# ИЗВЕСТИЯ ВЫСШИХ УЧЕБНЫХ ЗАВЕДЕНИЙ

T. 57, № 10/3

ФИЗИКА

2014

UDC 538.975: 539.234: 539.4.015.1: 629.7.023.[222+26]

V.P. SERGEEV\*,\*\*, M.P. KALASHNIKOV\*, A.V. VORONOV\*, I.A. BOZHKO\*,\*\*, E.V. RIBALKO\*, Yu.F. KHRISTENKO\*\*\*

## MAGNETRON DEPOSITION OF PROTECTIVE COATINGS ON THE BASIS OF SI-AL-N ON GLASSES OF WINDOWS OF SPACE VEHICLES<sup>1</sup>

Refractory coatings with a high coefficient of elastic recovery and low thermal coefficient of linear expansion on the basis of Si–Al–N are formed on glass substrate by pulse magnetron deposition method. The structure-phase states were investigated by TEM, SEM, X-ray. It was established that there are crystallites of the AlN phase with hcp crystalline lattice and  $Si_3N_4$  phase having amorphous structure in the form of layers between grains of the AlN phase. These coatings have high microhardness, modulus of elasticity and elastic recovery ratio.

Keywords: protective coatings, space vehicles, structure-phase state, microhardness, craters.

#### Introduction

Clashes of spacecraft (SC) with meteoroids of natural origin and space products of anthropogenic pollution are among the major factors causing the damage and destruction of SC [1, 2]. The greatest the variety optical elements such as windows, solar panels, etc. suffer from strikes particles. High-velocity kick of microparticles, regardless of its origin, causes mechanical and plasma processes when the crater is formed on the surface, a shock wave propagates; the microcracks are generated [3]. This leads to the degradation of the optical and mechanical properties of glass windows in the operation of spacecraft. One way to solve this problem is deposition of refractory coatings with a high coefficient of elastic recovery and low thermal coefficient of linear expansion, clear in the visible region of the spectrum [4, 5].

Purpose of this work is to improve impact durability of quartz glass under high-velocity (3-5 km/sec) microparticle bombardment of iron used in the ports of the spacecraft, using magnetron sputtering on them transparent coatings on the basis of Si–Al–N.

#### **Experimental**

Studies were carried out on two types of specimens made of quartz glass: with coating on the basis Si–Al–N 10 µm in thickness and without coating. Coating deposition was performed by magnetron reactive sputtering composite targets on the vacuum unit UVN-05MI "KVANT" (Scientific-Production Enterprise Tehimplant Ltd., Russia) [6]. Power was supplied from the magnetron pulse bipolar source with a frequency up to 100 kHz and stabilization of power (OOO "Applied Electronics", Russia).

Structural-phase state was investigated by X-ray using a DRON-7 (Scientific-Production Enterprise "Burevestnic", Russia) in CoK<sub>a</sub> radiation and TEM JEOL-2100 instrument (Jeol, Japan). The average grain size  $\langle d \rangle$  was determined using darkfield images obtained by TEM. Microhardness, modulus of inelastic buckling and elastic recovery coefficient of coatings and glass substrates was measured using NanoHardnessTester (CSM Instruments, Switzerland) under a load on the indenter 20 mN (Table 1). For

Table 1 Average values of microhardness  $H_m$ , modulus of inelastic buckling E\*, elastic recovery coefficient  $k_v$ 

Samples	<i>H</i> <sub>m</sub> , ГПа	<i>Е</i> *, ГПа	$k_y$
Glass without coatings	9.26±0.58	75.1±3.8	$0.48{\pm}0.07$
Glass with coatings Si–Al–N	31.6 ± 3.6	264.7±19.5	0.91±0.12

testing of experimental samples the special tooling was designed: the object table, which was placed in a vacuum chamber at a fixed position relative to the light-gas gun barrel. This table is provided for the si-

<sup>&</sup>lt;sup>1</sup> Work was performed in the scope of basic scientific research of the state academies of sciences for 2013–2020.

multaneous deployment of four original glasses and four glasses with coatings in strictly defined positions relative to the light-gas gun barrel.

As for the particle for bombardment of experimental samples the microparticles of iron powder were selected with an average size of  $(56.3 \pm 8.2) \mu m$ , with a particle shape close to spherical. Portion of the powder for each shot was constant  $(60.0 \pm 0.1) mg$ , shot velocity was 3–5 km/s.

### 3. Results and discussion

By X-ray diffraction and TEM (Fig. 1) it was established that the Si–Al–N coating have two phase nanostructure consisting of AlN crystallites with hcp lattice and phase  $Si_3N_4$ , being in an amorphous state in the form of layers between the grains of AlN. Using darkfield images obtained by the TEM, the average grain size of AlN phase was determined, which is in the range of 5–12 nm. The microstructure and phase composition of the coatings differ from coatings produced previously on the basis of Si–Al–N [7] due to differences in chemical composition and mode of preparation. Their characteristics are closer to coatings on the basis of Si–Al–N studied in [8].



Fig. 1. TEM bright field image and selected area diffraction pattern of coating on the basis of Si-Al-N(a) its X-ray pattern (b).

For silica glass with the Si–Al–N coatings and without it the microhardness  $H_m$  was measured, modulus of inelastic buckling E\* and was determined. These data are shown in Table 1. It is seen that microhardness of samples with coatings is higher 3.4 times and elastic recovery factor is greater 1.8 times

compare with samples without coatings. As for modulus of inelastic buckling, its value for samples with coating is greater compare with samples without coatings 3.5 times. After the shelling of glasses by the Fe microparticles the silver thin film (conductive layer) was deposited (10–20 nm) on their surface with craters. Then surface morphology of the samples were studied by SEM. Investigations were carried out in secondary electron mode with magnification 100 at an accelerating voltage of 20 kV. Each sample series of images was photographed and then joint together into a panorama. After joint the area about 100 mm<sup>2</sup> was chosen on each from the 8 samples and measurements were made of transverse size of craters. After that the results for all samples with coatings were tabulated and treated a single array. These data were compared with similar data obtained on 4 of the original glass without coating. Fig. 2 shows an image obtained by SEM, where circles outlined craters appearing after the shelling. It was established that the surface density of craters of glasses with coatings is significantly lower than for glasses without coatings at the same test conditions (Fig. 1). Surface density  $\rho$  for glasses with coating is 0.39·10<sup>6</sup> m<sup>-2</sup> and for glasses without coatings is 1.08·10<sup>6</sup> m<sup>-2</sup>.



Fig. 2. Craters images formed under flux of the iron particles on samples without (a) and with coating (b).

### Conclusions

In work it is shown that obtained by pulsed magnetron sputtering of the composite targets the coatings on the basis of Si–Al–N have two-phase nanostructure. There are crystallites of the AlN phase with hcp crystalline lattice and  $Si_3N_4$  phase having amorphous structure in the form of layers between grains of the AlN phase. Average grain size of AlN is in the range of 5–12 nm. These coatings have high values of microhardness, modulus of elasticity and elastic recovery ratio. Deposition them to the quartz glass can increase its impact resistance against the effects of iron microparticles with an average size of about 56  $\mu$ m, moving with velocities of 3–5 km/s. The obtained data has shown that deposition of coating on the basis of Si–Al–N on quartz glass leads to decrease in surface density of craters 2.7 times at bombard-ment of glass using light gas guns.

#### REFERENCES

- 1. Silverman E.M. Space //Environmental Effects on Spacecraft: LEO Materials Selection Guide, NASA Contractor Report 4661, Part 1, August 1995. pp.116.
- 2. Novikov L.S. // Exposure to particulate matter of natural and artificial origin on the spacecraft. M., 2009.
- 3. Romanchenko V.P., Brovkin A.G., and Lyulin I.B. // Modeling the influence of factors of anthropogenic pollution of near space on the structural elements and systems of the spacecraft. Gidrometeoizdat, 1992.
- Panin V.E., Sergeev V.P, Rizakhanov R.N., Sergeev O.V., Panin S.V. Barmin A.A., Golikov A.N., Pochivalov Yu.I., and Polyansky M.N. // Collection of articles based on the Second International Conference "Deformation and fracture of materials and nanomaterials". – M., 2007. – P. 357.
- 5. Silicon nitride materials and on its basis / eds. R.A. Andrievskii, Spivak I.I. M.: Metallurgy, 1984. P. 136.

- 6. Sergeev V.P., Yanovsky V.P., Paraev Yu.N., et al. // Physical Mesomechanics. 2004. V.7. Spec. issue. Part 2. P. 333-336.
- Sergeev V.P., Fedorischeva M.V., Sungatulin A.R., Nikalin A.J., and Neufeld V. // Proceedings of the TPU. - 2011. - V. 319. - No. 2. - P. 103-108.
- Musil J., Šašek M., Zeman P., Čerstvý R., Heřman D., Han J.G., and Šatava V. // Surface & Coatings Technology. - 2008. - V. 202. - P. 3485-3493.
  - \*Institute of Strength Physics and Materials Science SB RAS, Tomsk, Russia
    \*\*National Research Tomsk Polytechnic University, Tomsk, Russia
    \*\*\*Scientific Research Institute of Applied Mathematics and Mechanics of Tomsk State University, Tomsk, Russia
    E-mail: vserg@mail.tomsknet.ru

Sergeev Victor, Ph.D., professor, head of the laboratory; Kalashnikov Mark, chief engineer; Voronov Andrey, research associate; Bozhko Irina, Ph.D., senior researcher; Ribalko Evgeniya, graduate student; Khristenko Yurii, Ph.D., professor.