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Characteristics of RF Magnetron Sputtered VO₂ Thin Films for Potential Applications of Solar Control Coatings

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Abstract. Properties of vanadium dioxide or vanadia (VO₂) thin films prepared using radio frequency (RF) magnetron sputtering with a pure VO₂ target were analysed. The properties consisted of crystal structure, surface topography, surface morphology as well as optical and thermochromic properties of VO₂ thin films. The deposition was conducted in an argon atmosphere of flow rate of 30 sccm, substrate temperature of 500 °C and RF sputtering power of 100 W. The film, which was deposited with film thickness of 50 nm, exhibited a single orientation of (110) orientation. The film surface featured quite homogeneous and continuous surface topography, and spherical-like grains that represented grains in VO₂(M) phase. The film had low surface roughness resulting in quite high optical transmittance and relatively low optical reflectance, at room temperature throughout visible and near-infrared regions. Meanwhile, the film also showed a phase transition at ~65.0 °C with hysteresis width of 38 °C.

1. Introduction

Solar control coatings have been widely known for the ability to significantly reduce near-infrared (NIR) transmittance as well as outdoor heat inflow through windows while maintaining or improving visible transmittance when the temperature of the coatings is higher than its transition temperature (T_t). Vanadium dioxide (VO₂) thin films as solar control coatings have been discovered to exhibit T_t of 68 °C for a VO₂ single crystal and are still under investigations to efficiently reduce the T_t in order to produce intelligent coatings for architectural glazing. Nonetheless, accessibilities of deposition techniques to grow stoichiometric VO₂ thin films are still crucial.

Studies on the effects of deposition parameters on crystal structure, surface morphology, surface topography, optical and thermochromic properties of VO₂ films using DC magnetron sputtering have been intensively conducted [1-2]. Similarly, depositions and characterisations of thermochromic VO₂ thin films by using RF magnetron sputtering with pure vanadium (V) [3-5] and vanadium pentoxide (V₂O₅) [6,7] target materials have also attracted much attention.

However, only a few studies reported on RF magnetron sputtering with pure VO₂ target. For instance, Ko et al. [8] reported growth of VO₂ films with (002) preferential orientation on aluminium oxide (Al₂O₃) substrates by RF magnetron sputtering from a VO₂ target with Ar and O₂ gas mixture. The films were prepared at substrate temperature of 550 °C and RF sputtering power of 150 W. The T_t of the prepared VO₂ thin films was ~62.0 °C.



Hence, this work investigates the structural, morphological, optical and thermochromic properties of VO₂ thin films deposited on glass substrates using a RF magnetron sputtering with a VO₂ target as solar control coatings.

2. Experimental Details

2.1. Film Preparation

VO₂ thin films were prepared by means of RF magnetron sputtering using a VO₂ target (99.9 % purity) with a diameter of 76.2 mm and thickness of 3.0 mm. Prior to depositions of the films, Corning 2947 glass substrates were cleaned with ethanol in an ultrasonic bath for 15 mins and rinsed in distilled water. Then, the glass substrates were dried with nitrogen gas of 99.9 % purity and then clamped on a substrate holder in the deposition chamber. The chamber was evacuated to 10⁻⁴ Pa by a turbomolecular pump. The VO₂ target was pre-sputtered in an argon (Ar) atmosphere for 10 mins to remove any impurities or oxide layers on the surface of the target. During the film depositions, the working pressure and RF sputtering power were kept constant at 1.2 Pa and 100 W, respectively. Commercial Ar gas flow rate was supplied as sputtering gas into the deposition chamber during the depositions and maintained at 30 sccm which was controlled by a mass flow controller. The substrate temperature was varied from room temperature to 500 °C. Film thickness was kept constant at 50 nm and monitored with a quartz thickness meter supplied by Inficon.

2.2. Film Characterisation

Film thicknesses were measured by a surface profilometer (Alpha-Step IQ) with a stylus force of 0.03 N (3.0 mg) and measurement accuracy of ± 1.5 nm. The structural, topological and morphological properties of the films were also measured. The structural properties of the deposited VO₂ films were measured using Shimadzu X-Ray Diffractometer 7000 with a monochromatic high-intensity CuK α radiation ($\lambda = 1.54056\text{\AA}$) radiation operated at 40 kV and 30 mA. The grazing incidence X-ray diffraction (GIXRD) patterns of the films were recorded at a scanning rate of 2 °/min in the range of $2\theta = 20 - 60^\circ$ and an incident angle of 1.0 °. The surface topography of the films were investigated using a commercially available AFM system from CSM instruments in contact tapping mode with a constant load force on the cantilever of 2.0 nN. The surface morphology of the films was analysed using a JEOL JSM-7610F field emission scanning electron microscope. As for studying optical properties for the thermochromic coatings applications, characterisation on transmittance-temperature spectra is the preferred measurement to observe changes in optical properties with respect to temperature. The optical and thermochromic properties of the films were obtained using a CARY 5000 UV-Vis-NIR spectrophotometer (Agilent Technologies) equipped with a SPECAC 4000 SeriesTM high stability temperature controller to measure optical transmittance against temperature in the wavelength range of 350 – 2500 nm and scanning rate of 250 nm/min. The heating and cooling rate for the measurements was ~5 °C/min. The optical reflectance was also taken in the wavelength range of 300 – 800 nm at room temperature.

3. Results and Discussion

3.1. Crystal Structure of the RF Sputtered VO₂ Thin Films

Figure 1 shows the diffraction pattern of the VO₂ thin films deposited on glass substrates using RF magnetron sputtering and VO₂ target. Only one diffraction peak could be seen which corresponded to VO₂ (110) orientation plane. The (110) crystallite orientation was used to estimate crystallite size of the deposited films by using the Scherrer formula. The estimated crystallite size of the deposited VO₂ thin films was 58.3 nm. Similar GIXRD pattern has been reported in which the VO₂ films were deposited using DC magnetron sputtering and VO₂ target [2].

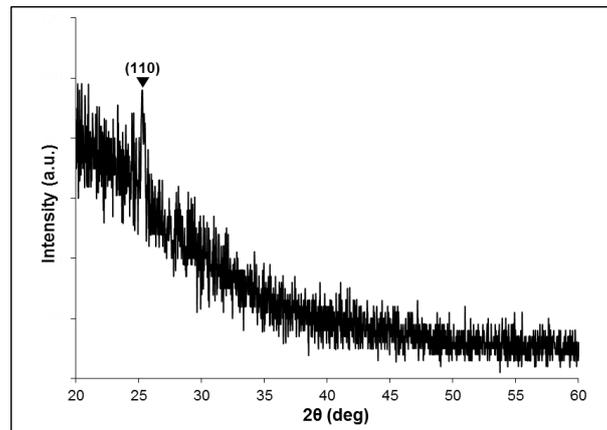


Figure 1. GIXRD patterns of the RF sputtered VO₂ thin films.

3.2. Surface Topography of RF Sputtered VO₂ Thin Films

Surface topography and surface roughness of the films were analyzed by using the representative AFM image of the VO₂ thin films shown in Figure 2, with a scan area of 4 μm². To study the AFM image, the dark regions were assumed to represent areas with zero or near zero height value in positive direction, whereas the bright regions represented higher areas such as the top of bulging grains. The film surface was quite homogeneous and continuous. The RMS surface roughness of the films was detected to be ~1.35 nm.

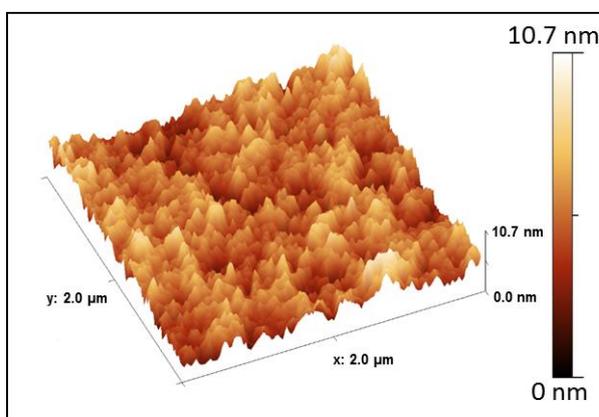


Figure 2. AFM 3D-image of the upper surface of the RF sputtered VO₂ thin films.

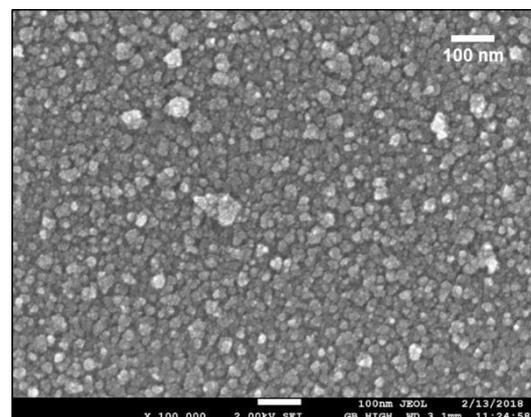


Figure 3. FE-SEM image of the RF sputtered VO₂ thin films.

3.3. Surface Morphology of RF Sputtered VO₂ Thin Films

Figure 3 represents the FE-SEM image of the RF sputtered VO₂ thin films. The film surface composed of uniform grain distributions with the estimated average grain size of 22.1 nm. Spherical-like grains could be seen which represented grains in VO₂(M) phase. As for VO₂(B) and V₂O₅ phases, very flat and no particular features and elongated grains would grow, respectively [9]. The application of VO₂ target for depositions of VO₂ thin films, in this study, using RF magnetron sputtering appeared to have similar surface morphology and spherical grains to the study of DC magnetron sputtered VO₂ thin films [2].

3.4. Optical and Thermo-chromic Properties of VO₂ Thin Films

Figure 4(a) displays the optical transmittance spectra (in the wavelength range of 350 – 2500 nm) of the RF sputtered VO₂ thin films at room temperature and 100 °C, i.e. in the semiconducting and

metallic states, respectively. The deposited VO₂ films had quite high transmittance throughout the wavelength regions. Low surface roughness detected in the AFM analysis might cause low light-scattering loss which then results in high optical transmittance [10]. Nevertheless, as the temperature increased from room temperature to 100 °C, only a slight decrease in the transmittance of the films was measured.

The optical transmittance at a fixed wavelength of 1500 nm during heating and cooling for the RF sputtered VO₂ thin films are illustrated in Figure 4(b). The red and blue lines represent the heating and cooling processes, respectively. Only a slight variation in the hysteresis loop was found. This was also observed for DC magnetron sputtered VO₂ thin films [2]. The films showed T_t of ~65.0 °C with hysteresis width, ω of 38 °C. The T_t was a bit lower by ~3 °C relative to a VO₂ single crystal with T_t of 68 °C.

Moreover, Figure 5 shows the optical reflectance spectrum of the RF sputtered VO₂ thin films, which was only measured at ambient temperature in air. The reflectance of the films was relatively low whereby about 13 % at 400 nm in the visible region and about 10 % at 800 nm in the near-infrared region could be detected. Thus, this could be correlated to surface characteristics of the films, for instance low surface roughness.

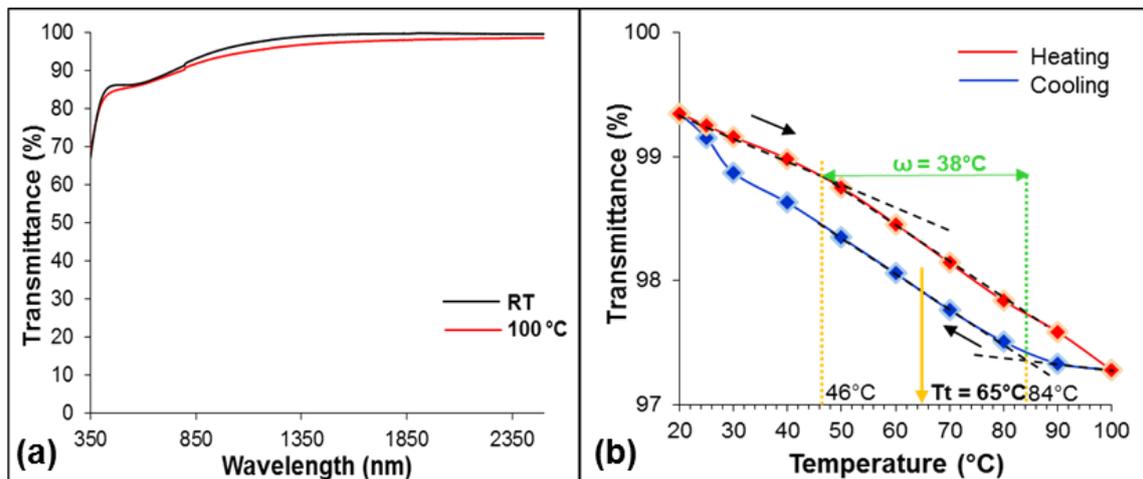


Figure 4(a). Optical transmittance spectra of the VO₂ films at room temperature (RT) and 100 °C, and **(b).** Optical transmittance at a fixed wavelength of 1500 nm as a function of temperature for the films.

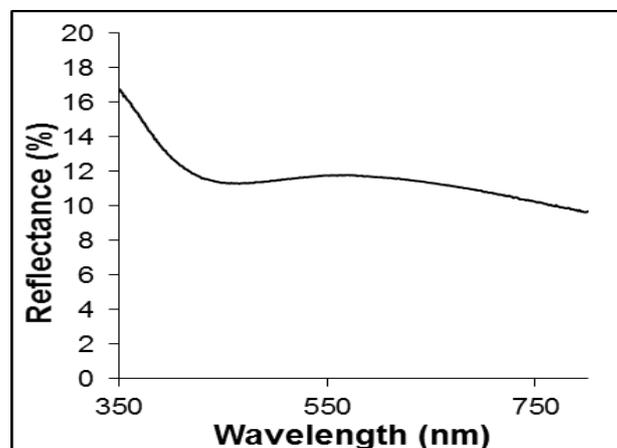


Figure 5. Optical reflectance spectrum of the RF sputtered VO₂ thin films measured at room temperature.

4. Conclusion

VO₂ thin films with a single orientation of (110) orientation were prepared using RF magnetron sputtering with a high purity VO₂ target at substrate temperature of 500 °C. The estimated crystallite size of the films was 58.3 nm. The film surface was quite homogeneous and continuous with RMS surface roughness of ~1.35 nm. The film also composed of uniform grain distributions with the estimated average grain size of 22.1 nm. The films showed quite high transmittance and relatively low reflectance throughout visible and near-infrared regions, at room temperature. Only a slight decrease in the transmittance and a slight variation in the hysteresis loop were measured between room temperature and 100 °C. The films had T_t at ~65.0 °C with hysteresis width of 38 °C. The T_t was a bit lesser than T_t of a VO₂ single crystal. Hence, as for solar control coatings, this study has shown a potential alternative technique to fabricate VO₂ thin films with a lower T_t using RF magnetron sputtering and a VO₂ target in an Ar atmosphere without the presence of any reactive gases such as oxygen.

Acknowledgments

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