

Inductance Characteristic of Annealed Titanium Dioxide on Silicon Substrate

Chih Chin Yang, Lan Hui Huang, Bo Shum Chen, Jia Liang Ke, and Chung Lun Tsai

Abstract—The control of oxygen flow rate during growth of titanium dioxide by mass flow controller in DC plasma sputtering growth system is studied. The impedance of TiO₂ films for inductance effect is influenced by annealing time and oxygen flow rate. As annealing time is increased, the inductance of TiO₂ film is the more. The growth condition of optimum and maximum inductance for TiO₂ film to serve as sensing device are oxygen flow rate of 15 sccm and large annealing time. The large inductance of TiO₂ film will be adopted to fabricate the biosensor to obtain the high sensitivity of sensing in biology.

Keywords—Annealed, Inductance, Silicon substrate, Titanium dioxide.

I. INTRODUCTION

BECAUSE the titanium dioxide is with the non-toxic characteristic and well stability in chemical reaction, it will be the material of potential applications in electrochemical field, photovoltaic devices, photo-catalytic, and memory cell capacitors [1]. The silicon based ultra large scale integrated (ULSI) circuits is with a high limitation for the thickness of oxide layer in scale of ULSI circuit, because of gate dielectrics in the metal oxide semiconductor (MOS) or ionic sensing metal oxide semiconductor (ISFET) devices in application of biosensor field. The breakdown voltage of MOSFET will be decreased with decreasing the gate oxide thickness. The cause is that the electric field of gate is increased at the same bias at the gate of MOSFET. The leakage current is also important factor with reaching the limit of tunneling thickness of titanium dioxide. Therefore, the physical thickness of oxidation layer must be increased at the gate terminal for the MOSFET device. In this paper, the inductance, so call leakage impedance, is measured and study to improve the leakage current effect of MOSFET when the TiO₂ thin film is used to as the insulator of MOS and the sensing layer of ISFET [2]. The TiO₂ films are with two different phases for the films structure including rutile phase and anatase phase. In general, the rutile phase of titanium dioxide is usually with stable phenomenon in the thermal mechanism than the anatase phase of titanium dioxide [2]. The decision of phase for the titanium dioxide film is depended on growth temperature, oxygen concentration and annealing conditions. The different phase and morphology of titanium

dioxide film will be also affected the sensing effect of ISFET absolutely. The titanium dioxide films are ever grown and deposited on many kinds of substrate by using the pulsed laser deposition (PLD) [3], atomic layer deposition (ALD) method [4], chemical vapor deposition (CVD) method [2], and DC magnetron sputtered method [5], and RF magnetron sputtered method [1]. The different materials including silicon dioxide [2], quartz glass [1], silicon carbide [6], and SnO₂:F coated glass-slides [7], as the substrates of deposited titanium dioxide layer which don't use the single crystal silicon of with mature fabrication process of integrated circuit in usual. In this research, the deposited titanium dioxide films with different annealing condition and gas flow rate of oxide source on single crystal silicon substrate are proposed.

II. EXPERIMENTAL

The TiO₂ thin film were prepared on the silicon substrate of (100) orientation at growth temperature of 500°C using titanium target with purity of 99.99% by using the DC reactive sputtering method with DC power of 80 W. The substrate was separated by a distance of about 10 cm from the centers of the target holder. The titanium target has diameter of 2 inch and 6 mm thick. The growth pressure in the chamber was firstly evacuated to 1×10^{-6} Torr before thin film growth. The work pressure was at room temperature, about 4.5×10^{-3} Torr. After purging the chamber, the TiO₂ films were deposited on the n-type silicon (100) substrates by the reactive DC magnetron sputtering method. Before loading the target and substrate, the substrates were cleaned by the BOE solution at room temperature with ultrasonic agitation cleaner before the TiO₂ film grown on silicon substrate. The substrates was taken into the BOE solvent solution, then the substrate is put into the agitation of ultrasonic cleaner and cleaned about 5 minute at first. And then the substrates were rinsed by using the solvent solutions of acetone, methanol and DI water in sequence respectively. After cleaning the substrate, we dried up the silicon wafer by using nitrogen gun. The target of reactive DC magnetic sputter was used titanium element and the oxygen was flowed into chamber to growth the TiO₂ thin film on silicon wafer. The oxygen source was arranged with the flow rate into chamber of 5 to 35 sccm in various. The growth time of TiO₂ thin film is about 10 minutes. The argon of about 10 sccm in flow rate as plasma is used to bomb the target.

The impedance value of TiO₂ thin film is measured in probe station system by using the LCR instrument in sample area of

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2cm×2cm with interface of ohmic contact between probe and films. The change of inductance of TiO₂ films was also examined by the measurement of LCR meter after TiO₂ films were annealed. We measure the inductance of TiO₂ films which are set into the annealing environment about 30 minutes and one hour. The inductance value of TiO₂ thin film is with some changes as increasing annealing time which we confirmed in this report. The annealing of TiO₂ thin film is completed by the rapid thermal annealing (RTA) system. The annealing temperature of RTA process was arranged at the temperature of 450°C and the annealing time was not including the rise time and fall time for the reach and recover of annealing temperature. In this process, we use RTA system without compensation gas.

III. RESULTS AND DISCUSSION

Fig. 1 shows the films thickness of grown TiO₂ as the function of oxygen flow rate varied from 5, 15, 25, to 35 sccm at growth temperature of 500°C and growth time of ten minutes. It is clear that the film thickness is raised as increased the oxygen flow rate at fixed argon flow rate. The growth rate is obviously increased when the oxygen flow rate is more than 15 sccm. This suggested that the increased oxygen molecule will result in the more frequency reaction with sputtered titanium ionic. The growth rate of TiO₂ films grown by using the DC sputtering system is more than about 0.16μm thick per hour.

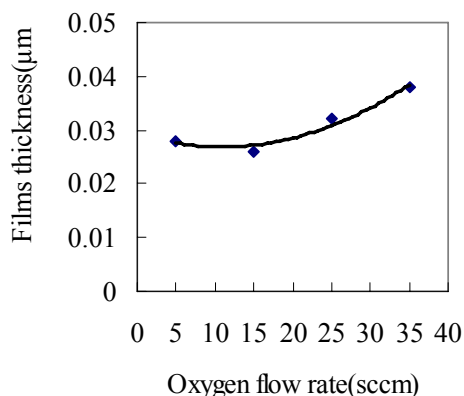


Fig. 1 The TiO₂ films thickness versus oxygen flow rate at growth temperature of 500°C and growth time of ten minutes

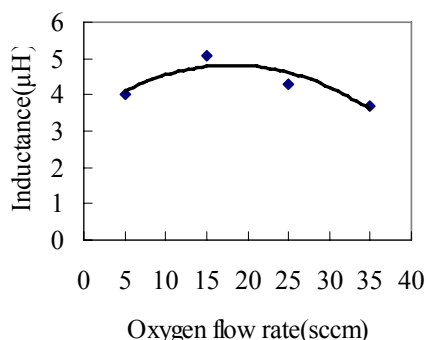


Fig. 2 The inductance of TiO₂ films without annealing process for the different oxygen flow rate from 5 sccm to 35 sccm with increment of 10 sccm

The inductance of TiO₂ films without heat treatment versus oxygen flow rate in impedance measurement is presented in Fig. 2. The oxygen flow rate is varied from 5 sccm to 35 sccm with increment of 10 sccm. The maximum inductance is occurred at about oxygen flow rate of 15 sccm. When the oxygen flow rate is less than 15 sccm, the inductance of TiO₂ films on surface is slightly decreased. We conclude that the TiO₂ films are approached to the conductor because of the rare of oxygen molecular in TiO₂ films. As the oxygen flow rate is more than 15 sccm, the inductance of TiO₂ films will be decreased with increasing the oxygen flow rate. The cause of decrease of inductance is due to the increase of capacitance effect.

The annealing effect of TiO₂ films at annealing temperature of 450°C and annealing time of 30 minutes is shown in Fig. 3. The inductance of TiO₂ film grown at higher and lower oxygen flow rate is seriously influenced by annealing process. In the Fig. 3, the inductance of TiO₂ film is increased twice or three times obviously. The reason is that the TiO₂ film is formed the image part of impedance value at these ranges, which can be applied in sensor with capacitance type or inductance type.

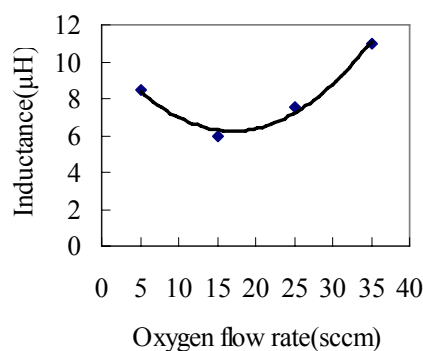


Fig. 3 The effect of annealing process of grown TiO₂ films at annealing temperature of 450°C and annealing time of 30 minutes

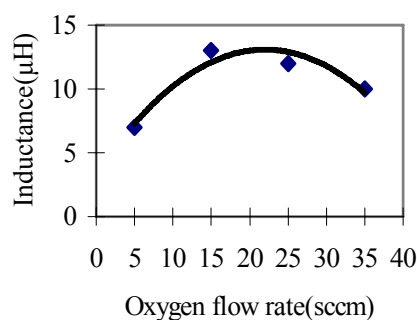


Fig. 4 The effect of annealing process of grown TiO₂ films at annealing temperature of 450°C and annealing time of 60 minutes

In Fig. 4, the TiO₂ film is treated in RTA system at annealing temperature of 450°C and annealing time of one hour. The inductance of TiO₂ film after process of annealing time in one hour is slightly changed merely at the higher or lower oxygen flow rate. As the oxygen flow rate is 15 sccm or 25 sccm, the inductance will be changed in twice than it without annealing process. The phenomenon can be suggested that the TiO₂

compound will decrease the institute impurity and titanium-rich effects for a long annealing time at the growth condition of oxygen flow rate of 15 sccm or 25 sccm.

Table I shows the results of inductance effect on the TiO₂ film to apply it in biosensor devices. It's demonstrated that the inductance is with maximum and optimum at oxygen flow rate of 15 sccm. The inductance of TiO₂ films is increased as the annealing time is increased, because of well anatase structure of annealed TiO₂ films.

TABLE I
CHARACTERISTICS OF INDUCTANCE OF TiO₂ FILMS AT THE ANNEALING ENVIRONMENT WITH VARIOUS FLOW RATES OF OXYGEN SOURCE AND ANNEALING TIME (THE UNIT OF INDUCTANCE IS INMH PER SAMPLE AREA OF 2CM×2CM)

O ₂ flow rate (sccm) \ annealing time	5 (sccm)	15 (sccm)	25 (sccm)	35 (sccm)
Without	4.0	5.1	4.3	3.7
30 min	8.5	6.0	7.5	11
60 min	7.0	13.0	12.0	10.0

The measurements of the inductances were completed by using the LCR instrument as shown in Table I. It was clearly that the different between with high and low flow rate or annealing time. The measured result is obviously for the inductances which has the reduction in the environment of without annealing time. We believe that the oxygen molecular was absorbed by the titanium molecular to form the TiO₂ thin film. The absorption of oxygen molecular will have influenced for the dielectrics of the TiO₂ thin film. Hence, it changes the capacitances of TiO₂ film. Therefore, we suggest that the TiO₂ thin film has a ability as humidity sensor.

IV. CONCLUSION

In this paper, the oxygen flow rate during growth of titanium dioxide is controlled by mass flow controller in DC plasma sputtering growth system. The impedance of TiO₂ films for inductance effect is studied. The annealing time is the more, the inductance of TiO₂ film is the more. The growth condition of optimum and maximum inductance for TiO₂ film to as sensing device is oxygen flow rate of 15 sccm. The large inductance of TiO₂ film will be adopted to fabricate the biosensor to obtain the high sensitivity of biosensor.

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REFERENCES

- [1] J. M. Jung, M. Wang, E. J. Kim, C. Park, and S. H. Hahn, "Enhanced photocatalytic activity of Au-buffered TiO₂ thin films prepared by radio frequency magnetron sputtering," *Applied Catalysis B: Environmental*, vol. 84, pp. 389–392, 2008.
- [2] M. K. Bera and C. K. Maiti, "Electrical properties of SiO₂/TiO₂ high-k-gate dielectric stack," *Materials Science in Semiconductor Proceeding*, vol.9, pp. 909–917, 2006.
- [3] M. F. Brunella, M. V. Diamanti, M. P. Pedferri, F. Di Fonzo, C. S. Casari, and A. Li.Bassi., "Photocatalytic behavior of different titanium dioxide layers," *Thin Solid Films*, vol. 515, pp.6309-6313, 2007.
- [4] I. Jogi, K. Kukli, J. Araik, A. Aidla, and J. Lu, "Precursor-dependent structural and electrical characteristics of atomic layer deposited films: Case study on titanium oxide," *Materials Science in Semiconductor Proceeding*, vol.9, pp. 1084–1089, 2006.
- [5] B. Karunakaran, R. T. Rajendra Kumar, V. Senthil Kumar, D. Managlaraj, Sa. K. Narayandass, G. Mohan Rao, "Structure characterization of DC magnetron-sputtered TiO₂ thin films using XRD and Raman scattering studies," *Materials Science in Semiconductor Proceeding*, vol.6, pp. 547–550, 2003.
- [6] M. H. Weng, R. Mahapatra, P. Tappin, B. Miao, s. Chattopadhyay, A.B. Horsfall, and N. G. Wright, "High temperature characterization of high-kdielectrics on SiC," *Materials Science in Semiconductor Proceeding*, vol.9, pp. 1133–1136, 2006.
- [7] M. F. Hossain, S. Biswas, T. Takahashi, and A. Fujishima, "Investigation of sputter-deposited TiO₂ thin film for the fabrication of dye-sensitized solar cells," *Thin Solid Films*, vol.516, pp.7149–7154, 2008.

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