Journal of Advances in Applied Sciences and Technology

ISSN : 2393-8188 (print)
2393-8196 (online)
www.milliyasrcollege.org.journal.php

STRUCTURAL PROPERTIES AND ANALYTICAL INFORMATION OF PIEZOMAGNETIC AND PIEZOELECTRIC COMPOSITES USING EDS

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ABSTRACT

The particulate composite of piezomagnetic and piezoelectric phases having the general formula (1-x) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (x) $BaTiO_3$ magnetoelectric (ME) composite have been prepared using conventional double sintering ceramic process where x is fraction of components that varies as 0.00, 0.25, 0.50, 0.75 and 1.00. The presence of constituent phases, i.e. cubic spinel structure of piezomagnetic phase and tetragonal perovskite structure of piezoelectric phase was confirmed by X-ray diffraction (XRD) technique. Energy Dispersive Spectroscopy (EDS) analysis provides the analytical information including qualitative analysis, quantitative analysis and elemental mapping. From the standard quantitative analysis, the molar composition of piezomagnetic and piezoelectric phases is confirmed.

KEYWORDS **:** Piezoelectric; Piezomagnetic; Composite

1. INTRODUCTION

Magnetoelectric composites consist of two phases such as piezoelectric and piezomagnetic. The deformation of piezomagnetic phase causes polarization of piezoelectric phase in the composites and the electrical polarization of piezoelectric phase causes change in magnetization of piezomagnetic phase due to strong mechanical coupling between the piezomagnetic and piezoelectric phases [1]. The ME effect is a property of ME composites, which is absent in their constituent phases [2]. Such magnetoelectric composites find many applications in ME data storage and switching, radio electronic device, Modulation of amplitudes, optoelectronic, microelectronic, spin wave generation, frequency conversion, amplification, transducers, etc. [3, 4]. The ME effect occurs due to the interaction between the magnetic and electric dipoles [5, 6, 7]. In the present communication, we have chosen $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ as a piezomagnetic phase and $BaTiO_3$ as the piezoelectric phase. Energy Dispersive Spectroscopy (EDS) is a standard procedure for identifying and quantifying elemental composition of sample areas as small as a few cubic micrometers. Characteristic X-ray are produced when a material is bombarded with electrons in an electro beam instrument, such as a SEM. Detections of the X-ray can be accomplished by an energy dispersive spectrometer, which is a solid state device that discriminates amount of X-ray energies[8,9] . EDS provides the analytical information including qualitative analysis, quantitative analysis, line profile analysis and elemental mapping [10, 11].

2. EXPERIMENTAL TECHNIQUES

2.1 PREPARATION OF ME COMPOSITES

The piezomagnetic material chosen as a piezomagnetic phase $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ has been prepared by standard double sintering ceramic method using AR grade oxides. The oxides were mixed in stoichiometric proportion and wet ground for about 2-3 hours in an agate mortar and pestle. The mixed fine powder is pre-sintered at 925^0C for 9 hours. The sintered powder is again reground and finally sintered at 1080^0C for 16 hours followed by slow cooling to room temperature to obtain pure single phase piezomagnetic samples. The single phase cubic spinel structure formation of piezomagnetic was confirmed by X- ray diffraction technique.

The piezoelectric phase Barium Titanate ($BaTiO_3$) was prepared by standard doubling sintering ceramic method using AR grade oxides/carbonate. Barium carbonate ($BaCO_3$) and Titanium-dioxide (TiO_2) were taken in molar proportion. The mixed powder of Barium carbonate ($BaCO_3$) and Titanium-dioxide (TiO_2) was ground using agate in mortar and pestle and pre-sintered at 900^0C for 12 hours. In the final sintering the material was held at 1050^0C for 16 hours. The sintered samples were allowed to cool to room temperature. Analysis of XRD pattern of $BaTiO_3$ revealed the formation of single phase tetragonal perovskite structure.

The composite of piezomagnetic and piezoelectric phase (1-x) $CoMnFe_2O_4$ + (x) $BaTiO_3$ with (x= 0.25, 0.50 and 0.75 mole %) was prepared by standard doubling sintering ceramic method using prepared piezomagnetic and piezoelectric phases. The fine powders of piezomagnetic ($CoMnFe_2O_4$) and piezoelectric phase ($BaTiO_3$) were mixed thoroughly in molar proportion and ground for above 3 hours. The composite phase of piezomagnetic and piezoelectric was pre-sintered at 925^0C for 12 hours and is cooled at room temperature. The composite powder is then reground again and finally sintered at 1080^0C for 24 hours. The magnetoelectric composites of piezomagnetic and piezoelectric prepared by mixing the constituent phase were ground for 2-3 hours and mixed with 2-3 drops of polyvinyl alcohol as a binder. The composite powder is then pressed into pellets of thickness around 2-3 mm and diameter 10mm using a hydraulic press. A pressure of 6 ton /cm2 was applied for 10 to 15 minutes. The pellets are finally heated up to 600^0C for 6 hours to remove the binder and cooled at room temperature. The prepared composite of piezomagnetic $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ and piezoelectric $BaTio_3$ was characterized by X-ray diffraction technique and was used for further investigations of structural, electrical and magnetoelectric properties of piezomagnetic, piezoelectric and their composites.

2.2 CHARACTERIZATION

The presence of constituent phases in the composites as well as the crystal structure of constituent phases and their composites was determined by X-ray diffraction using a Rigaku Miniflux-II and λ=1.5406Å.

2.3 ENERGY DISPERSIVE SPECTROSCOPY

EDS is a standard procedure for identifying and quantifying elemental composition of sample areas as small as a few cubic micrometers. The model of the EDS which was used is JEOL JXA 84C.

3. RESULTS AND DISCUSSION

3.1PHASE IDENTIFICATION

The XRD pattern of the representative composite is shown in figure 1. The pattern revels the presence of both piezoelectric as well as piezomagnetic phases. All the peaks are indexed and no additional peaks were observed. This indicates the absence of impurities or intermediate phases in the composite. Piezoelectric phase has tetragonal perovskite structure and piezomagnetic phase has a cubic spinel structure. The lattice parameters of piezoelectric phase are a=4.001 and c=4.005 and c/a ratio is 1.001 the lattice parameter for piezomagnetic phase is a=8.366Å.

As all the peaks of the composites are identified it conforms the formation of composites with two distinct phases. Increased molar percentage of piezoelectric in composites leads to increase in the intensity of (110) piezoelectric peaks. From XRD pattern it observed that with increase in piezoelectric percent in composite, number of piezoelectric peaks increases also the intensity of most intense piezoelectric peak (110) also increases.

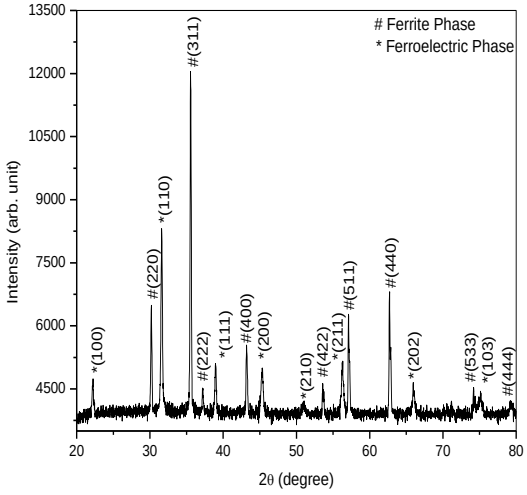

Fig.1 *X-Ray Diffraction pattern of (0.75) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (0.25) $BaTiO_3$ composite.*

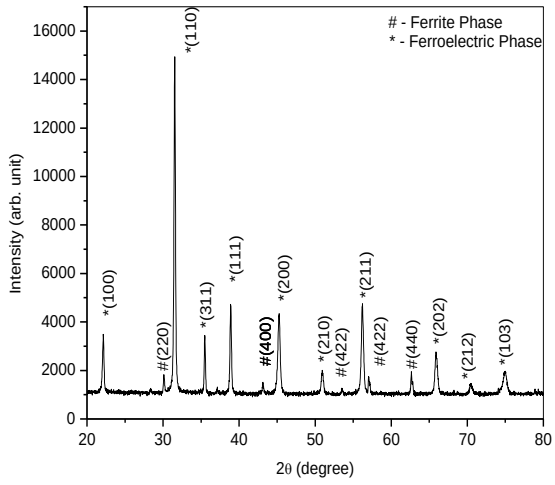

Fig.2 *X-Ray Diffraction pattern of (0.25) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (0.75) $BaTiO_3$ composite.*

The XRD pattern shows the presence of two phases i.e. piezomagnetic and piezoelectric, no single phase formation of composite material is observed, further it can be observed from figure that the intensity of

all the reflections of piezomagnetic and piezoelectric phases decreases as compared to their individual phases [12,13] . Using XRD data, lattice constant for all the values of composite x and for piezomagnetic and piezoelectric phases were calculated using the following relations (Eq. 1). For the cubic spinel structure the inter-planner distance 'd' and lattice constant 'a ' and the Miller indices (h k l) of the planes are related by equation ,

$$d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}} \quad \text{-- (1)}$$

For tetragonal structure, the inter-planner distance 'd', lattice constants 'a' and 'c' and the Miller indices (h k l) of the planes is related by equation

$$\frac{1}{d^2} = [(h^2 + k^2)/a^2] + (l^2/c^2) \quad \text{-- (2)}$$

Table 1 contains the values of lattice constant for piezomagnatic phase and piezoelectric phase. It is evident from Table 1 that lattice constant of piezomagnatic phase increases from x= 0.00 to x = 0.75. The increase in lattice constant of the piezoelectric phase x = 0.25 to x = 1.00 found to increases marginally. The slight increase in lattice constant is attributed to increase in piezoelectric contents in the composite.

Table 1 Lattice constant of piezomagnetic, piezoelectric phase of (1-x) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (x) $BaTiO_3$. (x= 0.00 - 1.00) composite.

Comp 'x'	Lattice parameters (Å)			
	Piezomagnetic		Piezoelectric	
	a	a	c	c/a
0.00	8.366	---	---	---
0.25	8.376	4.000	4.000	1.000
0.50	8.387	4.000	4.006	1.002
0.75	8.389	4.001	4.005	1.001
1.00	---	4.002	4.003	1.003

3.2 Energy Dispersive Spectroscopy

The results of Energy dispersive spectroscopy (EDS) of piezomagnetic phase is shown in Fig.3. It is evident from Fig. 3 that the results of the matrix show strong Co, Mn, Fe peaks maintaining the stoichiometry of the piezomagnetic phase ($Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$).

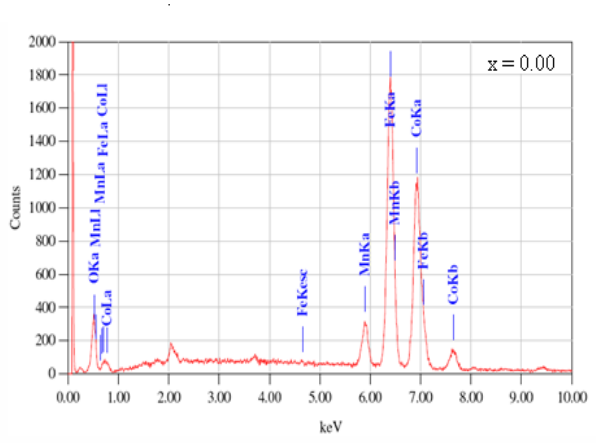

Fig 3 *Energy Dispersive Spectroscopy pattern of (1-x) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (x) $BaTiO_3$. (x = 0.00) composite.*

In Fig.5, the composition of piezoelectric (i. e. x = 0.50) reveals the presence of peaks of Ba and Ti ions as well as Co, Mn and Fe with their expected proportions.

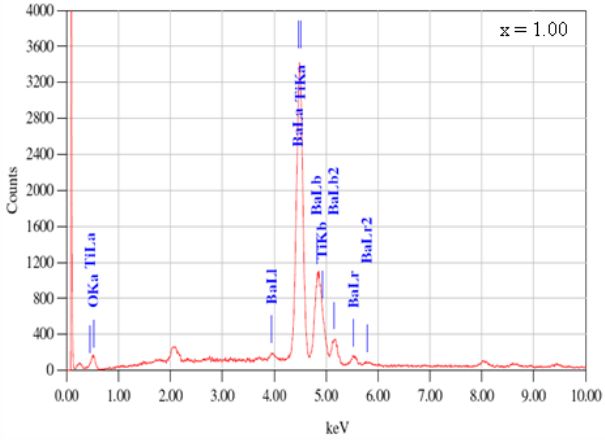

Fig 4 *Energy Dispersive Spectroscopy pattern of (1-x) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (x) $BaTiO_3$. (x = 1.00) composite.*

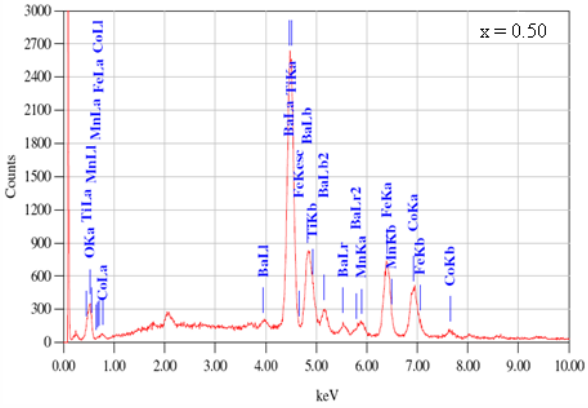

Fig 5 *Energy Dispersive Spectroscopy pattern of (1-x) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (x) $BaTiO_3$. (x = 0.50) composite.*

As piezoelectric $BaTiO_3$ content 'x' is increased in the composites the intensity of $BaTiO_3$ phase

increases as shown in Fig.5. It is observed from Fig. 4 that $BaTiO_3$ exist separately as pure at single phases. The elemental percentage of piezomagnetic and piezoelectric phase is shown in Table 2. From the standard quantitative analysis, the molar composition of piezomagnetic and piezoelectric phase is confirmed [14, 15].

Table- 2 *Elemental percentage for (1-x) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (x) $BaTiO_3$ (x= 0.25 – 1.00) composite system.*

Comp. x	Piezomagnetic			O^{2-} %	Piezoelectric	
	Co^{2+}%	Mn^{2+}%	Fe^{3+}%		Ba%	Ti%
0.00	39.26	6.08	48.77	5.88	-	-
0.25	25.65	3.91	31.97	7.92	21.18	9.38
0.50	13.1	2.34	16.34	7.62	43.27	17.24
0.75	5.91	0.75	7.00	8.13	55.36	22.86
1.00	--	--	--	5.31	67.07	27.62

4. CONCLUSIONS

The composites of piezomagnetic and piezoelectric with formula (1-x) $Co_{1.2}Mn_{0.2}Fe_{1.6}O_4$ + (x) $BaTiO_3$ has been successfully synthesized by ceramic technique with the presence of two distinct phases of piezomagnetic and piezoelectric as evidenced by the x ray diffraction techniques. From EDS, the standard quantitative analysis suggests that, the molecular composition of piezomagnetic and piezoelectric phase is confirmed.

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