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A NEW APPROACH TO LOW DRIFT Ta₂O₅ pH-ISFET BY RF REACTIVE SPUTTERING AND SOL-GEL METHOD

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SUMMARY

Thin films, such as SiO₂, ZrO₂, Al₂O₃, Si₃N₄ and Ta₂O₅, were known as hydrogen ion sensing membranes for pH-ISFET[1]. Among them, Ta₂O₅ thin film was reported to have the better sensing characteristics than those of other hydrogen ion sensing membranes.

In this research, the Ta₂O₅ hydrogen ion sensing membrane were formed by RF reactive sputtering and Sol-Gel method. Ta₂O₅ pH-ISFETs by RF reactive sputtering and Sol-Gel method showed a good linearity in wide pH range(pH 2 to 12) and had high sensitivities of 58~59mV/pH and 57mV/pH, respectively. Especially, Ta₂O₅ pH-ISFET by RF reactive sputtering showed stable long-term sensitivity(57~59mV/pH) during 45 days and long-term stability during 60 days (0.05pH/day).

RF REACTIVE SPUTTERING METHOD

Table 1 is the optimum conditions for the formation of the Ta₂O₅ membrane by RF reactive sputtering. The conditions were determined from investigation on the physical properties(refractive index, etching ratio, dielectric constant and composition ratio) and electrical properties (C-V characteristics and leakage current) of the

RF power	50W
Working pressure	50mtorr
Gas mixture ratio(Ar/O ₂)	4 : 1 sccm
Substrate temp.	200°C
Annealing conditions	O ₂ , 400°C, 1hr

Table 1. Ta₂O₅ sputtering and annealing conditions[2].

thin films.

Hydrogen ion sensing characteristics of the Ta₂O₅ pH-ISFET by RF reactive sputtering is shown in figure 1. This sensor showed a good linearity within the wide pH range (pH 2 to 12) and high sensitivity (about 59mV/pH), which was very close to the theoretical Nernst potential.

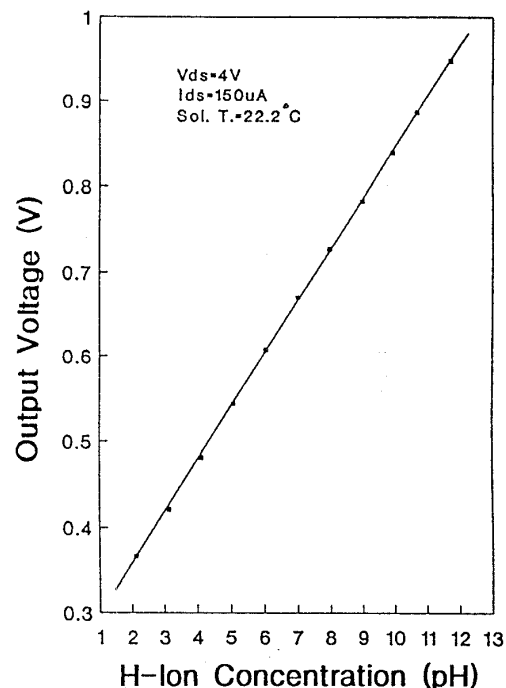


Figure 1. Hydrogen ion sensing characteristics of Ta₂O₅ pH-ISFET by RF reactive sputtering.

The long-term stabilities of the pH-ISFET measured in the pH 7 solution are shown in figure 2. The pH-ISFET annealed by conditions shown in table 1 had the best stability of about 0.05pH/day during 60 days. XRD analysis showed that the crystal

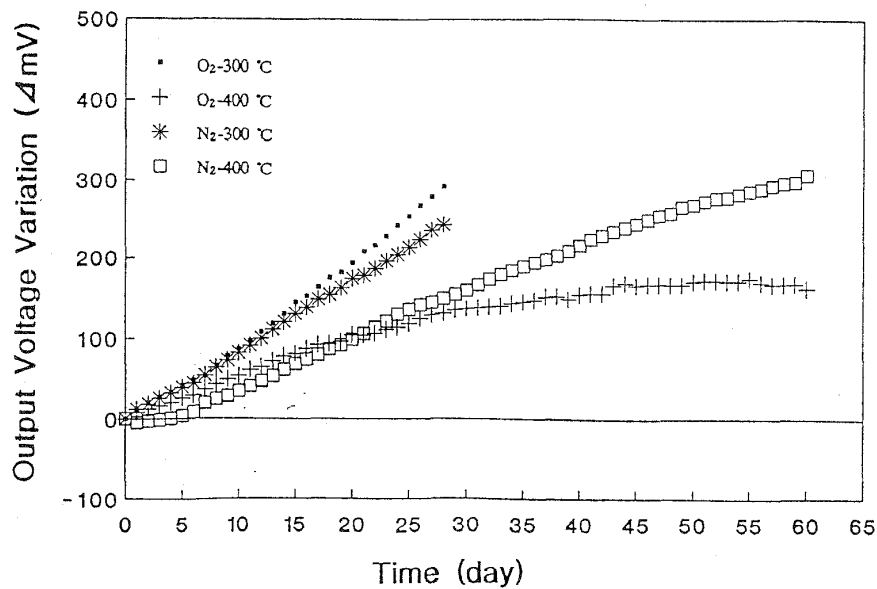


Figure 2. Long-term stabilities of the Ta_2O_5 pH-ISFET by RF reactive sputtering.

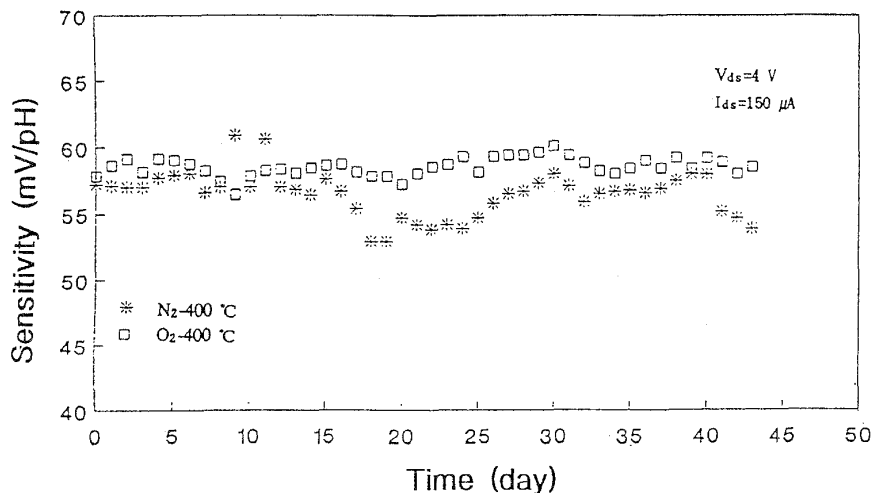


Figure 3. Long-term sensitivities of Ta_2O_5 pH-ISFET by RF reactive sputtering.

structure of the annealed Ta_2O_5 film was changed from amorphous to poly-crystalline structure in the vicinity of $500^\circ C$.

As shown in figure 3, the long-term sensitivities of pH-ISFET annealed in O_2 ambient was maintained at about $57\sim 59 mV/pH$, while that of pH-ISFET annealed in N_2 ambient much varied with time.

SOL-GEL METHOD

From the previous results, we can see that the

Ta_2O_5 gate pH-ISFET by RF sputtering has the better sensing characteristics than the results of the reference[3], but it needs to be still improved in the long-term stability. The dominant factor of the drift will be the hydration or memory effect of the membrane[4]. If the ultra-thin membrane is formed, paradoxically the hydration will be completed within a very short time, and the above-mentioned problems may be solved. In this work, we tried to form the ultra-thin Ta_2O_5 membrane by using Sol-Gel method. The solution for the fabrication of Ta_2O_5 films is prepared as

described in figure 4. The prepared solution was spin-on coated on the Si_3N_4 gate pH-ISFET and heat-treated at 400°C to remove the remaining organics. The membrane formed with this process had ultra-thin thickness (about 100\AA), which was measured by ellipsometric method. Figure 5 is XRD patterns of Ta_2O_5 film according to heat treatment conditions. The film showed an amorphous structure with heat treatment temperature below 600°C , while poly-crystalline structure was formed by heat treatment at 800°C in the O_2 ambience. We determined heat treatment conditions to be 400°C (O_2 ambience, 1 hour) because the leakage currents began to increase in the films annealed above 400°C . Figure 6 is an AES depth profile of Ta_2O_5 thin film heat-treated at 400°C . This figure shows that oxygen, the sensing site for hydrogen ions, is more rich at the surface than in the bulk.

Hydrogen ion sensing characteristics of Ta_2O_5 pH-ISFET by Sol-Gel method are shown in figure 7. This sensor showed a good linearity in the wide pH range (pH 2 to 12) and high sensitivity (about 57mV/pH). From these results, we see that Ta_2O_5 thin film by Sol-Gel method can be applied to hydrogen ion sensing membrane.

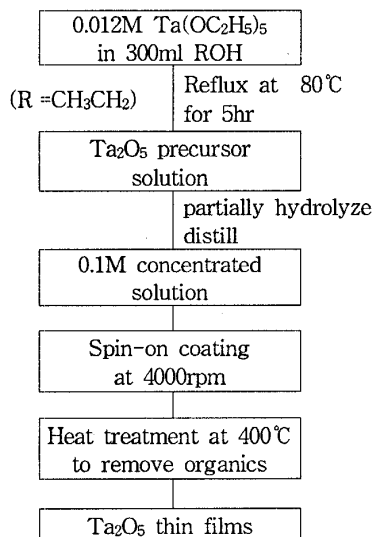


Fig. 4. Flow diagram for the fabrication of Ta_2O_5 thin films

CONCLUSION

The Ta_2O_5 pH-ISFET by RF reactive sputtering had a good linearity in the wide pH range (pH 2 to 12), high sensitivity (about $58\sim 59\text{mV/pH}$), stable long-term stability (about 0.05 pH/day), and good

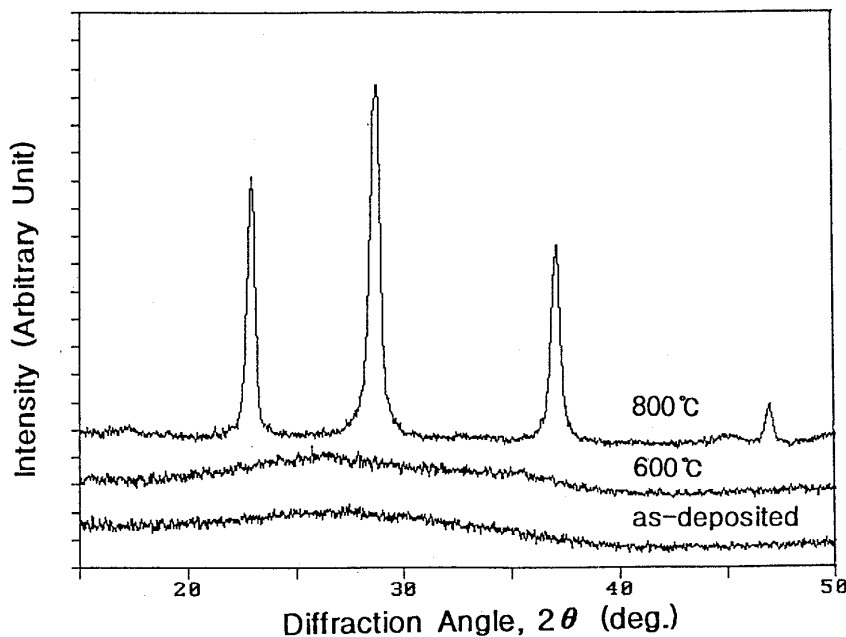


Figure 5. XRD patterns of Ta_2O_5 films

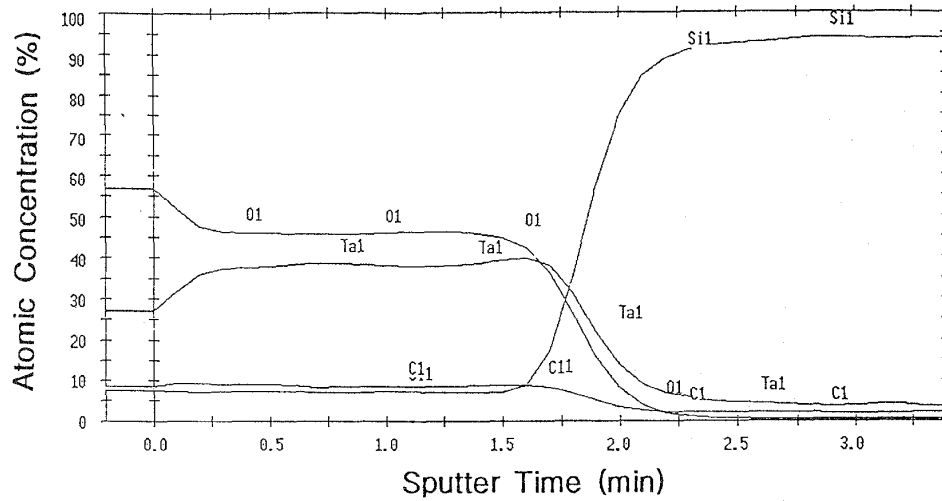


Figure 6. AES depth profile of Ta_2O_5 films

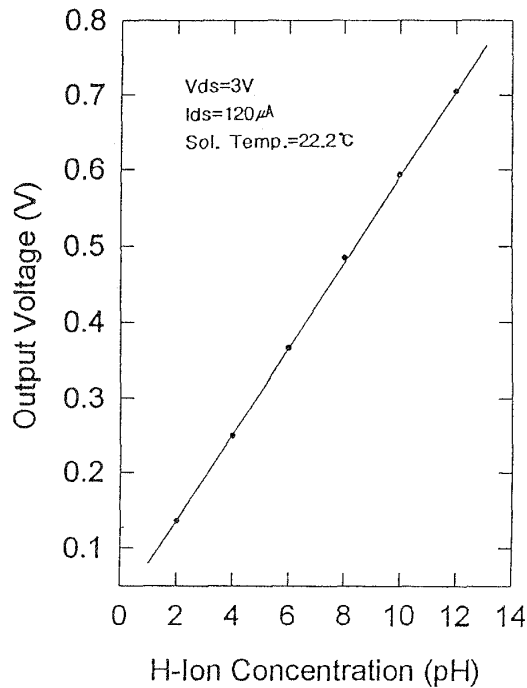


Figure 7. Hydrogen ion sensing characteristics of Ta_2O_5 pH-ISFET by Sol-Gel method.

long-term sensitivity(57~59mV/pH). These results were better than those of previously reported papers[3].

In this research, we tried to form ultra-thin Ta_2O_5 membrane for improving the drift of pH-ISFET by Sol-Gel method. By introducing Sol-Gel method, Ta_2O_5 film having about 100Å thick was formed. This sensor showed a good linearity and high sensitivity(about 57 mV/pH) in the range of pH 2 to 12. From above result, we see that Ta_2O_5 membrane by Sol-Gel method can be used in the hydrogen ion sensing membrane.

More detailed studies are now in progress to improve the long-term stability by Sol-Gel method.

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