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Growth and Structure of TiO_2 Thin Films Deposited Inside of Borosilicate Glass Tubes by Spray Pyrolysis

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Since the early work of Fujishima et al. [1] on photoelectrochemical reactions at semiconducting TiO_2 , attention has been focused on this material into the area of environmental photocatalysis, including recently self-cleaning, and antifogging surfaces [2]. Photocatalytic TiO_2 has been used to decompose organic contaminants [3] and/or to sterilize microbial cells [4] on air or water flows. Nevertheless, for the purification and treatment of polluted air and water the use of photo catalytic thin films have some advantages, because the active material are immobilized on the substrate avoiding the separation process needed when powders are used. In addition the thickness, then the amount, of the active material can be optimised in order to use the minimum required to obtain the best activity. In this work, undoped TiO_2 thin films were deposited inside of borosilicate glass tubes, by a very simple and reproducible spray pyrolysis (SP) technique. MIcrostructural characterization of the films were performed by high resolution transmission electron microscopy (HRTEM), selected area electron diffraction (SAED), high angle annular dark field (HAADF), and scanning electron microscopy (SEM). Film thickness was determined by fibre optic reflectance spectrometry in contact mode.

Titanium dioxide thin films were obtained inside of borosilicate glass tubes by very simple SP technique, similar to that reported for the deposit of TiO_2 and ZnO thin films inside of silica fused tubing [5]. The over all dimensions of the borosilicate tubes were external diameter of 25 mm and length of 120 cm. Films were uniform on almost 80% of the tube length, transparent, non-light scattering, and well adhered to the substrate. The spraying system is illustrated in figure 1. The borosilicate glass tube (a) has been coupled to a medical nebulizer (b), which was used as atomizer. This tubing was heated by a three zone cylindrical furnace (c), with a very precise temperature control (±1 K). Deposition temperature was fixed between 500 and 625 K in the different zones of the furnace. The starting solution was a 0.1 M dilution of titanyl acetyl acetonate in absolute ethanol. The process starts from aerosol generation of precursor solution in the nebulizer. This aerosol is subsequently conveyed by the carrier gas, and injected directly into the heated tube inside of the cylindrical furnace. The carrier gas was micro-filtered air; their pressure was kept at 310 kPa and the flux varied between 133 to 233 cm^3 s^{-1}. Table 1 summarizes the principal parameters to deposit the films. SAED analysis shows that titanium oxide films were polycrystalline and their structure corresponds to TiO_2 Anatase phase. HRTEM and HAADF images show that the microstructure of the films was composed of both irregular and round shaped closed packed grains, of around 100 to 300 nm of characteristic length. Cross section of the films were analyzed by scanning electron microscopy and HAADF, it is shown that columnar growth of crystallites takes place.

References

[1] A. Fujishima, K. Honda, Nature 238 (1972) 37.
[2] A. Fujishima, T. N. Rao, D. A. Tryk, J. Photochemistry and Photobiology C, 1 (2000) 1.
[3] Y. Paz, A. Heller; J. Mater. Res. 12 (1997) 2759 and references there in.
[4] W. A. Jacoby, P. Ching Maness, E. J. Wolfrum, D. M. Blake, J. A. Fennell, Environ. Sci. Technol. 32 (17), (1998) 2650
[5] M. Miki-Yoshida et al., Thin Solid Films 419 (2002) 60.

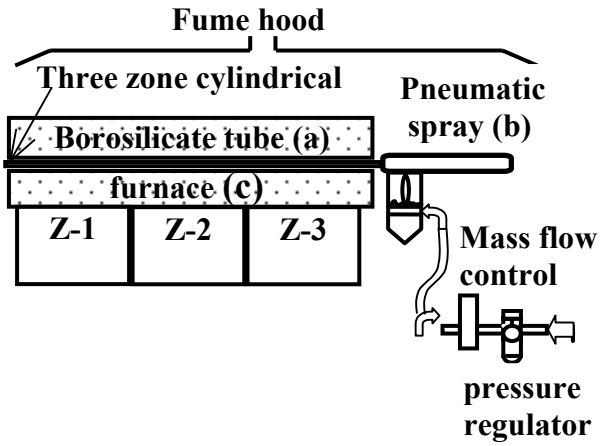

Figure 1. Schematic diagram of the spray pyrolytic system. See text for details.

Sample	415	438	427	431
T_{z-1} [K]	623	623	623	623
T_{z-2} [K]	544	542	551	548
T_{z-3} [K]	505	506	514	510
Air flux [cm^3 s^{-1}]	133	167	200	233
t [min]	10	8	8	8
Thickness [nm]	168	137	153	162
Deposition rate [nm s^{-1}]	0.28	0.29	0.32	0.34

Table 1. Deposition parameters of TiO_2 films. T_{z-N}: Furnace temperature in zone N. Air flux . t: deposition time. Average thickness and average deposition rate.

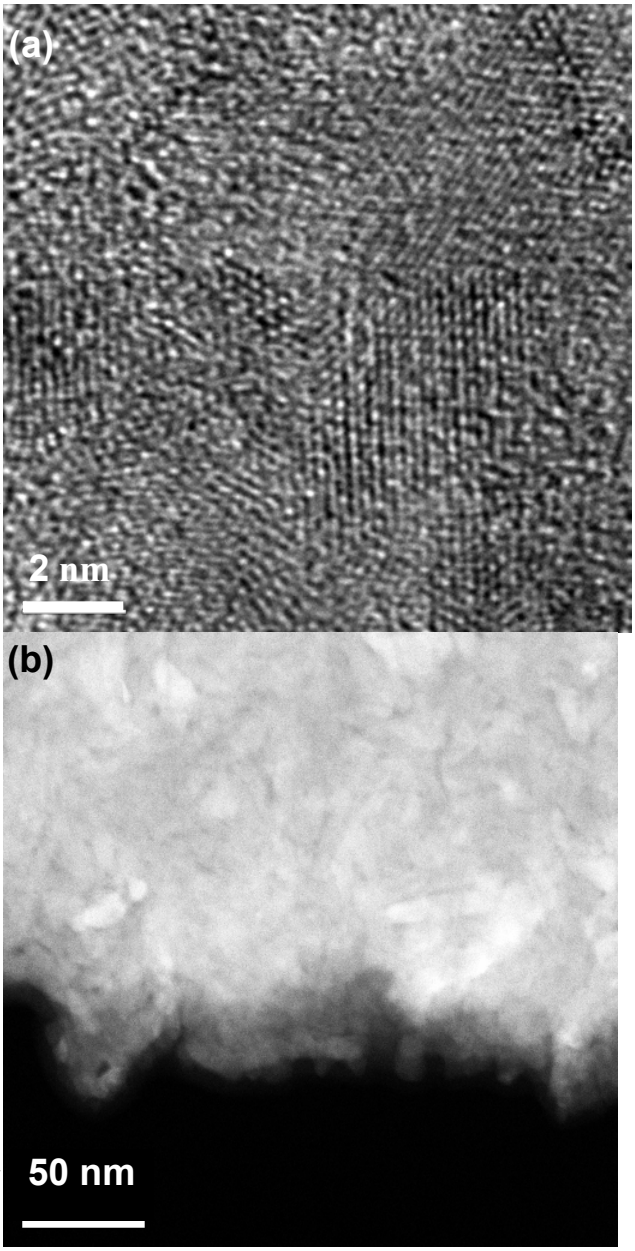

Figure 2. Typical images of TiO_2 films deposited inside of borosilicate tubes. a) HRTEM and b) HAADF.

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