CONTENTS

List of Figur	es xv	vii-xix
List of Table.	S	xxi
List of Abbre	eviations xxi	iii-xxv
List of Symbo	ols xxvii-	-xxviii
Preface	xxi	ix-xxx
CHAPTER	1	1-34
Introducti	on and Scope of the Thesis	
1.1	Introduction	3
1.2	Sensing Materials and Working Mechanism for Hydrogen Sensors	5
1.2.1	Metal Oxides for Hydrogen Sensor	6
1.2.1.1	Role of TiO ₂ in Hydrogen Gas Sensing	8
1.2.1.2	Role of ZnO in Hydrogen Gas Sensing	9
1.2.2	Nanostructured Materials for Hydrogen Sensor	10
1.2.2.1	Quantum Dots	12
1.2.2.2	Nanostructured Noble Metals	13
1.2.3	Working Mechanism of Hydrogen Sensor	13
1.2.3.1	Room Temperature Based Hydrogen Sensor	14
1.2.3.2	Temperature Based Hydrogen Sensor	15
1.3	Fabrication Techniques for Thin Film Devices	16
1.3.1	Thermal Evaporation	17
1.3.2	Electron Beam Evaporation	18
1.3.3	Spin Coating	19
1.4	Characterization Techniques for Thin Films and Devices	21
1.4.1	Atomic Force Microscopy	21
1.4.2	Scanning Electron Microscopy	22

1.4.3	X-Ray Diffraction	23
1.4.4	Electrical Characterization Techniques	24
1.5	Literature Review	25
1.5.1	Review of TiO ₂ Based Hydrogen Sensors	25
1.5.2	Review of ZnO Based Hydrogen Sensors	27
1.5.3	Major Observation from the Literature Survey	29
1.5.4	Challenges in Metal Oxide Based hydrogen Sensors	30
1.6	Motivation and Problem Definition	31
1.7	Scope of the Thesis	32
CHAPTE	R 2	35-50
	n Sensing Properties of Sol-Gel Derived TiO ₂ Thin	
Interdigi	tated MSM Device	
2.1	Introduction	37
2.2	Experimental Details	38
2.2.1	Synthesis of Sol-Gel Solution of TiO ₂	38
2.2.2	Substrate Cleaning	40
2.2.3	Device Fabrication	40
2.3	Results and Discussion	41
2.3.1	Thin Film Characterization	42
2.3.2	Electrical and Gas Sensing Characterization	45
2.4	Conclusion	49
CHAPTE	R 3	51-63
Room-To	emperature Hydrogen Sensing Properties of Therm	ally
Evapora	ted TiO ₂ Thin Film Based MOS Device	
3.1	Introduction	53
3.2	Experimental Details	54

3.2.1	Thin film Deposition and MOS Fabrication	54
3.3	Results and Discussion	55
3.3.1	Thin Film Characterization	56
3.3.2	Electrical Characterization	58
3.3.3	Gas Sensing Characterization	59
3.4	Conclusion	63
CHAPTEI	R 4	65-80
Room-Te	emperature Hydrogen Sensing Properties of Ele	ctron Beam
Evapora	ted TiO ₂ Thin Film Based MOS Device	
4.1	Introduction	67
4.2	Experimental Details	68
4.2.1	Materials and Sensor Fabrication	68
4.2.2	Film and Sensor Characterization	69
4.3	Results and Discussion	70
4.3.1	Thin Film Surface Characterization	70
4.3.2	Electrical Characterization of MOS Sensor	72
4.3.3	Hydrogen Sensing of MOS Sensor	73
4.3.4	Sensitivity of MOS Gas Sensor	75
4.3.5	Selectivity of MOS Sensor	78
4.4	Conclusion	80
CHAPTEI	R 5	81-96
Hydroge	n Sensing Properties of Colloidal ZnO Quantum	n Dots Thin
Film Bas	sed Interdigitated MSM Device	
5.1	Introduction	83
5.2	Experimental Details	84
5.2.1	Synthesis of ZnO QDs	84

		Content
5.2.2	Thin Film Deposition and Device Fabrication	85
5.3	Results and Discussion	86
5.3.1	Thin Film Characterization	87
5.3.2	Electrical Characterization of MSM Sensor	89
5.3.3	Gas Sensing Characterization of MSM Sensor	91
5.4	Conclusion	96
СНАРТЕ	R 6	97-104
Summer	y and Conclusion	
6.1	Introduction	99
6.2	Chapter-Wise Major Observations	99
6.3	Future Scope of Work	103
References	S	105-120
Author's Relevant Publications		121-122

LIST OF FIGURES

Figure 1.1:	Unit cell of the structures (a) rutile (b) anatase, and (c) brookite.	8
Figure 1.2:	Unit cell of the structures (a) Wurtzite and (b) Zinc blend.	9
Figure 1.3:	Types of NMs based on the number of dimensions in the nanoscale regime.	11
Figure 1.4:	Illustration of the effective surface to volume ratio in nanostructure materials.	15
Figure 1.5:	General schematic diagram of thermal evaporation.	17
Figure 1.6:	Schematic diagram of electron beam evaporation.	19
Figure 1.7:	Spin-coating process steps (a) Spin coating unit (b) Sample loaded on the spin coating unit and (c) Coating on the sample and sample with the spin-coated film.	20
Figure 1.8:	Sol-gel process.	20
Figure 1.9:	Working process of AFM.	21
Figure 1.10:	Working process of SEM.	22
Figure 1.11:	Working process of XRD.	23
Figure 1.12:	Block diagram of gas sensing chamber	25
Figure 2.1:	A simplified flow chart to demonstrate the Sol-gel synthesis process.	39
Figure 2.2:	Synthesized Sol-gel Solution of TiO ₂ .	39
Figure 2.3:	Cross-sectional view of the as-fabricated sensor.	41
Figure 2.4:	XRD image of the TiO ₂ thin film on SiO ₂ /p-Si substrate.	43
Figure 2.5:	AFM image of the TiO ₂ thin film annealed at 450°C.	43
Figure 2.6:	SEM image of sol-gel derived TiO_2 thin film on SiO_2 / p-Si substrate annealed at 450°C.	44
Figure 2.7:	EDS image of sol-gel derived TiO_2 thin film on SiO_2 / p-Si substrate.	45
Figure 2.8:	<i>IV</i> characteristic of fabricated sensor for 0% and 1% hydrogen gas under the ambient condition at room temperature.	46

Figure 2.9:	IV characteristic at different temperatures without hydrogen exposure condition.	47
Figure 2.10:	IV characteristic at different temperature underexposure of 1% H_2 gas.	47
Figure 2.11:	IV characteristic at different H ₂ concentration and fixed temperature of 175°C.	48
Figure 2.12:	(a) Gas response Vs H_2 concentration plot at bias voltage of 5 V and temperature of 175°C and (b) gas response Vs H_2 concentration plot at different voltage and fixed temperature of 175°C.	49
Figure 3.1:	(a) Device structure of fabricated MOS sensor and the camera image of the top surface of the final film. (b) Camera (real) image of fabricated MOS devices on the p-Si substrate packaged in a plastic case.	55
Figure 3.2:	AFM image of 50 nm thin TiO ₂ film on the p-Si substrate.	56
Figure 3.3:	SEM image of TiO ₂ film deposited on the p-Si substrate.	57
Figure 3.4:	EDS image of TiO ₂ thin film on the p-Si substrate.	58
Figure 3.5:	C-V characteristics at 1 MHz frequency for different concentrations of H_2 .	59
Figure 3.6:	G-V characteristics at 1 MHz frequency for different concentrations of H_2 .	59
Figure 3.7:	Sensitivity vs. H ₂ concentration.	61
Figure 3.8:	Gas response using capacitance and conductance for 4% concentration of H_2 .	62
Figure 4.1:	(a) Device structure of the MOS sensor and (b) Packaged MOS sensor.	69
Figure 4.2:	AFM image of (a) TiO_2 film on p-Si and (b) Pd dots on p-Si/ TiO_2 film.	71
Figure 4.3:	SEM image of Pd dot on TiO ₂ film deposited on a silicon substrate.	71
Figure 4.4:	EDS image of Pd dot on TiO ₂ film deposited on silicon substrate, while inset shows position of mapping.	72
Figure 4.5:	(a) <i>C-V</i> and (b) <i>G-V</i> characteristics of MOS sensor at a different frequency.	73

Figure 4.6:	(a) <i>C-V</i> characteristics and (b) <i>G-V</i> characteristics at 1 MHz frequency of MOS sensor for the various concentration of H ₂ gas.	73
Figure 4.7:	(a) Capacitance and conductance vs. H_2 concentration and (b) Sensitivity vs. H_2 concentration.	76
Figure 4.8:	Sensitivity vs. applied voltage at 4% concentration of H ₂ gas in ambient air and nitrogen atmosphere calculated from (a) capacitance and (b) conductance.	77
Figure 4.9:	Capacitance vs. relative humidity for 0% and 4% H_2 at an applied voltage of -1 V.	78
Figure 4.10:	Selectivity characteristics of the hydrogen sensor in the presence of common interferences.	79
Figure 5.1:	Flowchart of ZnO QD synthesis under the inert environment.	85
Figure 5.2:	Synthesized ZnO QDs.	85
Figure 5.3:	The cross-sectional device structure of as-fabricated ZnO QDs based MSM sensor.	86
Figure 5.4:	XRD image of the ZnO QDs thin film on SiO ₂ /p-Si substrate.	87
Figure 5.5:	EDS image of ZnO QDs deposited on the silicon substrate.	88
Figure 5.6:	SEM image of ZnO QDs thin film on Si/SiO_2 substrate annealed at $450^{\circ}C$.	88
Figure 5.7:	AFM image of the ZnO QDs thin film annealed at 450°C.	89
Figure 5.8:	Current-voltage characteristic at different temperatures under unexposed conditions. The stability of the MSM sensor in an open-air atmosphere at 175°C and 5 V bias voltage in the inset.	90
Figure 5.9:	Current-voltage characteristic at different temperature and exposure of $1\%~H_2$ gas.	91
Figure 5.10:	Current-voltage characteristic at different H ₂ concentration and fixed temperature of 175°C.	92
Figure 5.11:	Gas response - H ₂ concentration plot at different bias voltage and temperature of 175°C. Inset shows the response-recovery characteristics for H ₂ gas at an applied bias of 1 V.	94
Figure 5.12:	Gas response of the hydrogen gas sensor over common interference gases and organic vapors at a temperature of 175°C.	95

LIST OF TABLES

Table 1.1:	Comparison of the various dimensions of NMs.	11
Table 2.1:	Surface parameters for Sol-Gel derived TiO ₂ films.	43
Table 2.2:	Details of respective elements present in the fabricated device.	45
Table 3.1:	Deposition rate for the thermal evaporation.	54
Table 3.2:	Surface parameters for deposited TiO ₂ films.	56
Table 3.3:	Comparison of TiO ₂ based H ₂ sensor.	63
Table 4.1:	Surface parameters for deposited TiO ₂ /Pd films.	70
Table 4.2:	Comparison of the Oxide Based MOS Capacitative Sensor for Hydrogen gas.	80