



A photochromic single glass pane

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ABSTRACT

The transmittance of a photochromic (PC) window is reduced in sunlight but returns to its original value in the dark similar to photo-sensitive sun glasses. A 30 cm × 30 cm double glazed window containing a PC multi-layer coating on a single glass pane has been developed. A single PC glass pane can be used to build a window with a low heat transfer coefficient, e.g. double or triple glazed unit, using existing technology. To develop the PC glass with a multi-layered coating that combines chromogenic and photoactive materials we employed sol-gel chemistry approaches and up-scaled the synthesis. Double glazed windows have been assembled using PC glass with two different layer configurations. The transmittance (T_{vis}) was reduced from 76% to 35% under outdoor solar radiation (750 W/m²) within 15 min.

1. Introduction

Smart windows bring a dynamic function to the building envelope that enables control over excessive illumination of the interior and control over the building's heat loss when there is surplus solar energy. Electrochromic (EC) windows have been well-researched and commercial systems that allow the active management of sunlight are available [1,2]. However, varying the transmittance of an EC window requires an external electrical power supply and the wiring together of individual EC units, which adds significantly to the complexity and cost of the building envelope. A solution would be a self-powered smart window. Advanced approaches have focused on combining the components of a dye sensitized solar cell (DSSC) and a chromogenic layer to create a photoelectrochromic (PEC) device [3–6]. In a PEC device the solar cell provides the energy necessary to colour the electrochromic layer when illuminated by generating electrons that reduce the electrochromic layer leading to the formation of colour centres and consequently to the coloration of the PEC layer. The PEC device was first reported by Bechinger et al. in 1996 [3] and has been significantly modified by Hauch et al. [4]. In the Bechinger's PEC device the photoactive and electrochromic layer were positioned on separated electrodes while Hauch et al. [4] placed the electrochromic layer beneath the photoactive layer. This modification enables a faster response, a deeper colouration of the PEC and allows the device to colour without having to short circuit the electrodes. Tuning of the window transmittance under illumination is possible by short circuiting the electrodes or adding a resistor. In addition, a PEC device can be made to function as a traditional EC window by applying an external voltage [4]. So far, the

best performance from a PEC device was achieved using porous sol-gel materials resulting in a change in transmittance (T_{vis}) of 62% (bleached state) to 6% (coloured state) within 10 min under 1 sun illumination [5]. The first PEC device with a high optical contrast prepared using sputtered layers was reported in 2017 by Bogati et al. [6]. Other studies have investigated exchanging components of the DSSC with materials used in perovskite solar cells and as such these PEC systems are often referred to as photovoltacromic devices [7]. A critical review of PEC devices and the different photoactive and chromogenic materials used on their preparation is given by Cannavale et al. [8].

It should be emphasized that both EC and PEC devices are characteristic “sandwich” type devices, i.e., two TCO substrate electrodes (typically glass) separated by an electrolyte. In an energy efficient window with a low U-value (double or triple glazed unit) such a device replaces one of the glass panes. Therefore, the addition of a chromogenic function significantly adds to the weight of the window. In this regard, a single chromogenic glass is highly advantageous, since it can be used to build a smart double glazed unit (DGU) using existing technology without adding additional weight. There have been some attempts to create EC [9] and PEC [10] based on a single substrate device, but until now they have been limited in size and performance [9,10].

This study reports on the design of a photochromic (PC) window, based on a simplified version of a PEC device [11–15] capable of self-powering and colouring under solar illumination, without the option to vary its transmittance. A commercially important advantages of such a device are: the two TCO electrodes are not needed and the realisation of a single smart glass pane. In such a PC system two competitive reactions

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influence its transmittance, the generation and the recombination of electrons (loss reactions). When illuminated the generation of electrons prevail leading to coloration while in the absence of light the loss reactions bleach the device. A detailed description of the working principles of the PC system is given in [11,15]. The goal, therefore, was to develop two large PC demonstrators (30 cm × 30 cm) with different configurations of an electrochromic layer, dye sensitized TiO₂ and an electrolyte [4–6].

2. Experimental

All the materials for the PC coatings were prepared using sol-gel chemistry. The up-scaling of the sol-gel synthesis (1 l) was necessary to prepare 30 cm × 30 cm PC demonstrators. For the electrochromic layer, a modified WO₃ sol with the addition of titanium, c-WO₃, was chosen due to the best fit of its redox potential [13]. The use of a three-dimensionally stable solid gel electrolyte was essential for making a single substrate PC device. Solidification of the redox electrolyte is important also for upscaling and for the stability of the device [16]. The SiO₂ barrier on the float glass substrate was applied to prevent the diffusion of sodium ions from the glass into the layers during the annealing step.

Two PC demonstrators with the following configurations were prepared:

(PC1) glass/SiO₂/c-WO₃/TiO₂/dye/electrolyte//N₂//glass and
(PC2) glass/SiO₂/TiO₂/c-WO₃/dye/electrolyte//N₂//glass.

2.1. Materials

One litre of sol and 100 ml of electrolyte were prepared according to the following recipes:

SiO₂:

Abst. EtOH (puriss. p.a., absolute, ≥ 99.8%, Honeywell), 0.01 M nitric acid (ACS, Sigma-Aldrich) and tetraethyl orthosilicate (reagent grade, 98%, Aldrich) were mixed in a volume ratio of 9:2:6, followed by 5 min of stirring. The stock solution was left for 10 h, after which the sol was diluted with abst. EtOH in a volume ratio of 1:2.

c-WO₃:

The EC layer was a highly symmetric crystalline, cubic WO₃. Symmetry was induced by incorporating titanium into the WO₃ matrix (c-WO₃) via the peroxy sol-gel route. The details of the synthesis are described in full in [13,14].

TiO₂:

A TiO₂ sol was obtained by mixing titanium isopropoxide (puriss, Fluka), ethyl acetoacetate (for synthesis, Merck), 2-propanol (99.5%, Sigma-Aldrich) and poly(propylene glycol) bis(2-amino propyl ether), 2-APPG (Aldrich, average Mw ~ 4000). Full details are given in [14].

Electrolyte:

Preparation of the three-dimensionally stable electrolyte involved two steps. The first step involved the synthesis of a modified silane network – a ormosilane. According to the procedure reported in [17], the ormosilane was prepared by acylation of 3-(triethoxysilyl)propyl isocyanate (95%, Sigma-Aldrich) and 2-APPG. The reaction forms a precursor composed of poly(propylene)oxide bis-endcapped with triethoxysilane groups. Afterwards, LiI (puriss, Fluka) and I₂ (puriss, Merck) were dissolved in abst. EtOH and the solution mixed with the ormosilane in a mass ratio 1:1 to give a final concentration of LiI and I₂ in the electrolyte of 0.5 M and 0.005 M, respectively [5]. The hydrolysis/condensation reactions were catalysed using acetic acid. Full details can be found in [18].

2.2. Assembling a 30 cm × 30 cm PC device

All layers (SiO₂, c-WO₃, TiO₂) were deposited on a pane of float glass (30 cm × 30 cm or 5 cm × 5 cm, Pilkington UK) using the dip-coating technique. For this, a special in-house designed glass vessel

with an expanded top section was built to reduce the quantity of the sol required. The vessel contains approximately 600 ml of sol. Pulling speeds were: 12.5 cm/min for SiO₂ and 11.0 cm/min for TiO₂ and WO₃. Each dip-coated layer was then annealed at 450 °C prior to the deposition of the next layer. To avoid the glass substrate breaking during annealing the layers were annealed twice, first by heating from room temperature to 450 °C at 3.5 °C/min and then for 1 h at 450 °C. On inspection, the layers were found to be transparent, homogenous in thickness and without cracks. The thickness of the WO₃ and TiO₂ layers was 300 nm and 200 nm, respectively.

The glass/SiO₂/c-WO₃/TiO₂ (PC1) and glass/SiO₂/TiO₂/c-WO₃ (PC2) were then immersed in 1 × 10⁻⁴ M solution of a ruthenium complex dye N719 (di-tetrabutylammonium cis-bis(isothiocyanato)bis(2,2'-bipyridyl-4,4'-dicarboxylato)ruthenium(II), Solaronix) in ethanol for 15 h.

After dying, the electrolyte was coated directly on top of the multi-layers. This enabled the deposition of a homogenous, transparent and three-dimensionally stable electrolyte coating with a thickness of 50 μm. Finally, the “PC (multi-layered) glass” was dried overnight.

The PC glass pane and a second uncoated glass pane were bonded together using double-sided sticky tape (3 M™ VHB™ 4910 P, 9 mm × 1 mm). Polyisobutylene was placed around the edges to act as a second seal with barrier properties (EGOFERM 1 × 6 mm, 30MX22/K, GRAU) and the cavity between the two glass panes filled with N₂. Fig. 1 shows a schematic of the PC1 demonstrator. The PC2 schematic is the same as for PC1, but the order of the TiO₂ and WO₃ layers is reversed.

For optical characterization, smaller PC devices (5 cm × 5 cm) were fabricated using an identical layer composition to PC1 and PC2, and flushed with N₂.

2.3. PC characterization

All PC samples were exposed to outdoor solar irradiation (750 W/m²). The spectral responses of PC1 and PC2 devices in coloured and bleached state were measured using a UV-Vis spectrometer (Lambda 950, Perkin Elmer). The *L*, *a*, *b* and *C* values of the PC devices were extracted from the measured UV-vis spectra. The kinetics were evaluated indoor by exposing the PC1 and PC2 devices to 1 sun illumination (AM 1.5, 1000 W/m²) using a solar simulator Oriel 69907 (Newport) and a Si-photodiode [13].

3. Results and discussion

The study confirmed that up-scaling of the sol-gel synthesis to 1 l for the materials used to fabricate a 30 cm × 30 cm PC device is possible. The c-WO₃, TiO₂ and SiO₂ sols used for dip-coating were stable for > 12 months. The dip-coated layers were transparent, homogenous in thickness and without cracks. A solid ormosilane electrolyte based on ormosilane was used since this kind of electrolyte containing a iodine/iodide redox couple has been successfully used in PEC devices [5,16].

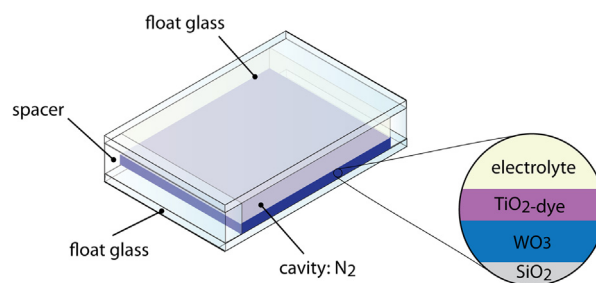


Fig. 1. Schematic representation of a photochromic demonstrator (PC1). The multi-layer sol-gel coatings are deposited on a single glass substrate and covered with a second glass pane using a spacer. The window cavity is filled with N₂.

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