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Gas sensing properties of magnesium ferrite prepared by co-precipitation method

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ABSTRACT

Polycrystalline magnesium ferrite ($MgFe_2O_4$) was prepared by the co-precipitation method. The synthesized compound was characterized for their phase and morphology by X-ray diffraction and scanning electron microscopy, respectively. Conductance responses of the ($MgFe_2O_4$) were measured towards gases like hydrogen sulfide (H_2S), liquefied petroleum gas (LPG), ethanol vapors (C_2H_5OH), SO_x, H_2, NO_x, NH_3, methanol, acetone and petrol. The gas sensing characterstics were obtained by measuring the sensitivity as a function of various controlling factors like operating temperatures and concentrations of gases. It was found that the sensor exhibited various responses towards these gases at different operating temperatures. Furthermore; the $MgFe_2O_4$ based sensor exhibited a fast response and a good recovery towards petrol at temperature 250 °C. The results of the response towards petrol reveal that ($MgFe_2O_4$) synthesized by a simple co-precipitation method, would be a suitable material for the fabrication of the petrol sensor.

1. Introduction

Applications of gas sensors have been growing at a consistent pace in the recent years. The mixed-metal oxide gas sensors offer advantages over other gas sensors devices due to their simple implementation, low cost and good reliability for real time control systems [1]. Gas sensors have a great influence in many areas such as environmental monitoring, domestic safety, public security, automotive applications, spacecrafts, houses and sensors networks [2]. Detection is necessary in different fields such as industrial emission control, household security, vehicle emission control and environmental monitoring [3–6].

Spinel ferrites synthesized by the conventional ceramic method have a limitation as a gas sensor. The literature survey reports response of ferrites as sensors towards various gases. Gopal Reddy et al. [7] reported nickel ferrite exhibiting good response towards chlorine. Mulla and co-workers [8] synthesized zinc ferrite which gives sensitivity towards H_2S gas. While Liu et al. [9] reported doped noble metal nickel ferrite to be sensitive toward H_2S. Recently, Xiangfeng et al. [10] synthesized nanotubes and nanorodes of nickel ferrite using a hydrothermal method that was found to be sensitive towards triethylamine. Most of the researchers have focused on detection of LPG, SO_x, H_2S, H_2, NO_x and NH_3 because of their toxicity, their relation with atmospheric composition or to their high levels in some environments. Organic vapors such as methanol, acetone and ethanol have also been detected [11,12].

In the present work, we report magnesium ferrite prepared by a co-precipitation method. The resulting compounds have been studied for their gas sensing properties under different conditions.

2. Experimental

The magnesium ferrite has been synthesized by using co-precipitation technique. A.R. grade magnesium sulphate and ferrous sulphate were dissolved in the appropriate proportion. The metal salts were then precipitated as hydroxides using 10% NaOH solution maintaining pH 10. Hydroxides were then oxidized using 30% H_2O_2 (100 Vml) solution. The precipitate was washed and filtered till it became free from sulphate and excess alkali. The precipitate was dried under vaccum at 110 °C followed by sintering at 900 °C for 4 h.

2.1. Phase identification

X-ray powders diffraction patterns were recorded on a diffractometer (Philips PW 1730) with microprocessor controller, using CuKα radiation (λ = 1.5428 Å).

2.2. Characterization

The variation in the DC resistivity with temperature (RT to 500 °C) was measured by the two-probe method.

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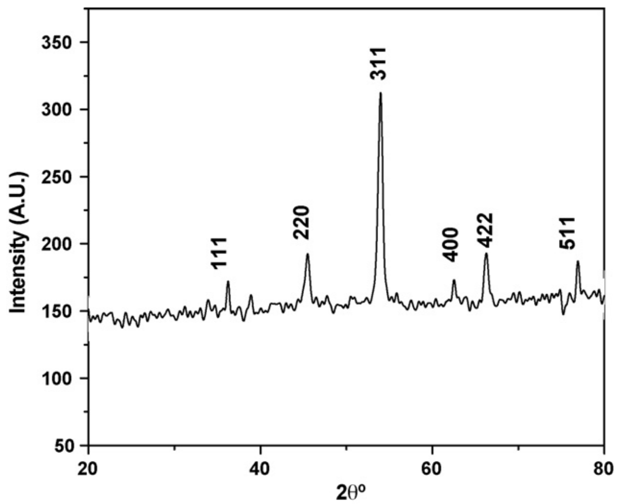

Fig. 1. X-ray pattern for $MgFe_2O_4$ sample sintered at 900 °C.

2.3. Gas sensing Characterization

The gas sensor was made by pressing the powder in the form of pellet. The gas sensing characteristics with reference to time at different operating temperatures and concentrations were recorded. The gas response (S) for a given test gas was calculated using following equation.

$$S = \frac{R_a}{R_g}$$

where, R_a and R_g are the resistance of the sensor in air and in the test gas, respectively.

3. Results and discussion

3.1. Structural properties

The X-ray diffraction pattern of the powder sintered at 900 °C for 4 h is shown in Fig. 1. It indicates formation of single phase spinel structure, matching with the standard data card No. # 73-1720 [13]. The lattice constant have been calculated from the spectrum and found to be a = 8.34 Å, which are in good agreement with the reported values. While the average crystallite size calculated by using Scherrer formula is about 40 nm.

Scanning electron micrograph for the sample is shown in Fig. 2.indicates irregular shape of fine particles. The particle size

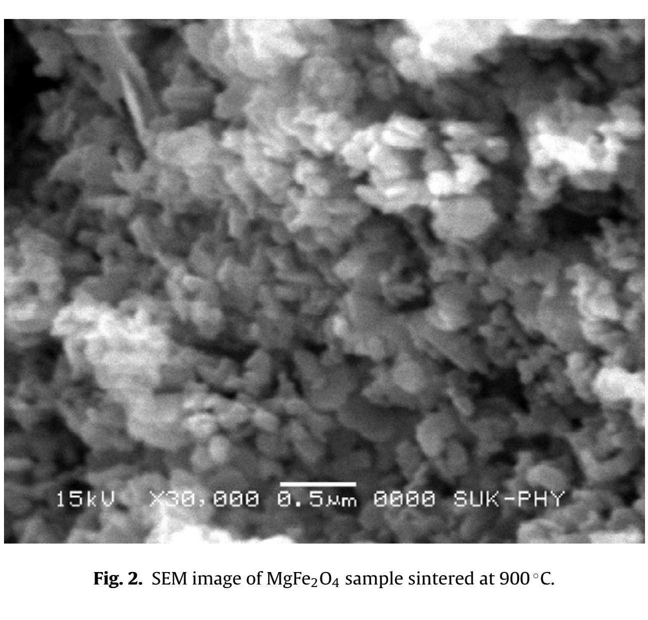

Fig. 2. SEM image of $MgFe_2O_4$ sample sintered at 900 °C.

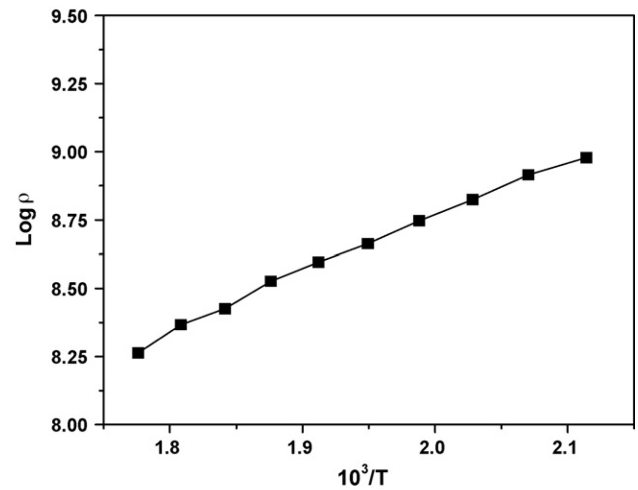

Fig. 3. Log ρ vs. $10^3/T$ for $MgFe_2O_4$ sample.

calculated by using Cottrell's method is about 1 μm. It suggests formation of grains by aggregation of small crystallites of 40 nm size.

3.2. Electrical properties

The variation of DC resistivity with temperature (RT to 500 °C) was measured by the two-probe method. The graph of log ρ vs. $10^3/T$ (Fig. 3.) shows that resistivity increases with rise in temperature. It indicates semiconducting nature of the spinel ferrite.

3.3. Gas sensing properties

The gas sensing mechanism is based on conductance of the ferrites. The oxygen adsorbed on the surface of the material influences conductance of the magnesium ferrite. The amount of oxygen on the surface of the material depends on the particle size, specific area and operating temperature of the sensor. The interaction between the sensing material and gas to be detected determines the gas response. The interaction includes physical and chemical adsorption. The chemical interaction typically involves exchange between adsorbed gas molecule and metal oxide semiconductor, resulting change in the band bonding near interfaces and hence the electric property of the sensing element [14].

The magnesium ferrite interacts with the oxygen, by transferring electrons from the conduction band to adsorbed oxygen atoms, resulting in the formation of ionic species such as O_2^- or O^-. The oxygen adsorbed on the surface of oxide gas sensor undergoes following reaction [15].

$$O_2(\text{gas}) \Leftrightarrow O_2(\text{ads}) \quad (2)$$

$$O_2(\text{ads}) + e^- \Leftrightarrow O_2^-(\text{ads}) \quad (3)$$

$$O_2(\text{ads}) + e^- \Leftrightarrow 2O^-(\text{ads}) \quad (4)$$

The electrons transfer from the conduction band to the chemisorbed oxygen resulting into the decrease in electron concentration. As a consequence, an increase in the resistance is observed [16].

The sensitivities of the gas sensors based on $MgFe_2O_4$ for different gases/vapors such as ethanol, methanol, petrol, LPG, ammonia and hydrogen are shown as a function of operating temperatures (Fig. 4). The sensitivities of ferrite to different gases greatly depend upon operating temperature. In case of magnesium ferrite the sensitivity increases with increasing operating temperature, and goes to maximum at 250 °C and then again decreases with

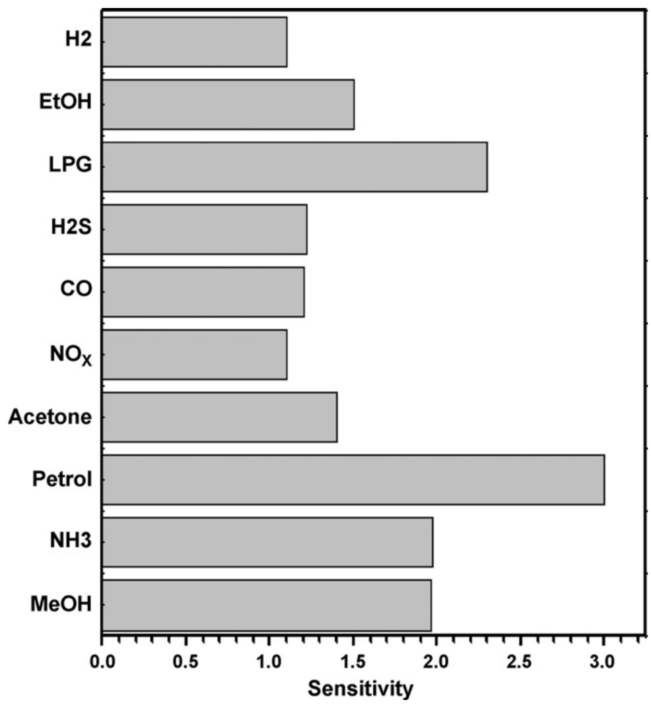

Fig. 4. Sensitivity of $MgFe_2O_4$ towards different test gases at 250 °C.

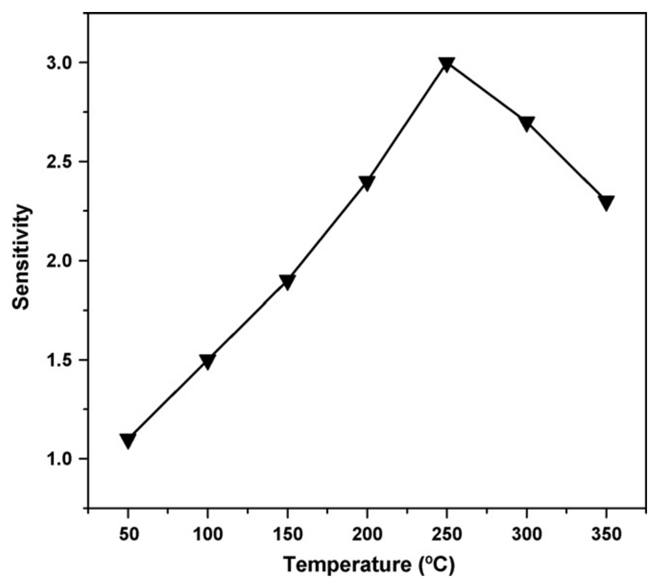

Fig. 5. Sensitivity of $MgFe_2O_4$ for different temperatures to petrol vapors.

increase in temperature. The sensitivity towards ethanol, CO, H_2, NO_x, methanol, LPG, ammonia and hydrogen are much lower than petrol. Fig. 5 indicates that quantity of adsorbed gas increases with increase in the operating temperature, because the gas sensing mechanism depends on the working temperature [17]. The response towards the gas depends on the concentration of the test gas. The graph of response vs. concentration of petrol is nearly linear as seen in Fig. 6. In this work different concentration of petrol was tested for different working temperatures. The $MgFe_2O_4$ shows high sensitivity towards higher concentration of petrol than the lower concentration. It is found that; magnesium ferrite is much more sensitive towards petrol, LPG, methanol, ethanol and ammonia gas and less sensitive towards H_2, CO and NO_x.

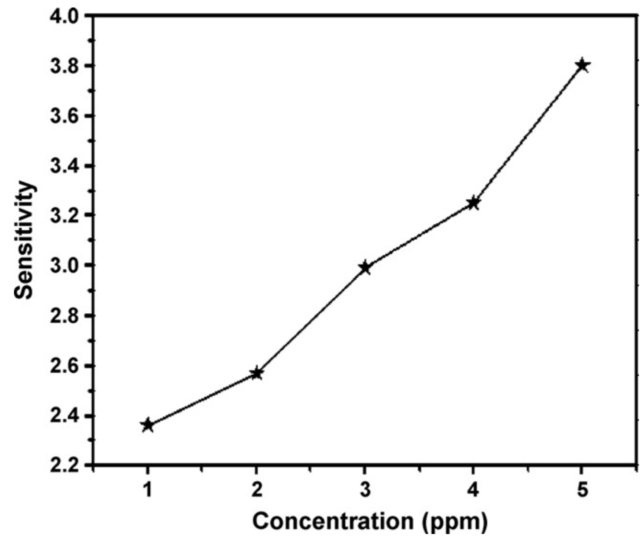

Fig. 6. Sensitivity of $MgFe_2O_4$ for different concentrations of petrol vapors.

4. Conclusions

The results discussed show that it may be possible to use ferrites materials efficiently to detect various types of gases such as petrol, LPG, ethanol, methanol, NH_3, H_2. The sensors based on this material exhibit a marked response towards petrol at temperature of 250 °C. The operating temperature and concentrations of gases significantly affect the sensitivity of the synthesized magnesium ferrite powder towards the petrol vapors. Thus the resulting ferrite have potential as a gas sensor for domestic and industrial purpose.

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