CNT-Ni-Pd nanocomposite films for optical gas sensor

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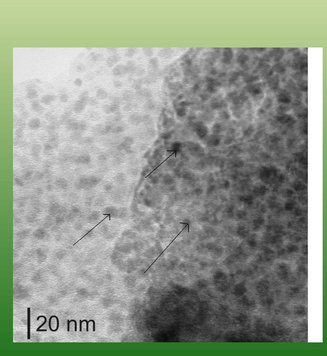
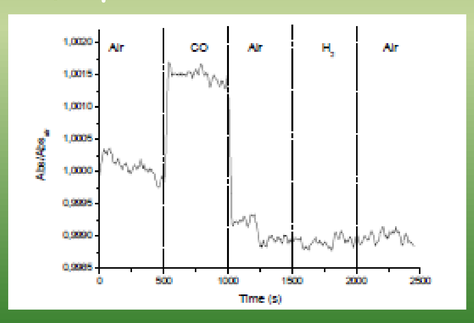
We proposed new type of materials designed for optical gas sensor. These materials are nanocomposite carbon nanotubes - nickel - palladium films. These films were prepared by PVD/CVD mixed methods. The initial film was prepared by physical vapor deposition (PVD) method using two separated sources: fullerene C60 (as carbon source) and nickel acetate (as metal source). Next, to obtain carbon nanotubes (CNTs) initial film was modified by chemical vapor deposition (CVD) with xylene. Then, on the carbon nanotubes film, palladium-fullerene films were deposited by PVD method.
Obtained film's morphology and topography were studied with SEM and EDS analysis. Optical gas sensing properties of the nanocomposites films have been tested measuring the variation of the absorbance of different gases (H2, CO, NO2).

Sample S1

Preparation - S1 was deposited on quartz substrates by PVD method. The process was performed under dynamic pressure of 10-3 Pa. Nickel acetate was used as a precursor of metal and fullerite C60 powder was applied as a precursor of carbon. S1 sample are very smooth and composed of very fine Ni nanocrystals (few nm in diameter) placed in carbonaceous matrix.

TEM image of S1





Dynamic scan of the sample S1 at operating temperature OT=80°C, λ=2325nm

Samples A1 and B1 were obtained by modification of sample S1 in a quartz reactor by CVD method using xylene C_8H_{10} as an additional source of carbon for the growth of carbon nanotubes on Ni grains. CVD process was performed at atmospheric pressure in the temperature of 650°C applying argon flow (a rate of flow = 40l/h) as the carrier gas for xylene vapor. After CVD modification the growth of carbon nanotubes was observed. The feed rate of xylene was 0.1ml/2 min and duration of CVD was t=15 minutes for both samples. The amount of using xylene was 0.5 ml.

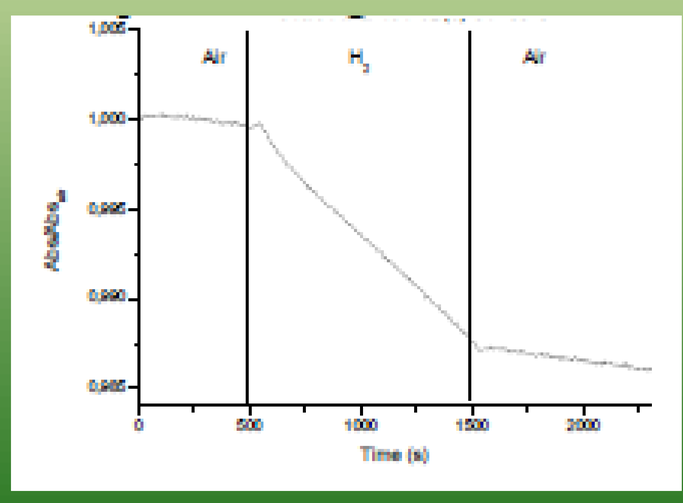
sample A1 (nanotubes marked with white arrows)

Optical gas sensing tests were conducted by measuring optical absorbance in the 250-2500 nm wavelength range on films deposited on SiO_2 substrates using a Harrick gas flow cell coupled with a Jasco V-650 spectrophotometer. The operating temperature (OT) was varied between room temperature (RT) and 80 °C and different gas concentrations up to 1% volume of CO and H_2 and up to 0.01% volume of NO_2 in dry air were utilized. The substrate size was approximately 1 cm × 2 cm and the incident spectrophotometer beam was normal to the film surface and covered a 9 mm × 1.5 mm area of the film.
For all the samples it was first measured the transmittance in air and then in gas in order to determine the wavelengths at which the absorbance variation was larger. For such wavelengths, dynamic measurements have been performed in order to compare the response time for the different gases

A2 and B2 samples - A1 and B1 samples were covered with palladium in PVD process performed in the same way as initial PVD process with this difference that in a place of nickel acetate was used palladium acetate.

Sample A2 sample B2

Pd nanograins covering nanotubes are marked with white arrows



Dynamic scan of the sample B2 at OT=80°C and λ=1050nm

CONCLUSIONS

1. All the samples coated with Pd did not give a stable variation of the absorbance under the different gases, showing irreversible variation and/or very large drift, especially in the case of H_2.
2. A1 and B1 samples were sensitive for CO and NO_2.
3. S1 sample showed very good response to CO