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Modulating light absorption and multiferroic properties of BiFeO₃-based ferroelectric films by the introduction of ZnO layer

Kexin Li, Wanyun Zhang, Kaixin Guo, Ruirui Cui, Chaoyong Deng * and Xu Wang *

Key Laboratory of Electronic Composites of Guizhou Province, College of Big Data and Information Engineering, Guizhou University, Guiyang 550025, Guizhou, People’s Republic of China

* Authors to whom any correspondence should be addressed.

E-mail: cydeng@gzu.edu.cn and xuwang@gzu.edu.cn

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Abstract

Pure bismuth ferrite (BiFeO₃, BFO) and ZnO thin films, as well as BFO/ZnO and ZnO/BFO composite thin films were successfully deposited by a sol-gel process on Pt/Ti/SiO₂/Si and FTO/glass substrates, respectively. The chemical composition, surface morphology, optical properties, and multiferroicity were systematically investigated. X-ray diffraction and electron microscopy measurements were used to determine the crystalline phase and to analyze the surface morphology. Evidently, the absorption edges of both BFO/ZnO and ZnO/BFO films show a redshift, broadening the absorption range. The leakage current density decreases with the introduction of ZnO, and the ferroelectricity was significantly improved of the bilayers. Thereinto, BFO/ZnO and ZnO/BFO show the highest saturate polarization ($2P_s$) of 46.7 μC cm⁻² and the maximum remanent polarization ($2P_r$) of 18.5 μC cm⁻², respectively. Meanwhile, the magnetization measurement revealed that both BFO/ZnO and ZnO/BFO exhibiting an enhanced magnetization, especially, BFO/ZnO displays the highest saturation magnetization ($2M_r$, 68.87 emu cm⁻³) and remanent magnetization ($2M_r$, 4.87 emu cm⁻³).

1. Introduction

BiFeO₃ (BFO), a unique single-phase multiferroic material exhibiting both ferroelectricity and ferromagnetism at room temperatures due to its high Curie temperature ($T_C \sim 1103$ K) and Néel temperature ($T_N \sim 643$ K) [1, 2], is widely studied for the varied functional characteristics such as ferroelectricity [3, 4], magnetism [5], multiferroism [6], optical functionality [7], high dielectric susceptibility [8] and piezoelectricity [9], etc, presents a great potential for pragmatic applications in nonvolatile ferroelectric memory devices [10], memristors [11], and photovoltaic devices [12]. Moreover, compared with most classical ferroelectric materials, BFO has a relatively lower bandgap ($2.18 \sim 2.67$ eV as reported) [13], large remnant polarization ($P_r, \sim 100$ μC cm⁻²) [14] and high transparency. Additionally, BFO presents a G-type antiferromagnetic order at room temperature due to the super-exchange interaction among the neighboring half-full d-shell of Fe³⁺ cations and O²⁻ anions [15]. However, the polycrystalline BFO thin films usually exhibit a low polarization due to the high leakage current caused by oxygen vacancies, conductive impurity phases, and valence fluctuation of Fe ions (from 3⁺ to 2⁺) [16, 17], at the meantime its weak ferromagnetism cannot be observed due to its space-modulated spin structure and long-wavelength of 62 nm [18], which limit its application in devices.

To obtain potential multiferroic and optical properties, special attention has been paid to the composite structure with other materials, such as BaTiO₃ [19], PbTiO₃ [20], and BiMnO₃ [21], which is an effective way to optimize the ferroelectric polarization and multiferroic characteristics of BFO [22]. Various studies have investigated the possibility of coupling BFO with metal oxide semiconductors like TiO₂ [23], Fe₂O₄ [24] and NiO [25], which are commonly used to modulate the built-in electric field in the BFO/semiconductor interfaces. By far, many characteristics have been found to be promoted in BFO-based composited structures including the optical, ferroelectric, magnetic with the effect of the interfaces of the heterostructures. Meanwhile,
zinc oxide (ZnO), a semiconductor oxide with a piezoelectric wurtzite structure and an irreversible spontaneous polarization [26, 27], has attracted great interest as a functional material due to the extensive active adsorption sites, superb electronic mobility, and tunable morphology [28]. In the visible region, ZnO exhibits a wide bandgap (~3.37 eV), high transmittance, high excitonic binding energy (60 meV), and meanwhile it presents excellent mechanical and thermal stability [29]. Therefore, it is of significant importance to investigate the feasibility of fabricating ZnO/BFO and BFO/ZnO thin films to improve the multiferroic and optical properties by solution–gelation (sol–gel) technique.

In this study, a simple one-step spin-coating and annealing process was used to prepare BFO, ZnO, BFO/BFO, and BFO/ZnO thin films, which possess enhanced ferroelectricity, magnetization and light absorption with the composition of the ZnO layer. The microstructure, surface morphology, ferroelectricity, magnetism, and optical bandgap of the thin films were discussed in detail.

2. Materials and methods

Preparation of ZnO precursor solution and thin films: Firstly, zinc acetate [Zn(CH3COO)2] (99.99%) was dissolved into 2-methoxyethanol [CH3OCH2CH2CH3] (99%), then ethanolamine was added to stabilize the solution. After stirring at 50 °C for 5 h, a transparent and faint yellow precursor solution with a concentration of 0.2 M was obtained. The solution was spin-coated at 4000 rpm for 30 s on Pt/Ti/SiO2/Si and FTO/glass substrates, respectively, and then preheated at 350 °C for 10 min. The spin coating and pyrolysis process were repeated to prepare ZnO thin films with suitable thicknesses. The last step was annealing the films at 600 °C for 1 h in the atmosphere.

Preparation of BFO precursor solution and thin films: Stoichiometric iron acetate [Fe(CH3COO)2] (90%) and bismuth acetate [Bi(CH3COO)3] (99.999%) were utilized as raw materials and dissolved into 10 ml propionic acid [CH3CH2COOH] (99.5%) solvent. The mixture was stirred at 70 °C for 10 min. Moderate ethanolamine was added as a stabilizer, and then another 6 h were spent stirring the mixture at room temperature. It is worth noting that 5 mol% excess Bi was used to compensate for evaporation during heating. The concentration of the precursor solution was adjusted to 0.2 M as well. Then BFO layers were deposited onto substrates, respectively, and then preheated at 350 °C for 10 min. The spin coating and pyrolysis process were repeated to prepare BFO thin films with suitable thicknesses. The last step was annealing the films at 600 °C for 10 min in air and then annealed at 500 °C for 30 min for crystallization.

Preparation of ZnO/BFO and BFO/ZnO thin films: The BFO/ZnO bilayers were fabricated by spin-coating BFO precursor solution on ZnO/substrate. In the same process, ZnO/BFO bilayer nanofilms obtained by depositing ZnO on BFO/substrate.

2.1. Measurements

An x-ray diffractometer (XRD, D/max-2500V Rigaku) with Cu–Kα radiation was used to determine the phase composition of the samples. The microstructure was measured using a scanning electron microscopy (SEM, Hitachi SU-8100). For the measurement of the optical absorption and transmittance spectra, an ultraviolet-visible spectrophotometer (Hitachi U-4100) was used. Several Pt electrodes with a diameter of 0.5 mm were deposited onto the surface of the films by ion sputtering to measure the electrical properties. An analytical multiferroic tester (MultiFerroic200 V, Radiant Technologies) was used to measure the ferroelectric and leakage current properties of the samples. The out-of-plane PFM measurements were performed using an atomic force microscope (AFM, Bruker MultiMode 8). The comprehensive physical property measurement system (PPMS DynaCool-9T, Quantum Design) without liquid helium was used to measure the magnetization of the thin films.

3. Results and discussion

3.1. Surface, interface and structure analysis

Figures 1(a), (c), (e), and (g) demonstrate the XRD patterns of BFO, ZnO, BFO/ZnO and ZnO/BFO thin films deposited on Pt/Ti/SiO2/Si substrates, respectively. At room temperature, BFO presents a rhombohedral crystal structure, belonging to the R3c space group, with a 111-oriented spontaneous polarization for the most part [29]. The diffraction peaks located at 22.5°, 31.8°, 32.1°, 39.6°, 46.0°, 51.7° and 56.5° in figure 1(a) correspond to the (012), (104), (110), (202), (024), (122), and (300) crystal planes of BFO respectively, matched well with the standard card (ICPDS No. 71-2494), and no secondary phases were detected. All the peak positions and their relative intensities are obviously well matched with the rhombohedral phase, whose lattice constants are: a = b = c = 5.588 Å, c = 13.867 Å. The XRD spectrum (figure 1(c)) of ZnO shows a well-defined diffraction peak at about 34.4° which corresponds to the (002) plane of ZnO (ICPDS No. 36–1451), whose lattice constants
are: \( a = b = 3.249 \text{ Å}, c = 5.207 \text{ Å} \). It can be demonstrated from this intensive and sole peak that the ZnO film shows a high crystallinity. Additionally, except for the substrate peaks shown in figure 1(e), only BFO and ZnO diffraction peaks are available, confirming that the BFO/ZnO bilayer has been deposited successfully on the substrate. The diffraction pattern of ZnO/BFO is in accord with that of BFO/ZnO thin film, in which all the peaks presented in BFO and ZnO single layers can be found. However, due to the higher temperature of crystallizing of ZnO than BFO film, the diffraction peak of impurity appeared in the XRD pattern of the ZnO/BFO composited structure. Figure 1(i) exhibits the preparation schematic diagram of the BiFeO\(_3\)/ZnO and ZnO/BiFeO\(_3\) bilayers deposited on FTO/glass substrates.

The surface morphologies and cross-sections of BFO, ZnO, BFO/ZnO and ZnO/BFO thin films were examined by scanning electron microscope (SEM). Figures 1(b), (d), (f) and (h) show the surface morphologies of the films and the inset of figures 1(f) and (h) display the cross-section of both BFO/ZnO and ZnO/BFO bilayer thin films, respectively. The thickness of the BFO/ZnO and ZnO/BFO composited films is about 280 nm, in which the thickness of the ZnO layer is 80 nm and the BFO layer is 200 nm. The surface morphology appears to be dense and crack-free, while clear interfaces between the films and the substrates can be observed from the cross-sections of the films. Compared with the single-layered ZnO and BFO, the BFO/ZnO, and ZnO/BFO composited structures presented denser surface morphologies. Meanwhile, it can be obviously seen in the cross-section graphs that the interface between BFO and ZnO is vaguer in the ZnO/BFO bilayer than which in the BFO/ZnO bilayer, and can be ascribed to the exchange and recombination of the ions at the interface of the composited structure and the consequent generation of the impurity caused by the higher formation temperature of ZnO layer.

### 3.2. Ferroelectricity and leakage current

Figure 2(a) presents the variations in the leakage current density (\( J \)) of BFO, ZnO/BFO, and BFO/ZnO in relation to the applied electric field (\( E \)). The bilayers composed of BFO and ZnO exhibit decreased leakage current density compared with BFO thin film. At 300 kV cm\(^{-1}\), the leakage current densities of BFO, ZnO/BFO and BFO/ZnO thin films are \( 2.98 \times 10^{-4} \text{ A cm}^{-2} \), \( 3.40 \times 10^{-5} \text{ A cm}^{-2} \) and \( 1.30 \times 10^{-5} \text{ A cm}^{-2} \), respectively. Due to the existence of oxygen vacancies and the promoted undulation of Fe\(^{2+}/\text{Fe}^{3+}\) valence cause by the electronic instability of the Fe ions, the BFO films present a large leakage current \([30, 31]\). On the contrary, the leakage current densities of ZnO/BFO and BFO/ZnO bilayers were found to be lower than that of the BFO thin film. As the ZnO layer was added, a prominent decrease in the leakage current density was observed, which can be put down to the movement blocking of the oxygen vacancies and electrons of the ZnO layer which acted as an
Figure 2. Ferroelectric properties of the BFO, BFO/ZnO, and ZnO/BFO thin films. (a) $E$-$J$ characteristic curves. (b) $P$-$E$ hysteresis loops. (c) The variation of the coercive electric field ($2E_c$), saturation polarization ($2P_s$), and leakage current density ($J$).

efficient insulator between BFO and the Pt substrates and electrodes [32]. In addition, the reducing the leakage current density may be caused by a potential barrier formed at the interface between BFO and ZnO, which blocked the conduction of carriers in the composite and 46.7 $\text{ef}$ BFO has increased slightly compared with BFO ferroelectric ZnO layer, which induces a significant insulator between BFO and the Pt substrates and electrodes. Therefore, the results of the ferroelectric measurements illustrated ascribed to the dense microstructure, reduced leakage current, improved crystallization, and the interface coupling between the two consisting layers. Therefore, the results of the ferroelectric measurements mainly illustrated that the composite structure of ZnO and BFO yields better ferroelectric properties than BFO thin film.

In addition, the observed larger $2E_c$ of ZnO/BFO composited structure can be attributed to the non-ferroelectric ZnO layer, which induces a significant effect in which the ferroelectric BFO layer’s coercive field is substantially increased as a result of the formation of stress and defects, particularly in the vicinity of the interface region. Moreover, the p–n heterojunction formed in the ZnO/BFO bilayer generated a depletion region between ZnO and BFO and formed the built-in electric potential at the interface of the composited structure. In this way, the tendency for the $P$-$E$ loop to shift is enhanced by the addition of ZnO [34].

3.3. Magnetically ordered materials

Figures 3(a) to (c) shows the hysteresis loops ($M$-$H$) of BFO, ZnO/BFO, and BFO/ZnO films tested at 300 K. The coercivity ($2H_c$), saturation magnetization ($2M_s$), and remanent magnetization ($2M_r$) of the films are presented in figure 3(d), and the specific values can be found in table 1. Evidently, the $H_c$, $M_s$, and $M_r$ of the composite thin films are all higher than those of BFO film, showing an enhancement of the ferromagnetism due to the introduction of the ZnO layer, which has largely modified the spin structure of the BFO layer in ZnO/BFO and BFO/ZnO films. Meanwhile, the magnetism incensement of BFO/ZnO is more remarkable than ZnO/BFO, which may be put down to the impurity and modified interface of the composited structure yielded during the formation of ZnO because of the high temperature. The ferromagnetic property is comparable to a recent work that fabricated Ho and Mn co-doping BiFeO$_3$ thin film with a $2M_s$ of about 61.6 emu cm$^{-3}$ and 23 emu cm$^{-3}$, respectively. [35] In addition, the temperature dependence of magnetic susceptibility was conducted in the range of 4 ~ 300 K under the condition of zero-field cooling, as presented in the insets of figures 3(a) to (c), and it is obvious that the magnetization decreases along with the temperature.

BFO possesses a magnetic structure that exhibits an antiferromagnetic order since its antiferromagnetic axis rotates along the crystal axis with a 62 nm long-wavelength period. Incidentally, an increase in magnetization is observed when the BFO grain size is smaller than 62 nm [36]. The BFO moment may be explained by the diffusion of Fe ions into the interface. Nevertheless, the synthesis temperature of our BFO is lower than 880 °C needed for Fe diffusion into ZnO [37]. Furthermore, we exclude the possibility of the diffusion of mixed-valence of Fe$^{2+}$/Fe$^{3+}$, because Fe$^{2+}$ ions are derived from oxygen vacancies within the films, Fe$^{2+}$ disappeared when the films exceeded 100 nm thick according to the previous study [38]. As a result, the magnetization observed in ZnO/BFO and BFO/ZnO films originates exclusively from BFO. Therefore, the antiferromagnetic order in BFO can be disrupted when it is compounded with ZnO suppressing the cycloidal spin structure and inducing a
magnetic moment, which partly comes from super-exchange interaction among the interfaces between d-shell of Fe$^{3+}$ cations and O$_2^-$ anions [15].

### 3.4. Bandgap analysis

To measure the optical performance of the BFO, ZnO, BFO/ZnO and ZnO/BFO films, the exact same structured films were deposited on FTO/glass substrates. The optical absorption and transmission spectra of the BFO, ZnO, BFO/ZnO and ZnO/BFO films prepared on FTO/glass were measured in the wavelength range of 200–800 nm, as depicted in figure 4. The optical transmittance properties correlate with the electronic structure and bandgap. As shown in figure 4(a), the UV–vis transmittance spectra of the ZnO, BFO, BFO/ZnO and ZnO/BFO films were investigated. There are three distinct regions divided in the transmittance spectra marked by 'I', 'II', and 'III', respectively: strong absorption, low transmission, and high transmission, and the transmittance of all the films decreases to zero at the wavelength of 270 nm. Furthermore, the absorption edges of both BFO/ZnO and ZnO/BFO films show a redshift compared with ZnO film. The composite thin films are highly absorbent in violet and blue regions of the spectra, which could be used for photovoltaic and optoelectronic devices. Moreover, the transmittance of BFO/ZnO and ZnO/BFO composite films decrease gradually with the decrease in wavelength.

![Figure 3. Ferromagnetism of the samples. (a) to (c) Hysteresis loops of BFO, ZnO/BFO, and BFO/ZnO thin films at 300 K, the insets are the temperature dependence of magnetic susceptibility. (d) The variation of the coercivity ($2H_c$), saturation magnetization ($2M_s$) and remanent magnetization ($2M_r$) of the films.](image-url)

Table 1. The coercivity ($2H_c$), saturation magnetization ($2M_s$), and remanent magnetization ($2M_r$) of the films.

| Films     | $2H_c$ (Oe) | $2M_s$ (emu cm$^{-3}$) | $2M_r$ (emu cm$^{-3}$) |
|-----------|-------------|------------------------|------------------------|
| BFO       | 146.81      | 35.81                  | 3.50                   |
| ZnO/BFO   | 154.88      | 45.02                  | 4.37                   |
| BFO/ZnO   | 183.97      | 68.87                  | 4.87                   |
of working wavelength. In the visible light region, the ZnO film possesses a high transmittance of 90%. Although the transmittance of both BFO/ZnO and ZnO/BFO are lower than that of ZnO films, an obvious enhancement compared with BFO thin film, revealing that composite with ZnO is an effective way to obtain a higher transmittance of BFO thin film.

Meanwhile, in the UV–vis absorption spectra, as presented in figure 4(b), the absorption region of BFO/ZnO film is wider and the excitation wavelength is extended into the visible region compared with ZnO film, and on the other hand, the intensity of light absorption of ZnO/BFO in the near UV region is improved compared with BFO film. Additionally, the estimated bandgap of the thin films were extrapolated from the linear part of the corresponding Tauc plots. As the band edge absorption increases rapidly, a direct bandgap characteristic can be inferred. The corresponding optical bandgap ($E_g$) of each thin film was estimated by classical Tauc law, as shown in the equation below [39],

$$ (a h \nu)^n = B(\nu - E_g) $$

where $\alpha$ is the absorption coefficient given by $A/t$ ($A$ is absorptivity and $t$ is the thickness of the film), $n$ is 2 as for a direct bandgap, $h \nu$ is the photon energy, $B$ is Planck constant, and $E_g$ is the bandgap. As shown in figures 4(c) to (f), the corresponding bandgap energies of ZnO, BFO, BFO/ZnO, and ZnO/BFO are estimated to be 3.25, 2.39, 2.37, and 2.88 eV, respectively. The measured bandgap of BFO and BFO/ZnO thin films is comparative to recent work, in which a bandgap of 2.39 eV was observed in BiFeO$_3$ thin film fabricated by Ceballos-Sanchez et al [40]. Obviously, in the visible light range, the BFO/ZnO heterostructure exhibits a stronger optical response, indicating the optical absorption of the compositied structure is better than that of BFO and ZnO, thus leading to a potential photocatalytic application in photovoltaic devices. Furthermore, the low bandgap enables the increase of the number of photons harvested from the broad solar spectrum and the reduction of the recombination of charge carriers.
4. Conclusion

In this study, BFO, ZnO, ZnO/BFO and BFO/ZnO films were successfully fabricated on Pt/Ti/SiO₂/Si and FTO/glass substrates by a sol-gel process. The XRD analysis revealed that the thin films have been prepared on the substrates successfully, while the SEM images demonstrated that the thin films have dense and smooth surface morphologies. According to the properties analyses, fabricating the composite structure was found to be a valid way to improve the ferroelectric, magnetic, and optical properties of BFO thin films. Compared with BFO film, the absorption edges of both BFO/ZnO and ZnO/BFO films show a redshift, indicating a potential application in optoelectronic devices. Additionally, the introduction of ZnO can reduce the leakage current density of the two bilayers. Among all the thin films, BFO/ZnO thin film shows the highest 2P, of 46.7 μC cm⁻². The strongest 2M₁ and 2M₂ are 68.87 and 4.87 emu cm⁻³, respectively. The ZnO/BFO thin film shows the maximum 2P, of 18.47 μC cm⁻². The strategy of employing metallic oxide composed with self-polarized ferroelectric materials like BFO provides a facile way to optimize the properties of the thin films and fabricate devices for practical applications.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Data accessibility

All data related to this research are contained in the manuscript.

ORCID iDs

Chaoyong Deng https://orcid.org/0000-0003-4745-2568
Xu Wang https://orcid.org/0000-0001-6324-7359

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