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Facile preparation of double-sided VO_2 (M) films with micro-structure and enhanced thermochromic performances

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ABSTRACT

The fundamental challenge for VO_2-based thermochromic smart windows is the ideal combination of high luminous transmittance (T_{lum}) and high solar modulate ability ($\triangle T_{sol}$). Satisfying these competing demands is commonly achieved by sacrificing the luminous transmittance by optimizing the thickness through doping, composition, porosity adjustment, bio-antireflection, and multi-layer design. Here, we demonstrate an effective strategy for the preparation of VO_2 (M) films on two sides of fused silica substrates with ice crystals like micro-structure, which play a key role in anti-reflection. The films have been prepared using a dip-coating method followed by annealing in argon atmosphere. The resulting micro-patterned VO_2 (M) films show an ultrahigh T_{lum} (75.5%) and a suitable $\triangle T_{sol}$ (7.7%), derived from the sub-wavelength micro-structure on two sides of the substrate. In addition, the effects of the thickness, single-, or double-sided patterns on the optical performances of the VO_2 (M) films have also been investigated. These encouraging results show great potential for developing thermochromic smart windows with high performance for practical applications.

1. Introduction

Non-renewable energy has almost been exhausted, while increasing carbon dioxide emissions are leading to global warming. Nearly 30–40% of primary energy is used to maintain thermal comfort in buildings, such as heating, cooling, lighting, and ventilation [1]. The management of solar irradiation using thermochromic thin-film coatings on building glass (namely smart windows) can significantly decrease the energy consumption of buildings.

An ideal smart window needs a high transmittance in the visible region and performs thermal control ability to regulate the solar radiation in the near infrared (NIR) region [2]. Vanadium dioxide has a significant advantage as a coating material because of its insultor-metal transition (IMT), known as the thermochromic property, which changes from monoclinic phase with IR-transparent as a semiconductor to rutile phase that reflects IR as metal around 341 K [3].

Typically, the main optical performances of VO_2 thermochromic smart windows, including luminous transmittance (T_{lum}, 390–780 nm) and solar modulate ability ($\triangle T_{sol,}$ 250–2500 nm), are obtained using the following equations:

$$T_{lum/sol} = \int \phi_{lum/sol}(\lambda) T(\lambda) d\lambda / \int \phi_{lum/sol}(\lambda) d\lambda \quad (1)$$

$$\Delta T_{sol} = T_{sol}(30°C) - T_{sol}(100°C) \quad (2)$$

where $T(\lambda)$ is the recorded film transmittance, φ_{lum} is the standard luminous efficiency function for the photopic vision of human eyes, and φ_{sol} is the solar irradiance spectrum for air mass 1.5 (corresponding to the sun at 37° above the horizon).

Unfortunately, for thermochromic VO_2-based films, it is difficult to achieve a sufficiently high T_{lum} while preserving the $\triangle T_{sol}$ [4]. It has been calculated that the VO_2 (monoclinic M) film has a noticeable solar energy modulation limit of $T_{lum} \sim 40\%$, and the modulation of $\triangle T_{sol}$ does not exceed ~10% [5]. In order to increase the energy efficiency, several approaches have been designed to improve the optical performance of VO_2, including doping [6–9], composition [10–14], porosity adjustment [15–17], bio-antireflection [18] and multi-layer design [19–21]. Although the above solutions show excellent results in enhancement of $\triangle T_{sol}$, T_{lum} is still not high enough to achieve daylight quality in commercial buildings, which will increase the lighting energy needs, thus, resulting in negative energy conservation from a practical point of view [22,23].

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Recently, VO_2 (M) films with sub-wavelength structures have been widely investigated to enhance T_{lum} by reducing the reflection at the interface between air and VO_2 (M) as a low effective-index (n) layer [18,24–28]. For example, Long et al. prepared VO_2 (M) films with moth-eye structures, which had a gradient refractive index and enhanced both $\triangle T_{sol}$ and T_{lum} [18]. Gao et al. investigated a series of VO_2 (M) films fabricated by polymer assisted sol-gel method [15,16,24,29], and a surface with micro-roughness was obtained by phase separation, which lowered the optical constants and resulted in excellent optical performance [24]. Zhou et al. introduced a periodic porous structure into a VO_2 (M) film. The pores allowed light to pass through directly, and a favorable $\triangle T_{NIR}$ at 2000 nm with only a little attenuation of T_{vis} was achieved [25]. Zhang et al. synthesized a self-assembled 2D ordered VO_2 (M) film using a hydrothermal method, and an increased $\triangle T_{sol}$ of 10.7% with a T_{lum} of 46.7% was achieved [26]. Tao et al. prepared porous W-doped VO_2 films with simultaneously enhanced visible transparency and thermochromic properties. The method is facile and impressive [27,28].

However, to the best of our knowledge, there is no research reports on the construction of micro-patterned VO_2 (M) film on two sides of the substrates, which will have a better anti-reflection effect [30–32], thus, obtaining simultaneous enhancement in $\triangle T_{sol}$ and T_{lum}.

Here, for the first time, we have prepared VO_2 (M) films with micro-structure on both sides of a fused silica substrate by a one-step dip-coating method, followed by annealing in argon atmosphere. The surface morphology of the VO_2 (M) film was ice crystal-like after annealing at 500 °C for 5 h. As expected, the T_{lum} of the micro-patterned and double-sided VO_2 (M) film increased significantly, up to 75.5%, with a comparable $\triangle T_{sol}$ of 7.7%.

2. Experimental details

2.1. Materials

V_2O_5 powder (AR, Tianjin Fu Chen Chemical Reagents Factory) was used as the starting material to prepare the precursor sol. Polyvinylpyrrolidone (PVP K30, Sigma-Aldrich) was used as a sol stabilizer. All the reagents were used without further purification.

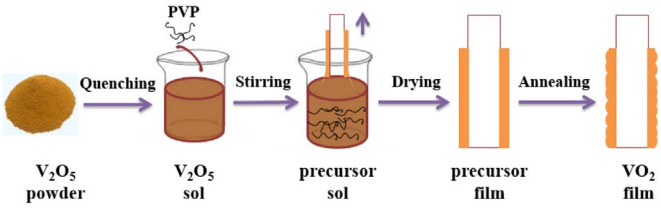
2.2. Preparation of the precursor sol

V_2O_5 sol was fabricated by a water quenching method: 10.0 g V_2O_5 powder was placed in a crucible and melted at 850 °C for 10 min. The fused V_2O_5 liquid was added to 400 mL de-ionized (DI) water at room temperature (RT). The obtained slurry was stirred vigorously for 1 h, and then, diluted with DI water to 10 g/L. PVP (K30 6 wt%) was added to the sol to stabilize the V_2O_5 sol at room temperature. The precursor sol was obtained after vigorous stirring for 10 h. The concentration of the precursor sol was adjusted from 2.5 g/L to 10.0 g/L to regulate the thickness by adding DI water. All the samples used in this work were synthesized using these precursor sols, unless otherwise noted.

2.3. Fabrication of the VO_2-based film

The VO_2 films were dip-coated on fused-silica substrates (1×4 cm^2) using a dip coater (KSV Instruments) at a speed of 500 μm/s. After drying at RT for 30 s, the same process was repeated 4 times. The smooth precursor films were then dried for 30 min at 80 °C to remove the excess solvent. Then, the films were annealed under argon atmosphere (purity of 99.99%) at different conditions to transform the V_2O_5 phase into the VO_2 (M) phase. The annealing temperature was varied from 400 °C to 550 °C with an interval of 50 °C at the rate of 1 °C/min. The annealing time was 1 h, 5 h, and 10 h. Pretreatment was performed at 260 °C for 1 h to obtain smooth films. The general fabrication process is illustrated in Scheme 1.

Single sided VO_2 films were also prepared for comparison by washing out one side with DI water. The samples prepared are listed in Table 1.



Scheme 1. Schematic illustration for the preparation of the micro-patterned VO_2 (M) films on both sides of the fused-silica substrates.

Table 1
Samples prepared in the experiment.

Samples	Sol concentration g/L	Annealing Temperature °C	Annealing time h	Film sides	No. of dips
D1	10.0	400	1	2	4
D2	10.0	400	5	2	4
D3	10.0	400	10	2	4
D4	10.0	450	1	2	4
D5	10.0	450	5	2	4
D6	10.0	450	10	2	4
D7	10.0	500	1	2	4
D8	10.0	500	5	2	4
D9	10.0	500	10	2	4
D10	10.0	550	1	2	4
D11	10.0	550	5	2	4
D12	10.0	550	10	2	4
D13	2.5	500	5	2	1
D14	5.0	500	5	2	1
D15	7.5	500	5	2	1
D16	10.0	500	5	2	1
D17	5.5	500	5	2	1
S17	5.5	500	5	1	1

2.4. Measurements

The crystalline phases of the films were determined using X-ray diffraction (XRD, PANalytical B.V. Model X′pert Pro, primary monochromatic Cu-Kα radiation) at an X-ray grazing angle of 2.0 °. Surface morphologies and thickness of the films were examined by a field-emission scanning electron microscope (FEI Helios Nanolab600i) and an atomic force microscope (AFM, Dimension Icon, Bruker). Fourier transform infrared (FT-IR) spectroscopy was performed using a FT-IR system (VERTEX-70, Bruker) from 4000 to 400 cm^{-1}. X-ray photoelectron spectroscopy (XPS) was performed with a PHI 5700 ESCA System using Al Ka radiation (1486.6 eV). XPS data were calibrated to the C1s peak and analyzed using Casa XPS software. Thermogravimetry-differential thermal analysis (TG-DTA) was performed using a NETZSCH STA449F3 at a heating rate of 10 °C/min in argon atmosphere.

The transmittance of the VO_2 films was measured using a UV–vis–NIR spectrophotometer (Lambda-950, Perkin Elmer) equipped with a film heating unit over the wavelength range of 250–2500 nm. Hysteresis loops were measured by collecting the transmittance of films at a fixed wavelength (2000 nm) at an approximate interval of 2.0 °C using a fiber optic spectrometer (Ocean optics, NIRQuest 256-2.5). The temperature was measured with a thermocouple in contact with the film and was controlled by a temperature-controlling unit. The temperature errors were smaller than 0.5 °C based on repeated measurements.

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