A layered structure surface acoustic wave for oxygen sensing

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Abstract - A novel layered structure Surface Acoustic Wave (layered SAW) transducer has been employed for an oxygen sensing application. It is a $SiO_2(0.36\mu m)$/ST-cut quartz crystal transducer. The dominant mode propagating in the transducer is a combination of Rayleigh and Love modes. Such a structure has the advantage of confining acoustic wave energy to the top selective layer, which increases the sensitivity of the device. A TiO_2 thin film deposited by the sol-gel process has been used as the oxygen sensitive layer.

A. Introduction

Micro-sensors that use acoustic waves comprise a very versatile class of sensors. Because they are highly sensitive to surface perturbations, they show extensive applications as gas sensors [1]. In recent years, a new class of layered SAW transducers opened a new era in terms of enhancing the sensitivity of acoustic wave sensors in gas media [2],[3]. They show high sensitivity to any sort of boundary condition perturbation caused by adsorption of gas molecules to the selective layer. The present paper uses this advantage to realise an oxygen gas sensing system.

SAW gas sensors are based on the interaction of the gas molecules with a selective film, which is deposited on a piezoelectric substrate. The propagation characteristics of the electro-mechanical wave travelling along the surface of the sensor are dependent on this interaction. Small changes in the conductivity of the selective layer introduce a perturbation in the electromagnetic boundary condition on the surface of the device, which alters the resonant frequency of the system.

The key design issue of the layered SAW delay line sensor is the ability to launch and detect a single mode acoustic wave. The layered SAW transducer has been designed and fabricated to operate as a delay line in a closed loop system to form an oscillator (Fig. 1). The system design consists of two transducers; one as the sensor and the other as the reference.

The measured output signal is the difference between the frequencies of the reference and the sensor delay line oscillators. TiO_2 deposited on the surface of the sensor as the sensitive layer, adsorbs oxygen molecules and changes the conductivity of the surface [4]. This alters the electrical boundary conditions on the active area of the sensor. As a consequence the speed of propagation of the acoustic waves is changed. The change in speed is reflected in a shift in the operational frequency of the sensing delay line oscillator, a shift that is proportional to oxygen concentration.

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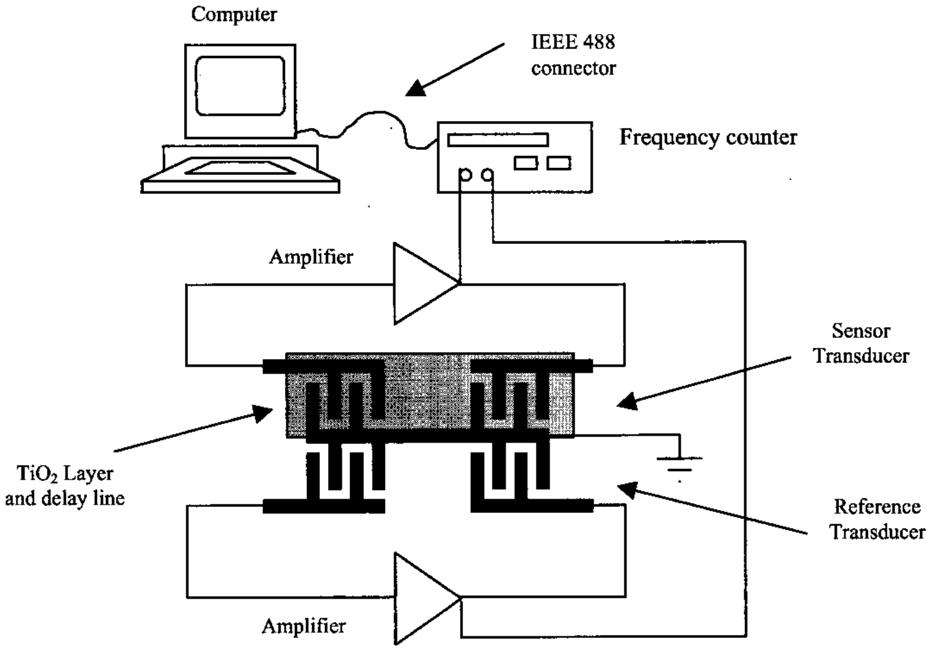
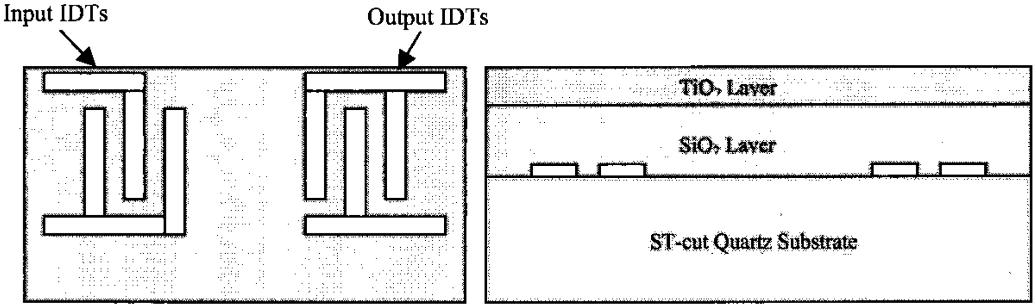


Fig. 1. Overall system set up.

B. Fabrication of the transducer

The device consists of double line layered SAW transducers. It is fabricated on a 14×19 mm^2 and 0.5 mm thick ST-cut quartz substrate. The transmit and receive interdigital transducers consist of 64 and 16 finger pairs respectively, with the width and separation 12.5 μm. The electrode metal layers were deposited by an electron beam evaporation process: Ti (200Å), Cr (300Å) and Au (500Å). The finger-pairs were then patterned using photolithographic techniques. The IDT aperture was 2.5 mm and the IDT centre to centre length was 4 mm. One transducer was used as a reference and the other has a TiO_2 selective layer. The substrate was a ST-cut quartz crystal, which is a piezoelectric with good thermal stability. The IDT patterns were fabricated directly onto the substrate surface. A SiO_2 layer was deposited on the top of the patterned area using an electron beam evaporation technique. The thickness of the SiO_2 layer was 3000 Å. This layer isolates the sensitive top layer (TiO_2 is a semiconductor) from the inter-digital transducers (Fig. 2).



a) b)

Fig. 2. Layered SAW sensor: a) Top view b) Cross section.

C. TiO_2 sensing layer

The sol-gel process was employed to prepare a TiO_2 thin film on the transducer. The precursor solution was prepared using the starting material of titanium butoxide ($Ti(OC_4H_9)_4$), with a purity of 99%. Analytical purity butanol was used as the solvent. The solution was spun coated (2500 rpm, 30 s) on the transducer to form a TiO_2 thin film. The film was annealed at 450°C for one hour to crystallise.

D. System set up

A Keithley 775A dual input frequency counter was used to measure both reference and sensor delay line oscillator frequencies. It was connected to a PC through a GPIB card and an IEEE-488 bus. A virtual instrument media programmed in LabView (National Instrument) software controls the system (Fig. 1).

E. Results and discussions

Figure 3 shows the frequency response as a function of time when the sensing layer is exposed to different oxygen concentrations. The optimum sensing temperature for measurements was found to be about 320°C. This temperature was provided by a micro heater fabricated on a sapphire substrate touching the back of the sensor.

The device was mounted inside an enclosed environmental cell. At a temperature of 320° C the nominal oscillation frequency was 63.04 MHz. All measurements were achieved using a gas flow of 0.1 LPM through the measuring chamber. Fig. 3 shows the frequency response as a function of time. The sensor was exposed to alternate concentrations of oxygen of 100ppm, 1000ppm and 10000ppm and between each measurement a purging gas of nitrogen was used. Frequency shifts were about 4 kHz, 16 kHz and 47 kHz.

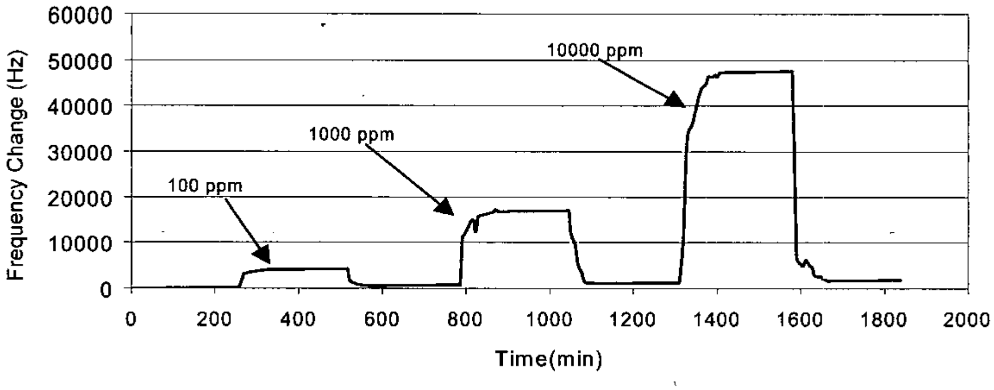


Fig. 3. Frequency response of the system to different oxygen concentrations.

The system shows high sensitivity. The response time for the 63% of the maximum peak value is about 25 seconds. The electromechanical coupling coefficient, K^2, for the layered gas sensor was equal to 0.023. For such a K^2, the calculated frequency shift [5] for exposure to 100ppm, 1000ppm and 10000ppm oxygen in nitrogen gas is equal to 3kHz, 15kHz and 45kHz, respectively. The theoretical results are in good agreement with the experimental results.

F. Conclusion

A system based on layered SiO_2/ST-cut quartz crystal transducers has been successfully fabricated for oxygen sensing. Nanocrystalline TiO_2 thin films have been successfully deposited by spin-coating a nano-particle TiO_2 suspension. The response to O_2 gas was fast and the sensitivity was remarkably high. Frequency shift values obtained from theoretical calculations were in agreement with the experimental results. Further investigations are planned using different SiO_2 thicknesses and different materials as the guiding layer.

Acknowledgment

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