OBTAINING AND CHARACTERIZATION OF TiO_2 BY DIP COATING FOR UV-BLOCKING APPLICATIONS

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Abstract

This study reports on the synthesis and characterization of thin films of titanium dioxide (TiO_2) with a focus on their ultraviolet (UV) blocking properties. The thin films were obtained through a sol-gel method, deposited by dip-coating, and subsequently annealed to enhance structural and optical properties. Various precursor solutions, solvents, and deposition speeds were investigated to optimize film quality. The films were analyzed using X-ray diffraction (XRD), Raman spectroscopy, and UV-Vis-NIR spectroscopy. Results showed that the deposited films were well-crystallized, highly transparent in the visible range, and had efficient UV-blocking properties. The results suggest that these thin films are promising candidates for applications in protective coatings and UV filters for high-temperature working materials, combining effective UV shielding with favorable optical transparency in the visible spectrum at high temperatures.

Introduction

Historically, quartz glass envelopes have been doped with cerium to block harmful ultraviolet (UV) radiation. However, this method has significant drawbacks: it lowers the annealing point of the quartz, reducing the lamp's lifespan and efficiency. The cerium dopant also absorbs, rather than reflects, the UV energy, which leads to wasted light and further degradation of the material. A solution to these problems is to use a high-temperature coating that effectively reflects UV emissions while maintaining the transmission of visible light [1].

This study explores a promising alternative: the synthesis and characterisation of thin films of titanium dioxide (TiO_2). These films were produced using a sol-gel method and are designed to provide efficient UV shielding with high transparency in the visible spectrum. The materials were analysed to confirm their well-crystallised structures and excellent UV-blocking properties. The results suggest these thin films are promising candidates for protective coatings and UV filters on high-temperature materials, offering a superior alternative to traditional cerium-doped quartz [2,3].

Experimental

Synthesis of Thin Films

Titanium dioxide, TiO_2, was prepared by the sol-gel method using the hydrolysis of titanium tetra-isopropoxide ($Ti(OCH(CH_3)_2)_4$ – TTiP; 98+%; Thermo Scientific) with deionised water. The solvents used for the synthesis were isopropanol ($(CH_3)_2CH_2OH$ – iPOH; ACS; Carlo Erba Reagents) and absolute anhydrous ethanol (CH_3CH_2OH – EtOH; HPLC Plus;

Carlo Erba Reagents), while acetylacetone ($CH_3COCH_2COCH_3$ – AcAc; >99.0%; TCI Europe N.V.) was used as a stabiliser. Two TiO_2 solutions were prepared, with concentrations of 3% and 10%, respectively. The code of samples represented the deposition rate (s1, s2, and s4), the solvent (iP and Et being isopropanol and ethanol), and the concentration of TiO_2 in the solution (3 and 10 g TiO_2 per 100 ml of colloidal solution).

Table 1. The sample's parameters.

Sample Name	Deposition speed (mm/s)	TiO_2 Concentration (g/100 ml)	Solvent
s1iP3	s1	3	iP (isopropanol)
s2iP3	s2	3	iP (isopropanol)
s2Et3	s2	3	Et (ethanol)
s2iP10	s2	10	iP (isopropanol)
s4iP3	s4	3	iP (isopropanol)
s4Et3	s4	3	Et (ethanol)

Film Deposition and Characterisation

The thin films were deposited onto quartz glass slides using a dip-coating technique, **Solgelway** dip coater (Model ACEdip 2.0). The slides were cleaned, immersed in the precursor solutions, and withdrawn at controlled speeds of 1, 2, or 4 mm/s. After deposition, the samples were dried at 100 °C for 30 minutes, followed by a final annealing step at 800 °C for 15 minutes. The films were then thoroughly characterised. **UV-Vis-NIR spectroscopy** was used to measure their transparency and UV-blocking ability. **X-ray diffraction (XRD)** and **Raman spectroscopy** confirmed their crystalline phases of anatase (TiO_2). The surface structure was examined using **optical microscopy**.

Results and discussion

X-ray diffraction (XRD) analysis confirmed the formation of pure, crystalline phases (fig. 1).

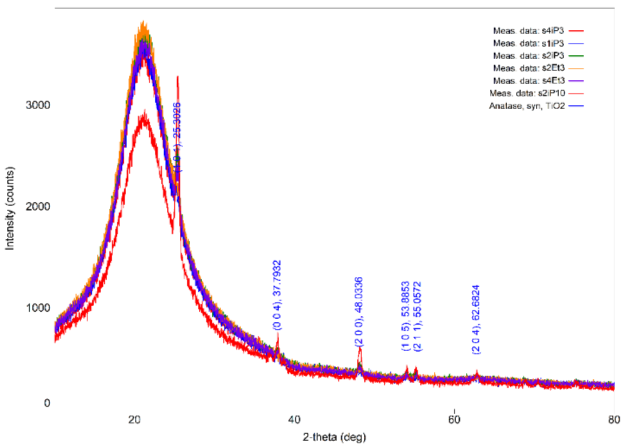

Figure 1. RX spectra for coatings of all TiO_2 samples.

The crystalline phase in the heat-treated TiO_2 films was identified as the **anatase** phase with no impurities detected. The peaks in the XRD patterns for TiO_2 matched those of the anatase structure. **Rietveld refinement** of the XRD data showed that the lattice parameters and crystallite sizes were consistent with those found in the literature, confirming the high structural quality of the films.

The Raman spectra (fig. 2) for the TiO_2 films also confirmed the presence of the anatase phase through characteristic vibration modes at 144, 197, 399, and 513-519 cm^{-1}, indicating high crystallinity.

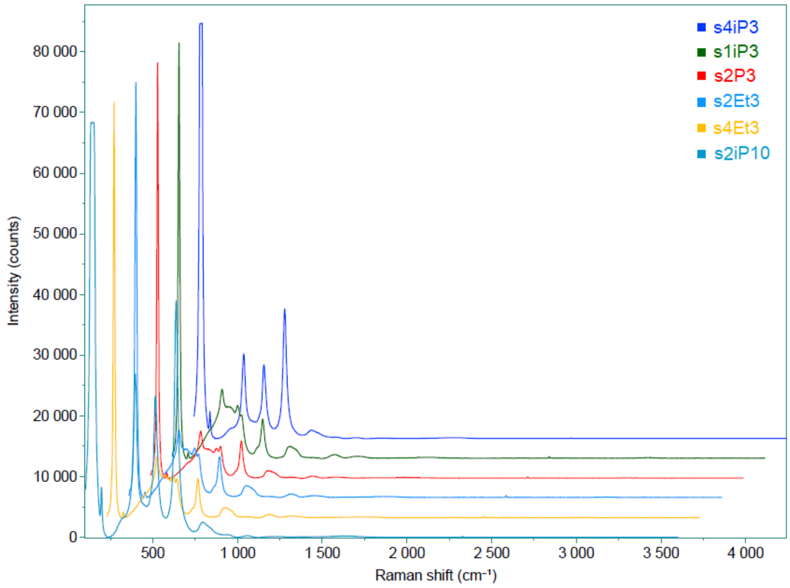

Figure 2. Raman spectra of the quartz glass with TiO_2 thin films.

Fig. 2 shows the Raman spectra of titanium dioxide, which highlight several distinct peaks characteristic of the anatase phase, a crystalline form of TiO_2. These peaks correspond to the vibration modes of the crystal lattice: 144 cm^{-1} (E_g), a strong and sharp peak frequently used as a reference for identifying anatase; 197 cm^{-1} (E_g), of lower intensity; 399 cm^{-1} (B_{1g}), relatively weak; and 513 cm^{-1} and 519 cm^{-1} (B_{1g} and A_{1g}), which usually appear as a single broad peak due to their proximity. The presence of these well-defined peaks confirms the high crystallinity of the analysed samples.

The UV blocking properties depend on the band gap value of TiO_2, which is around 3.2–3.3 eV, corresponding to a wavelength of 387–366 nm. In this range, the energy of UV radiation absorbed by atoms in TiO_2 is high enough to excite electrons from the valence band to the conduction band. The analysis of the spectra in Fig. 3 shows that, in the TiO_2 series, the highest transmittance values in the visible range are obtained for films deposited from ethanolic solutions at a speed of 2 mm/s, but these films also have a relatively high transmittance in the UV range, which makes them less favourable for UV-block applications.

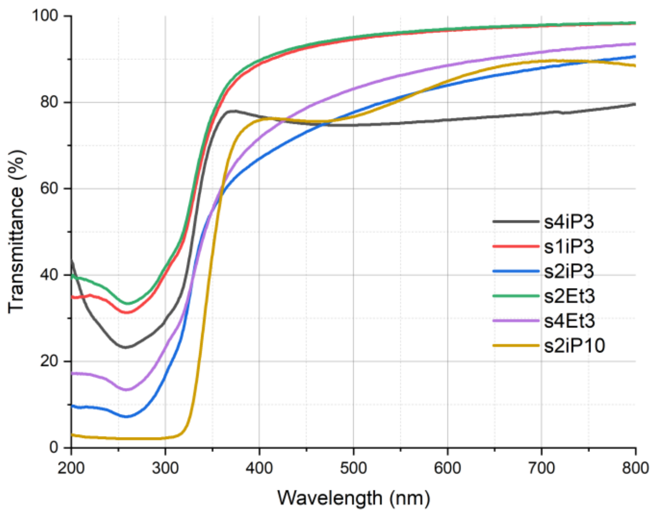

Figure 3. UV-Vis spectra for coatings of all TiO_2 samples.

Fig. 4 shows optical micrographs of thin films, taken at low (20×) magnification, highlighting the influence of deposition parameters on surface morphology and uniformity.

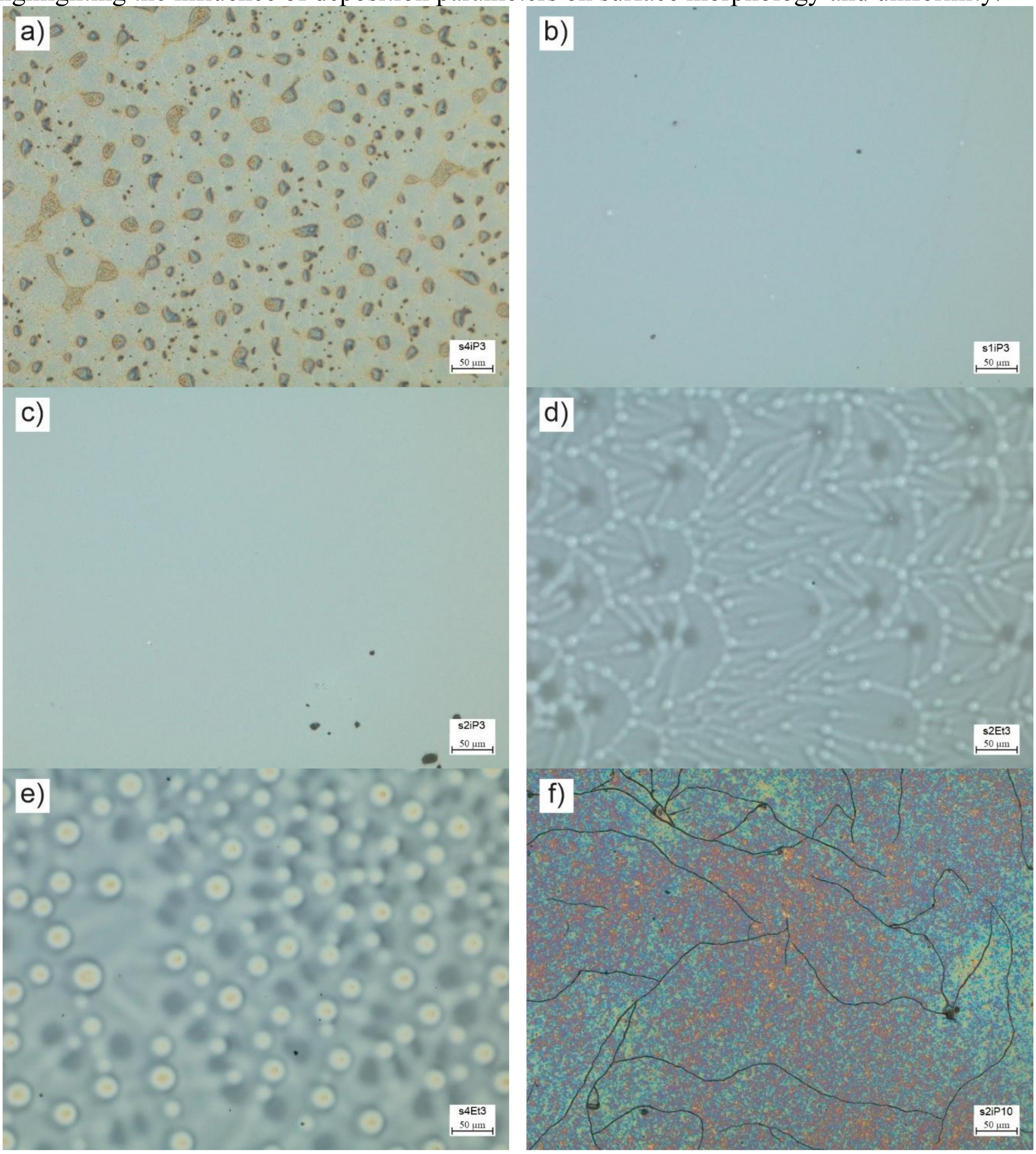

Figure 4. Optical micrographs of the samples

(s4iP3): This image shows a **non-uniform** film with distinct droplet-like structures and agglomerates. This morphology suggests a fast deposition speed or a high concentration of the precursor solution, which led to the formation of separate islands of material rather than a continuous, smooth film.

(s1iP3): This film appears **highly uniform and smooth**, with very few visible defects. This is a sign of an excellent and well-controlled deposition process. This kind of film would be ideal for applications that require optical clarity and a defect-free surface, like anti-reflective coatings.

(s2iP3): Similar to the top-right image, this film is also **very smooth and uniform**. There are some small defects, but overall, the quality is high, indicating optimised deposition parameters that resulted in a homogeneous film.

(s2Et3): This micrograph reveals a unique **"dendritic" or "spider-web" pattern**. This structure is likely the result of a specific deposition parameter, possibly a fast solvent evaporation rate, which caused the TiO_2 particles to self-assemble into this interconnected network. Films with this porous morphology are valuable for applications where a large surface area is beneficial, such as in catalysis or gas sensing.

(s4Et3): This image shows a film with numerous **spherical agglomerates** or "beads." This could be a result of the precursor solution's properties, like surface tension or viscosity, and the interaction with the substrate during the deposition process. This film is less uniform than others.

(s2iP10): This film displays a **complex, porous, and highly interconnected network** with distinct colours. The pattern suggests a rapid, non-equilibrium growth process. The colours are likely due to thin-film interference, which indicates variations in the film's thickness across the sur

face. This type of morphology is often used in applications like solar cells or sensors.

Conclusion

This study successfully demonstrated the synthesis of highly crystalline titanium dioxide (TiO_2) thin films using a simple and effective sol-gel dip-coating method. The annealing process at 800 °C yielded pure **anatase** phase TiO_2 films, as confirmed by both **X-ray diffraction (XRD)** and **Raman spectroscopy**. The high structural quality was further validated by Rietveld refinement, which showed that the films' lattice parameters and crystallite sizes were consistent with published literature.

The films exhibit strong **UV-blocking properties**, which are directly linked to the material's band gap. While all films absorb UV radiation, those deposited from ethanolic solutions at 2 mm/s demonstrated the highest transparency in the visible spectrum. However, this highly visible transparency came with a compromise: relatively high UV transmittance, making these specific films less effective for UV-blocking applications. Overall, the results confirm that these TiO_2 thin films are a promising alternative to traditional UV-filtering methods, offering a combination of high crystallinity, good optical transparency, and effective UV absorption for use in high-temperature protective coatings.

Acknowledgements

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