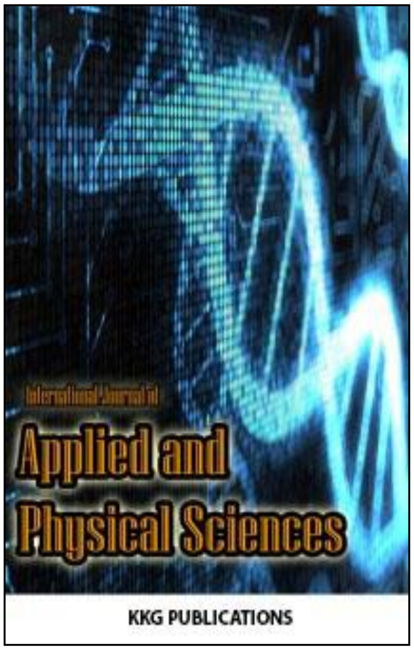

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MEASUREMENT OF THERMAL DIFFUSIVITY OF V_2O_5 THIN FILMS USING NANOSECOND THERMOREFLECTANCE TECHNIQUE

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Abstract. The nanosecond thermoreflectance technique is a very useful method to measure the thermophysical properties of thin films because of rapid measurement of changed reflectance by generated heat in the thin film caused by a nanosecond pulse laser. In this study, the nanosecond thermoreflectance system based on rear heating-front detection technique using pulsed DPSS laser was composed, and the thermal diffusivity of vanadium pentoxide (V_2O_5) thin films were measured using this system. V_2O_5 thin films are applied as a chemical sensor, thermometer, or thermal imaging sensor since its outstanding chemical, electrical, and thermal properties. V_2O_5 is the most stable compound among the vanadium oxide systems, and when V_2O_5 thin film is growing by sputtering, it is crystallized with orthorhombic structure at 773 K temperature. Crystallized V_2O_5 film shows metal-insulator transition (MIT) phenomenon near 550 K. The structural properties of V_2O_5 thin film samples with a thickness of 200 and 244 nm grown by RF magnetron sputtering were verified using SEM, XRD, and Raman spectrum. The thermal diffusivities of 200, 244 nm thickness samples were measured from 300 K to 680 K, and the values at 300 K were 1.67×10-7 m2/s and 1.87×10-7 m2/s, respectively, and they were not changed until to 590 K. However, at 620 K, the values suddenly increased to 7.33×10-7 m2/s and 13.2×10-7 m2/s, respectively. From this result, we think that the MIT of V_2O_5 thin films occurred at about 590 K.

INTRODUCTION

Thermal diffusivity is one of the main thermophysical properties of materials therefore, for the accurate analysis of material's thermal property, the thermal diffusivity have to be measured precisely. Especially, as the thermal diffusivities of bulk and thin film are completely different [1], [2] the measurement technique proper to the state of material is required. [3] developed thermoreflectance technique firstly using a picosecond laser for the accurate measurement of thermal diffusivity of thin film [3]. In this method, changed reflectance by generated heat in the thin film caused by instantaneously short pulse laser beam is measured. This technique is called as the most proper method because of high accuracy and fast measurement.

In this study, the nanosecond thermoreflectance system based on rear heating-front detection technique using DPSS(diode-pumped solid-state) laser was composed, and the thermal diffusivity of vanadium pentoxide(V_2O_5) thin films was measured. Since the outstanding thermal, chemical and electrical properties, the V_2O_5 thin film can be applied to chemical sensor, temperature measurement sensor, and thermal detection image sensor etc. [4], [5] and [6]. V_2O_5 is the most stable compound among the vanadium oxide systems [7], [8] and when V_2O_5 thin film is growing by sputtering, it is crystallized with orthorhombic structure at 773 K. Crystallized V_2O_5 film shows metal-insulator transition (MIT) phenomenon near 553 K [9], [10], [11] and [12] therefore, in order to observe the variation of thermal diffusivity caused by the MIT phenomenon, the thermal diffusivities of 200, 244 nm thickness thin film samples were measured from 300 K to 680 K.

MEASUREMENT THEORY

In the Parker's thermal diffusivity analysis method, because the radiation heat loss caused by the heating of thin films was not considered, the fitting of real data containing the radiation heat is impossible in full time region. For this reason, to consider the radiation heat loss, Cape and Lehman corrected the Park's equation as follow [13] and [14];

$$T(t) = \Delta T \sum_{n=1}^{\infty} (A_n) \exp\left(- X_n^{\ 2} \frac{t}{\tau_0} \right) \tag{1}$$

here

$$\begin{aligned} A_n &= 2(-1)^n X_n^{\ 2} \left(X_n^{\ 2} + 2Y + Y^2 \right)^{-1}, \\ X_0 &= (2Y)^{\frac{1}{2}} \left(1 - \frac{1}{12} Y + \frac{11}{1440} Y^2 \right)^{-1}, \\ X_n &= n\pi + \frac{2Y}{n\pi} + \frac{4Y^2}{(n\pi)^3} \\ &\quad + \left[\frac{16}{(n\pi)^5} - \frac{2}{3(n\pi)^3} \right] Y^3 \\ &\quad + \left[-\frac{80}{(n\pi)^7} - \frac{16}{3(n\pi)^5} \right] Y^4 \qquad (n \geq 1) \end{aligned}$$

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Using the non-dimensional parameter of biot number, Cape and Lehman the radiation heat loss occurred in the film was corrected. Because the fitting in full time region can be possible by the Cape-Lehman's corrected equation, the accurate thermal diffusivity values can be obtained from the thermoreflectance data.

EXPERIMENTAL DETAILS

Composition of Nanosecond Thermoreflectance System

For the measurement of thermal diffusivity of V_2O_5 thin film samples, the rear heating-front detection nanosecond thermoreflectance system was composed as Fig. 1, The pulsed DPSS laser (1064 nm wavelength, Eos IR, Innolight) was used as heating source and 785 nm CW laser diode (LP785-SF20, Thorlabs) was used as a probe laser for the detection of reflectance change in the sample film. The sample films were fixed to the holder in vacuum chamber (VPF-700, Janis research). The variation of probe laser reflectance was detected by oscilloscope (DPO7254C, Tektronix) and the thermal diffusivity was obtained after fitting the data by Cape-Lehman equation.

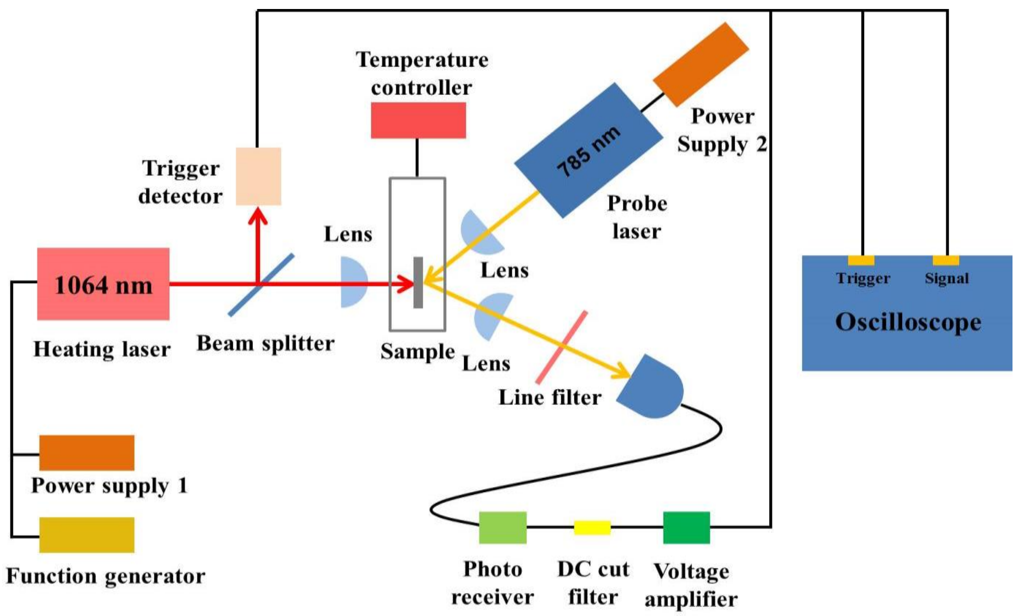

Fig. 1. Schematic diagram of the rear heating-front detection nanosecond thermoreflectance system.

Preparation of V_2O_5 Thin Films

The V_2O_5 thin films were deposited on quartz substrate by RF magnetron sputtering using 10 cm vanadium metal target. Ar(99.999%) and O2(99.999%) gases were used and for the crystallization of V_2O_5 thin film the substrate temperature was maintained as 500 ℃. To deposit two thicknesses film samples, the sputtering was carried out for 180 and 240 minutes. The thicknesses of films were investigated by Ellipsometer (Uvisel, Horiba Jobin-Yvon) and found as 200 and 240 nm.

Because of high transmittance of V_2O_5, the measurement is difficult therefore, for the absorption of laser beam from rear surface and reflection from front surface, aluminum thin films with thickness of 50 nm were coated on both sides of V_2O_5 layer as Fig. 2.

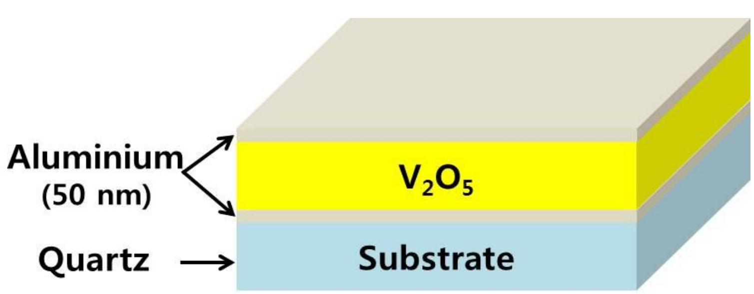

Fig. 2. The structure of measured film sample.

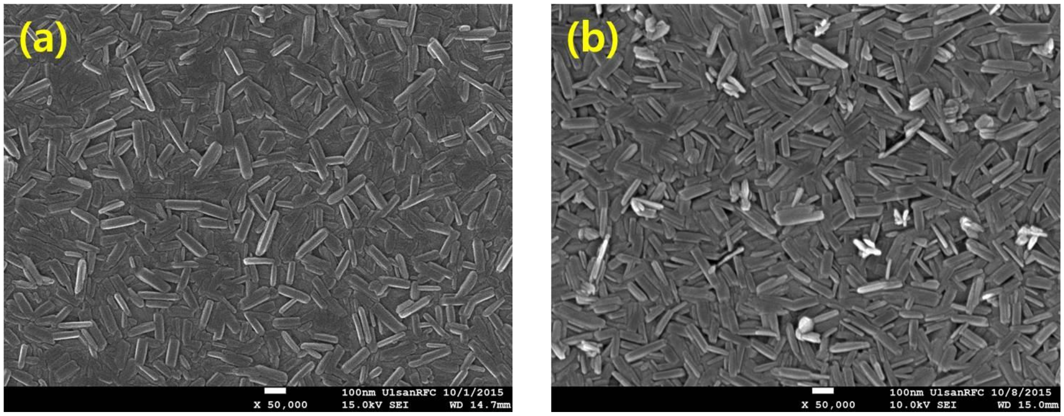

Fig. 3. The SEM microphotograph of deposited V_2O_5 thin film samples. (a) 200 nm and (b) 244 nm.

RESULTS AND DISCUSSION

The structures of deposited V_2O_5 thin films were characterized by SEM, XRD, and Raman spectrum. Figures 3(a) and 3(b) are SEM microphotographs of 200 nm and 240 nm film samples, respectively and both shows nearly uniform rod-like crystallines with 111 nm length and 24 nm width

The XRD spectra are shown in Fig 4 and both spectra show (001) peak near 20.53°. This peak appears from orthorhombic structure of α-V_2O_5. From this result, it was identified that the sample films were well crystallized with orthorhombic structure. Figure 5 is the Raman spectra of deposited V_2O_5 thin films.

All the observed peaks in Raman spectra have direct relations with the film structure and by analyzing these peaks, it was also identified that the samples were well crystallized c-axis α-V_2O_5 films with orthorhombic structure.

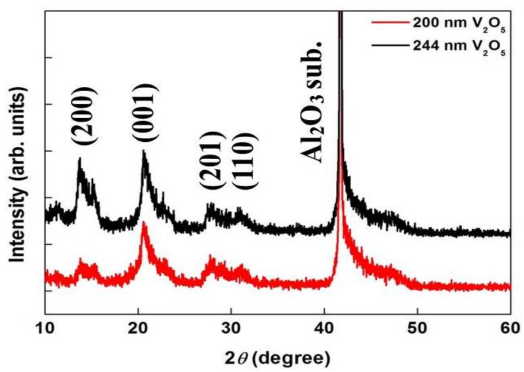

Fig. 4. The XRD spectra of deposited V_2O_5 thin film samples

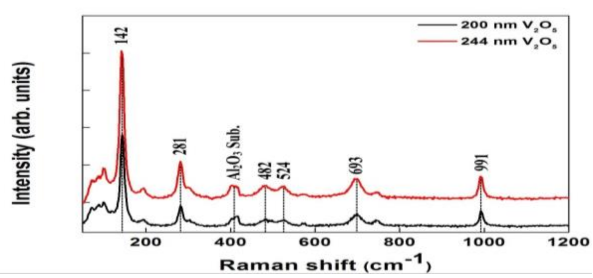

Fig. 5. The raman spectra of deposited V_2O_5 thin films.

The thermal diffusivities of 200 and 244 nm thickness samples were measured from 300 K to 680 K. The thermoreflectance signal from the film sample was detected and the thermal diffusivity was obtained by fitting the detected signal using the Cape-Lehman model. Figure 6 shows the measured and well fitted thermoreflectance signals at 300 K. The thermal diffusivities of 200 nm- and 244 nm-thick V_2O_5 films were 1.67×10-7 m2/s and 1.87×10-7 m2/s, respectively. To investigate the temperature-dependent thermal diffusivity of the V_2O_5 thin film, we measured the thermoreflectance signal over the temperature range of 300-680 K, and the result is shown in Fig. 7. The thermal diffusivities of both V_2O^5 films do not nearly change up to 590 K. However, at 620 K, the values abruptly increased to 7.33×10-7 m2/s for 200 nm-thick film and 13.2×10-7 m2/s for 244 nm-thick film, respectively. This result is regarded as an MIT in V_2O_5 film [9]-[10].

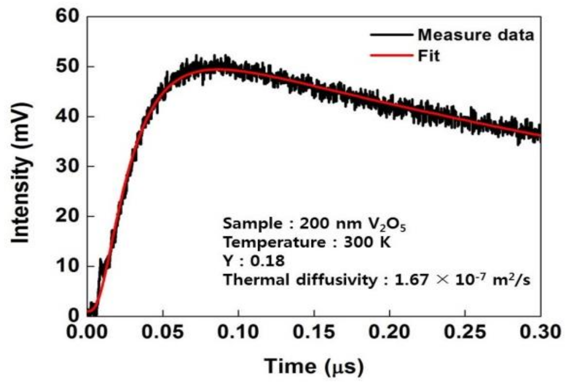

Fig. 6. Measured and fitted thermoreflectance signals of the 200 nm-thick V_2O_5 film at 300 K.

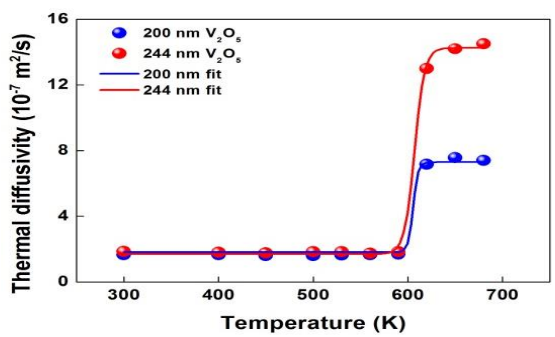

Fig. 7. Temperature-dependent thermal diffusivities of V_2O_5 thin films with thicknesses of 200 nm and 240 nm.

The abrupt change in the thermal diffusivity between the 590 and 620 K implies that the films undergo a transition to the metallic state. From our result, we conclude that the MIT in V_2O_5 thin film occurs near 590 K.

CONCLUSION

In this study, the temperature dependent thermal diffusivity of sputtered V_2O_5 thin films with thicknesses of 200 nm and 240 nm on quartz substrate were measured using the composed nanosecond thermoreflectance system from 300 K to 680 K.

The obtained thermal diffusivities at 300 K were 1.67×10^{-7} m^2/s and 1.87×10^{-7} m^2/s, respectively and they were not changed until to 590 K. However, at 620 K, the values suddenly increased to 7.33×10^{-7} m^2/s and 13.2×10^{-7} m^2/s, respectively. From this result, we think that the MIT of V_2O_5 thin films occurred at about 590 K and this technique can be widely utilized to analyze the thermophysical properties of various functional thin films.

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