

Magnetron Deposition of Metal-Ceramic Protective Coatings on Glasses of Windows of Space Vehicles

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Abstract. Transparent refractory metal-ceramic nanocomposite coatings with a high coefficient of elastic recovery and microhardness on the basis of Ni/Si–Al–N are formed on a glass substrate by the pulse magnetron deposition method. The structure-phase states were investigated by TEM, SEM. It was established that the first layer consists of Ni nanograins with a fcc crystalline lattice, the second layer is two-phase: 5–10 nm nanocrystallites of the AlN phase with the hcp crystalline lattice in amorphous matrix of the Si₃N₄ phase.

Keywords: magnetron deposition, protective coating, glass, space vehicle, structure-phase state, microhardness, crater density

INTRODUCTION

Continuous bombardment of the illuminator glasses of a spacecraft by high-velocity micrometeoroids leads to the formation of craters and then microcracks form on the surface and propagate deeper into the glass [1–3]. This leads to the degradation of the optical and mechanical properties of the glass windows of a spacecraft. One way to solve this problem is the deposition of refractory coatings with a high coefficient of elastic recovery and a low thermal coefficient of linear expansion transparent in the visible region of the spectrum [4, 5]. Composite coatings consisting of metal and ceramic layers can have a wide range of properties.

In this paper the possibility of application of the magnetron deposition method of two-layer metal-ceramic coatings on the basis of Ni/Si–Al–N to protect quartz glass from the high-velocity flow of hard microparticles used in the ports of a spacecraft is considered.

EXPERIMENTAL PROCEDURE

Plates of quartz glass of the KV type in the form of disks 15 mm in diameter and 4 mm in thickness were used as experimental samples. A two-layer coating was deposited on one side of the glass. The first metal layer was 50 nm in thickness and consisted of nickel (pure 99.99 wt.%) It was deposited in the argon using a magnetron with unipolar pulsed power with a frequency of 50 kHz.

TABLE 1. Average values of the microhardness H_m , the modulus of inelastic buckling E^* , the elastic recovery coefficient k_y ,

Samples	H_m , GPa	E^* , GPa	k_y
Glasses without coatings	9.26 ± 0.58	$75.1. \pm 3.8$	0.48 ± 0.07
Glasses with coatings Si–Al–N	31.6 ± 3.6	264.7 ± 19.5	0.91 ± 0.12

The second ceramic 8 μm layer, consisting of silicon nitride-aluminum with the ratio of the atomic concentrations of Si : Al = 1 : 4, was deposited by magnetron reactive sputtering of composite targets using the vacuum unit UVN-05MI KVANT (NPP Tehimplant Ltd., Russia) [5]. The power was supplied from a magnetron pulse bipolar source with a frequency up to 100 kHz and was stabilized (OOO “Applied Electronics”, Russia).

The microstructure and the phase composition were investigated by transmission electron microscopy (TEM) a JEM-2100 instrument (Jeol, Japan). The average grain size $\langle d \rangle$ was determined using darkfield images obtained by TEM.

The phase and the chemical composition of individual structural components and the thin layers in the coating were determined using the modes of microdiffraction and the electron microprobe analysis by TEM. Foils for investigation were prepared by the “cross-section” method using an ION SLISER EM-09100IS device (Jeol, Japan).

The microhardness, the elasticity modulus and the elastic recovery coefficient of the coatings and the glass substrates were measured using NanoHardness Tester (CSM Instruments, Switzerland) under 20mN a load on the indenter (Table 1).

To test a sample for impact resistance, a special tooling was designed, which was a subject table placed in a vacuum chamber at a fixed position relative to the light-gas gun barrel. This table was designed for simultaneous deployment of four original glasses and four glasses with coatings in strictly defined positions relative to the light-gas gun barrel. Bombardment particles for the experimental samples were microparticles of iron selected with an average size of $56.3 \pm 8.2 \mu\text{m}$, with a particle shape close to spherical. The portion of powder for each shot was constant $60.0 \pm 0.1 \text{ mg}$, the shot velocity was 3–5 km /s.

RESULTS AND DISCUSSION

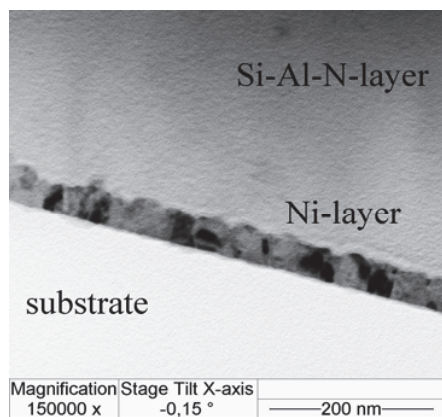
It was established by TEM (Fig. 1) that the first metallic layer in the coating was nanocrystalline and monophase. There were nanograins with the fcc crystalline lattice (Fig. 1(e, f)). The second layer of Si–Al–N coating had a two-phase nanostructure consisting of AlN crystallites with the hcp lattice and the Si_3N_4 phase in the amorphous state in the form of layers between the AlN grains. Using the dark-field images obtained by TEM, the average grain size of the AlN phase was determined, which was in the range of 5–10 nm.

The microstructure and the phase composition of the coatings differed from the coatings produced previously on the basis of Si–Al–N [6] due to the differences in the chemical composition and the mode of preparation. Their characteristics were closer to the coatings on the basis of Al–Si–N studied in [7].

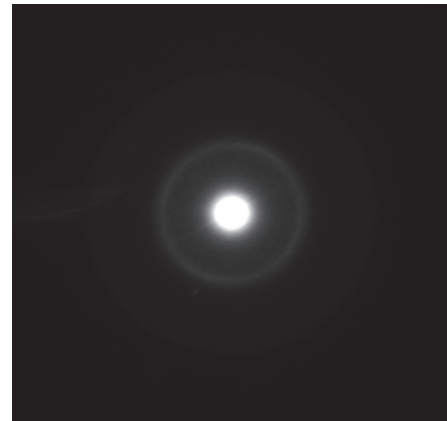
The coefficient of light transmission is measured by the UVIKON 943 spectrophotometer (Kontron Instruments) in the visible area of wavelengths no less than 0.85 ± 0.02 and is close to the value for glass without a coating (0.90 ± 0.02).

The microhardness H_m was measured for the Si–Al–N silica glass with and without a coating, the elastic recovery factor k_y and the modulus of inelastic buckling E^* were determined. These data are shown in Table 1. It is seen that the microhardness of the samples with coatings is 3.4 times higher and the elastic recovery factor is 1.8 times greater compared with the samples without coatings. As for the modulus of inelastic buckling, its value for the samples with coatings is 3.5 times greater compared with the samples without coatings.

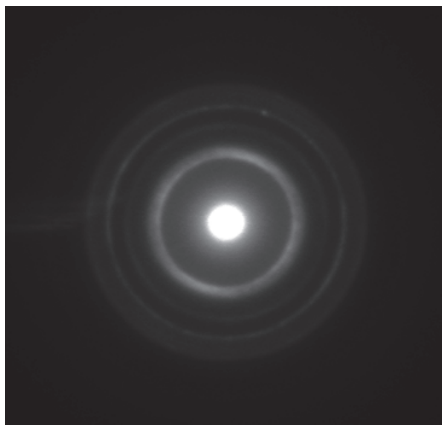
After shooting of the glasses by Fe microparticles a 10–20 nm thin silver film (a conductive layer) was deposited on their surface. Then the surface morphology of the samples was studied by SEM. Investigations were carried out in the secondary electron mode with the magnification 100 at an accelerating voltage of 20 kV. Each sample series of images was photographed and then joined together to produce a panorama. After that an area about 100 mm^2 was chosen on each of the 8 samples and measurements of the transverse size were made. The results for all samples with coatings were tabulated and treated as a single array. These data were compared with similar data obtained for 4 samples of the original glass without coatings. Figure 2 shows an image obtained by SEM, where the circles outlined the craters appearing after shooting. It was established that the crater surface density of glasses with coatings is significantly lower than for the glasses without coatings at the same test conditions (Fig. 1). The surface density ρ for the glasses with coating was $0.39 \times 10^6 \text{ m}^{-2}$ and for the glasses without coatings was $1.08 \times 10^6 \text{ m}^{-2}$.



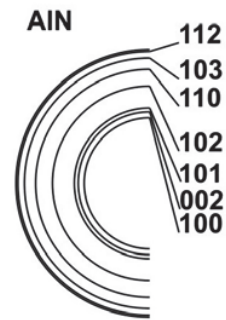
(a)



(b)



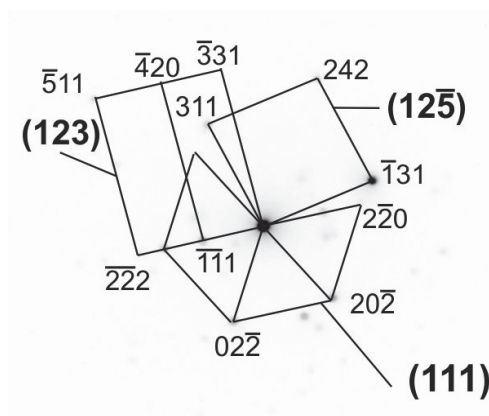
(c)



(d)



(e)



(f)

FIGURE 1. TEM image of the cross-section of the two-layer coating Ni/Si-Al-N: bright-field (a) microdiffraction pattern of the amorphous layer (b), microdiffraction pattern of the coating on the basis of Si-Al-N (c, e) and schemes of their indexing (d, f)

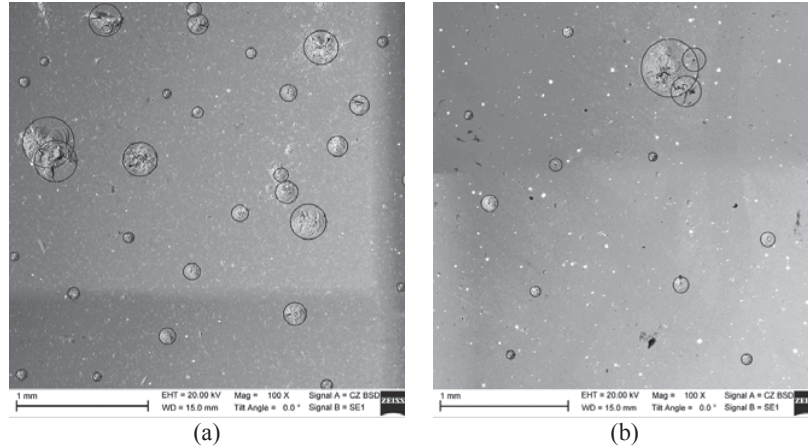


FIGURE 2. The images of craters formed under a flux of the iron particles on the samples without (a) and with coatings (b)

Thus, the deposition of two-layer metal-ceramic nanocomposite coatings decreases ~ 2.7 times surface density of the craters, formed on the surface of the silica microparticles during shooting by Fe microparticles moving at a velocity of 3–5 km/c.

CONCLUSION

In this work it is shown that the metallic-ceramic coatings on the basis of Ni/Si–Al–N obtained by pulsed magnetron sputtering of composite targets have a two-layer nanostructure consisting of the Ni monophase nanograin layer 50 nm in thickness, crystallites of the AlN phase with the hcp crystalline lattice and the Si_3N_4 phase having the amorphous structure in the form of layers between the grains of the AlN phase 8 μm in thickness. The average grain size of AlN was in the range of 5–10 nm.

These coatings are transparent in the visible spectrum and they have high values of microhardness, the modulus of elasticity and the elastic recovery ratio. Their deposition on quartz glass increases the glass impact resistance to iron microparticles with an average size of about 56 μm moving at a velocity of 3–5 km/c. The obtained data have shown that the deposition of coatings on the basis of Si–Al–N on quartz glass leads to the decrease in the surface density of the craters 2.7 times at the bombardment of the glass with light gas guns.

ACKNOWLEDGEMENTS

The work was supported within the scope of the basic scientific research of state academies of sciences for 2013–2020 performed within the scope of state task “Science NRTPU”.

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