

# Room temperature XRD and magnetic investigations of NBT-Zn-Cr-CFO multiferroic composites

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## ABSTRACT

The multiferroic composite consist of ferroelectric phase contain lead free  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$  (NBT) and ferromagnetic phase of Zn-Cr substituted cobalt ferrite  $\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  (Zn-Cr CFO). The ferroelectric phase (NBT) of composite is synthesized by solid state reaction method and ferromagnetic phase (Zn-Cr-CFO) is prepared by sol-gel auto combustion method. The multiferroic composite of (75)  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$  + (25)  $\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  were prepared by mixing the two phases according to its stoichiometric concentration. The structural and morphological properties were investigated by using X-ray diffraction and scanning electron microscope with EDAX. X-ray diffraction pattern confirms the presence of both phases in composite. The magnetic properties were studied by using vibrating sample magnetometer (VSM) the value of magnetic saturation increases with increasing ferromagnetic phase in composite.

**Key words:** Sol-gel method, XRD, SEM, VSM

## INTRODUCTION

The multiferroic materials increase the interest of researcher due to their unusual properties in different field of modern technology. Magnetolectric composite consists of a suitable combination of ferroelectric material with ferromagnetic material. Magneto-electric effect in these composites is the result of mechanical coupling between piezoelectric (ferroelectric) and magnetostrictive (ferromagnetic) materials. Under applied magnetic field a strain is developed in ferromagnetic (piezomagnetic) material through magnetostriction, which passed to ferroelectric (piezoelectric) material results into stress in it. Thus extra charges are developed across multiferroic composite when magnetic field is applied which in turn generates induced voltage due to the piezo- electric effect [1].

The lead free materials are preferred due to concern of human health and for taking care of environment. Sodium bismuth titanate (NBT) is a new kind of perovskite ( $ABO_3$  - type) ferroelectric discovered by Smolenskii et al. in 1960 [2].  $Na_{0.5}Bi_{0.5}TiO_3$  (NBT) exhibits a rhombohedral symmetry at room temperature. It undergoes a series of phase transitions: (i) ferroelectric rhombohedral to antiferroelectric tetragonal around 230 °C, (ii) antiferroelectric tetragonal to nonpolar tetragonal around 320 °C, (iii) non-polar tetragonal to cubic around 520 °C [3]. NBT is considered as one of the good candidates for lead-free piezoelectric ceramics due to its large remnant polarization ( $P_r = 38 \mu C cm^{-2}$ ) at room temperature and high Curie temperature ( $T_c = 320$  °C). Various composite systems have been reported and they mostly correspond to Co and Ni ferrites with P (ZrTi) O<sub>3</sub> (PZT) [4-7],  $BaTiO_3$  [8-11] or  $BiFeO_3$  [12-15] with combinations of both ferrites and piezoelectrics. The NBT based composite have great prospect due to this it used in various application.

However, PZT-CFO magneto- electric composites have shown a low Magnetolectric voltage co-efficient due to the large magnetic anisotropy and coercivity of CFO which restricts the domain wall motion process [16, 17].

Substitution of Co by Zn at tetrahedral site in CFO is known to reduce magnetic anisotropy and coercivity while maintaining the value of magnetostriction co-efficient comparable to that of CFO. Thus, the Zn substituted cobalt ferrite is a good choice for a magnetostrictive phase due to its higher magnetostrictive response. However, it is well known that the substitutions of  $Co^{2+}$  ions by  $Zn^{2+}$  ions and  $Fe^{3+}$  ions by  $Cr^{3+}$  in  $CoFe_2O_4$  may manipulate spinal structure, cation distribution between A and B sites and also the magnetic properties. The substitution of  $Zn^{2+}$  ions increases the resistivity of ferrite phase [20], at the same time  $Zn^{2+}$  ions at lower substitution level for  $Co^{2+}$  will also increase the saturation magnetization due to canting effect.

In the present work Zn-Cr substituted Co ferrite with a chemical formula  $Co_{0.75}Zn_{0.25}Cr_{0.2}Fe_{1.8}O_4$  has been made composite with  $Na_{0.5}Bi_{0.5}TiO_3$  is successfully synthesis by sol-gel method and solid state reaction method respectively. Hence the composite of sodium bismuth titanate and Zn-Cr substituted cobalt ferrite were study the structural, magnetic and electric properties.

## METHODOLOGY

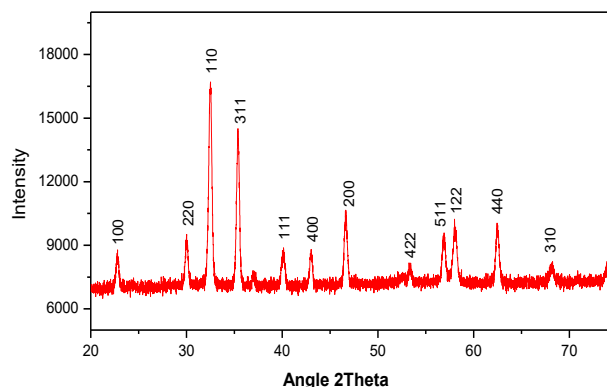
The composite material having chemical composition  $(1-x) Na_{0.5}Bi_{0.5}TiO_3 + (x) Co_{0.75}Zn_{0.25}Cr_{0.2}Fe_{1.8}O_4$  ( $x = 0.25$ ) is synthesized by two different methods. The ferromagnetic phase consists of Zn-Cr Substituted with cobalt ferrite is synthesis by sol-gel auto combustion method and ferroelectric phase sodium bismuth titanate is obtained by solid state reaction method. The nanoparticles of  $Co_{0.75}Zn_{0.25}Cr_{0.2}Fe_{1.8}O_4$  and  $Na_{0.5}Bi_{0.5}TiO_3$  are combined by varying  $x$  ( $x = 0.025$ ). This composite material is grinded for couple of hour's and finally annealed at 1100° C for 06 hours. By grinding it is converted into fine powder of composite. The pellets of composite material were prepared by taking 0.35 gm powder of sample and PVA binder is added to get good strength. The all pellets are 0.1 cm diameter and it has thickness about 0.90 mm are formed.

### Characterization:

To study the structural analysis of prepared samples were characterized by X-Ray Diffraction technique, X ray diffraction pattern were recorded at room temperature in the  $2\theta$  range  $20^\circ$  to  $80^\circ$  using Cu-K $\alpha$  radiation ( $\lambda = 1.5404 \text{ \AA}$ ). The microstructures of all the composite samples were studied by using Scanning Electron microscope (SEM). Elemental compositions were estimated using X-ray electron diffraction analysis (EDAX). The magnetic properties of samples are studied were by using vibrating samples magnetometer (VSM) at room temperature with an applied magnetic field of 1 Tesla.

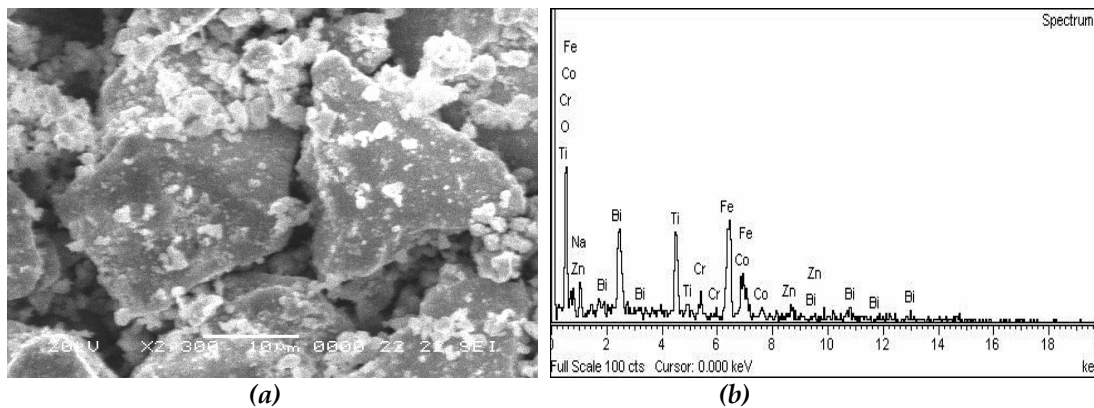
## RESULTS AND DISCUSSIONS

Fig.1 shows the X-ray diffraction pattern of (75)  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$  + (25)  $\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  multiferroic composite at room temperature in the  $2\theta$  range  $20^\circ$  to  $75^\circ$ . The X-ray diffraction analysis of sintered multiferroic composite shows separate diffraction peaks of the constituent parent phases. The x-ray diffraction patterns consist some peaks of Zn-Cr Substituted in cobalt ferrite it has cubic spinal structure with diffraction peaks at angle  $2\theta$  is observed at  $30.04$  (220),  $35.45$  (311),  $43.03$  (400),  $53.39$  (422),  $56.39$  (511),  $62.521$  (440) (as per reference data from ICSD Card No. 22-1086). Similarly, remaining peaks of ferroelectric phase NBT has a single perovskite phase with rhombohedral structure and its peaks are indexed with angle  $2\theta$  are

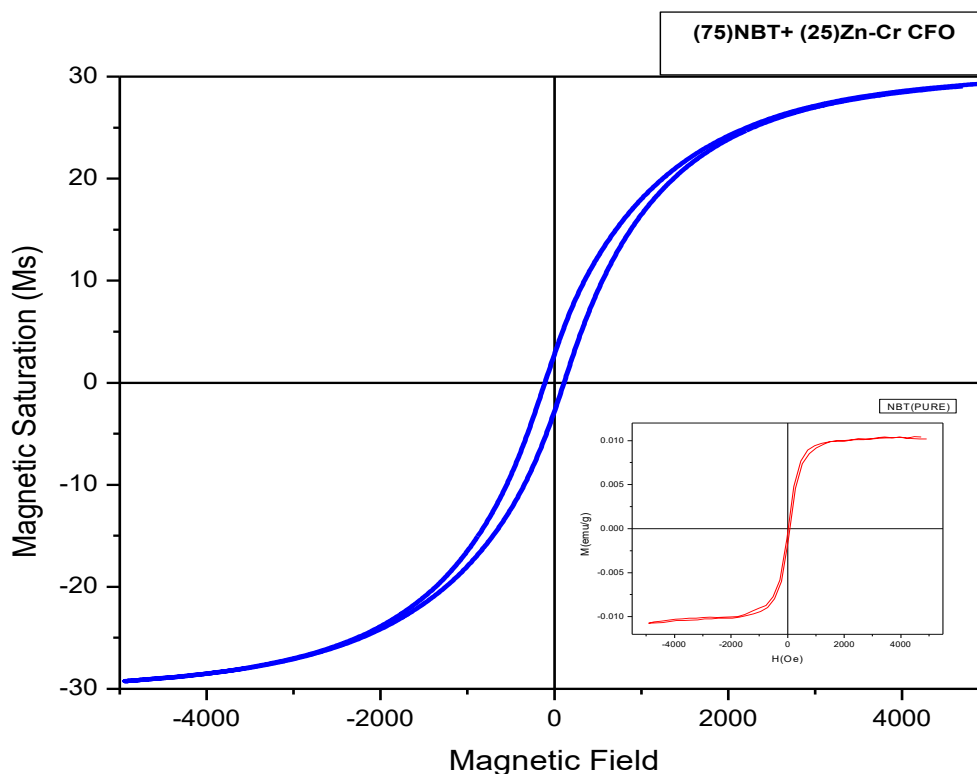


**Fig. 1:** XRD pattern of (75)  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$  + (25)  $\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  multiferroic composite sintered at  $1150^\circ\text{C}$ .

$22.808$  (100),  $32.58$  (110),  $40.239$  (111),  $46.699$  (200),  $58.194$  (211) according to ICSD file no.280983. All the XRD peaks in the patterns could be indexed to one of the  $\text{CoZnCrFe}_2\text{O}_4$  and NBT phases, no extra peak are present in the composite it shows that no impurity phases were identified or no significant chemical reaction is takes place between ferroelectric and ferrite interface in composite. The prepared sample shows sharp peaks of XRD it means that material has crystalline in nature. The lattice constant of NBT phase is matched with reported value in different journals and for the ferrite phase it slightly changes from  $8.4158 \text{ \AA}$  to  $8.3951 \text{ \AA}$ . The Debye Scherrer equation is used to calculate crystalline size of composite particle size of ferroelectric phase  $20.8040 \text{ nm}$  and crystalline size of ferrite phase  $21.6272 \text{ nm}$ .



**Fig. 2:** (a) Shows SEM micrographs and (b)EDAX of (75)  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$  + (25)  $\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  multiferroic composite



**Fig. 3: M-H hysteresis curve of (75)  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$  + (25)  $\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  multiferroic composite sintered at  $1150^\circ\text{C}$ . Inset fig. shows variation of Ms of Pure NBT with applied field.**

Fig. 2 shows SEM micrographs and EDAX of (75)  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$  + (25)  $\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  multiferroic composite sintered at  $1150^\circ\text{C}$ . The Morphology and microstructure of prepared sample were studied by using scanning electron microscope (SEM). The image obtain from SEM it is clear that both the NBT and Zn-Cr CFO phases of composite material are present. NBT material shows larger grain size were as Zn-Cr CFO shows smaller grain size and it is observed that material has homogeneous microstructure, dense in nature, small pores are detected and no cracks is found in composite. The Energy dispersive X-ray diffraction analysis (EDAX) patterns the NBT and Zn-Cr CFO phases of composite material shows only main elements are detected. This shows that prepared sample does not contain any type of impurity. The atomic weight of bismuth ions decreases in composite due interaction of with oxygen ions.

#### Magnetic Measurements:

Fig.3 shows the magnetic Hysteresis loop of (75)  $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$  + (25)  $\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  multiferroic composite at room temperature. The magnetic properties of ZnCr CFO and Sodium Bismuth Titanate nanocomposite material were studied by using Vibrating Sample Magnetometer (VSM).

NBT has ferroelectric in nature it shows very low response to magnetic field when we added ferromagnetic phase Zn-Cr CFO added in composite it gives small change in magnetic saturation. The magnetization of composite material increases due to the dilution effect of the ferroelectric component in the sample. The coercivity of multiferroic material is depends on anisotropy of material. This composite material is soft in nature due to this it is used in AC electric components, transformer and magnetic core to

increase the efficiency of equipment and decreases the energy losses.

## CONCLUSION

The multiferroic composite of  $(1-x)\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3 + (x)\text{Co}_{0.75}\text{Zn}_{0.25}\text{Cr}_{0.2}\text{Fe}_{1.8}\text{O}_4$  is successfully synthesis by sol-gel and solid state method. The XRD analysis confirms the crystalline nature due sharp peaks and no chemical interaction is occurring between both the phases. The XRD analysis confirms the NBT has perovskite structure and cubic spinal structure of Zn-Cr CFO. The intensity of NBT peaks decreases with increasing ferromagnetic content in composite. The SEM analysis shows material has crystalline in nature, dense and small pores are observed in composite. The remanance magnetization and saturation magnetization of multiferroic composite increases with increasing Zn-Cr CFO concentration.

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