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Title: **Chemical Processing and Functionalization of Carbon-nanotubes for Optimization of Gas Sensing Properties**

Abstract

In Ph.D. work three types of MWCNTs composites i.e. MWCNT-ceramic, MWCNT-polymer and MWCNT-metal oxide composites, were synthesized and resistive type gas sensors were fabricated in order to investigate their gas sensing behavior. Different series of MWCNTs ceramic (alumina) composites had been synthesized by following sol-gel route as described below:

1) Series one was prepared by functionalizing MWCNTs to monitor the effect of functionalization on gas sensing behavior of these composites.

2) Series two was prepared by varying concentration of MWCNTs in composites to investigate the effect of MWCNTs concentration on gas sensing properties of the composites.

3) Series three was prepared by varying the annealing temperature of the composites to observe the effect of annealing temperature on ammonia gas sensing properties of the composite.

The MWCNT-alumina composite based sensors were found to have a good reproducible response at room temperature and easily detected 6 ppm of NH3 gas with response time of around 10 minutes. The thermal treatment protocol was employed to achieve complete and fast recovery with proper selection of temperature during thermal treatment and we have been successful in our attempt to acquire complete and fast recovery (in ~20 minutes) by selection of suitable thermal treatment regime. In this work two types of MWCNT-polymer composites were fabricated using both non-conducting and conducting polymers. Non-conducting polymer, PVP (poly vinyl pyrrolidone) and two types of conducting polymers, poly (3, 4-ethylenedioxythiophene)–polystyrene sulfonic acid (PEDOT: PSS) and Polyaniline (PANI) were used to prepare MWCNT-polymer composites following solution casting method. Two series of MWCNT-conducting polymer composites were prepared to examine the effect of functionalization and defects and effect of MWCNTs-HNO_3 treatment time and different types of defects on MWCNTs on gas sensing properties of MWCNT-PEDOT: PSS composite based gas sensor. Additionally, thermal stability study was carried out for both the composites and MWCNT–PEDOT: PSS composite was found to show better thermal stability than MWCNT–PANI composite. To avoid any polymer degradation at high temperature,

a suitable thermal treatment protocol along with application of small DC bias voltage was employed for MWCNT–PEDOT: PSS composite sensor and recovery time was reduced to 20 min. The MWCNT–PEDOT: PSS composite sensor was found to exhibit excellent response for trace level sensing (1–50 ppm) of ammonia gas.

Following major conclusions have been drawn from the research work reported in the present Ph.D. thesis:

- Structural defects and defect sites on the surface of nanotubes (created during the acid treatment) are supposed to increase the MWCNTs surface activity, resulting in significant improvement in gas sensing response of CNT based gas sensors. Hence for CNT based gas sensor fabrication HNO_3 fuctionalization may get the preference over HCl and (3:1) H_2SO_4: HNO_3 functionalization.
- Two types of conducting polymer composite based ammonia gas sensors were integrated in resistive sensor design, using poly (3, 4-ethylenedioxythiophene)–polystyrene sulfonic acid (PEDOT: PSS) and Polyaniline (PANI). We conclude that MWCNT-PEDOT: PSS polymer is the most promising candidate for gas sensor fabrication. Hence we prefer to work only with PEDOT: PSS polymer rather than PANI.
- Sensor recovery posed a great problem at room temperature and a new approach is proposed to get complete recovery. A suitable thermal treatment protocol along with application of small DC bias voltage was employed for MWCNT-PEDOT: PSS composite sensor and recovery time was reduced to 20 min. The MWCNT-PEDOT: PSS composite sensor was found to exhibit excellent response for trace level sensing (1-50 ppm) of ammonia gas.
- A systematic study based on the defect sites created on MWCNTs by means of acid treatment when refluxed in HNO_3 for different times and its effect on the gas sensing properties of the corresponding MWCNT-PEDOT: PSS composite based gas sensor was also conducted.
- Gas sensing response of MWCNT-polymer composite was found to increase when acid treatment time was increased up to 10 h but dramatically decreased thereafter. Concluding that addition of defects in a controlled manner may improve the gas sensing response of carbon nanotubes up to the great extent but extensive acid treatment may damage as well as break the carbon nanotube into small segments, resulting in degradation of gas sensing response.
- The reported work of this thesis has many applications in the development of carbon nanotubes based gas sensors for low level gas detection at room temperature. The results reported in this thesis work mainly indicates the dependence of sensing response of CNT based sensors on the structure and disorders of CNTs, dispersion, interaction and concentration of CNTs in host matrix and structural configuration of the composite, which may be useful in commercialization of such types of sensor technology.