Formation of optically transparent nanocomposite protective coatings on glass produced by ionic implantation and magnetron sputtering methods for space applications

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Abstract. The purpose of this research is design of a magnetron deposition technology for quartz glasses of optically transparent nanocomposite protective coatings based on Al-Si-N with preliminary modification of the substrate surface by a high-energy Ni^+ ion beam. The coating improves the mechanical properties and resistance of the glass to the impact of high-speed iron microparticles. The structural-phase state and elemental composition of the coatings and the surface layer of the substrate are investigated by electron microscopy, electron-probe analysis, and secondary ion mass spectrometry.

1. Introduction

Continuous bombardment of glass windows of spacecraft in space by the flow of high-speed micrometeoroids and space debris leads to the craters formation with the subsequent occurrence of cracks on the glass surface [1]. This phenomenon results to degradation of the optical and mechanical characteristics of the quartz glass and their failure. A possible way to solve the problem can be the deposition of protective coatings from materials with high impact resistance and melting point, which are transparent in the visible region of the spectrum [2, 3].

The possibility of using ion implantation and pulse magnetron deposition of nanocomposite coatings on the basis of the Al-Si-N system for protection of quartz glasses against the flow of high-speed solid microparticles is studied.

2. Experimental procedure

As experimental samples, industrial quartz glass plates were used, on which Al-Si-N coating with a thickness up to 10 μm with Al:Si = 3:1 atomic concentrations was formed. The deposition of the coatings was implemented using the pulse magnetron method by sputtering of the mosaic targets on the basis of aluminum with silicon inserts using the UVN-05MI "KVANT" vacuum magnetron sputtering equipment (Techimplant Ltd., Russia). A bipolar pulsed power supply with a frequency up to 100 kHz was used.

Before the coating deposition, the surface of the quartz substrate was bombarded by high-energy nickel ions using a pulsed vacuum arc ion source "DIANA-3" (Techimplant Ltd., Russia), at first the accelerating voltage was 80 kV and a fluence 10^{17} cm^{-2} and then 40 kV and $2{\cdot}10^{17} cm^{-2}$.

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The structure and phase composition of the samples was investigated by the transmission electron microscope JEOL-2100F (Jeol Ltd., Japan) in Department of Nanomaterials and Nanotechnologies in Tomsk Polytechnic University. The average grain size <d> was determined using the dark-field images obtained by the TEM. To determine the phase and chemical composition of individual structural components and local micro regions in the coating the modes of microdiffraction and micro-X-ray analysis (INCA-Energy X-act, INCA-Wave, Oxford Instruments, GB) were used. The foils were prepared by the «cross-section» method using the Ion Sliser-EM-09100IS installation (Jeol Ltd., Japan). Work was performed in the "Nanotech" user center of scientific instruments of ISPMS, SB RAS.

The microhardness, the modulus of elasticity and the elastic recovery coefficient of the coatings and the quartz substrates were measured using the NanoHardnessTester (CSM Instruments, Switzerland) under a load on the indenter 20 mN.

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 [4] (Institute of Applied Mathematics and Mechanics affiliated to Tomsk State University). The iron microparticles of the spherical form with an average diameter of 56 μm with the velocity of 6–8 km/s were used for a bombardment.

3. Results and discussion

As a result of ion implantation in the above mode, Ni^+ ions are introduced into the surface layer of quartz plate on depth up to 150 nm (figure 1). In this case, the microhardness H_m and the elastic modulus E^* of the surface layer of quartz plate increase 1.2 time. A slightly smaller increase in the elastic recovery coefficient K_e is observed (table 1). However it has enhanced substantially an adhesion of Al-Si-N coatings with a quartz substrate.

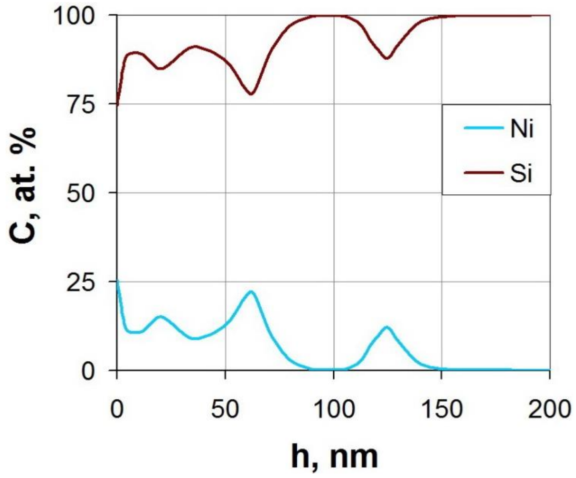

Figure 1. Concentration profile of implanted Ni^+ ions in the surface layer of SiO_2 quartz plate, obtained by the secondary ion mass spectroscopy method (SIMS).

Using the TEM method we determined (figure 2) that the Al-Si-N coating has a two-phase structure consisting of AlN nanograins (figure 2(a)) with a hcp crystalline lattice (figure 2(c), (d)) and Si_3N_4 phase in an amorphous state (figure 2(a), (b)) in the form of interlayers between nanograins of AlN similar to that observed in coatings Al-Si-N with a low Si content [5]. The average grain size of AlN was determined using the dark-field images and it was about 6 nm.

Measurements using UVIKON 943 spectrophotometer, the light-transmission factor of the quartz plate with Al-Si-N nanocomposite coating in the visible wavelength region is not less than 0.85 ± 0.02, which is close to its value for quartz without coating 0.90 ± 0.02.

Table 1 shows the values of the microhardness H_m, the elastic modulus E^*, and the elastic recovery coefficient K_e. Microhardness of ion-implanted samples with Al-Si-N coating 3.5 times and K_e 1.6 times is higher than on the initial samples without coating. The value of the elastic modulus for an ion-implanted quartz substrate with Al-Si-N coating exceeds its value for the initial quartz glass 3.1 times. After impacts on the glass samples with Al-Si-N coatings and without them by the Fe high-speed microparticls the craters are formed in the glass surface as we see in figure 3. These data were obtained using LEO EVO 50 scanning electron microscope (Carl Zeiss Group, Germany).

Table 1. An average values of the microhardness H_m, elastic modulus E^*, elastic recovery coefficient K_e

Kind of a samples	H_m, GPa	E^*, GPa	K_e	ρ, 10^6 m^{-2}
Quartz as initial without coatings	9.3 ± 0.6	78.2 ± 5.9	0.51 ± 0.04	26.9
Quartz as treated by a Ni^+ ion beam	11.1 ± 0.8	93.5 ± 9.2	0.60 ± 0.06	–
Quartz as treated by a Ni^+ ion beam with an Al-Si-N coating	32.4 ± 1.9	241 ± 25	0.82 ± 0.09	8.85

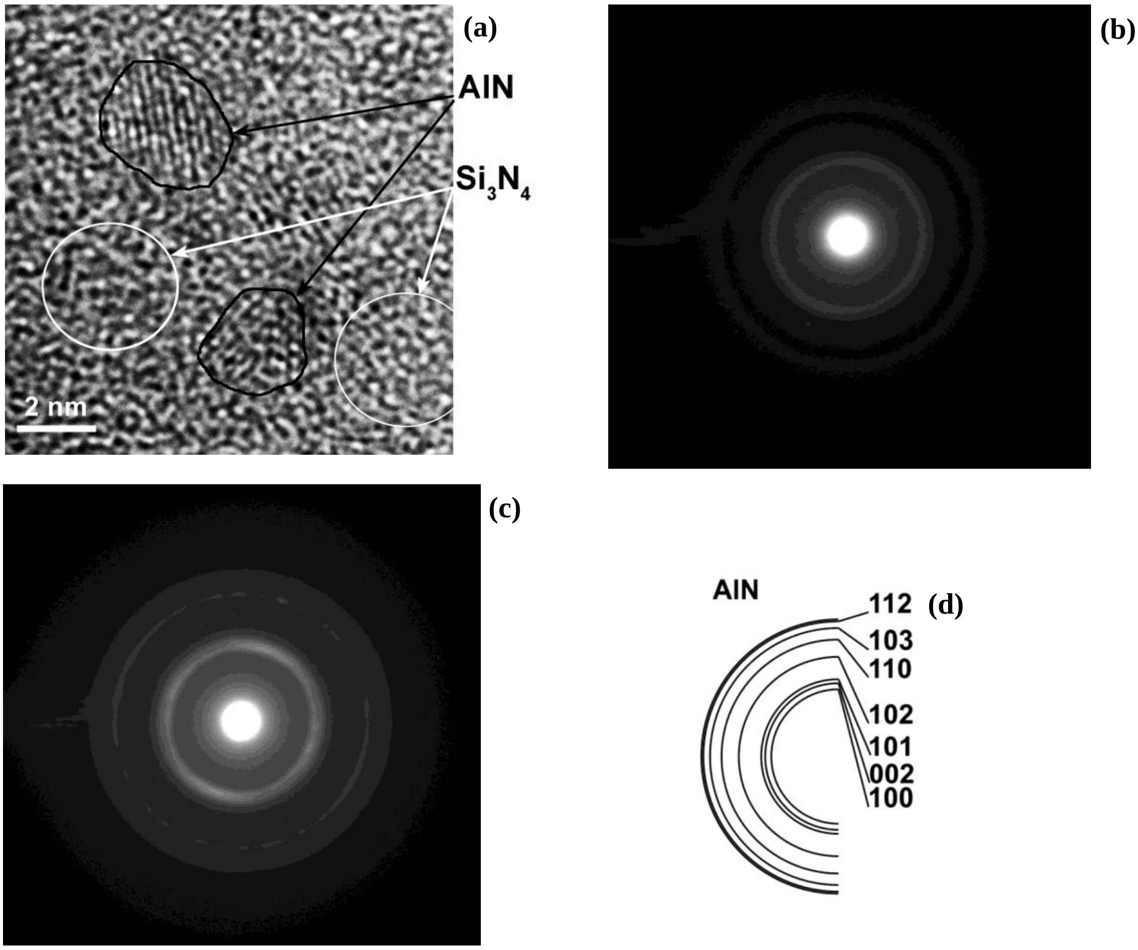

Figure 2. TEM bright-field image of cross-section of coating on the basis of Al-Si-N **(a)**, micro diffraction patterns obtained from areas of Si_3N_4 amorphous phase **(b)** and micro diffraction patterns obtained from areas of nanocrystalline AlN phase **(c)** and their indexing schemes **(d)**.

One can see (figure 3) that ion-implanted quartz glasses with coating and without them have the different surface density of the craters ρ under the same test conditions.

The surface density of craters formed on the surface of quartz glass under impacts of Fe microparticles with speed up to 6–8 km/s was decreased about 3 times by the Ni^+ ion implantation and magnetron deposition of Al-Si-N nanocomposite coatings (table 1).

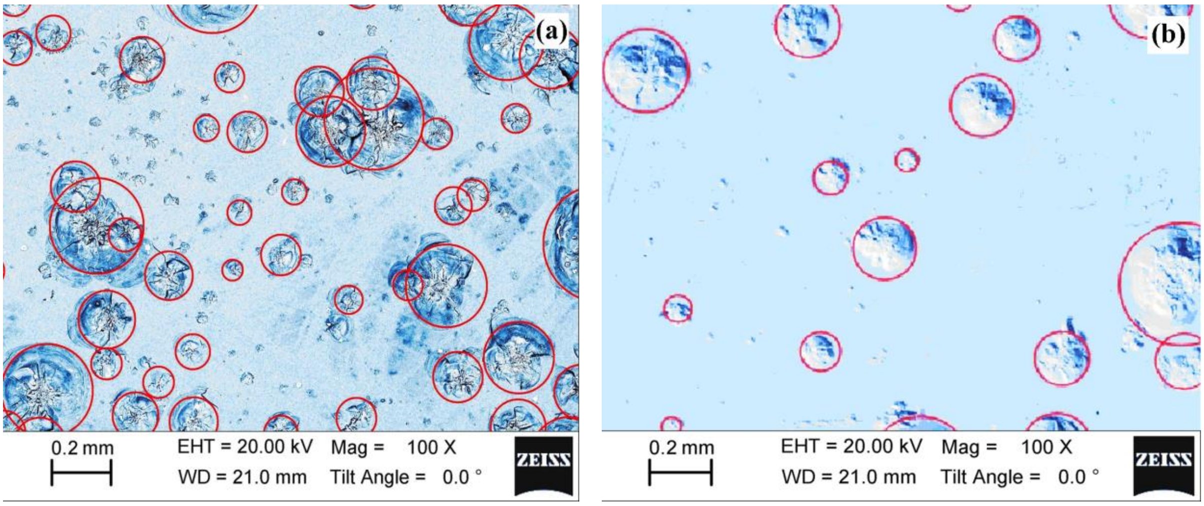

Figure 3. SEM images of the craters formed on the surface of the samples under the action of the high speed flow of the Fe microparticles: **(a)** – part of a surface of the initial quartz sample without the coating; **(b)** – part of a surface of quartz sample with preliminary Ni^+ ion implantation and with Al-Si-N coating. The craters on the SEM images are marked by circles.

4. Conclusions

The possibility of using ion implantation and pulse magnetron deposition of nanocomposite coatings on the basis of the Al-Si-N system for protection of quartz glasses against the flow of high-speed solid microparticles was studied. By SIMS method it has been established that Ni^+ ions are introduced into the surface layer of quartz glass on depth up to 150 nm. In this case, the microhardness, elastic modulus and elastic recovery coefficient of the surface layer of Ni^+ ion implanted quartz plate increase weakly. But it has enhanced substantially an adhesion of Al-Si-N coatings with the Ni^+ ion implanted quartz substrate.

By TEM it was shown that there is two-phase structure consisting of AlN nanograins with a hcp crystalline lattice and Si_3N_4 phase in an amorphous state having form of interlayers between nanograins of AlN in the Al-Si-N coating. Microhardness of ion-implanted samples with Al-Si-N coating increases 3.5 times and elastic recovery coefficient 1.6 times compare with initial samples without coating. The value of the elastic modulus for an ion-implanted sample with Al-Si-N coating exceeds its value for the initial quartz glass 3.1 times.

By SEM it was shown that the surface density of craters formed on the surface of quartz glass under impacts of Fe microparticles with speed up to 6–8 km/s decreased about 3 times by the Ni^+ ion implantation and pulse magnetron deposition of Al-Si-N nanocomposite coatings. The light-transmission factor of the quartz plate with Al-Si-N nanocomposite coating in the visible wavelength region is not less than 0.85 ± 0.02, which is close to its value for quartz without coating 0.90 ± 0.02.

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