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Analysis on the effect of ZnO on Carbon nanotube by spray pyrolysis method

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Abstract

Background: ZnO/CNT nanocomposites were prepared using Zinc acetate source materials and with the assistance of copper plate, glycine and sugar solution. The combined behavior between these two materials may give rise to the production of advanced materials with a wide range of applications in electronics and optoelectronics.

Methods: The ZnO-CNT nanostructures are successfully prepared by simple perfume spray pyrolysis method on copper substrate. The possible growth mechanism of ZnO-CNT nanocrystals formation by this method has been tried to explore the sensor and optical properties has been demonstrated.

Results: The as-synthesized ZnO-CNT nanostructures were characterized using the scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray diffraction (XRD) pattern measured with Cu Kα radiation. Studies of the morphologies of the ZnO-coated CNTs revealed no significant change in the internal structures single walled graphite sheets and the diameters of the CNTs, but the ZnO appeared to form a layer of thinfilm single crystalline particles attaching to the surface of the nanotubes. The photoluminescence (PL) measurements excited by the 380 nm were done at room temperature. CNTs are easy to be entangled and agglomerate due to their long length and low diffusive mobility in base fluids.

Conclusion: The lower mobility was found to occur for the ZnO/CNT composite where a linear sensitivity behavior was measured and it reaches high at the temperature of 200 °C. The samples luminescence is dominated by well-structured ultraviolet band emission and almost no deep level emission was observed, revealing a high optical quality of the produced structures.

Keywords: ZnO-CNT, Photoluminescence studies, Morphological studies and sensor studies

Background

The carbon nanotubes (CNTs) have also strained much attention since their unique fundamental physical structures, eminent mechanical and electronic properties which leading to potential high-technology applications (Odaci et al. 2008; Wei et al. 2008; Iyakutti et al. 2009). The ZnO is one of the most important functional metal oxides for their versatile practical applications, ranging from photodetector (Jun et al. 2009), transparent electrode (Oh et al. 2005), spintronic devices (Gupta et al. 2008), surface acoustic wave devices (Krishnamoorthy and Iliadis 2008), and thin film gas sensors(Tang et al. 2006), attributed to their outstanding properties such as wide direct optical band gap, large exciton binding energy, excellent chemical and thermal stability, and excellent piezoelectric properties(Gupta et al. 2009). When the ZnO metal oxide is combined with CNT, it is marvelous that, the novel extraordinary properties of ZnO-CNT composite is appear.

In recent years, nano structured materials such as ZnO-CNT nano composites have also been incorporated into electrochemical sensors for biological and pharmaceutical analyses (Suchea et al. 2006). While they have many properties similar to other types of materials, they offer unique advantages including enhanced electron transfer, large edge plane/basal plane ratios and rapid kinetics of the electrode processes (Banks et al. 2006). Nanocomposites of a variety of shapes, sizes and compositions are changing modern bioanalytical measurement (Moradi et al. 2013).

Ching-Feng Li, Chia-Yen Hsu, Yuan-Yao Li et al. reported that, an 80 nm-thick ZnO film was prepared via the sol–gel method at 500 °C using zinc acetate, 2-methoxyethanol, and mono ethanolamine as precursors. Characterization of the film showed that it was composed of 20–30 nm sintered

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ZnO nanoparticles with good crystallinity. The NH_3 sensing properties of gas-sensing devices with a 5 μm gap that utilized the prepared ZnO film were examined. The highest sensor response (57.5 %) was achieved with 600 ppm NH_3 in air at 150 °C. The response and recovery times were 160 s and 660 s, respectively. This study also examined the effects of NH_3 and oxygen concentration as well as the temperature on the sensor response performance. The findings show that oxygen plays an important role in the conductivity of ZnO thin films, and thus affects the sensor response toward NH_3 (Lia et al. 2014).

Herran, I. Fernandez, E. Ochoteco, G. Cabanero, H. Grande et al. reported that, the role of water vapour in ZnO nanostructures for humidity sensing at room temperature is presented and discussed. Experimental and theoretical results demonstrate that ZnO nanoparticles and nanorods, show different physico-chemical behaviour under different relative humidity atmospheres. While electrical current density increases as R_H does in the case of the ZnO nanoparticles, ZnO nanorods show inverse behaviour. These facts are related to the capillary condensation and water electric dipole moment effects, respectively. Additionally, a simultaneous validation between the sensor developed and a commercial device corroborates the potential application of this kind of low-cost sensing nanostructures presented in this work (Herran et al. 2014).

Ganesh Kumar Mani et al. (Ganesh Kumar and John Bosco Balaguru 2014) reported that, randomly interconnected zinc oxide (ZnO) nanoplatelets were successfully deposited on glass substrates using simple chemical spray pyrolysis technique. X-ray diffraction (XRD) pattern confirmed that the nanoplatelets were highly polycrystalline in nature with hexagonal wurtzite structure. Field emission scanning electron microscope (FE-SEM) image revealed the formation of randomly interconnected nanoplatelets with no visible defects on their surface. The thickness of the nanoplatelets was found to be in the range of 110–130 nm. The optical absorbance spectra showed no sharp absorption edge and the optical band gap was found to be 3.23 eV. Acetaldehyde sensing characteristics of ZnO nano platelet sat room temperature were investigated. The selectivity of ZnO nanoplatelets towards acetaldehyde was found to be significant in comparison with the other gases like ethanol, methanol, ammonia, acetone, formaldehyde and toluene.

Mohsen Asad, Mohammad Hossein Sheikhi reported that a surface acoustic wave (SAW) based H_2S gas sensor with excellent selectivity and recovery/response time developed using single wall carbon nanotube decorated with copper nanoparticles (Cu NP-SWCNT). A thin film of Cu NP-SWCNT was deposited onto a lithium niobate ($LiNbO_3$) piezoelectric substrate via a drop-casting technique. Sensing of H_2S gas was carried out by measuring acousto electric perturbation of SAWs traveling along the $LiNbO_3$ piezoelectric substrate and Cu NP-SWCNT sensing layer. H_2S gas of concentrations as small as 5 ppm could be readily measured. The effect of temperature on the SAW sensor response was also investigated for a range of temperatures from 70 to 200 °C. The optimum operating temperature was 175 °C, in which, a relatively rapid response (7 s) and recovery time (9 s) was recorded. The selectivity of the proposed Cu NP-SWCNT gas sensor was examined by assessing the sensor response upon exposure to hydrogen, acetone, ethanol, and H_2S gas species in air background and a large selectivity toward H_2S gas was observed (Mohsen and Mohammad Hossein 2014).

In this research work, the ZnO-CNT composites are synthesized from spray pyrolysis method and their fundamental physical and optical properties have been investigated by X-ray diffraction (XRD), SEM, TEM, EDAX, PL spectra and FT-Raman analytical tools.

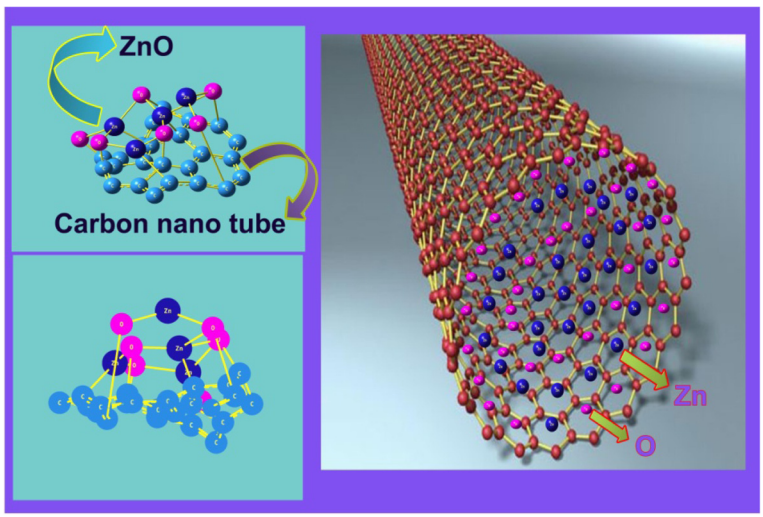

Fig. 1 Molecular strutural analysis of ZnO-CNT asprepared sample

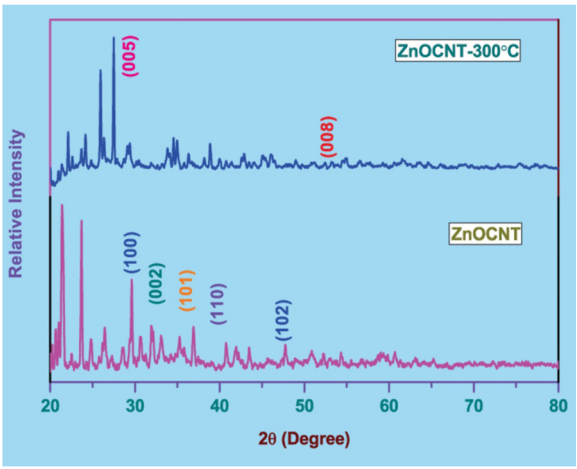

Fig. 2 XRD analysis of ZnO-CNT asprepared and annealed sample at 300 °C

Methods

In the static position of this spray nozzle, the substrate size may be 1 cm × 1 cm for coating. But here we used 2.5 cm × 2.5 cm copper substrate for spraying. So a slight vertical and horizontal movement required for constant spraying. The droplets (mist) hit the copper substrate, where the solvent is entirely vaporized leading to the deposition of a rough film in which the transmission decreases markedly. At the optimum air flow rate the size of the mist particle is also optimum. So the thermal energy gained by the droplet is in such a way that it vaporizes just above the copper substrate and gives a good quality of powdered particles on the surface. They form a powdery precipitate on the substrate resulting in the decrease in transparency in the present work it has been observed that which gives highly transparent, good powdered particles by spraying (Shanti et al. 1999).

A specially designed glass tube is used as a carrier tube for the chemical mist generated by the perfume sprayer. The length of the tube is about 25 cm horizontal and 15 cm vertical length with diameter of 14 mm and the bottom of the vertical tube act as a spray nozzle with a diameter of 7 mm. In the above tube the spray nozzle glass walls cross section should be pure flat structure. i.e. the 7 mm nozzle outlet must be as a perfect circle. Then only the spray outlet will be a stream lined. The heat waves from the hot plate may affect the mist coming through vertical tube.

0.5 Molar solution of Zinc acetate dehydrate and 0.5 Molar solution of Glycene was dissolved in a 25 ml of distilled water and stirred for half an hour by using magnetic stirrer. The solution was well mixed with stirred then 5 ml of diethanolamine was added in it. This solution is filled with perfume sprayer of tube and sprayed continuously in equal time intervals on the hot plate. For the Zinc nanoparticles Zinc acetate dehydrate and diethanolamine was the solution for CNT Glycene was well stirred with zinc acetate dehydrate and sprayed on the copper plate without diethanolamine.

The crystalline phases of the materials were investigated using X-ray powder diffraction (XRD) PAN analytical X-ray diffractometer using CuK_α radiation (λ =1.5406 nm). The morphological of the materials was observed using a scanning electron microscope (SEM, JEOL JSM 6500-F). A transmission electron microscope (TEM JEOL, JEM – 2010-F) was used to characterize the ZnO, ZnO-CNT and CNT powdered particles. A Raman spectrometer (Raman, Dongwoo, optron, Co) and an electron spectroscopy for chemical analysis (ESCA, VG Scientific Microlab 310 F) were used to investigate the surface chemical composition and characteristics of the samples.

Results and discussion

XRD analysis

Generally, the ZnO existed in both the form of cubic zinc-blende and hexagonal wurtzite structure whereas the CNT is in the form of hexagonal rings as shown in the Fig. 1. The XRD of ZnO-CNT composite is shown in the shown Fig. 2. The XRD pattern of the present compound showed the peaks corresponding to cubic and hexagonal geometrical planes (100), (002), (101, (110) and (102) which are presented in the Fig. 2. In this figure, the peaks of the pattern showed the strong reflections of the interplanes of cubic ZnO and weak reflections of hexagonal CNT lattice structures. From this view, it is confirmed that, the ZnO is captured by the hexagonal C patterns of CNT lattice. It is also observed that, the electrochemical bonds are formed between CNT and ZnO compounds and ZnO is directly coupled with C in the continuous chain of CNT lattice. The Figure shown a large quantity of ZnO seeds is distributed over the CNT. Therefore, the peaks at 44.0°, 51.2° and 75.3° can be observed, which are corresponding to carbon. Moreover, characteristic graphitic peak at 2θ = 26.4° referred to (002) exposes the perfect crystallinity of

Table 1 Structural parameters of ZnO-CNT

Position	FWHM	D Å		Plane Spacing	Diffraction planes	Grain Size (D) (10^{-9}) nm	Dislocation Density (δ) (10^{15}) lines/m^2	Strain (ε) (10^{-3})
		Std	Obs					
31.8994	0.4015	2.8943	2.8055	2.8137	100	21.504	2.1625	1.5635
43.4871	0.3346	1.9111	2.0810	1.9108	110	26.709	1.4017	0.9318
47.8036	0.6528	1.9111	1.9011	1.9108	002	13.910	5.1679	1.6357

the CNT. This result is supported by the earlier work (Yanping et al. 2009).

In CNT, the carbons are coupled directly and form the hexagonal chain. In the chain, the subsequent carbons are making themselves positive and negative and thus electrochemically connected continuously. In the case of ZnO structure, the Zn is positive and O is negative. The Zn is coupled by bond with negatively charged carbon and O is connected by bond with positively charged carbons in the hexagonal frame as in the Fig. 1. In this case, the sample is annealed at 300 °C and the XRD is also taken for the same, in the XRD pattern, the peaks are observed with weak intensity which is not preferred to take in to analysis. XRD signal pattern strongly represent the presence of ZnO and weakly symbolize the CNT.

The CNT, ZnO-CNT and ZnO powdered particles, its full width at half maximum (FWHM) β is used in Scherrer formula,

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

The crystal size (D) and its related parameters are calculated for the compounds without and with annealing temperature of 300 ° C are tabulated in the Tables 1 and 2 respectively. The variation in grain size is less appreciable and it decreases nominally about 21 nm, 26 nm and 13 nm. Whereas the grain size of the ZnO-CNT particles are 51 nm, 22 nm and 16 nm at the annealing temperature 300 ° C. from these two results, it is observed that, the grain size is grown due to the annealing process. In the case of interplanes spacing, the value of the compound with annealing are found be increased considerably. From this result, it can be inferred that, the interplanes are drastically expanded over the CNT surface due to the applied thermal energy. From the table, it is also observed that, there is no change in the dislocation density and strain over the lattice due to the annealing temperature. From this observation, it can be noticed that, though thermal forces applied in the lattice sites, there is no considerable strain found and the structure does not changed.

SEM analysis

The SEM image of the present compound ZnO-CNT composite is displayed in the Fig. 3. Usually the diameter of the CNT s is about 10 nm levels (Yanping et al. 2009). When metal oxides added on a surface its circumference may be changed (Green et al. 2006). The CNT network structures have large surface area and are able to decorate ZnO nano dots over the area. Since the CNT is the chain of continuous domain in which the ZnO nano dots are deposited and thus CNT is converted in to the form of ZnO reinforced CNT. The SEM image showed the clear spherical shape of ZnO over the CNT. The particles are appeared transparently and the CNT does not appear visibly due to the densely packed ZnO grains. After the annealing process, however, the presence of the spherical shaped particles is found to be more uniform and the particles are more consistent in size and shape. From the Figure, it is concluded that, there appears to be minute change in the morphology of the CNT itself and the diameter of the tubes not remains the same and bulged little bit. There are numerous particles appear on the surface of the nanotubes like beads on a necklace which contributes in homogeneity over the surface of carbon nanotube due to ZnO coating.

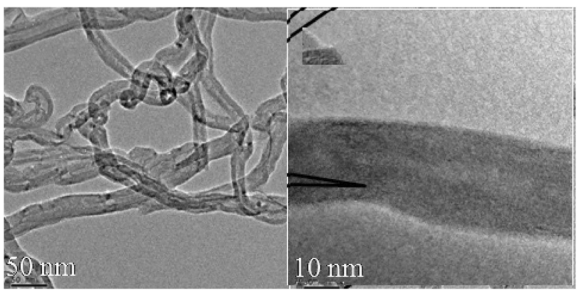

Fig. 3 SEM Analysis of ZnO-CNT annealed at 300 °C

TEM profile

To gain further deep knowledge about the internal structure of the ZnO-CNT, the TEM analysis is used to characterize the material. The TEM image of the present compound is presented in the Fig. 4. The picture of the CNT can be viewed clearly and looks like a long silver chain. The sticking of fine droplets of ZnO over the vicinity of CNT is to be found. The figure shows clumps of the droplet of ZnO nano dots that are formed on the surface of the tube before and after the annealing process. A thick nebulous layer appeared on the surface of the CNT as

Table 2 Structural parameters of ZnO-CNT annealed at 300 °C

Position	FWHM	D A°		Plane spacing	Diffraction lines	Grain Size (nm)	Dislocation density (δ) (10^{15}) lines/m^2	Strain (ε) (10^{-3})
		Std.	Obs.					
27.5053	0.1673	3.348	3.2429	3.348	005	51.085	0.3832	0.7633
42.7882	0.4015	2.11	2.1134	2.1100	-	22.206	2.0279	1.1407
45.1937	0.5333	2.061	2.0064	2.0615	-	16.798	3.5438	1.4346
51.2405	0.6691	1.896	1.7829	1.8961	008	13.759	5.2818	1.5565

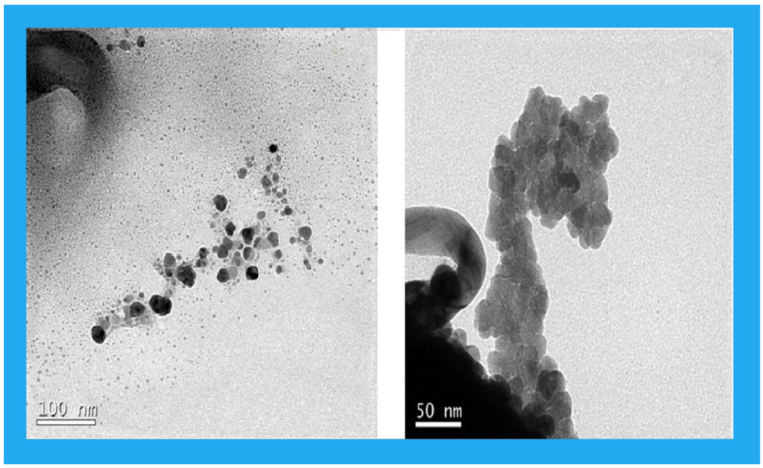

Fig. 4 TEM analysis of ZNO-CNT at 300 °C

well in the Fig. 4. From the figure it is also observed that the ZnO nano dots on the surface slightly deform the shape of CNT. Basically the ZnO has more sensing action, so the CNT coated with ZnO will be sensed well. In this case, the material under study can be used as good sensor.

Conductivity analysis

The conductivity of the material can be detected by drawing the graph between resistivity and temperature. The Fig. 5 shows the resistivity response of the ZnO-CNT against temperature. The graph shows the non linear behavior of the conducting property of the compound. The figure showed the comparative graph of ZnO, CNT and ZnO-CNT materials. The blue line of the graph displays the resistance characteristics of ZnO-CNT. Initially, the resistance is directly proportional to the temperature. It is constant between 200° - 220 °C and after that; the resistance is inversely proportional to the temperature. From the graph, it is found that, the conductivity of the material can be tuned by the controlling of the temperature. Simultaneously, the transconductance of the material also is controlled.

Gas sensing applications

The Fig. 6 shows the Sensitivity variations of CNT, ZnO-CNT and ZnO of samples for methanol concentration of 75 ppm for different temperatures. The graph shows the linear behavior of the sensing property of the compound. The projection of blue line in the graph reflects the sensing efficiency of the present materials. The

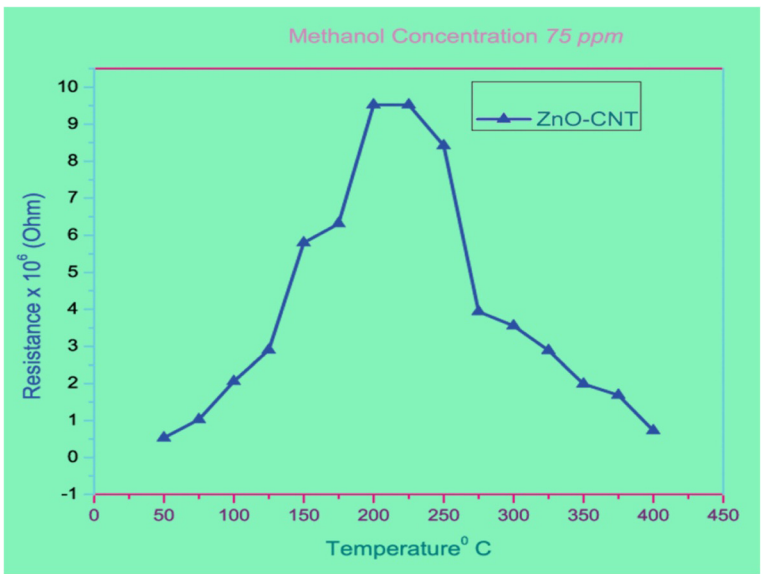

Fig. 5 Conductivity for annealed sample ZnO-CNT at 300 °C

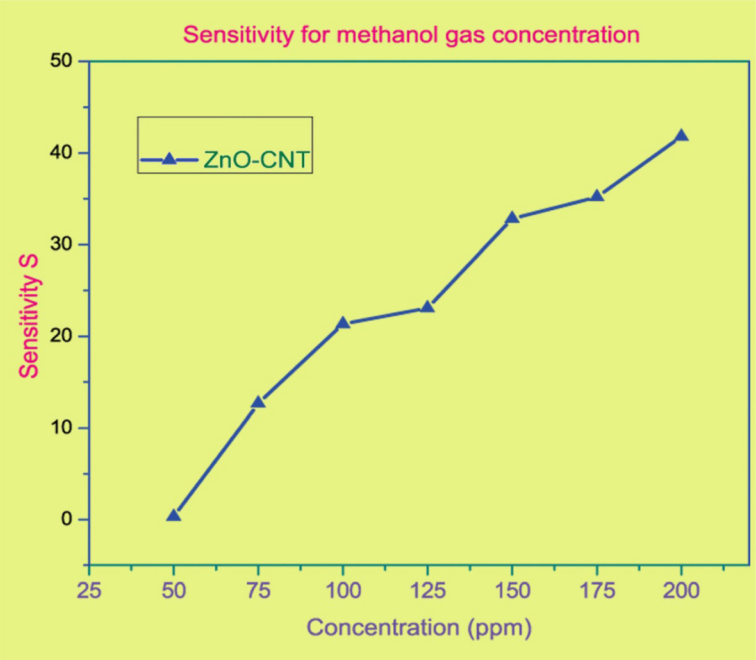

Fig. 6 Sensitivity Analysis of ZnO-CNT annealed at 300 °C

sensitivity of the ZnO-CNT material is measured up to 220 °C and it is observed that, the sensitivity is more at 200 °C. From the analysis, it is concluded that, the sensitivity of the present compound is more than pure ZnO and CNT. The sensitivity of the ZnO is added with sensing ability of the CNT compound and if this material used as sensor, the device efficiency will be more.

PL analysis

Photoluminescence is used for characterization purposes to measure the quantities of semiconducting nanotube species in a sample. The Photoluminescence (PL) optical spectra of ZnO, CNT and ZnO-CNT are obtained at an excitation wavelength of 345 nm is shown in Fig. 7. The interaction between nanotubes or between a nanotube

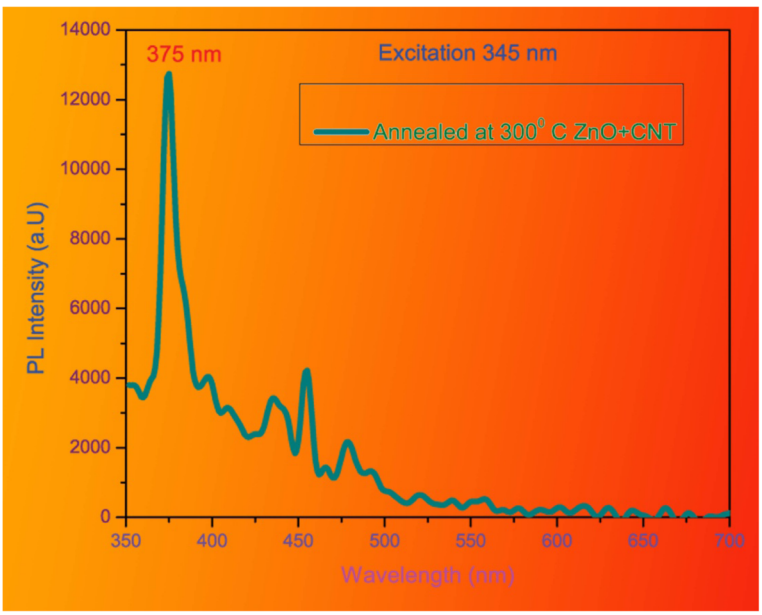

Fig. 7 PL Analysis of ZnO-CNT annealed at 300 °C

and another metal oxide material may quench or increase PL (Satishkumar et al. 2007). Usually, the PL spectrum emission peak in the UV-Visible spectrum is observed at 375 nm for ZnO-CNT (Mahato et al. 2009), in this case, the green-shifts of PL spectra by 0.37 eV with resulting emission peak found at 460 nm. The PL in such carbon systems is a consequence of geminate recombination of localized electron–hole pairs in sp^2 clusters which essentially behave as the luminescent centres.

The PL intensity increases with the increase of sp^2 content in the disordered carbon systems (Eda et al. 2010). This is certainly due to the presence of CNT aggregated ZnO nano dots. The blue shift is expected normally, whereas in this work, due to the presence of ZnO on CNT, the green photoluminescence shift is observed which is also due to the transition in defect states, particularly oxygen vacancies (Luo et al. 2009). In this case, blue shift is significantly quenched. This suggests that an additional pathways for the diminishing the charge carriers dominates because of the interactions between the excited ZnO and CNT. Since the ZnO act as n-type semiconductor due to oxygen vacancies and zinc interstitials (Look et al. 1999) and CNT behaves as a p-type semiconductor, hence, n-p depletion layer is formed at the ZnO: CNT interface. This set up making very narrow band gap and it is a root cause of the application involving opto-electronics.

Raman analysis

The Raman spectroscopy is the most popular technique of carbon nanotube characterization is shown in Fig. 8. The Raman scattering in CNTs is resonant, i.e., only those tubes are probed which have one of the band gaps equal to the exciting laser energy (Souza Filho et al. 2004). Similar to photoluminescence mapping, the energy of the excitation light can be scanned in Raman measurements, thus producing Raman maps (Fantini et al. 2004). The Raman spectra of ZnO-CNT as prepared and annealed at 300 °C are shown in Fig. 8. Usually, the G line around at 1570 cm^{-1} which is assigned to E_2g phonon of C sp^2 atoms and D line around at 1350 cm^{-1} as a breathing mode of k-point phonons of A1g symmetry (Barone et al. 2005) which is assigned to local defects and disorder especially at the edge of graphene oxide (Yanagi et al. 2006) in Raman spectra. In the present Raman spectra, the peaks are observed at 1400 cm^{-1} and 1500 cm^{-1}. From this assignment, it is observed that, the G and D lines are present with some shift in the spectrum. The shift is contributed by the addition of ZnO compound. This is also confirmed that, the presence of CNT and adopted with ZnO which are capable of having semiconducting property.

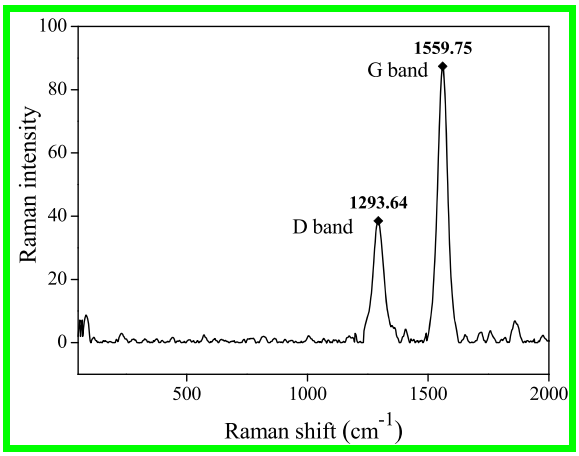

Fig. 8 Raman Analysis of ZnO-CNT annealed at 300 °C

Conclusion

The ZnO-CNT nano material has been prepared by simple perfume spray pyrolysis method on copper substrate. The effect of the structural, morphological, and sensor properties have been studied extensively. The XRD, SEM and TEM images evidenced the formation of crystalline ZnO-CNT with nano grained structure. The SEM examination of the present compound reveals that, there are numerous particles appear on the surface of the nanotubes like beads on a necklace which contributes in homogeneity over the surface of carbon nanotube due to ZnO coating. The Raman analysis confirmed that, the presence of CNT and adopted with ZnO which are capable of having semiconducting property. From the conductivity graph, it is found that, the conductivity of the material can be tuned by the controlling of the temperature. Simultaneously, the trans-conductance of the material also is controlled. From the analysis, it is observed that, the ZnO-CNT is active material for sensing gas.

Competing interests

The authors declare that they have no competing interests.

Authors' contribution

AA (first author) carried out the experimental method and sensor studies, participated in the sequence alignment and drafted the manuscript. Head, Department of Physics, Khadir Mohideen College, Adirampattinam, 614701, affiliated with Bharathidasan University, Thiruchirappalli, 620024, Research and Development Center, Bharathiyar University, Coimbatore, 641046, India. DS (Second author) carried out the experimental section to prepare ZnO-CNT nanoparticles. KS (third author) has been involved in revising it critically for important intellectual content and improving the figures and participated in the sequence alignment of the manuscript. SS (fourth author) participated in the design of the study and performed the calculations. BR (fifth author) conceived of the study, and helped to draft the manuscript. All authors read and approved the final manuscript.

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