Influence of Sputtering Power on the Structure and Electrical Properties of $\text{Bi}_2\text{Fe}_4\text{O}_9$ Thin Films

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Abstract Bismuth ferrite ($\text{Bi}_2\text{Fe}_4\text{O}_9$) thin films were grown on p-type Si (100) substrate by radio-frequency magnetron sputtering at 873 K. X-ray diffraction, field emission scanning electron microscopy and Raman spectroscopy studies revealed that the grown films have single-phase polycrystalline nature and are crystallized in orthorhombic structure. The grain size of the grown thin films was found to increase (56–130 nm) with sputtering power. Atomic force microscopy images clearly illustrated that the grown thin films have smooth surface. Energy-dispersive X-ray analysis revealed the presence of Bi, Fe and O elements with desired ratio and also the absence of impurities in the grown films. Analysis of ferroelectric hysteresis loops revealed that the remanent polarization and coercive field increase with the increase in sputtering power. Vicker's hardness analysis showed that the hardness of films strongly depends on the grain size and film thickness, which are mainly determined by the sputtering power. The above observations revealed that $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin film deposited at higher sputtering power has good crystallinity and shows better electrical properties.

KEY WORDS: $\text{Bi}_2\text{Fe}_4\text{O}_9$; Thin films; Ferroelectric; Sputtering; X-ray diffraction; Hardness

1 Introduction

Multiferroic materials exhibit simultaneously ferroelectric and ferromagnetic behaviours, which can be correlated with each other through their interaction [1–3]. These materials can be used in ferroelectric random access memories and also in novel magnetic memory devices due to their multifunctional properties [1, 4–7]. In addition, these materials enable the invention of novel energy-efficient multistate memory devices, which allow electrical writing and non-destructive magnetic reading operations through their coupling between electrical and magnetic

orders [3, 8, 9]. Nowadays, the magneto-electric materials possess an essential role in various device applications like sensors, actuators, microelectromechanical and nanoelectromechanical systems, spintronics and data storage devices [10]. BiFeO_3 is the solitary and studied extensively for efficient multiferroic device application at room temperature. The BiFeO_3 thin films exhibit low electrical resistivity and low fatigue perseverance, which allows to utilize them in advanced device applications [1, 11]. In this manner, revelation of new multiferroic materials with predominant ferroelectric and ferromagnetic properties is inevitable. Recently, $\text{Bi}_2\text{Fe}_4\text{O}_9$ is found to be a potential multiferroic material and additionally, it can be utilized for multifunctional applications, such as an electrolyte in solid oxide fuel cells, a photocatalyst for the decomposition of organic pollutants, as a low-cost catalyst for ammonia oxidation, and also in gas sensors and gas separation membranes around room temperature [12–14]. A unit cell of $\text{Bi}_2\text{Fe}_4\text{O}_9$ contains FeO_4 tetrahedra and FeO_6 octahedra, which are uniformly disseminated in orthorhombic structure with a

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Fig. 1 XRD patterns of the $\text{Bi}_2\text{Fe}_4\text{O}_9$ films deposited at various sputtering powers

space group of *pbam* and belongs to the family of mullite type [13, 15, 16]. It is understood from the previous reports that the magnetic moment in the perovskite BiFeO_3 is due to the arrangements of antiparallel electron spin of Fe^{3+} -ion at six closest neighbour Fe^{3+} -ions, which brings antiferromagnetic property along (001) direction [17]. The indirect exchange interaction between Fe^{3+} -ion and O^{2-} -ion causes G-type antiferromagnetic structure in the BiFeO_3 [17]. The magnetic transition temperature of BiFeO_3 is reported as ~ 643 K, while the $\text{Bi}_2\text{Fe}_4\text{O}_9$ phase shows an antiferromagnetic transition at ~ 264 K and ferroelectric hysteresis circles at ~ 250 K [18] due to the presence of spirited exchange interaction between FeO_6

octahedra and FeO_4 tetrahedra, which results in a geometrically spin-frustrated antiferromagnetic order [19, 20]. Moreover, the *sp* hybridization between the *s* orbital of the Bi ion and the *p* orbital of the O ion can be ascribed to the adjustment of non-centrosymmetric ferroelectric mutilation in the $\text{Bi}_2\text{Fe}_4\text{O}_9$ compound [12]. The studies on the electrical and multiferroic properties of $\text{Bi}_2\text{Fe}_4\text{O}_9$ bulk ceramics are scarce [12, 16]. It is worth mentioning here that this phase is frequently observed as a secondary phase of BiFeO_3 , which is most stable and environmental friendly material [7, 21]. In fact, bismuth ferrite has many possible phases like Bi_2O_3 , Fe_2O_3 , $\text{Bi}_{12}\text{Fe}_{27}\text{O}_{13}$. Most of the studies on $\text{Bi}_2\text{Fe}_4\text{O}_9$ mainly focus on enhancing its magnetic as well as ferroelectric properties, since the material suffers of high leakage current, high dielectric loss and negligible magnetization for its potential device applications [22–24], such as electronic information storage, integrated micro-electronics memory devices, spintronics devices and so on [5, 25]. In the present work, the influence of sputtering power on the structural and electrical properties of single-phase $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films grown on p-Si substrate by RF magnetron sputtering technique was investigated.

2 Experimental Procedure

$\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films were grown on p-type Si (100) substrate maintained at 873 K by RF (radio frequency) magnetron sputtering. Target for sputtering was prepared through the solid state reaction of the mixture of Bi_2O_3 (purity, 99.99%) and Fe_2O_3 (purity, 99.99%) powders using PVA (polyvinyl alcohol) as a binder. Initially, the fine powder was pelletized and the same was annealed at 923 K for 4 h. Polished single crystalline p-Si (100) wafers, which were ultrasonically cleaned using distilled water, acetone and ethanol and then dried using the hot plate, were used as substrates. The cleaned substrates were centred with respect to the centre of the circular target. The target to substrate distance was fixed as 5 cm. The RF sputtering was carried out in argon (Ar) atmosphere for 30 min with the working pressure of 2×10^{-3} mbar by varying the sputtering powers as 40, 60, 80 and 100 W.

Table 1 Calculated parameters of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films from XRD patterns

Sample	Strain ($\times 10^{-2}$)	Dislocation density lines/ m^2	Crystalline size nm (from XRD)	Grain size nm (from FE-SEM)	Film thickness (nm)
A	3.6536	3.1×10^{-4}	56	60–70	220
B	2.7939	1.8×10^{-4}	74	78–85	246
C	1.7193	7.9×10^{-5}	112	110–120	332
D	1.7192	6.9×10^{-5}	120	120–130	350

Fig. 2 Crystalline size as a function of sputtering power

The substrate temperature was controlled by PID temperature controller.

Crystal structure of the sputtered film was confirmed by X-ray diffraction (XRD, Rigaku Rigaku Ultima III) and Raman spectroscopy (NRS-3100; Jasco, Germany) at room temperature. The XRD spectra were recorded in θ - 2θ mode in the range 10° – 80° with a step size of 0.05° . Microstructural parameters of the grown films were analysed using field emission scanning electron microscope (FE-SEM). The surface topography of the grown films was examined by atomic force microscopy (AFM). Constituent elemental ratio was determined by EDAX (Bruker) analysis. Ferroelectric hysteresis loops were traced using Radiant technologies (RT-66A) ferroelectric tester. Silver circular dots of diameter 0.2 mm were formed at a suitable place to develop top–top electrode configuration using RF sputtering and silver target.

Fig. 3 FE-SEM images of $\text{Bi}_2\text{Fe}_4\text{O}_9$ films deposited at sputtering power of **a** 40 W, **b** 60 W, **c** 80 W, **d** 100 W

Fig. 4 Raman scattering spectra of the $\text{Bi}_2\text{Fe}_4\text{O}_9$ **a** powder, **b** thin films

3 Results and Discussion

3.1 XRD Analysis

Figure 1 shows the XRD patterns of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films deposited on p-Si (100) substrate maintained at 873 K using different sputtering powers of 40, 60, 80 and 100 W. For the sake of convenience, the samples deposited with sputtering power of 40, 60, 80 and 100 W are labelled as A, B, C and D, respectively. The well-defined peaks at 61.42° and 74.52° in the XRD pattern reveal the polycrystalline nature of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films. The observed peaks are found to match very well with the characteristic peaks corresponding to (233) and (062) planes of the phase $\text{Bi}_2\text{Fe}_4\text{O}_9$. It is also observed that the films deposited at 40, 60, 80 and 100 W exhibit reflections from the planes (233) and (062) and importantly, the intensity of the reflection (233) is found to increase with sputtering power. It reveals that the grains are initially oriented in random directions and tend orient mostly in [233] direction with the increase in sputtering power. From the literature, it is observed that the grain size changes with the film thickness [26], in general, and the grain size increases with sputtering power, in particular. In fact, the energy of sputtered atoms, which are obtained from the collision of positive ions with target, increases with sputtering power. Moreover, all other detectable diffraction peaks were found to match well with the standard JCPDS data (# 74-1098) of $\text{Bi}_2\text{Fe}_4\text{O}_9$ phase. The above observations reveal that the obtained $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films are crystallized in orthorhombic structure. The dominant peak observed at 2θ of 68.90° is attributed to reflection from substrate. From the peaks observed in the XRD pattern, grain size (D), strain (ε) and dislocation density (δ) were calculated using the following equations [27, 28] and are tabulated in Table 1.

$$D = \frac{k\lambda}{\beta \cos \theta}, \quad (1)$$

$$\varepsilon = \frac{\beta \cos \theta}{4}, \quad (2)$$

$$\rho = \frac{1}{D^2}, \quad (3)$$

where k is shape factor (taken as constant value of 0.94), λ is the wavelength of X-ray used ($\lambda = 1.54 \text{ \AA}$), β is the full width at half maximum (FWHM) and θ is the Bragg's diffraction angle. The crystallite size of the grown films is found to increase from 56 to 130 nm with sputtering power, and the values are given in Table 1. From Table 1, it is noted that the dislocation density and strain decrease with sputtering power [29] and this behaviour can be attributed to the improvement in crystallinity of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films. Figure 2 shows the variation of crystalline size of the $\text{Bi}_2\text{Fe}_4\text{O}_9$ films with sputtering power.

The thickness of the grown films was measured using surface profilometer, and the values are tabulated in Table 1. It is observed that the thickness of films increases with the sputtering power [30]. FE-SEM images of the prepared thin films are shown in Fig. 3. The recorded images clearly show that the grain size increases with the increase in sputtering power. The result is in good agreement with the same obtained from XRD analysis and is tabulated in Table 1.

3.2 Raman Analysis

Raman spectra of the $\text{Bi}_2\text{Fe}_4\text{O}_9$ powder and thin films were recorded at room temperature using a solid state laser with the wavelength of 514 nm as shown in Fig. 4. From the group theoretical calculations, the irreducible representations for the orthorhombic structure of $\text{Bi}_2\text{Fe}_4\text{O}_9$ can be

written as $12A_g \oplus 12B_1g \oplus 9B_2g \oplus 9B_3g$, which leads to 42 active Raman modes in total [31]. The observed Raman shift of active modes for the $\text{Bi}_2\text{Fe}_4\text{O}_9$ powder and thin films deposited at sputtering power of 100 W is in good agreement with the reported values for orthorhombic crystal structure with space group *pbam* [31, 32]. The Raman active modes for $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films deposited with sputtering power of 100 W are given in Table 2 and are in good agreement with the Raman shifts of the corresponding modes reported by Iliev et al. [31] and Friedrich et al. [32]. In $\text{Bi}_2\text{Fe}_4\text{O}_9$, the low-frequency and high-frequency Raman modes are attributed to the bismuth vibrations and oxygen motions, respectively [31]. All the modes are considered as the atomic displacement modes, which are attributed to the motion of O and Fe atoms along various crystallographic axis of $\text{Bi}_2\text{Fe}_4\text{O}_9$ compound. Raman modes observed above 500 cm^{-1} are attributed to the stretching vibrations of oxygen atoms [15]. In both the powder and thin films of $\text{Bi}_2\text{Fe}_4\text{O}_9$, the observed active Raman modes are 122, 190 and 628 cm^{-1} . The active Raman modes observed at 173 and 324 cm^{-1} are related to the atomic motion of Fe and oxygen in the FeO_6 octahedron of the $\text{Bi}_2\text{Fe}_4\text{O}_9$. The mode presented at 229 cm^{-1} is attributed to the Fe–O vibration in the FeO_4 tetrahedron. In conclusion, the analysis of Raman spectra substantiates the results obtained from XRD studies that the $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films are crystallized in orthorhombic structure.

3.3 AFM Analysis

Typical AFM topographic images of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films were acquired in 1 cm^2 area in order to understand the effect of RF sputtering power on the surface morphologies of grown thin films as shown in Fig. 5. The micrographs (Fig. 5a–d) indicate the surface morphologies and grain growth of the $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films. It can be seen that the sputtering power has made a vast impact on film surface structure. In the lower sputtering power (40 W), the morphology of the $\text{Bi}_2\text{Fe}_4\text{O}_9$ film shows a uniform layer and few grains exist with random orientation as shown in Fig. 5a. On the other hand, the films grown at 60, 80 and 100 W (Fig. 5b–d) show the formation of grains on the film surface and this phenomenon is found to have higher intense with the increase in sputtering power [33]. The $\text{Bi}_2\text{Fe}_4\text{O}_9$ film deposited at higher sputtering power helps to increase the surface mobility of adatom, which is required to form continuous films. As the sputtering power increases, the crystallinity of the films increases and the crystalline size becomes larger. That is, the increase in sputtering power promotes the growth of crystalline and induces an improvement in crystallinity of the film [34]. The surface diffusion of these sputtered films adatom is then enhanced by higher sputtering power. All the films

Table 2 Raman active modes observed for the $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films

Raman frequencies (cm^{-1})											
Raman modes	B1 g(2)	B3 g(2)	A _g (4)	B _{2g} (3)	B _{1g} (4)	A _g (5)	A _g (6)	A _g (7)	A _g (5)	A _g (8)	B _{1g} (9)
					B _{3g} (4)	B _{2g} (6)	B _{1g} (5)	B _{1g} (6)		B _{3g} (6)	
							B _{2g} (6)				
$\text{Bi}_2\text{Fe}_4\text{O}_9$ (100 W)	120	182	229	244	306	324	337	362	398	437	618
											647
											726
											740
											826

Fig. 5 AFM images of $\text{Bi}_2\text{Fe}_4\text{O}_9$ films deposited at various sputtering powers of **a** 40 W, **b** 60 W, **c** 80 W, **d** 100 W

exhibit a uniform microstructure with no cracks. The values of the roughness root mean square (RMS) are given in Fig. 6. The AFM images have a relatively good surface profile with almost uniform and compact grains. The surface roughness of the films decreases with the increase in RF power.

Quantitative elemental composition of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films were analysed by EDAX and the corresponding spectrum is shown in Fig. 7. The quantitative elemental analysis at different points on the sample approximately validated with a ratio of 1:2 between Bi and Fe. This result reveals the homogeneity of chemical composition of the

Fig. 6 Dependence of the roughness as a function of sputtering power

prepared films. The atomic percentage of the elements present in the film is given in the inset of Fig. 7.

3.4 Electric Properties

Figure 8a–d shows the effect of sputtering power on the ferroelectric behaviour of Ag/Bi₂Fe₄O₉/Ag capacitors, which is examined by measuring the polarization as a function of applied electric field (P – E) at room temperature. The ferroelectric hysteresis loops were measured at different electric fields of 2, 4, 6, 8 and 10 V. Well-

saturated P – E loops of the film deposited at higher sputtering power were obtained with an applied voltage of 4 V. This can be attributed to the lower leakage current and higher breakdown electric field [11, 35]. When the applied voltage is increased above 4 V, the P – E loop obtained for the films deposited at lower sputtering power is very poor. On the other hand, the films grown at higher sputtering powers have spin orientation and show a good P – E loop, due to the increase in applied voltage. It is concluded that unshaped and unsaturated P – E loops were acquired for the films deposited at lower sputtering power due to high leakage current and low breakdown electric field. The remanent polarization and coercive field were increased with increasing the sputtering power up to 80 W. The saturation polarization (P_s), remnant polarization (P_r) and coercive field (E_c) values of Bi₂Fe₄O₉ thin films are measured at 4 V as listed in Table 3.

Figure 9 shows the influence of sputtering power on hardness of Bi₂Fe₄O₉ thin films, which were measured by an optical microhardness tester. Vicker's hardness number was calculated using the equation [36]

$$HV = \frac{1.854p}{d^2} \text{ (kg/mm}^2\text{)}, \quad (4)$$

where p is the applied load value of 200 g with 10 s loading time and d^2 is the average length of the diagonal. The indentation is made on five different points for each sample to obtain its average hardness value. The hardness of the deposited films is found to increase with sputtering power. This may be due to the loose structure caused by

Fig. 7 EDAX spectrum of Bi₂Fe₄O₉ film

Fig. 8 P-E hysteresis loop of $\text{Bi}_2\text{Fe}_4\text{O}_9$ films deposited at various sputtering powers of **a** 40 W, **b** 60 W, **c** 80 W, **d** 100 W

Table 3 Ferroelectric properties of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films (100 W at 4 V)

Ag/ $\text{Bi}_2\text{Fe}_4\text{O}_9$ /Ag	A	B	C	D
P_s ($\mu\text{C}/\text{cm}^2$)	3.47	2.69	3.01	4.12
P_r ($\mu\text{C}/\text{cm}^2$)	0.16	0.13	0.28	1.32
E_c (kV/cm)	2.12	2.85	7.53	22.88

too much high-energy sputtering particles or oxidation [33].

4 Conclusion

In the present work, the $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films were grown on p-type Si (100) substrate maintained at 873 K by RF magnetron sputtering by varying the sputtering power as 40, 60, 80 and 100 W. It is found that the sputtering power influences the structure, surface morphology and the electric properties of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films. Based on the results of XRD, FE-SEM and Raman spectroscopy studies, all the films have single-phase polycrystalline nature and are crystallized in orthorhombic structure. The crystalline size is found to increase, whereas the stress and the dislocation density are observed to decrease with sputtering power. The elements presented in the grown thin films (bismuth, ferrite and oxygen) and the elemental ratio of grown films were determined by EDAX analysis. The AFM analysis showed that the roughness of the deposited film decreases with an increase in sputtering power. The P - E loops revealed that the films deposited with higher sputtering power have well-saturated hysteresis loops. The micro-hardness of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films was measured, and it

Fig. 9 Hardness of $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin films deposited at variation of sputtering powers

showed an increasing trend with an increase in sputtering power. The higher hardness of the film is due to the grain size effect, highly textured grains and compressive residual stress in the films. In conclusion, the results of investigation have clearly revealed that $\text{Bi}_2\text{Fe}_4\text{O}_9$ thin film deposited at higher sputtering power has good crystallinity.

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