Fabrication and Characterization of C2H2 Gas Sensor Based on PdO doped In2O3 as Prepared by Flame Spray Pyrolysis

K.Inyawilert1, A. Wisitsoraat2, C. Liewhiran1, S. Phanichphant3,*

1 Department of Physics and Materials Science, Faculty of Science, Chiang Mai University, Chiang Mai 50202, Thailand
2 Nanoelectronics and MEMS Laboratory, National Electronics and Computer Technology Center, National Science and Technology Development Agency, Klong Luang, Pathumthani 12120, Thailand
3 Materials Science Research Center, Faculty of Science, Chiang Mai University, Chiang Mai 50202, Thailand

sphanichphant@yahoo.com*

Abstract:

0–1.0wt% PdO doped In2O3 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 In2O3 spherical and cubic morphologies were found to be ranging from 5 to 20 nm and PdO might form solid solution with In2O3 lattice. Gas-sensing characterization were studied at the operating temperatures ranging from 200 to 400°C in dry air, acetylene sensing characteristics of In2O3 nanoparticles is significantly improved as Pd content increased from 0 to 1.0wt.%. The 0.75wt% PdO doped In2O3 sensing film showed an optimum C2H2 response of ~948 at 3% acetylene concentration and 350°C operating temperature. In addition, PdO doped In2O3 sensing films exhibited good selectivity towards hydrogen and acetylene gas.

Key words: Flame spray pyrolysis, PdO doped In2O3, Gas sensor, C2H2 sensor

Introduction

Acetylene (C2H2) 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 C2H2 to avoid more serious accidents at incipient faults. In this study the optimization of the doping conditions for PdO doped In2O3 nanoparticles are produced by flame spray pyrolysis. The effect of PdO on gas sensing performances are systematically studied and optimized for selective detection of H2 and C2H2 gas.

Materials and Methods

Firstly, 0–1.0wt% PdO doped In2O3 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 In2O3 nanopowders were thoroughly mixed and ground with the binder solution. The resulting paste was spin coated on Al2O3 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 H2, C2H2, C2H4, C2H5OH, H2S and NO2 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$_2$O$_3$ lattice ((2 2 2), (4 0 0) and (4 4 0)). Fig. 1 shows BF-TEM image with corresponding SAED pattern of pure In$_2$O$_3$ and optimal PdO doped In$_2$O$_3$ 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$_2$O$_3$ nanoparticles. The observed In state can be assigned to the highest oxidation state of In$^{3+}$ for In$_2$O$_3$. 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$_2$H$_2$ response of In$_2$O$_3$ nanoparticles with different In PdO doping indicated that the 0.75 wt% PdO doped In$_2$O$_3$ sensor exhibits the highest response of ~948 to 3 vol% C$_2$H$_2$ at high temperature of 350°C. PdO doped In$_2$O$_3$ sensing films exhibited good selectivity towards hydrogen and acetylene.

![Fig.1](image1.jpg)

**Fig.1.** TEM images of the FSP-made (a, b) undoped In$_2$O$_3$, (c, d) 0.75 wt% PdO doped In$_2$O$_3$ nanoparticles. Insets: the corresponding SAED patterns.

![Fig.2](image2.jpg)

**Fig.2.** The change in resistance of In$_2$O$_3$ sensing films with different PdO doping concentrations under exposure to various C$_2$H$_2$ concentrations ranging from 0.15 to 3 vol% in terms of backward and forward concentrations at the operating temperature of 350°C.

![Fig.3](image3.jpg)

**Fig.3.** Selectivity histograms: sensor response of 0–1.0 wt% PdO doped In$_2$O$_3$ sensors to 1,000 ppm C$_2$H$_4$, 200 ppm C$_2$H$_5$OH, 1 vol% C$_2$H$_2$, 1 vol% H$_2$, 10 ppm H$_2$S and 5 ppm NO$_2$ at the optimal operating temperatures of 350°C.

**References**

