REAL-TIME HERMITICITY DETECTION USING TITANIUM DIOXIDE THIN FILM RESISTOR AS BOTH OXYGEN AND PRESSURE SENSORS

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ABSTRACT

In-situ hermiticity detection using Titanium dioxide (TiO_2) thin film resistors has been investigated for the first time. Titanium dioxide is widely used as an oxygen sensor, with accurate partial pressure reading at high temperatures. In addition to being oxygen sensitive at room temperature, TiO_2 thin film resistors were found to be excellent pressure gauges at typical bonding pressures in the range of 1 Torr to 760 Torr. Combining the pressure sensitivity with the oxygen selectivity, Titanium dioxide thin film resistors constitute perfect hermiticity solutions for MEMS devices capped in an oxygen deficient environment at sub-atmospheric pressures. This unprecedented demonstration opens up new opportunities for monitoring real-time hermiticity in packaged MEMS devices, understanding the various failure modes during operation, and implementing the necessary diagnostic solutions.

INTRODUCTION

Hermiticity constitutes a critical precursor for the reliability of MEMS sensors. Ensuring hermiticity is a mandatory requirement for most encapsulated MEMS devices including automotive-grade sensors. Hermiticity failures emanates from two major sources: (1) Defects in the bonding material and (2) Cracks due to stress. A breach in hermiticity can change the gas content and pressure surrounding the MEMS device which in turns leads to performance shifts. In our previous work [1], we have found that any slight modification in the water content of the capped MEMS devices can lead to failure due to stiction. In addition, it is reported that the performance of RF MEMS switches is deleteriously affected when the devices are non-hermitically sealed due to the effect of humidity and small particles [2].

The state of the art hermiticity detection technique is Helium presence determination using mass spectroscopy [3]. However, this technique is not suitable for micro-cavities due to fast Helium diffusion rate. Several hermiticity detection techniques have been investigated. Elger *et al.* have investigated hermiticity existence optically using a non-contact profilometer with a chromatic confocal sensor. The pressure change inside the sealed cavity due to gas leakage causes a built-in membrane to deflect [4]. Pinter *et al.* have developed the technique of measuring the gas viscosity inside the cavity by the Quality factor (Q) of a vibratory device built inside the sealed MEMS devices [5]. Any breach in hermiticity will cause viscosity to change which is consequently detected by Quality factor shift. In addition, Lellouchi *et al.* adopted a Fourier transform infrared spectroscopy (FTIR) to quantify the concentration and composition of the gases in the sealed cavity [6]. These techniques do not allow for monitoring hermiticity in real-time during use and require expensive analysis equipment.

Titanium dioxide is an intrinsic semiconductor whose conductivity varies with the change in the concentration of the oxygen gas in the surrounding environment when deposited in the Anatastase phase [7]. In this work, Titanium dioxide thin films are used to quantify the pressure and oxygen content of the sealant gas by simple resistance measurement enabling real-time hermiticity checking both during manufacturing and in-operation. In contrast to the previous publications, this is the first paper that investigates

in-situ hermiticity detection of sealed MEMS devices using TiO_2 thin film resistors integrated inside the MEMS cavity for devices capped in a sub- atmospheric pressure and in an oxygen deficient environment.

Figure 1: Layout schematic of a TiO_2 test structure.

FABRICATION PROCESS

A Titanium dioxide thin film with metal electrodes was built on insulated substrate. The test structure was measured in a sealed pressure chamber that mimics the environment within a sealed MEMS device in order to validate the proposed hermiticity detection concept. Figure 1 shows the layout schematic of a $3800\mu\text{m} \times 3800\mu\text{m}$ TiO_2 block test structure. The fabrication process starts by thermally growing $1.47\mu\text{m}$ of oxide followed by the deposition of $1200\text{\AA}$ of stoichiometric nitride to form the insulation layers. A $2200\text{\AA}$ thick Titanium dioxide film was then deposited on the wafer using Plasma Enhanced Chemical Vapor Deposition (PECVD), and consequently annealed at 500°C for 30min in a vacuum environment in order to form the Anatastase phase [8]. The TiO_2 film was then patterned with a wet etchant composed of 5:1:4 HNO_3 : HF: H_2O at 70°C . A $200\text{\AA}/2000\text{\AA}$ of Ti/Au was then deposited and patterned by lift-off to form metal contacts to the TiO_2 film resistor.

The integration of the TiO_2 thin film resistors inside a capped MEMS sensor is proposed to be achieved using *Through Silicon Via* (TSV) technology as shown in the process flow outlined in Figure 2. The TiO_2 thin film is deposited on the cap side of the sealed MEMS devices. The processing of the cap wafer starts by forming front side alignment marks on the front side of the wafer. Vias are then etched on the back side of the wafer up to a depth of two-thirds the total wafer thickness using Deep Reactive Ion Etching (DRIE). Via isolation is achieved by a thin thermally grown oxide of 250nm in thickness. The cavity is later filled with a thin conductor seed layer followed by copper electroplating. The deposited copper is grinded down to the underlying oxide liner

using Chemical Mechanical Polishing (CMP). Titanium dioxide is deposited on the back side of the wafer and the front side is grinded down using CMP to get access to the copper bond pads. The cap wafer is finally bonded to the device wafer.

Figure 2: Fabrication Process of TiO_2 thin film resistors on cap side using Through Silicon Via (TSV) technology.

RESULTS AND DISCUSSION

The thin film resistor were tested in a Lake Shore low temperature probe chamber Model CPX-HF equipped with a temperature controller Model 332 with built-in probes and gas inlets, where both O_2 and N_2 gases can be flown and chamber pressure was monitored as shown in Figure 3. An Agilent B1500A parametric semiconductor analyzer was used to measure the I-V curves necessary to extract the thin film resistivity where the voltage was swept between 0 and 1.5V at an increment of 50mV.

Figure 3: Digital picture showing testing setup of TiO_2 thin film resistors inside an enclosed chamber.

Figure 4: The TiO_2 thin film resistance vs. temperature at atmospheric pressure.

The resistance of the Titanium dioxide thin film resistor was measured at both room temperature (25°C) and 177°C as shown in Figure 4. Both tests were done are normal pressure of 1 atm with the chamber lid open. At room temperature the resistance of the thin film resistor as outlined in the schematic shown in Figure 1 was measured as 200GΩ. The resistivity can be then deduced as follows:

$$R = \frac{\rho W t}{L} \quad (1)$$

where R is the resistance of the thin film resistor, ρ is the film resistivity, W is the width of the resistor with a value 3800 μm for this resistor structure, t is the thickness of the film with a value of 220nm, and L is the resistor length with a value of 3800 μm . As such the resistivity of the Titanium dioxide film at room temperature is calculated as $4.4 \times 10^6 \Omega\text{-cm}$. At 177°C, the resistance is measured 4GΩ, and as such the resistivity is calculated as $88 \times 10^3 \Omega\text{-cm}$. The resistivity decreased 50 times as the chuck temperature increased from room temperature to 177°C

at atmospheric pressure as can be inferred from the I-V curves shown in Figure 4. The resistivity of sputtered thin film of Titanium dioxide has been reported in the literature with values of $4.95 \times 10^6 \Omega\text{-cm}$ and $20 \times 10^3 \Omega\text{-cm}$ at 25°C and 177°C , respectively [9]. The value measured at room temperature is very close to the value reported in the literature. The discrepancy between the measured value and the value reported in the literature at 177°C is attributed to the lower equilibrium temperature of the Titanium dioxide thin film due to the silicon substrate and epoxy glue separation between the heated chuck and the tested sample.

Figure 5: Response of TiO_2 thin film resistors capped at 100 Torr of pure N_2 , 100 Torr of pure O_2 , and normal room pressure at room temperature of 25°C .

Figure 6: Response of TiO_2 thin film resistors capped at 250 Torr of pure N_2 , 250 Torr of pure O_2 , and room pressure at a temperature of 177°C .

In order to reproduce micro-cavity gas environment conditions, the chamber was evacuated and then filled with pure nitrogen gas (N_2) up to 100 Torr pressure, a typical gas content and pressure level of many capped MEMS devices. As shown in Figure 5, current flowing through the TiO_2 thin film at 1.5V DC Bias and room temperature dropped from 40pA at bonding environment to roughly 10pA when the control chamber was opened and exposed to atmospheric pressure. As such the resistance dropped 4 times as hermeticity was breached and sensor

environment changed from pure Nitrogen at 100 Torr to a room temperature and pressure environment. The resistivity of the Titanium dioxide thin film was also measured at 100 Torr of pure Oxygen gas. There is a distinct shift in thin film resistivity as shown in Figure 5 with the Titanium dioxide resistance increasing from $48\text{G}\Omega$ in a Nitrogen environment to $58\text{G}\Omega$ in an Oxygen environment. This shows the selectivity of the thin film sensor to oxygen at room temperature.

Figure 7: TiO_2 thin film resistors I-V curves for varying N_2 pressures showing increase in resistance as N_2 pressure inside the control chamber increases.

Figure 8: TiO_2 thin film resistors I-V curves for varying O_2 pressures showing increase in resistance as O_2 pressure inside the control chamber increases.

As the temperature increased to 177°C , the current flowing through the TiO_2 thin film dropped from 1480pA for a hermitically sealed die capped at 250 Torr of pure Nitrogen to 360pA for a leaky die exposed to atmospheric pressure as shown in Figure 6. This corresponds to a resistance drop from roughly $4\text{G}\Omega$ to $1\text{G}\Omega$ as hermeticity is breached. Response of TiO_2 thin film resistors to pure O_2 environment with same pressure as pure N_2 capped devices is also measured. The current flowing through the TiO_2 thin film resistor was 270pA at 1.5V of bias. This corresponds to a resistance value of $5.54\text{G}\Omega$. The higher resistance measured in a pure oxygen environment at a pressure

level of 250 Torr relative to the value measured at normal atmospheric conditions is an indication of sensitivity of the thin film to oxygen content in the surrounding environment.

The variation of TiO_2 film resistivity to control chamber pressure was studied. As shown in Figure 7, TiO_2 thin film resistors act as perfect pressure sensors at room temperature and N_2 pressure varying from 1 Torr to 760 Torr. The resistance increased as the nitrogen pressure increased. The resistance values were 0.313G Ω , 0.786G Ω , 1.078G Ω , and 1.72G Ω , at a pressure of 1 Torr, 10 Torr, 100 Torr, and 760 Torr, respectively.

In addition, TiO_2 thin film resistors are sensitive to O_2 pressure varying from 0.1 Torr to 100 Torr, with signal saturating above 100 Torr indicating sensor selectivity to O_2 at room temperature. The resistance increased as the oxygen pressure increased. The resistance values were 0.533G Ω , 2.132G Ω , and 4.000G Ω , and 6.993G Ω , at a pressure of 0.1 Torr, 1 Torr, 10 Torr, and 100 Torr, respectively. After 100 Torr, the measured I-V overlapped indicating saturation in the output signal. It is also observed that the resistance values at the same pressure are higher in the presence of the oxygen gas vs. nitrogen gas.

The transduction phenomenon of Titanium dioxide thin films and their oxygen sensitivity is a surface phenomenon. A structure with parallel TiO_2 line resistors is proposed in order to maximize open surface area as shown in Figure 9.

Figure 9: Layout schematic of a proposed TiO_2 test structure with improved oxygen sensitivity.

CONCLUSION

A real-time hermeticity solution has been investigated for the first time. The proposed technique is based on the resistance measurement of a thin film Titanium dioxide resistor at a DC bias voltage as small as 1V. Titanium dioxide is an extrinsic semiconductor with some missing oxygen atoms in its crystalline structure. The variation of the oxygen concentration in the surrounding environment will modulate the resistivity of the thin film due to the change in the free carrier concentration. MEMS devices capped at sub-atmospheric pressure in an oxygen deficient environment will experience an abrupt change in built-in Titanium dioxide resistance as the film is exposed to atmospheric pressure and a 21% oxygen concentration. This is a clear indication of hermeticity and it can be used to monitor MEMS devices during operation versus the other techniques which check for hermeticity during manufacturing. Titanium dioxide thin films with metal electrode were built on insulated substrates, and their behavior to varying gas pressures and composition was studied in a control

chamber. The testing results show clear distinction between resistance of Titanium dioxide films at room temperature at pressure versus environments with pure nitrogen gas and sub-atmospheric pressure levels. The thin films were very sensitive to both nitrogen and oxygen gas pressures in the range between 1 Torr to 760 Torr and 0.1 Torr to 100 Torr, respectively. In addition, the thin films were selective to oxygen with higher resistances measured at the same pressure level for oxygen gas as compared to nitrogen gas. A process flow to integrate the Titanium dioxide with the MEMS capping process is proposed using Through Silicon Via (TSV) technology with the thin film built on the cap side of the sensor. It is envisioned that this novel study will resolve many issues related to hermeticity by enabling in-use hermeticity monitoring in order to understand the root-cause of failures in field and part returns.

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