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LETTERS TO THE EDITOR

Sol-Gel Synthesis of TiO_2 Mesoporous Thin Films on Silicon

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Abstract—Conditions were determined for the synthesis of TiO_2 mesoporous thin films on silicon by the sol-gel method in the version of coating by dipping silicon plates in a TiO_2 sol (dip-coating).

Keywords: nanolayer, nanoparticle, titanium dioxide, mesoporous nanofilms, sol-gel method

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Film titanium dioxide has found application in many advanced fields, like photovoltaics, sorption technology, photocatalysis, biomedicine, and sensor analyzers [1–3]. The reason of it, first and foremost, is a good combination of physical and chemical properties of the material. Currently nanostructured materials based on TiO_2, nanolayers and nanoparticles, are intensely investigated [1–3].

We have determined conditions for the synthesis of TiO_2 mesoporous nanofilms with developed surface on silicon by its dip coating from a TiO_2 sol. In the synthesis of TiO_2 sols tetraisopropyl titanate, polyethylene glycol (*M* 3000 and 4000, mole fraction 0.003–0.05), diethanolamine, HNO_3, and isopropyl alcohol are usually used [4–6].

The synthesis was carried out at the molar ratio $(i\text{-PrO})_4\text{Ti–PEG–}(\text{NOSN}_2\text{CH}_2)_2\text{NH–}H_2O\text{–}HNO_3\text{–}i\text{-PrOH}$ 1 : *Y* : 0.5 : 150 : 1.5 : 18 [4–6]. The surface area of pores was regulated by changing the PEG mole fraction (*Y*) and its molecular weight. Specific surface area and diameter of pores were measured by the method of nitrogen capillary adsorption (see table).

It was found by the scanning electron microscopy method that the size of TiO_2 particle in the sol is 7±2 nm. Films of TiO_2 on silicon obtained from the sol by dip coating were calcinated in air at 500°C for 2 h with the aim of removing PEG from the pores. Film thickness measured by the method of scanning electron microscopy of the film edge was 200 nm with a surface roughness of ±20 nm.

According to the X-ray photoelectron spectroscopy data, the resulting TiO_2 films are homogeneous and cover the entire surface of the silicon substrate. The study of the phase composition of silicon samples coated by TiO_2 films showed the presence of a crystalline phase (anatase).

It follows from the above data that it is possible to regulate the specific surface area of pores in a TiO_2 mesoporous nanofilm by variation of the PEG mole fraction and molecular weight. The use of PEG 4000 makes it possible to reach the pore specific surface area of 248 m^2/g.

Structural characteristics of the TiO_2 mesoporous film on silicon were investigated by the methods of

Dependences of pore diameter and surface area of the TiO_2 mesoporous film on the PEG molecular weight and mole fraction

M	Parameter	PEG mole fraction in a sol				
		0.003	0.01	0.02	0.03	0.05
3000	Diameter of pores, nm	3.7±0.7	3.7±0.9	3.7±0.8	3.7±1.0	3.7±0.8
	Specific surface area, m^2/g	192	200	180	130	110
4000	Diameter of pores, nm	3.8±0.7	3.8±0.9	3.8±1.1	3.8±0.8	3.8±0.7
	Specific surface area, m^2/g	185	248	236	217	178

scanning electron microscopy and X-ray phase analysis using a Zeiss Merlin scanning electron microscope and a Xcalibur Agilent Technologies (Oxford Diffraction) single crystal diffractometer, respectively. Specific surface areas of samples were measured on an ASAP 2020 adsorbometer. Chemical composition and homogeneity of the films were determined by the X-ray photoelectron spectroscopy method on a Thermo Fisher Scientific Escalab 250Xi Auger electronic spectrometer.

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