# Vanadium Dioxide Films Deposited on SiO<sub>2</sub>- and Al<sub>2</sub>O<sub>3</sub>-coated Si Substrates Using Reactive RF-Magnetron Sputter Deposition Technique

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### 1. Introduction

Vanadium dioxide (VO<sub>2</sub>) has been known as a Mottinsulator showing an abrupt metal-insulator transition (MIT) near  $68^{\circ}$ C [1]. The metallic high-temperature phase of VO<sub>2</sub> is tetragonal, whereas its insulating phase is monoclinic. It has recently been reported that the MIT temperature can be varied by applying electric field or by irradiating infrared light [2]. The variability provides a large potential to MIT materials, such as VO<sub>2</sub>, for programmable thermal or photo sensors.

The earlier works reported that VO<sub>2</sub> films deposited by pulsed laser deposition (PLD) method revealed superior quality compared to the films deposited by other techniques [3]. However, PLD is not appropriate for manufacturing practical devices on a large area substrate. RF-magnetron sputter deposition technique is considered as one of the most promising techniques for large-area uniform deposition with high packing density and strong adhesion to underlying layer. The use of Si wafer as substrate is also very useful for cost-effective fabrication of sensors. However, VO<sub>2</sub> films deposited on amorphous SiO<sub>2</sub>-coated Si substrate have been known to show inferior quality to that deposited on a single crystalline sapphire substrate. Therefore, it is required to develop a sputter deposition process of VO2 films on amorphous films for the production of commercial sensor devices.

In the present work, the deposition of VO<sub>2</sub> film on Si wafer coated with a thermally grown amorphous-SiO<sub>2</sub> film (SiO<sub>2</sub>/Si) or amorphous Al<sub>2</sub>O<sub>3</sub> film (Al<sub>2</sub>O<sub>3</sub>/Si) was investigated using reactive RF-magnetron sputter deposition technique. The VO<sub>2</sub> film quality sensitively depends on the oxidation environment during deposition. In this work, the effect of operating pressure, that is one of process parameters governing oxidation ambient, on the film quality was evaluated to obtain VO<sub>2</sub> films showing abrupt MIT behavior. The dependence of film characteristics on the underlying layer was also investigated.

### 2. Experiments

The SiO<sub>2</sub> film was formed on Si wafer by thermal oxidation at  $925^{\circ}$ C and amorphous Al<sub>2</sub>O<sub>3</sub> film on Si was deposited at 150°C using plasma-enhanced atomic layer deposition (PEALD) technique. The reactive sputter deposition of VO<sub>2</sub> films was carried out using V-metal

target of 4-inch diameter (99.9%, Kojyundo Kagaku) in the atmosphere of  $O_2$  gas mixed with Ar. The  $O_2$  gas fraction was 12.3 %, and the operating pressure was varied from 2 to 30mtorr. The RF power was 300W. During deposition, the substrate was rotated for uniform deposition and heated by IR-lamps. The films were post-annealed at 490°C with  $O_2$  flow rate of 50 sccm and pressure of 30 mtorr. The thickness of VO<sub>2</sub> films was approximately 110 nm. The resistance was measured by standard four-point probe method.

### 3. Results and Discussion

Although VO<sub>2</sub> film easily coexists with and is converted to different vanadium oxides, such as V<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>, and V<sub>3</sub>O<sub>7</sub> etc., only VO<sub>2</sub> reveals MIT at 68°C. Therefore, MIT characteristics, such as abruptness and magnitude of resistance change near 68°C, have been considered to be measures of VO<sub>2</sub> film quality.

First, the resistance (R) of VO<sub>2</sub> films fabricated on SiO<sub>2</sub>/Si was measured with increasing temperature (T), and a typical R-T curve showing abrupt resistance change near 68°C is illustrated in Fig. 1(a). The film was deposited at a deposition temperature (T<sub>s</sub>) of 400°C with the pressure of 5 mtorr and post-annealed at a annealing temperatue (T<sub>a</sub>) of 490°C. The R-T curve in Fig. 1(a) demonstrates the VO<sub>2</sub> film of a quality good enough to be used in practical sensor devices [2]. The ratio of resistances at 27 and 97°C,  $\Delta$ R, was approximately 1.3 x 10<sup>4</sup>. The value is as large as that of VO<sub>2</sub> films deposited on c-sapphire [4].



Figure 1. R-T curves of VO<sub>2</sub> films deposited (a) on SiO<sub>2</sub>/Si at  $400^{\circ}$ C;(b) on Al<sub>2</sub>O<sub>3</sub> at  $450^{\circ}$ C (pressure:5mtorr; T<sub>a</sub>: 490°C)

Figure 1(a) also shows the hysteresis between R-T curves obtained while heating and cooling. The thermal hysteresis is attributed to the evolution and absorption of latent transition heat [5]. The discrepancy between two curves is less than 10°C.

In Fig. 1(b), the R-T curve of VO<sub>2</sub> film on Al<sub>2</sub>O<sub>3</sub>/Si is compared to that on SiO<sub>2</sub>/Si annealed at the same condition. The inferior abruptness and magnitude of MIT of VO<sub>2</sub> film on Al<sub>2</sub>O<sub>3</sub> is thought to be the low density of underlying Al<sub>2</sub>O<sub>3</sub> layer deposited at 150°C compared to SiO<sub>2</sub> film formed at 925°C.

The effect of operating pressure on resistance change of VO<sub>2</sub> films by MIT was evaluated as shown in Fig. 2. The earlier works on the sputter deposition of VO<sub>2</sub> films reported that the use of V-metal target revealed very narrow process window, and recommended to rather use V<sub>2</sub>O<sub>3</sub> or V<sub>2</sub>O<sub>5</sub> target [6]. However, the present work indicated that VO<sub>2</sub> films of  $\Delta R > 6.3 \times 10^3$  could be obtained with a relatively wide range of pressure, 5 – 25mtorr, in the deposition process, as shown in Fig. 2. The wide process window of the parameter governing the oxidation ambient is very important to establish a reproducible fabrication process of VO<sub>2</sub> film.



Figure 2. The resistance change,  $\Delta R$ , of VO<sub>2</sub> films deposited on SiO<sub>2</sub>/Si at T<sub>s</sub> of 400°C.



Figure 3. SEM photographs of  $VO_2$  films as-deposited with operating pressures of (a) 5 mtorr and (b) 20 mtorr.

In Fig. 3, the surface microstructure of 110nm-thick films as-deposited at 400°C was observed using the scanning electron microscopy (SEM). The images show crystalline submicron-grains having rectangular or hexagonal facets. The roughness was also measured using atomic force microscopy (AFM). The rms roughnesses of

 $VO_2$  films deposited on SiO<sub>2</sub>/Si with operating pressure of 5 and 20 mtorr were 15.9 and 20.4 nm, respectively. The roughness is higher than that (7 - 8 nm) of a VO<sub>2</sub> film formed by sol-gel method. The rough surface morphology of VO<sub>2</sub> films shown in Fig. 3 might be due to the formation of polycrystalline grains during deposition. The roughness was not considerably increased by post-annealing.



Figure 4. SEM photographs of VO<sub>2</sub> films as-deposited on Al<sub>2</sub>O<sub>3</sub>/Si with operating pressures of (a) 5 mtorr (O<sub>2</sub>: 12.3%;  $T_s$ : 450°C) and (b) 20 mtorr (O<sub>2</sub>: 9.1%;  $T_s$ : 400°C).

Figure 4 illustrates SEM images showing the surface morphology of VO<sub>2</sub> films as-deposited on Al<sub>2</sub>O<sub>3</sub>/Si. Although the underlying Al<sub>2</sub>O<sub>3</sub> layer was amorphous, grains well-ordered toward two directions as indicated by arrows are observed. The rms rougness of the sample in Fig. 4(b) was 3.4 nm. Further study is required to clarify the effect of underlayer on the grain growth of VO<sub>2</sub> film.

## 4. Conclusion

The present work demonstrated that the reactive RFmagnetron sputter deposition technique is very promising for depositing VO<sub>2</sub> films on amorphous layers on Si. VO<sub>2</sub> films showing  $\Delta R$  as large as 1.3 x 10<sup>4</sup> could be obtained on SiO<sub>2</sub>/Si substrate after post-annealing at 490°C. In the deposition process using V-metal target, VO<sub>2</sub> films of  $\Delta R$ > 6.3x10<sup>3</sup> could be obtained in the operating pressure range as wide as 5 – 25 mtorr.

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