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SPECTROSCOPIC INVESTIGATIONS ON SELF-CLEANING FILMS FOR PHOTOVOLTAIC GLASSES

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ABSTRACT

Titanium dioxide thin coatings are widely used in different fields for the manufacture of various products: electrochromic displays, photocatalytic systems, photosensitized solar cells and many others. At the same time, the possibilities for obtaining protective coatings based on TiO_2 are of interest. The chemical and physical characteristics of the TiO_2 coatings depend on a large extent of the applied technological method and the temperature values of the heat treatment used. Coatings are prepared by sol gel method applying dip coating technique. The present study characterizes self-cleaning TiO_2 thin films for the photovoltaic application. The studies were carried out at different ratios of the components involved, rate of application of the solution on the glass substrate and different curing and holding temperatures. The coatings are dense and nanocrystal line according to AFM studies and optically transparent according to UV-Vis-NIR spectra.

Keywords: self-cleaning, photovoltaic, TiO_2 coatings, UV-Vis-NIR spectra, AFM.

INTRODUCTION

The active role of photovoltaic systems represents an essential aspect of the strategy for sustainable energy development and the priorities for limiting the use of conventional energy carriers and the application of renewable energy sources [1 - 5]. The efficient operation of photovoltaic systems is determined by several factors: positioning of the solar modules, meteorological and climatic conditions, contamination of the working surface of the solar panels, etc [1, 2]. Predominant surface contaminants are various fractions of dust particles [6, 7], different local surface deposits, products of the life cycle of various organisms and others. Existing automated and robotic cleaning systems [1, 7] require additional investments for high-tech equipment subject to periodic maintenance and repair. Manual cleaning systems are primarily applicable to relatively small solar systems. The development of long-lasting self-cleaning coatings [8 - 14] with high weather resistance and other functional characteristics [15 - 17] (hydrophobic, hydrophilic, antireflective, anti-dust, anti-frost, anti-fogging and others) is considered as a promising and cost-effective approach to maintaining solar panels in a highly efficient state.

Various TiO_2 nanostructured, self-cleaning [8 - 10, 13], antireflective [11, 14] coatings have been developed for photovoltaic installations, solar cells, solar concentrators [10] and other equipment [13]. The role of the obtained nanoscale structure for determining the operational characteristics of the layers was analysed [11]. Experimental tests have been performed for the presence of self-cleaning properties in doped TiO_2-coatings deposited on glassy substrates [8]. By applying the sol-gel method [18] and atmospheric pressure chemical vapor deposition (APCVD) [19, 20], TiO_2 anti-reflective coatings [18 - 20] were obtained, providing an increase in the productivity of monocrystalline [18, 19] and polycrystalline [20] silicon solar cells.

The efficiency and technological possibilities of deposition of double-layer self-cleaning [21] TiO_2/SiO_2 and ZrO_2/SiO_2 antireflective [22] thin films applicable for photovoltaic modules are investigated. Experimental self-cleaning [23], the reflective two-layer [23, 24] (SiO_2/TiO_2 and SiO_2-TiO_2/SiO_2-TiO_2) and three-layer [25, 26] (SiO_2/SiO_2-TiO_2/TiO_2 and $SiO_2/TiO_2/SiO_2$-TiO_2), coatings for solar cells and other applications in solar energy were obtained by the sol-gel method [24 - 26]. The anti-reflective properties of developed multilayer coatings with a complex "stacks" structure (TiO_2-$SiO_2/SiO_2/SiN_x$) applicable to polycrystalline silicon solar cells were investigated [27].

By applying magnetron sputtering, experimental three-layer thin films (with TiO_2/SiO_2/Ag structure) with self-cleaning properties and high reflectivity applicable for solar frontal reflectors have been obtained [28].

Super hydrophobic self-cleaning composite coatings obtained in the binary oxide system reduced graphene oxide-titanium oxide (rGO-TiO_2) were prepared [29]. Double-sided photovoltaic panels with self-cleaning coatings (based on graphene) and presence of anti-dust characteristics have been developed. Nanoscale antireflective coatings with the participation of Ta_2O_5 and TiO_2 suitable for monocrystalline [30] and polycrystalline [31] silicon solar cells have been obtained. Nanoscale anti-reflective coatings with WO_3-TiO_2 composition and photocatalytic properties, potentially applicable for sputtering high-efficiency thin films for silicon photovoltaic modules, have been investigated [32]. Experimental anti-reflective TiO_2/Ni coatings have been developed for photovoltaic cells [33]. The possibilities of increasing the performance of silicon solar cells by applying antireflective, surface coatings of the type Al_2O_3/In and NPs/TiO_2 have been analysed [34].

In the present work, the experimental results of the performed laboratory studies of self-cleaning TiO_2 thin films, deposited by the sol-gel method on glass plates are presented. Obtaining self-cleaning coatings for photovoltaic modules is considered as a major possibility for potential application of the development. The structure, micromorphology and optical properties of the obtained experimental samples were investigated by applying AFM and Ultraviolet Visible Near Infrared Spectroscopy (UV-Vis-NIR) methods.

EXPERIMENTAL

Materials

All compounds were supplied as analytical grades and were used without further modification. Thin film coatings are performed on glass slides from AGC Glass Europe. TiO_2 precursors and solvents were 99 % titanium butoxide - $Ti(OBu)_4$ and 99.9 % ethanol (C_2H_5OH), all purchased from Alfa Aesar Germany. The hydrolysing agent is distilled water, and the stabilizer is nitric acid /HNO_3 /. Preparation of the glass substrate. Before being cleaned, the slides were cut to size (20 mm x 70 mm x 2 mm). Substrate cleaning is important prior to the deposition process, as this allows the cleaning and removal of submicron particles from the substrate surface. This is decisive for the quality of the bonding film on the substrate. The glass substrates were cleaned/ washed/ in 99.9 % ethyl alcohol for 15 min and acetone for 15 min and then dried for 30 min at 100^{o}C. Cleaning the glass substrates improves the adhesion of the coating material to the substrate and prevents contamination on the formed coating.

Preparation of TiO_2 sol. The aim of the synthesis is to obtain coatings of TiO_2 on glass substrates applying sol-gel method. Mixture of precursor - 5 mL of 99 % $Ti(OBu)_4$ and 5 mL of DEA /$C_4H_{11}NO_2$/ - was added dropwise to the acid solution, made of ethanol, water and nitric acid in a ratio of 1:1:0.5 for prevent gel formation. To ensure adequate hydrolysis, the mixture was continuously stirred at 500 rpm with a magnetic stirrer for 2 hours under air pressure to produce a pink translucent solution. Schematics of the process of obtaining a thin film of sol-gel with TiO_2. Depiction of the chemical synthesis of TiO_2-based sol. A dip coating machine (Dip Coating technology) at a speed of 42.8 mm/min was used to deposit thin layers of TiO_2 on the substrates. A lower speed of the applied solution on the glass substrate helps the coating to be uniform, while a higher rate helps the solution to become thinner. The deposited films were then heated at a temperature of 400^{0}C for 2 hours /schema 1/.

Research related equipment

Characterization methods

Atomic force microscopy (AFM)

By measuring the surface of the materials at the nano level by atomic force microscopy (AFM), the roughness of the investigated sample is quantified. AFM imaging was performed on the NanoScope V system (Bruker Ltd, Germany) operating in tapping mode in air at a room temperature. Silicon cantilevers (Tap 300Al-G, Budget Sensors, Innovative Solutions Ltd., Bulgaria) with 30 nm thick aluminium reflex coatings were used. According to the producer's datasheet, the cantilever force constant and the resonance frequency are in the range of 40 N m^{-1} and 300 kHz, respectively. The tip radius was less than 10 nm. The scan rate was set at 1 Hz and the images were captured in height mode with 512 × 512 pixels in JEPG. Subsequently, all images were flattened using NanoScope software. The same software was also used for section and roughness analysis.

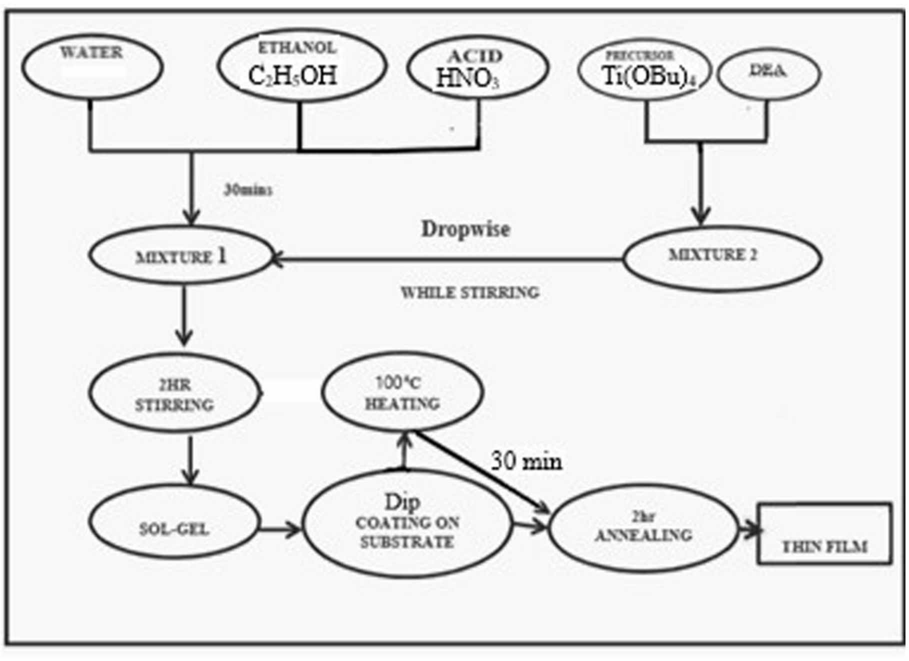
The optical transmittance spectra of the obtained films in the wavelength range of 200 nm to 800 nm were measured at room temperature at normal light incidence using a UV-Vis-NIR spectrophotometer (Cary 5E, Varian, Sydney, Australia).

RESULTS AND DISCUSSION

Analysis of transmittance and absorption spectra

Fig. 1 show the transmittance spectra of the films. As it is shown, all films are transparent in visible region, although their transparency degree and region are slightly different. Focusing on the transmittance spectra reveals that the films have higher transparency, so that all 5 samples of different treatment, thickness are highly transparent. The transparency of reference glass is 90.9 % and the transparency of all films is in the range of 76.03 % - 65.29 % at 415nm, indicating the successful synthesis of highly transparent thin films.

The decrease in the transmittance of the films GLS3



Scheme 1. Schematics of the process of obtaining a thin film of sol gel with TiO_2.

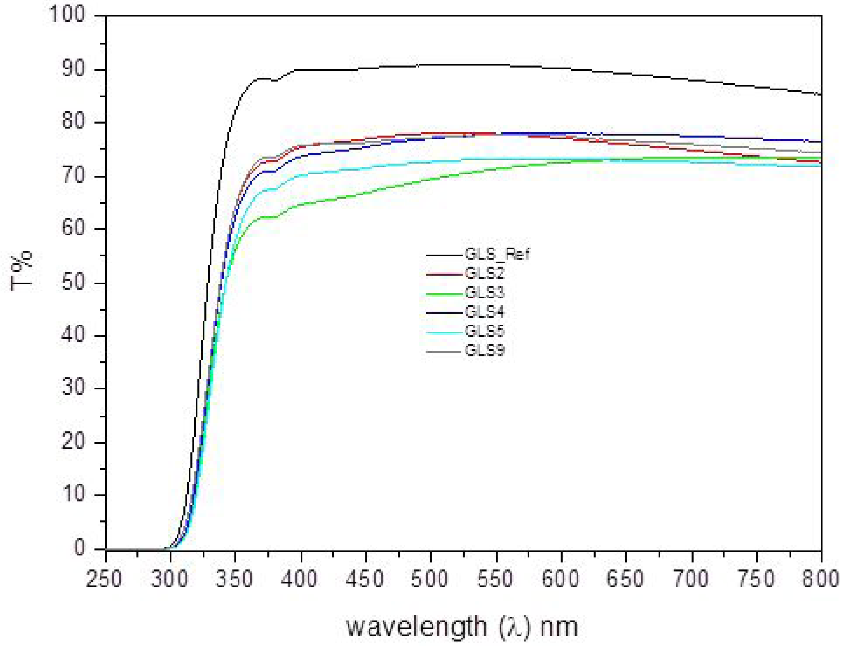


Fig. 1. Transmittance spectra of the films.

and GLS5 is structurally distinct and can be associated with the increase in particle size since bigger particles scatter the light more [35]. Apart from the changes in transparency degree of the prepared glass slides, one more point can be extracted from the transmittance spectra (isosbestic point), and it is maximum wavelength of transparency of the coatings: for GLS2 and GLS9 the isosbestic point at 416nm; for GLS2, GLS4 and GLS9 at 552nm; for GLS3 and GLS5 at 631nm and relative to this point excluding GLS2 and GLS9, the transmission region of the films is shifted to the longer wavelengths. This red shift of the transmittance spectra and absorption edge can be attributed to TiO_2 crystal growth [36], confirmed by XPS and AFM measurements. Parallel investigation of transmittance and absorption spectra (Fig. 2) illustrates that along with the increase in absorption of the films, transparency decreases. There is a dramatic rise in the absorption below 370 nm which is related to the intrinsic band gap absorption [37]. According to the highest transparency wavelength of the films, that are highly transparent in the wavelength ranges of 370–500 nm is preferred because they transmit visible light which is the most intense radiation reaching the earth's surface [38].

The topography and surface roughness of glass samples with different coatings GLS-2, GLS-3, GLS-4, GLS-5 and GLS-9 were investigated using atomic force microscope. The surface for the samples from the GLS series was measured with a surface scan area 15 μm × 15 μm and presented in Fig. 3. The roughness values R_a and R_q were estimated from the AFM images of the coatings- TiO_2 deposited coatings with different percentages in titanium tetrabutoxide solution GLS2 and GLS3, as well as TiO_2 deposited coatings with different percentages in titanium isopropoxide solution GLS4 and GLS5. The roughness values R_a and R_q were presented in Table 1.

The roughness analysis gives the value R_a which is the arithmetic mean of the absolute values Z_i of the surface height deviations measured from the mean plane formula 1 i.e.

$$R_a = \frac{1}{N}\sum_{i=1}^{N}|Z_i| \qquad (1)$$

while R_q is the root-mean-square value of the height deviations taken from the plane of the average images date formula 2 [39].

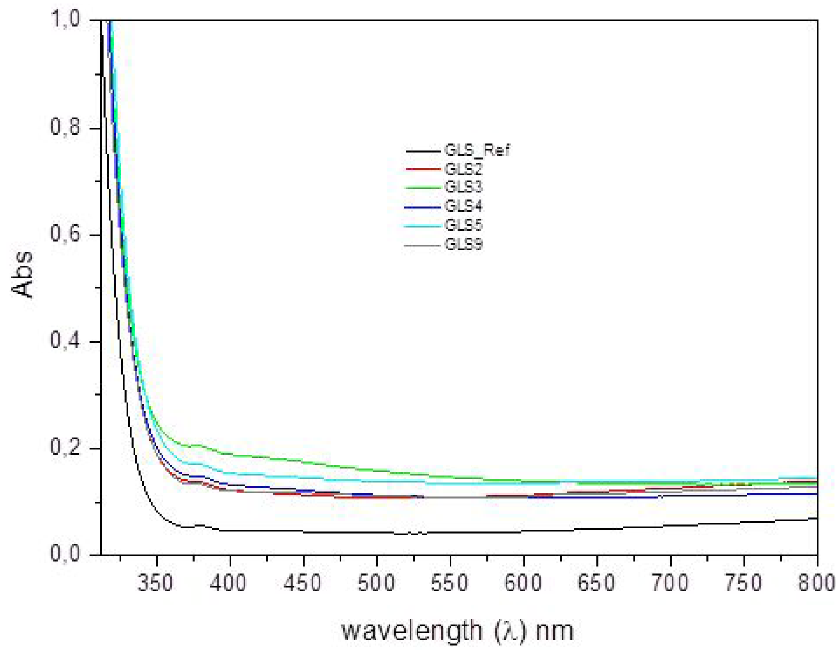


Fig. 2. Absorption spectra.

Table 1. Table for samples.

Samples	Main composition	Drying time	T°C Treatment	Number layers
GlS-2	$Ti(OBu)_4$	30 min	400°C-	2
GlS-3	$Ti(OBu)_4$	30 min	400°C-	3
GlS-4	$Ti(OBu)_4$	30 min	500°C-	1
GlS-5	$Ti(OBu)_4$	30 min	500°C-	3
GlS-9	$Ti(OBu)_4$	30 min	520°C-	3

$$R_q = \sqrt{\frac{1}{N}\sum_{i=1}^{N} Z_i^2} \quad (2)$$

The morphology of the coatings GLS-2 and GLS-3 with applied organic precursor of titanium tetrabutoxide is with a smooth surface with the presence of spherical structures with dimensions in the range of 50 nm to 100 nm, depending on the percentage content of the precursor. The roughness R_q values for the GLS-2 and GLS-3 coatings are 5.51 nm and 14 nm, respectively (Fig. 3 and Table 2).

Analogy, the morphology of the coatings GLS-4 and GLS-5 with applied organic precursor of titanium isopropoxide is with a smooth surface. Those coatings have clusters of the spherical structures with diameter in the range of 1.0 µm to 2 µm, depending on the percentage content of the precursor. The roughness R_q values for the GLS-4 and GLS-5 coatings are 11.7 nm and 38.7 nm, respectively (Figure 3 and Table 2).

The roughness values R_q and R_a for coatings GLS-4 and GLS-5 are higher compared to those for coatings GLS-2 and GLS-3, which is due to the precursor used as well as the processing temperature.

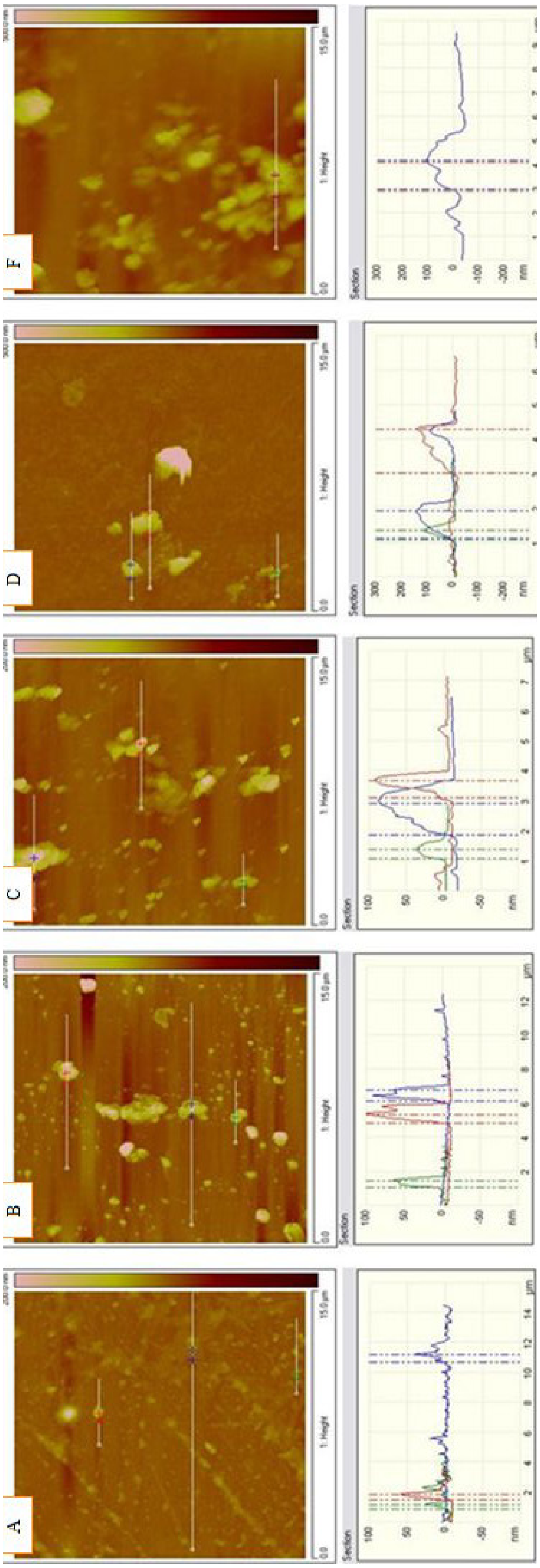


Fig. 3. 2D AFM images and section analysis of the coatings glass samples: (a) GLS-2, (b) GLS-3, (c) GLS-4, (d) GLS-5 and (f) GLS-9.

Table 2. Roughness values for R_a and Rq of coatings glass samples: GLS-2, GLS-3, GLS-4, GLS-5 and GLS-9.

Sample	Roughness R_q, nm	Roughness R_a, nm
GLS 2	5.51	3.35
GLS 3	14.0	6.74
GLS 4	11.7	6.16
GLS 5	38.7	11.4
GLS 9	27.8	18.1

In addition, contact angle measurements of wetting coatings on glass substrates and ellipsometry were performed [40]. Experimental vitreous samples with the presence of self-cleaning coatings applied by the sol-gel method were tested in a real environment under cyclic atmospheric conditions in different TPPs / TPP-Sofia and TPP-Republika, Pernik. Contact angle measurements show that the GLS9 coating is superhydrophilic with a contact angle of 8.3 degrees /8.3°/. Electrons in the valence band of TiO_2 are excited to the conduction band under UV irradiation conditions, leading to superhydrophilic behaviour [41].

CONCLUSIONS

The self-cleaning coatings are prepared by the sol-gel method, applying the dip coating technique. The present study characterizes self-cleaning TiO_2 thin films for photovoltaic application.

- The tests were carried out at different ratios of the components involved, speed of application of the solution on the glass substrate and different curing and holding temperatures
- The experimental results /together with our previous research/ represent a prerequisite for the development of a series of additional compositions and a detailed technological regime for obtaining various modifications of resistant, long-lasting self-cleaning coatings, potentially applicable to photovoltaic panels.

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