

# **Nb<sub>2</sub>O<sub>5</sub> Sensing Membrane in Extended Gate Field Effect Transistor Biosensor With Post Rapid Thermal Annealing**

Lien Tai Kuo, Chyuan Haur Kao, Titisha Chakraborty, Munmun Das, Chun Fu Lin, Chun Hsiang Wang, Yi Wen Liu

Department of Electronic Engineering, Chang Gung University  
259 Wen-Hwa 1<sup>st</sup> Road, Kwei-Shan, Tao-Yuan 333, Taiwan, R.O.C.  
Phone: +886-3-2118800 ext. 5783 E-mail: [chkao@mail.cgu.edu.tw](mailto:chkao@mail.cgu.edu.tw)

## **Abstract**

**The post-annealing effect of high-k Niobium Oxide sensing membrane applied to Extended Gate field effect transistor (EGFET) by RF sputtering was studied for different annealing conditions. The most interesting sensing performance in different ionic, as well as urea and glucose solutions, was achieved at 700°C annealing temperature.**

## **1. Introduction**

The EGFET has a lot of advantages, such simple to passivize and package, a flexibility of shape of the extended gate area, etc. Various high-k materials replacing the gate SiO<sub>2</sub> layer have been widely investigated due to their large conduction band offset. Recently, a substantial advancement has been achieved in the preparation of a new class of material called Niobium Oxide (Nb<sub>2</sub>O<sub>5</sub>) whose dielectric constant is in the order of forty provides a high capacitance and its good chemical stability and corrosion resistance in both acid and base solutions makes it useful for electrochemical biosensor applications.

In this work, the high-k Nb<sub>2</sub>O<sub>5</sub> dielectric deposited on polycrystalline silicon by reactive radio frequency (RF) sputtering applied to an EGFET and post-deposition rapid thermal annealing effect has been studied.

## **2. Experiment**

First, the wafers were cleaned by HF-dip (HF: H<sub>2</sub>O = 1:100) to remove the native oxide, followed by thermal oxidation with an oxidized layer of 600nm. Then, a polysilicon film of 300nm was deposited at 625°C by low pressure chemical vapor deposition (LPCVD) system. Next, a 30-nm Nb<sub>2</sub>O<sub>5</sub> was deposited by RF reactive sputtering on the n-type silicon wafer at Ar: O<sub>2</sub> (20:5) ambient. Then, rapid thermal annealing (RTA) in O<sub>2</sub> ambient for 30 sec was done for temperatures ranging from 600°C to 900°C. A 300nm thick Aluminum was used for the back contact of the Si wafer. The sensing area was defined by the standard photolithography process and mounted on the copper by a silver gel. The detail EGFET structure is illustrated in Fig. 1.

## **3. Results and Discussion**

The XRD analysis in fig. 2 shows the strongest peak intensity at 700°C signifies a well-crystallized Nb<sub>2</sub>O<sub>5</sub> formed with a preferential orientation of the (180), (510), (112) and (381) plane of cubic Nb<sub>2</sub>O<sub>5</sub> structure because of a stronger and better-defined bonding (Nb-O).

Fig. 3 shows the XPS spectra demonstrating the O 1s peak intensity corresponding to Nb was increased upon increasing the RTA temperature at 700°C indicating the reaction of oxygen with Nb atoms forming a Nb<sub>2</sub>O<sub>5</sub> film.

The Atomic Force Microscopy (AFM) images in fig. 4 and fig. 5 shows the increment of the number of surface sites with surface roughness by raising the temperature.

Fig. 6 shows the normalized I-V curves showing sensitivity 47.90mV/pH in various pH values at 700°C determines the formation of a thinner low-k interfacial layer and thus a high density of surface hydroxyl groups.

The sample was subjected to a pH loop of 7-4-7-10-7 over a period of 25 minutes annealed at 700°C repairs the bonding and eliminates porous structures resulting in the least hysteresis deviation of 7.7 mV and the lowest drift rate of 4.53 mV/hr depicted in fig. 7 and 8.

The pNa and pK sensitivity of the film, RTA at 700°C, are 19.76mV/pNa and 17.4mV/pK, respectively. We compare the different ion sensitivity in fig. 9 showing the sensing film has more responsive to H<sup>+</sup> relative to Na<sup>+</sup> and K<sup>+</sup>.

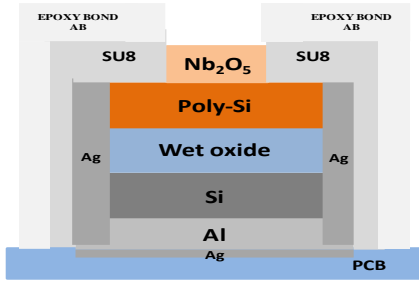
pUrea and pGlucose responses of enzyme-immobilized (Urease and Glucose Oxidase respectively) Nb<sub>2</sub>O<sub>5</sub> by covalent bonding are discussed in fig. 10 and 11 respectively. The hydrogen ions and ammonium produced by enzyme-catalyzed reaction ions may be used for urea detection leading to the changes of the output signal of the sensor. The glucose biosensor measured the glucose concentration by detecting the variation in pH caused by the generation of hydrogen ions by the dissociation of glucose acid.

## **4. Conclusions**

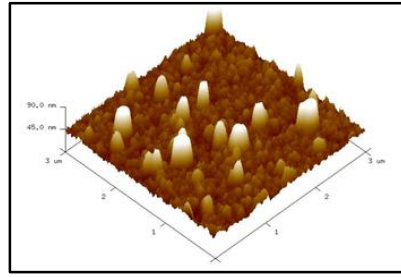
Finally, we may conclude that 700°C is the optimum annealing temperature to achieve high sensitivity, high linearity, low hysteresis voltage, and low drift rate for the high-k Nb<sub>2</sub>O<sub>5</sub>-EGFET device in different ionic as well as urea and glucose solutions and proves itself as an efficient biosensor.

## **References**

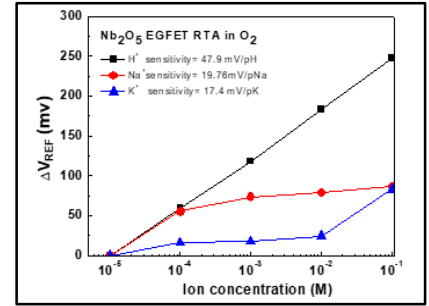
- [1] P. Bergveld, "Development of an ion-sensitive solid state device for neurophysiological measurement," IEEE Trans. Biomed. Eng., vol.17, pp.70-01, 1970.
- [2] P. Temple-Boyer, A. Benyahia, W. Sant, M.L. Pourciel-Gouzy, J. Launay, and A. Martinez, "Modelling of urea-EnFETs for haemodialysis applications", Sens. Actuators B, 131, pp. 525-532, 2008.



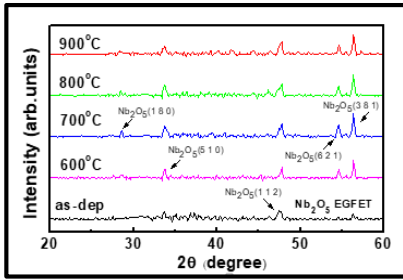
**Fig. 1** The Nb<sub>2</sub>O<sub>5</sub> EGFET structure



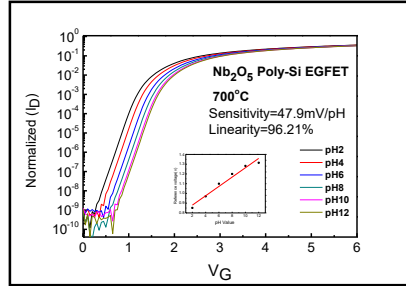
**Fig. 5** RTA700°C in O<sub>2</sub> R<sub>rms</sub>=9.88 (nm)



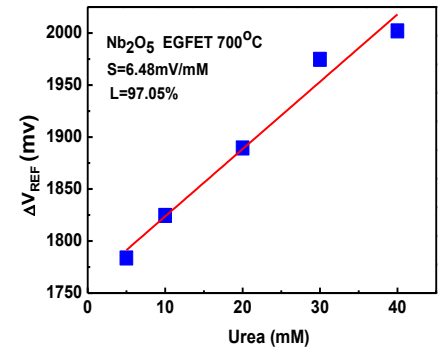
**Fig. 9** The different ion sensitivity of Nb<sub>2</sub>O<sub>5</sub> sensing membrane annealed 700°C in O<sub>2</sub> ambient



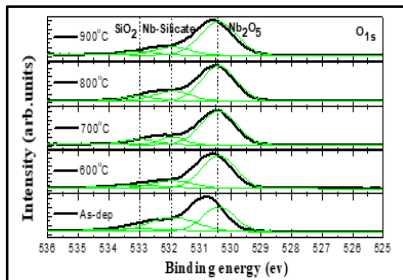
**Fig. 2** XRD of the Nb<sub>2</sub>O<sub>5</sub> film after Annealing at various temperatures on Poly crystalline silicon in O<sub>2</sub> ambient for 30 sec.



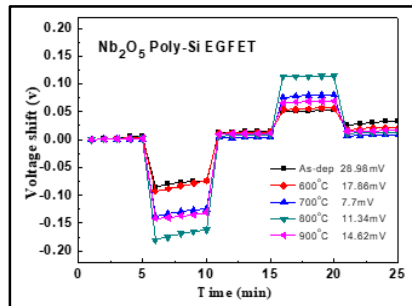
**Fig. 6** Normalized I-V curve at RTA 700°C in O<sub>2</sub> ambient. Inset shows Sensitivity and linearity.



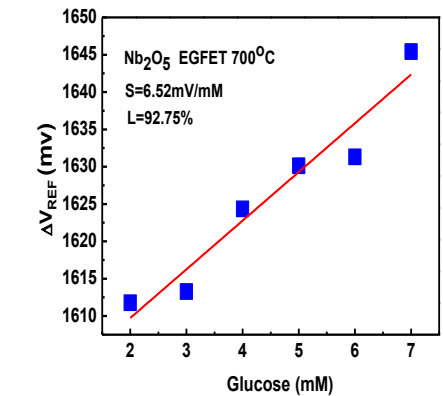
**Fig. 10** pUrea-responses of enzyme-immobilized Nb<sub>2</sub>O<sub>5</sub> (the film was annealed at 700°C in O<sub>2</sub> ambient) EGFET structure by covalent bonding method.



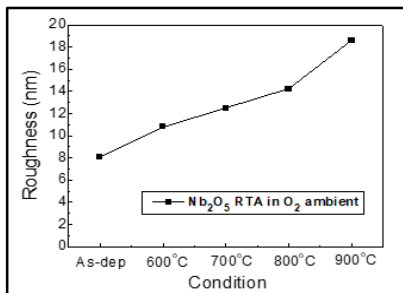
**Fig. 3** XPS results of Nb<sub>2</sub>O<sub>5</sub> film (a) Nb 3d (b) O 1s in O<sub>2</sub> annealing



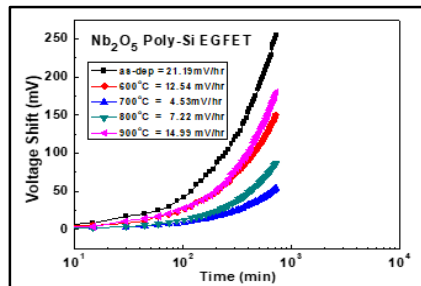
**Fig. 7** The hysteresis of Nb<sub>2</sub>O<sub>5</sub> film with various RTA temperatures during the pH loop of 7→4→7→10→7 over a period of 25 minutes



**Fig. 11** pGlucose-responses of enzyme-immobilized Nb<sub>2</sub>O<sub>5</sub> (the film was annealed at 700°C in O<sub>2</sub> ambient) EGFET structure by covalent bonding method.



**Fig. 4** AFM of high-k Nb<sub>2</sub>O<sub>5</sub> surface on poly crystalline silicon after RTA at different annealing temperatures



**Fig. 8** the drift voltage of Nb<sub>2</sub>O<sub>5</sub> sensing membrane annealed with various RTA Temperatures.