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Vacuum-plasma synthesis of functional coatings using targets obtained by the sol-gel method

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Abstract. To prepare coatings by electron-beam sputtering, targets have been designed and synthesized by a sol-gel method, based on SiO_2 and TiO_2 materials. The optimum basic compounds for the synthesis of stable silica sol and titanium are reported. Spectroscopic studies have been conducted for the coatings aiming at the characterization in terms of optical characteristics, such as refractive index, reflectance, transmission and absorption coefficients. The morphology of the layers was investigated by atomic force (AFM) and scanning electron (SEM) microscopies.

Introduction

The growing demand for new functionality films used in various technological applications, such as surface protection and decoration, tribology, data storage microelectronics offers a strong motivation to begin the basic and applied research on the synthesis of functional films, as well as the development of advanced deposition techniques.

The current method of vacuum plasma coating synthesis based on active gas phase allows for a technological cycle to growing complex systems based on organic and inorganic doped layers. Such configurations cannot be used by other methods, or are associated with a large number of difficulties. On the contrary, the proposed method does not require additional post-deposition high-temperature heat treatment and use of solvents. Consequently, virtually no undesirable influence is exerted on the technological factors, on the structure and morphology of the substrate. The quality and purity of the target material result in the quality of the coating and its functional properties.

To obtain coatings by electron-beam, sputtering targets were designed and synthesized in the current experiments, made of SiO_2 and TiO_2 prepared by a sol-gel type method. The optimum organic basic compound for preparing a stable silica sol and titanium was found, along with the optimal mode of gelation, drying, heath treatment. This ensured the synthesis homogeneous targets with thickness of 5÷7 mm and a diameter of 35 mm, with a mean surface area of 500 m^2/g.

Deposition of single-layer coatings was carried out using the VU-1A (Smorgon, Belarus) vacuum coaters, equipped with an UELI-I electron-beam evaporation source and a photometric thickness control Iris 0211 (Essent Optics Ltd, Belarus). The UELI-I electron beam evaporation source is specifically designed for vacuum evaporation of refractory oxides, semiconductors and metals, featuring a maximum accelerating voltage of 12 kV and a maximum load current of 500 mA. The coatings were deposited on substrates of polished silicon, quartz, optical glass BK7 [1].

Figure 1 shows the X-ray Photoelectron Spectroscopy (XPS) survey spectrum of the SiO_2 precursor surface, showing the oxygen and silicon *s* and *p* XPS peaks, along with an Auger oxygen peak (O KLL). The deconvolution of the high-resolution XPS spectra of the O 1s and Si 2p peaks allowed us to calculate the elemental composition at the surface of the coatings.

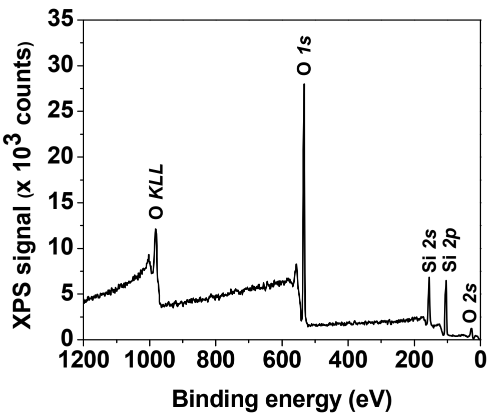


Fig.1 The XPS survey spectra of SiO_2 precursor

Investigation of the optical properties of the sampless was carried out using a spectrophotometer PHOTON RT (Belarus). Figure 2 shows the dependences of the refractive index on wavelength for four different coatings.

The AFM images of the surface of the samples are shown in Fig.3, and the numerical results on the topology of the coatings are shown in Table 1.

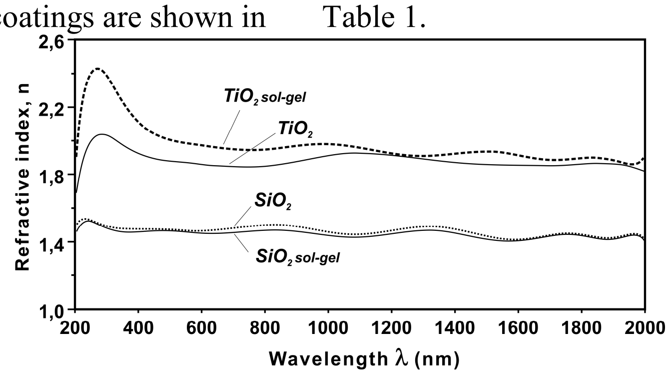
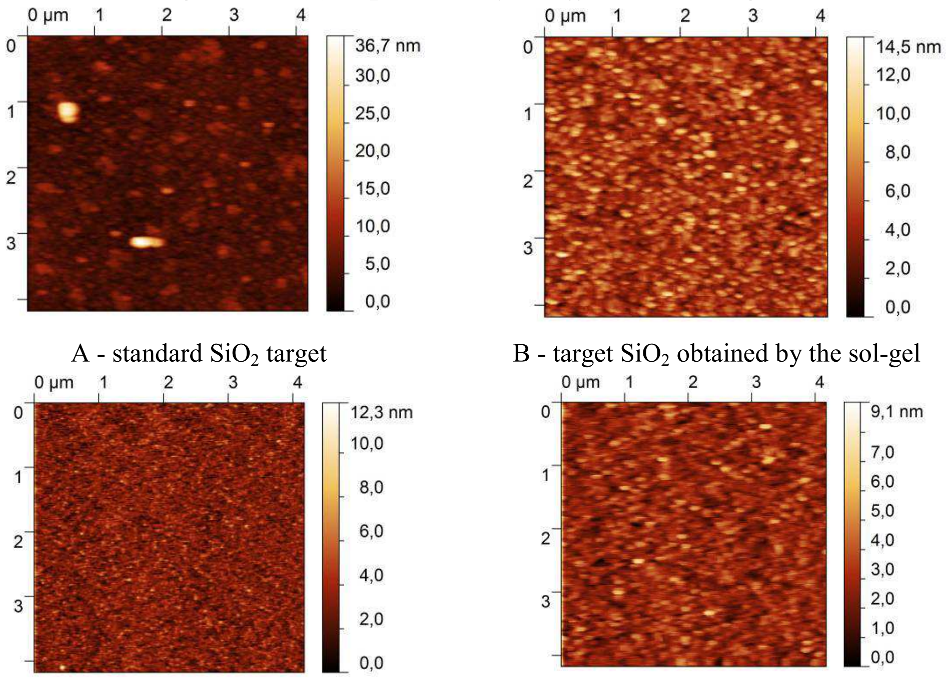


Fig.2 The n(λ) dependence for different coatings.



A - standard SiO_2 target

B - target SiO_2 obtained by the sol-gel

C - standard TiO_2 target

D - target TiO_2 obtained by the sol-gel

Fig.3 Images of the surface coatings obtained by atomic force microscopy.

Table 1 - Topological parameters of the investigated coatings

Type of coating	Mean height, Z, nm	Roughness, Ra, nm
SiO_2	5,79	0,97
SiO_2 (sol-gel)	5,37	1,21
TiO_2	3,76	0,79
TiO_2 (sol-gel)	2,86	0,64

The surface pictures in Fig. 3 show that the SiO_2 coating (sol-gel) is formed of relatively large granules, with an average size of about 80±7 nm. The coating synthesized from the standard target is characterized by a mild-grained structure of equally-sized granules of similar shape, with an average granule size of about 40±5 nm, in the presence of significantly larger fragments ranging in size from 200 to 350 nm. This difference is probably due to the peculiarities of the electron-beam evaporation of monolithic SiO_2 target, compared to the one prepared by sol-gel method.

The AFM images of the TiO_2 coatings look similar to the SiO_2 samples, i.e. the coatings leaving from the TiO_2 (sol-gel) targets are characterized by granules of typical size of about 80 nm. The TiO_2 coatings prepared from a standard TiO_2 target feature as average granule size of about 30 nm. However, the characteristic difference in morphology of the SiO_2 and TiO_2 coatings is that granules of the TiO_2 prepared from the standard target have a larger height, i.e. the granule structure of the coating is more evident.

Based on data analysis by atomic force microscopy, one can conclude that all types of coatings have low values of surface subroughness (about 1 nm). Low radiation loss when dispersed on the surface of the coatings, along with the high adhesive strength characteristics, allow the application of these coatings in various optical elements of a functional purpose.

Summary

It has been shown that the use of the target synthesized by a sol-gel method allows one to grow coatings with superior optical properties, compared to the coatings grown using conventional targets of SiO_2 and TiO_2. In such a way, multilayered interference systems can be fabricated from a single material, provided that this is made of adjacent layers with different optical characteristics.

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