Pathways to Tailor Photocatalytic Performance of TiO₂ Thin Films Deposited by Reactive Magnetron Sputtering

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1. Materials and Methods

1.1. Thin Film Deposition

For the deposition of the TiO₂ thin films, either electron beam evaporation and pulsed unipolar DC magnetron sputtering from a metallic target in a reactive O₂/Ar atmosphere was applied. The details on the deposition of TiO₂ thin films by evaporation are discussed in our previous work [1] and details on deposition by reactive sputtering from a metallic Ti target are described in our earlier publications [1–3].

1.2. Nanoparticle Deposition

The deposition of nanoparticles was realized by following either a sol-gel approach (in the case of TiO₂, as reported by Suresh et al. [4]) or a gas phase synthesis approach (Al and TiO₂) using unipolar DC magnetron sputtering and an in-house gas aggregation source (GAS). Details on the deposition of Al or TiO₂ nanoparticles from GAS can be found in the work of Ghor et al. and Polonskyi et al. respectively [5,6]. For the deposition of TiO₂ nanoparticles from solution a sol-gel approach, 7.18 mL of titanium (IV) butoxide (5593-70-4, purity > 97.0%, Fluka Chemie AG, Buchs, Switzerland) was given into a glass beaker and 12.0 mL acetic acid (64-19-7, purity > 99.8%, Merck KGaA, Darmstadt, Germany) was added as a solvent and protecting agent for the titania precursor under continuous stirring. After 30 min, 37.5 mL deionized water was added drop wise and under vigorous stirring. The solution was subsequently heated on a hot plate (70 °C for 90 min). Afterwards, the solution was transferred into a screw cap flask and stored under continuous stirring until it was coated onto the sputter-deposited TiO₂ thin film by spin coater (WS-650MZ-23NPPB, Laurell Technologies Corporation, North Wales, PA, USA). Consecutively, a heat treatment step (700 °C, 1 h, LE 4/11/R6, Nabetherm GmbH, Lilienthal, Germany) was performed.

1.3. Characterization

In order to achieve a thorough characterization of the fabricated TiO₂ thin films and nanocomposites, atomic force microscopy (AFM, NanoWizard 3, JPK, Berlin, Germany) was applied to obtain information regarding the surface topography and Raman spectroscopy, as well as X-ray diffraction (XRD, Rigaku, Billerica, MA, USA), were applied to study the presence of crystalline TiO₂ polymorphs. In addition, the morphology of the TiO₂ samples was investigated by scanning electron microscopy (SEM, Zeiss Supra 55VP, New York, NY, USA) in top view and cross-sectional configuration and the photocatalytic, UV induced, degradation of organic dyes (at the example of methylene blue, MB) was studied in order to evaluate the photocatalytic performance. Details on the applied thin film characterization methods can be found in our earlier work [1–3].
References

1. Henkel, B.; Neubert, T.; Zabel, S.; Lamprecht, C.; Selhuber-Unkel, C.; Rätzke, K.; Strunskus, T.; Vergöhl, M.; Faupel, F. Photocatalytic properties of titania thin films prepared by sputtering versus evaporation and aging of induced oxygen vacancy defects. *Appl. Catal. B Environ.* **2016**, *180*, 362–371.

2. Henkel, B.; Vahl, A.; Aktas, O.C.; Strunskus, T.; Faupel, F. Self-organized nanocrack networks: A pathway to enlarge catalytic surface area in sputtered ceramic thin films, showcased for photocatalytic TiO₂. *Nanotechnology* **2018**, *29*, 035703.

3. Vahl, A.; Dittmann, J.; Jetter, J.; Veziroglu, S.; Shree, S.; Ababii, N.; Lupan, O.; Aktas, O.C.; Strunskus, T.; Quandt, E.; et al. The impact of O₂/Ar ratio on morphology and functional properties in reactive sputtering of metal oxide thin films. *Nanotechnology* **2019**, *30*, 235603.

4. Suresh, C.; Biju, V.; Mukundan, P.; Warrier, K. Anatase to rutile transformation in sol-gel titania by modification of precursor. *Polyhedron* **1998**, *17*, 3131–3135.

5. Ghori, M.Z.; Veziroglu, S.; Hinz, A.; Shurtleff, B.B.; Polonskyi, O.; Strunskus, T.; Adam, J.; Faupel, F.; Aktas, O.C. Role of UV Plasmonics in the Photocatalytic Performance of TiO₂ Decorated with Aluminum Nanoparticles. *Acs Appl. Nano Mater.* **2018**, *1*, 3760–3764.

6. Polonskyi, O.; Peter, T.; Mohammad Ahadi, A.; Hinz, A.; Strunskus, T.; Zaporojtchenko, V.; Biederman, H.; Faupel, F. Huge increase in gas phase nanoparticle generation by pulsed direct current sputtering in a reactive gas admixture. *Appl. Phys. Lett.* **2013**, *103*.

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