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Substrate-Temperature Effects on Optical and Photoinduced Hydrophilic Properties of Sputtered TiO_2 Films on ITO Glass

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Abstract: ***Titanium dioxide (TiO_2) thin films combine UV-activated photocatalysis with photoinduced hydrophilicity, making them attractive for self-cleaning glass coatings. We grew 500 nm TiO_2 thin films on ITO-coated glass substrates via RF magnetron sputtering. Using a rutile-phase target, we carried out the deposition across five substrate temperatures, from room temperature (22 °C) up to 200°C. Optical absorption and transmittance were characterised by UV–V is spectrophotometry over 200–450 nm, and photoinduced wettability was quantified from the water contact angle after 3 min of UV irradiation (365 nm, 400 W). All five films absorbed strongly in the UV region while remaining approximately 90 % transparent in the visible range, supporting their use as glazing. The post-illumination contact angle decreased monotonically with substrate temperature, from 38.7° at 22 °C to 13.6° at 200 °C, indicating a denser, more photoreactive surface at higher growth temperatures. A companion sol–gel-derived TiO_2 film of matched composition reached a higher steady-state contact angle under the same illumination, showing stronger hydrophilicity for the sputtered film. These results support RF sputtering as a reproducible route to transparent, self-cleaning TiO_2 coatings on conductive glass and identify substrate temperature as a practical parameter for tuning photoinduced hydrophilicity.***

Keywords: Titanium dioxide; RF magnetron sputtering; substrate temperature; photocatalysis; photoinduced hydrophilicity

1. Introduction

Titanium dioxide (TiO2) has been at the center of photocatalysis research ever since Fujishima and Honda's landmark discovery of UV-driven water splitting on a TiO2 electrode [1]. Under illumination with photons of energy exceeding its band gap, TiO2 generates electron–hole pairs capable of driving strong reduction and oxidation reactions at its surface, which underlies its use in air and water purification, self-cleaning coatings and dye-sensitised solar cells [3, 2]. Compared with other wide-band-gap photocatalysts such as CdS, SnO2, PbS and ZnO, TiO2 is favoured because it is chemically stable, non- toxic, resistant to photocorrosion, and has a band gap (∼3.2 eV for anatase, ∼3.0 eV for rutile) well matched to near-UV excitation [2]. Because both the photocatalytic activity and photo-induced wettability of TiO2 depend heavily on film crystallinity and available surface-active sites, the underlying deposition technique and growth parameters play a critical role in deciding final application performance. This drives the need for a systematic study on how synthesis conditions- especially substrate temperature- impact the optical and surface behavior of TiO2 thin films deposited via a scalable physical vapor deposition route.

2. Literature Survey

2.1. Principle of Photocatalysis

Photocatalysis begins when a material absorbs light with photon energy higher than its band gap. This promotes an electron from the valence band into the conduction band, simultaneously generating a hole in the valence band. (figure 1). The photogenerated hole is a strong oxidiser and the photogenerated electron a strong reductant; when these charge carriers reach the film surface they drive oxidation and reduction half-reactions with adsorbed species (typically O_2 and H_2O), pro-ducing reactive radicals such as superoxide ($O_2^{\bullet-}$) and hy-

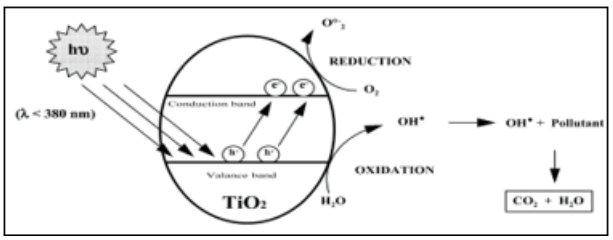

Figure 1: Mechanism of T_1O_2 photocatalysis under UV excitation ($\lambda < 380$ nm). The resulting photogenerated

e^-/h^+ pairs migrate to the surface to drive oxidation and reduction processes with adsorbed moisture and oxygen.

droxyl ($OH^•$) that can mineralise adsorbed organic pollutants to CO_2 and H_2O [3]. Because this process scales with the density of photoexcited carriers that reach the surface before recombining, particle or grain size strongly affects photocatalytic efficiency: reducing the feature size toward the nanometre regime increases the surface-to-volume ratio and hence the achievable reaction rate [2]. This has motivated extensive interest in nanostructured TiO_2 thin films for applications including dye-sensitised solar cells and micro-fluidic passive valves.

2.2. Photoinduced Hydrophilicity

When UV light illuminates a TiO_2 surface, the electron–hole pairs produced at the surface can be trapped at bridging lat-tice oxygen sites, generating surface oxygen vacancies. Water molecules adsorb dissociatively at these vacancy sites, and the resulting hydroxyl (Ti–OH) groups accumulate on the surface, progressively lowering its water contact angle until the surface becomes hydrophilic, or in the limiting case superhydrophilic (contact angle below 5°) [5, 4]. When UV illumination stops, the film gradually relaxes: atmospheric oxygen refills the vacancies, fewer hydroxyl groups are regenerated, and the hydrophilicity decays back toward the pre-illumination value [5]. Because this mechanism depends on the surface density of accessible oxygen-vacancy/ hydroxylation sites, it is directly sensitive to film density, crystallinity and microstructure – all of which are influenced by the substrate temperature used during growth.

2.3. TiO_2 Thin Film Deposition Techniques

Several routes have been used to prepare TiO_2 thin films, including sol–gel deposition, spray pyrolysis, chemical vapour deposition and physical vapour deposition by sputtering [4]. Among these, RF magnetron sputtering is attractive because it gives dense, well-adherent, highly reproducible films whose thickness, composition and microstructure can be controlled independently of substrate chemistry, and it scales readily to large-area, industrially relevant coatings [6]. Sputtered TiO_2 films deposited directly onto glass and onto transparent con-ducting substrates have previously been reported to show use-ful photocatalytic and optical properties [8, 9, 10, 11], and the substrate temperature during growth has been identified as an important parameter governing film crystallinity, optical transmittance and band gap [7]. Doped TiO_2 systems (e.g. with SiO_2 or Al_2O_3) have achieved even lower steady-state con-tact angles, below 2°, by further increasing the density of sur-face hydroxylation sites [5], and comparative studies of sputtered versus sol–gel TiO_2 films report systematic differences in film density and photoresponse arising from the two deposition routes [12].

3. Problem Definition

Although TiO_2 thin films have been widely investigated, very few works examine the effect of substrate temperature on ITO-supported sputtered films while jointly monitoring their optical clarity and photo-wettability. Additionally, benchmarking these films directly against a compositionally matched sol–gel reference is rarely reported. In particular, it is not well established, within a single controlled experiment, how far a simple and industrially practical process variable– substrate-holder temperature during RF sputtering – can be used on its own to tune the photoinduced hydrophilicity of a TiO_2 coating without compromising the visible-range transparency required for glazing applications. The present study addresses this gap by depositing 500 nm-thick TiO_2 films on ITO glass by RF magnetron sputtering at five substrate temperatures (22–200 °C), characterising their UV–Vis optical response, and quantifying their photoinduced hydrophilicity through water contact angle measurements before and after UV illumination, with a direct comparison to a sol–gel-derived film.

4. Methodology / Approach

4.1. Principle of RF Magnetron Sputtering

RF magnetron sputtering deposits a thin film by physical vapour transport (figure 2). An RF field (typically 13.56 MHz) sustains a low-pressure argon plasma between a grounded chamber and a target electrode; a magnetic field confines energetic electrons close to the target, increasing the local ionisation rate. Argon cations (Ar^+) generated in this plasma are accelerated by the electric field toward the negatively biased target and bombard its surface, ejecting target atoms (and, for a compound target, associated fragments) by momentum trans-fer. The ejected species traverse the chamber and condense on the substrate, building up the film atom by atom [6].

Because the target material is sputtered rather than melted or evaporated, the process is highly reproducible and gives dense, well-adherent films with good thickness uniformity over large areas, which is why it was chosen for the present study.

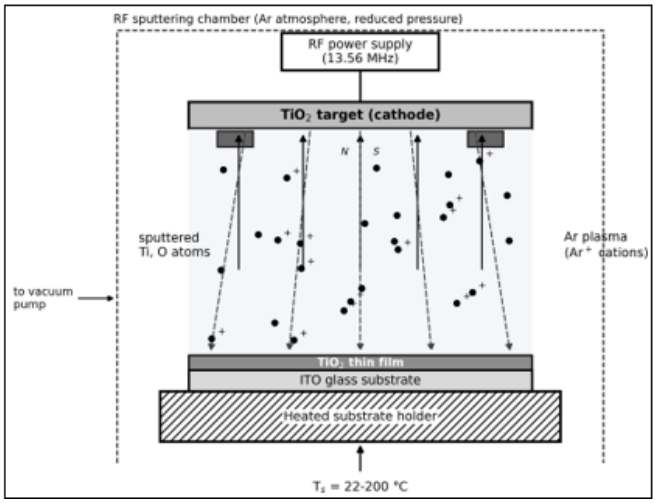

Figure 2: Schematic breakdown of the RF magnetron sputtering setup. The diagram shows Ar+ ions from the RF plasma striking the target to eject the Ti and O coating species. These then condense and build up on the heated ITO glass substrate.

4.2. Sputtering Target Characterisation

In this study, a high-purity (99.99 %) sintered TiO_2 target (Ko-jundo Chemical Laboratory Co., Ltd, Japan) was used as the sputtering source. X-ray diffraction of the as-received target (figure 3) confirmed a rutile crystal structure, indexed to the (110), (101) and (220) reflections. It is important to emphasize that the crystal structure of the sputtering target does not directly dictate the structure of the deposited film. During sputtering, the target lattice breaks down into individual ejected species, which then re-nucleate and condense on the substrate. Consequently, the phase of the final film depends primarily on the substrate temperature and overall growth kinetics, rather than the original target phase [2]. TiO_2 is thermodynamically most sta-ble in the rutile phase, whereas the anatase phase, favoured at lower growth or annealing temperatures, is metastable and converts irreversibly to rutile on annealing above approxi-mately 600–800 °C [2, 7].

4.3. Substrate Preparation

ITO-coated glass (sheet resistance ≈ 7 Ω/sq) was cut into 3 cmx 2 cm substrates. Substrates were cleaned with isopropyl alcohol to remove surface particulates while preserving the electrical conductivity of the ITO layer.

4.4. Thin Film Deposition

TiO_2 films were deposited by RF-gun sputtering of the tar-get described in Section 4.2, at a fixed nominal thickness of 500 nm. Five sets of samples were prepared, differing only in the substrate-holder temperature during growth: 22, 50, 100, 150 and 200 °C (22 °C being the unheated, ambient condition).

4.5. Optical and Wettability Characterisation

The optical absorption and transmittance of each film were measured with a UV–V is spectrophotometer over 200–450 nm, spanning both the UV band relevant to photocatalytic activation and the near-visible range relevant to glazing transparency.

Photoinduced wettability was assessed by water contact angle goniometry. A total of six samples- the five sputtered TiO2 films along with a bare ITO glass reference- were exposed to a 365 nm, 400 W UV source for 3 minutes. The spectral power distribution of this source is presented in Figure 4. Immediately after illumination, the water contact angle was measured for each sample A smaller contact angle indicates stronger photoinduced hydrophilicity and, by extension, a more active photo-catalytic surface [5]. As a benchmark, an additional TiO_2 film of matched nominal composition prepared by the sol–gel route was measured under the same illumination protocol at three exposure times (0, 5 and 10 min) for comparison with the sputtered film.

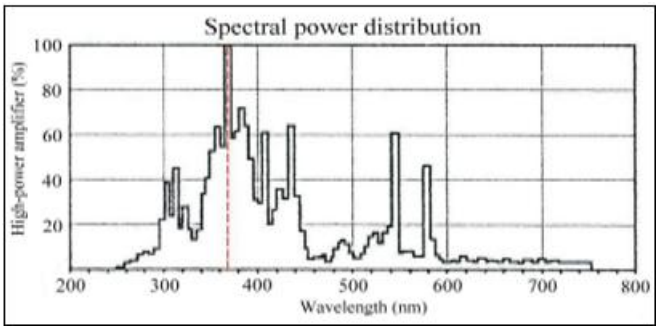

Figure 4: Measured spectral power distribution of the 365 nm UV-illumination source used for the photoinduced-hydrophilicity tests; the peak emission (dashed line) is centred at 365 nm.

5. Results and Discussion

5.1. Structural Considerations

As noted in Section 4.2, only the sputtering target (rutile phase, figure 3) was structurally characterised by XRD in this study; grazing-incidence XRD of the deposited films themselves was not performed. This is a limitation of the present data set: because the substrate temperatures used here (22–200 °C) are well below the temperature range typically required to crystallise sputtered TiO_2 into a clearly resolved anatase phase (annealing or growth temperatures of order 300–600 °C in com-parable studies [7]), the films in this study are expected to be amorphous or only weakly crystalline throughout the temperature range investigated. The trends discussed below (Sections 5.2–5.4) should therefore be interpreted as arising primarily from temperature-dependent changes in film density, packing and surface defect chemistry rather than from a bulk anatase–rutile phase transition.

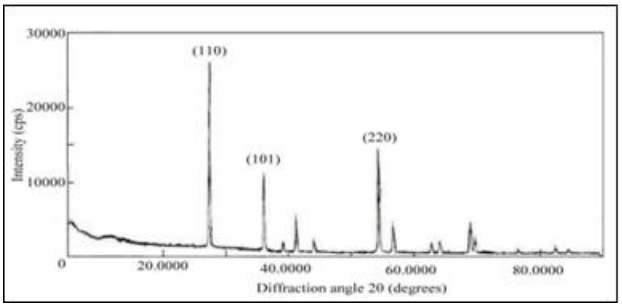

Figure 3: X-ray diffraction pattern of the sintered TiO_2 sputtering target, showing rutile-phase (110), (101) and (220) reflections.

5.2. Optical Absorption and Transparency

Consistent with the strong UV absorption expected of TiO_2, all five films showed pronounced absorption in the 200–250 nm and 280–360 nm bands, with the substrate temperature having little discernible effect on absorption below 300 nm (figure 5). This UV-absorbing behaviour is consistent with the strong sub 380 nm absorption reported for sputtered TiO_2 films on glass and ITO substrates elsewhere [8, 11].

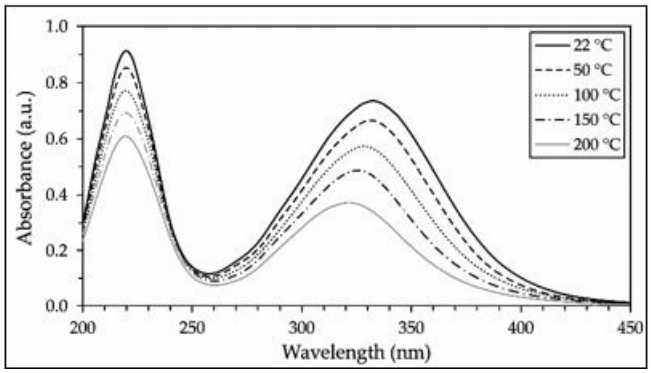

Figure 5: Placeholder for the UV–Vis absorption spectra referenced in the text. The image file for this measured curve was not present among the figures supplied for this revision and must be inserted by the authors from the original spectrophotometer output.

Across the same five samples, visible-range transmittance was approximately 90 % (figure 6), comparable to the high visible transmittance reported for RF-sputtered TiO_2 films on glass in the literature [7], and supports the suitability of sputtered TiO_2 on ITO glass for glazing applications where both optical clarity and UV-activated self-cleaning are required.

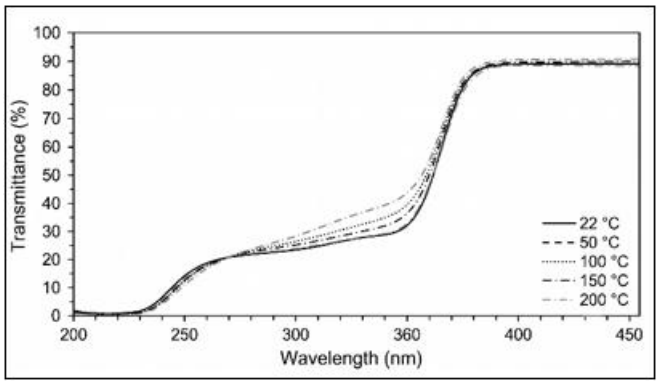

Figure 6: Placeholder for the UV–Vis transmittance spectra referenced in the text. As with figure 5, this measured curve must be inserted by the authors from the original spectrophotometer output before the manuscript is submitted.

5.3. Photoinduced Hydrophilic Property

Figure 7 displays the water droplet profiles recorded to measure the contact angle of the bare ITO glass reference and the five sputtered TiO 2 films right after 3 minutes of UV exposure. Figure 8 summarizes these results by plotting the post-illumination contact angle as a function of substrate temperature. The uncoated ITO glass reference showed the largest contact angle (59.2°), while every TiO_2-coated sample was substantially more hydrophilic. Within the coated series, the contact angle fell monotonically with increasing substrate temperature, from 38.7° at 22 °C to 32.1° at 50 °C, 24.7° at 100 °C, 18.0° at 150 °C and 13.6° at 200 °C.

This trend is consistent with the mechanism described in Section 2.2: a higher substrate temperature during sputtering is expected to give adatoms more surface mobility during growth, favouring a denser film with fewer voids and a larger density of accessible surface sites for oxygen-vacancy formation and subsequent hydroxylation under UV exposure. Similar temperature-dependent improvements in the photoresponse of sputtered and other physical-vapour-deposited TiO_2 films have been reported elsewhere [7, 10].

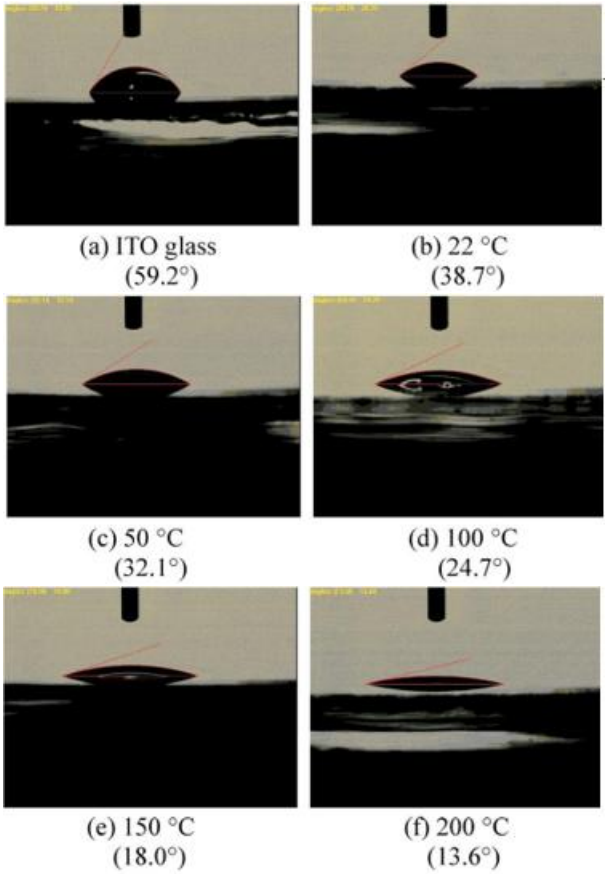

Figure 7: Water droplet contact-angle images recorded immediately after 3 min of UV illumination (365 nm, 400 W): (a) uncoated ITO glass, 59.2°; TiO2 films grown at (b) 22 °C, 38.7°; (c) 50 °C, 32.1°; (d) 100 °C, 24.7°; (e) 150 °C, 18.0°; (f) 200 °C, 13.6°.

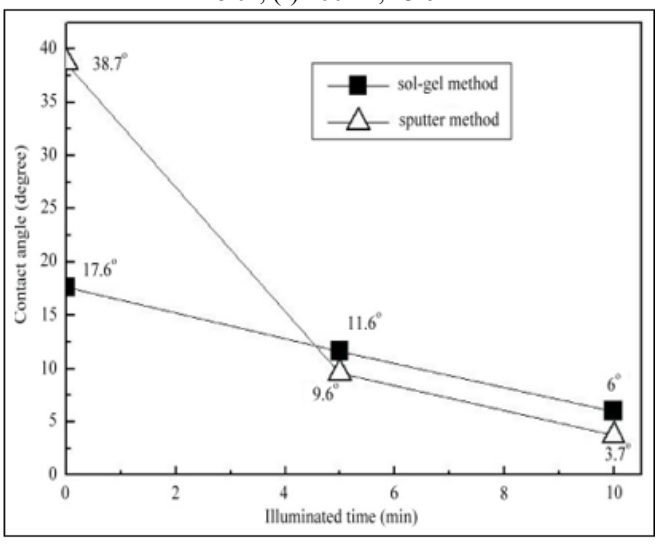

Figure 8: Effect of substrate temperature on the post-illumination water contact angle of sputtered TiO_2 films. Higher growth temperatures lead to a steady, monotonic drop in contact angle (higher surface wettability).

5.4. Comparison with a Sol–Gel-Derived TiO_2 Film

Figure 9 compares the contact-angle recovery of the RF-sputtered TiO_2 film (grown at 22 °C) with a TiO_2 film of matched nominal composition prepared by the sol–gel route, both illuminated under the same protocol. Before illumination, the sol–gel film exhibited a smaller contact angle (17.6°)

than the as-deposited sputtered film. After 5 min of UV exposure the two films were similar (11.6° sol–gel vs. 9.6° sputtered), and by 10 min the sputtered film had reached a lower contact angle (3.7°) than the sol–gel film (6°). This indicates that, although the sol–gel film started more hydrophilic, the sputtered film became comparatively more hydrophilic un-der continued UV exposure. Because sputter-deposited oxide films are generally denser and more compositionally uniform than sol–gel films, which often retain residual porosity and organic precursor by-products from calcination, this is a plausible explanation for the difference, consistent with comparative sputtered-vs.-sol–gel TiO_2 studies reported elsewhere [12]; a direct structural comparison (e.g. by AFM or porosimetry) of the two film types was not performed here and would help con-firm this interpretation.

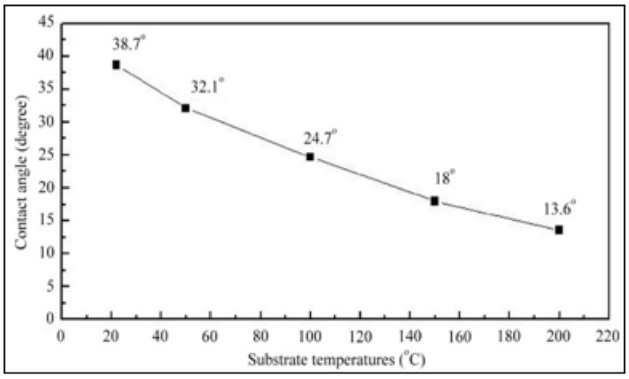

Figure 9: Time-dependent reduction in water contact angle under UV light for the 22 °C RF-sputtered TiO 2 film, plotted alongside a compositionally matched sol–gel TiO 2 control

6. Conclusion

TiO2 thin films (500 nm) were deposited on ITO glass by RF magnetron sputtering of a rutile target at five substrate temperatures between 22 °C and 200 °C. All films combined strong UV absorption with approximately 90 % visible transparency, supporting their use as transparent, self-cleaning glazing coatings. The water contact angle measured immediately after UV illumination decreased monotonically with substrate temperature, from 38.7° at 22 °C to 13.6° at 200 °C, showing that substrate temperature is an effective and simple process parameter for tuning the photoinduced hydrophilicity of sputtered TiO2 films. Relative to a sol–gel-derived film of matched composition, the sputtered film reached a lower steady-state contact angle under continued UV exposure, indicating a stronger sustained hydrophilic response. Together, these results support RF magnetron sputtering as a reproducible route to transparent, photocatalytically self-cleaning TiO2 coatings on conductive glass, with substrate temperature.

7. Future Scope

Several extensions of this work would strengthen and broaden its conclusions. First, the complete measured UV–Vis absorption and transmittance data sets for all five samples (Sections 5.2, figures 5 and 6) should be obtained from the original spectrophotometer records and included in the final manuscript, since the present revision only carries the qualitative trends described in the results text. Second, film-level structural characterisation– grazing-incidence XRD together with AFM or SEM imaging of surface morphology- is recommended to confirm directly whether the observed hydrophilicity trend correlates with changes in crystallinity, grain size or surface roughness, as proposed in Section 5.1. Third, conducting direct photocatalytic degradation tests on a model organic pollutant- such as methylene blue, following established protocols- would help quantify the photocatalytic efficiency of these films. This would allow us to correlate their degradation performance directly with the photo-induced hydrophilicity trends reported in this work [13]. Finally, compositional modification of the sputtered films- for instance doping with SiO_2 or Al_2O_3, which has been shown to push TiO_2 coatings toward full superhydrophilicity (contact angle below 2°) [5] – could be combined with substrate-temperature control to further optimise the self-cleaning performance of sputtered TiO_2 coatings on conductive glass.

Beyond self-cleaning glazing, the dense, transparent, ITO-compatible TiO2 films demonstrated here are structurally similar to the thin compact “blocking layer” that is sputter-deposited beneath the mesoporous photoanode in dyesensitised solar cells (DSSCs) to suppress interfacial charge recombination and improve device efficiency [14]. This points to a further application direction for the present RF-sputtering process: fabricating compact TiO2 layers for coloured, semitransparent DSSC modules used as external building facades. Unlike conventional opaque photovoltaic cladding, semi-transparent, dye-tinted DSSCs can be produced in a range of colours while still transmitting a substantial fraction of incident daylight into the building interior, combining on-site electricity generation with an aesthetically customizable façade and the potential to reduce daytime artificial-lighting demand [15, 16]. Combining such a DSSC-integrated façade with the substrate-temperature-tuned photoinduced hydrophilicity reported in the present study could additionally provide a self-cleaning outer surface, helping to limit soilingrelated efficiency losses over long-term outdoor exposure.

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