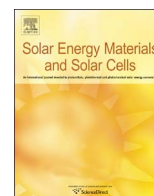




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Thermochromic vanadium dioxide thin films prepared by electric field assisted atmospheric pressure chemical vapour deposition for intelligent glazing application and their energy demand reduction properties



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ABSTRACT

Thermochromic vanadium dioxide thin films were deposited from the electric field assisted atmospheric pressure chemical vapour deposition reaction of vanadyl acetylacetonate and 2%O₂/98%N₂ at temperatures between 350 and 530 °C on to fluorine doped tin oxide coated glass substrates. A potential difference was applied between the top plate and substrate during the deposition to generate an electric field with a positive bias applied to the substrate. The films produced were analysed and characterised by X-ray diffraction, scanning electron microscopy, atomic force microscopy Raman spectroscopy, contact angle and variable temperature UV/Visible spectroscopy. It was found that the presence of an electric field during deposition could lead to a marked change in the microstructure particularly a reduced crystallite size and a reduced thermochromic transition temperature. The optical data collected in this study is used in building energy demand simulation to predict the potential energy savings achievable by using these novel thin films compared to standard industrial products, for locations with different climates. The results suggest that such glazing can have a significant energy saving effect compared to current approaches across a wide range of climate types.

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1. Introduction

Vanadium dioxide has been suggested as an intelligent glazing material for a number of years [1,2]. This application is postulated as vanadium dioxide has a reversible phase transition that occurs in the single crystal material at 68 °C [3]. This phase transition is from a lower temperature monoclinic phase to a higher temperature rutile phase [3,4]. This phase transition also results in a change in optical properties that derives from the electronic properties of the two phases [5]. The lower temperature monoclinic phase is semiconducting, whilst the higher temperature rutile phase is metallic. As such the higher temperature rutile phase becomes somewhat reflective towards light in the near infrared region of the spectrum, whilst the lower temperature

monoclinic phase is much more transmissive. As a result, films of vanadium dioxide may modulate the solar heat gain entering into buildings thus reducing the need for cooling in the summer and heating in the winter [6].

A number of challenges persist that currently prevent successful implementation of such materials in glazing applications. The first is that at 68 °C the transition temperature is too high. Energy modelling studies have indicated that a transition temperature in the region of 20–25 °C is ideal, although does depend on the local climate [7,8]. A number of reports have demonstrated that metal doping can reduce this transition temperature dramatically, with tungsten being the most efficient typically reducing the transition temperature by 22–25 °C per at% doping, although it is noted that this has a negative impact on the hysteresis behaviour of the transition [9–11]. Alternatives to metal doping to reduce the transition temperature include inducing preferential growth [12] or strain [13] into the deposited material by assiduous control of the deposition parameters or the application of electric

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fields [14] – these tend to have a less profound effect on the hysteresis behaviour of the deposited films.

Hysteresis behaviour is the second challenge. Recent energy modelling reports suggest that the ideal transition is immediate and with no hysteresis, although it was noted that transitions that were relatively sharp (complete change from transmissive to reflective states occurring over 1 °C or less) and with a low hysteresis width (3–5 °C) could still give a significant energy benefit [8,15,16]. Very few reports of vanadium dioxide films satisfy these criteria [2]. Approaches to improve these properties generally take the form of controlling crystallite size and particle size distribution [2].

The third challenge for vanadium dioxide based glazing is the colour of the film. Vanadium dioxide is a brown/yellow colour that is not considered to be saleable in Western glazing markets, although this may not be the case in China and other Eastern markets. A number of approaches have been taken to overcome this such as the use of fluorine or magnesium doping [17–20], nano-scaling [21,22] and the addition of gold nanoparticles [23,24] although these approaches have other challenges associated with them e.g. deleterious effect on the transition (doping), complexity and cost of synthesis (nano-scaling) and cost (gold nanoparticle composites).

Numerous synthetic methods have been used to prepare vanadium dioxide thin films including physical vapour deposition [25], sol-gel chemistry [26], nanoparticle suspensions [21,22], aerosol assisted chemical vapour deposition [27], atmospheric pressure chemical vapour deposition [28,29], atomic layer deposition [30] and pulsed laser deposition [31]. But none of these have been able to comprehensively solve the challenges noted above. In this work we present for the first time the synthesis of vanadium dioxide thin films using electric field assisted atmospheric pressure chemical vapour deposition. Previous work on electric field assisted chemical vapour deposition techniques has demonstrated that these techniques can be used to gain control over crystallite size, crystallographic orientation and microstructure that can have greatly beneficial consequences for the materials functional properties [14]. We further perform energy demand modelling and compare the results for the thermochromic films with some current industrial products.

2. Experimental section

2.1. Film synthesis

The experimental setup for this work is shown in Fig. 1. All of the handling lines, regulators and flow lines were made of stainless steel and were 1 cm in diameter. Gases came directly from the cylinders

and were preheated in tubing before use. The temperatures of all gas inlet lines were monitored by Pt-Rh thermocouples and Eurotherm heat controllers.

The precursor, Vanadyl Acetylacetonate [VO(acac)₂] (Aldrich 99.99%), was loaded into the stainless steel bubbler in air and heated by a heating jacket to 150 °C. The precursor was introduced to the reaction chamber by diverting hot nitrogen through the bubbler with a flow rate of 10 L min⁻¹. The oxygen source came from a plain line flow of 2 L min⁻¹ of nitrogen with 2% oxygen (BOC) which was flowed through heated tubes and met the precursor flow in the mixing chamber where they were both then transported to the reaction chamber. The reaction chamber was designed to ensure even reactor wide laminar flow. Gas flows were adjusted using suitable regulators and gas flow controllers. Exhaust from the reaction was vented directly into the fume cupboard. The reaction chamber was heated to 530 °C using a graphite block containing a Whatman heating cartridge, with temperature gradients of up to 100 °C between the top plate and the substrate. The substrate temperature was monitored using Pt-Rh thermocouples. The substrate and top plate were glass sheets (90 mm × 45 mm × 4 mm) with a fluorine doped tin oxide transparent conducting oxide layer (Pilkington-NSG). A potential difference was applied between the top plate and substrate with an AC or DC voltage with positive bias on the substrate. The top plate and substrate were separated by 1 cm allowing the precursor and carrier gas to pass between them. Thin sheets of alumina were used to stop short-circuiting between the electrodes and the reaction chamber. The samples prepared in this work are tabulated in Table 1. These were chosen from a larger range of samples [32] due to their desirable optical properties. Details for the results of this optimisation study can be found in the electronic [Supplementary information](#).

2.2. Film characterisation

The samples were cut into 1 cm × 1 cm pieces and used for analysis. Energy dispersive analysis of X-rays (EDAX) and wavelength dispersive analysis of X-rays were conducted using a Phillips XL30 ESEM instrument. Scanning electron microscopy (SEM) images were acquired on a Jeol 6301F field emission instrument. X-ray diffraction patterns were measured on using monochromated (CuK_{α1+2}) radiation in the reflection mode using a glancing incident angle 5°. Raman spectra were acquired on a Renishaw Raman system 1000 using a helium-neon laser of wavelength 632.8 nm. The Raman system was calibrated against the emission lines of neon. Ultraviolet/visible (UV/Vis) spectroscopy was conducted on a Perkin Elmer Lambda 950 instrument. Variable temperature measurements were made using a custom-built heater cell that has been described previously [12].

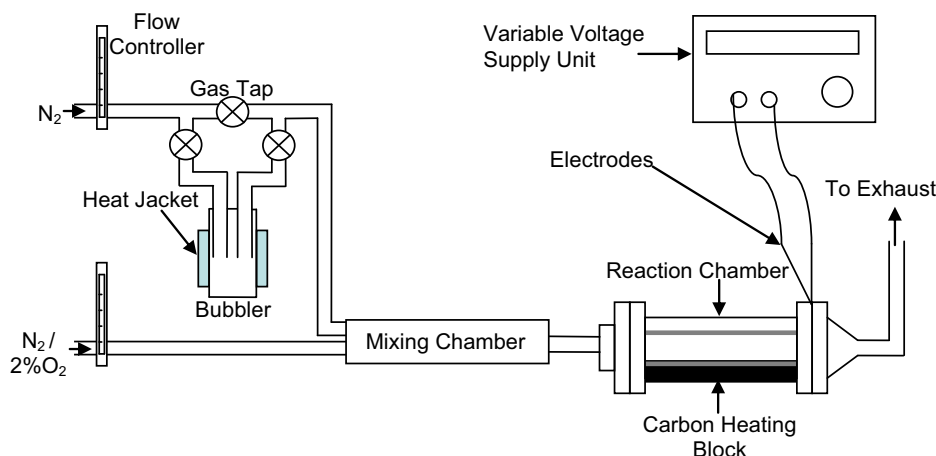


Fig. 1. Electric Field EA(AP)CVD rig schematic.

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