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Gas sensitivity of sol-gel prepared mesoporous WO₃ thin filmM. Takács^{a,b,*} and A. E. Pap^a^a*Institute of Technical Physics and Materials Science – MFA, Centre for Energy Research
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Abstract

Tungsten oxide thin film was prepared by sol-gel method from tungsten hexachloride (WCl₆) precursor material in analytical grade ethanol. In order to form the porous nanostructured WO₃ thin film Pluronic F127 was added to the solution. The sol-gel solution was deposited by spin coating on micro-hotplates containing interdigital platinum electrodes on top to measure the sensing layer conductivity. The porous tungsten oxide films were sensitized with ~20 nm platinum nanoparticles. Sensor responses of pure and sensitized porous WO₃ were measured and their responses for 100 ppm H₂S in synthetic air were compared in the 140–260°C operation temperature range. The presence of platinum nanoparticles significantly increased the sensitivity for H₂S gas. Test results of different operating temperatures were investigated in terms of sensitivity, stability and response time.

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1. Introduction

Semiconductor metal-oxide (SMO) thin films are among the most intensively investigated materials for gas sensing devices. The best SMO gas sensors exhibit quick sensing response, simple implementation, low manufacturing cost and have stable chemical and thermal properties over extended period. A large variety of deposition methods has been proposed to prepare thin films, such as spray pyrolysis, physical vapor deposition,

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pulsed laser deposition, sol-gel technique, etc [1]. As the thickness of space charge layer at the crystal surfaces plays crucial role in the nano-porous material, these layers are more sensitive than the compact grain structured thin films. Moreover, the high surface-to-volume ratio enhances the sensing area and the gas easily diffuses through the surface into the bulk. In contrary, the gas can not diffuse in the compact grain structured materials. It is well-known that addition of nano-sized catalyst, such as noble metals typically enhances the gas sensitivity of the thin layer. In this work we prepared non-doped and platinum sensitized mesoporous WO_3 thin films by sol-gel technique and investigated the effect of Pt on gas sensing properties.

2. Experimental

2.1. Mesoporous WO_3 thin film

The WO_3 thin films were synthesized by sol-gel technique. Different amount of Pluronic F127 block copolymer were dissolved in 10 ml analytical grade ethanol. 1g tungsten hexachloride (WCl_6) powder was added to the solution and stirred for 1h, followed by ageing for another 24h at room temperature [2]. The WO_3 sol was transferred onto the surface of the micro-hotplates by spin-coating technique (2000 rpm). Combined with lift-off technique, sensing layers of 75 μm diameter and 150 nm thickness were formed on the top of the hotplate. Applying 5 $^\circ\text{C}/\text{min}$ ramp up heating the sensors were annealed at 300 $^\circ\text{C}$, 400 $^\circ\text{C}$ and 500 $^\circ\text{C}$ in air for 90 minutes to form the final mesoporous structured WO_3 layers (Fig. 1a.). Finally, the chips were exposed to O_2 plasma treatment ($P=400\text{ W}$, $t=5\text{ min}$) to remove the residues of solvent and all organic ligands.

2.2. Platinum nanoparticles sensitized WO_3 thin film

3.6 ml of a 0.2 M solution of chloroplatinic acid hexahydrate was added to 46.4 ml of boiling distilled water. After 1 min, 1.1 ml of 1 M sodium citrate solution was added, then 0.55 ml freshly prepared sodium borohydrate (0.08 M) solution containing 1 M sodium citrate was quickly injected. After 10 min the product was cooled down to room temperature. These nano-particles were used as seeds to grow the larger ($\sim 20\text{ nm}$) particles. 1 ml of the platinum seed solution was dissolved in 29 ml distilled water at room temperature and mixed with 0.045 ml of a 0.4 M chloroplatinic acid hexahydrate. This solution was mixed with 0.5 ml 1 M sodium citrate and 1.25 M L-ascorbic acid. Under stirring, the temperature was slowly increased to the boiling point ($\sim 10\text{ }^\circ\text{C}/\text{min}$) [3]. The synthesized platinum ($\sim 20\text{ nm}$) nano-particles were dropped onto the above described WO_3 sensing layer onto the middle of the membrane. Finally, the sensors were annealed at 300 $^\circ\text{C}$ for 15 minutes and exposed to O_2 plasma treatment ($P=400\text{ W}$, $t=5\text{ min}$) to remove the solvent and to form separated platinum nanoparticles on the WO_3 films (Fig. 1b,c).

3. Results

The micro-sensor is built on a sandwich structured, non-perforated membrane with embedded Pt heater and Pt interdigitated electrodes on top. The comb-like Pt electrodes serve for measuring the resistance of the sensing layer. The membrane was released by deep reactive ion etching of Si underneath. The Pt filament and the Pt interdigitated electrodes were positioned in the middle of the membrane.

The obtained WO_3 thin layers have different porosities due to the different concentrations of the F127 surfactant molecule. The WO_3 films were annealed at different temperature to remove the Pluronic block copolymer to get the porous thin layer. In order to prepare WO_3 films of the best porosity we optimized these last two parameters (F127 concentration and annealing temperature). Fig. 1a shows that the highest porosity is provided by solution containing 1g F127 if annealed 300 $^\circ\text{C}$. The WO_3 layer loses its integrity and cracks if the annealing temperature exceeds 300 $^\circ\text{C}$.

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