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Structure and optical-mechanical properties of the Zr-Y-O coatings deposited by pulse magnetron sputtering on optical glass

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Abstract. The results of the research of the phase composition and the mechanical properties of the coatings on the basis of the Zr-Y-O system produced by pulsed magnetron sputtering on the K208 glass substrates are presented in this paper. By the X-ray diffraction method it has been discovered that there is the ZrO_2 phase in two modifications in the coatings. The deposition of the Zr-Y-O coating system allows not only to increasing the microhardness of the surface layer of the K208 glass up to ~19 GPa, but also maintain a high level of the elastic properties (We> 0.70). The laboratory tests have been carried out on the impact of the high-speed flows of iron microparticles on the Zr-Y-O protective coating produced by pulsed magnetron sputtering. When a high-speed flow of iron microparticles impact the samples with the Zr-Y-O coatings the surface density of the craters decreases ~ 7 times compared with initial state.

1. Introduction

In near-Earth space, outer elements of spacecraft are almost continuously subjected to impacts of high-speed micrometeoroids and particles of anthropogenic pollution. Optical elements [1], such as glass windows, lenses, as well as photoelectric converters of solar batteries suffer the most. As a result of such collisions, mechanical damage in the form of craters and cracks is formed on the glass surface. An increase in their density during long-term operation leads to a significant erosion of the surface, a decrease in transparency and a degradation of the mechanical properties of optical elements.

It was shown [2], that surface modification by protective Al-Si-N coatings can significantly increase the erosion resistance of quartz glasses used in spacecraft windows. However, the significant difference in the thermal expansion coefficient of the Al-Si-N coating and the K208 glass [3] complicates the task of deposition of high-quality coating on thin plates of optical glass. The Zr-Y-O system as a material for deposition of a protective coating has a closer coefficient of thermal expansion to K208 glass. It also has a unique set of properties [4], such as high strength, wear resistance and transparency. It may be a suitable option for the K208 protecting glasses of the solar cells of the spacecraft from the impact of high-speed microparticles.

The purpose of the work is to study the structural state, optical and mechanical properties of coatings based on the Zr-Y-O system, as well as the effect of these coatings on the resistance of K208 optical glasses against the impact of solid microparticles moving with a speed close to the first cosmic one.

2. Experimental procedure

The investigated coatings were deposited on substrates, which were polished discs ∅15 × 4 mm in size made from the K208 radiation-resistant glass, using pulsed magnetron sputtering on a UVN-05MD KVANT vacuum unit (Techimplant Ltd) [5] in an Ar + O_2 gas mixture using a target based on Zr-Y. The temperature of the substrates during the coating process was 573 K. The thickness of the coatings was 5–6 µm.

The structural-phase state of the samples was investigated by X-ray using the DRON-7 device (Burevestnik Innovation Center, JSC). X-ray investigation of the coatings was carried out under continuous 2θ - scanning with the Bragg - Brentano focusing at Co Kα radiation (wavelength λ= 1.78897 A). The data base JCPDS PDF-2 (The Joint Committee on Powder Diffraction Standards) was used for interpretation of the diffractograms.

Analysis of the optical properties of magnetron coatings of the Zr-Y-O system on quartz glass substrates was based on measurements of the transmittance T = f (λ) in the wavelength range λ = 200-1100 nm using the SF-256 UVI spectrophotometer (LOMO Photonics). The principle of measuring the ratio of two light fluxes that have passed through the investigated and the standard sample respectively was taken as a basis.

The mechanical properties of the coatings and the glass substrates were measured using the NanoHardnessTester (CSM Instruments, Switzerland) under the load on the indenter 20 mN, with the depth of indentation of the coated samples not exceeding 220 nm. In order to obtain reliable results for each sample at the above loading no less than 10 prints were made and the final result was the arithmetic mean value. The microhardness, the modulus of elasticity and the elastic recovery coefficient of the coatings were calculated on the basis of the analysis of the unloading branch obtained by the Oliver - Pharr method [6].

To assess the impact-protective properties of the coatings, the experimental samples were bombarded by high-speed microparticles in the light-gas gun MPH23/8 [7]. The gun was developed in the Institute of Applied Mathematics and Mechanics affiliated to Tomsk State University. The experimental samples were bombarded by iron microparticles of the spherical form with an average size of 56 µm at the velocity of 5-8 km/s. After the shock impact test the images of the craters formed on the surface of the samples were investigated using a scanning electron microscope LEO EVO-50XVP (Karl Zeiss Group, Germany), and the surface density of the craters (ρ) was calculated.

3. Results and discussion

The X-ray diffraction analysis previously established that there were ZrO_2 phases in various structural modifications in the coating based on Zr-Y-O [8,9]. The phase ZrO_2 was represented by two modifications: tetragonal (t) and monoclinic (m). The quantity of tetragonal and monoclinic phases was 44 and 56%, respectively. Figure 1 shows the X-ray diffraction patterns of the Zr-Y-O coating with a thickness of coating about of 6 µm.

TEM shows that the grain structure of the coating based on the Zr-Y-O system is columnar. The longitudinal grain size of the ZrO_2 phase coincides with the growth axis of the coating and is determined by the thickness of the layer, and the transverse grain size does not exceed ~ 80 nm. Figure 2 shows the electron microscopic image of the columnar structure of a coating based on the ZrO_2.

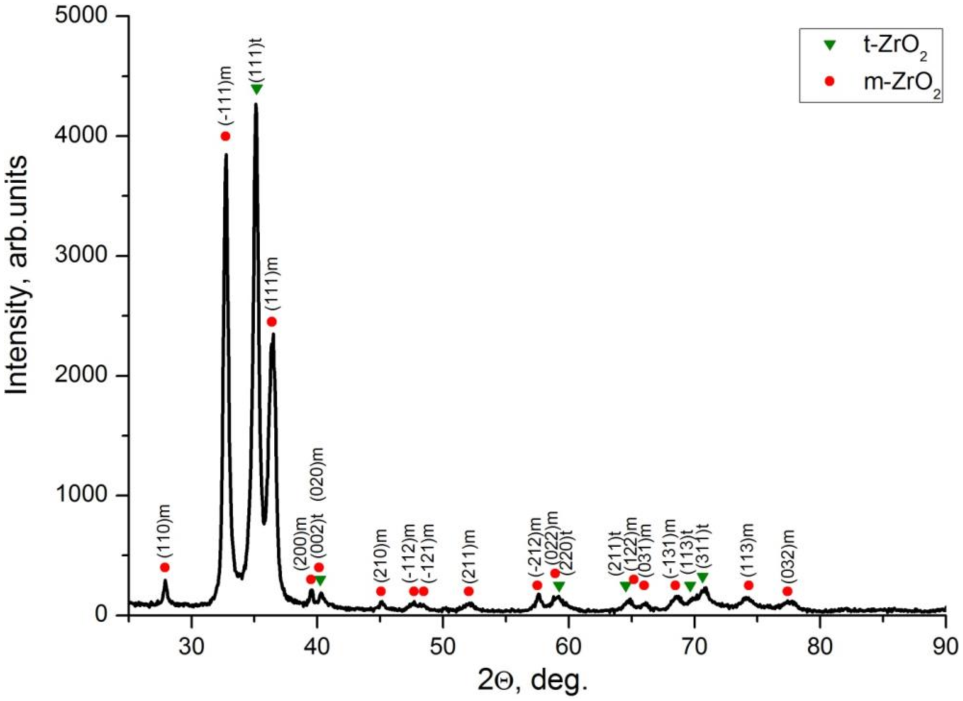

Figure 1. X-ray patterns of coatings on the basis of Zr-Y-O on the K208 glass substrate.

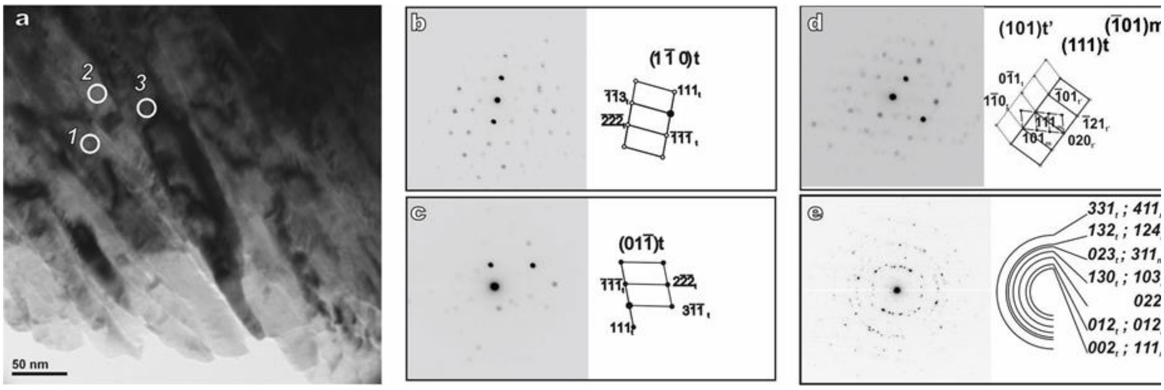

Figure 2. TEM image of the coating layer of based on Zr-Y-O in the initial state; a is the bright-field image of the cross section; b is the microdiffraction pattern and the scheme of its indexing of area 1; c is the microdiffraction pattern and the scheme of indexing of area 2; d is the microdiffraction pattern and the scheme of the indexing of area 3; e is the ring microdiffraction and the scheme of indexing of the whole region.

The circles in figure 2 show the bright-field image of the cross-section of the foil and the microdiffraction pattern of the Zr-Y-O coating from sections 1, 2, 3 in the initial state. There is the phase ZrO_2 in area 1 with the crystalline lattice parameters a = 5.12 Å and c = 5.25 Å. In area 2 there is the same phase but one can see another plane. In area 3 there are two tetragonal phases (t and t' with a smaller lattice parameter a = 3.64 Å, b = 3.64 Å, c = 5.27 Å) and a monoclinic modification of ZrO_2 with the lattice parameters a = 5.31 Å, b = 5,21 Å, c = 5.15 Å. These data are consistent with the results of the X-ray diffraction analysis.

Figure 3 shows the transmission spectra of the investigated samples in comparison with the transmission of light for a k208 glass substrate. One can see that the initial glass K208 (figure 3, curve 1) has a high transmittance of 87-90% in all investigated visible wavelength interval. The deposition of protective coatings of the Zr-Y-O system results in an insignificant decrease in the light transmittance of K208 glass samples (down to 60-80%) in the visible region of the spectrum (figure 3, curve 2). The oscillating character of the experimental transmission spectra observed for samples of K208 glass with protective coatings in the visible region of the spectrum is due to the phenomenon of interference of light in the coatings.

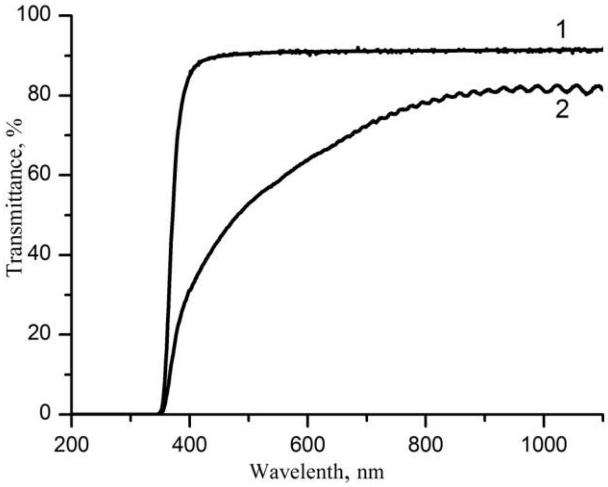

Figure 3. Spectra of transmission of K208 glass samples with and without protective coatings: 1–K208 glass; 2 – the same with Zr-Y-O coatings.

The results of the investigation of the mechanical properties of the Zr-Y-O are shown in Table 1. One can see that the microhardness for the Zr-Y-O coatings exceeds the microhardness of the initial K208 glass more than 2 times and the coefficient of elastic recovery - by 1.3 times. It is important to note that for all Zr-Y-O coatings a high microhardness is combined with a high coefficient of recovery ($W_e > 0.70$).

It is known [1, 2] that at the impact of high-speed particles at the target surface a dispersing shock wave is formed, which propagates deeply into the material. As a result, a crater is formed of the impact spot on the target surface. Figure 3 shows the images of the craters formed on the surface of the samples as a result of the exposure to high speed iron microparticles flow.

Table 1. Average values of the microhardness H_m, the elastic modulus E*, the elastic recovery ratio W_e, the surface density of the craters ρ on the K208 glass substrate with the Zr-Y-O coating as compared to the substrate without a coating.

Sample	Microhardness, H_m GPa	Young's modulus, E*, GPa	Coefficient of elastic recovery, R_e	The surface density of the craters ρ, mm^2
Glass K208	8.40 ± 0.15	93.0 ± 0,8	0.53	20.19
Glass K208 with Zr-Y-O coatings	18.81±0,69	318.6±8,8	0.72	3.04

One can see that the craters formed on the K208 glass surface have a shape characteristic of brittle materials. There are a large number of craters having various sizes (figure 4a) on the surface of the initial K208 glass. On glass substrate with the Zr-Y-O protective coating the number of the craters decreases by ~ 7 times. The quantity of the craters whose cross sectional dimension does not exceed 50 µm decreases to large degree.

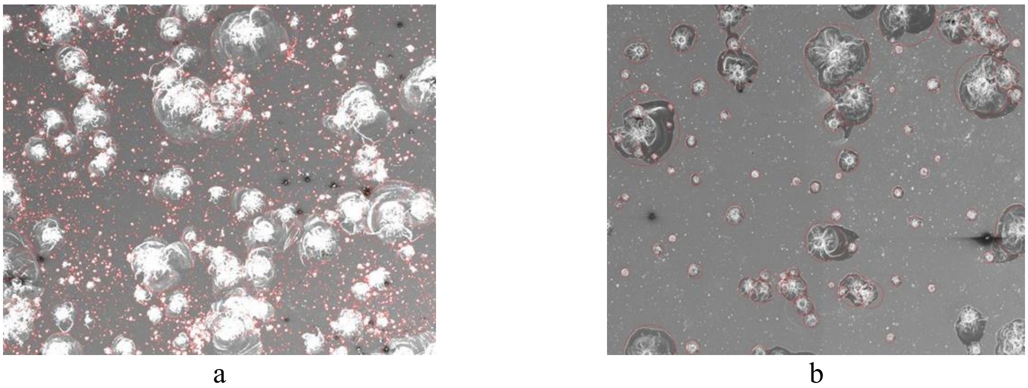
a b

Figure 4. SEM images of craters formed on the samples surface as a result of high-speed impact flow by iron microparticles: a - uncoated sample, b - Zr-Y-O coated sample (craters are circled by red circles).

4. Conclusion

The Zr-Y-O coatings deposited on K208 glass by pulsed magnetron sputtering have a crystalline multiphase structure including the ZrO2 phases in monoclinic and tetragonal modifications. The deposition of these coatings leads to significantly increase in the erosion resistance of K208 glass. The decrease in the surface density of craters on the surface of the samples after impact of high-speed iron microparticles by ~ 7 times compared to the initial ones indicates this. The microhardness of the K208 glass substrate with deposited Zr-Y-O coating was 18.81 GPa.

Acknowledgments

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