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Fe Doped WO₃ Thin films Prepared by Spray Pyrolysis towards Ethanol Sensing

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Abstract : WO_3 and Fe doped WO_3 thin films were deposited on glass substrate using spray pyrolysis technique. The structural properties reveals the crystalline nature with preferred oriented (200) plane along c-axis direction. There is a shift in the band edge towards longer wavelength after Fe incorporation is confirmed from optical properties. Compare to WO_3 , Fe doped WO_3 shows large sensor response towards ethanol.3

Keywords: Fe Doped, WO₃, Thin films, Spray Pyrolysis, Ethanol Sensing.

Introduction:

Medical detection, environmental protection and food industries give a prior place to ethanol sensors. Ethanol present in human breath can be easily detected by breath alcohol analysis in which detection of ethanol is an important feature¹. Present researches include the development of ethanol sensors at lower operating temperature with better selectivity and sensitivity. Metal oxide semiconductors (MOS) promises a new generation of gas sensing devices among which tungsten trioxide WO₃ has great attention^{2,3}. WO₃ doped with other elements has high performance and efficiency. The sensing properties of the film can be upgraded significantly which becomes the main objective of doping the films with catalytic ion in presence of ethanol. However no enhancement of ethanol sensing performance of with the catalytic doping was observed.

Fe is one of the most warrant materials with WO_3 to enhance the physical and chemical properties for optoelectronics device application⁴. Compare to other techniques, spray pyrolysis is the most suitable method to produce metal oxide thin film with huge number of pores and fabricate metal oxide sensors. The growth of the film use a novel spray pyrolysis deposition route with a spray nozzle and crystal structure, optical properties of Fe doped WO₃were investigated.

Experimental Procedure

Pristine and Fe activated films fabricated on glass substrate at 400 route using (0.3 M) tungsten chloride 0.5 iron chloride precursor. The carrier gas flow rate is fixed at 0.3 Kg cm². The precursor volume is 100 ml using the solvent ethanol and solution feeding rate is 4 ml/min. The distance between the substrate and the spray nozzle is kept 20 cm with the deposition time of 12 min. The spray nozzle is designed in such a way

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with two concentric glass pipes. After the deposition, the films were characterized using X-ray diffractometer (XRD), UV-vis spectrophotometer. The Fe: sensor is fabricated to measure the sensitivity towards the ethanol using the standard formula⁵.

Results and Discussions

Structural Properties

Fig. 1 shows the X-ray diffraction pattern of the deposited films. Single crystalline nature and a strong peak was observed at 24.8 of (200) phase of Absence of Fe and peaks was observed. Periodicity, textured structure and deformation of lattice led to strong intensity of (200) peak and this was also due to small difference between ionic radii. The estimable Fe intercalation on W site and distortion in crystal lattice leads to lower diffraction angle which creates a number of defects favorable for gas sensing. The crystallite size was calculated using Scherrer's formula⁶. The value of average crystallite D of film is 46.5 nm which increases to 48.3 nm after Fe doping which increases the amount of oxygen vacancies which is favorable for gas sensing.



Fig. 1. XRD pattern of (a) WO₃ and (b) Fe doped WO₃ thin films by spray pyrolysis technique

Optical Properties.

The changeover from valence band to conduction band regards to sharp absorption edge while absorption relates to certain energy levels. Fig. 2 shows the absorption spectra of the deposited films. The addition of Fe assimilates into which causes a shift towards longer wavelength owing to reduced band gap. The average transmittance greater than 70% and UV-cut off at approximately 310 nm was observed. The transmittance spectral owes to the light interference between films and substrate. The optical band gap (Eg) is found using Tauc's relation⁷. A donor level is created to narrow the band gap and this owes to doping of Fe ions leading to reduction in Eg from 2.87V to 2.60 eV. The top view of films reveal tightly packed particles. Fe incorporation produces porous structure which enhances gas sensing properties.



Fig. 2 Transmission spectrum of (a) WO₃ and (b) Fe doped WO₃ thin films by spray pyrolysis technique.

Gas Sensing Properties

Fig. 3 shows the sensor response of the resultant film with concentration of ethanol. The sensor response increases and decreases corresponding to ethanol sensing mechanism. Fe doping with crystallite size is added which increases the magnitude of sensor. The orientation in desired manner and crystallinity is improved by Fe stimulator. Exposure of Fe: films in air, partly transforms oxygen molecules into chemisorbed oxygen species by capturing



Fig. 3. Gas sensor response of (a) WO₃ and (b) Fe doped WO₃ thin films by spray pyrolysis technique.

electrons from conduction band which leads to high resistance in air. The formation of depletion region on the surface is due to negatively charged oxygen ions and positively charged W ions. The exposure of sensors to ethanol leads to reaction between ethanol molecules and oxygen species which compensates the electrons released from oxygen ions⁸. The original value retracts back, due to the reduction of resistance re-absorption of adsorbed oxygen species takes place. Better reproducibility and baseline stability are observed due to smooth crystal structure and adherent nanostructures. The sensing performance of films is much higher than pristine. Output response of the sensor is improved by Fe catalyst. The lattice oxygen which enhances the sensing properties of is promoted by the interaction between and Fe components of the sensor. The gas sensing mechanism is surface controlled. Contrast to WO_3 , Fe doped WO_3 has more sensor response. The response increases with increasing concentration of ethanol gas which indicates that the range of interaction of gas molecules is high.

Conclusion

The development of highly oriented and Fe activated films is accomplished and XRD analysis proves the formation of (200) plane with enhanced crystal quality. Compared to pristine Fe activated shows a red shift in absorption edge and a decrease in band gap due to quantum size effect. The nucleation of islands also reveals promising results with improved sensing response towards ethanol. Also FE doped sensor shows fast recovery towards ethanol at room temperature which promises the use of ethanol as sensors in practice.

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