Influence of Mn Substitution on Structural and Electrical properties of $MgAl_2O_4$ Synthesized by sol-gel Auto Combustion method

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Abstract

Nanostructured manganese substituted magnesium aluminate ($Mg_{1-x}Mn_xAl_2O_4$, where x = 0.00,0.25,0.50,0.75,1.00) were prepared by sol-gel autocombustion method.The prepared samples were characterised by XRD,SEM,EDAX,TEM and IR spectroscopy.The study of XRD shows the formation of single cubic spinel phase with average crystalite size 28 nm.The composition of $Mg_{0.50}Mn_{0.50}Al_2O_4$showspherical interlinked fibrous morphology.Partical size obtained from TEM analysis was found to be 27 nm.The IR spectra shows two strong characteristic absorption bands at tetrahedral and octahedral sites.The elemental compositions was studied by energy dispersive X-ray analysis (EDAX).

Key words: Sol-gel method, XRD, EDAX, SEM, IR.

1. Introduction

Magnesium aluminate oxide spinel has been widely studied as it has a specific combination of desirable properties such as: excellent optical properties, good mechanical strength at room temperature as well as high temperatures, high melting point (2135^oC), high chemical inertness against both acidic and basic slag's, low dielectric constant, low thermal expansion and good catalytic properties [1-6]. Therefore, it has been extensively used for various purposes as structural material in fusion reactors, refractory material, vacuum induction furnaces, steel ladles, luminescent host, cement rotary kilns, active element in humidity sensor, excellent transparent ceramic material for high temperature arc-enclosing envelops and catalyst or catalyst support in the field of environmental catalysis [7-11].

In recent years, various wet-chemical techniques or wet-chemical assisted techniques, such as hydroxide co precipitation [12-13],flame spray pyrolysis, modified pechini process [14], sol-gel of metal alkoxides or inorganic salts [15-16], have been developed and successfully used for the production of pure spinel powders.

In this article, we report the structural, electrical and morphological propertise of manganese substituted magnesium aluminates synthesized by sol-gel auto combustion method.

2. Experimental

Nanocrystaline $Mg_{1-x}Mn_xAl_2O_4$samples were prepared by simple sol-gel autocombustion method. The A. R. grade citric acid ($C_6H_8O_7.2H_2O$), Alluminium nitrate[$Al(NO_3)_36H_2O$], Magnesium nitrate [$Mg(No_3)_26H_2O$], and Manganese nitrate [$Mn(No_3)_24H_2O$] were used as a starting material. The samples were prepared by sol-gel autocumbustion method as prepared in our previous work [17].

3 Results and discussion

3.1 XRD study

X-ray diffraction patterns of $Mg_{1-x}Mn_xAl_2O_4$(where x = 0.00,0.25,0.50,0.75,1.00) are shown in Fig.1. The diffraction patterns and data proved that the samples have been formed in a cubic spinel structurebecause of their small size giving rise to high specific-surface-area.These particles may be sinterable at a temperature lower than 1400^0C.The crystallite size of the prepared powders is calculated from XRD line broading of (311) peak using Scherrer's equation [18].

$$D = 0.9\lambda/\beta\cos\theta$$

Where β is the FWHM of the most intense peak (311), θ is the Bragg angle for the (311) peak and λ is the wave length of Cukα = 1.54 A^0.From above equation the average crystal size of the $Mg_{1-x}Mn_xAl_2O_4$ was estimated to be 28 nm.The lattice parameter as a function of composition is tabulated in Table 1. From this table one can clearly see that, lattice parameter and X-ray density increases with increasing Mnconcentration.

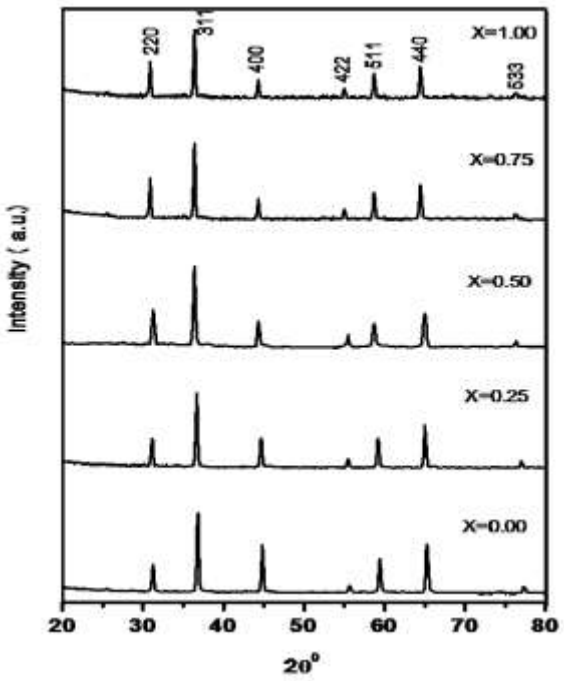

Fig.1. XRD patterns of the$Mg_{1-x}Mn_xAl_2O_4$(where x = 0.0,0.25,0.50,0.75,1.0).

3.2 SEM and EDAX analysis

Scanning electron microscopy (SEM) is an excellentmethod to study morphology of the sample. The SEMmicrograph of Mg0.5Mn0.5Al2O4are shown in Fig. 2.The EDAX results suggested that the precursors have fully undergone the chemical reaction to form the expected alluminate material. The reason for making EDAX characterization of the samples was to ratify the purity and surety of the chemical composition of the prepared fine particle powders. Fig.3 shows the SEM microphotograph of the Mg-Mn alluminate(x = 0.50) sintered at 900 ^{0}C. It is clearly seen that densified alluminate with a fine-grained microstructure behavior.

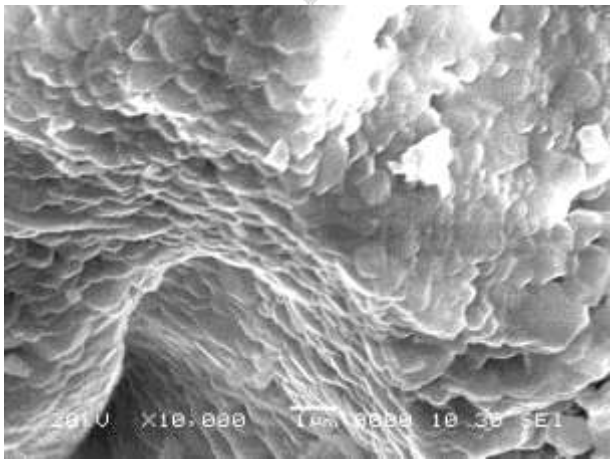

Fig.2. SEM Photographs of $Mg_{1-x}Mn_xAl_2O_4$(x = 0.50)

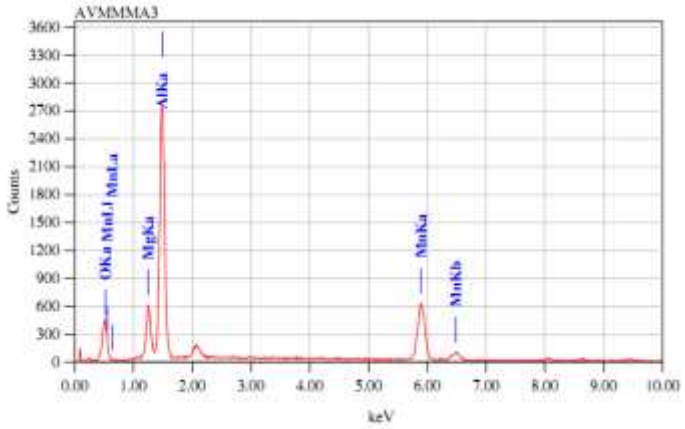

Fig.3. EDAX pattern for the sample $Mg_{0.5}Mn_{0.5}Al_2O_4$(X = 0.50)

3.3 Infrared analysis

The FTIR spectra were recorded in the range of 350-700 cm^{-1}. The band wavenumber ν_1 and ν_2 observed in the range 625-550 $cm^{-1}$and 450-375 cm^{-1} respectively. ν_1 band corresponds tointrinsic stretching vibrations of the metal at the tetrahedral site, Mtetra ↔ O,where as ν_2 lowest band is assigned to octahedral-metal stretching, Mocta ↔ O**[**19].The decreases of wavenumber with the increases of Mn^{+2} was shown in Fig.4.

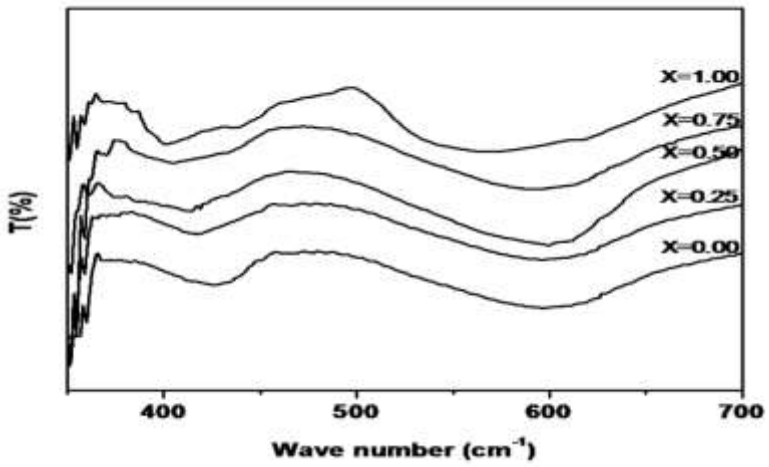

Fig.4.Infrared spectra of ($Mg_{1-x}Mn_xAl_2O_4$,where x = .0,00.25,0.50,0.75.1.00)

3.4 Electrical Properties

The temperature dependent variation of dc resistively for$Mg_{1-x}Mn_xAl_2O_4$is shown in Fig.5. Linear decrease in resistivity withincreasing temperature reflects semiconducting nature of ferrites.The conduction mechanism in ferrites is explained on the basis ofVerwayDeBoher mechanism that involves exchange of electronsbetween the

ions of the same elements present in more than onevalence state and distributed randomly over equivalent crystallographiclattice sites. The decrease in resistivity with increase intemperature is attributed to increase in drift mobility of the chargecarriers.

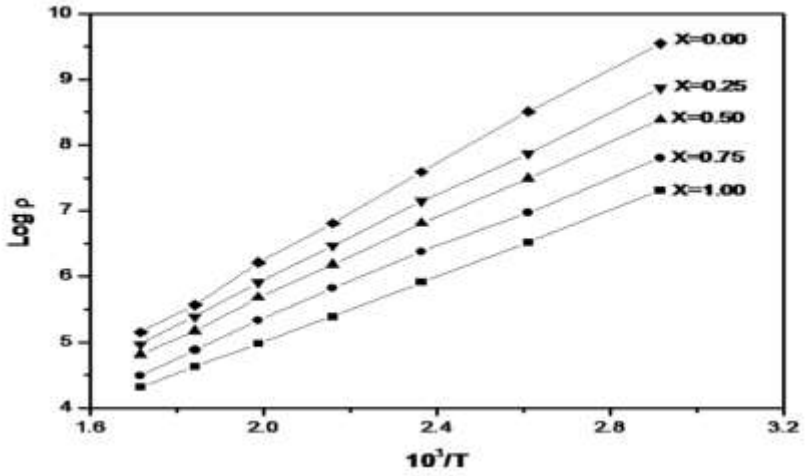

Fig.5. Electrical conductivityof the$Mg_{1-x}Mn_xAl_2O_4$(where x = 0.0,0.25,0.50,0.75,1.0).

4. Conclusion

Spinel alluminate of $Mg_{1-x}Mn_xAl_2O_4$was synthesized by simple sol-gel autocombustion method.XRD analysis shows that, as the composition of Mn^{+2} increases then crystal sizedecreases and X-ray densesity increases.The average crystalite size of the system was 27 nm.SEM analysis shows fine grained microstructure.EDAX analysis of the nanomaterial confirm that the material is composed of Mg,Mn,Al and O without any impurity.Unit cell parameter increases with increases in Mn concentration.

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