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Growth and infrared switching properties of deposited VO_2 films at various sputtering power with a VO_2 target by RF magnetron sputtering

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Abstract. In this work, structural and morphological properties as well as phase transition temperature and hysteresis width of VO_2 thin films grown with a VO_2 target by RF magnetron sputtering were studied under the influence of relatively low sputtering power of 80 – 140 W. It was observed that as sputtering power increased, the crystallinity of the films improved with the presence of obvious diffraction peaks of VO_2, and a slight increase in the average crystallite and grain sizes of the films. The deposited films revealed a slight change in infrared transmittance during heating and cooling cycles. Films deposited at sputtering power of 140 W exhibited the lowest Tt of 60.5 °C with a hysteresis width of 41.0 °C.

1. Introduction

Vanadium dioxide or Vanadia (VO_2), due to intelligent thermochromic properties, has received a great attention over the last few decades. The properties make it very beneficial for the electrochemical energy storage, smart windows as well as aerospace and microelectronics industries [1-2]. However, studies on optical properties such as infrared transmittance, and surface properties such as morphology, topography and crystal structure, of VO_2 thin films are crucial for those applications [3-4].

Many investigations on preparations and developments of VO_2 thin films have been reported [5]. For example, studies on surface and optical properties of direct-current (DC) [6-7] and radio-frequency (RF) [8-9] magnetron sputtered VO_2 thin films have been investigated. VO_2 films have also been prepared using RF magnetron sputtering to study its structural, morphological, optical and thermochromic properties for the application of solar control coatings [10]. Meanwhile, VO_2 thin films deposited with high sputtering power of 330 – 400 W by RF magnetron sputtering have also been studied [11]. Control of sputtering power was highly significant to achieve high purity monoclinic VO_2 films. In addition, at such high sputtering power of 400W, target poisoning happened due to a chemisorption of oxygen on the surface of the target causing a reduction in deposition rate and film thickness with large grain size, and an increase in formation of defects. Thus, this leads to films with porous structure [12]. Hence, in order to prevent porous films, this present work will study depositions of VO_2 films at low sputtering power to ensure pores filled during the film depositions.

This work will investigate the influence of low sputtering power of 80 – 140 W on structural, morphological and infrared switching properties of VO_2 films grown with a VO_2 target by RF magnetron sputtering, which has not been considered yet.

2. Details of experiments

2.1. Preparation of films

The method for preparing VO_2 films was reported as in our previous papers [6,10]. VO_2 films were prepared on Corning 2947 glass substrates by RF magnetron sputtering using a high purity VO_2 target (99% purity) of 76 mm diameter and 5 mm thickness. The substrates were ultrasonically cleaned with ethanol for about 15 min and rinsed with distilled water. The substrates were then blow-dried using high purity nitrogen gas (99.9% purity), and screwed onto a substrate holder in the sputtering chamber. Commercial high purity argon, Ar (99.9% purity) was supplied into the chamber as the sputtering gas. The sputtering chamber was evacuated to 10^{-4} Pa by a turbomolecular pump that was backed by a mechanical pump. The Ar gas flow was kept constant at a gas flow rate of 40 sccm by a mass flow controller. Before depositing VO_2 onto the substrates, Pre-sputtering of the VO_2 target in Ar atmosphere was performed for 10 min to eliminate any oxide layers and impurities. The substrates were rotated at 10 rpm throughout the film deposition in order to get films with uniform thickness. The film thickness during deposition was maintained at 50 nm and monitored using a quartz crystal thickness meter with 1 Angstrom (Å) resolution reliably supplied by Inficon (Model SQM-160). During sputtering, the chamber pressure and substrate temperature were 10^{-1} Pa and 500 °C, respectively. The films were grown on the substrates at various sputtering power of 80 – 140 W.

2.2. Characterization of films

A surface profilometer (Alpha-Step IQ) was employed to ensure film thickness of the deposited VO_2 films by applying a stylus force of 0.03 N with measurement accuracy of ± 1.5 nm. Crystal structure of the films was analyzed by grazing incidence X-ray diffraction (GIXRD) on a Shimadzu XRD-7000 using monochromatic high-intensity Cu Kα (λ = 1.54056Å) radiation operated at 40 kV and 30 mA. GIXRD patterns of the films were detected in the range of 2θ = 20 – 40° and an incident angle of 1.0 ° at a scanning rate of 2 °/min. Surface morphology was studied by field-emission scanning electron microscopy (FE-SEM) using a JEOL JSM-7610F field-emission scanning electron microscope. The infrared switching properties, i.e. phase transition temperature and hysteresis width in infrared region, were recorded using a CARY 5000 UV-Vis-NIR spectrophotometer (Agilent Technologies) assembled with a SPECAC 4000 Series™ high stability temperature controller. The temperature-dependent transmittance spectra were taken at a wavelength of 2.5 μm with temperature varied from room temperature of 25 °C to 100 °C.

3. Results and discussion

3.1. Crystal structure

The GIXRD patterns of the VO_2 thin films deposited at sputtering power of 80 – 140 W are detected as shown in figure 1. It could be seen that all films have an obvious diffraction peak at 2θ = 27.9 ° of monoclinic VO_2(M) phase. Thus, it can be deduced that the deposited films have a prominent peak of VO_2(M) corresponding to (011) orientation plane. The peak enhanced with increasing sputtering power. As the sputtering power stepped-up from 100 to 140 W, another peak appeared at 2θ = 29.2 ° of metastable VO_2(B) phase. When the sputtering power was 80 W, the deposition rate was 0.72 nm/min. The films were found to have low film crystallinity as seen from the low peak intensity. This was also reported by Ahmadipour et al. [13], in which the low sputtering power leads to very low formation energy which was insufficient to cause production of a high crystalline thin film. As the sputtering power rose up to 140 W, the deposition rate seemed to increase up to 1.32 nm/min. This might be due to more energetic particles bombarded with high driving energy and settled on suitable lattice sites resulting in high film crystallinity [14]. In addition, the average VO_2 crystallite size can be estimated using the (011) crystallite orientation and Scherrer formula. The average crystallite size of the films at sputtering power of 80, 100, 120 and 140 W was 14.9, 25.6, 39.0 and 43.1 nm, respectively. It has also been reported by several studies that enhancement of crystallite size was obtained by increasing the RF sputtering power [13,15-16].

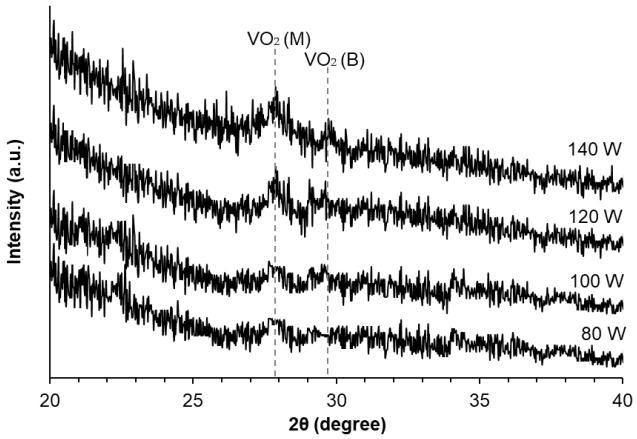

Figure 1. GIXRD patterns of monoclinic VO_2(M) and metastable VO_2(B) thin films at sputtering power of 80 – 140 W.

3.2. Surface morphology

Figure 2 represents the FESEM images of the films at sputtering power of 80 – 140 W. Spherical-like grains in VO_2(M) phase are grown on the film surface with a slight increment in the grain size of the VO_2 as the sputtering power increased. Irregular and dark areas, which indicate deep and wide trenches, are seen on the surface of the films grown at sputtering power of 80 W. As the sputtering power stepped-up, from 100 to 140 W, uniform grain distributions are observed on the film surface. It has been reported that, at high sputtering power, more ionized Ar gas particles bombarded on the VO_2 target, and hence more energetic target atoms are dominant causing the formation of nuclei on the film surface [14,16]. Thus, this indicates more sites for growth of the grains. In other words, when the energetic atoms implanted into the surface of the films, this induced a thermal effect in the substrate [11]. This will cause the grain size to increase and the defect density to decrease with elevating sputtering power.

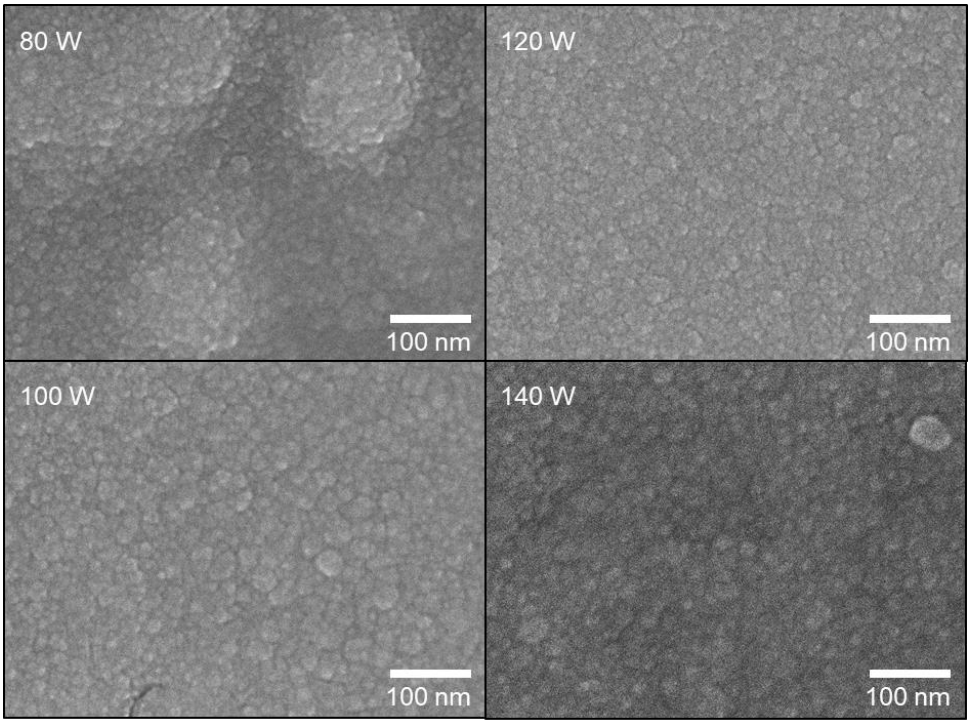

Figure 2. FESEM images of VO_2 thin films grown at sputtering power of 80 – 140 W.

3.3. Temperature-dependent transmittance spectra

Figure 3 exhibits transmittance spectra measured in heating and cooling cycles at a wavelength of 2.5 μm for the films prepared at various sputtering power. Hysteresis loops could also be obviously observed. According to Luo et al. [11], grain size could influence the hysteresis width. When the films have bigger grain size, the hysteresis width will be narrower. Particularly, the transition temperature of the VO_2 films was reported to correlate with microstructures such as defect density and grain boundary of the films [11]. By increasing sputtering power, the defect density could be reduce and lower transition temperature could be achieved. In addition, Table 1 shows the infrared switching properties i.e. phase transition temperature and hysteresis width, of the prepared films in infrared region with respect to the figure 3. Infrared transmittance of the films varied with sputtering power at the measured wavelength. The film deposited at 80 W showed a slightly high transmittance than the other films. The Tt of the films prepared at 80 W is 65.5 °C with a hysteresis width of 57.0 °C; while that of the films deposited at 100 W is 69.5 °C with a hysteresis width of 43.0 °C; and the films deposited at 120 W is 65.0 °C with a hysteresis width of 44.0 °C. The lowest Tt of 60.5 °C with a narrowest hysteresis width of 41.0 °C was observed for the films prepared at sputtering power of 140 W. This might due to high crystallinity with larger average crystallite size of the VO_2 films sputtered at sputtering power of 140 W. Furthermore, the nonmonotonic variation of transition temperature against sputtering power obtained in this study was also reported in a study whereby VO_2 films prepared at sputtering power of 330 – 400 W [11].

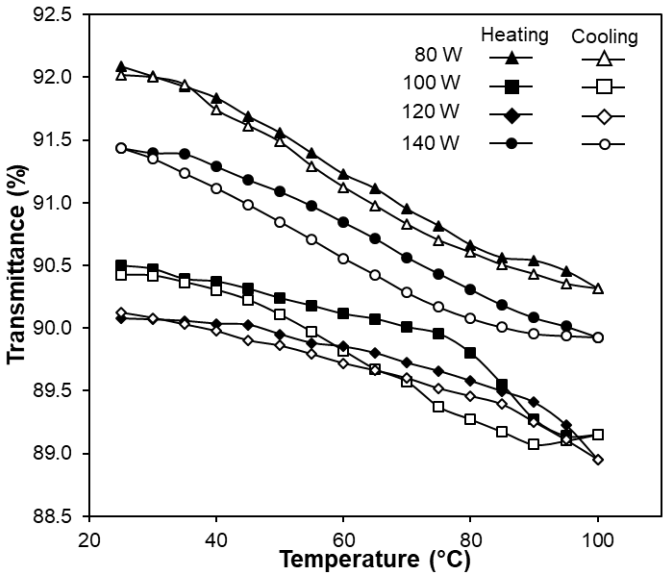

Figure 3. Temperature-dependent transmittance of VO_2 thin films at sputtering power of 80 – 140 W. Colored and uncolored triangles represent heating and cooling cycles, respectively.

Table 1. Phase transition temperature and hysteresis width of VO_2 thin films deposited at various sputtering power, measured at a wavelength of 2.5 μm.

Sputtering power (W)	Phase transition temperature (°C)	Hysteresis width (°C)
80	65.5	57.0
100	69.5	43.0
120	65.0	44.0
140	60.5	41.0

4. Summary

The influence of relatively low sputtering power on several properties of VO_2 thin films prepared with a VO_2 target by RF magnetron sputtering were analysed. This study also showed the ability of the technique employed to control sputtering power in order to obtain desired VO_2 thin films. As sputtering power increased, the film crystallinity enhanced revealing diffraction peaks of VO_2, and a slight increase in the average crystallite sizes. A slight change in infrared transmittance of the deposited films during heating and cooling cycles was observed. By increasing sputtering power, the average crystallite and grain sizes of the films rose which contributes to narrow hysteresis width. In addition, the enhancement in sputtering power resulted in lower transition temperature due to decrease in defect density. Among the prepared films, films prepared at sputtering power of 140 W have the lowest Tt of 60.5 °C with a hysteresis width of 41.0 °C.

5. References

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Acknowledgments

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