Preparation, phase and optical characterization of Sm_2O_3-ZrO_2 coatings on glasses obtained by sol-gel technology using the Dip Coating method

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Abstract: *Obtained experimental self-cleaning coatings (based on compositions with the participation of Sm_2O_3), applied by the sol-gel method on glass slides. This study attempts to improve the overall efficiency of a photovoltaic solar panel by using a Sm_2O_3 doped ZrO_2 (SDZ)-based coating. The optical characterization and phase composition of the obtained experimental samples were investigated using UV-VIS-NIR, XRD and XRF methods. The coatings are nanocrystalline according to XRD and XRF analyzes and show transmittance close to that of pure glass when tested with a UV–VIS–NIR spectrophotometer The experimental results 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.*

Keywords: SOL GEL, XRD, XRF, UV–VIS–NIR, DIP COATING, PHOTOVOLTAICS, ZrO_2, Sm_2O_3

1. Introduction

Zirconium oxide is one of the most well studied transition-metal oxides with promising applications in the development of innovative materials, products with high operational reliability and components for high-tech devices [1-7].

Zirconium oxide thin films are characterized by the presence of a significant variety of functional properties [8-27]: high hardness, excellent mechanical properties, high melting point, corrosion resistance, low electrical conductivity, high dielectric constant, high refractive index, high transparency in the visible and near-infrared regions, large optical band gap, high laser-induced damage threshold, low optical loss, good thermal properties, chemical inertness, biocompatibility, good thermodynamic stability in contact with silicon, etc.

Due to their characteristics, various thin films obtained on the basis of ZrO_2 find application as wear resistance coatings, corrosion protective layers, thermal barriers [28], as well as in the production of various electronic components, biocompatible implants and a number of other products.

At the same time, due to their unique optical properties, thin films of ZrO_2 have significant potential for use in the field of optics and opto-electronics [15,19,29-31] for the development of broadband interference filters, selective reflection coatings, active electro-optical devices, including light emitting diodes, oxygen sensors, high power laser, passive and active waveguides, buffer layers in micro-electronic devices, scintillators, etc.

The operational properties of thin films are determined by their phase composition and structure, which depend on the components used, the presence of modifiers and the applied technological methods for coating deposition.

There are many different techniques for deposition thin films [3,15-19,29-38], such as chemical vapor deposition (CVD), physical vapour deposition (PVD), electrochemical deposition, hydrothermal processing, electron beam evaporation, magnetron sputtering, spray pyrolysis, plasma spraying, pulsed laser deposition, atomic layer deposition, liquid phase deposition method, ion-beam aid deposition and sol-gel method, etc.

Among them the sol-gel method [3,16,32-34,37] provides a number of significant technological advantages such as relatively low processing temperatures, high optical quality of the obtained coatings, capability of coating large surface areas, simplicity and comparative low cost, etc. In order to regulate the solvent evaporation rate, the formation of various cracks and crystal defects and to suppress of the process of the grains growth and unfavorable aggregation, appropriate low-volatility organic compounds are introduced to the used sol-gel precursors: polyvinyl alcohol, polyethylene glycol, hydroxypropyl cellulose, polyvinyl-pyrrolidone [19]. In addition, this technique is suitable to preparation almost any homogeneous and uniform single- or multi-component oxide coatings (ZrO_2, SiO_2, etc.) prepared by spin and dip coating method [31].

The ion Zr^{4+} does not exhibit pronounced photoluminescence properties, but this can be significantly affected by the formed local defects [20]. In most cases they represent oxygen vacancies, various types of impurities, free radicals, etc., which favor broad band PL emission [20].

The sol-gel deposition method is known to provokes the formation of large number of surface defects (for example-oxygen / Zr vacancies or interstitials) in the thin ZrO_2 coatings, which improves their photoluminescence characteristics [19].

Another technological method to activate the PL emission is incorporation of appropriate luminescent agents (including lanthanide ions and others) into the host lattice of ZrO_2 [20]. The existing low-phonon energy of ZrO_2 creates the possibility for the implementation of more efficient luminescence from the incorporated activator ions. The considered effect is determined by the fact that the lower the phonon energy of the specific host, the higher the probability of performing a radiative transitions of the introduced rare earth 3+ ions [20].

In this aspect, a current technological interest is the study of the characteristics of modified Sm-doped ZrO_2 experimental samples and the possibilities for obtaining thin films of Sm_2O_3.

Samarium-doped zirconia samples were prepared using two different technological approaches. By applying atomic layer deposition, thin crystalline films (doped by using ion implantation) with a predominantly monoclinic structure were prepared. While by skull-melting method, bulk polycrystalline experimental samples with presence of monoclinic and tetragonal phases of ZrO_2 were obtained [35]. Photoluminescence measurements of Sm emission in the prepared samples were performed by using pulsed laser excitation at 405, 320 and 230 nm. The obtained spectra may be interpreted as superpositions of two different sets of lines attributable to the presence of Sm^{3+} centers in different phases of ZrO_2. The research of the fine structure in the observed luminescence spectra of Sm^{3+} ions in the experimental sample showed the existence of at minimum two different luminescence centers related to monoclinic and tetragonal phases of ZrO_2 [35]. Excitation at 405 nm provokes the direct excitation of Sm^{3+}, whereas the excitation at 230 nm is interpreted as host-sensitized excitation.

A samarium doped polycrystalline ZrO_2 experimental sample was studied for its phase composition and optical characteristics. From micro-Raman measurements performed the presence of tetragonal as well as monoclinic phase was confirmed [36]. The obtained emission spectrum of Sm^{3+} suggest a significant changes under excitation near the spectral region of fundamental absorption. Combined excitation-emission spectroscopy measurements shows three different sites for the used dopant ions to be present in the experimental sample [36].

Innovative thin films of zirconia oxide ZrO_2 and samarium oxide Sm_2O_3 on glass substrates have been obtained by applying sol-gel technology and using dip coating method [37]. Excitation and emission spectra of ZrO2 films consists of broad bands with maximum values of 318 and 365 nm. The photoluminescence measurements performed of the deposited Sm_2O_3 thin films reveals that can be efficiently excited with 700 nm and consist of emission bands centered at 567, 603, 639 and 707 nm, consistent with characteristic Sm^{3+} transitions [37]. The established values of the chromaticity coordinate points of the prepared Sm_2O_3 and ZrO_2 thin films are located in the orange and blue region. The obtained Sm_2O_3 thin films is characterized by a high color purity of 96% [37].

Samarium oxide Sm_2O_3 thin coatings were deposited on silicon substrates by using pulsed laser deposition method at a temperature range of 25, 200, 400 and 680°C [38]. The performed XRD analysis shows that the structure of Sm_2O_3 coating obtained at 25°C is amorphous, while the coating prepared at 200°C is a partially crystallized. Monoclinic structure was the established predominant phase for Sm_2O_3 coatings obtained at temperatures of 400°C and 680°C [38]. The polycrystalline Sm_2O_3 coating prepared at 680°C is very dense, while coatings obtained at lower temperatures (25, 200 and 400°C) have higher porosity. The established values of index of refraction n and extinction coefficient k (at λ=633 nm) are 1.867 and 0.0660 for the coating prepared at 680°C and are in a range of 1.5~1.6 and 0.01~0.04 respectively for films deposited at lower temperatures [38].

2. Experimental procedures

All of the chemicals were utilized exactly as they were supplied, in analytical grades. AGC Glass Europe chemicals were used for thin film coatings. Alfa Aesar Germany supplied the 99.9% ethanol C2H5OH), ZrOCl2.8H2O, and Sm2O3. Nitric acid (HNO3) is used as a stabilizer. Getting the glass substrate ready. The slides were trimmed to size (20 mm × 70 mm x 2 mm) before to cleaning. In order to clean and remove submicron particles from the substrate surface, substrate cleaning is crucial before the deposition process. The quality of the sticky film on the base is determined by this. After being cleaned or washed for 15 minutes in 99.9% ethyl alcohol and 15 minutes in acetone, the glass substrates were dried at 60°C for 10 minutes. Cleaning the glass substrates helps the coating material stick to the substrate better and keeps the coating from being contaminated. The production technology is presented in the scheme. They used sol-gel technology. After dissolving ZrOCl2.8H2O in H2O and pure alcohol, a 0.01–0.02 molar solution was produced. Sm2O3 was added 30 minutes later. It was vigorously stirred while Sm2O3 was added gradually. The ratio was 6.5 weight percent Sm2O3 to 93.5 weight percent ZrO2. Nitric acid was added or agitated during the hydrolysis process until the solution turned clear, meaning it had been fully dissolved. The glass sample was coated with the resultant solution using the Dip Coating technique. The substrates were immersed in the prepared solutions for 30 s and then pulled out at a speed of 0.8 mm.s-1 and dried to evaporate the organic solvents - the glass substrates were dried at 120°C for 30 minutesThe coating procedure was repeated three times. After that, the sample was treated at 420^0C.

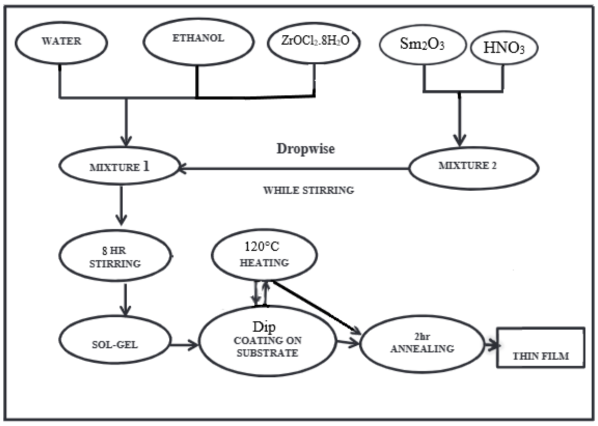
3. Results and Discussion

The samples were examined using a Philips X-ray diffractometer PW 1030, with an X-ray source of Cu Kα radiation (λ = 1.5406 Å) and θ−2θ Bragg-Brentano geometry. The scan step size was 0.025°, 10 s in the range 25–65° 2θ.The diffractograms were analysed using the PDF-2 2022 database.

The thickness of the layer and its elemental composition were measured using energy-dispersive X-ray fluorescence spectrometry (EDXRF) with an EPSILON 1 spectrometer (PANalytical, Malvern, UK).The Omnian and Stratos software modules were used for quantitative and layer thickness analysis respectively.

The size of the crystallites is calculated according to the Scherrer formula:

Schematics of the process of obtaining a thin film of sol gel with ZrO_2 and Sm_2O_3



$$D = 0.9\lambda/B.\cos\theta$$

D - average crystallite size /Average Crystallite size/

λ - wavelength (in angstroms) - 1.54178 Å /X-Ray wavelength/

θ- Bragg angle (degrees) - 2θ/2 /Bragg angle/

B - line broadening FWMH (2θ), /Line broadening/ must be of dimension radians (rad=2θ*π/180)

Figures 1-3 present the results of XRD and XRF analyses of the surface of the glass samples. The SDZ3 /Figure 1/ layer is thinner and amorphous. X-ray diffraction (XRD) analysis confirmed the presence of zirconium and samarium oxides. The distinct peak at 31.91° 2θ in the diffractogram of sample SDZ5 /Figure 3/corresponds to the (120) plane of orthorhombic ZrO_2 [ref. code: 00-049-1746]. A broad, lower-intensity peak at 31.12° 2θ suggests the crystallization of an additional phase on the surface. Weaker reflections at 34.92°, 50.21°, and 59.67° 2θ correspond to the (111), (200), (220), and (311) planes of cubic ZrO_2 [ref. code: 00-089-9069]. The broadening of the main peak at 31.12° 2θ (111) indicates the fine-grained nature of the coating, but it could also be related to the formation of monoclinic Sm_2O_3, whose peaks overlap with those of ZrO_2. The diffractogram of SDZ4 /Figure 2/ shows the formation of a cubic ZrO_2 layer, with samarium possibly present either as a dopant in the cubic zirconium oxide lattice or as monoclinic Sm_2O_3.

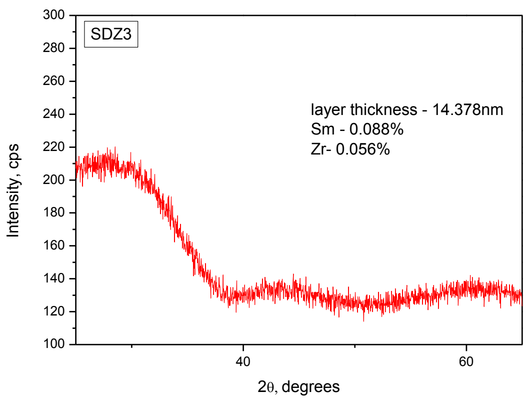


***Figure 1**. XRD and XRF analyses of the surface of the coating on glass / **SDZ3** /*

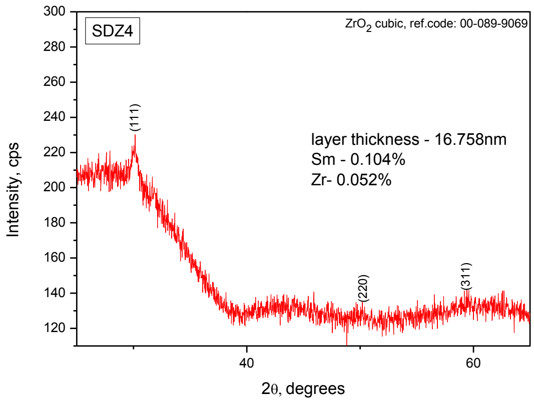


*Figure 2. XRD and XRF analyses of the surface of the coating on glass / **SDZ4** /*

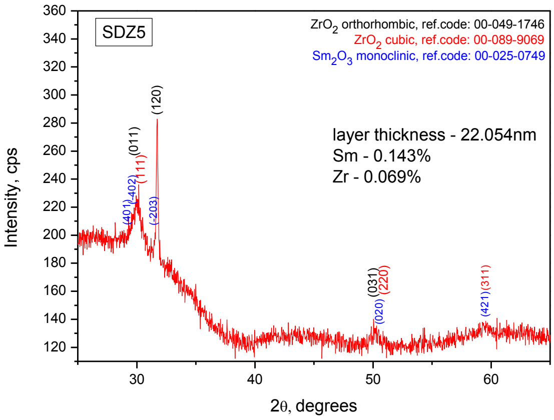


*Figure 3. XRD and XRF analyses of the surface of the coating on glass / **SDZ.5**/*

The figures show the percentage composition of ZrO_2 and Sm_2O_3 and the thickness of the coating on the modified glass surfaces. X-ray fluorescence analysis confirms the incorporation of Zr and Sm ions into the surface, forming a coating with a thickness of 14 to 22 nm.

The crystallite sizes (t) were calculated using Scherer's equation: $t = \kappa\lambda/B\cos\theta$. Here, κ is the shape factor (κ = 0.9 for spherical crystals with cubic symmetry); λ (in Å) is the wavelength; θ is the diffraction angle of the peak; and B (in radians) is the line broadening at the full width at half maximum (FWHM) values of the peaks. The crystallite sizes ranged from 9 to 76 nm for ZrO_2 and Sm_2O_3 (Table.1).

Table 1.

Samples	°2θ	hkl	t (nm)
SDZ5	31.12	(111)	9
	31.91	(120)	76
SDZ4	31.12	(111)	10

Optical characterization was performed with UV–VIS–NIR Shimadzu 3600 double-beam spectrophotometer (Shimadzu Corporation, Kyoto, Japan) in the spectral region of 240-1800 nm. The transmittance spectra of the samples were taken against air. The reflectance spectra were measured by using the specular reflectance attachment (5° incidence angle) and Al coated mirror as reference. On the figures 4,5 present the results of optical characterization.

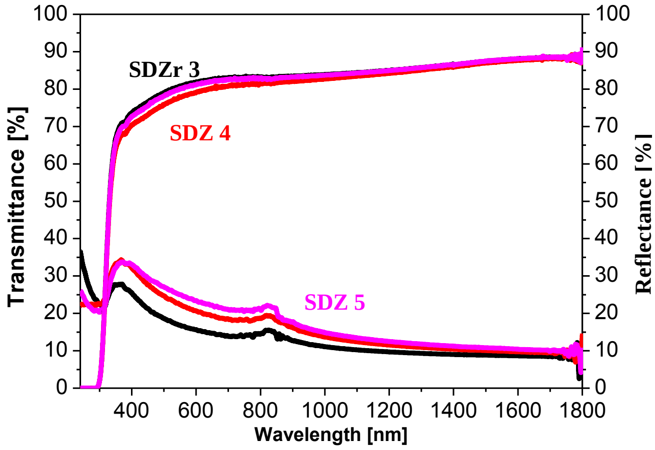


*Figure 4. The figure shows the transmission and reflection spectrum of an experimental coating on a glass substrate in the region 400 - 1800 nm / **SDZ.3, SDZ.4 and SDZ. 5** /*

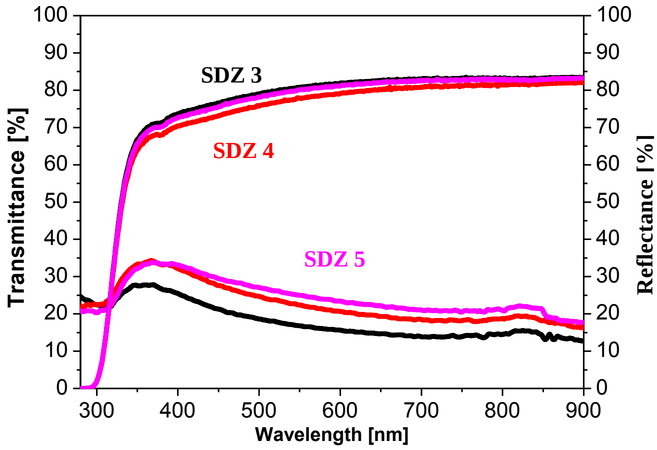


*Figure 5. The figure shows the transmission and reflection spectrum of an experimental coating on a glass substrate in the region 300 - 900 nm / **SDZ.3, SDZ.4 and SDZ.5** /*

4. Conclusions

This study attempts to improve the overall efficiency of a photovoltaic solar panel by using a ZrO_2-based coating, with Sm_2O_3.

1. The coatings are prepared by sol gel method with Dip Coating.

2.The coatings are dense and nanocrystalline, according to XRD and XRF analyses.

3. The coatings of SDZ.3, SDZ.4 and SDZ.5 samples show good transmittance by UV–VIS–NIR spectrophotometer examination.

The experimental results 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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