Synthesis of SnO_2 Nanosized Powder by Mechanochemical Method for Sensing of H_2S

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Abstract

In a mechanochemical process $SnCl_2$ and $Ca(OH)_2$ powders were milled in a ball-mill at room temperature and in an air atmosphere. No X-ray diffraction peaks were observed for the as-milled powder but heat treatment above 300 °C causes the appearance of only SnO_2 and $CaCl_2$ phases confirming that solid-state displacement reactions have been started during a ball-milling without external heating. Removal of the $CaCl_2$ by-product was carried out by washing the powder with distilled water using a centrifuge. The powders obtained were impregnated with an aqueous solution of ($AgNO_3$ or $La(NO_3)_3{\cdot}6H_2O$) followed by heat treatment at a low temperature(450 °C) which was still sufficient to decompose the salt and to distribute the surface additive on a fine scale. Higher decomposition temperatures may cause agglomeration of the additive particles. Surface-doped SnO_2 powders were then mixed in polyvinyl alcohol (PVA) solution to form pastes and coated onto Al_2O_3 substrates with comb-type Au electrodes and sintered. On exposure to 1 ppm of H_2S gas the Ag_2O-SnO_2 thick film has higher sensitivity compared to that of the undoped and lanthanum-doped SnO_2. High sensitivity to H_2S gas was gained due to fine crystallites of synthesized tin dioxide powder with unagglomerated surface microstructure. The BET particle size was less than 25 nm. Surface doping by impregnation seemed to be effective since the small SnO_2 crystallite size was maintained and it was controlled by varying the concentration of solution and time. The results show that by an appropriate powder synthesis method and control of surface doping of SnO_2 can be modified for use as a H_2S gas sensor in the concentration range below 1 ppm.

Key words: ball-milling, heat treatment, by-product, SnO_2, surface doping, XRD, H_2S adsorption, electronic conductivity.