetization behaviour can also be interpreted as ferrimagnetic like, possibly due to the dipolar interaction of Cr and Fe domains oriented in opposite directions [13]. Although, the lack of any published report on YFCO or LFCO thin films precludes any detailed comparative evaluation of magnetic behaviour. Nevertheless, the slight loop opening without the saturation of magnetization observed in M-H curve of ceramic sample, confirms the weak ferromagnetism in YBFCO system. Further, the temperature dependent DC magnetization measurements were carried out on ceramic sample to confirm the magnetic transition temperature of the material. Figure 3(b) showed the magnetization versus temperature (M-T) curves under FC and ZFC conditions in the temperature range of 5-350K, where the abrupt increase in the FC curve around the onset of the magnetic ordering temperature ($T_N \sim 302\text{K}$) confirms the presence of the weak ferromagnetic interaction [10]. A temperature induced magnetization reversal was observed around $T_{\text{irr}} \sim 268$ K. On further cooling the sample, the magnetization was found to decrease and showed zero magnetization at compensation temperature (T_{comp}) ~ 247 K below which the sign of magnetization changes to negative, indicating opposite direction of magnetization that of the applied field. The substitution of Cr ion at the Fe site leads the competition between the antisymmetric D-M interaction and the single ion magnetic anisotropy (mainly due to the Cr^{3+}) where the equal proportion of Fe and Cr ions makes such competition more

prominent [9]. As an effect of this competition, the net moments produced by the antisymmetric D-M interaction and the single ion magnetic anisotropy are remained in opposite direction with the different temperature dependence of magnetization. Therefore, negative magnetization takes place at low field (at 100 Oe) while decreasing the temperature in FC mode. In YBFCO, T_N was found to be higher than as observed in YFCO ceramic whereas a slight shift in compensation temperature was observed towards lower temperature possibly due to Bi doping effect, as observed by Vijayanandhini et al. in $(La_{1-x/2}Bi_{x/2})(Fe_{0.5}Cr_{0.5})O_3$ system [8,13]. Furthermore, temperature dependent dielectric measurements were carried out on Pt/YBFCO/LNO thin film capacitor structure. Figure 4(a) and (b) showed the temperature dependence (80-350 K) of the real part of dielectric constant (ϵ') and dielectric loss ($\tan \delta$) in the frequency range of 500-100 KHz. The YBFCO film showed high dielectric constant but low dielectric loss as compared to YFCO ceramics [8,10]. The dielectric constant versus temperature curves was found to exhibit a hump around T_{irr} . Such an anomaly near magnetization reversal temperature indicates the magnetodielectric effect in YBFCO thin film.

The current-voltage characteristics were performed to study the leakage current behaviour of the YBFCO film. Figure 5(a) depicted the variation of leakage current density (J) as a function of applied electric field (E) measured on Pt/YBFCO/LNO metal-insulator-metal structure. To clarify the conduction mechanisms of the film, the J - E curve in the positive voltage region was replotted in a log-log scale in Fig. 5(b). The $\log(J)$ - $\log(E)$ curve can be well fitted as linear segments with different slope values, as shown in Fig. 5(b). This kind of J - E behaviour can be explained based on the trap-controlled space-charge-limited current conduction (SCLC) mechanism [20-22]. According to trap-controlled SCLC mechanism, the linear J - E behaviour at low voltage region can be mainly ascribed to Ohmic conduction ($I \sim V$), whereas in the intermediate voltage region the conduction is dominated by

the Child's square law ($I \sim V^2$), and the steep increase in current is observed at high voltage region. The current density for trap-controlled SCLC emission can be expressed as follows [23]:

$$J = \theta(9/8)\epsilon_r\epsilon_o\mu \frac{V^2}{d^3} \quad (2)$$

Where, $\theta = (N_c/N_t) \exp - (E_c - E_t)/kT$ is the ratio of free electron to trap electron, N_c is the effective density of state in conductive band and N_t is the density of emptied electron traps, ϵ_r is the static dielectric constant, ϵ_o is the permittivity of free space, μ is electron mobility, V is the applied voltage, and d is the film thickness. In Child's law region, the density of free electrons due to carrier injection greatly exceeds the density of thermally stimulated electrons, and therefore, contributes extra current in the film. Electron traps are formed due to defects (mainly from oxygen vacancies) in perovskite films [21,22]. These traps are located below the conduction band and have tendency to absorb injected electrons [23]. On further increase of applied electric field to reach the value to fill the traps, large field square-law conduction takes place due to remaining excess charge carriers leading to drastic increase of the current. The voltage corresponding to drastic increase in current is defined as the so called trap filled limit voltage (V_{TFL}), which can be expressed as [20]:

$$V_{TFL} = \frac{eN_t}{2\epsilon_r\epsilon_o} d^2 \quad (3)$$

where e denotes the effective charge of electron. Thus, the conduction behaviour of YBFCO film can be explained on the basis of trap-controlled SCLC mechanism.

4. Conclusions

Multiferroic, dielectric, and electrical-transport properties of $(Y_{0.9}Bi_{0.1})(Fe_{0.5}Cr_{0.5})O_3$ (YBFCO) thin films have been studied. The YBFCO film exhibited room temperature

multiferroic properties with weak ferroelectric and weak ferromagnetic characters. Temperature dependent dielectric measurements (80-350K) at different frequencies showed an anomaly near magnetization reversal temperature (T_{irr}) $\sim$ 268 K, divulging magnetodielectric effect in YBFCO film. In addition, the conduction mechanism for the leakage current density observed in insulating YBFCO film was described by trap-controlled SCLC mechanism. Our studies open up the promising way to fabricate mixed rare-earth orthochromites and orthoferrites based high quality room temperature multiferroic thin films.

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Figure captions:

Fig. 1(a) X-ray diffraction patterns of (100)-oriented LNO buffer layer grown on LAO (100) substrate and YBFCO thin film on LNO/LAO, respectively. Inset shows the 3-D AFM image of YBFCO film. (b) The cross-sectional view of YBFCO/LNO/LAO structure and, (c) schematic diagram of Pt/YBFCO/LNO metal-insulator-metal capacitor structure.

Fig. 2(a) Room temperature P-E hysteresis loops of Pt/YBFCO/LNO capacitor under applied electric field of different amplitudes, (b) the butterfly nature of C-V curve, (c) phase-PFM image after applying ± 12 V, and (d) phase and amplitude PFM-signals versus voltage curves.

Fig. 3(a) Magnetization versus field (M-H) curve of YBFCO film at 300 K. Inset shows the M-H curve of ceramic YBFCO sample. (b) Temperature dependence of dc magnetization with ZFC and FC protocols for ceramic sample in an applied field of 100 Oe. Inset shows the zooming view around 270 K.

Fig. 4(a) Temperature dependence of (a) dielectric constant (ϵ'), and (b) dielectric loss ($\tan \delta$) of YBFCO film at different frequencies. Inset of (a) shows the dielectric anomaly around T_{irr} in a reduced scale.

Fig. 5(a) (a) Leakage current density (J) as a function of electric field (E) measured on Pt/YBFCO/LNO metal-insulator-metal structure, and (b) $\log(J)$ – $\log(E)$ plot fitted as linear segments into three different regions.

Fig. 1 (a)-(c)

Fig. 2 (a)-(d)

Fig. 3 (a) and (b)

Fig. 4(a) and (b)

Fig. 5(a) and (b)