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Oxygen Sensing Properties of the WO_3 Thick Films

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Abstract: This paper presents oxygen gas sensing properties of WO_3 thick films. In this work, the WO_3 thick films were prepared by standard screen-printing method. These films were characterized by X-ray diffraction (XRD) measurements and scanning electron microscopy (SEM). The oxygen gas sensing properties of these thick films were investigated at different operating temperatures and oxygen gas concentrations. The WO_3 thick films exhibit excellent oxygen sensing properties with a maximum sensitivity of ~ 35.75 % at 350 °C in air atmosphere with fast response and recovery time. *Copyright © 2009 IFSA.*

Keywords: WO_3 thick film, Screen printing, Oxygen gas

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1. Introduction

Concern over environmental pollution and health issues has driven legislation over the past two decades and significant research and development efforts have been undertaken to address environmental issues. Oxygen sensing is a very important issue because it can prevent the ignition of explosive gases. In particular, oxygen sensors have played a key role in pollution control through automobile engine management, optimizing industrial boilers, steel, cement industries, biological and food processing plants and control of chemical processes [1]. It is also important in some areas of medical applications, mainly in equipment for medical ventilation assistance used for pulmonary diseases. Based on the number of sensors in operation, the predominant use of oxygen sensors is in the control of air-fuel mixture in the combustion engine of the automobiles and is an integral part of the 'on board diagnostic' (OBD) of the exhaust emission control system [2, 3].

Semiconductors oxide based gas sensors prepared by the screen printing method have certain advantages with respect to other type of gas sensors. To detect small concentration of a reactive gas in air, the surface reactions are much more relevant than bulk changes, so the specific surface of gas sensitive elements must be as high as possible. The screen printing technology is adequate for satisfying such a primary requirement. In addition to high sensitivity, thick film sensors have high stability and simple construction at low cost [4, 5].

Tungsten trioxide (WO_3) films are reported to have promising electrical and optical properties for various applications like efficient photolysis, electrocromic devices, selective catalysts and gas sensors [6]. Amorphous and polycrstalline WO_3 films are particularly attractive as gas sensors because they show a high catalytic behaviour both in oxidation and reduction reactions [7]. Electrochromatic devices which exploit WO_3 are tyoically in an amorphous form, whereas electrical devices such as gas sensors, are in a crystalline form [8]. Tungsten also forms other oxides such as WO, W_2O_3 and W_4O_3, however, in gas sensing the stable WO_3 form is used.

In recent years, WO_3 thick films have been considered to be the most promising materials for detecting reducing gases like NO_2, CH_4, NH_3, H_2S, CO, CO_2, LPG and organic vapors [9-15].

The aim of the present study is to prepare the WO_3 thick films by using screen printing method onto alumina substrates and to investigate their sensing properties for oxygen gas.

2. Experimental

The WO_3 powder (purity ~ 99.99 %) was mechanically milled in an acetone medium using Fisher type electric agate pestle and mortar for 24 hours. After drying at 200^0C for 20 min, the powder (99.5 %) was thoroughly mixed with some organic and inorganic compounds to achieve proper thixotropic properties of the paste. The paste, thus formed was screen printed onto an alumina substrate. The films were allowed to stabilize at room temperature for 24 hours and then the films were cured at 800 ^{0}C for 1h. Our group has reported the details of the preparation method of the WO_3 thick films and it was found that strongly adherent WO_3 thick films can be obtained on the alumina substrate [14].

The structural properties of the WO_3 thick films were investigated using X-ray diffraction (XRD) technique. The X-ray diffraction patterns were recorded with a Rigaku diffractometer (Miniflex Model, Rigaku, Japan) having Cu K_α (λ = 0.1542 nm) radiation. The scanning electron microscopy (SEM) was employed to characterize the surface morphology with a Leica Cambridge 440 Microscope (UK). The thickness of the WO_3 thick film was measured by a light section microscope (Nikon Optical Microscope, M44).

The gas sensing studies were carried out using a static gas chamber to sense the oxygen gas in air ambient. The WO_3 thick films were used as the sensing elements. The sensing element was kept directly on a heater in the gas chamber and the temperature of the sensing element was monitored by chromel-alumel thermocouple placed in contact with the sensing element. The known volume of the oxygen gas was introduced into the gas chamber pre-filled with air and it was maintained at atmospheric pressure. The electrical resistance of the sensing element was measured by simple two probe configuration, before and after exposure to oxygen using a sensitive digital multi meter (METRAVI 603). The electrical contact leads were fixed 1cm apart on the surface of the film. The sensitivity (S) of the sensing element is defined as

$$S(\%) = \frac{R_g - R_a}{R_g} \times 100 ,$$

where R_a and R_g are the resistance values of the sensor element in air and in the presence of oxygen, respectively.

3. Results and Discussions

The XRD pattern of the WO_3 thick film is shown in Fig. 1. It indicates the diffraction peaks at 2θ values of 23.6^o, 24.2^o, 27.2^o, 28.2^o, 33.8^o, 42.2^o, 48.8^o, 50.8^o 54.0^o, 54.6^o, 55.2^o, 56.4^o, 61.6^o and 62.6^o, which reveals the formation of the triclinic phase of WO_3 (JCPDS Data card 83-0948).

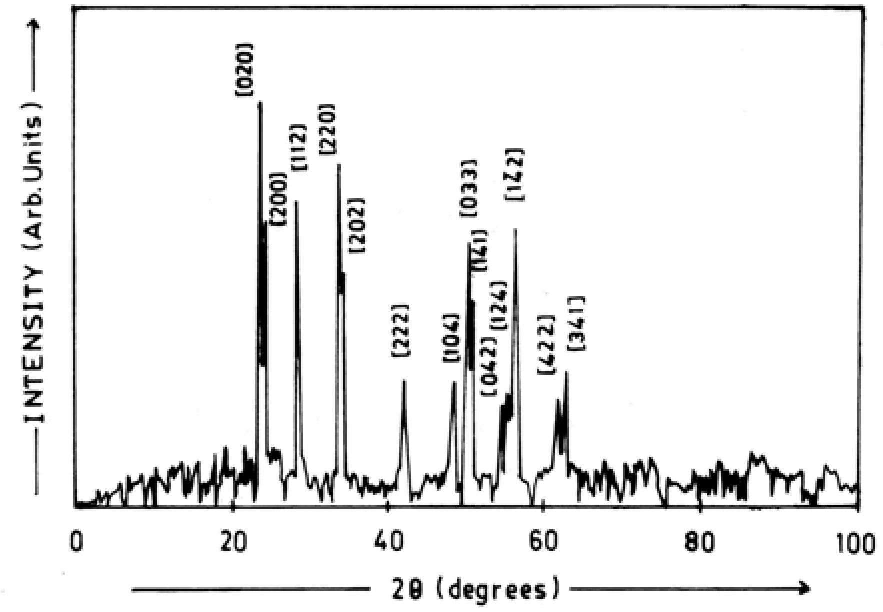

Fig. 1. The X-ray diffraction pattern of the WO_3 thick film.

The surface morphology of the screen printed WO_3 thick film deposited onto the alumina substrate is as shown in Fig. 2. It clearly shows that the surface of WO_3 films is porous and forms channels with bundles of grains.

The dependence of sensitivity of WO_3 thick film towards 50 ppm oxygen gas on the operating temperature is shown in Fig. 3. It is observed that the sensitivity of WO_3 thick film to 50 ppm oxygen gas increases as the operating temperature is raised from 200- 350^oC. At 350^oC, the sensitivity reaches to maximum and it is found to be ~35.75 % at 350^oC. With further increase in the operating temperature, the sensitivity significantly decreases. The response time of the WO_3 thick film to oxygen gas was nearly 10 s and the recovery time was found to be nearly 15 s.

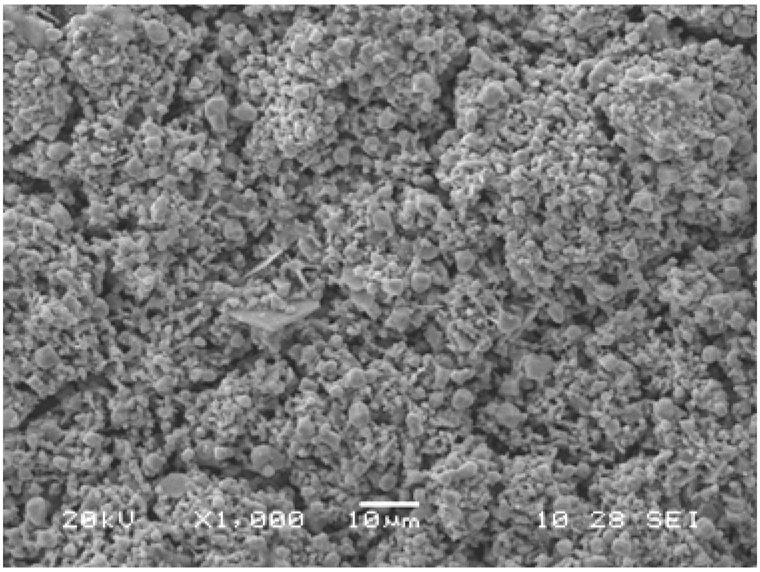

Fig. 2. SEM image of the screen printed WO3 thick film on the alumina substrate.

The relationship between the sensitivity of the WO_3 thick film and the oxygen gas concentration for an operating temperature of 350°C is shown in Fig. 4. It is observed that the sensitivity increases linearly up to 50 ppm of oxygen gas concentration and after that it remain constant up to 75 ppm and after that it decreases. It is obvious from Fig. 4 that not only temperature but also the oxygen gas concentration plays a role in determining the sensitivity of the WO_3 thick film. The WO_3 thick film is able to detect up to 25 ppm oxygen gas with reasonable sensitivity at an operating temperature 350°C. The linearity of the sensitivity in the low oxygen gas concentration range (25-50 ppm) suggests that the screen printed WO_3 thick films can be reliably used to monitor the concentration of oxygen gas over this range.

It was found that the resistance of the WO_3 thick film increases upon exposure to oxygen. It may be due to the increase in height of the potential barrier due to concentration of oxygen species present on the surface of the material [16-17].

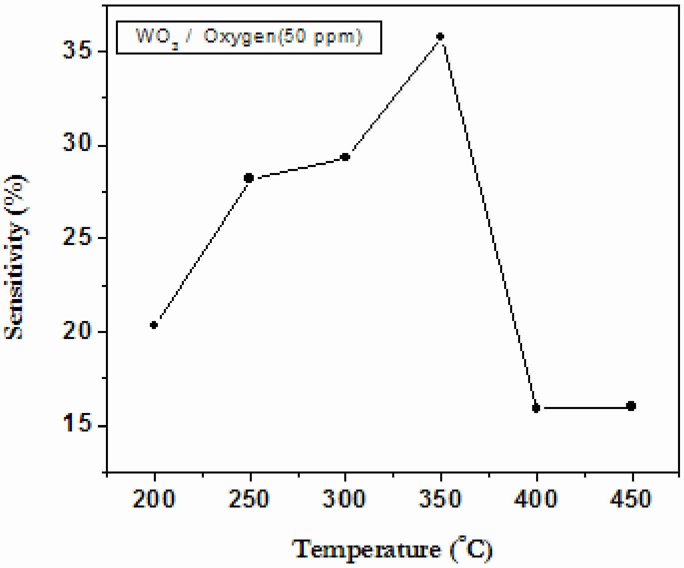

Fig. 3. Sensitivity verses Temperature graph of the WO_3 thick film for oxygen sensing.

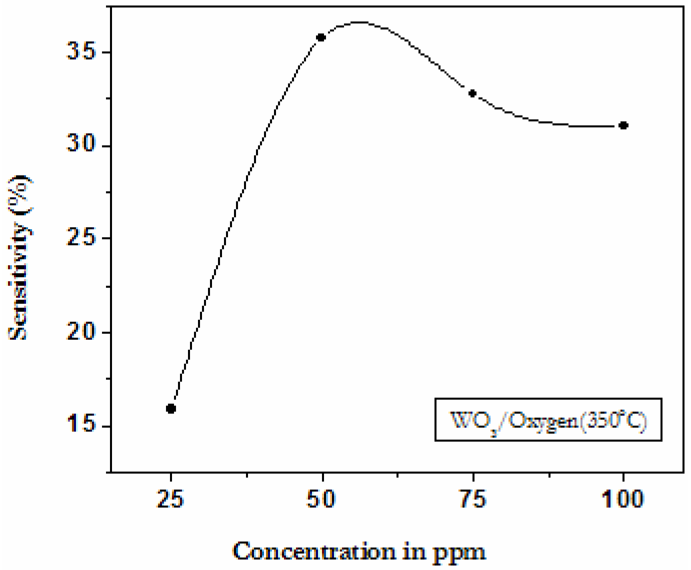

Fig. 4. Sensitivity verses Concentration graph of the WO_3 thick film for oxygen sensing.

4. Conclusions

In summary, following conclusions have been drawn from the present investigations –

- The oxygen sensing properties of screen printed WO_3 thick films were investigated.
- The highest sensitivity was obtained at operating temperature 350°C.
- All the results have been found to be repeated for three times indicating the reliability of the sensor element.
- This study demonstrates the possibility of utilizing WO_3 thick film as a sensor element for the detection of oxygen.

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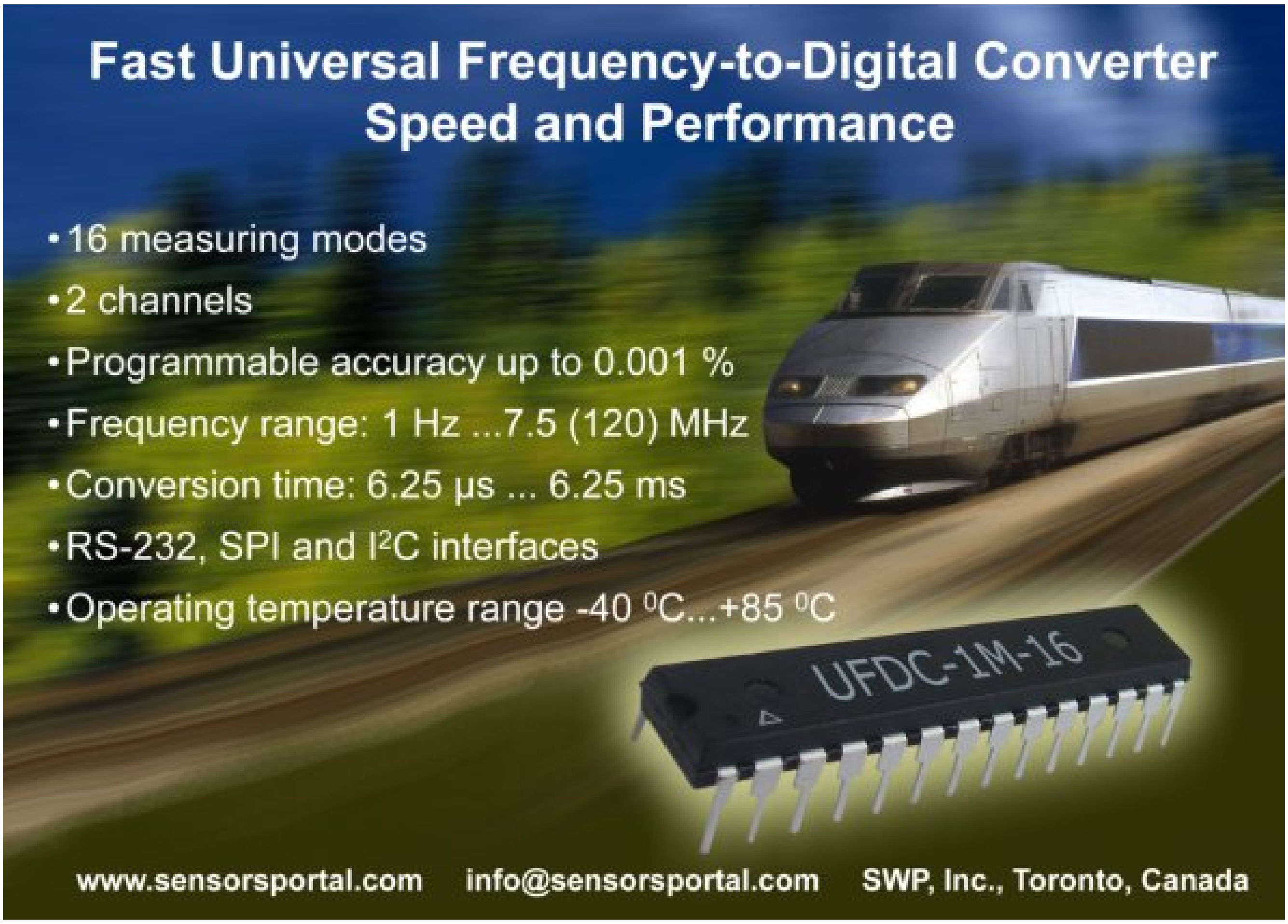

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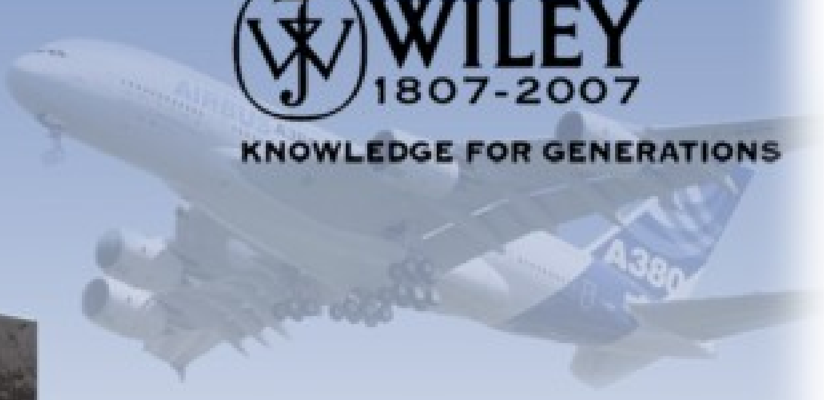

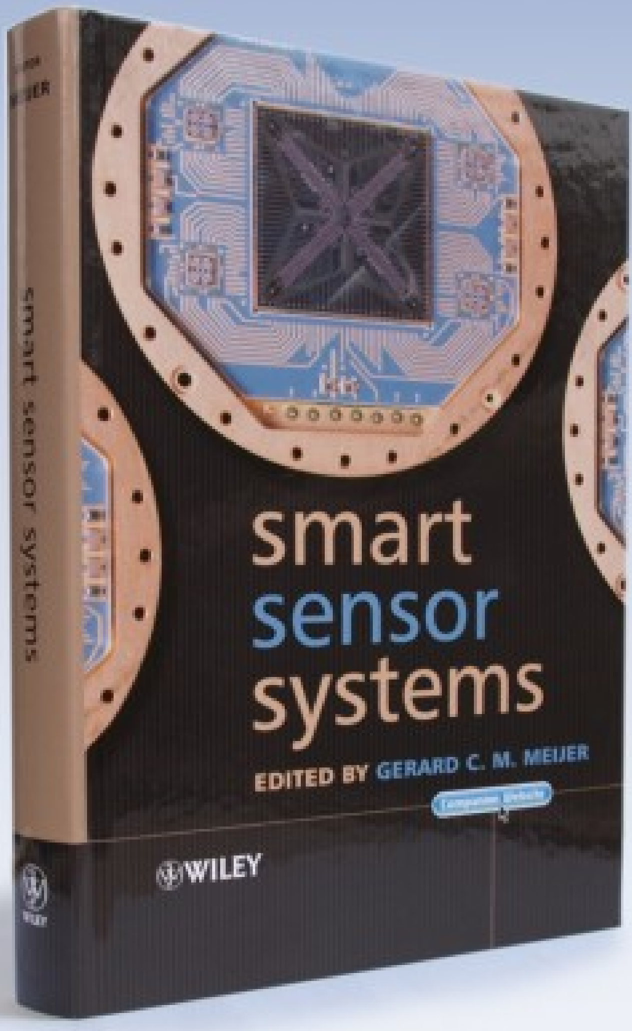

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