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Acetone and alcohol sensitivity of nanocrystalline nickel ferrite synthesized by sol-gel combustion technique

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ABSTRACT

Nanocrystalline ($NiFe_2O_4$) was synthesized by sol-gel self-combustion technique. X-ray diffraction (XRD) was utilized for structural characterization. Broad diffraction peaks indicating smaller particle size was evident form x-ray pattern. Particle size estimated using Transmission electron microscopy (TEM) was in the range 15-25 nm. Sensitivity was found to increase with temperature before being maximum at a particular operating temperature. Significantly high sensitivity of ~88% was observed in presence of 200 ppm acetone at an operating temperature of 350°C. Maximum sensitivity of ~76% was observed for 200 ppm alcohol at an operating temperature of 300°C.

KEYWORDS

Nickel ferrite;
XRD;
TEM;
Acetone sensitivity.

INTRODUCTION

Conventional semiconducting metals oxides (particularly tin oxide and zinc oxide) have been well studied as sensor material to detect most of the reducing gases. Although incorporation of metal ions (primarily noble metal ions) impart high sensitivity to these materials, most of these traditional ceramic oxides have poor sensitivity at low gas concentration (< 200 ppm), requires high operating temperatures (>300°C) and lack long-term stability apart from the major problem of selectivity[1]. In recent years the ferrites have demonstrated to be good materials for gas sensing applications[2-4]. In the present study, nickel ferrite investigated as gas sensor for acetone and alcohol vapor. The spinel structure, with two differently sized cations, is amenable to a variety of dopant additions. This flexibility allows for control of the structural, transport and catalytic properties, which are important for improving sensor performance. Apart from easy amenability to variety of dopants, another particular advantage of these materials is their high stability compared to conventional semiconducting oxides. Among the ferrites, nickel ferrite with inverse spinel structure seems to be a promising candidate for volatile organic compounds[4]. Several research reports[5,6] have also confirmed the beneficial effect of nanostructure on the sensor performance. In this work we have synthesized nanocrystalline nickel ferrite powder by sol-gel self-combustion technique. The synthesized materials have been characterized using XRD, SEM, TEM etc. Gas sensing characteristics in presence of acetone and alcohol has been carried out.

EXPERIMENTAL

Nickel ferrite was synthesized by sol-gel self-com-

bustion technique from analytical pure grade of metal nitrates. The ingredients were weighted according to the desired composition and dissolved in distilled water. An aqueous solution of 5% polyvinyl alcohol was used to make a colloidal solution. A small amount of ammonia solution (25% concentration) was added drop wise to adjust the pH value to about 8. By coprecipitation, a sol of metal hydroxides and ammonium nitrate occurs Ammonia addition was carried out under continuous stirring in a magnetic stirrer until a viscous gel was obtained. The gel was dried at 150°C for 12 hours. The dried gel so obtained was ignited in a corner on a heated surface and an exothermic reaction takes place at ~225°C between polyvinyl alcohol and ammonium nitrate. A combustion front spontaneously propagates and all the gel is burnt out. After the completion of the exothermic reaction a loose powder is obtained containing very fine crystallites. During the combustion reaction, the metal hydroxides are converted into metal oxides. The fusion between oxides forms the spinel ferrite. After quick combustion, the finely dispersed powder is calcined at 600°C in air, for one hour, to eliminate the residual organic compounds.

The phase identification and crystalline properties of the films were studied by the X-ray diffraction (XRD) method employing a Philips PW 1830 x-ray diffractometer with CuK_α radiation (λ=1.5418 Å). The experimental peak positions were compared with the standard JCPDS files and the Miller indices were indexed to the peaks. Scanning electron microscopy (SEM, Model S530, Hitachi, Japan) was used to illustrate the formation of crystallites on the film surface. Transmission electron microscopy (TEM) was used for particle size analysis.

For gas sensing measurements, the powder (0.15 gm.) was pelletized in a cold press pellet under a pressure of 4 ton for 5 minutes. Silver (AG) paste was used for electroding on both sides of the sample. Copper wires were used for measurement of resistance using a Keithley 6514 DMM. The gas sensing measurements were performed in closed tube furnace. Calibrated Spancan (USA) cylinders of 200 ppm acetone and alcohol with regulated flow meters (1 LPM) was used carrying out the experiment.

RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction pattern

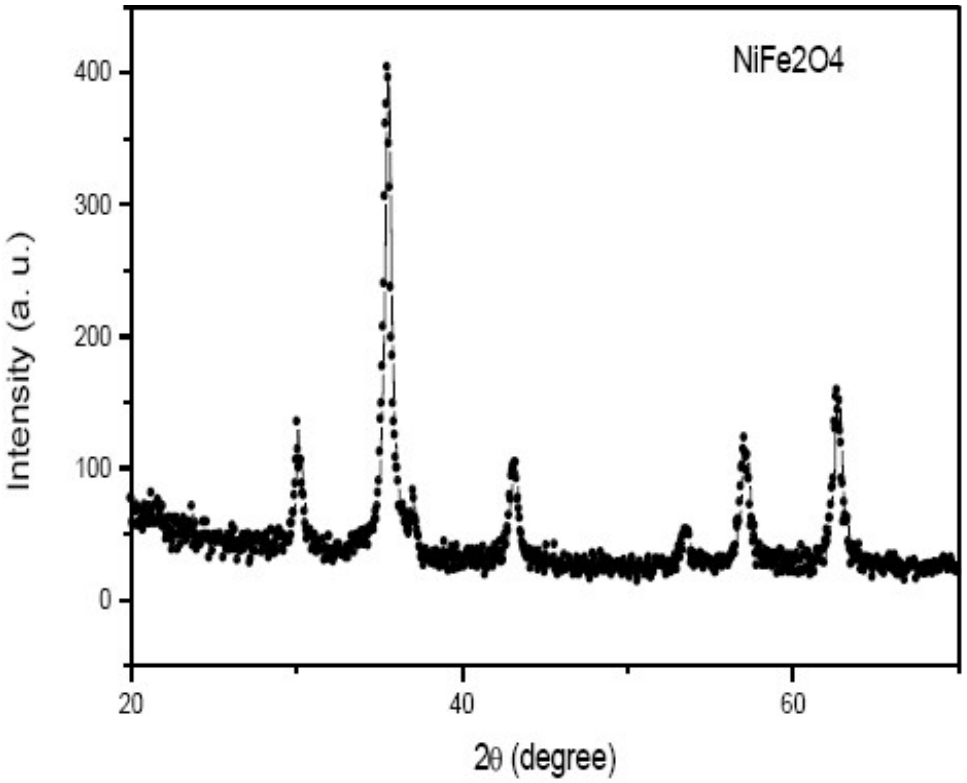

Figure 1 : XRD pattern of $NiFe_2O_4$

of $NiFe_2O_4$. The diffraction peaks observed at 30.05°, 35.5°, 37.05°, 43.25°, 53.60°, 57.05° and 62.65° respectively and can be associated with nickel ferrite structure[7,8]. The material is phase pure without presence of any secondary phase. Broad diffraction peaks in the x-ray diffraction pattern indicates low particle size of the synthesized powder material.

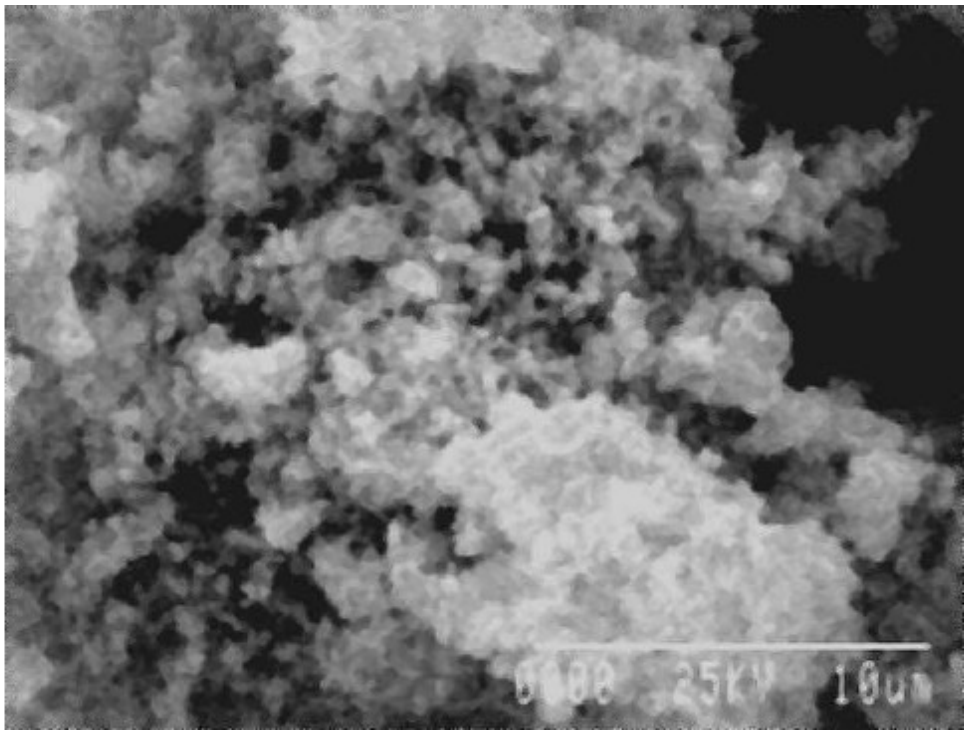

Figure 2 : SEM image of $NiFe_2O_4$

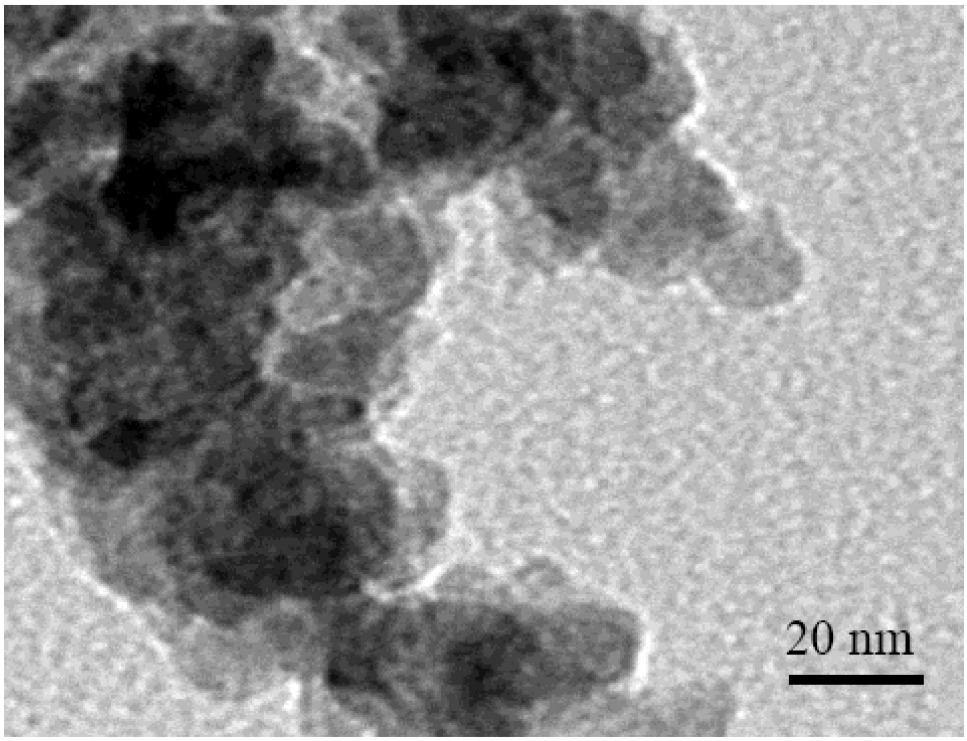

Figure 3 : TEM image of $NiFe_2O_4$

Figure 2 shows the SEM micrograph of nickel ferrite. The image was taken at normal incidence with magnification $\times$5000. One can notice the presence of irregular-shaped aggregates formed by aggregation of very fine particles. The Tem image is shown in Figure 3. Particle sizes ranging between 15 to 25 nm was observed in the TEM micrograph.

Figure 4 shows the plot of resistance against time

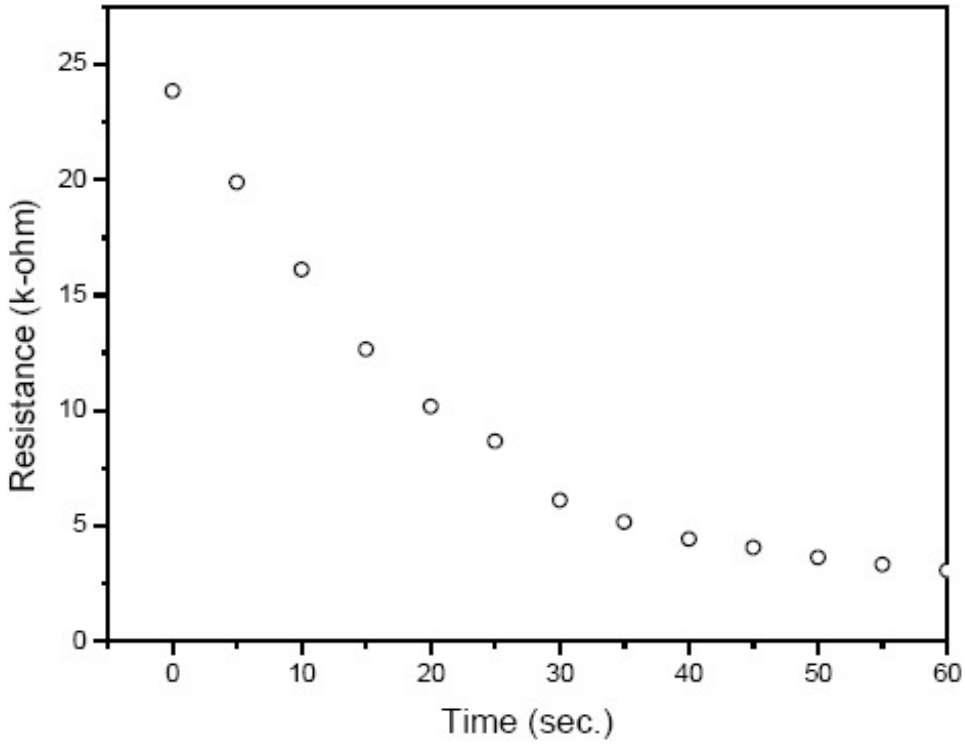

Figure 4 : Sensing for 200 ppm acetone

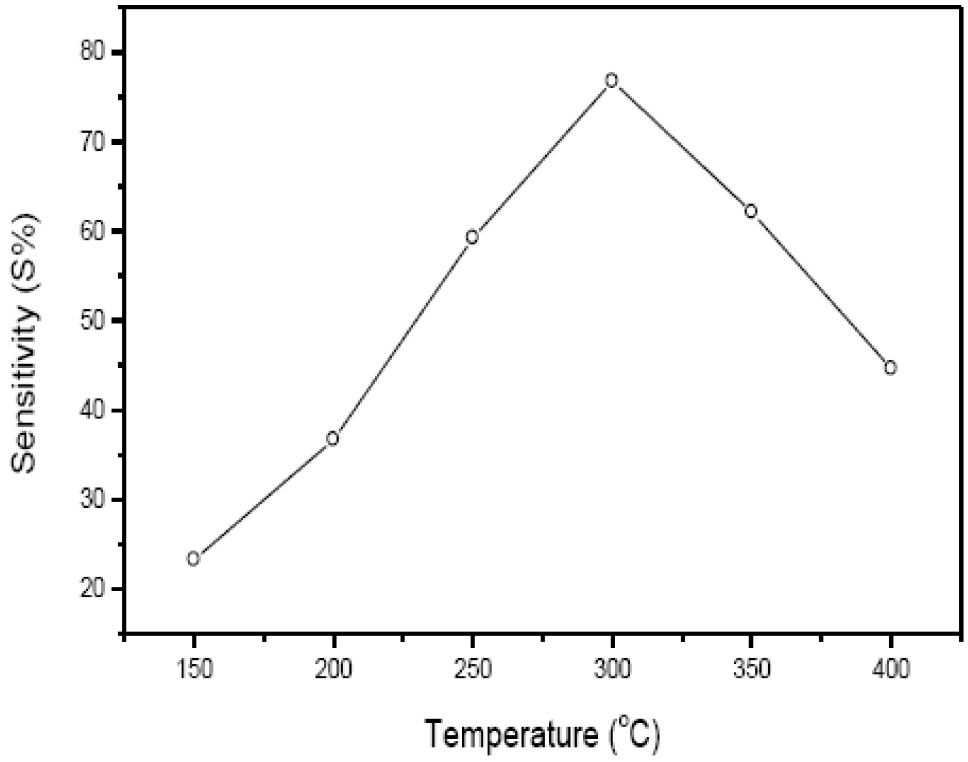

Figure 5 : S% vs. T for 200 ppm alcohol

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(in seconds) for 200 ppm acetone gas at an operating temperature of 350°C. The resistance initially falls sharply and decreases slowly and almost stabilizes after one minute. If R_{air} and R_{gas} represents the equilibrium sample resistance in ambient air and under test gas respectively, the percent sensitivity (or the percent reduction of sensor resistance in presence of test gas) can be expressed as[9,10]

$$S\% = \frac{R_{air} - R_{gas}}{R_{air}} \times 100$$

In the present experiment, R_{air} was ~23.85 M-ohm and R_{gas} was ~3.05 M-ohm giving sensitivity of ~88%. The sensitivity was found to increase with increasing operating temperature and reaches a maximum value around 350°C. With further increase in temperature above 350°C, sensitivity was found to decrease. Usually, the gas sensing mechanism depends on the work temperature, because this mechanism isothermally activated[4]. Figure 5 shows the variation of percent sensitivity against temperature for 200 ppm alcohol in air. Maximum sensitivity of ~76% was observed at 300°C. It is evident from our present experiment that the sensitivity of the ferrite to acetone is better than that to alcohol. Iftime et. al.[4] also showed that among acetone, alcohol, LPG and methane, nickel ferrite shows best sensitivity to acetone.

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