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Thermochromic vanadium oxide coatings grown by APCVD at low temperatures

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Abstract

Amorphous vanadium dioxide coatings were deposited on SnO_2-precoated glass substrates at 400 ^oC by atmospheric pressure chemical vapor deposition for various vanadium precursor flow rates. The coatings were characterized by X-ray diffraction, Raman spectroscopy, Fourier transform infrared spectroscopy and scanning electron microscopy. Their thermochromic performance has been found to be independent on the vanadium (V) triisopropoxide flow rates. All vanadium dioxide films present a reversible transition behavior at 68 ^oC as derived from the transmittance studies.

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Keywords: Atmospheric pressure chemical vapor deposition; Vanadium dioxide; Thermochromics; Transition temperature.

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1. Introduction

A lot of attention is given to materials, which can respond in a desired way to an external stimulus (i.e. temperature). As such, thermochromic VO_2 coatings exhibit a reversible semiconductive state to a metallic state at a critical temperature called transition temperature (T_c), while maintaining visible transparency (Morin, 1959; Nag et al., 2008).

Chemical vapor deposition (CVD) routes are attractive for the production of VO_2 on glass, since the characteristics of the materials can be simply controlled via the growth temperature and the vapor flow in the coating zone (Manning et. al., 2004; Vernardou et. al., 2006). In addition, the simplicity of CVD, particularly when performed at atmospheric pressure (APCVD), makes such a process compatible with in-line glass manufacturing processes.

The aim of this work is to study the effect of vanadium (V) triisopropoxide ($VO(OC_3H_7)_3$) vapor carrier gas flow rate on the structure, morphology and thermochromic behavior of the coatings grown at 400 ^oC by APCVD.

2. Experimental

The APCVD reactor used in this work is an in-house design and consists of a cold-wall reactor connected to an arrangement of stainless-steel heated pipes, valves and bubblers as reported previously (Vernardou et. al., 2006; Vernardou et. al., 2007; Vernardou et. al., 2011). $VO(OC_3H_7)_3$ bubbler was heated at 50 ^oC, while the gas lines were kept at 60 ^oC to avoid any condensation. The carrier gas was nitrogen, which was passed through the reactor during all depositions. The vanadium oxide growth was performed at 400 ^oC for vanadium precursor flow rate of 2.5, 3, 3.5 and 4 $l.min^{-1}$. The deposition time for all samples was 30 min. The substrates used during the APCVD experiments were commercial SnO_2-precoated glass (Uniglass, Greece), all of dimensions 2 x 2 x 0.4 cm^3. Prior to deposition, all substrates were cleaned with H_2O and detergent, rinsed thoroughly with H_2O and deionised H_2O, and allowed to dry.

X-ray diffraction (XRD) measurements were carried out in a Siemens D5000 Diffractometer for 2θ = 10.00-60.00 ^{o}, step size 0.02 ^{o} and step time 30 s/^{o}. Raman spectroscopy was done in a Nicolet Almega XR micro-Raman system using a 473 nm laser. Fourier transform infrared spectroscopy (FTIR) was performed in a Bruker IFS 66v/S spectrometer at a resolution of 4 cm^{-1}, sample and background scan time of 30 scans in the mid-IR region. Surface characterization was accomplished in a Jeol JSM-7000F field-emission electron microscope. The transmittance was measured using a Perkin-Elmer Lambda 950 UV-Vis spectrophotometer in the region of 300-1100 nm at 25 and 90 ^oC. Finally, transmittance/temperature studies were fulfilled at 1100 nm.

3. Results and Discussion

The coatings produced during the APCVD reaction of $VO(OC_3H_7)_3$ were green, adhesive and passed the Scotch tape test. They were insoluble in water and common organic solvents. Higher vanadium precursor flow rates led to better coating uniformity and coverage over the whole substrate surface. This behaviour may be due to the increased collision rate of the species in the gas-phase with those adsorbed in the substrate resulting in higher vanadium species concentration. By increasing the precursor flow rate from 2 to 4 $l.min^{-1}$, the thickness of the coatings raised as indicated from the respective decrease in transmittance by half (not shown here).