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i-Butane Sensor Made of SnO_2/Multiwall-Carbon-Nanotube Nanocomposite

V.M. Aroutiounian[1], V.M. Arakelyan[1], G.E. Shahnazaryan[1], E.A. Khachaturyan[1], M.S. Aleksanyan[1], L. Forro[2], A. Margez[2], K. Hernadi[3], Z. Nemeth[3]
[1]Yerevan State University (YSU), 1 A. Manoukyan, Yerevan, Armenia
[2]Ecole Polytechnique Fédérale de Lausanne, CH-1015 Lausanne, Switzerland
[3]Department of Applied and Envitomental Chemistry, University of Szeged, Rerrich B. tér 1, Szeged H-6720, Hungary
Corresponding e-mail: kisahar@ysu.am

Abstract

The *SnO_2/multiwall-carbon-nanotube* (MWCNT) *nanocomposite* powder was prepared by the sol-gel method and gas sensitive structures (ceramic and thick film) were manufactured made of it. The morphology of as-provided carbon nanotubes (CNTs) and prepared nanocomposite powder was investigated by scanning electron microscopy (SEM). The EDX and XRD analyses were also carried out, the element structure of a nanocomposite has been determined. The sensibilization of the nanocomposite SnO_2/MWCNT in the $Ru(OH)Cl_3$ water solution leads to the appearance of rather high sensitivity to *isobutane* already at 120°C. The sensitivity of prepared sensors was measured at different gas concentration. The influence of variation of the sensibilized solution concentration and the work body temperature on the *sensor sensitivity* was investigated.

Introduction

SnO_2 is the most widely studied metaloxide among all other oxides used for the manufacture of gas sensors, 42% of publications in last 30 years focused on SnO_2. Unique properties allow using CNTs in many different applications, in particular, for the development of the new sensor to detecting of different gases. We did not detect i-butane by our sensors made of both pure SnO_2 and CNTs. But we expected to realize some syn-energetic results in hybrid sensors made of nanocomposite consisting both non-functionalized CNTs and inorganic nanoparticles like SnO_2 [1]. Here we reported about the manufacture and investigations of hybrid sensors made of nanocomposite MWCNT/SnO_2/Pd structure.

Experimental results and discussion

Millimeter long MWCNT grown by CVD were used to prepare membranes by vacuum filtration form a suspension in isopropanol [2]. These membranes were employed for the preparation of the nanocomposite SnO_2/MWCNT powder by the sol-gel method [1].

SEM micrographs of the SnO_2/MWCNT nanocomposite powder prepared in YSU show the presence of metal oxide grains and CNTs in a few regions. Results of EDX analysis have shown that the fraction of CNTs in the investigated SnO_2/MWCNT nanocomposite powder was equal 4-5 wt.%. Using the XRD measurements, we could confirm that heat treated nanoparticles have the SnO_2 crystal structure. Wide diffraction peaks testify that the nanocomposite SnO_2/MWCNT powder contain both tin oxide and MWCNT phases.

Tablets are pressed from one part of a powder. Some tablets were sensibilized at the 0.01 and 0.03M $Ru(OH)Cl_3$ water solution. Then tablets were exposed to the second heat treatment in air (400°C, 10 hours). Powder polypropylene (2.5-3 wt.%) as binder was added to the another part of nanocrystalline powder with the aim of the manufacture of samples with sufficient density and stabilization of sensors parameters. For the manufacture of gas sensor, palladium catalytic nanoparticles and gold inter-digital ohmic contacts have been created on the surface of tablets and films made of the nanocomposite SnO_2/MWCNT powder using the ion-plasma sputtering method. The gas sensing properties of the sensors made of the nanocomposite MWCNT/SnO_2/Pd structures were tested in the presence of i-butane. Sensor responsiveness (S) was determined as the ratio between the sensor resistance in air (R_{air}) and the sensor resistance in the presence of the pollutant after reaching a steady state (R_{gas}). All investigations of the sensitivity were

performed via computer control of the gas concentration and temperature of the work body.

Results of these investigations have shown that sensors made of the MWCNT/SnO_2/Pd structure sensibilized with the 0.03 M $Ru(OH)Cl_3$ water solution have rather high sensitivity to isobutane already at 120°C. The resistance of sensor was equal ~700 kOhm at this temperature. It is decreased in 10 times through ~10 s after injection of i-butane. The sensor sensitivity increases together with increase of the temperature, their characteristics are stabilized. The endurance in the environment of isobutane gas for 24 h, despite some decrease in the sensitivity, leads to stabilization of sensors parameters and, in particular, to essential reduction of the system recovery time up to 2-3 minutes. For sensors prepared with used the powder polypropylene as binder the largest sensitivity is observed at 300°C (Fig.1a). Dependence of the sensitivity on the concentration of i-butane has almost linear character (Fig.1b). For these sensors, almost identical system times of the response and recovery were observed. They are equal ~30-40 s at low concentration of gas.

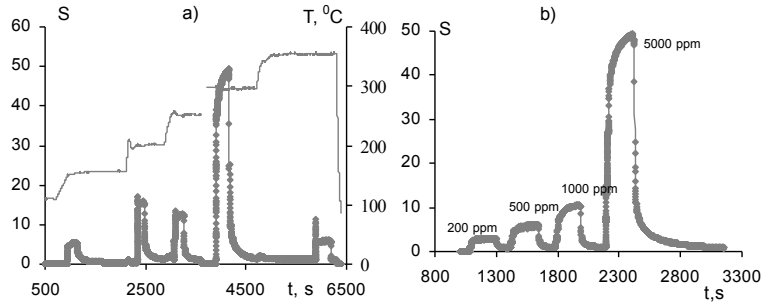

Fig. 1: Sensitivity for the sensors made of the MWCNT/SnO_2/Pd nanostructure prepared by use of binder and sensibilized with the 0.01 M $Ru(OH)Cl_3$ solution at different temperature (a, 5000 ppm) and different concentration of isobutane (b, 300°C).

We manufactured thick films made of the MWCNT/SnO_2 powder nanocomposite by the spray coating method on glass ceramics substrates. The solution 0.5 g SnO_2/MWCNT powder in 10 ml absolute dimethyl formamide was prepared. Three layers were precipitated consistently on the substrate for preparing of samples with necessary conductivity. The sensors made of these films have shown sufficient sensitivity to i-butane at temperatures more 200°C. The sensibilization of films in the 0.01 M $Ru(OH)Cl_3$ solution led to the best sensitivity. These sensors characteristics steady enough, times of the response and recovery are equal ~30 s.

Conclusion

Nanocomposite SnO_2/MWCNT were prepared by the sol-gel method. Results of SEM, EXD, the element structure and XRD investigations shown that the nanocomposite SnO_2/MWCNT powder can be considered as prepared by the sol-gel tin oxide doped with MWCNTs. A technology for the manufacture of ceramic and thick film hybrid i-butane sensors made of the MWCNT/SnO_2/Pd nanostructure was developed. The prepared ceramic sensors are sensitive to isobutane already at its concentration 200 ppm at 120°C. The thick film sensors made of the MWCNT/SnO_2/Pd had the sensitivity to isobutane more than 5-7 at the temperature of the work body 250°C.

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