DOI 10.5162/IMCS2018/P2GS.5

Fabrication and Characterization of C_2H_2 Gas Sensor Based on PdO doped In_2O_3 as Prepared by Flame Spray Pyrolysis

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Abstract:
0–1.0wt% PdO doped In_2O_3 nanoparticles have been successfully produced in a single step by flame spray pyrolysis (FSP) technique using indium(III) nitrate hydrate and palladium(II) acetylacetonate, as precursors dissolved in ethanol and their acetylene sensing characteristics have been investigated. The particle and sensing film properties were analyzed by XRD, BET, TEM and XPS. The sensing films were prepared by spin coating technique. The crystallite sizes of In_2O_3 spherical and cubic morphologies were found to be ranging from 5 to 20 nm and PdO might form solid solution with In_2O_3 lattice. Gas-sensing characterization were studied at the operating temperatures ranging from 200 to 400°C in dry air, acetylene sensing characteristics of In_2O_3 nanoparticles is significantly improved as Pd content increased from 0 to 1.0wt.%. The 0.75wt% PdO doped In_2O_3 sensing film showed an optimum C_2H_2 response of ~948 at 3% acetylene concentration and 350°C operating temperature. In addition, PdO doped In_2O_3 sensing films exhibited good selectivity towards hydrogen and acetylene gas.

Key words: Flame spray pyrolysis, PdO doped In_2O_3, Gas sensor, C_2H_2 sensor

Introduction

Acetylene (C_2H_2) is a colorless with a faint garlic-like odor widely used as fuel and in many industrial applications, such as a raw material for the production of experimental electrically conducting plastics, used with high purity air as a fuel for the flame in atomic absorption flame spectroscopy, used in water and biological research laboratories. It is quite unstable in pure form and usually handled in solution and becomes highly explosive when it is liquefied, compressed, heated or mixed with air. For this reason, special safety measure is vital during its production and handling. At the same time, the range of interest for its detection is much wider, typically 100–100,000 ppm, allowing for early leakage warning and explosive indication. So it's essential to detect the content of C_2H_2 to avoid more serious accidents at incipient faults. In this study the optimization of the doping conditions for PdO doped In_2O_3 nanoparticles are produced by flame spray pyrolysis. The effect of PdO on gas sensing performances are systematically studied and optimized for selective detection of H_2 and C_2H_2 gas.

Materials and Methods

Firstly, 0–1.0wt% PdO doped In_2O_3 nanoparticles were prepared by flame spray pyrolysis technique, which was previously established by our group. For sensor fabrication, flame-made 0–1.0wt% PdO doped In_2O_3 nanopowders were thoroughly mixed and ground with the binder solution. The resulting paste was spin coated on Al_2O_3 substrates equipped with Au interdigitated electrodes to form a sensing film. The resulting substrates annealed at 450°C for 2 h in an oven for binder removal prior to sensing test. The gas-sensing performances of all sensors were characterized towards H_2, C_2H_2, C_2H_4, C_2H_5OH, H_2S and NO_2 under atmospheric conditions by the standard flow through technique in stainless steel chamber at operate temperature in range of 200–400°C.

Results and discussion

Characterizations significantly confirmed Pd^{2+} was formed solid solution with In_2O_3 lattice ((2 2 2), (4 0 0) and (4 4 0)). Fig. 1 shows BF-TEM image with corresponding SAED pattern of pure In_2O_3 and optimal PdO doped In_2O_3 nanoparticles. It is seen that the spherical and cubic nanoparticles with the particle size in the range of 5–20 nm. The corresponding SAED patterns display diffraction rings of polycrystalline In_2O_3 nanoparticles. The observed In state can be assigned to the highest oxidation state of In^{3+} for In_2O_3. For Pd element can be assigned to Pd^{2+} of PdO and Pd^{4+} of PdO_2 consistent with some other reports on XPS study. The effect of operating temperature ranging from 200 to 400°C on C_2H_2 response of In_2O_3 nanoparticles with different In PdO doping indicated that the 0.75 wt% PdO doped In_2O_3 sensor exhibits the highest response of ~948 to 3 vol% C_2H_2 at high temperature of 350°C. PdO doped In_2O_3 sensing films exhibited good selectivity towards hydrogen and acetylene gas.

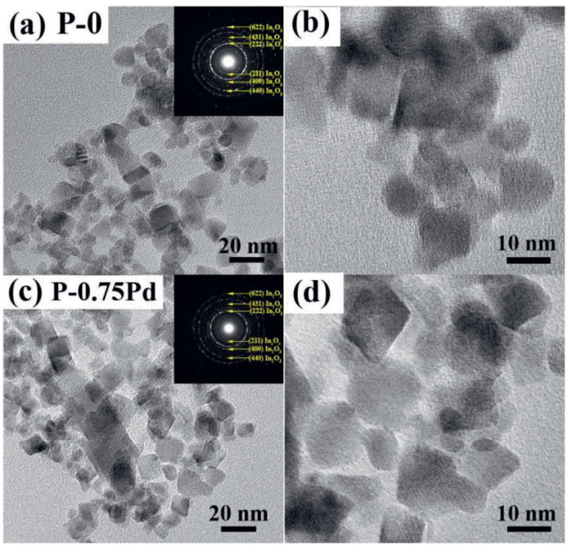

Fig.1. TEM images of the FSP-made (a, b) undoped In_2O_3, (c, d) 0.75 wt% PdO doped In_2O_3 nanoparticles. Insets: the corresponding SAED patterns.

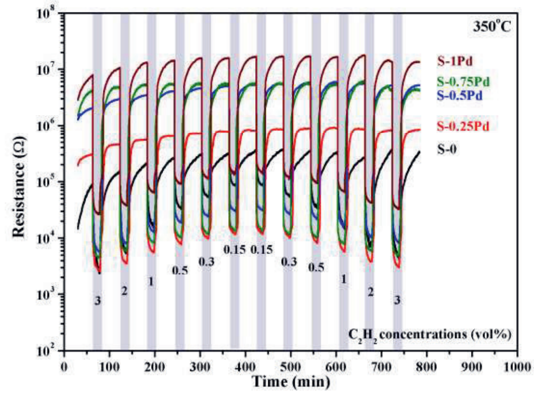

Fig.2. The change in resistance of In_2O_3 sensing films with different PdO doping concentrations under exposure to various C_2H_2 concentrations ranging from 0.15 to 3 vol% in terms of backward and forward concentrations at the operating temperature of 350°C.

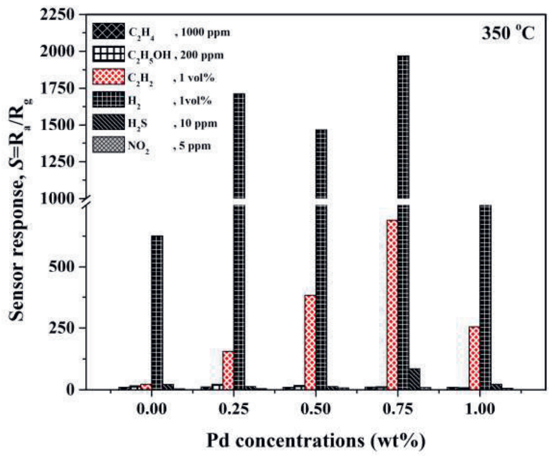

Fig.3. Selectivity histograms: sensor response of 0–1.0wt% PdO doped In_2O_3 sensors to 1,000 ppm C_2H_4,200 ppm C_2H_5OH, 1vol% C_2H_2,1 vol% H_2, 10 ppm H_2S and 5 ppm NO_2 at the optimal operating temperatures of 350°C.

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