

FABRICATION AND STRUCTURAL STUDIES OF SPIN-COATED COBALT FERRITE LAYERS

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Abstract

Cobalt ferrite coating of various layers is fabricated on fluorine-doped tin oxide (FTO) substrate using a spin coater. A precursor sol of 0.1 M metal ion concentration is employed to develop layers of cobalt ferrite on FTO substrate. Various layers of cobalt ferrite are deposited on the FTO substrate at a rotation speed of 3000 revolutions per minute for 30 seconds. After deposition of each layer, the sample is kept for thermal baking at 200°C for 5 min. Three samples of three different coats ranging from single layer to three layers are fabricated and thermally baked. These samples are then annealed at 500°C for 7 hours in air ambient. Structural studies namely phase formation, chemical element analysis and surface morphology of the sample is carried out respectively using X-ray diffractometer (XRD), Energy-dispersive X-ray analysis (EDX) and Field Emission Scanning Electron Microscope (FESEM). These structural characterizations confirm the phase formation of cobalt ferrite. Spin-coated layers of cobalt ferrite can be widely employed in memristive applications due to its excellent resistive switching characteristics. The typical memristive applications are resistive random-access memory (RRAM), artificial intelligence (A.I.), neuromorphic computing, and hardware security. Such deposited cobalt ferrite layers also find applications in gas sensor fabrication, magneto-optic recording technology, and spintronics devices.

Keywords: Spin coating; Cobalt ferrite; Memristor; Neuromorphic Computing; Artificial Intelligence

Introduction

Magnetic spinel (XFe_2O_4) where; X is a divalent metal cation viz Co^{2+} , Cu^{2+} , Zn^{2+} , Ni^{2+} , and Mn^{2+} , exhibit extraordinary physio-chemical (Mondal *et al.*, 2023), physio-mechanical (Gharieb *et al.*, 2023), optical (Munawar *et al.*, 2023), electrical (Kamran and Anis, 2023), dielectric (Zahid *et al.*, 2023) and magnetic properties (Padhan *et al.*, 2023). Therefore, they are extensively employed in cutting-edge technological applications namely hyperthermia (Jamir *et al.*,

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2023), electrochemical sensors (Padhan *et al.*, 2023), wastewater treatment (Sonia *et al.*, 2023), magnetic resonance imaging (MRI) (Nandwana and Dravid, 2023), photodetector (Tran *et al.*, 2024), biomedical applications (Salih and Mahmood, 2023), microwave applications (Fedorchuk *et al.*, 2024), solar cells (Liu *et al.*, 2023), super capacitor (Jeevanantham *et al.*, 2023), and data storage devices (Angadi *et al.*, 2023). Additionally, they show an almost rectangular magnetic hysteresis loop (Bharathi *et al.*, 2023). Such a type of magnetic hysteresis properties can be harvested for magnetic switching as well as resistive switching applications (Kaiser, 2023). Particularly, spin-coated magnetic spinels found their applications in Spin Transfer Torque Magnetic Random Access Memory (STT-MRAM) (Adiba *et al.*, 2023; Zheng *et al.*, 2023) and Resistive Random Access Memory (RRAM) Devices (Hu *et al.*, 2012). Recently, spin-coated magnetic spinels namely nickel ferrite, zinc ferrite, and cobalt ferrite have been examined for resistive switching applications (Gayakvad *et al.*, 2023). Cobalt ferrite is one of the magnetic spinels having an almost rectangular magnetic hysteresis loop and hence nowadays, it is widely explored for its application in data storage devices, microwave applications, magnetorestrictive devices, biomedical applications, spintronics applications, (Munjal and Khare, 2017; Raut *et al.*, 2021). Especially, spin coated cobalt ferrite on FTO substrate exhibits good quality resistive switching properties (Munjal and Khare, 2017; Gayakvad *et al.*, 2023). Therefore, we have fabricated and studied structural characterizations of spin-coated cobalt ferrite layers.

Materials and Methods

The analytical grade chemicals and reagents of HIMEDIA have been harvested for the experimentation purpose. We have used double distilled water (DDW), ferric nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, cobaltous acetate $\text{C}_4\text{H}_6\text{CoO}_4 \cdot 4\text{H}_2\text{O}$ and 2-methoxyethanol $\text{C}_3\text{H}_8\text{O}_2$. The solvent 2-methoxyethanol was taken in order to prepare a sol of 0.1 M metal ion concentration. To deposit layers of cobalt ferrite using a spin coater, a Sol of 0.1 M metal ion concentration with Co: Fe=1:2 proportion was synthesized. The Sol was prepared using Cobalt Acetate and Ferric Nitrate as precursors respectively for Co and Fe and 2-methoxy ethanol as solvent. The Sol of proper viscosity was then employed for the casting of spin-coated cobalt ferrite layers on the FTO substrate. The spin coater was adjusted for 3000 rpm for 30 seconds. After casting of each

layer, the as-deposited sample was kept under thermal baking at 200 degree celsius for 5 minutes. In this way, we have prepared cobalt ferrite layers ranging from single coats to twelve coats. Finally, all these samples were kept in the furnace for annealing purposes. The samples were annealed at 500 degrees Celsius for 7 hours in air ambient. The photographs of annealed samples of various layers are shown in Figure 1. The final look of the annealed samples was brownish and the degree of brown color increased with several layers. We can also conclude that, with an increase in the number of layers that is beyond eight layers, the sample gets pilled off, and hence breaking of the layers starts as shown in Figure 1.



Figure 1. Spin-coated cobalt ferrite of two(n=2)to twelve (n=12) layers Structural Studies

The phase confirmation, presence of chemical elements, and morphology of the sample (n=2) were respectively examined using an X-ray diffractometer (XRD), Energy-dispersive X-ray analysis (EDAX), and Field Emission Scanning Electron Microscope (FESEM).

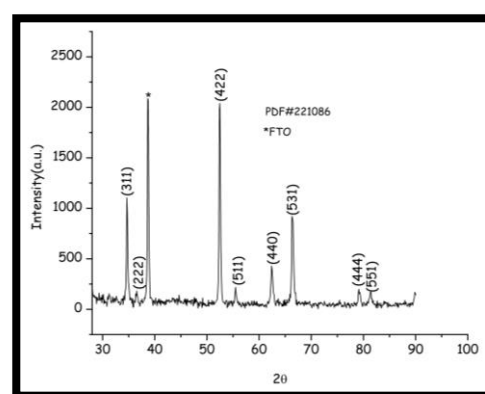


Figure 2. X-Ray Diffraction spectrum

The XRD characterization of the sample was carried out using Cu- K_{α} X-ray wavelength of $1.54056 \text{ \AA}$. The observed XRD pattern is as shown in Figure 2.

The peak designated by asteric mark(*) was observed out of the way of spinel structure. It may be due the reflection from FTO substratet. However, most of the peaks with (hkl) planes namely (311), (222), (422), (511), (440), (531), (444), (551) were found to be approximately matching with standard PDF number 221086. Therefore, we can conclude that, the synthesized sample is of spinel nature having Face Cantered Cubic (FCC) crystal structure with space group $Fd3m$. The most intense peak corresponds to the (422) miller plane and therefore the crystallite size was calculated concerning peak position and Full Width Half Maximum of the (422) peak using the Debey-Sherrer equation as follows.

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Where, D is the crystallite size, K is the Sherrer constant, λ is the wavelength of X-rays, β is Full Width Half Maximum in radian, and θ is peak position in radian. The calculated crystallite size turns out to be 31.4472 nm and the estimated lattice constant was resulted as 8.45 angstrom. Additionally, the lattice strain is also determined with the help of the following formula,

$$\varepsilon = \frac{\beta_{hkl}}{4 \tan \theta}$$

For the most intense peak (422) positioned at 26.2373° , the lattice strain was calculated and found to be 0.1375 radian. Further calculation was done for the density of the crystal material which was found to be 0.036 gm per cubic centimetre. The availability of the constituent elements was confirmed by Energy Dispersive X-ray Analysis.

Energy Dispersive X-ray(EDX) Analysis

The presence of Cobalt (Co), Ferric (Fe), and Oxygen(O) were confirmed using EDX spectrum of the sample. The EDX spectrum accompanied with atomic and weight percentage is as shown in Figure 3. We could see that the atomic ration in the EDX is deviated from the stoichiometry of cobalt ferrite. This may be due to impurities incorporated during the sample fabrication and its characterization.

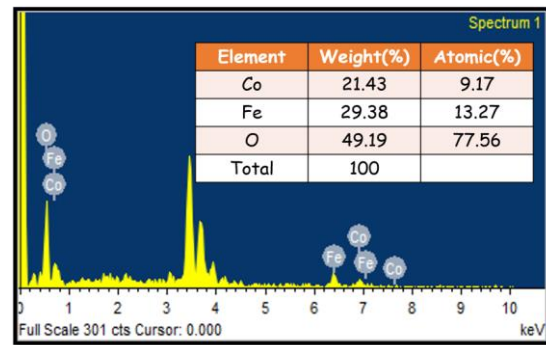


Figure 3. EDX of spin-coated cobalt ferrite

Morphology study

The surface morphology of the sample under consideration was examined with the help of Field Emission Scanning Electron Microscopy (FESEM). The sample was studied for different magnifications as shown in Figure 4.

As the magnification increases, the squareness of the crystallites increases and hence spherical geometry of the grain tends to rectangular shape. The size of the grain ranges from 72.73 nm to 345.46 nm. This implies the polycrystalline nature of the synthesized material. The average grain size estimated from the FESEM image is found to be 148.05 nm.

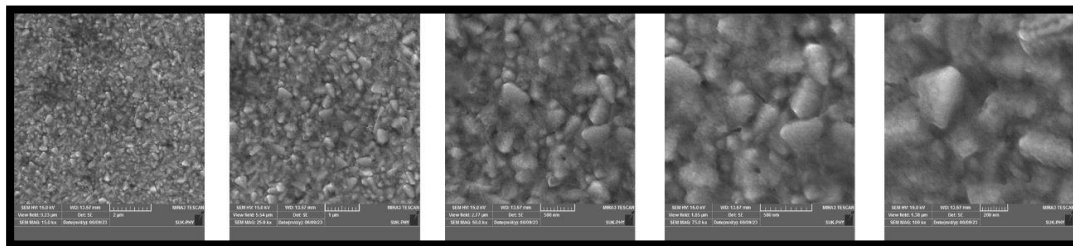


Figure 4. FESEM of cobalt ferrite layer under different magnification

Applications of spin-coated cobalt ferrite

Spin-coated cobalt ferrite layers find applications in diverse fields of modern science and technology. As shown in Figure 5, Cobalt ferrite layers can be employed in spintronic devices, biomedical applications, Optoelectronic devices, Energy storage and energy conversion devices, Wastewater treatment, Gas sensors, Photodetectors, and Memristive devices. Furthermore, Memristive devices can be employed for their applications in artificial intelligence (A.I.) and neuromorphic computing.

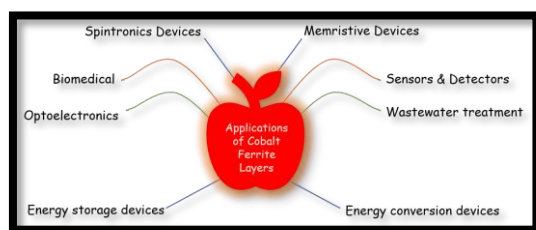


Figure 5. Applications of spin-coated cobalt ferrite

Results and Discussion

We have successfully fabricated cobalt ferrite layers using the spin coating technique. The optimized parameters are found to be as follows. The 0.1 M Sol can be deposited on rotating FTO substrate. The optimized angular speed of rotating substrate 3000 revolution per minute for 30 second. Each coat was followed baking at 200 degree Celsius for 5 minute. The XRD study of annealed sample (n=2) at 500 degree Celsius for 7 hours shows the phase formation of spinel structure. The EDX characterization of the synthesized sample indicates the presence of Co, Fe, and O. The surface morphology study shows spherical granular nature of crystallites. These type of cobalt ferrite layers can be used in diverse fields of 21st century technology.

Conclusions

Spin-coated cobalt ferrite layers can be successfully fabricated on FTO substrate for their applications in cutting-edge science and digital technology.

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