CHAPTER 6

Summary and Conclusion

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6.1 Introduction

The objective of the present thesis is to fabricate and characterize some TiO_2 and ZnO thin film based hydrogen gas sensors with special emphasis on the room-temperature or low-temperature sensing characteristics. Two commonly used device structures, namely MSM and MOS, are fabricated. The sol-gel and physical vapor deposition methods are used for the thin film depositions. This chapter presents the chapter-wise major findings of the thesis as described in the following section.

6.2 Chapter-Wise Major Observations

Chapter-1 introduces the different types of sensors and the materials/techniques required to fabricate hydrogen sensors. Metal oxides and their importance in electronic devices for gas sensing applications are discussed. The working mechanism of metal oxides-based hydrogen sensors and the role of TiO_2 and ZnO thin films in hydrogen gas sensing are also discussed in this chapter. The advantages of nanostructures and noble metals in gas sensing are compared with conventional bulk materials. Various deposition techniques like thermal evaporation, electron beam evaporation, and spin coating, as well as different thin film characterization techniques such as AFM, SEM, and XRD have been briefly introduced. A detailed literature survey on TiO_2/ZnO metal oxide-based hydrogen gas sensor has been presented. Based on the research gaps observed from the literature survey, the scope of the thesis is outlined at the end of this chapter.

Chapter-2 reports the fabrication and characterization of a sol-gel derived TiO_2 film based MSM structure with Pd based interdigitated electrodes for hydrogen gas sensing applications. The sensor is fabricated on the SiO_2 coated Si substrates by depositing sol-gel derived TiO_2 thin film using the spin coating technique. The major observations and findings from the chapter are summarized below:

- The surface and structural morphologies are analysed by XRD, EDX, SEM, AFM confirm the suitability of the sol-gel derived TiO_2 film for electrical and gas sensing applications.
- The resistance of the film is decreased to enhance the value of current with the increased hydrogen concentration.
- The fabricated sensor shows a negligible hydrogen gas response at room temperature. However, the device gives the maximum gas response of 26.75% at 4% hydrogen at 175°C for 5 V of the applied voltage.
- At room or lower temperature O_2^{-1} is the principal species of chemisorbed oxygen, whereas chemisorbed O_2^{-1} donates an electron to TiO_2 at the higher temperatures which increase the conductivity of the sol-gel derived TiO_2 film.
- The hydrogen gas response of the device is dependent on the applied bias voltage, and the maximum gas response is observed for 5 V bias voltage.
- The sol-gel derived TiO_2 film-based MSM sensor's gas response is not enough to detect a low concentration of hydrogen at room temperature or low bias operation. Hence further improvement in gas response is required by film/device structure engineering.

Chapter-3 reports the investigation of the electrical and hydrogen sensing characteristics of the thermally evaporated TiO_2 thin film based MOS device. The major finding of this chapter can be summarized as follows:

- Thermally grown nanostructured TiO_2 based Pd/TiO_2/Si/Al-based MOS sensor provides better hydrogen gas response over MSM sensor discussed in Chapter-2. The maximum hydrogen gas response at RT is 84% using conductance, whereas 65% using capacitance (which is much larger than 26.75% at 4% hydrogen at 175°C of the MSM sensor) when 4% hydrogen gas is exposed.
- The maximum shift in the capacitance and the conductance are observed around 0 V for 50 nm thin TiO_2 oxide layer. The reduction in the capacitance and the conductance is found for the increasing exposer of hydrogen gas.
- The obtained gas response is high enough for practical application at room temperature operating conditions. However, a very low capacitance value is obtained due to 50 nm thin TiO_2 film. Hence further improvement is required by film/device engineering.

Chapter-4 investigates the electrical and hydrogen gas sensing characteristics of electron beam evaporated (EBE) TiO_2 film based MOS device. The performance of the EBE deposited TiO_2 film-based sensor has been compared with that of the spin-coated sol-gel derived TiO_2 film based MSM structure sensor and thermally evaporated TiO_2 film based MOS structure sensor. The major observations are summarized below:

- The EBE grown TiO_2 film based Pd/TiO_2/Si MOS sensor with 10 nm thin oxide layer provides better hydrogen gas response over MSM sensor discussed in Chapter-2 and thermally evaporated TiO_2 film based MOS sensor discussed in Chapter-3.

- The capacitance measurements show the maximum room-temperature sensitivities of 51% and 64% in ambient air and nitrogen gas for 4% H_2 gas concentration, respectively. However, the conductance measurements give the maximum room-temperature sensitivities of 84% in ambient air and 90% in nitrogen gas. The performance parameters are observed to be better than those of thermally evaporated TiO_2 film based MOS sensor considered in Chapter-3.
- The measured values of the response and recovery times for 4% hydrogen gas in an N_2 environment are ~52 s and 48 s, respectively.
- The fabricated sensor is less sensitive to the organic vapors such as acetone, chloroform, ethanol, isopropanol, and methanol due to selective response in electron beam deposited TiO_2 and thermally evaporated Pd films.
- All the performance parameters of the EBE TiO_2 film-based MOS hydrogen sensor are observed to be superior to those of the spin-coated sol-gel derived TiO_2 film based MSM sensor and thermally evaporated TiO_2 film based MOS sensor considered in Chapter-2 and Chapter-3, respectively.

Chapter-5 investigates the electrical and hydrogen gas sensing properties of colloidal ZnO quantum dots (QDs) based MSM structure with interdigitated electrodes. The colloidal ZnO QDs are synthesized by low-cost hot-injection method. The colllidal ZnO QDs are deposited on a SiO_2 coated p-Si substrate by spin-coating method. The major observations of this chapter are listed below:

- The XRD and EDS are used to confirm the presence of ZnO whereas the SEM and AFM analyses are used to analyze the surface morphology of the ZnO QDs thin film.
- The proposed MSM sensor shows a negligible hydrogen sensitivity at room temperature. However, it gives the maximum sensitivity of 83.2% for 4% hydrogen at 175ºC. The values of response and recovery are calculated as ~27 s and ~56 s, respectively under the exposure of 4% H_2 gas.
- The proposed ZnO QDs based MSM sensor also shows a very good H_2 selectivity with respect to other interference gases, namely ammonia, sulfur dioxide, and various organic vapors. The hydrogen response of the proposed sensor is found to be nearly independent of the applied bias voltage.

We have fabricated 10 set of each type of sensor devices considered in this thesis. The maximum percentage deviation in the responses of various devices of a particular sensor type was found within ±2%. This shows the repeatability and reusability of our fabricated devices. Further, the fabrications and characterizations of all the sensors studied in this thesis were carried out in the open environment conditions. The encouraging results confirm that the proposed sensors investigated in the present thesis can be reliably used in harsh environments. It is also important to mention that our measurement techniques were calibrated by using pre-characterized commercially available gas sensors. We have observed that the measured results are highly accurate with a slight variation of less than ±1%.

6.3 Future Scope of Work

We will now propose some future scopes of work related to the present thesis. They are listed below:

- Other TiO_2 nanostructures such as QDs, NPs, Nanowires can be used in the MSM/MOS structures for hydrogen sensing.
- Nanocomposite films of ZnO and TiO_2 can be explored for hydrogen sensing applications.
- Multi-layered noble metal can be used on the TiO_2 and ZnO for gas sensing application.
- The metal doping in TiO_2 and ZnO can be used to improve the hydrogen gas response.
- The nanomaterials-based gas sensor can be fabricated on a flexible substrate for wearable and smart sensor technology applications.