The Characteristics of MgO Sensing Membrane applied in Electrolyte-Insulator-Semiconductor after Rapid Thermal annealing in O₂ and N₂ ambient

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Abstract

EIS structure with high-K Magnesium Oxide (MgO) as a sensing membrane fabricated by RF sputtering and the characteristics of the subsequent post rapid thermal annealing (RTA) treatment in various ambient (N_2 , O_2) with various temperature have been investigated in the proposed work. Furthermore, this EIS structure annealed at 800°C in different ion (Na⁺, K⁺, Urea, Glucose) solution exhibits high sensitivity and high linearity, low hysteresis and low drift voltage.

1. Introduction

The enzyme biosensor named as enzyme field-effect transistor (ENFET) largely depends on the performance of the ISFET-based pH sensing mechanism. For chemical stability and low leakage current, various kinds of high-k metal oxide materials such as HfO₂, Y₂O₃, Pr₂O₃, Gd₂O₃, and WO₃ have been introduced. Among various ferroelectric materials MgO has been considered because of its good perovskite structure, high dielectric constant, high electronegativity and good chemical and thermal stability with Si material which will improve ion sensing performance to meet the requirement of commercial biomedical industrial applications.

2. Experiment

EIS structure with MgO membrane was fabricated on 4-inch n-type (100) Si wafers and then the wafers were cleaned by using HF-dip (HF: $H_2O = 1:100$). A 50-nm MgO was deposited by RF sputtering on a silicon wafer in the ambient of Ar: O_2 at 25:0. Next, samples were annealed at different temperatures (600°C, 700°C, 800°C) by rapid thermal annealing in different O_2 and N_2 ambient for 30 sec. The back-side contact of the Si wafers was deposited by Al film and the sensing area was defined by a standard photolithography process. Finally, the samples were fabricated on the copper lines of the printed circuit board (PCB) by using silver gel. The detail EIS structure is illustrated in Fig. 1.

3. Result and Discussion

XRD of MgO film after post-RTA treatment for various temperatures in N_2 and O_2 ambient for 30 sec was done, where at the highest temperature of 800°C clearly exhibited a stronger peak (220) at 61.70°, shown in the Fig. 2 and Fig. 3.

When the RTA temperature was at 800°C, Mg 2p and O1s XPS spectra exhibit the strongest binding intensity of MgO. From Fig. 4 and Fig. 5 it can be easily observed that

after post-RTA treatment the crystalline structure of MgO was better in O_2 ambient rather than in N_2 ambient because oxygen can easily combine with Mg atoms for its electronegativity property, and thus reduce SiO₂ and silicate formation.

In AFM analysis, the surface roughness of MgO sensing membrane after post-RTA treatment at 800°C is better in O_2 ambient than in N_2 shown in Fig. 6 and Fig. 7.

The C-V curves of the sample shown in Fig. 8 and Fig. 9 where RTA annealed (at 800°C) MgO film was immersed in a buffer solution with various pH values which causes the threshold voltage shift of MgO membrane, reveals higher sensitivity (61.64 mV/pH) and higher linearity(99.79%) under O₂ atmosphere than N₂ ambient (the sensitivity is 54.61 mV/pH and the linearity is 99.09%). With different pH loop of $7\rightarrow 4\rightarrow 7\rightarrow 10\rightarrow 7$, the sample shows lower hysteresis voltage in case of O₂ (5.03 mV) compared to N₂ (6.26 mV) at 800°C. The sample annealed at 800°C also shows the lower drift rate in oxygen ambient (1.55mV/hr) rather in N₂ ambient (3.71mV/hr) because the sample with oxygen treatment reduced the traps and the dangling bonds generated at the surface of the membrane.

In case of the sodium and potassium ions solution, the calculated pNa and pK sensitivity of MgO sensing membrane annealed in O_2 and N_2 environment were shown in Fig.10 and Fig. 11 where the EIS with MgO sensing film is more responsive to H⁺ relative to Na⁺ and K⁺. Fig. 12 and Fig. 13 shows the pUrea-responses of enzyme-immobilized MgO with RTA temperature at 800°C in O_2 and N_2 ambient where Fig. 14 and Fig. 15 shows the pGlucose-response immobilization of MgO by covalent bonding methods.

4. Conclusions

The MgO sensing membrane after annealing at 800° C in O_2 ambient not only shows higher sensitivity, higher linearity, higher H⁺ selectivity, lower hysteresis voltage and lower drift rate but also shows improved bonding intensity, stabilized crystalline structure and formation of stronger lattice to enhance the peak intensity.

References

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Fig. 1 EIS structure



Fig. 2 XRD of the MgO film after annealing at various temperatures on single crystalline silicon in O_2 ambient for 30 sec



Fig. 3 XRD of the MgO film after annealing at various temperatures on single crystalline silicon in N_2 ambient for 30 sec



Fig. 4 XPS results of MgO film (a) O 1s in O_2 ambient, (b) O 1s in N_2 ambient



Fig. 5 XPS results of MgO film (a) Mg 2p in O_2 ambient (b) Mg 2p in N_2 ambient



Fig. 6 AFM of high-k MgO surface after RTA at 800°C in O₂ R_{rms}=4.33(nm)



Fig. 7 AFM of high-k MgO surface after RTA at 800°C in N₂ R_{rms}=2.85 (nm)



Fig. 8 The normalized C-V curve of MgO sensing membrane with RTA at 800°C in O₂, the inset figure represents the sensitivity and linearity



Fig. 9 The normalized C-V curve of MgO sensing membrane with RTA at 800°C in N₂, the inset figure represents the sensitivity and linearity



Fig. 10 The different ion sensitivity of MgO sensing membrane annealed 800° C in O_2 ambient



Fig. 11 The different ion sensitivity of MgO sensing membrane in N_2 ambient



Fig. 12 pUrea-response of enzymeimmobilized MgO annealed at 800°C in O₂ ambient



Fig. 13 pUrea-response of enzymeimmobilized MgO annealed at 800 $^{\circ}\text{C}$ in N_{2} ambient



Fig. 14 pGlucose-responses of enzymeimmobilized annealed at 800°C in O₂ ambient



Fig. 15 pGlucose-responses of enzymeimmobilized annealed at 800° C in N₂ ambient