Structural and magnetic behavior of spinel CuMn$_2$O$_4$ synthesized by co-melting technique

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ABSTRACT

Spinel type CuMn$_2$O$_4$ compound was prepared by co-melting technique. The product was synthesized by the fusion of high purity copper and manganese metals in the presence of oxygen atmosphere. The formation of CuMn$_2$O$_4$ has been confirmed through X-ray diffraction analysis and a small percentage of secondary phases of oxides of Cu and Mn have been observed. The magnetic measurements carried out by VSM magnetometer shows the room temperature antiferromagnetic behavior. The occurrence of antiferromagnetism in such a compound is possibly due to the super exchange interaction between Mn ions.

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1. Introduction

Spinels are very well known for its flexibility in incorporating various ions into their crystal structure. The magnetic spinel ferrites have been used in electrical engineering like microwave devices and inductors. But magnetic, electric, thermoelectric and catalyst properties [1–3] of copper manganite spinels demand a wide range of applications in various engineering sectors. The multivalent nature of Mn and Cu in CuMn$_2$O$_4$ spinels makes the material to be special for engineering applications and become the source of the various unique properties. Especially the orientation of the site of Mn and Cu in copper manganite spinels is the main cause of magnetic properties [4–7]. Sometime, the processing technique [4,5,8] also evolves the unique properties of these samples. The unique properties of these samples depend upon the structure. It is observed that the crystal structure of CuMn$_2$O$_4$ is usually tetragonal due to Jahn-Teller distortion, but under some preparation conditions, it can become cubic [9]. The importance of spinel type CuMn$_2$O$_4$ has been attracting a lot of attention to the researchers due to its possible application as catalysis for oxidation of CO at ambient temperature and for respiratory protection, particularly in the mining industry [10–12]. Especially, in the mining industry, volatile organic compounds (VOCs) are recognized as major air pollutants, because of their toxic properties. VOCs are carcinogenic and can cause disease to skin, neural systems, eyes and other organs of living being.

Thermal incineration and catalytic incineration are the highly efficient techniques for destroying VOCs [13,14]. The common catalytic incineration used to destroy the VOCs is CuMn$_2$O$_4$ [15] and Copper manganese oxides are highly active catalysts for ethylene oxidation [11]. The fabrication of single phase CuMn$_2$O$_4$ is a challenging one as there is more probability of formation of the secondary phases of Mn during synthesis. To avoid such a difficulty, researchers have been focused for the synthesis of such compound by the fusion of copper with manganese metals. However, the slow step sintering process adopted for this often produces secondary phases. As, it is known that in a spinel structure of CuMn$_2$O$_4$, Cu$^{2+}$ is located in tetrahedral sites and Mn$^{3+}$ in octahedral sites. This kind of elemental distribution is known as normal spinel, the corresponding general formula is written as A$^{2+}$B$^{3+}$O$_4$ [16]. The high activity of this spinel compound is attributed to the amounts of Mn$^{4+}$ species, and compositional homogeneity. Incorporation of copper enhances the activity of resultant bimetallic oxides. Hence, it is very much important to choose a suitable synthesis technique to prepare high pure single phase CuMn$_2$O$_4$.

Keeping view on above aspects, we have adopted a simple fusion technique to prepare CuMn$_2$O$_4$ out of Cu and Mn metals using a high temperature conventional furnace through oxygen atmosphere. The structural analysis has been done to know the purity of the compound and the antiferromagnetic contribution obtained in the compound has been discussed through super exchange interaction.

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2. Experimental details

High pure Cu plate and Mn metal powders were taken for the synthesis of CuMn₂O₄ compound. Both Cu and Mn metals were taken in a stoichiometry weight ratio of 1:2 in a recrystallized alumina crucible. The crucible was placed in the hot zone of a high temperature furnace for co-melting at a temperature of 1250 °C with a variation of ± 1 °C. It is known that the melting point of Cu (1085 °C) is less than melting point of Mn (1246 °C). To fuse both the metals simultaneously, the temperature of the furnace was raised to 1250 °C with a heating rate of 600 °C per hour in presence of oxygen atmosphere. The materials were kept at the same temperature for five hours for uniform mixing of Cu and Mn melt and then it was rapidly cooled by switching off the furnace at high temperature. The fused samples were now considered for various characterizations. The phase and structural analysis of the samples was carried out using X-ray diffractometer (XRD) where Cu Kα (1.540 Å) is taken as the X-ray irradiation source. The room temperature magnetic study has been done by suing VSM magnetometer. Electron Spin Resonance (ESR) signals was obtained by using a Bruker EMX spectrometer, operating at 9.45 GHz and a field modulation frequency of 100 Hz. The DC magnetic field was swept from 0 G to 10 kG.

3. Results and discussion

The XRD pattern of CuMn₂O₄ compound recorded using CuKα radiation is shown in Fig. 1. The most intensified peak of CuMn₂O₄ is observed at a diffraction angle of 36.40° with a shoulder peak at 36.95° which indicates that the synthesized sample exhibit tetragonal structure. The lattice parameter ‘a’ and ‘c’ calculated using the FullProf software for CuMn₂O₄ are 5.820 Å and 8.694 Å respectively which is close agreement with the corresponding reported values of 5.818 Å and 8.658 Å (JCPDF # 711142). The average crystallite size calculated using Scherrer’s equation [17] is about 24 nm. Apart from CuMn₂O₄ diffraction peaks, a small percentage of secondary phases of oxides of Cu and Mn have been observed. SEM micrographs of CuMn₂O₄ spinel compound is shown in inset of Fig. 1. The SEM study shows that fused micrograins are observed with porous structure. The grains are well connected and the average grain size is in the order of 5 µm. FTIR spectrum of CuMn₂O₄ spinel compound is shown in Fig. 2. Five absorption bands in the spectrum range of 450–4000 cm⁻¹ were investigated. The 1st band is appeared at around 588–606 cm⁻¹ which corresponds to the Mn–O [18]. The 2nd band appears at around 846–860 cm⁻¹ shows the signal of Cu–Mn ions and 4th band in the range 1000–1633 cm⁻¹ shows the signal of OH⁻ group. The 5th band signal shows the presence C–O and C=O groups when band in a range greater than 1633 cm⁻¹ in the spectrum. The observation of FTIR signifies the formation of CuMn₂O₄ spinel compound with appearance of oxides of Cu and Mn.

ESR spectrum of CuMn₂O₄ spinel compound is shown in Fig. 3. Low field absorption observed in the spectrum (inset Fig. 3) represents the compound has inherent magnetic properties. The broad nature of peak to peak width (750 G) indicates the inhomogeneous arrangement of magnetic phases and the antiferromagnetic nature of the compound which is confirmed from the VSM measurement. The magnetic field dependent magnetization at 300 K is shown in Fig. 4. The room temperature antiferromagnetic behavior with coercivity of 1085 Oe is observed in the M–H plot. The antiferromagnetic behavior is due to super exchange interaction between Mn ions. It is also evidenced by the ‘g’ factor (3.38) obtained from ESR data analysis. The ‘g’ value above than the free electron value confirms the presence of exchange interactions.

Fig. 1. X-ray diffraction pattern of CuMn₂O₄ compound (* indicates the impurity oxide phases of Cu and Mn) and inset shows the scanning electron micrograph.

Fig. 2. FTIR spectrum of CuMn₂O₄ compound.

Fig. 3. Electron spin resonance spectrum of CuMn₂O₄ compound.

Fig. 4. Room temperature magnetic behavior with coercivity of 1085 Oe is observed in the M–H plot.
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