Gas sensing performance of nanostructured NiO thin films

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Abstract:

A simple and inexpensive spray pyrolysis technique was employed to deposit nanostructured NiO thin films from nickel chloride hexadihydrate solution on to the glass substrates heated at 350 °C. The sensing performance of the films was tested for various hazardous gases. The sensor showed high gas response (S = 678 at 250 °C) on exposure of 10 ppm of NO_2. Its response time was short (~8 s) and recovery was also fast (~12 s). The results are discussed and interpreted.

Keywords: Nanostructured NiO, NO_2 gas sensing, response and recovery time.

1. Introduction

Sensors have attracted a great deal of attention for scientists and engineers in the recent years. Even in future it is expected to grain importance in view of the construction subsystems. Nanostructured nickel oxide also possesses excellent chemical and thermal stability [1-2]. Because of its unique properties, Nickel oxide is employed to make device, applicable in different fields of science and daily life. Nanostructured NiO is widely used as for making gas sensors, light emitting diodes acoustic wave filters, UV photo detectors, field effect transistor, intermolecular p-n junction diodes, schottky diodes, photo diodes, optical modulator wave guides. Nickel oxide (NiO) is an interesting chemically and thermally stable n-type semiconductor with large excitation binding energy, large bang gap energy, and high sensitivity to toxic combustible gases.

Nickel oxide (NiO) is the most exhaustively investigated transition metal oxide. It is a NaCl-type antiferromagnetic oxide semiconductor. It offers promising candidature for many applications such as solar thermal absorber, catalyst for O_2 evolution photoelectrolysis and electrochromic device. Nickel oxide is also a well-studied material as the positive electrode in batteries [3].

In this work, nanostructured NiO thin films with different volume of the solution were prepared by spray pyrolysis technique. Crystal structure and grain sizes were studied from X-ray diffraction (XRD) and Transmission electron microscopy (TEM). These nanostructured NiO thin films were tested for sensing different gases and were observed to be most sensitive to NO_2 gas.

2. Experimental details

Nickel oxide thin films of various thicknesses were deposited by varying spraying time of solution between 10 to 40 minutes. The solution was prepared by dissolving Nickel chloride hexadihydrate ($NiCl_2.6H_2O$) [Merck] in deionized water so as to get desired solution concentration (0.05 M). The spray produced by nozzle was sprayed onto the glass substrates heated at 350^oC. Various parameters such as solution concentration (0.05 M), spray rate (7 ml/ min), nozzle to and fro frequency (14 cycles/ min), nozzle to substrate distance (30 cm), etc. were optimized to obtain good quality films. The films with different volume of the solutions: 5 ml, 10 ml, min, 20 ml and 30 ml were obtained and were referred to as S1, S2, S3 and S4, respectively. As synthesized NiO thin films were annealed in air at 500^oC for 1 hrs.

3. Determination of film thickness

Film thickness was measured by using a gravimetric weight difference (considering the density of the bulk nickel oxide). The films were deposited on clean glass slides whose mass was previously measured. After the deposition the substrate was again weighted, determining the quantity of deposited NiO. Measuring the surface area of the deposited film, taking account of NiO specific weight of the film, thickness was determined using the relation:

$$T = M / A.\rho \quad \text{----------} (1)$$

Where

A is the surface area of the film [cm^2]

M is the quantity of the deposited nickel oxide

ρ is the specific weight of NiO

The measured thickness of the thin film samples S1, S2, S3 and S4 were observed to be 450, 789, 833, 978 nm respectively.

4. Results

4.1 Structural characterization

Fig.1 shows the X-ray diffractogram of nanostructured NiO thin film sample S3. It clear from figure that the films exhibited strong orientation along c-axis (200).The average crystalline size was calculated from Scherer's formula and observed to be 23 nm.

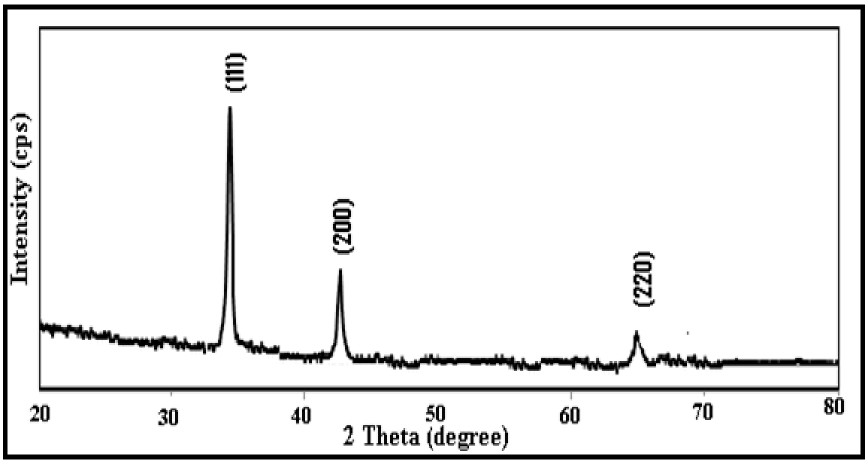


Fig. 1: X-ray diffractogram of most sensitive thin film (Sample =S3)

4.2 Microstructure properties using TEM

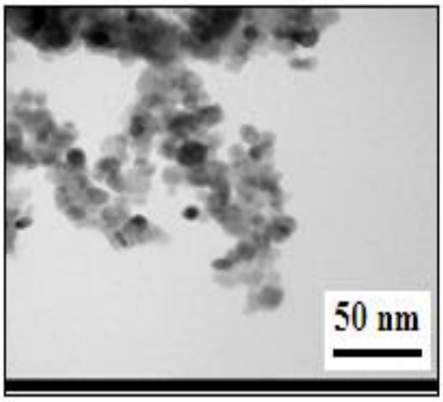


Fig.2. TEM images most sensitive thin film sensor (S3)

Fig. 2 shows the TEM image of most sensitive nanostructured NiO thin film sample S3. The morphology of the grains was spherical in shape. Particle size was observed to be in the range of 10 nm to 16 nm respectively.

5. Sensing Performance of the sensor

5.1 Gas response with operating temperature

Fig.3 shows variation of percentage of gas response with operating temperature of nanostructured NiO thin film samples S1, S2, S3 and S4 on exposure of 10 ppm NO_2. It is clear from Fig.3, that the NO_2 response of sample S3 is higher at 350°C as compared to those of S1, S2, S3 and S4. Due to the greater surface area of nanostructured materials, its interaction with the adsorbed gases is stronger, leading to higher gas response [4].

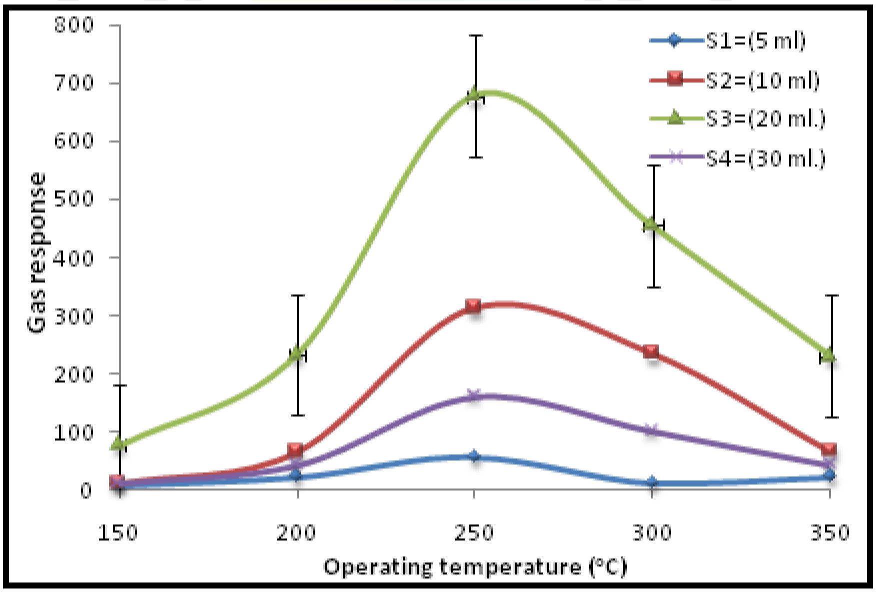
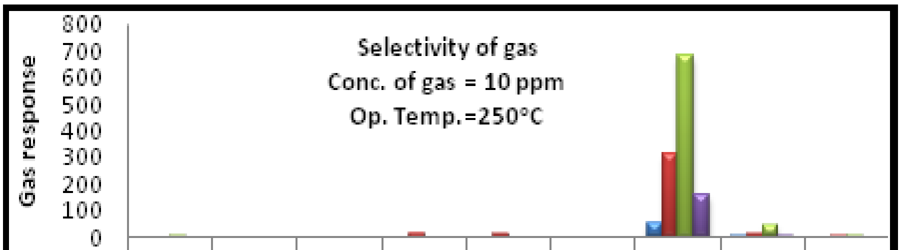


Fig. 3. Gas response of pure nanostructured NiO thin films with operating temperature.

5.2 Selectivity



	LPG	CO2	H2	NH3	C2H5 OH	Cl2	NO2	NO	SO2
S1	0.66	0.23	0.16	0.05	0.07	0.13	56	1.2	0.9
S2	0.44	0.79	0.2	17	12	0.16	312	13	1.3
S3	0.95	0.8	0.36	0.18	0.26	0.27	678	42.3	1.36
S4	0.15	0.5	0.16	0.07	0.3	0.22	160	7.5	0.56

Fig. 4. Selectivity of nanostructured NiO thin films for different gases.

Fig. 4 depicts the bar diagram to indicate NO_2 selective ability of the sensor as compared to other conventional gases.

5.3 Response and recovery of the sensor

The response and recovery of the nanostructured NiO thin film (sample S3) sensor on exposure of 10 ppm of NO_2 at 350^{o}C are represented in Fig. 5. The response is quick (~8 s) and recovery is fast (~12 s).

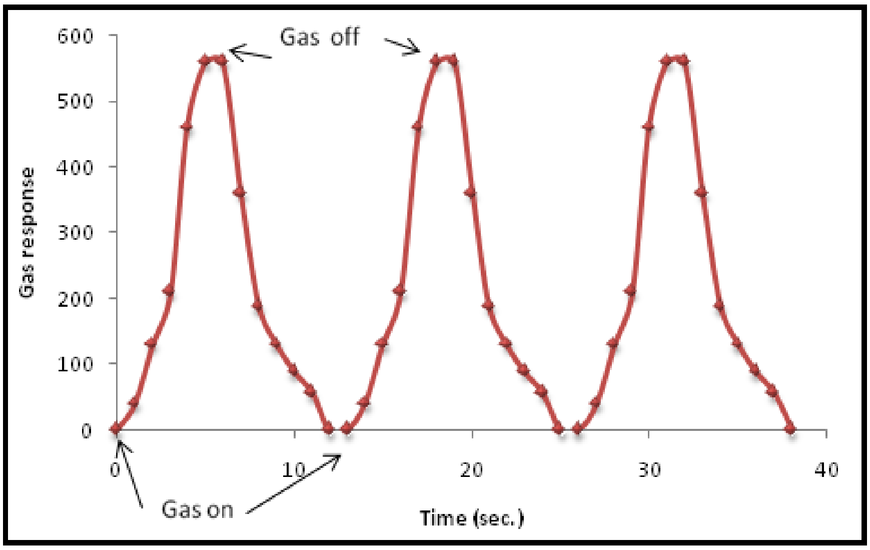


Fig. 5. Response and recovery of the sensor.

6. Conclusions

Nanostructured NiO thin films could be prepared by simple and inexpensive spray pyrolysis technique. Nanostructured grains are found to be important for obtaining enhanced response characteristics. NiO thin film thin film based sensor structure have been designed for the trace level (10 ppm) detection of NO_2 gas at operating temperatures (<250 ^{o}C) and exhibit the response of S= 678.The nanostructured NiO thin films exhibit rapid response–recovery which is one of the main features of this sensor

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