Nanocrystalline ZnO based MEMS Gas Sensors with CMOS ASIC for Mining Applications

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Abstract:

In this paper a nanocrystalline (nc) zinc oxide based hybrid gas sensor with signal conditioning ASIC has been reported for sensing and transmitting the information about methane concentration from the underground coalmine environment. A low power, low temperature nc zinc oxide MEMS based gas sensor has been designed, fabricated and tested for the purpose with a power consumption of ~70mW and sensitivity of 76.6 % at 1.0% methane concentration at a sensor operating temperature of 150^0C. For transmitting the output of the gas sensor, a voltage controlled oscillator (VCO) chip integrated with a low noise amplifier has been fabricated in 0.35μm CMOS technology to convert the voltage output of the gas sensor to desirable frequency. The power consumption of the chip has been obtained to be around 3mW. The amplifier gain is set suitably ~13 to apply the desirable control voltage (~1.2V-3.2V)to the VCO. The noise of the amplifier has been obtained to be around $2\mu V/Hz^{1/2}$. The output frequency of the VCO varies from 20kHz to 100kHz for the change in methane concentration from 0 to 1%. The output of the VCO chip can be applied as a modulating signal to a commercially available transceiver, which transmits the signal to the control room.

1. Introduction

Metal oxide gas sensors like zinc oxide and tin oxide have been used for various applications ranging from domestic to environmental monitoring and industrial applications [1-4]. These sensors which are mostly alumina substrate based, are commonly used for sensing inflammable hydrocarbon gases like methane [4] and other toxic gases like carbon monoxide [5]. However, they suffer from the principal limitations, of relatively high operating temperature (≥300° C) [6] and large power dissipation of around 0.5-1Watt [7]. Both these features are unacceptable for continuous gas monitoring in many environmental scenario such as underground coalmines. MEMS technology has been presently employed in sensor technology in miniaturization of the devices, low power consumption, faster response and greater sensitivity [8]. But these MEMS based sensors still are reported to operate at high temperature(≥ 300^0C).To achieve the lower operating temperature at a relatively low cost, a modified structure of sensor has been proposed employing nanocrystalline ZnO as the sensing material and nickel as microheater element instead of commonly used platinum or polysilicon and using sol-gel process for depositing nc zinc oxide [9] and silicon dioxide[10].

Also for hazardous environments like underground coalmines where continuous monitoring of hazardous gas concentration is required it is extremely desirable that the entire signal-processing unit capable of amplifying low signal level output from the sensor as well as transmitting the modified signal to remote control station, should be integrated along with the sensor platform [11,12].There are some reports on the development of integrated gas sensor systems with log- invertor circuits for linearising the output voltage[13]. Oscillator circuits have also been reported to be integrated with the gas sensor for conversion of voltage to frequency providing improved resolution [14].

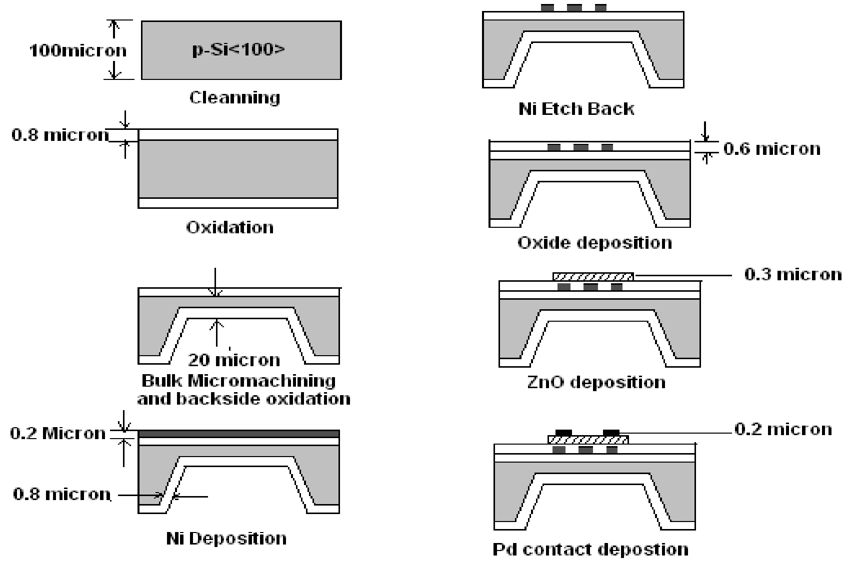
In this paper we also report the integration of the signal conditioning unit with the sensor output for transmission of the sensor data to control room. This has been achieved through coupling of the sensor output with an low noise amplifier integrated with VCO. The amplifier integrated VCO has been designed and fabricated in 0.35μm CMOS technology of Austria Microsystems. The amplifier has been designed to yield a low noise, good linearity and low gain using closed loop configuration of a two stage OpAmp. The VCO is a ring oscillator based configuration with tunable PMOS varactors, which provides improved sensitivity for low methane concentration. The output of the VCO is converted to a square wave through a zero crossing detector and can be applied as a modulating signal through buffer to a commercially available CHIPCON transceiver which transmits the signal at 2.4GHz center frequency.

2. Nanocrystalline ZnO based MEMS Gas Sensor Fabrication and Characterization

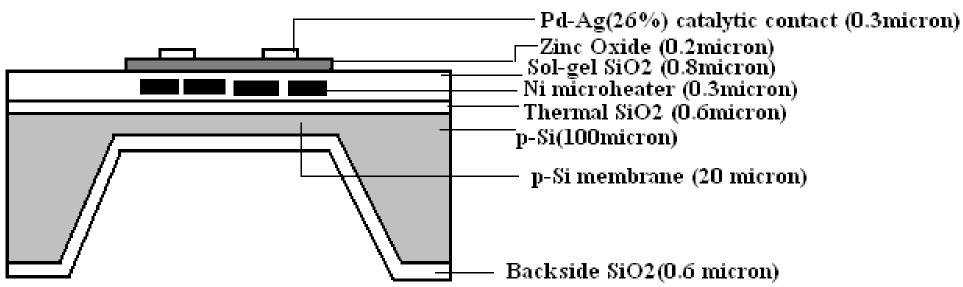
The flowchart for the fabrication of MEMS based nc zinc oxide gas sensor is shown in Fig.1.The starting wafer was p-Si <100> of resistivity 1Ω-cm (100μm thick) over which a thermal insulating SiO_2 layer (0.8μm) was grown by thermal oxidation. After opening window for micromachining by lithographic technique on the backside, bulk micromaching was carried out with EDP (Ehylene Diamine Pyrocatechol) solution at a temperature of 85°C, which results in a silicon membrane of 3mm by 3mm by 20μm dimensions.

A backside silicon oxide layer (0.8μm) is grown on the membrane to improve thermal isolation and reduce power dissipation. Nickel is used as a microheater element instead of platinum or polysilicon because of its relatively high resistivity, low cost, ease of fabrication and acceptable durability. Particularly, when the maximum desired temperature is around 100-150^{0}C, nickel film is good enough to act as the heating element.

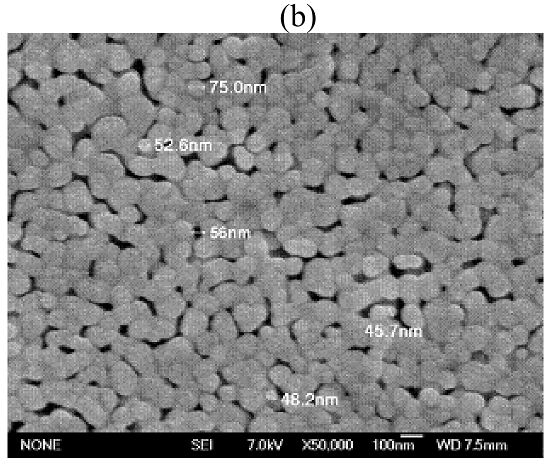
A 0.2 μm nickel layer was deposited on SiO_2 covered front side of the sample by e-beam (10^{-6} mbar) evaporation technique.The microheater was fabricated using conventional lithography followed by nickel etch back technique. A 0.6 μm SiO_2 layer, acting as an electrical isolation between the heater and the active layer, was then deposited on Ni microheater by sol-gel method. The active area was having a dimension of 1.3 mm × 1.3 mm at the center of the membrane. The total sensor area was 4 mm × 4 mm. The lines of meander shaped microheater were 50 μm wide and were separated also by 50 μm. The fabricated heater resistance was about 150 to 170Ω. The active ZnO layer was deposited by solgel method by spin coating technique.



(a)



(b)



(c)

Fig.1:(a) Process flow chart for the fabrication of nanocrystalline ZnO based micromachined methene sensor with embedded Ni microheater (b)Two-dimensional schematic view of nanocrystalline ZnO based MEMS methane sensor (not to scale)(c) SEM images of the nanocrystalline ZnO surface.

Finally the samples were annealed at 350^{o}C for 30 min for producing nanocrystalline ZnO. The entire process was repeated for three times and a ZnO film of ~900 nm thickness with the particle size ranging from 45 nm to 75 nm and average pore diameter of ~56 nm was produced (Fig. 1(c)). Pd-Ag (26%) catalytic contact was deposited on ZnO by an e-beam deposition method (10^{-6} mbar) using Al metal masks. A two dimensional schematic drawing of sensor structure fabricated is shown in Fig. 1(b)

For sensor study high purity (100%) methane gas and high purity (99.99%) N_2 in desired proportions were allowed to flow to the gas-sensing chamber through a mixing path via an Alicat Scientific mass flow controller and a mass flow meter for keeping the mass flow rate and thus the concentration of the methane gas constant throughout the experiments. The gas pressure over the sensor device was 1 atm during the experiments. The resistance of the sensors in the presence and absence of CH_4 was measured by a Keithley 6487 picoammeter/voltage source.

The variation of sensor resistance at an operating temperature of 150^{0}C with different concentration of methane in the gas is shown in Fig.2. The response magnitude S, is expressed in terms of sensor resistance in air (R_a) and in test gas (R_g) as follows

$S=(R_a - R_g) / R_a$

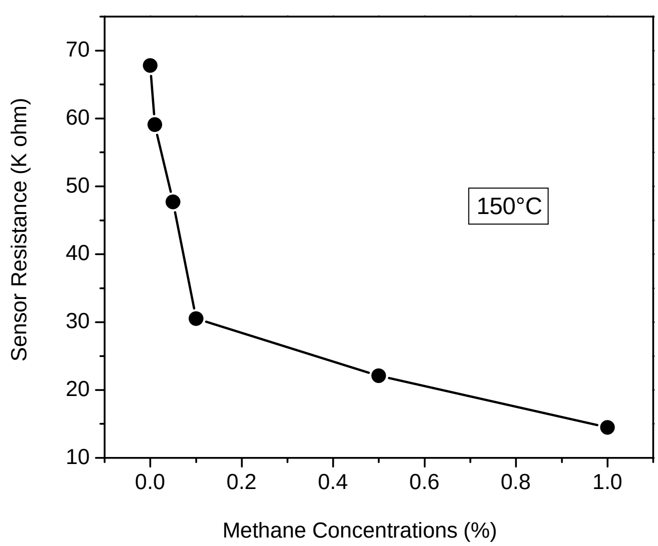


Fig.2: Sensor resistance as a function of methane concentrations at 150°C

The corresponding change in sensor resistance with respect to the gas (methane) concentrations is shown in fig2.

3. Integration with signal conditioning unit for transmission

The schematic of the signal conditioning system for transmission of gas sensor output from coalmine environment to control room is shown in Fig.3. The gas sensor is driven by a constant current source [15] of about 3μA implemented using standard IC chip to obtain a voltage in the range of 40mV-200mV for a change in methane concentration from 0 to 1%. The output of the gas sensor is applied to the input of a low noise amplifier, through a level shifter, which provides the control voltage to the VCO in the range of 1.2V to 3.2V. The VCO converts the voltage to frequency in the range suitable to be applied as a modulating signal to the transceiver chip for transmission of the signal. The following sections discuss the design and implementation of the amplifier, VCO and the transceiver blocks.

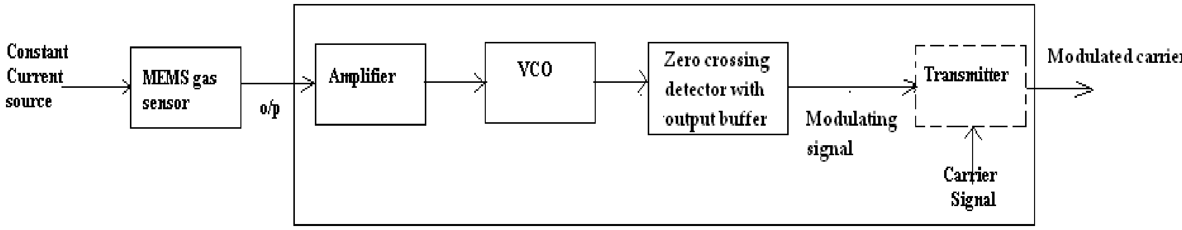


Fig 3. Block diagram of the MEMS sensor system

3.1 Design and Simulation of low noise amplifier

The output voltage of the MEMS gas sensor is the input to the low noise non-inverting amplifier through a level shifter. The level shifter circuit helps to provide dc bias of 1.2V to the input terminals of the amplifier [16] IN- and IN+ as shown in Fig.4. A two stage op-amp in the closed loop non-inverting configuration shown in Fig.4. has been selected for gain stability and better linearity. The desired gain has been adjusted to about 13 through the proper selection of feedback and input resistance

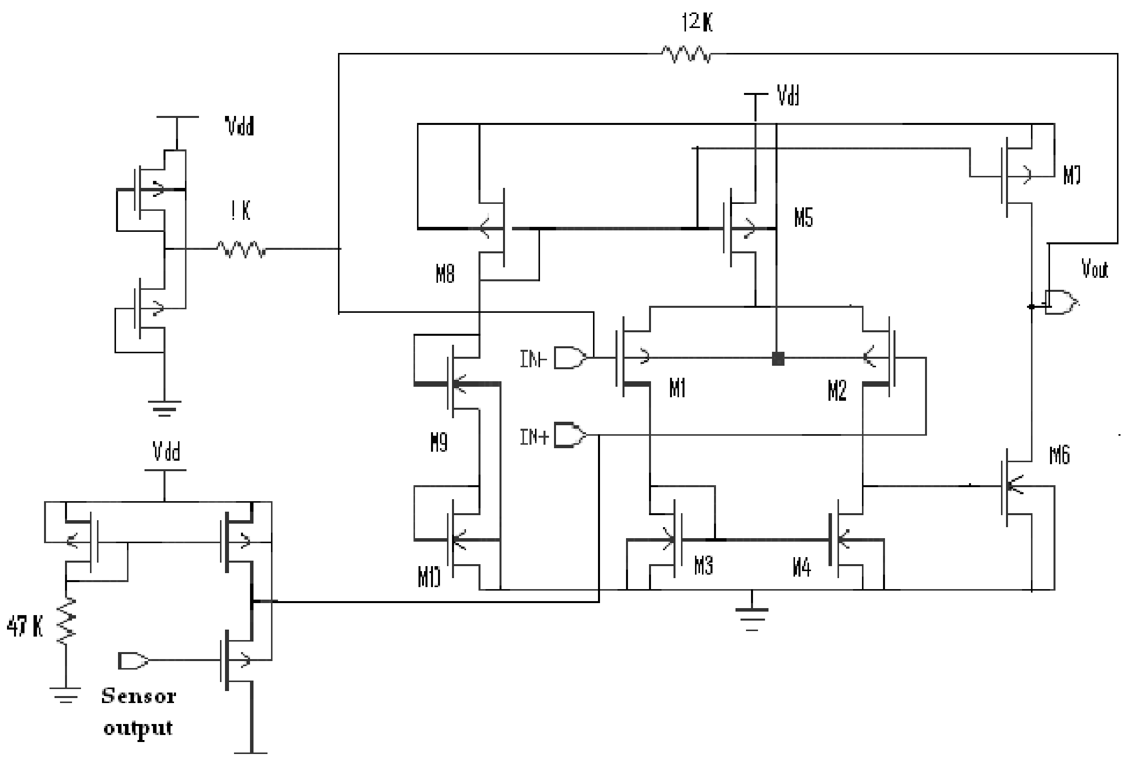


Fig.4: Closed loop operational amplifier

To reduce the equivalent noise of the open loop op-amp, the inputs are applied to PMOS transistors. The primary component of the noise at low frequency operation is the flicker noise and PMOS transistor has lower flicker noise coefficient compared to NMOS transistor [17]. The equivalent noise spectral density at the input terminal of the open loop operational amplifier is expressed as equation1 [17]:

$$e_{eq}^{2} = 2 * e_{n}^{2} * \left[1 + \frac{K_N B_N}{K_P B_P} \left(\frac{L_1}{L_3} \right)^2 \right] \qquad (1)$$

where e_n^2 is the noise voltage of the individual transistors, K_N and K_P are the transconductance parameters of NMOS and PMOS transistors respectively, B_N and B_P are the noise coefficients, L_1 and L_3 are the lengths of transistors M1 and M3 respectively. Further selecting the ratio of L_1 and L_3 much less than one gives an equivalent input spectral noise density of $2*e_n^2$. The noise voltage of the open loop operational amplifier has been obtained to be 2µV/ $Hz^{1/2}$.

The gain of the open loop operational amplifier has been obtained as 75dB. To obtain a stable and linear gain for sensing applications, the operational amplifier is used in a closed loop configuration through the 12K feedback resistor. The gain of the amplifier is around 13 as shown in Fig.5. The design has been simulated using 0.35µm CMOS technology of Austria Microsystems yielding an ICMR of 1-2.5V and a power consumption of 0.6mW.The passive resistors have been implemented using polysilicon resistors.

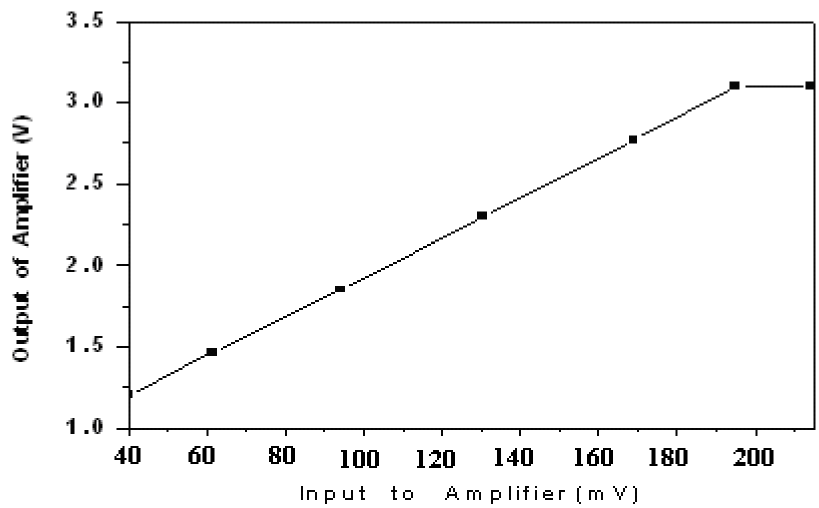


Fig 5: Output of amplifier

3.2 Design and Simulation of VCO

The output of the amplifier is the input to the VCO as the tuning voltage. The primary design needs of the VCO are: (a)low power consumption (b) wide tuning range from 1.2V to 3.2V since the amplifier output varies in this range for the input gas concentration (c) a frequency tuning from 20kHz to 100kHz to meet the desired resolution of detecting 0.01% gas concentration for concentration less than 0.1%.

The maximum frequency limit of the VCO is to be maintained lower than 125kHz since the interfacing transmitter chip CHIPCON 2500[18] has allowable data rate of 250kbps for binary frequency shift keying modulation scheme.
To achieve the above-mentioned characteristics, a three-stage differential ring VCO with frequency dependent PMOS capacitors have been used. The schematic of the ring oscillator is shown in Fig.6.Usually in a ring oscillator based MOS VCO, MOS resistors are employed as voltage dependent tunable elements but in this case, PMOS varactors have been used since they yield greater sensitivity compared to tunable resistors as explained below:
For the circuit to oscillate, the frequency dependent phase shift of each stage should contribute to an overall phase shift of 180^0. The oscillation frequency is given by equation (2).:

$$f = \sqrt{3}/(2\pi R C_p) \quad (2)$$

where R is the equivalent of the output impedance of the NMOS amplifier, PMOS resistor and the parasitic resistance of C_p where C_p is the equivalent capacitance of the PMOS varactor.
The variation of capacitance with applied voltage is given by:

$$C_p = k(V_\gamma + V_{DD} - V_C)^{\frac{-1}{2}} \quad (3)$$

where k depends on the dielectric constant of silicon and doping concentration of the substrate, V_γ is the built-in potential of the drain source-substrate junction, V_{DD} is the supply voltage and V_C is the control voltage at the shorted drain source terminal.

From equations 2 and 3 we obtain the frequency sensitivity as:

$$\Delta f / f \Delta V_c = -(1/2)(V_\gamma + V_{DD} - V_C)^{-1} \quad (4)$$

From equation 4 we observe that for higher values of control voltage, the frequency sensitivity is more unlike that of tunable resistors [19, 20] which is an advantage for sensing lower concentration of methane. This justifies the application of PMOS capacitor for frequency tuning.

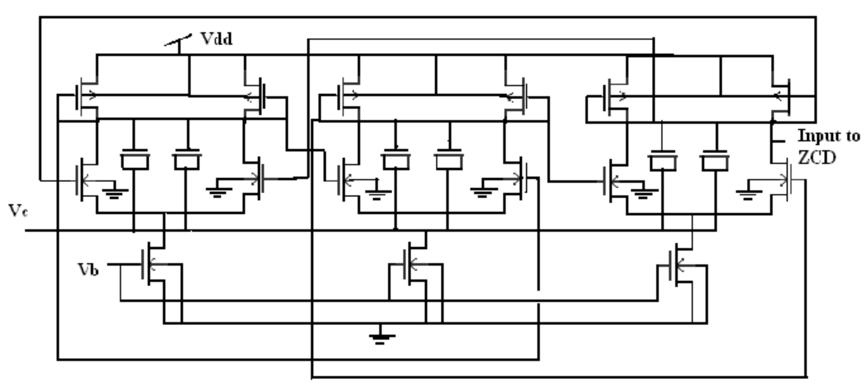


Fig. 6 Schematic of the VCO

The MOS varactor has been realized using a PMOS transistor with the drain and source terminals shorted. The bulk is connected to *Vdd* and the gate terminal is connected to the drain of the MOS amplifier.
The output frequency of the VCO varies from 20kHz to 100kHz for control voltage variation from 1.2V to 3.2V a shown in Fig.7. The sensitivity obtained is 40kHz/V.To obtain a resolution of 0.01% methane concentration detection for concentration less than 0.1%, the sensor output changes by about 12mV which causes the amplifier output to change by 156mV resulting in a frequency change at the output of the VCO by 5kHz. This change in frequency is detectable since the simulated phase noise performance of the VCO is obtained as -95dBc/Hz at 60Hz offset frequency from the center frequency of 60kHz.

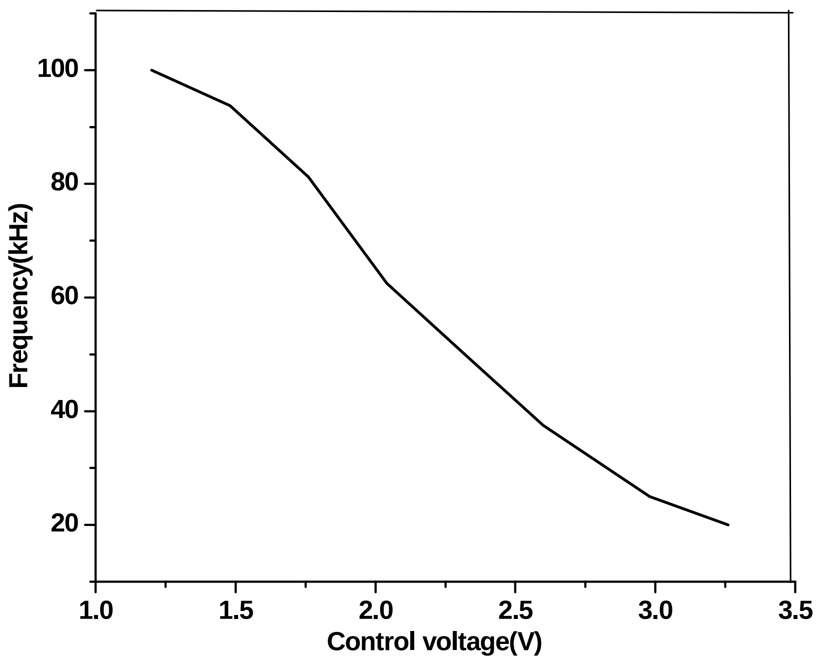


Fig.7 Change in frequency with control voltage

To obtain the output of the VCO from the external pin, the sine wave is converted to a square wave by a zero crossing detector which is followed by a buffer circuit to drive the output pin capacitance.

3.2.1 Zero Crossing Detector and output buffer

The zero crossing detector is essentially an operational amplifier [17] as shown in Fig.8a which converts the sinusoidal output of the VCO to a square wave. The amplifier has been designed as a comparator with two differential amplifiers and a cascode amplifier. To shape the square waves further, invertors are used which also act as output buffer. A three stage invertor is used to drive the output load capacitance of the pins a shown in Fig.8b.The power consumption of the entire VCO, zero crossing detector and the output buffer is 2.5mW in 0.35µm Austria Microsystems technology.

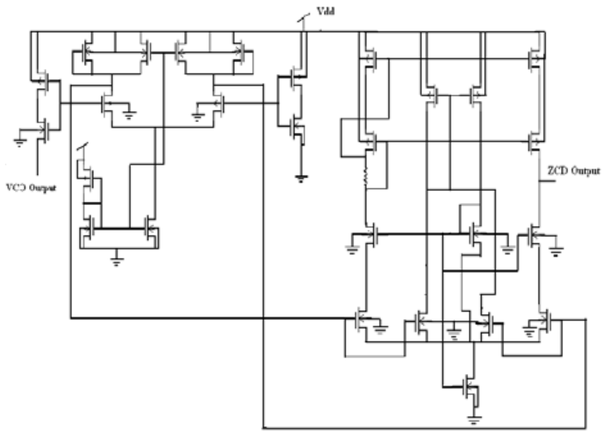


Fig.8(a) Zero Crossing Detector

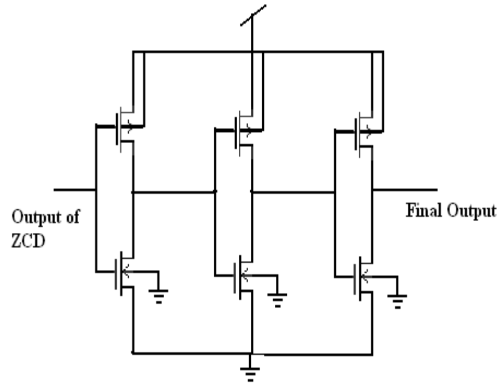


Fig. 8(b) Output buffer stage

3.3 Results of the amplifier integrated VCO chip

The overall schematic of the integrated chip is shown in Fig.9. An ASIC chip has been fabricated in Austria Microsystems 0.35µm technology and the chip tapeout is shown in Fig.10.

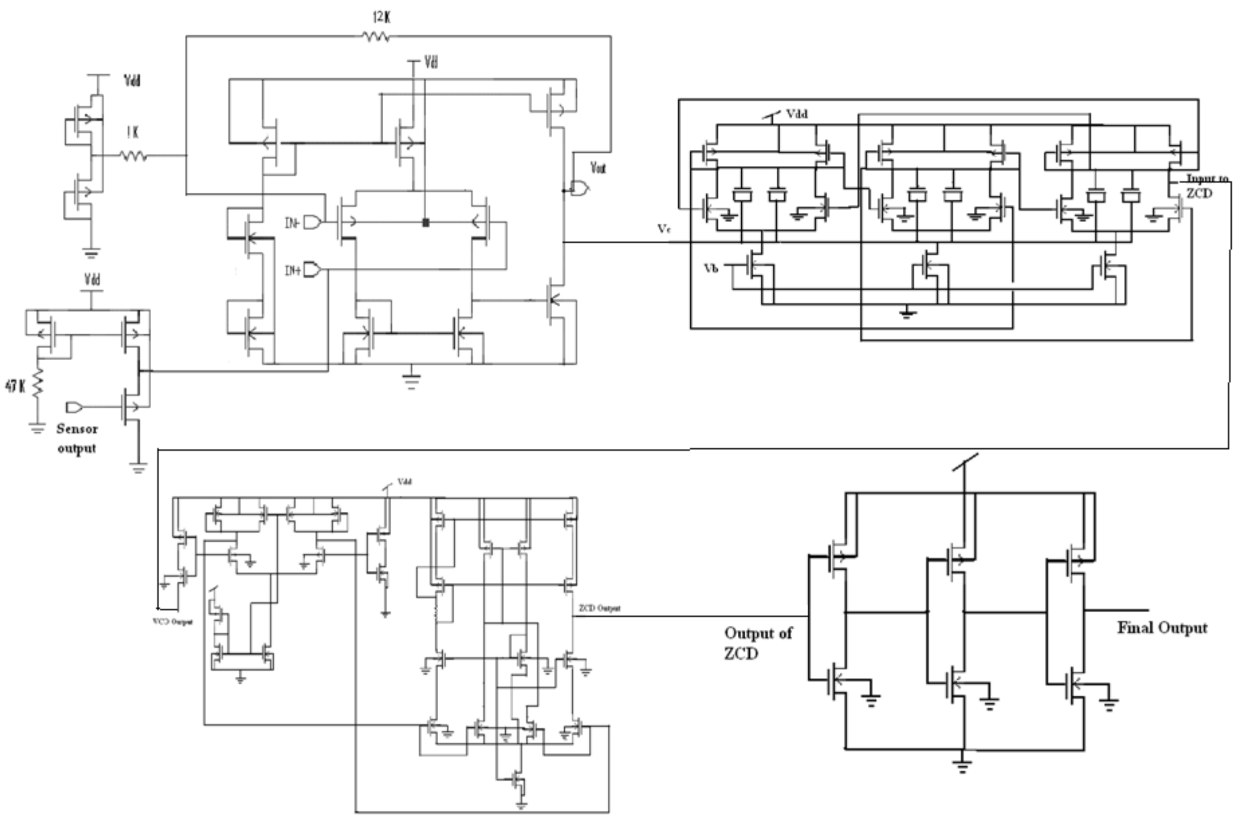


Fig. 9 Schematic of the total chip

The square wave obtained at the output pin for an input voltage of 70mV to the amplifier is shown in Fig.11a. The sensor output is provided at the input pin of the chip and the ultimate change in frequency with gas concentration is plotted in Fig 11b. It is observed that the frequency increases with increased methane concentration. This can be attributed to the fact that the resistance of gas sensor decreases with increased methane concentration leading to a decrease in control voltage which increases the oscillation frequency. Also the change in the frequency is more for lower methane concentration from 0 to 0.3% which helps to achieve a resolution of 0.01% methane concentration detection. The frequency of the VCO can be selected appropriately for detecting gases by using suitable sensor array.

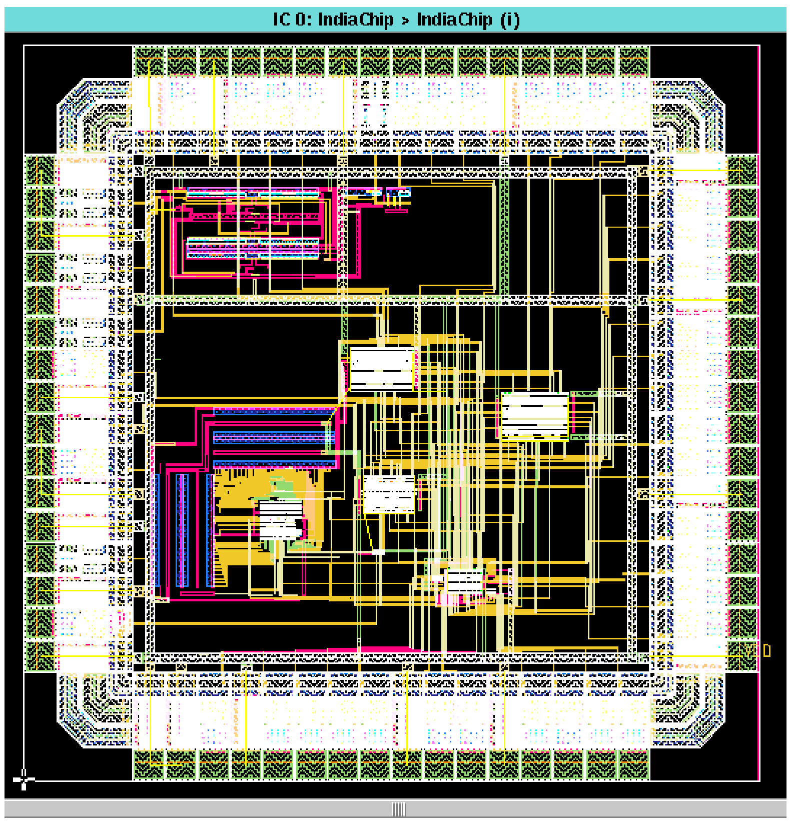


Fig.10 Chip tapeout of the amplifier integrated VCO

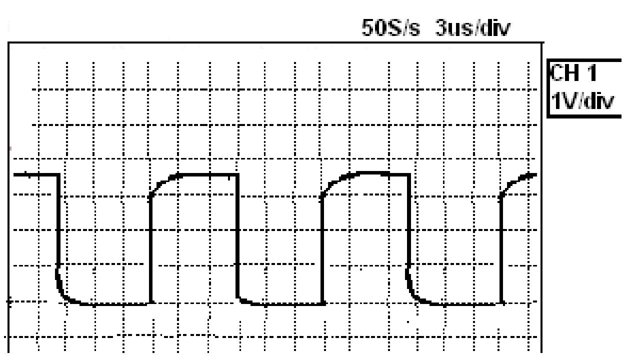


Fig.11(a) Square wave output of the ASIC chip for an input voltage of 70mV

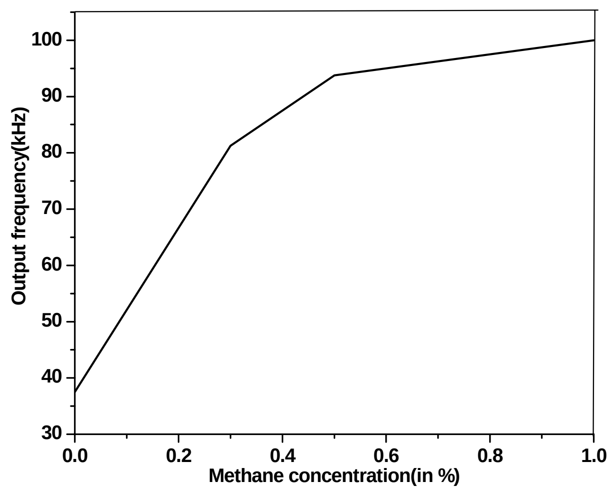


Fig.11(b) Variation of frequency with methane concentration at the output of the chip

For transmission, the output of the ASIC chip can be interfaced with the CHIPCON 2.4GHz transceiver. It operates with a 3.5V supply which is compatible with the ASIC chip. The output buffer at the output of the ASIC chip helps to avoid any impedance matching problem. The output of the VCO is provided at the input of the microcontroller which is interfaced with the CHIPCON CC2500 transceiver [18]. The CC2500 has been programmed in the unbuffered transmit mode with BFSK scheme of modulation with NRZ format of data.

4. Conclusion

A silicon MEMS based low power (≈ 70mW), low temperature(≈150^0C) gas sensor with nc-ZnO as sensing layer and nickel as microheater for use as sensor nodes for environmental monitoring has been fabricated and characterized for methane concentration in the range of 0 to 1% with a resolution of 0.01% for gas concentration less than 0.1%. The change in the resistance of the microsensor is converted to change of voltage using a precision constant current source. For transmission of the sensor output, a simple ASIC chip comprising of a low noise amplifier and a VCO with a zero crossing detector has been designed and fabricated in 0.35μm Austria Microsystems Technology. The power consumption of the chip is around 3mW. The frequency of the VCO can be selected appropriately for detecting gases by using suitable sensor array. The output of the ASIC chip is coupled through the microcontroller to a transceiver chip which can transmit the signal from the underground mining environment to the control room. The proposed low power, low temperature sensor system offers a low cost solution for continuous monitoring of different hazardous gases in mining environment by an array of sensors.

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