Research Article

Electronic Structure of BiFe$_{1-x}$Mn$_x$O$_3$ Thin Films Investigated by X-Ray Absorption Spectroscopy

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Received 7 March 2012; Accepted 21 April 2012

Academic Editor: Weichang Hao

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Multiferroic polycrystalline BiFe$_{1-x}$Mn$_x$O$_3$ (0 ≤ x ≤ 0.3) thin films have been prepared on the Pt(111)/Ti/SiO$_2$/Si(100) substrates by pulsed laser deposition method. The influence of Mn substitution on the electronic structure and magnetic properties has been studied. X-ray diffraction spectroscopy shows that Mn substitution slightly modulates crystal structure of the BiFe$_{1-x}$Mn$_x$O$_3$ system within the same structural phase. According to Fe L edge X-ray absorption spectroscopy, Fe ions are found to be formally trivalent for doping amount x in BiFe$_{1-x}$Mn$_x$O$_3$. The enhanced magnetization by increasing Mn content is attributed to an alternation degree of hybridization between Fe 3d-O 2p and Mn 3d-O 2p orbitals, basing on the carefully examined Fe L and O K edge X-ray absorption spectroscopy. The crystal structural and the electronic structural results show a causal relation between them by demonstrating intrinsic mutual dependence between respective variations.

1. Introduction

Multiferroics are a group of materials which simultaneously show up several kinds of ferroic properties such as ferroelectricity, ferromagnetism, and ferroelasticity in the same phase [1]. In ferromagnetic-ferroelectric multiferroics, magnetization can be tuned by an applied electric field and electronic polarization by magnetic field [2]. The magnetoelectric (ME) effects of multiferroics imply important technological application perspectives in multifunctional devices like high-temperature transistor gate, memory device, and spintronics. Few multiferroics exhibit as natural occurring phases, examples of which include YMnO$_3$, PbVO$_3$, BiCrO$_3$, BiMnO$_3$ and BiFeO$_3$ (BFO) [3, 4]. Among them only the BFO exhibits ME effects at room temperature. Although the BFO is considered to be a room-temperature multiferroics, its ME effect is too low because of weak ferromagnetism. Two different approaches have been used for enhancing the ME effect in BFO. One is to bring the ferroelectric $T_C$ or ferromagnetic $T_N$ transition temperature to around room temperature by cationic substitutions [5]. The other is first to achieve phase transition from antiferromagnetic to ferromagnetic and then increase magnetic properties while retaining the ferroelectricity through strain modification [6, 7]. Among these, Mn doping is the most common method to achieve an enhanced magnetization. This is because the BiMnO$_3$—a Mn centered compound of BiMO$_3$ series—exhibits ferromagnetism below ~105 K and ferroelectric at ~450 K [8]. The BiMnO$_3$ has a large ferromagnetic magnetization, implying that the Mn doping in BFO improves the magnetization and enhances the ME effects more effectively than other element substitution. Mechanisms for the increasing magnetization in Mn-doped BFO systems have been discussed in terms of canting of the antiferromagnetically ordered spins by structural distortion [9], double exchange interaction induced by hole doping effects [10], and variation of Fe$^{3+}$ ion concentration [11]. Investigation in terms of electronic structure is a direct way to understand the macroscopic properties of materials, due to intrinsic relationship between the electronic structure and macroscopic properties.
In this work, we prepared Mn-substituted BiFe$_{1-x}$Mn$_x$O$_3$ (BFMO) thin films by pulsed laser deposition (PLD). The crystal structure, surface morphology, magnetic properties, and electronic structure of the Mn-doped BFMO systems were investigated via X-ray diffraction (XRD), scanning electron microscopy (SEM), Alternating Gradient force Magnetometer (AGM), and X-ray absorption spectroscopy (XAS). Origin for the enhanced magnetization of the BFMO system with increased Mn amount of doping is investigated in terms of their electronic structural changes.

2. Experiment

The BiFe$_{1-x}$Mn$_x$O$_3$ (0 ≤ x ≤ 0.3) films were prepared by PLD method on the Pt(111)/Ti/SiO$_2$/Si(100) substrates in a PLD chamber connected to the photoemission spectroscopy (PES) system at 4B9B beam line of the Beijing Synchrotron Radiation Facility. Ceramic BFMO targets of 20 mm diameter 2 mm thick for PLD were prepared by sintering the mixtures of Bi$_2$O$_3$ (99.99 at.%), Fe$_2$O$_3$ (99.99 at.%), and Mn$_2$O$_3$ (99.99 at.%) at 820°C in air, following the solid state reaction routine way. Slight excess of Bi$_2$O$_3$ has been added to compensate the preferential loss of bismuth during sintering. The XRD result of the target material indicates that the sample is in single phase of polycrystalline BFMO.

Before deposition, the Pt(111)/Ti/SiO$_2$/Si(100) substrate was preheated at 700°C to eliminate surface contamination. The substrate temperature and oxygen ambient pressure were maintained at 550°C and 5.6 Pa, respectively. During the deposition, the laser fluence was at 2.3 J/cm$^2$ and the pulse repetition rates 1.5 Hz. After deposition, all the films were annealed at same condition for 30 minutes and then transferred under a background pressure of ~10$^{-8}$ Pa to the PES chamber to carry out in situ XAS measurements. The overall energy resolution was 0.2–0.7 eV, depending on the photon energies. All the spectra were recorded at room temperature, and the XAS measurement was carried out with total electron yield (TEY) detection mode. After the in situ measurements, the films were taken for ex situ crystal structure characterization by XRD, surface morphology by SEM, and magnetic AGM measurements.

3. Result and Discussion

3.1. Structure and Microstructure Characterization. XRD spectra were recorded at room temperature on an (MSAL-XD2) X-ray diffractometer with a Cu K$_\alpha$ source ($\lambda = 1.5418$ Å) in Bragg-Brentano geometry. Figure 1(a) shows the X-ray diffraction patterns of BiFe$_{1-x}$Mn$_x$O$_3$ (x = 0, 0.1, 0.2, and 0.3) thin films deposited on Pt(111)/Ti/SiO$_2$/Si(100) substrate without buffer layers. The BFO films, which are directly deposited on the Pt-buffered substrates, often exhibit a polycrystalline structure with one or more secondary phases [12, 13]. In comparison, in this work all the films are in single-phase polycrystalline structure with rhombohedral space group (R3c) and no other peak related to secondary phases was detected. It is also interesting to notice that a varied Mn substitution results in the regulation of the crystal orientation of the BiFe$_{1-x}$Mn$_x$O$_3$ thin films. Figure 1(b) shows the enlarged XRD patterns of films in the 2-theta range 20~24. Relative to that of pure BFO film, the (100) peaks of Mn doped show a slight shift to higher angle with a similar peak shapes and structures, confirming that the Fe ion substitution by Mn in BFO films. Except the rigid shift of the peak positions of doped sample compare to the undoped one, and a slight modulation of the (100) peak width in the doping range, crystalline structure of BFO has been affected little by the substitutional doping of Mn, in agreement with the rhombohedral structure of BFO unchanged up to 30% of Mn doping [5, 10].

The morphological SEM images of the BiFe$_{1-x}$Mn$_x$O$_3$ (x = 0, 0.1, 0.2, 0.3) thin films on Pt(111)/Ti/SiO$_2$/Si(100) substrate are shown in Figure 2. The SEM image of undoped BFO film, in Figure 2(a), reveals nonuniform growth of grains with wide pores between grains and an averagely different grain sizes. The Mn doping significantly affects the morphologies of BFMO thin films (Figure 2(b), 2(c), and 2(d)), the films become denser, the grain sizes tend to be smaller in (b) and (c), the grain boundaries are blurred, and the pores between grains are reduced. Larger pores between the grains of undoped and 30% Mn-doped thin films are consistent with the weaker diffraction intensity of those films in Figure 1(a).

The XRD peaks and the SEM images, as direct indicators for atomic long-range ordering level and macroscopic morphology of crystal, both provide mutual-revealing clues on what has happened in atomistic level of the systems along with variational condition, such as doping in the underlying case. The grain size variation observed in the SEM images shows a consistent relation with the peak width in the XRD; that is, the larger the grain sizes are, the wider the observed XRD peaks are, though the latter at a poorer statistical level. When all the samples keeping the phase of crystal structure within the same R3c symmetry group, the facts such as the rigid peak positions shift of (100), gradual disappearance of peak (110), and the various morphological show-up of the crystal films against the Mn doping imply the systems undergone certain kind of electronic structural modulation, instead of the results from simple mixing of different radius ball-like elements.

Change in 2θ implies a change in d-spacing of lattice parameters. The (100) lattice parameter a and the diffraction 2θ angle have the relation $\propto 1/\sin \theta$ the small amount up-shift of the 2θ implies a decrease in the a with the Mn doping. The rigidity of the shift by reaching a fixed position all at once reflects that, first of all, the sensitivity of the electronic structure to the doping rather than the doping amount, secondly, the dopant distribution uniformity of the substitution. We attribute the d-spacing decrease and the rigid shift of 2θ to electronic structural effects, rather than a different size elemental mixing effects. If it was the latter effects, as it has been shown clearly for Fe-Al mixing in [14], the smaller size Fe substitution of larger size Al drove a continuous decrease of lattice parameters with their relative percentage, in the underlying case we would have observed a scenario where the lattice parameters were increasing and the 2θ degree decreasing continuously following the fact that
the Mn atom has a larger radius than that of Fe. All these are clearly in a contrary way of the above simple elemental situation. The disappearance of the (110) indexing surface with the Mn doping can be taken to be a direct result in the electronic structural regulation. This can be due to randomization, distortion or breakdown of the (110) surface, but certainly not due to, continuously changing grain sizes; this is not the case shown in the measured SEM images. The electronic structural effects in the underlying case are mostly by modulation of the hybridization interactions between Fe-O and Mn-O two pair elements, as they are discussed through the XAS results at Fe L edge and O K edge in the following sections. The observed electronic structural variations with doping provide an affirmative standing point for the XRD and SEM observations.

3.2. Fe L Edge X-Ray Absorption Spectroscopy. The Fe 2p XAS spectra of BFMO system in Figure 3(a), along with the spectra of reference trivalent Fe2O3 (Fe3+) and divalent FeO (Fe2+) oxides [15], provide information on the unoccupied Fe 3d states. The Fe 3d orbitals split by exchange interaction into majority (spin up \( \uparrow \)) and minority (spin down \( \downarrow \)) states. Each state further splits by the octahedral crystal ligand field into \( t_{2g} \) and \( e_g \) orbitals. In the Fe L edges XAS spectra, the lower energy shoulder of Fe \( L_1 \) corresponds to the unoccupied \( t_{2g} \downarrow \) states and the sharper one to the unoccupied \( e_g \downarrow \) states. Similar to \( L_3 \) edge, the \( L_2 \) edge also splits into two \( t_{2g} \downarrow \) and \( e_g \downarrow \) states. The energy separation between \( t_{2g} \downarrow \) and \( e_g \downarrow \) states is called octahedral crystal field 10Dq value (or the distance between the \( t_{2g} \) and \( e_g \)). Measured energy separation between \( t_{2g} \downarrow \) and \( e_g \downarrow \) states for all the samples is \( \sim 1.6 \) eV. This leads the Fe 3d electrons to stay at high-spin configuration \( t_{2g}^3 e_g^2 (^6A_{1g}) \), like the Fe 3d electrons in LaFeO3.

The line shape of the Fe 2p XAS provides information on the valence state of the Fe ions. The spectra show that the line shapes of all the doped samples are similar to that of Fe2O3 and quite different from that of FeO. These indicate that the Fe ions keep formally in trivalent (Fe3+) states with the doping, consistent with literature report [16].

The change in peak intensity and peak width reflects a modulation in the electron density of state distribution between the Fe 3d and ligand O 2p with doping. In Figure 3(b), the \( t_{2g} \downarrow \) peak weight estimated by the \( I_{t_{2g}} / (I_{t_{2g}} + I_{e_g}) \) intensity ratio and the \( L_3 \) edge FWHM (full width at half maximum) by arithmetic sum of \( t_{2g} \downarrow \) and of \( e_g \downarrow \) peaks’ FWHM show explicit variation trends. As \( x \) increases, the intensity of \( t_{2g} \downarrow \) state increases from 0.364 at \( x = 0 \) to 0.408 at \( x = 0.3 \); at the same time, the \( L_3 \) edge peak width shrinks from 1.63 eV to 1.41 eV. Origin for these variations can be understood in terms of the doping-driven hybridization strength modification between the Fe 3d and O 2p states. The starting point is put on the valence band state of the \( x = 0 \) undoped BFO system. The BFO system is assumed to be Fe 3d0 + Fe 3d6L (L represents the ligand hole state O 2p0L formed by a charge transfer from O 2p to Fe 3d) mixture states derived by the Fe 3d and O 2p hybridization. The Fe 3d6L state is induced by charge transfer from the ligand O 2p to Fe 3d. The \( t_{2g} \downarrow \) state intensity change indicates an increase of the Fe 3d6L portion with the doping and correspondingly implies a decrease of the Fe 3d6L charge transfer state. The simultaneous peak width narrowing can be discussed analogously to the intensity change as result of the hybridization strength decrease. The O 2p ligand valence band is of 5.5 to 6 eV wide [17]; a stronger hybridization effect in the pure BFO induces higher portion of Fe 3d6L in the Fe 3d6 + Fe 3d6L mixed state then gives the observed weaker intensity and larger peak width due to delocalization. At the same time, the reduced hybridization effect along the Mn doping brings about an increase of the Fe 3d6 portion in the Fe 3d6 + Fe 3d6L states. Main causes of the current observation at the Fe L edge, in a self-consistent way with an increased hybridization strength between Mn 3d-O 2p, are clearly seen affirmatively through the below O K edge XAS measurement results.
3.3. Oxygen K Edge X-ray Absorption Spectroscopy. The O K-edge XAS spectra, shown in Figure 4(a), are labeled a, b, and c. The relative intensity change of the peak a’s shoulder and the FWHM of the double-peak a, as shown in Figure 4(b), are obtained with the same way as that of the Fe 2p XAS results shown in Figure 3(b). The M 3d
\textsuperscript{n+1}L characteristic peak intensity increases from \(~0.1\) to \(~0.21\) with the Mn content change 0 through 0.3, and at the same time the peaks’ width decreases from 1.95 eV to 1.25 eV.

The O 1s XAS measurement provides holistic information on the O 2p hybridization with transition metals M 3d
\textsuperscript{n} + M 3d
\textsuperscript{n+1}L states where the M 3d
\textsuperscript{n} + M 3d
\textsuperscript{n+1}L represents the [Fe 3d
\textsuperscript{5} + Fe 3d
\textsuperscript{6}L] + [Mn 3d
\textsuperscript{5} + Mn 3d
\textsuperscript{6}L] mixture states. The spectral structure reflects the density of states of M 3d
\textsuperscript{n+1}L portion in M 3d
\textsuperscript{n} + M 3d
\textsuperscript{n+1}L mixed states, since the hole state L of O 2p characteristic orbitals results in the charge transfer via hybridization interactions between M 3d and O 2p. The a double-peak structure including the arrow pointing shoulder is of the O 2p characteristic M 3d
\textsuperscript{n+1}L part in the M 3d
\textsuperscript{n} + M 3d
\textsuperscript{n+1}L states and these states are about the shoulder t\textsubscript{2g} \textsuperscript{1} and the main peak eg \textsuperscript{1} states, respectively [18].

When the density of the hole-state L at ligand side is high, the higher the intensity of peaks’ a is observable. The band b is attributed to the hybridization of O 2p with Bi 6sp states and the c at \(~542\) eV to the hybridization of O 2p with Fe 4sp, Mn 4sp orbitals.

In this section we pay special attention to the double-peak a due to it is about the M 3d
\textsuperscript{n+1}L state. The above Fe 2p XAS results, which are for the Fe 3d
\textsuperscript{5} portion of Fe 3d
\textsuperscript{5} + Fe 3d
\textsuperscript{6}L mixed states, have reached the conclusion that the Fe 3d
\textsuperscript{5}L density of state decreases and correspondingly the Fe 3d
\textsuperscript{5} state increases with the Mn doping. Now at O K edge, we see that the M 3d
\textsuperscript{n+1}L characteristic peak intensity increases, and simultaneously the peaks’ width decreases with the Mn doping, as shown in Figure 4(b). At the O K edge, the increase of t\textsubscript{2g} \textsuperscript{1} characteristic M 3d
\textsuperscript{n+1}L intensity with Mn doping reflects the fact that available L states at the ligand side are increasing, and this is by an enhanced hybridization effect which drives the charge transfer from ligand to metal ions. The M 3d
\textsuperscript{n+1}L state includes contributions from both states Fe 3d
\textsuperscript{6}L \textsuperscript{2} and Mn 3d
\textsuperscript{6}L \textsuperscript{2}. In the underlying system, the decrease of Fe 3d
\textsuperscript{6}L \textsuperscript{2} density of state and correspondingly the increase of Fe 3d
\textsuperscript{5}L \textsuperscript{2} with Mn have been proved by the XAS results at Fe L edge. The intensity increase of the M 3d
\textsuperscript{n+1}L state at O K edge, then, uniquely corresponds to the increase of Mn 3d
\textsuperscript{6}L \textsuperscript{2} density of state, and simultaneously a decrease of Mn 3d
\textsuperscript{5}L \textsuperscript{2} state with the doping. The double-peak width
Figure 3: (a) Comparison of the Fe 2p XAS of BiFe$_{1-x}$Mn$_x$O$_3$ (0 ≤ x ≤ 0.3) with those of FeO and Fe$_2$O$_3$; (b) FWHM of $L_3$ edge and spectral weight of $t_{2g}$ in $L_3$ edge as a function of x.

decrease at the O K edge implies that the localization of Mn 3d$^5$L states is enhanced with the Mn doping. These results indicate a scenario that the Fe 3d-O 2p hybridization strength weakens with the enhanced Mn 3d-O 2p hybridization effect in the BFMO thin films.

3.4. Magnetic Properties. The magnetic hysteresis (M–H) curves of the films measured at room temperature are shown in Figure 5. The M–H curves indicate typical ferromagnetic characters. The pure BFO thin film shows a weak ferromagnetic behavior in slim magnetic loops (Ms) of 28.7emu/cm$^3$, since it is in a rhombohedral distorted perovskite structure and allows a weak ferromagnetic ordering due to canting of the spins [19]. The magnetization value of pure BFO films in this work almost same with other reports [7]. The Ms of the samples at x = 0.1, x = 0.2, and x = 0.3 are 31.2, 40.3, and 47.7 emu/cm$^3$, respectively (inset of Figure 5). These values are higher than those of their corresponding bulk samples [20]. This increasing trend of Ms through Mn doping agrees with that of other reports about BFMO thin films and ceramics [11, 21, 22]. The coercive field (H$_C$) decreases with the increase of Mn composition.

The increase in macroscopic magnetization with the increase of Mn content (inset of Figure 5) was attributed to either structural transition or exhibition of certain amount of Fe$^{2+}$/Fe$^{3+}$ mixed ions in films [9, 11]. XPS results (not shown here) and the Fe L edge XAS show formal Fe$^{3+}$ only composition and no trace of Fe$^{2+}$ demonstrating. From the XRD results, however, there is no clear evidence for a phase transition such as from rhombohedral to tetragonal or other phases with Mn doping. These imply the BFMO thin films magnetization enhancement could not be from the different valence states of Fe ions. It also does not originate from the misfit strain by different films thickness [7, 23], since all the BFMO films were grown with a same film thickness, that is, the films deposition rates monitored in situ by crystal oscillator during growth process. Enhancement of the Ms with Mn doping is actually due to change in the electron density of state distributions between Fe-O and Mn-O pairs through variation of hybridization interactions, as discussed in section below.
Concerning the magnetic property of doped ceramic system, in an early work on the role of covalency in magnetic property of an A-site-doped ABO$_3$ (e.g., La$^{3+}$ substitution with Sr$^{2+}$ in the LaMnO$_3$ perovskite-type manganite La$_{1-x}$Sr$_x$MnO$_3$ [24], Goodenough assumed that inhomogeneity in the bonding nature of the central cations Mn$^{3+}$ and Mn$^{4+}$ with anion O$^{2-}$ by forming a mixed state of ionic, covalent, or semicovalent bonds could be the major driving force for ferromagnetic property observed in the system. The basic idea behind is that if the Mn$^{3+}$–O$^{2-}$ bond has more ionic character and the Mn$^{4+}$–O$^{2-}$ with more covalency or semicovalency, the bonding inhomogeneity with greater separation for ionic bonding and shorter for covalence will ease the barrier of ferromagnetic moment in the Mn$^{3+}$(3d$^4$)–O$^{2-}$–Mn$^{4+}$(3d$^3$) chain. The observed ferromagnetic results in the underlying BFMO system in the Mn$^{3+}$(3d$^4$)–O$^{2-}$–Fe$^{3+}$(3d$^5$) chain, analogous to the case suggested for the Mn$^{3+}$(3d$^4$)–O$^{2-}$–Mn$^{4+}$(3d$^5$) chain, demonstrate a good agreement to the assumption, showing an increase of ionic bond character for Fe$^{3+}$–O$^{2-}$ as observed at Fe 2$p$ edge XAS and an increase of covalence bonding for Mn$^{3+}$–O$^{2-}$ at O $K$ edge XAS. Apparently, the increase of the magnetism in the underlying system with the Mn doping associates closely to the bonding inhomogeneity created in the Mn$^{3+}$(3d$^4$)–O$^{2-}$–Fe$^{3+}$(3d$^5$) chains with the Mn doping.

4. Conclusion

In summary, the polycrystalline multiferroic BiFe$_{1-x}$Mn$_x$O$_3$ (0 $\leq$ x $\leq$ 0.3) thin films were grown by PLD techniques. The X-ray diffraction spectroscopy shows Mn content up to x = 0.3 is in single phase having a rhombohedral crystalline structure. The crystal structural variation with Mn doping is attributed to the electronic structural modulation, instead of different size elemental mixing. Mn doping brings about an increase of the Fe 3$d^5$ portion in the Fe 3$d^5$ + Fe 3$d^5$ states and spontaneous decrease of Mn 3$d^4$ portion in the Mn 3$d^4$ + Mn 3$d^5$ mixed states. The observed increase in Ms with increasing Mn concentration was closely related with the electronic density of state distribution changes. Increase in the Mn concentration reduces the charge transfer between the Fe 3$d$ and O 2$p$ and leads to the bond inhomogeneity in the Fe–O–Mn chains; these change driven by the hybridization interactions are responsible for the magnetization enhancement, as well as the observed slight modulation in crystal structures.
function of Mn-doping level.
0.2, and 0.3. The inset shows the magnetization (Ms) plotted as a

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National Natural Science Foundation of China (Grant no. H91G750Y21, and
Academy of Sciences under Grant no. H91G750Y21, and
The authors acknowledge the financial support from Chinese

Acknowledgments
The authors acknowledge the financial support from Chinese
Academy of Sciences under Grant no. H91G750Y21, and National Natural Science Foundation of China (Grant no. 11164026).

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Figure 5: Magnetic hysteresis (M-H) curves measured at room
temperature for the BiFe\(_1-x\)Mn\(_x\)O\(_3\) thin films with x = 0, 0.1,
0.2, and 0.3. The inset shows the magnetization (Ms) plotted as a
function of Mn-doping level.

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