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Liquefied petroleum gas sensor based on Cr^{3+} substituted mixed Mg-Cd spinel ferrites

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ABSTRACT

The Cr^{3+} substituted Mg-Cd ferrite powder was synthesized by standard ceramic route. The crystal structure and formation of spinel ferrite were examined by XRD and FT-IR techniques. The particle size has been estimated using Debye-Scherrer equation. All the peaks in the XRD pattern are narrow which means that particles are to nano range. The structure inferred from the XRD and FT-IR is cubic. The samples were tested for LPG gas. The sensitivity was measured at various temperatures. The sensitivity was found to maximum at 300^0C. The response time and recovery time was also measured.

Key words: gas sensing, Mg-Cd ferrites, Cr^{3+} substitutions.

INTRODUCTION

The ferrites are the metal oxides, which have been the subject of extensive investigations. These oxides possess unique magnetic, magneto-optical, magneto resistive, thermal, electrical, and mechanical [1-4]. Recently ferrites have been investigated for gas sensors. Many investigators reported the preparation and gas sensing properties of ferrites [5-7]. During the last decades many kinds of ceramic oxides have been investigated actively as humidity sensing materials [8]. Gadkari et al have reported the gas sensing property of Magnesium [6] and Mg-Cd ferrite [7]. They reported that the sensitivity of samples depends on the kind of ferrite, grain size and specific surface area. Kadu et al [9] showed that Mg-Zn nanomaterial has high response at operating temperature of 300^0C**.** Number of inorganic materials are used for the manufacturing of sensors. Among these, the use of ceramics for sensing purpose has attracted singnificant attention due to the low cost of raw materials. Ferrites with their inherent thermal stability and structural characteristics using phenomena of bulk and grain boundaries are suitable material for various gas sensors.

Many types of methods are being used for preparation of ferrites, including Ceramic synthesis, co-precipitation method, tartrate precursor method, hydrothermal method, combustion, auto combustion and sol gel technique etc. The standard ceramic method is widely used and is applicable for industry for preparation ferrite samples in bulk form.

In this communication we report the preparation, characterization and Liquefied Petroleum gas (LPG) sensing properties of Cr^{3+} substituted Mg-Cd ferrite.

MATERIALS AND METHODS

A ferrite samples with general formula $Cd_xMg_{1-x}Fe_{2-y}Cr_yO_4$ (x = 0.2, 0.4, and 0.6 ; y = 0.05 and 0.1) were prepared by double sintering standard ceramic method using starting oxides Fe_2O_3, $CdCO_3$, MgO and Cr_2O_3 (AR grade, LOBA Chemi India). These oxides were weighed on the single pan microbalance in the required molecular weight proportion and were mixed and milled thoroughly for about 2 hours. The powder was presintered in a temperature-controlled furnace at temperature 700 ^{0}C for 12 hours. The furnace temperature was measured by using Chromel-Alumel thermocouple. The furnace was cooled at the rate of 80 ^{0}C per hour to room temperature. Sintering process was carried out at the temperature 1050 ^{0}C for 24 hours. The sintered compositions were then mixed with 5% PVA and again milled thoroughly.

The pellets of 1cm diameter were prepared by using hydraulic press. These pellets were again sintered at 1050 ^{0}C for 24 hours for better compaction. Using soft metal paper, pellets were polished. These pellets were used as sensor element for testing of LPG.

Physical Measurements

The powdered samples of each composition was characterized by X-ray diffraction method (XRD) on Philips computerized X-ray diffractometer (PW 3710) using Cu-Kα radiations. The samples were also characterized by IR absorption spectroscopy at room temperature, in the range of 350 cm^{-1} to 800 cm^{-1} by using FTIR spectrophotometer

Gas sensing

The schematic diagram of the gas sensing set up is as shown in **Fig. 1.** It consist test chamber, made up of thick glass which is insulated properly to avoid the heat losses. The sensor element (ferrite sample) was placed on the heater. The contact electrodes were connected on the surface of the sensor element. The DC voltage of 5 volt was applied across the sensor element. The temperature was controlled with an accuracy of +/-1 ^{0}C. Dry air was used as a carrier gas and allowed with a constant flow rate. LPG and Ehtanol was injected in the chamber and the resistance change across pellet was measured before and after passing test gases through chamber in the temperature range 200^{0}C to 400^{0}C. The sensitivity of the sample was calculated by using the relation [10],

$$S (\%) = \Delta R/Ra *100 = Ra-Rg/Ra *100 \quad (1)$$

RESULTS AND DISCUSSION

Characterization

The X-ray diffraction pattern of $Cd_xMg_{1-x}Fe_{2-y}Cr_yO_4$ with x= 0.2 and y=0.05 is presented in **Fig. 1.**The analysis from the X-ray diffractogram reveals the formation of single phase cubic spinel structure. The average crystallite size is calculated by using the Debye-Scherrer formula $D= 0.9\lambda / \Delta \cos\theta$, Where, θ is angle of diffraction, λ= wavelength of XRD & Δ = measured in radian as a full width at half maximum of the diffraction line. It is nearly 0.05^0. The values are present in **Table 1.** The average crystallite size D is 74 nm. The grains are irregular in shape and separated by pores. These pores serve as adsorption sites and sensitivity depend on size of pores. The FT-IR spectrum is presented in **Fig.2,** which shows two absorption bands in the frequency range 350-800 cm^{-1}. The high and low frequency absorption band (υ_1, υ_2) are observed in the frequency range 560 to 580 cm^{-1} and 432 to 475 cm^{-1} respectively. These bands are characteristics of spinel ferrites [11].

Sensing mechanism

The sensor works on the principle of change in resistance or capacitance due to the adsorption gases [13]. It is well known that the gas sensing phenomena is surface effect of gas- solid interaction. It is assumed that the chemiabsorbed oxygen of related species such as OH^-, O^{2-} and O are responsible for the condition behaviour of gas sensor. When the sensor is exposed to reducing gases, they react with the chemiabsorbed oxygen, there by releasing the trapped elelctron back to the conduction band and decreases the resistance of the sensor materials. The following reactions will take place [14].

$$O_{2\,gas} \longleftrightarrow O_2^-{}_{ad}$$

$$O_{2ad} + \longleftrightarrow 2O^-{}_{ad}$$

$$O^-_{ad} + e^- \longleftrightarrow O^{2-}_{ad.}$$

The adsorption of the gas depends on the type of the test gas, sensitivity of the materials and response time. The variation of gas sensitivity with operating temperature of $Cd_xMg_{1-x}Fe_{2-y}Cr_yO_4$ sensor for LPG and ethanol is presented in the **Fig.3** and **Fig.4.** Theses figures shows that the sensitivity reaches maximum at temperature 300^0K optimum temperature and decreases at higher temperature for both gases. The temperature at which maximum desorption takes place is known as optimum temperature. This temperature is enough to complete the chemical reactions in the maximum absorption of LPG. As temperature increases further, the oxygen at the surface became less and hence the sensitivity reduces after optimum temperature. The reaction takes place during the adsorption and desorptions is given by the relation [15],

$$C_nH_{2n+2} + 2O^- \longleftrightarrow C_nH_{2n} - O + e^-$$

From the figures it is also found that the sensitivity increases with the addition of Cr^{3+} in Magnesium-cadmium ferrite. The increase in porosity may be responsible for more adsorption of gases. At low temperature, the gas response is limited by the rate of diffusion of gas molecules restrict the gas response. At optimum temperature the rate of chemical reaction and diffusion of gas molecules becomes equal, attaining the equilibirium at that temperature, the sensor shows maximum response.

The recovery time is the time taken by the sensor to reduce the sensitivity 90% of its maximum value. **Fig. 5** explains the response and recovery time for the ferrites under investigation at operating temperature 300^0C for LPG. From the figure it can be noticed that the response and time is shorter $MgFe_2O_4$.

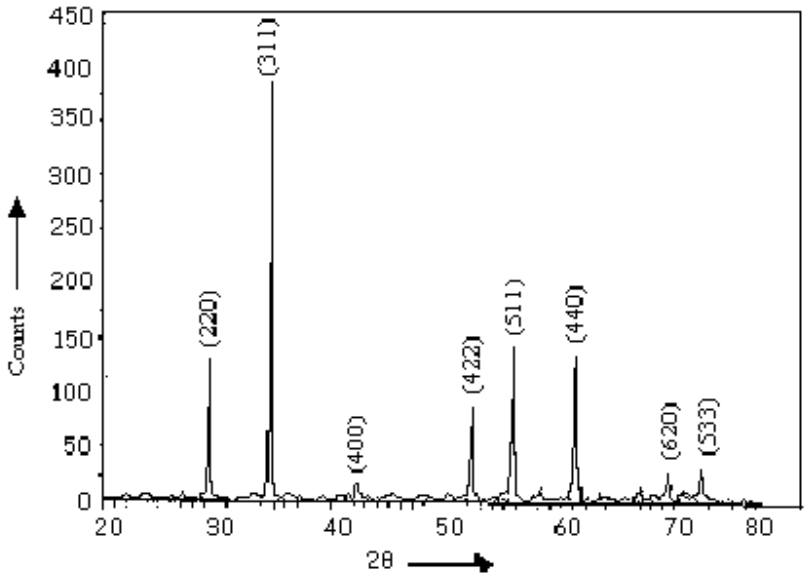

Fig.1 X-ray diffraction pattern of the ferrite sample $Cd_{0.4}Mg_{0.6}Fe_{1.9}Cr_{0.1}O_4$

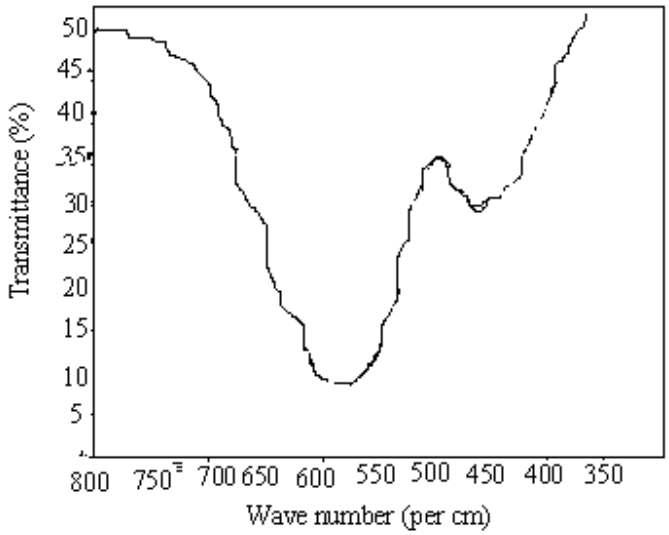

Fig. 2 FT-IR spectra of $Cd_{0.4}Mg_{0.6}Fe_{1.9}Cr_{0.1}O_4$

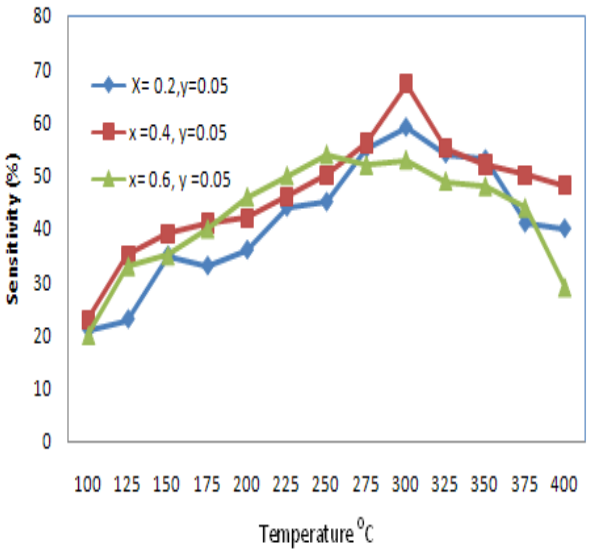

Fig.3 Variation of sensitivity with operating temperature

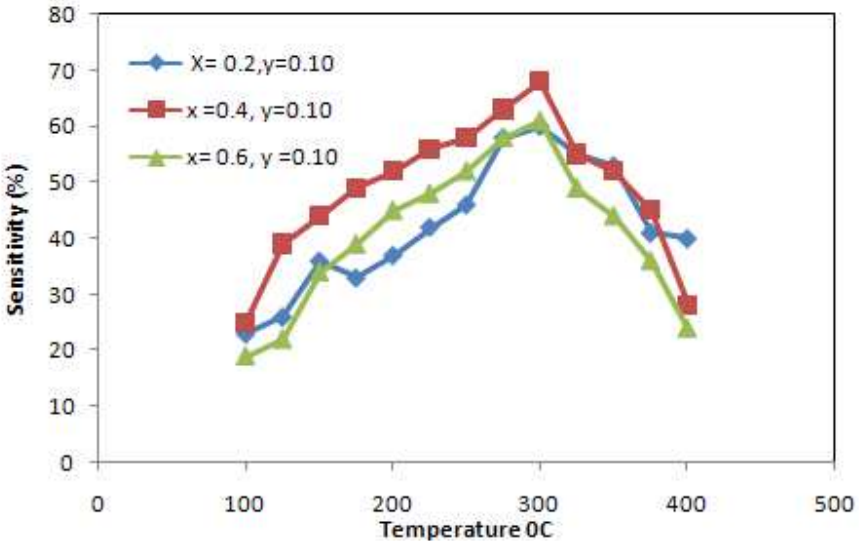

Fig.4 Variation of sensitivity with operating temperature

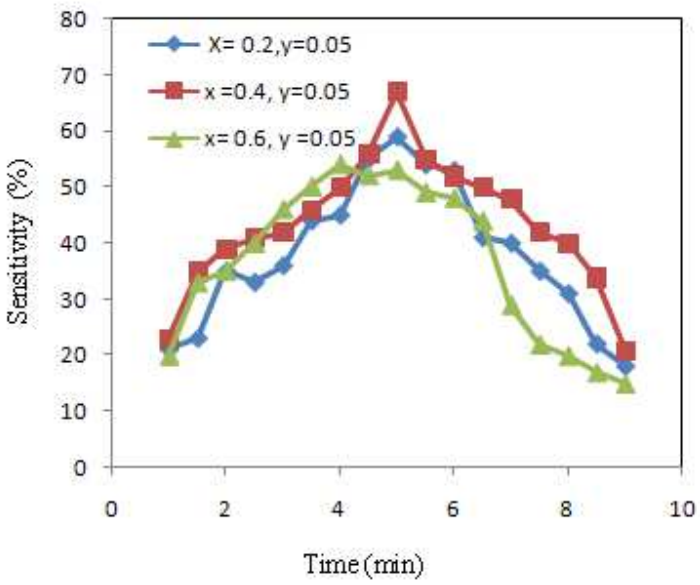

Fig. 5 Response and recovery time of $Cd_xMg_{1-x}Fe_{2-y}Cr_yO_4$ in presence of LPG

Table 1. Parameters estimated from XRD and gas sensing measurements of $Cd_xMg_{1-x}Fe_{2-y}Cr_yO_4$

Ferrite sample		Lattice constant A^0	Crystalline size nm	Operating temperature ^0C
x= 0.2	y = 0.05	8.42	75	300
x = 0.4		8.48	72	302
x = 0.6		8.54	64	310
x= 0.2	y= 0.10	8.40	71	299
x = 0.4		8.46	70	295
x = 0.6		8.52	66	300

CONCLUSION

The ferrite was prepared by standard ceramic method and was tested for LPG. The Cr^{3+} substituted Mg-Cd ferrite shows good response to the liquefied petroleum gas at the optimum temperature 300^0C. The response and recovery

time also reduced extent so that the chromium substitution in Mg-Cd ferrite is suitable for gas sensing purpose. From the study it found that the sensitivity depend on operating temperature and type of the gas and also microstructure.

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