

New Solid State Sensor for Gas Detection Based on Carbon Nano-Tube Fortified by Fe₂O₃-ZnO

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In this paper multi-wall carbon nano-tubes were prepared by using chemical-thermal vapour deposition method and utilizing molybdenum and iron catalysts and silica alumina-base. Methane was passed over catalyst in optimum condition including passing speed equal to 4 L/min and pressure of 1.27 bar for 2-25 min and temperature of furnace was equal to 980 °C. Diameter and specific area of nano-tubes were 10-70 nm and 400-560 m²/g respectively. Various quantities of Fe₂O₃-ZnO and 0.1 % palladium were inserted in carbon used for prepared nano-tubes by utilizing vacuum evaporation which its optimum quantity was [2.50 % (1/1 w/w) Fe₂O₃-ZnO] and 0.1 % palladium in order to enhance sensitivity of carbon nano-tubes relative to NH₃ to make sensors appropriate for low temperatures. For investigation instruments and determining physical properties, XRD, SEM and TEM equipments have been applied and prepared. NH₃ sensor is simple, low cost and ready to use in various environment.

Key Words: NH₃ gas sensor, Carbon nanotube, Fe₂O₃-ZnO.

INTRODUCTION

By discovering carbon nanotubes (CNT), a nonstop investigation was initiated on chemical and physical properties of carbon nanotubes. However, based on vibration and electronic characteristics on hybrid allotropy (pure sp^2 and sp^3) basis of graphite and diamond was carried out. Nevertheless, issues relevant to strong and special vibration of Raman have been studied. In addition to Raman spectroscopy, several microscopic efforts may be beneficial for determining selective properties of carbon nanotubes including transmission electron microscopy (TEM), scanning electron microscopy (SEM), which are applied for determining carbon morphology of multi wall carbon nanotubes¹⁻⁵. In this paper, we used diffuguen method for penetrating Fe₂O₃-ZnO and 0.1 % palladium in carbon nanotubes.

Many semiconductor oxides like ZnO, Fe_2O_3 , SnO_2 , Ga_2O_3 , *etc*.⁶⁻¹⁵ have been known to detect the polluting, toxic and inflammable gases. The gas sensing characteristics of the materials can be improved by surface functionalization or incorporating few additives^{16,17} into the toxic films. Ferric oxide have already reported¹⁷⁻²⁰ by many researchers as a gas sensing material. ZnO and Fe₂O₃ are the *n*-type semiconductors²¹. Fe₂O₃ occure as hematite.

As ammonia is utilized extensively in many chemical industries, fertilizer factories, refrigeration systems, *etc.* a lake in the system can result the health hazards. Ammonia is harmful and toxic in nature. The exposure of ammonia causes chronic lung disease, irritating and even burning the respiratory track, *etc.* It is therefore, needed to monitor ammonia gas and to develop the ammonia gas sensor. Among various additives tested, Fe₂O₃ is an outstanding promoter in enhancing the catalytic activity and gas sensing properties of ZnO for NH₃ detection.

Pure Fe_2O_3 has poor electrical conductivity and gas sensitivity. Catalysts like Pt, Pd, Ag, Ru and Cu are often added to the base material to improve the gas response and selectivity.

EXPERIMENTAL

After growing multi-walled carbon nanotubes with chemical vapour deposition (CVD), we took (SEM) and found diameter of tubes are about 10-70 nm, then we examine Raman spectroscopy and compared it with other references and found that it have 3-4 peaks and compared it with graphite Raman spectroscopy and found that graphite has only one peak in 1582 cm⁻¹ but multi walled carbon nanotubes have two peaks in this region the first one corresponds to D mode and second corresponds to P mode completely conformed with multiwalled carbon nanotubes (MWNT) and then we made one cylinder bulk of multiwalled carbon nanotubes.

All the common chemicals were on analytically grade and were commercially available. The water used in all experiments was double distilled with quartz heating tube. A model Cambridge stereo scan 360 scanning electron microscopy (SEM) of nanotubes. A model Hall effect Oxford Instrumentsaugust 1992, A model Raman spectroscopy Almega Dispersive Raman manufactory Thermo Nicollet Made in USA, was used.

Chemical vapour deposition method involves thermal decomposition of hydrocarbons (usually CH_4) at temperatures ranging form 500 to 1000 °C in the presence of a catalyst containing transition metals such as Fe and Mo. This process is more energy efficient that the electric arc-discharge and laser ablation methods, is ideal to generate well-defined structure of nanotubes. The yield and structure of nanotubes are affected by the type, purity and porosity of the catalyst. It has been shown that methane chemical vapour deposition process can be used to obtain *ca*. 200 % yield (2 g of MWNT s/g catalyst) of high quality carbon nanotubes. The methane chemical vapour deposition process shows promises for long-scale production of defect free carbon nanotubes.

Carbon nanotubes produced in a typical process are closed ended and are usually associated with other carbonaceous species such as nanoparticles, fullerenes and catalyst.

After preparing single walled carbon nanotubes in order to mounting Fe_2O_3 -ZnO and 0.1 % palladium and increasing sensibility of carbon nanotubes relative to ammonia gas in low temperatures, ferric chloride, palladium cloride and ZnCl₂ were applied.

Ferric chloride and $ZnCl_2$ was selected in various quantity and mounted by certain amounts of carbon of multi wall nanotubes by using vacuum evaporation and then were investigated by utilizing metallurgic optical microscope and SEM and optimum quantity of ferric chloride and $ZnCl_2$ required to be mounted on carbon nanotubes found to be (2.50 % 1/1 w/w and 0.1 % palladium).

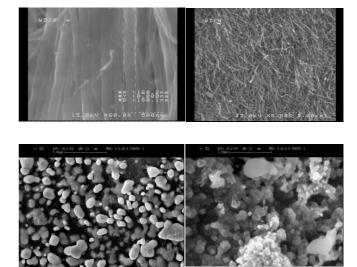
RESULTS AND DISCUSSION

Multi-walled carbon nanotubes prepared from chemical vapour deposition and used from them for review of sensitivity properties in SEM spectrum observed and clearly have seen exist of multi walled carbon nanotubes under 50 nm, since SEM device in researchment is in limit of 50 nm then we can not used from it for determine of correct dimension and done reviews. Transmission electron microscopy spectrum sample of multi walled carbon nanotubes and anything reason be more observed that carbon nanotubes have provided in about10 nm for next studies and reviews²².

We have seen obviously on different sample by TEM that seed scales of catalyst have essential role in status and grow of single-walled carbon nanotubes. Anything seed scales of catalyst from Gel-cell way are smaller and about nanometer, provided multi -walled carbon nanotubes samples will be desirable by scale. In image clearly scale of multi-walled carbon nanotubes is about 10 nm.

Raman spectrum of carbon nanotubes review to determine curves of type D that first state is related to multi-walled carbon nanotubes and next curve is marker of acidic corrosion in surface of carbon nanotubes²².

The scanning electron microscopy (SEM) photograph of the sensor materials sintered at 980 °C, indicated that the prosity and grain size of carbon nanotubes significant increases (SEM) photograph revealed (Fig. 1) qualitative the carbon nanotubes with Fe₂O₃-ZnO has greater and larger number of pores. By taking image from different parts of the s and 0.1 % palladium surface and driving the average size of carbon nanotubes with Fe₂O₃-ZnO particles in each image by foregoing software and average size particles in each sample was calculated and the mean particle size is about 10 nm. The particle are packed closely and well distributed on the disk. The size and morphology of the particles were characterized by TEM as shown in (Fig. 2) the uniformly of nano powder was confirmed by TEM observation.



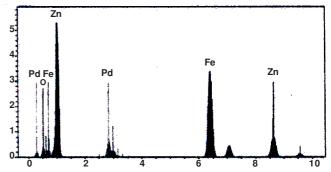


Fig.1. SEM Photograph before and after adding Fe₂O₃-ZnO and palladium

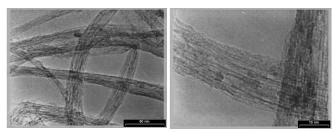


Fig. 2. TEM photograph of carbon nanotyube with Fe_2O_3 -ZnO and palladium

The evaluation of the response and recovering characteristics has been carried out. The DC resistance in ammonia gas alternatively helped to establish the response and recovering characteristics. The result (Fig. 3) shows that the invariant resistance in ammonia gas with time (s). Response and recovering times are 20 (s) and 45 (s) respectively.

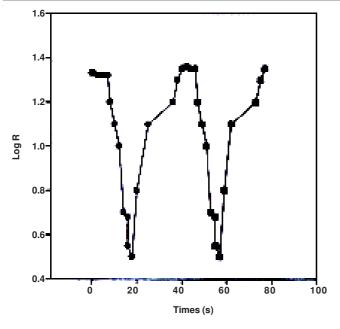


Fig. 3. Invariant resistance in 20000 ppm of ammonia

Conclusion

In this paper, a summary of multi walled carbon nanotubes provided and prepared by chemical vapour deposition method in area of 10-70 nm based on Sol-Gel catalyst. Device studies show desirability of sintered materials and ability applications, In review of provided XRD spectra, study of Hall effect phenomena and conductivity of provided multi-walled carbon nanotubes that can have very well of repeatability, high-level purity of material and development and bring ability of application in most of the industries. Carbon nanotubes were activated by Fe₂O₃-ZnO and 0.1 % palladium through utilizing diffusion method. Studying and investing carbon particles and sensor sensibility shows that decreasing grain size of carbon in nanotubes and Fe₂O₃-ZnO and 0.1 % palladium amounts leads to increase grain size of particles and sensibility of sensors²³⁻²⁵.

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