

Preface

In recent years, intense efforts have been made towards the development of miniaturized gas-sensing devices for hazardous gas detection. Carbon monoxide, which is a result of incomplete combustion, is a colourless, odourless, poisonous, explosive and highly toxic gas. It binds with haemoglobin to form carboxyhemoglobin, which reduces the oxygen-carrying capacity of blood and, finally, leads to death. Hence, there is a need to develop a portable and cost-effective gas sensor to detect low concentrations of CO gas. MEMS-based metal oxide semiconductor gas sensors offer several advantages compared to conventional optical and electrochemical techniques, such as compact size, low power consumption, quick response, high-temperature stability and low cost for mass production. However, a high-temperature is required for the optimum performance of metal oxide semiconducting gas sensors and such temperatures can be achieved with microheaters.

The present thesis work deals with the design, fabrication and characterization of a MEMS gas sensor for the detection of low concentrations of carbon monoxide gas. The work is focused on five specific objectives: a) growth and characterization of sensing film; b) sensitivity enhancement using noble metal additives and nanowire structures; c) design, fabrication and characterization of microheaters; d) development of microhotplate integrated MEMS gas sensor; e) gas sensor packaging. The investigations undertaken are as follows:

Titanium dioxide thin film sensing material is deposited using DC magnetron sputtering. The deposition conditions are optimized to obtain stoichiometric TiO₂ thin films and are characterized for carbon monoxide gas detection. The influence of the operating temperature, annealing temperature, thin film thickness and the interdigitated electrode geometry on the sensor response is investigated. The TiO₂ thin film sensor (annealed at 800 °C) shows a high response (79.5 %) to CO gas and a low response (<23%) to other reducing and oxidizing gases at 400 °C. It is observed that the sorption is completely reversible and the response and recovery times are of the order of 50 and 120 sec respectively.

Noble metal additives such as Au, Pt, and Pd are decorated on the TiO₂ surface to enhance the sensitivity and selectivity of the TiO₂ thin film gas sensors. The pristine TiO₂ thin film gas sensor (annealed at 400 °C) is found to exhibit a detectable response only when the operating temperature is above 300 °C; below this temperature there is no detectable change in resistance in the presence of 5000 ppb of CO gas. The response is found to increase with the operating temperature and it exhibits a maximum response of 58.6 % at 400 °C. The

surface- modified TiO_2 thin film based gas sensor shows a remarkable response event at $100\text{ }^\circ\text{C}$. The Au-TiO_2 and Pt-TiO_2 thin film gas sensor has been found to exhibit a maximum response of 83.46 and 79.64 % at 200 and $250\text{ }^\circ\text{C}$ respectively, whereas, the Pd-TiO_2 thin film gas sensor exhibits a n to p transition above $150\text{ }^\circ\text{C}$.

TiO_2 nanowires are synthesized using hydrothermal processes and are characterized for the detection of low concentrations of carbon monoxide gas. The TiO_2 nanowire-based gas sensor shows a detectable change in its resistance even at $150\text{ }^\circ\text{C}$ and its response is found to increase with the temperature. At $400\text{ }^\circ\text{C}$, the sensor is found to exhibit a maximum response of 80 % for 5000 ppb of CO. Further, under the same operating conditions, the sensor is found to exhibit a remarkable change in its resistance (a response of 5.6 %) for 100 ppb of CO. The response and recovery times of the sensor are of the order of 18 and 27 s respectively.

Microheaters are designed and simulated using the CoventorWare MEMS design and analysis tool to optimize the geometry of the microheater structure for uniform temperature distribution and low power consumption. Low resistive molybdenum thin films are deposited for high temperature microheater applications. The molybdenum microheaters are fabricated and their electro-thermo-mechanical characteristics are studied. The microheater membrane stability is analyzed by measuring its deformation under high thermal stresses using an optical profilometer. The microheater response is characterized in both pulsed and constant temperature modes of operation; it is found to exhibit a negligible resistance drift after 600 hours of continuous operation, indicating its long-term thermal and mechanical stability. The response and recovery times are in the order of a few milliseconds (19 and 34 ms), which make them suitable for gas-sensing applications.

Finally, microhotplate integrated MEMS gas sensors are fabricated, packaged and characterized for carbon monoxide gas detection at elevated temperatures (250 to $700\text{ }^\circ\text{C}$). The sensitivity, selectivity, repeatability and response and recovery times of the miniaturized MEMS gas sensors are investigated. The developed MEMS gas sensor is found to exhibit a high sensitivity and selectivity to CO gas compared to the TiO_2 thin film based gas sensor. It shows a highest response of 96.14 % for 5000 ppb of CO and a minimum response ($< 28\%$) to other reducing and oxidizing gases at $550\text{ }^\circ\text{C}$ optimized temperatures. The MEMS gas sensor is found to exhibit quick response and recovery times (9 and 21 s) compared to thin film-based gas sensors (50 and 120 s).