Hydrothermal growth of VO$_2$ nanoplate thermochromic films on glass with high visible transmittance

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The preparation of thermochromic vanadium dioxide (VO$_2$) films in an economical way is of interest to realizing the application of smart windows. Here, we reported a successful preparation of self-assembly VO$_2$ nanoplate films on TiO$_2$-buffered glass by a facile hydrothermal process. The VO$_2$ films composed of triangle-shaped plates standing on substrates exhibit a self-generated porous structure, which favors the transmission of solar light. The porosity of films is easily controlled by changing the concentration of precursor solutions. Excellent thermochromic properties are observed with visible light transmittance as high as 70.3% and solar modulating efficiency up to 9.3% in a VO$_2$ film with porosity of ~35.9%. This work demonstrates a promising technique to promote the commercial utilization of VO$_2$ in smart windows.

Energy consumption in the residential, commercial and other man-made buildings accounts for nearly 40% of total global energy use, making it the largest single component of energy use$^1$. The explosion in demand for air-conditioning units is aggravating this large energy consumption. Low-E window which exhibits high reflectivity of infrared light has been widely used in commercial buildings to achieve energy saving. However, its solar radiation reflection has no responding ability to environmental temperature change, limiting its application in different market requirements$^{1,2}$. Smart windows with thermochromic thin-film coatings on building glass provide an effective way to modulate the solar energy transmitted into the interior room.

It is well known that vanadium dioxide (VO$_2$) shows a reversible metal-insulator phase transition (MIT) at a phase-transition temperature ($T_c$) of 68 °C$^3$. When temperature below $T_c$, VO$_2$ is an insulator with a monoclinic structure (M phase, space group $P2_1/c$) which is transparent to infrared radiation (IR). As temperature above $T_c$, VO$_2$ transforms to a metallic state with a rutile structure (R phase, space group $P4_2/mmm$) which is reflective to IR radiation while maintains visible-light transparent$^4$. Such a MIT transition makes VO$_2$ an attractive material for smart windows$^5$. In order to promote the application of VO$_2$ based smart windows, various methods have been used to achieve the VO$_2$ coatings on transparent substrates for smart windows$^6$. In order to promote the application of VO$_2$ based smart windows, various methods have been used to achieve the VO$_2$ coatings on transparent substrates for smart windows. However, how to enhance the visible light transmission with little sacrifice of solar modulation ability and lower the cost of large scale coating are still two major challenges for researchers. Based on the vapor-based deposition techniques$^6,7$, multilayered structure (glass/TiO$_2$/VO$_2$/TiO$_2$/VO$_2$/TiO$_2$)$^8$, multifunctional TiO$_2$(R)/VO$_2$(M)/TiO$_2$(A)$^9$ and antireflection (AR) coatings on VO$_2$ films$^{10}$, were designed to meet the performance boost. But those methods were proved to be complicated and expensive due to the difficulties in controlling variable valences of V ions and costly equipment.

Recently, solution-based methods for depositing VO$_2$ coatings on substrates have been studied extensively because of its low-cost and up-scalable. Cao et al. and Kang et al. have utilized solution methods (dip-coated with freeze-drying and spin-coated with sol-gel, respectively$^{11,12}$) to obtain enhanced optical performance with high solar modulation ability. Their work demonstrated that creating tunable porosity in VO$_2$ films was a feasible way to meet the performance requirements for practical usage. However, complex processes with high temperature
and ethanol (80 ml) and stirred for 1 h. Finally a transparent and stable TiO2 sol was obtained. The sol was spin coated on a transparent polymer (e.g., VO2/SiO2 core-shell 19, VO2/ATO/polymer20 and polymer-assisted deposition 21–24).

Solution of acetone, 2-propanol and deionized water with volume ratios of 1:1:1. As-coated TiO2 precursor layer was heated under 400 °C for hours to achieve its crystallization and adhesion 32. The detailed preparation process for TiO2 buffers is as follows: firstly, tetrabutyl titanate (C4H9O4Ti, 10 ml) was added into the ethanol (3 ml) at room temperature and stirred for 30 min. Then the solution was transferred into a mixed solution of nitric acid (3 ml), deionized water (6 ml) and ethanol (80 ml) and stirred for 1 h. Finally a transparent and stable TiO2 sol was obtained. The sol was spin coated at 3500 rpm for 30 s on a glass with diameter of 2 inches, which was ultrasonically cleaned for 10 min in a solution of acetone, 2-propanol and deionized water with volume ratios of 1:1:1. As-coated TiO2 precursor layer was heated under 400 °C for 1 h to produce fine grained TiO2 layer. The glass with TiO2 buffers was used for the hydrothermal growth of VO2 films. In the hydrothermal process, the vanadyl oxalate precursors were prepared by dissolving V2O5 (0.182 g) in the aqueous solution (50 ml) containing oxalic acid (1.97 g) at 70 °C. The aqueous solution was diluted into 500 ml with deionized water, forming a 4 mmol/L solution with pH value ~2.4. The pH value was controlled by NH4OH. The vanadyl oxalate aqueous solution (60 ml) was transferred into a Teflon-lined autoclave (100 ml). The chemical reaction was carried out at 230 °C in an electric oven. After heating for 4 h, the autoclave was naturally cooled down in furnace. The side of TiO2 layer was covered by a uniform film. The wafer samples were cleaned up with deionized water and alcohol, and dried by nitrogen. The thermochromic VO2 windows were obtained through annealing the as-grown VO2 films in a short annealing furnace at 400 °C for 60 s in 4 * 104 Pa of air. Unless specifically noted in the article, all samples used here are prepared as mentioned above.

Instrumentation characterization. The morphology of the reaction product was examined by using scanning electron microscopy (SEM, Hitachi S–4800). The phase identification of the TiO2 and VO2 films was performed using X-ray diffraction (XRD, Bruker-AKS diffractometer, Model D8 ANVANCE) with Cu-Kα radiation source, Raman spectra (HR800, excitation wavelength: 633 nm, laser power: 1 mW) and Transmission Electron Microscope (TEM, FEI Tecnai G2 F20 S-TWIN). The chemical valence of vanadium ions was measured by XPS (PHI QUANTRA-II SXM) with Al-Kα radiation source (1486.6 eV). The porosity based on SEM images was calculated by using Image-Pro Plus (IPP) to compare the gray scale pixel of the area occupied by VO2 nanoplates and exposed TiO2 films. The optical transmittance spectra of samples at normal incidence from 300 to 3000 nm and were measured by using Shimadzu UV-3600 UV-VIS-NIR spectrophotometer with Heat Solid Transmission Accessory.

Result and Discussion

Figure 1a shows the morphology images of polycrystalline TiO2 buffers with grain size between 25 to 75 nm. The XPS full spectrum (Fig. 1b) of TiO2 reveals a high purity component. The obtained VO2 film is composed of nanoplates with an average thickness of ~40 nm, and a height of ~400 nm, which are regularly grown against substrates (Fig. 1c,d). There are smaller and more randomly oriented nanoplates close to the substrate, which is similar with the previous report for the growth of ZnO films 33. As identified by XRD (Fig. 1e), the characteristic peaks agree with those of M-VO2 in monoclinic structure (JCPDS No. 65-2358) and A-TiO2 in anatase phase.
(JCPDS No. 21-1272) respectively. The remarkable (020) peak of VO$_2$ indicates that the growth of VO$_2$ films are preferentially oriented on substrates. For a randomly oriented VO$_2$ polycrystalline sample the intensity of (020) diffraction is only ~2.4% of the strongest peak (011). The preferred orientation of the VO$_2$ films supports the conclusion that the VO$_2$ nanoplates are regularly grown on substrates as shown in the cross-section structure of VO$_2$ films in Fig. 1d. The XRD pattern of TiO$_2$ buffers indicates the (004)-preferred orientation of anatase TiO$_2$. It is known that the close-packed planes in anatase-TiO$_2$ (112) and rutile-VO$_2$ (200)/(020) are equivalent$^{34}$, so we can infer that there is a lattice-matching relationship between anatase TiO$_2$ and rutile VO$_2$ with $A$-TiO$_2$ (112)//$R$-VO$_2$ (200)/(020). In this case, it is possible for VO$_2$ to grow in a preferred orientation manner guided by the $A$-TiO$_2$ buffer under hydrothermal growth temperature (230 °C). The $M$-VO$_2$ is a polymorphic phase transformed from $R$-VO$_2$ through a small distortion$^{35}$. The $R$-VO$_2$ [200] planes correspond to the (020) and (002) planes in the $M$-VO$_2$ phase$^{36}$. For the (004)-preferred orientation of anatase TiO$_2$ as determined by XRD, the preferred orientation of $M$-VO$_2$ should be (011)$M$ considering the crystal distortion induced by the mismatch between TiO$_2$ and VO$_2$. The angle between (112) and (004) in $A$-TiO$_2$ is ~61° and no good lattice-match relation exist along other directions, therefore, the inclined growth of plate-like VO$_2$ nanocrystals are observed in Fig. 1c,d. While the VO$_2$ nanoplates show the strong preferred orientation of (020)$M$, it should be related to other orientations of TiO$_2$, i.e. (110) or (112) orientations of $A$-TiO$_2$. For $A$-TiO$_2$ (110) or (112) orientations the VO$_2$ nanoplates would grow perpendicular or parallel to the substrate. The corresponding growth of VO$_2$ nanoplates can be observed in Fig. 1c,d. The existence of (110)-orientation TiO$_2$ is verified by TEM in Fig. 2. XPS measurements were performed to examine the oxidation states of V ions in VO$_2$ thin films (Fig. 1f)$^{37}$. It is shown that the VO$_2$ thin films contain partial V$^{5+}$ ions together with V$^{4+}$ ions. The presence of V$^{5+}$ ions could be attributed to surface oxidization in the annealing process or storage in air and exist only on the surface as proved$^5$. 

Figure 1. (a) Compact TiO$_2$ thin films are composed of equiaxed grains with size distribution between 25 to 75 nm. (b) The XPS full spectrum of prepared TiO$_2$ thin film. (c,d) SEM images of the obtained VO$_2$ thin films and the corresponding cross section morphology, revealing a nanoplate structure. (e) XRD patterns of VO$_2$ thin films compared to TiO$_2$ thin films, indicating the orientated growth of the monoclinic VO$_2$ on anatase TiO$_2$ phase. (f) XPS spectrum of VO$_2$ thin films.
In order to understand more details about the oriented growth of VO$_2$ and TiO$_2$ layers, a cross-section sample of VO$_2$/TiO$_2$ films was prepared and investigated by TEM. TEM images (Fig. 2a,b) show the well-connected 3-layer structure. The TiO$_2$ thin film has a thickness ~12.8 nm (Fig. 2b). Two TiO$_2$ grains exist in the observation region, and they have different orientations as shown by the HRTEM images in Fig. S1 (supporting information). The VO$_2$ nanoplates show a triangle-like shape in Fig. 2a, which stand on the substrate. HRTEM images taken from two layers in Fig. 2c,e show clear lattice fringe, indicating good crystallinity of VO$_2$ and TiO$_2$ films. The interplanar spacing of 0.331 nm in Fig. 2c corresponds to the plane distance of (1–10) of monoclinic VO$_2$ (Fig. 2d). The interplanar spacings of 0.270 nm and 0.358 nm in Fig. 2e belong to the (−110) plane and (011) plane of anatase TiO$_2$ (Fig. 2f), respectively. For the present orientations of A-TiO$_2$ and M-VO$_2$ as shown in Fig. 2(c–f), the equivalent planes, i.e. A-TiO$_2$ (112) and M-VO$_2$ (002)/(020) are not in the matching orientations. However, the right-hand grain of A-TiO$_2$ as shown in Fig. 2(b) and Fig. S1(c) exhibits an orientation that the left-hand grain rotates about 15° clockwise. In this case, the M-VO$_2$ (002) plane is parallel to the A-TiO$_2$ (112) plane of the right-hand grain, indicating the growth of VO$_2$ in Fig. 2 is guided by the left-hand TiO$_2$.

Figure 2. (a,b) Cross-sectional TEM images of the VO$_2$/TiO$_2$ films on glass substrate, (a) shows the shape of the VO$_2$ nanoplates, (b) a VO$_2$ grain grown on the TiO$_2$ thin film, (c,e) High resolution TEM (HRTEM) images taken from different layers as marked by squares in (b). (d,f) FFT patterns correspond to (c) VO$_2$ nanoplate and (e) TiO$_2$ thin film respectively.
corresponding crystallographic relationship of VO₂ and A-TiO₂ is schematically shown in Fig. S2. The TEM analysis demonstrates the guided growth of VO₂ by buffer TiO₂.

To investigate the possible growth mechanism of VO₂ films, controllable hydrothermal processes were designed. Different precursor solutions and pH values were found to be key factors to affect the reaction process. The role of precursors in the hydrothermal process for preparing the VO₂ films were investigated, i.e. precursor solutions obtained from V(OH)₂NH₂ dissolved in HNO₃, hydrazine hydrate reacted with VOSO₄, NH₄VO₃ with 1,3-propylene glycol reduced in H₂SO₄, and V₂O₅ dissolved in oxalate acid solution. It is found that VO₂ films can be grown only in the vanadyl oxalate solution, which suggests that oxalate acid solution is a suitable solvent for the formation of VO₂ thin films.

The pH value of vanadyl oxalate solution was modulated by adding droplets of NH₄OH. Figure 3(a–e) show the SEM images of VO₂ films prepared at different pH values. The morphology of VO₂ nanoplates greatly changes with increasing pH values. Obviously, the growth of VO₂ is greatly influenced by the pH value. At pH 3.46, the VO₂ nanoplates in Fig. 3a are twice thicker than those grown at pH 2.4 (Fig. 1c), making the nanoplates more like nanorods (length was ~300 nm). When the pH value rises up to 4.56, the nanorods become shorter (length is 250 nm) and wider (Fig. 3b). As the PH value equals to 6.21, nanorods disappear instead of rectangle-like grains distribute randomly on the film (Fig. 3c). At pH 7.45, irregularly shaped particles are loosely attached to substrates. At PH 8.12, more area of substrate is exposed. Furthermore, experiments revealed that nothing could be grown on the substrate while pH values ≥ 8.5. Dobson et al. have examined the adsorption of low molecular weight (LMW) carboxylic acids to TiO₂ in aqueous solutions by infrared spectroscopic analysis, and reported the existence of strong adsorption of dicarboxylic acids (such as oxalic acid) to TiO₂. This result was demonstrated by Mendive et al., who pointed out that the pH value played an important role in the adsorption behavior. The strong adsorption of oxalate organic species on TiO₂ occurred only as the pH value less than IEP (the isoelectric point, a pH value at which a particular molecule or surface carries have no net electrical charge). Bandura et al. investigated the adsorption of H₂O on TiO₂, and reported that for adsorption of H₂O onto the surface of TiO₂, H⁺ and OH⁻ would produce positive (-O-H⁻) and negative (-Ti-OH⁻) surface sites, respectively. The IEP of TiO₂ is close to 6.2 as reported by Parks. When PH is lower than 6.2, positive charge sites should dominate on the surface, whereas negative charge sites would be in majority. The adsorption affinity decreased rapidly as the pH value larger than IEP. Although the concentration of oxalate acids and the presence of metal cations in solution can influence the IEP, the pH dependence of adsorption does not change. It indicates that the protonated...
surface of TiO$_2$ thin films is required for adsorption of organic anions. Our experimental results of different vanadic acid solutions and pH values are in consistence with the reported adsorption features of the organic acid solutions, indicating the chemical solution growth of VO$_2$ on TiO$_2$ is of adsorption dependence. The TiO$_2$ buffer is a key factor for adsorption and consequently for interface reactions in the chemical solution environment because its surface chemical state at low pH values facilitates adsorption of carboxyl group.

In the oxalic acid solution, the possible surface reaction would be like that: 1) the vanadyl oxalate species were adsorbed on the TiO$_2$ buffer. It is known that oxalate can form organic metallic cation complexes through the coordinating ability of the carboxyl group. In that case, the negatively charged organic vanadium complexes ([VO$_x$(C$_2$O$_4$)$_y$]$^{-y}$) should be adsorbed on the positive surface sites through the carboxylic group. 2) Undergoing the water shrinkage reaction between the adsorbed vanadyl oxalate and the neighboring hydrogen ions on the protonated surface, VO$_2^{+}$ were adsorbed on the TiO$_2$ substrate, and then crystallized to VO$_2$ thin films. The schematic diagram of the growth process is shown in Fig. 3g. The different vanadic precursor solutions mentioned above have no carboxylic group, so there is no effective species to play the role of bridge between vanadium ions and positive charge-terminated surface of the TiO$_2$ thin films for achieving the growth of highly adhesive VO$_2$ films.

The optical modulation properties of the prepared VO$_2$ films were investigated to evaluate its potential for the smart windows. For realizing the application of VO$_2$ in smart window a technological challenge is to improve the maximum visible transmittance ($T$-vis) to an acceptable value (>60%), while maintain the high solar modulating efficiency ($\Delta T_{sol}$) of VO$_2$. To improve $T$-vis, one way is to fabricate porous films and reduce the thickness of the continuous films of VO$_2$ to less than 80 nm. In this work, the standing nanoplate structure facilitates the penetration of solar light, namely apt to achieve high $T$-vis. The obtained VO$_2$ films are in fact self-generated porous films, which would produce excellent combination thermochromic property. Cao et al. reported a nanoporous VO$_2$ film exhibiting good thermochromic properties, the highest value of $T$-vis and $\Delta T_{sol}$ were 75% and 7.9% respectively. In our work, the $T$-vis can be easily adjusted by changing the porosity of VO$_2$ films through diluting the concentration of vanadyl oxalate in solution (Fig. S3). The porosity of VO$_2$ films on glass increases with decreasing the concentration of vanadyl oxalate. By comparing the area occupied by VO$_2$ nanoplates and exposed TiO$_2$ films, the calculated porosities for the VO$_2$ films grown in different concentration vanadyl oxalate solutions are shown in Fig. 4a. The samples are marked as: #1, 0.73 mmol/L; #2, 1.1 mmol/L; #3, 1.5 mmol/L; #4, 2.2 mmol/L; #5, 4.0 mmol/L, respectively. Such self-generated porous nanostructures exhibit a good combination property of thermochromism (combining visible light transmittance and solar modulating efficiency). Figure 4b shows temperature-dependent
transmittance of the porous VO₂ nanofilms. The right insets are the corresponding coating photos. The hysteresis loops of transmittance at 2000 nm for different VO₂ thin films are shown in Fig. 4c, the Tₜ and hysteresis loop width (∆T) of #5 is 70.1 °C and 12.9 °C respectively. Both of Tₜ and ∆T are increased as the porosity of thin films increasing, which is considered that the discontinuity of grain in thin films causes a loose grain boundaries limit propagation of MIT, and results higher Tₜ and wider ∆T [9]. The T-vis, ∆Tšal, and near-infrared (NIR) switching efficiency (∆T1000–2500nm) are shown in Fig. 4d, T-vis monotonously increases with the porosity of thin films as predicted. While the ∆Tšal shows a plateau for samples #2–#4. Pleasurable thermochromic properties are observed in the sample #2 with 35.9% porosity, the T-vis value is as high as ~70.3% with the ∆Tšal up to 9.3%. The results are even better than those of periodic and aperiodic porous VO₂(M) films fabricated by complicated chemical and physical processes [24,49], the multilayered TiO₂(or SiO₂)/VO₂/substrate films [8], and the VO₂-based composite thin films [20,31]. The excellent thermochromic properties of our VO₂ films benefit from the special nanofilms structure which provides pores to solve the issue of low visible transmittance, meanwhile keep the thickness of films up to ~400 nm.

The integrated solar transmittance (Tsol, 300–2500 nm) and the ∆Tsol values are obtained from the following equation:

\[ T_{sol} = \frac{\int \varphi_{sol}(\lambda) T(\lambda) d\lambda}{\int \varphi_{sol}(\lambda) d\lambda} \]

\[ \Delta T_{sol} = T_{sol(300\degree C)} - T_{sol(100\degree C)} \]

where \( T_\lambda \) denotes transmittance at wavelength \( \lambda \), \( \varphi_{sol} \) is the solar irradiance spectrum for air mass 1.5 (corresponding to the sun standing 37° above the horizon) [52].

**Conclusion**

In summary, we have successfully fabricated nanofilms VO₂ films on glass substrates with TiO₂ buffers, for the first time, by a facile hydrothermal method. The obtained VO₂ films show unique self-assembly porous structure with the porosity controllable by the concentration of the precursor solution. Excellent thermochromic properties are achieved with a visible light transmittance of 70.3% and a solar modulating efficiency of 9.3%. The investigation of growth process reveals that the appropriate adsorbent media, such as oxalate groups adsorbing on TiO₂ buffers, are necessary for the preparation of VO₂ thin films on glass by the hydrothermal technique. The preparation process of thermochromic VO₂ films adopted in this work is facile, low-cost and up-scalable. The experiments proved its potential in promoting the practical application of VO₂ in smart windows.

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Author Contributions
J.L., J.Z. and H.J. supervised and coordinated all aspects of the project. J.Z. synthesized and characterized the materials. P.C. carried out the measurement of electronic and optical properties. F.R. carried out the XRD and Raman characterizations and crystal structure analysis. Y.J., M.C. and Y.Z. carried out the TEM characterization and image analysis. All authors contributed to the writing of the manuscript.

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In this Article, the scale bars are omitted from Figures 1a,c,d and 2a. The correct Figures 1 and 2 appear below.
Figure 1.
Figure 2.

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