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Optimization of Urea-EnFET Based on Ta_2O_5 Layer with Post Annealing

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1. Introduction

Enzymatic field effect transistors (EnFETs) have been proposed as an attractive sensing platform for biomedical applications, such as penicillin, urea, glucose, etc [1]. The basements of EnFETs are usually pH ion sensitive filed effect transistors (ISFETs). Many materials have been applied for the sensing membrane of pH-ISFETs, in these materials Ta_2O_5 layer has the largest surface buffer capacity (β). In addition, wide applicable pH range (from pH -7 to 13.7) and high chemical resistance for the cleaning-in-place (CIP) test are the attractive advantages of Ta_2O_5 layers as a pH sensing membrane [2, 3]. Since Ta_2O_5 layer is a good sensing membrane for ISFET application, to minimize the light induced drift become subjects in many laboratories.

In this study, the sensing properties of the Ta_2O_5-ISFETs without and with post annealing were described. In addition, to concern about the biomedical application, the urease was immobilized on ISFETs with covalent bonding and compare with standard Si_3N_4-ISFET.

2. Experimental

The ISFETs were fabricated by Institute of Electron Technology (ITE), Poland. The structure and cross section are shown in Fig. 1. The ISFETs are designed with *p*-well arrangement on 3 inch (100) *n*-type wafers. The ratio of channel width and length was W/L= 600/16 (in μm). The 50 nm-thick buffering oxide layers were deposited by dry oxidation. The sensing membranes, Ta_2O_5 layers were deposited by rf sputtering, and a post annealing in N_2 ambient for 900^{o}C was performed as the best condition.

The procedures of covalent bonding were described as follows. At first, the surfaces of the sensing membrane were oxidized with H_2O_2 over night. Then, the pretreated surfaces were immersed in 9% APTS for 1 hour. Afterwards, these surfaces were immersed in 10% glutaraldehyde solution for 1 hour. Finally, ureases (E.C. 3.5.1.5) were immobilized on the surfaces. To avoid the deviation caused by the non-attached enzyme, the device was rinsed by phosphate buffer after final step. (Fig. 2)

To evaluate the performance of ISFETs and EnFETs, the I_{DS}-V_{GS} curves were measured by HP 4156, firstly. Afterwards, a constant drain-source voltage and constant drain-source current (CVCC) circuit (Fig. 3) was preferred in measurements with I_{DS} = 100 μA and V_{DS} = +0.5 V. Commercial pH buffer solutions from pH 2 to pH 12 were used for the pH sensing properties. For the light induced drift, the intensity of light source was controlled from 1500 lux to 4500 lux. The sensitivity of urea concentrations were measured from 1 to 10 mM, which with a set of urea concentration variation in 5mM phosphate buffer at pH 6.

3. Results and discussion

At first, the I_{DS}-V_{GS} curves of Ta_2O_5-ISFET measured in the buffer solutions from pH 2 to pH 12 are shown in Fig. 4. The I-V curves shifted to the left-side when the ISFETs were measured in buffer solutions of high pH values. The pH response is shown in the inert of Fig. 4. Afterwards, the sensing properties including sensitivity, hysteresis, and drift were measured by CVCC circuit. The results of ISFETs without and with post annealing were listed in Table I, in addition, the results of standard Si_3N_4-ISFET was also included. In this table, the ISFETs with annealed Ta_2O_5 layer showed a near Nernstian response and best response. To find out the mechanism of improved sensing properties, SEM and XPS spectra were analyzed as shown in Fig 5 and Fig 6, respectively. In these two figures, the increase of surface roughness and oxygen intensity could be the reason for the better properties. Moreover, the light induced drift was shown in Fig. 7. The low response was observed on the Ta_2O_5-ISFET with post annealing.

For the urea monitoring by EnFET, a similar I_{DS}-V_{GS} curves and transconductance were observed before and after urease immobilization as shown in Fig. 8. The urea response of output voltage to time variation is shown in Fig. 9. The higher concentration was testing, the more intense reaction was performed. In all concentrations, the output voltage was stable in 2 min. The phenomenon could also observed in Fig. 10. Fig. 11 shows the pC_{urea} sensitivity of EnFETs with different pH sensing membrane. In all cases, the linearity is over 99% between 1 to 7.5 mM of urea. As expected, the EnFET with annealed Ta_2O_5 layer shows the best urea response.

4. Conclusion

In this paper, the best pH sensing properties including sensitivity, hysteresis, drift, and light induced drift, were observed on Ta_2O_5-ISFET with post annealing. In addition, the best properties for urea detection were also performed on the EnFET with annealed Ta_2O_5 layer.

Acknowledgements

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References

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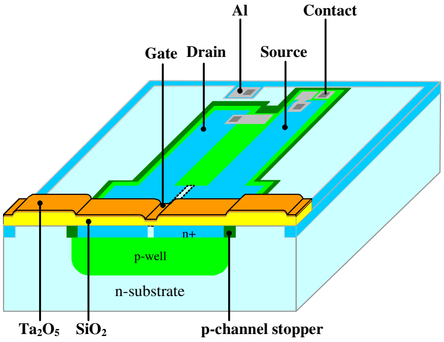


Fig. 1 Structure and cross section of Ta_2O_5-ISFET (ITE, Poland).

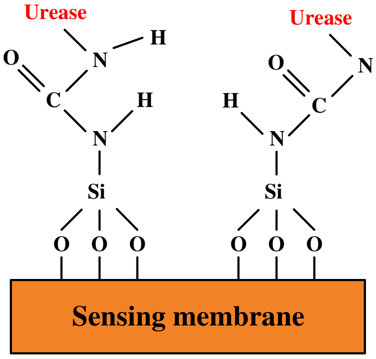


Fig. 2 Schematic of urease immobilized on the top of sensing membrane with covalent bonding.

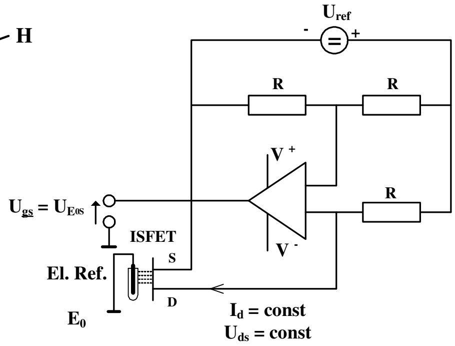


Fig. 3 CVCC circuit of ISFETs and EnFETs.

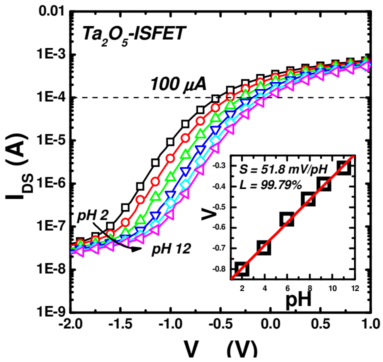
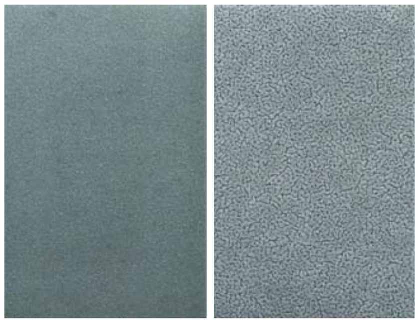


Fig. 4 I_{DS}-V_{GS} curve and sensitivity of Ta_2O_5-ISFET measured from buffer solitions of pH 2 to pH 12.

Table I Sensing properties of ISFETs, with Si_3N_4, Ta_2O_5, and annealed Ta_2O_5 layers

	pH sensitivity (mV/pH)	Hysteresis (7-4-7-10-7) (mV)	Drift (mV/h)
Si_3N_4	51.07	5.1	-0.18
Ta_2O_5	51.83	6.3	-0.43
Ta_2O_5 (anneal)	56.88	0.7	-0.75



(a) (b)

Fig. 5 SEM of Ta_2O_5 layers with (a) amorphous phase and (b) polycrystalline.

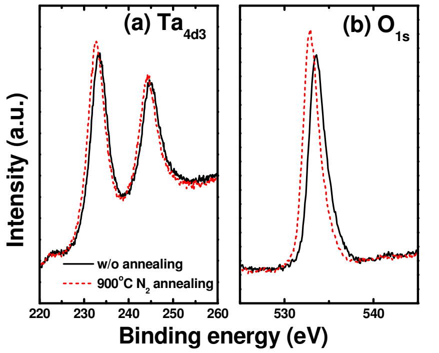


Fig. 6 XPS spectra of (a) Ta_{4d3} and (b) O_{1s} for the Ta_2O_5 layer without and with post annealing.

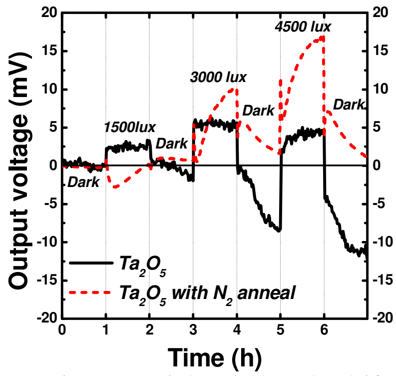


Fig. 7 Light induced drift of Ta_2O_5-ISFET without and with post annealing.

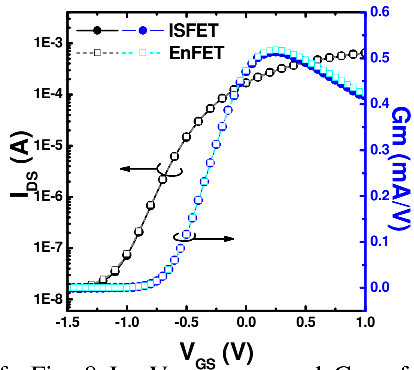


Fig. 8 I_{DS}-V_{GS} curves and Gm of ISFET and EnFET.

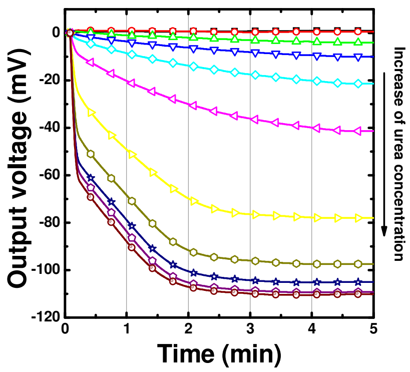


Fig. 9 Response of the output voltage to time variation for the EnFET with different urea concentrations.

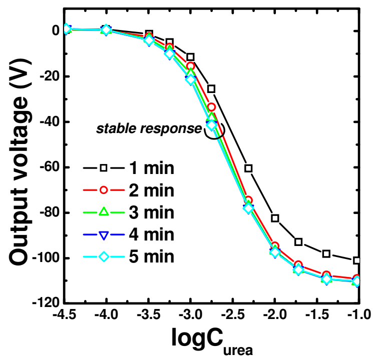


Fig. 10 Output voltage to urea concentrations for the EnFET measured with various time from 1-5 min.

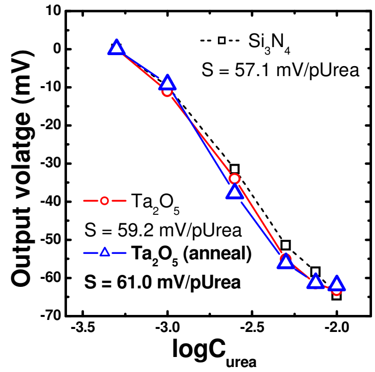


Fig. 11 Urea sensitivity slope of EnFET with Si_3N_4, Ta_2O_5, and annealed Ta_2O_5 layers.