Intelligent Window Coatings: Atmospheric Pressure Chemical Vapor Deposition of Tungsten-Doped Vanadium Dioxide

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Thin films of tungsten-doped vanadium(IV) oxide were prepared on glass substrates from the atmospheric pressure chemical vapor deposition of vanadium(IV) chloride, tungsten-(VI) ethoxide, and water at 500–600 °C. The films were characterized by Raman microscopy, glancing angle X-ray diffraction (GAXRD), X-ray photoelectron spectroscopy (XPS), Rutherford backscattering (RBS), scanning electron microscopy (SEM), and vis/IR reflectance–transmittance. The films showed a reduction in thermochromic transition temperatures from 68 °C in VO_2 to 42 °C in $V_{0.99}W_{0.01}O_2$—approaching that required for commercial use as an intelligent window coating.

Introduction

Vanadium dioxide exists in four polymorphic forms—$VO_2(M)$ and $VO_2(R)$ and two metastable forms $VO_2(A)$ and $VO_2(B)$.[1] The monoclinic $VO_2(M)$ converts to rutile $VO_2(R)$ at 68 °C.[2] This is a fully reversible metal-semiconductor phase transition (MST) that is associated with drastic changes in electrical conductivity and optical properties in the near-IR region.[3] Above 68 °C, VO_2 behaves as a semi-metal, is reflecting to a wide range of solar wavelengths below the MST it behaves as a semiconductor, and reflects significantly less energy. Incorporating thermochromic films into window products could have significant benefit to the consumer. For example, a window coating that became more reflective with increasing external temperature would reduce solar gain on hot days—thus reducing air conditioning costs.[4] For thin films of vanadium(IV) oxide to find practical use as intelligent window coatings, the MST needs to be reduced from 68 °C.

Doping metal ions into the VO_2 lattice has been shown to increase or decrease the MST temperature (T_c) depending on factors such as size[5] and charge[6] of the dopant ion and changes in electron carrier density.[7] Tungsten has been shown to be the most affective

dopant ion in manipulating the MST of VO_2; it can optimally lower the T_c to about 25 °C at 2 atom % loading. Tungsten-doped VO_2 films have been prepared by sol–gel[8,9] and physical vapor deposition.[10,11] Film thickness has also been shown to influence the thermochromic transition temperature of VO_2[12]—films of VO_2 300 nm or less display a thermochromic transition temperature of 50 °C—this has been explained in terms of a strain effect.[12]

Chemical vapor deposition has not been reported for the production of metal-doped vanadium oxide thin films. Atmospheric pressure chemical vapor deposition (APCVD) is an attractive method for applying thin films to glass substrates.[13] The process can be easily integrated into commercial float-glass production lines, as it has fast growth times and is directly compatible with on-line manufacture in a way that off-line coating methods such as physical vapor deposition and sol–gel are not.

We have shown in a previous paper[14] that single-phase VO_2 can be obtained from the APCVD reaction of VCl_4 and water. This paper details the formation of tungsten-doped VO_2 from the APCVD reaction of VCl_4, water, and a tungsten ethoxide precursor. It was found that tungsten could be doped into the VO_2 films by CVD causing a significant decrease in the thermochromic phase transition, from 68 to 42 °C.

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Table 1. APCVD Conditions Used To Prepare Tungsten-Doped VO₂ Thin Films from VCl₄, [W(OC₂H₅)₆], and Water

[W(OC₂H₅)₆] flow rate (L min⁻¹); bubbler temp (°C)	reactor temp (°C)	gas-phase VCl₄:H₂O ratio	phase from Raman and XRD	W atom % in film by XPS (±0.1)	transition temp ± hysteresis width (°C)
1.2−180	500	1:10.7	VO₂(M)	0.6	52 ± 11
1.0−184	550	1:10.2	VO₂(M)	0.6	52 ± 12
1.0−185	550	1:9.2	VO₂(M)^a and VO₂(B)	0.9	45 ± 15
1.0−185	550	1:9.8	VO₂(M)	0.8	42 ± 15
1.2−189	600	1:5.5	VO₂(M)	0.3	58 ± 9
1.3−194	600	1:4	VO₂(M)	1.2	49 ± 16

^{a} VO₂(B) was confined to the leading edge.

Experimental Section

All CVD studies were performed on a custom-built apparatus using a previously described procedure.[14] Vanadium-(IV) chloride (99%, Aldrich, UK) and [W(OC₂H₅)₆] (99.9%, Alfa Aesar, UK) were placed into separate bubblers. Distilled water was injected into the plain-line gas flow using a fixed-rate syringe driver (1.33 cm min⁻¹) and a 2 cm³ syringe. Nitrogen (99.999%, BOC UK) was used as the system gas in all CVD reactions.

The substrate was float glass that had a 50 nm thick SiO₂ barrier layer to stop diffusion of ions from the glass into the film (Pilkington Glass, UK). The glass had dimensions 15 cm × 4.5 cm × 0.3 cm and was cleaned before use by wiping with a water-soaked tissue and then a propan-2-ol soaked tissue and then rinsed with propan-2-ol. It was allowed to dry in air prior to mounting in the CVD chamber.

A series of films were prepared by varying the carrier gas flow rate through the tungsten precursor bubbler. All other conditions were kept constant to previous work.[14] The gas-phase VCl₄:H₂O concentration ratio was between 1:5 and 1:10 for all CVD reactions. The bubbler containing [W(OC₂H₅)₆] was set to 190 °C. The flow rate through the tungsten bubbler was 0.5 L min⁻¹ or greater to ensure a significant vapor pressure of the reagent to be transported to the CVD reactor.

Film Analysis and Characterization. Analysis of the resulting films consisted of UV/vis, adhesion tests (scratch and abrasion resistance, Scotch tape test), vis/IR reflectance−transmittance, micro-Raman spectroscopy, scanning electron microscopy and energy dispersive analysis of X-rays (SEM/EDX), glancing angle X-ray diffraction (GAXRD), X-ray photoelectron spectroscopy (XPS), and Rutherford backscattering spectroscopy (RBS).

UV/vis absorption measurements were obtained on a Thermospectronic Helios α spectrometer between 300 and 1100 nm. Reflectance−transmittance measurements were performed on a Hitachi U4000 spectrophotometer between 240 and 2600 nm. Transmittance−temperature studies were performed on a Perkin-Elmer 457 grating spectrometer set to 4000 cm⁻¹. An aluminum temperature cell controlled by *RS* resistive heaters, Eurotherm temperature controllers and *k*-type thermocouples was used to manipulate sample temperature. Sample temperature was measured by a *k*-type thermocouple taped to the film surface. Raman spectroscopy was performed on a Renishaw 1000 spectrometer using a 632.8 nm laser at 2 mW and 50× magnification; sample temperature was controlled by a Linkam THMS600 variable temperature cell with a liquid nitrogen pump.

SEM images to determine film thickness were obtained on a Hitachi S-570 scanning electron microscope at 20 kV accelerating voltage. Samples were prepared by deeply scoring the film side of the substrate to cause shelling of the film and hence a distinct edge between the film and the substrate. Energy dispersive analysis of X-rays (EDX) was performed on a Philips XL30 ESEM instrument using Inca analytical software (Oxford Instruments).

X-ray analysis of the films was determined on a Siemens D5000 machine using primary Cu Kα radiation at 1.5418 Å with a 1.5° incident angle. Diffraction patterns at different temperatures were obtained at station 2.3 of the CCLRC synchrotron radiation source at Daresbury, UK, using 1.2981 Å radiation with a 1.5° incident angle. The temperature was controlled using the aluminum sample holder described above. X-ray photoelectron spectrometry was performed on a VG ESCALAB 2201 XL instrument using monochromatic Al Kα X-rays with a pass energy of 50 eV. XPS data were analyzed using CasaXPS software version 2.0.11. Rutherford backscattered spectra were obtained using a 2 MeV accelerator; the analyzing beam was 2 MeV He⁺ with the sample being analyzed at normal incidence with a scattering angle of 168° in IBM geometry. Quark software version 1.3[15] was used for the simulation.

Results and Discussion

Film Preparation and Characterization. The goal of this work was to prepare tungsten-doped VO₂ films by APCVD. It was found that a substrate temperature at or above 500 °C was required for formation of single-phase VO₂ thin films from the APCVD reaction of VCl₄ and H₂O.[14] Gibbs free energy calculations indicate that VO₂ is the most stable phase of vanadium oxide under the conditions of the CVD reactor despite the somewhat confined formation window attributed to VO₂ in the vanadium−oxygen phase diagram.[16] Thin films of tungsten-doped vanadium(IV) oxide were obtained from the atmospheric pressure chemical vapor deposition of VCl₄, [W(OC₂H₅)₆], and water at 500−600 °C. As both precursors were shown individually to form films,[14,17] it was anticipated that the tungsten content could be controlled by altering the flow rates through the bubblers. A summary of the conditions used to prepare the films and their properties is given in Table 1. The films were yellow to dark yellow in color, passed the Scotch tape test, but were scratched with a steel scalpel and a brass stylus. The films were stable in air for over 6 months and were impervious to common solvents (water, acetone, thf, toluene). Film coverage was predominately over the half of the substrate closest to the gas inlet to the CVD coater, corresponding to the hottest area of the coater. In virtually all experiments, tungsten-doped VO₂(M) was obtained. The introduction of a tungsten precursor into the APCVD system appeared not to affect the phase of vanadium oxide obtained, and tungsten was incorporated into the VO₂(M) lattice−rather than being phase separated. The reaction conditions and film growth showed good reproducibility−runs were typically duplicated three times each. However on one occasion a repeat run gave an unexpected result as the film had a deep blue area at the leading edge of the substrate (ca. 1 cm in width and a similar thickness to the yellow areas) that was identified as VO₂(B) by X-ray diffraction−

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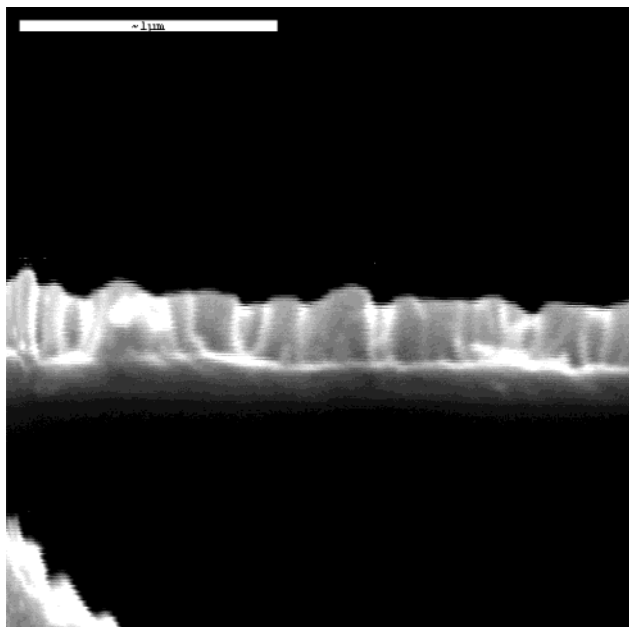(17) Riaz, U. *Thin Solid Films* **1993**, *235*, 15.

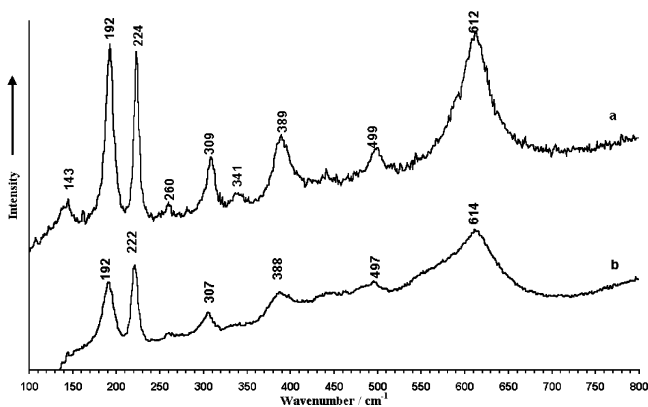**Figure 1.** Edge-on SEM of typical tungsten-doped VO$_2$ thin film on glass substrate to determine film thickness.

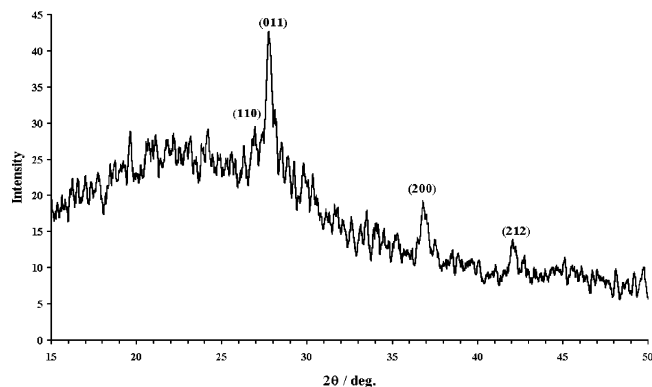**Figure 2.** Raman spectra of (a) undoped VO$_2$ and (b) tungsten-doped VO$_2$.

Figure 3. Glancing angle XRD of tungsten-doped VO$_2$ thin film on glass substrate.
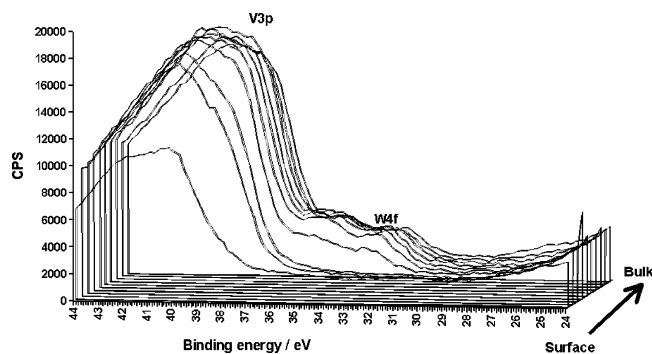
Figure 4. XPS depth profile of V3p–W4f region of 0.9 atom % tungsten-doped VO$_2$ thin film on glass substrate.

the rest of the film was VO$_2$(M). The VO$_2$(B) area did not show any thermochromic switching properties in the temperature range investigated (<80 °C).

Film growth rates, as measured by SEM imaging (Figure 1), were typically 150–200 nm min^{-1} at 600 °C. Film morphology was that congruent with the island growth mechanism typical of films grown on highly nucleating substrates. The relatively easy scratching of the films indicates relatively poor adhesion of the film to the glass surface.[18]

Monoclinic VO$_2$, was observed by Raman spectroscopy at room temperature.[19] Some broadening of the Raman bands were observed for the tungsten-doped vanadium dioxide when compared to the undoped VO$_2$, as shown in Figure 2. All Raman bands are assigned to an A_g symmetry mode.[19] The slight broadening of the Raman

bands is probably due to an increase in defects caused by the dopant ions in the lattice.

X-ray diffraction studies showed that monoclinic VO$_2$ (Figure 3) was formed in all depositions, matching database spectra (JCPDS file 43-1051). No preferred orientation was observed. Insufficient reflections were observed to give accurate cell constants. No reflections were observed that could be attributed to oxides of tungsten. EDX elemental mapping showed homogeneously distributed vanadium and tungsten, suggesting that a solid solution of tungsten in VO$_2$ is formed with tungsten ions substituting for some of the vanadium ions in the lattice. This is the ideal situation for tungsten to have an affect on the thermochromic switching temperature of the VO$_2$ thin films—phase-separated WO$_x$–VO$_2$ composites would be expected to show no reduction in the MST temperature.

XPS studies indicated that the films did not contain chlorine within the detection limits of the instrument (0.1 atom %). Also carbon contamination from the alkoxide precursor was not observed. A peak due to adventitious carbon, which served as a peak position reference, was seen on the surface of the films but was not observed after the first argon ion etching. Depth profiling by XPS showed that the tungsten was largely incorporated into the bulk of the film and was not segregated in the very uppermost surface layers. Figure 4 illustrates the tungsten content of the film increasing with depth. The depth profiling results are confirmed by RBS modeling (Figure 5). The RBS modeling indicated an overlayer of 10–20 nm of V$_2$O$_5$ on top of the main film of V$_{0.99}$W$_{0.01}$O$_2$, which was ca. 400 nm thick. The contents of the tungsten(VI) ethoxide containing bubbler were

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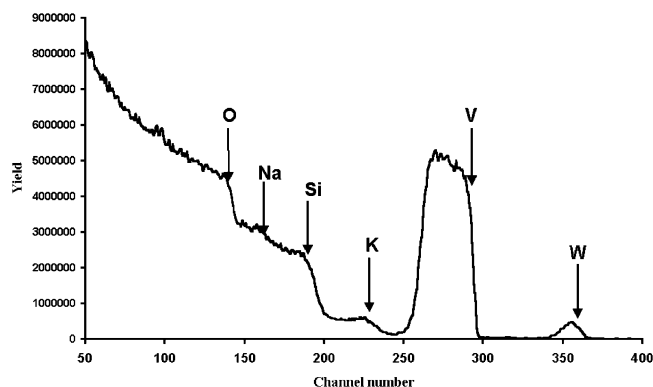
Figure 5. Rutherford backscattering spectrum of 0.9 atom % tungsten doped VO_2 on glass substrate. Na, Si, and K from glass substrate.
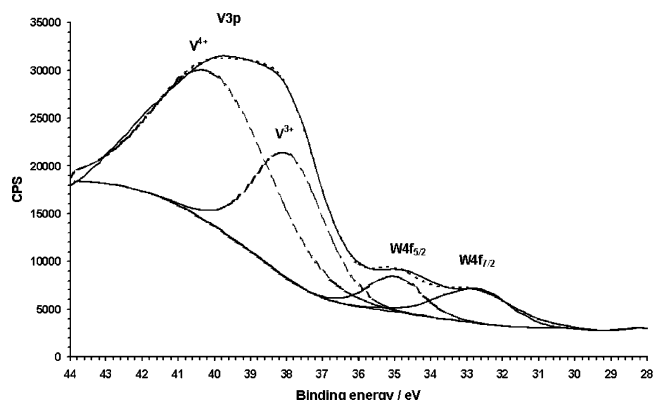

Figure 6. Fitted high-resolution XPS of V3p−W4f region. W4f peaks correspond to W^{4+}, and V3p peaks correspond to V^{4+} and V^{3+}. (- -) Deconvoluted peaks, (···) fit, (—) data.

checked after deposition and were found to contain precursor. Furthermore, as no gas delivery lines were blocked, it implies the tungsten is partially bulk segregated.

XPS was also used to elucidate the oxidation states of the ions present (Figure 6). It was found that, in the tungsten-containing layers of the thin films, vanadium was present predominantly as V^{4+}, with a V3p binding energy of 40.0 eV, and a reduced vanadium species assigned as V^{3+} ions with a binding energy of 37.9 eV. A broadening of the V2p and V3p peaks toward lower binding energy is observed as etching progresses due to the formation of reduced vanadium species that we interpret as resulting from the preferential sputtering of oxygen. This broadening increases markedly at layers containing tungsten out of proportion to that attributable to preferential sputtering, suggesting that the tungsten may also be influencing the oxidation state of the vanadium ions (control experiments with undoped VO_2 films confirm these findings). The surface showed mostly V^{4+} with a small amount of V^{5+} due to atmospheric oxidation of the VO_2 thin films. Tungsten 4f peaks were assigned as W^{4+}, with binding energies of the W4f5/2 and W4f7/2 at 34.7 and 32.4 eV, respectively. All assigned binding energies agree well with the literature values.[20] We appreciate that preferential sputtering affects can alter the oxidation states and
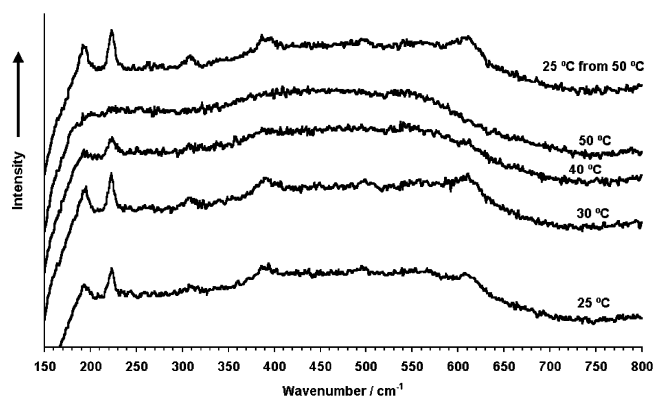

Figure 7. Raman spectra of a 0.3 atom % tungsten-doped VO_2 thin film on glass at 25−50 °C.

relative composition. However, the depth profile did agree well with the RBS model for the film. Also the positions of the tungsten peaks did not appear changed as the film was etched away and appeared to be unaffected by the etching process.

These observations relating to the oxidation states of the vanadium and tungsten are complimentary to that proposed for powdered samples of tungsten-doped VO_2. Tang et al.[21] suggest that the $V^{4+}-V^{4+}$ pairs of the monoclinic phase are disrupted by tungsten doping forming a $V^{3+}-W^{6+}$ pair and a $V^{3+}-V^{4+}$ pair. This destabilizes the semiconducting monoclinic phase of VO_2 to lower the temperature at which the MST occurs. This model may not be compatible with the films formed by this APCVD process. It appears from the XPS data that in the APCVD material the tungsten incorporates as W^{4+} in the VO_2 thin films. It should be noted that tungsten oxide films prepared from tungsten(VI) ethoxide are blue; partially reduced WO_{3-x} indicating reduced tungsten (W^{5+}) species are readily formed by CVD.[17]

The amount of tungsten present in the VO_2 films was found to be directly related to the thermochromic transition temperature. Tungsten(VI) ethoxide was found to be able to dope VO_2 with W by APCVD. However an amount of W at 1 atom % seems the maximum that can be doped using this particular precursor and CVD method. This is because of the low tungsten(VI) ethoxide vapor pressure and its thermal instability.

Thermochromic Transition Temperature. To observe the MST phase transition by Raman microscopy, a heating cell was used to raise the temperature of the films on the glass substrates at a rate of 3 °C min⁻¹. The cell was allowed to dwell at the chosen temperature for 10 min before the spectrum was obtained. A typical set of spectra is shown in Figure 7, where a VO_2(M) thin film doped with 0.3 atom % tungsten has a transition temperature of below 50 °C. As the temperature of the film is increased, the Raman bands for the monoclinic phase of VO_2 decrease in intensity until the switching temperature is achieved whereby the monoclinic VO_2 Raman bands are not observed. The tetragonal phase of VO_2 does not show a Raman pattern.[19] When cooled, the Raman bands for VO_2 (M) are again observed, illustrating the reversible nature of the transition. This

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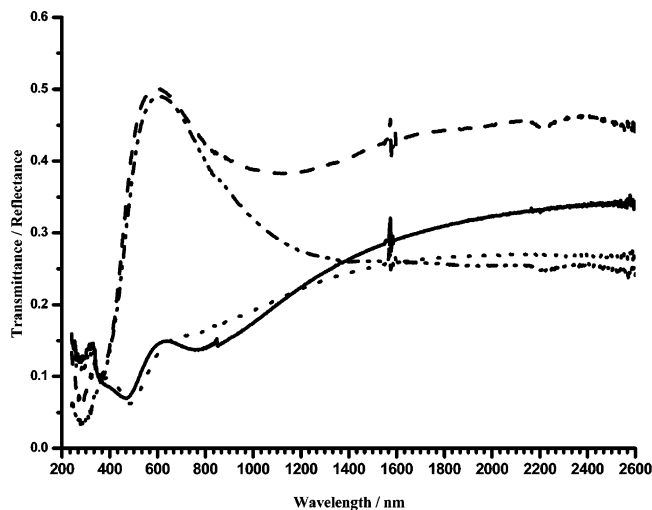
Figure 8. Reflectance−transmittance spectra of a 0.6 atom % tungsten-doped VO$_2$ thin film on glass substrate, above and below the MST temperature. (- -) Transmittance below T_c; (− · · −) transmittance above T_c; (···) reflectance below T_c; (—) reflectance above T_c.
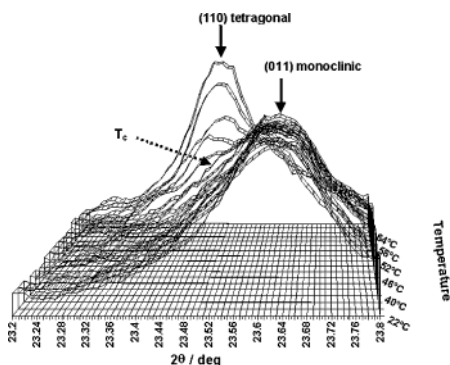

Figure 9. XRD pattern at 22−66 °C of VO$_2$ doped with 0.6 atom % tungsten. The arrow labeled T_c indicates the transition temperature of the film where the monoclinic and tetragonal reflections are approximately equal in intensity.

phase transition could be multiply cycled with no change in MST or film degradation.

A measurement of the reflectance−transmittance spectra of the films above and below the metal-to-semiconductor transition is shown in Figure 8. This indicates the films display the properties required for intelligent window coating applications—namely, a dramatic decrease in IR transmittance and an increase in IR reflectance but with little or no change in the visible region.

The same thermochromic transition was also observed from the monoclinic-to-tetragonal structural change in the X-ray diffraction pattern. This was particularly easy to see for the VO$_2$(M) (011) reflection as shown in Figure 9. The conversion of VO$_2$(M) to VO$_2$(R) removes the (011) reflection from the monoclinic phase; however, a new reflection due to the (110) tetragonal reflection is seen at slightly lower 2θ.

The thermochromic transition temperature was also monitored by the change in transmittance at 2.5 μm. The transition is particularly large at a wavelength of 2.5 μm and so is easy to observe. This wavelength is also in the region corresponding to thermal energy and so gives an indication of the effectiveness of the film as a window coating. The sample temperature was in-
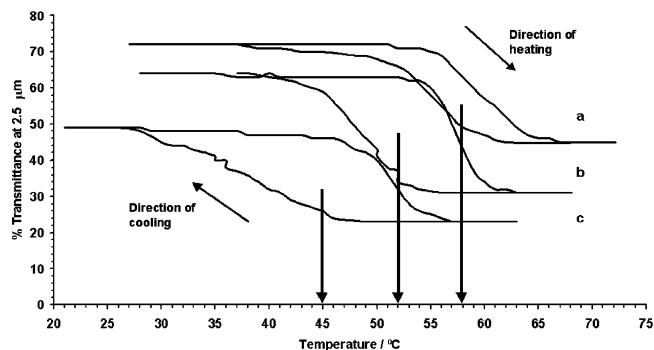

Figure 10. Transmittance at 2.5 μm against temperature for (a) 0.3, (b) 0.6, and (c) 0.9 atom % tungsten-doped VO$_2$ thin films on glass substrates. Arrows indicate T_c for each film.

creased by 2 °C min^{-1} until no further change in transmittance was observed and then allowed to cool to room temperature. Some results of this study are displayed in Figure 10.

The extent of the reduction in thermochromic transition temperature could be related to the tungsten content of the film as shown in Table 1 and Figure 10. The transition temperature was taken to be at the center of the hysteresis loop and is quoted together with the width of the hysteresis interval (i.e., $T_c \pm \Delta T$ in Table 1). A reduction of the thermochromic transition by an average of 25 °C/1 atom % W was observed for the tungsten-doped VO$_2$ thin films prepared in this study. This correlates with tungsten-doped VO$_2$ films prepared from sol−gel[8,9] and PVD studies[10,11] which generally show a 20−25 °C/1 atom % W decrease in the thermochromic transition temperature. Repeated cycling generated the same pattern each time—indicating reversibility. The width of the hysteresis loops broadened markedly with increasing tungsten content. This is discordant with results from PVD films, which show narrow hysteresis loops for tungsten-doped VO$_2$ thin films.[9] The thin films of tungsten-doped VO$_2$ prepared by APCVD in this study have a broad range of particle sizes, which may account for the wide hysteresis loops observed. This has been observed in sol−gel studies where different crystallite sizes cause variations in hysteresis width.[22] A further factor influencing the hysteresis width was experimental—as the IR beam used for the measurement heated the sample—and gave a slight disparity between the temperature measured by the thermocouple and that of the area of the film actually interrogated by the IR beam. Increasing the tungsten content of the VO$_2$ thin films decreased the transmittance of the low-temperature semiconducting phase, in agreement with films prepared by PVD methods.[10] The temperature at which the thermochromic transition of the VO$_2$ thin films occurs has been reduced to a value approaching room temperature, which may facilitate their use in a commercial product.

The lower MST temperature we assign purely to the effect of W doping rather than to a combination of doping and strain effects. This is because all films produced in this work had a thickness of 400 nm and above. Strain-induced reductions in MST from VO$_2$ prepared by PVD were only seen in films less than 300 nm in thickness.

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The maximum decrease in IR transmittance observed was 45% on passing through the metal-to-semiconductor transition for a 0.3 atom % W-doped VO_2 film with a typical decrease in transmittance of 30% being observed. The large changes in infrared transmittance and reflectance observed on passing through the metal-to-semiconductor transition would affect the heating effect of sunlight passing through the window on which the coating was applied. The minimal change in the transmittance and reflectance in the visible region of the spectrum means the color of the film remains unchanged and predictable. Improvements to the system could however be made, and further precursor combinations are planned to create APCVD VO_2 films with greater tungsten loadings.

Conclusions

Thin films of tungsten-doped vanadium(IV) oxide on glass substrates can be prepared from the atmospheric pressure chemical vapor deposition reaction of vanadium(IV) chloride, tungsten(VI) ethoxide, and water. The films displayed a reduced metal-to-semiconductor transition temperature as compared to undoped VO_2. The tungsten doped VO_2 films prepared in this study show many of the properties necessary for commercial applications as intelligent window coatings, such as a transition temperature approaching room temperature and large changes in infrared transmittance and reflectance, with minimal changes in visible properties, on passing through the metal-to-semiconductor transition.

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