"Synthesis and Structural Characterization of (1-x) $CuFe_2O_4$ + (x) $KNbO_3$ Magneto electric Composites"

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Abstract

Magnetoelectric composite materials with mixed spinal copper ferrite($CuFe_2O_4$)and perovskite potassium niobate ($KNbO_3$) as co-existing phases have been grown by solid state reaction over wide region compositions of the $CuFe_2O_4$+$KNbO_3$ system. An estimate of phase formation was obtained by X-ray diffraction (XRD).The lattice constant, X- ray density, Bulk density, Porosity such structural analysis of both the phases and their composites were estimate. The scanning electron microscopy (SEM) used to study the microstructure of the composite.

Keyword: *X-ray diffraction, Bulk density, Porosity, Scanning electron microscopy (SEM).*

Introduction:

The ME effect is a property of ME composites, which is absent in their individual constituent phase [1], such magneto –electric composites are widely used in many applications like radio electronic devices, photo electronic, microelectronic, transducers etc [2].The magneto-electric composites with piezoelectric and magneto-strictive material are of interest because of their application in many fields. Magneto-electric(ME) materials posses two or more iron based properties like polarization, magnetization, or strain as a result of electric field, magnetic field and stress.[3,4]

The ME effect occurs due to the interaction between the magnetic and electric dipoles [5]. The interrelation between two spontaneous effects Viz. Ferroelectric and ferromagnetic allows magnetic control of ferroelectric properties and vice versa. Recently, laminated ME composites synthesized by using piezoelectric and magneto-strictive materials have gained attention because they exhibit superior ME response [6-8]. It is seen from the literature survey that the higher value ME conversion factor results in the combinations having

molar percentage of ferrite phase between 10 to 50 [9–11]. It is well known that the strain caused in the piezoelectric is being mechanically coupled to induce a stress in the piezoelectric, as result of which an electric voltage is generated. In the present work the strain caused in the ferrite lattice is mainly due to the Jahn-Teller distortion caused in the ferrite lattice by Jahn-Teller ions like Cu^{2+}. These Jahn-Teller ions also induce good elastic coupling. Boomguard et al [12] have reported that the resulting signal in the magnetoelectric composites depends on various factors such as particle size, dc resistivity and mole ratio. These parameters in turn depend upon the sintering temperature and time. Thus, ME effect depends on the structural properties.

Preparation of ME composites:

ME composites of (1-x) $CuFe_2O_4$+(x) $KNbO_3$ with x= 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 mole % were prepared by solid state reaction method. Fine powders of copper ferrite ($CuFe_2O_4$) and potassium niobate ($KNbO_3$) were mixed thoroughly in molar proportion and ground for about 3 hours, 2wt. % polyvinyl alcohol was used as a binder in the mixed powders. The mixed powder is then pressed into pellets of thickness of around 2-3 mm and diameter 10 mm using a hydraulic press. A pressure of 5 ton/cm^2 was applied. The palletized samples were sintered at 1050^o C for 24 hours in a programmable furnace. The pellets were cooled slowly to room temperature to obtain heterogeneous composite.

Characterizations:

The prepared ME composite of (1-x) $CuFe_2O_4$+(x) $KNbO_3$ were characterized by using X-ray diffractometer (Philips Model PW1710). The X-ray diffraction patterns were recorded in the 2θ range of 20^o to 80^o using CuK_α radiation. The XRD patterns are taken at room temperature. The x-ray diffraction (XRD) pattern of the composite phase containing20% $CuFe_2O_4$+80%$KNbO_3$ is as shown in Fig.1.

Result and Discussion:

Structural Characteristics:

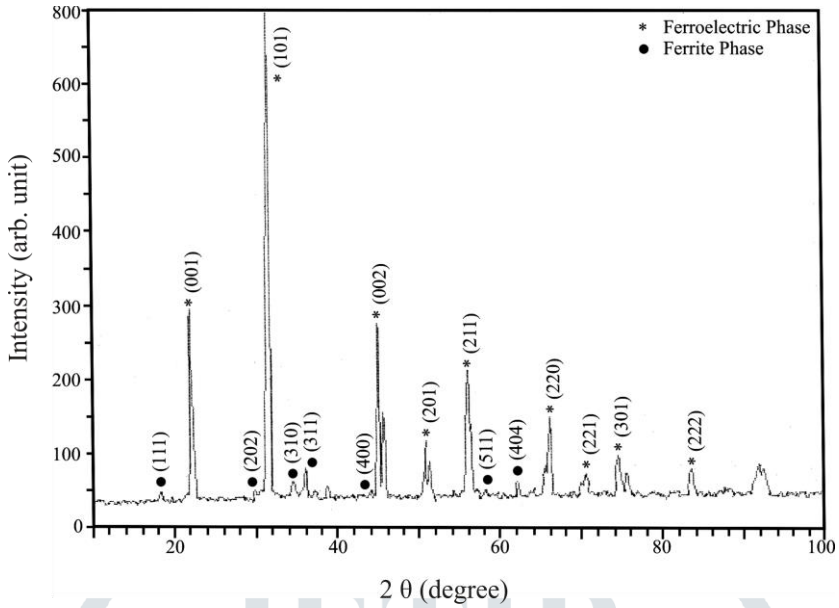

Fig.1: XRD Patterns of 20% $CuFe_2O_4$+80%$KNbO_3$ composite

Magneto-electric composite of copper ferrite ($CuFe_2O_4$) and potassium niobate ($KNbO_3$) was characterized by X-ray diffraction method. Fig.1 depicts X-ray diffraction patterns of composite (1-x) $CuFe_2O_4$+(x) $KNbO_3$ for x=0.0, 0.2, 0.4, 0.6, 0.8 and 1.0. It is reveled from the XRD patterns that the composite consist of $CuFe_2O_4$+ $KNbO_3$ as predominant phases. A close examination of XRD pattern indicates that the intensity of ferroelectric ($KNbO_3$) (101) plane increases with increasing the percentage of $KNbO_3$. On the other hand the intensity of ferrite ($CuFe_2O_4$) (311) plane decreases with decreasing percentage of ferrite. The XRD patterns of composite material under investigation are as per ASTM (06-0545 for $CuFe_2O_4$, 71-0945 for $KNbO_3$) data. The XRD pattern clearly shows two separate phase belonging to spinel ferrite ($CuFe_2O_4$) and ferroelectric ($KNbO_3$).

The XRD patterns were used to determine the structural parameter like lattice constant of ferrite phase and ferroelectric phase separately. All the peaks of XRD patterns are well indexed, using Bragg's law. The values of lattice constant obtained from XRD data are given in Table 1. The values of lattice parameters 'a' and 'c' for x = 0.0, i.e. for pure copper ferrite phase are in close agreement with the reported values [13]. The lattice constant of ferroelectric phase was also calculated using XRD data and the values are also given in the table. The ferroelectric $KNbO_3$ is also a tetragonal and the values of lattice parameter 'a' and 'c' are almost same for all the compositions. The values of lattice parameter of ferroelectric $KNbO_3$ is also in good agreement with the literature value.

Table1. Structural data for (1-x) $CuFe_2O_4$+(x) $KNbO_3$

Comp. 'x'	Lattice parameter (Å) Ferrite	Ferroelect.	X-ray density 'd_x' (gm/cm^3)	Bulk density 'd' (gm/cm^3)	Porosity P (%)	Particle size 't' (Å)	Average grain size (μm)
0.0	a = 8.233 c = 8.727	------ -----	5.373	4.019	25.188	234	----
0.2	a = 8.229 c = 8.723	a =3.961 c = 4.027	5.197	3.640	29.970	191	1.38
0.4	a = 8.218 c = 8.711	a =3.989 c = 4.056	5.061	3.989	21.183	218	1.46
0.6	a = 8.203 c = 8.696	a = 3.999 c = 4.056	4.981	3.691	25.897	213	1.67
0.8	a = 8.159 c = 8.649	a = 3.991 c = 4.057	4.794	3.447	28.105	227	1.82
1.0	------ -----	a = 3.991 c = 4.058	4.625	3.345	27.680	208	-----

The volume of the samples was calculated using the values of lattice parameter (a and c). The estimated values of volume of ferrite, ferroelectric and composite samples are used to find the X- ray density of ferrite phase and ferroelectric phase was calculated using the values of molecular weight and volume of the respective samples. The values of X-ray density for ferrite phase, ferroelectric phase and composite phase are given in Table1. It is observed from table that the X-ray density of ferrite phase decreases with the addition of $KNbO_3$. Similarly the X-ray density of ferroelectric phase and ME composite phase also decreases with the addition of $KNbO_3$. The decrease in X-ray density of ferrite phase is related with the decrease in mass overtakes the decreases in volume of the ferrite phase. The decrease in X-ray density of ferroelectric phase is related with the molecular weight and volume of the ferroelectric phase. The molecular weight of ferroelectric phase increases with the addition of $KNbO_3$, at the same time volume also increases with the addition of ferroelectric phase. The decrease in X-ray density of ferroelectric phase is due to the predominance of increase in volume over the increase in molecular weight.

The molecular weight of composite material goes on decreasing with the addition of $KNbO_3$; similarly volume of composite also decreases with addition of $KNbO_3$, both mass and volume of composite decreases with increase in $KNbO_3$. The decrease in mass (molecular weight) overtakes the decrease in volume and hence the X-ray density of composite decreases with addition of $KNbO_3$. Further it is noticed that the rate of decrease of X-ray density with composition of ferrite and ferroelectric is fast where as it is slow for composite material.

The values of bulk density for composite material under investigation were obtained by Archimedes liquid immersion method**.** The percentage porosity 'P' of composite material was also determined for each composition 'x' and the values are also presented in table 1. The porosity values are in the range 21-29 %.

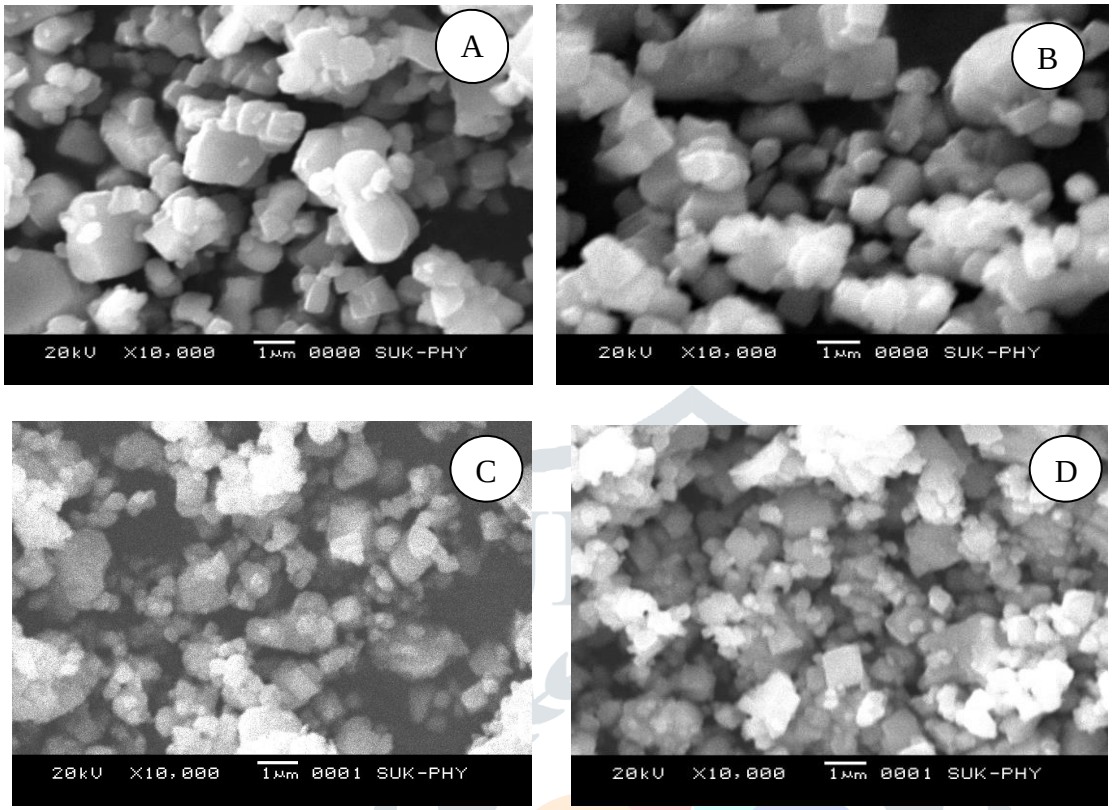

Fig.2. SEM micrographs of (1-x) CuFe2O4 + (x) KNbO3 With (A) x= 0.2, (B) x = 0.4, (C) x= 0.6 , (D) x = 0.8

The scanning electron microscopy is one of the powerful techniques used to study the microstructure of the composite. Fig. 2 shows SEM micrographs of the (1-x) $CuFe_2O_4$+(x) $KNbO_3$ for typical samples (x =0.2, 0.4, 0.6 and 0.8). These SEM photographs were used to obtain grain size using intercept method. The average grain size calculated from SEM is listed in Table 1. The grain size is found to increase with increasing $KNbO_3$ content. The grain size for (1-x) $CuFe_2O_4$+(x) $KNbO_3$ with x = 0.2, 0.4, 0.6 and 0.8 lies between 1.82 to1.38µm. The grain size of composites is found to decrease with increasing ferrite content. This may due to the fact that the average grain size of the ferrite phase is smaller than average grain size of the ferroelectric phase and the composites are prepared in ferroelectric rich region.

The results of structural studies of the composite ($CuFe_2O_4$+$KNbO_3$) are in analogues with the reported results [14-16].

Conclusions:

The composite material of (1-x) $CuFe_2O_4$ + (x) $KNbO_3$ was successfully synthesized by standard ceramic technique. The XRD patterns revealed the formation of clearly distinguishable two phase diffraction peaks corresponding to ferrite phase and ferroelectric phase. The structural parameters like lattice constant, X-ray density, bulk density, and porosity derived in the present study closely agrees with reported values. Well defined grains appeared in the SEM photographs. Average grain size derived from SEM images gives grain size of around 1.5 µm.

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