Resistance investigation of diamond-like carbon coatings to cyclic temperature changes

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Abstract. Optical elements used in outer space must be designed considering the effects of such factors as space vacuum, atomic oxygen in low Earth orbit, solar and space radiation, large temperature drops, gas release of spacecraft materials and structural elements, space dust and debris. In order to harden and protect mirror surfaces of optical elements from external factors, it has been promisingly applied diamond-like carbon coatings on their surface. These coatings are characterized by high strength and wear-resistant properties, in particular, high hardness, low friction coefficient, high wear resistance and chemical inertness. This leads to their widespread use in various fields of science and technology, including optical instrumentation. This paper presents the results of testing an aluminum mirror with a diamond-like carbon coating under the effect of cyclic temperature changes for determining their ability to withstand a rapid cyclic ambient temperature change, and specifically, to maintain optical and mechanical properties.

Keywords: diamond-like carbon coating, temperature cycling, optical properties, mechanical properties, hardness, reflection coefficient, roughness, shape deviation.

1. Introduction
Optical elements used in outer space must be designed to withstand such factors as space vacuum, atomic oxygen in low Earth orbit, solar and space radiation, large temperature drops, gas release of spacecraft materials and structural elements, space dust and debris [1].

Thermal cycling is one of the main factors affecting the performance of the spacecraft optical elements. The temperature of optical surfaces in space, depending on the field of application, can vary from -150 °C (and even lower in devices for deep space exploration) to several hundred degrees. These temperature fluctuations can cause a shift in spectral lines at lower temperatures, which can be compensated by installing heaters, and also can cause a change in the geometric dimensions of the deposited layers, which will lead to a change in the refractive index. In addition, the difference in the coefficients of thermal expansion of the deposited layers materials on other layers will produce an increase in internal stresses, causing the destruction of the applied coating. In this regard, the choice of deposition technological modes or, if necessary, the application of intermediate subcoatings play a decisive role in ensuring the coating durability [1].

A promising option for protecting reflective mirror surfaces operating in the visible and infrared ranges is the deposition of diamond-like carbon (DLC) coatings on their surface. This type of nanostructured coatings, consisting of carbon atoms with both diamond (sp³) and graphite-like (sp²) bonds, is characterized by high strength and wear-resistant properties, in particular, high hardness from 5000 to 10000 Hv, low friction coefficient from 0.15 up to 0.08, high wear resistance and chemical inertness. This leads to their wide application in various fields of science and technology,
including optical instrumentation [2-5]. At the same time, the refractive index of diamond-like carbon coatings is in the range from 1.55 to 2.20 [6-8], depending on the manufacturing technology, and the absorption coefficient is close to zero in a wide range of IR wavelengths [9-11]. Such unique properties make it possible to improve the reliability indicators (durability and trouble-free operation) of optical elements while maintaining the performance indicators (transmission or reflection coefficients).

This paper presents the results of testing mirrors with a diamond-like carbon coating under the effect of cyclic temperature changes for determining their ability to withstand ambient temperature changes, and specifically, to maintain optical and mechanical properties.

2. Methods
The procedure for testing mirrors with a diamond-like carbon coating under the effect of cyclic temperature changes was developed on the basis of a series of the Russian Federation State Standards (GOST R) [12-14]. The tests were carried out in 3 stages:
  • preliminary measurements;
  • impact of temperature cycling;
  • final measurements.

Preliminary and final measurements included:
  • measurements of light reflectance;
  • measurements of surface roughness;
  • measurements of shape deviations;
  • measurements of hardness and Young's modulus.

The test procedure under the effect of temperature cycling included the following main stages:
  1. The test sample, which has the same temperature as the laboratory air, is immersed in a cold bath with a liquid temperature of -196 °C.
  2. The sample is kept in the cold bath for 18 minutes.
  3. Then, the sample is removed from the cold bath, kept in normal atmospheric conditions for 2-10 minutes, and transferred to the heat chamber.
  4. The heat chamber must be kept at a high temperature of 100 °C for 2 minutes.
  5. During the next cycle, the sample is kept in normal atmospheric conditions for 2-10 minutes and transferred to the cold chamber.
  6. One cycle includes two dwell times and two transfer times.
  7. The sample is exposed to at least three cycles.
  8. At the end of the last cycle, the sample is kept under normal atmospheric test conditions for the time necessary to achieve thermal equilibrium.

An experimental diagnostic stand (MKF-3M) was used as a heat chamber to study the degradation of materials under the influence of the outer space factors. The holding chamber at a low temperature was a Dewar vessel with liquid nitrogen and a boiling point of -196 °C. The test samples were always immersed in liquid.

The testing objects were samples of flat mirrors made of silicon carbide SiC with an aluminum coating deposited on them and a protective diamond-like carbon coating of 50 nm thickness (figure 1). The aluminum layer was deposited 200 nm by magnetron sputtering. The diamond-like carbon film was obtained by laser-triggered cathodic arc deposition and plasma separation [15].
The test sample was subjected to temperature cycling in accordance with the developed methodology. Three cycles were carried out.

The procedure described in GOST R 54164-2010 was taken as the reference for assessing the reflection coefficient. The measurements of the spectral reflectance before and after the tests were performed in the wavelength range from 780 to 1700 nm with a step of 5 nm on a PHOTON RT spectrophotometer.

The procedure for measuring hardness and Young’s modulus is based on GOST R 8.748-2011 (ISO 14577-1: 2002) “Metals and alloys. Measurement of hardness and other characteristics of materials under instrumental indentation. Part 1. Test method”. The procedure consists in introducing a geometrically and physically certified pyramid (Berkovich pyramid with an apex angle of 65.3° and a radius of curvature of 200 nm) into the material and determining with high accuracy the load-deformation relationship (indentation depth). The hardness before and after testing was measured using a Nanovea nanohardness tester according to the Oliver-Pharr method [16].

The roughness was measured in accordance with [17] by the method of coherent correlation interferometry using a TALYSURF CCI 6000 profilometer, which belongs to the type of non-contact optical interference devices. In this case, 10 sectors of the mirror surface with an area of 0.35 × 0.35 mm were selected for measurements.

The measurement of shape deviations of the samples was carried out using a Fizeau interferometer [18], designed to control the shape parameters of flat optical surfaces. The controlled parameter was the standard deviation of the mirror shape from the plane.

3. Results
It was determined by means of Raman spectroscopy that the diamond-like carbon coating of studied sample has sp$^3$ fraction equal 8% (figure 2).
Figure 2. Study results of the DLC coating by the Raman method.

The measurement results of the reflection coefficients before and after thermal tests are shown in figure 3.

The averaged values of the spectral reflection coefficient of the sample $\rho (\lambda)$ in the wavelength range from 780 to 1700 nm, as well as the coating hardness, roughness and shape deviation before and after testing are given in table 1.

The differences obtained in the investigated parameters of the tested mirror are small and commensurate with the instrumental and measurement methods errors.

Figure 3. Light reflectance of the sample before and after thermal tests.
Table 1. Investigated parameters before and after testing.

| Parameter                  | Before test value | After test value |
|----------------------------|-------------------|------------------|
| Integral reflectance, %    | 89.21             | 89.19            |
| Hardness, GPa              | 2.39              | 2.41             |
| Elastic modulus, GPa       | 157               | 181              |
| Roughness Ra, nm           | 2.12              | 1.79             |
| RMS flatness deviation, nm | 26.08             | 25.78            |

4. Conclusions
The tests were carried out on mirrors with a diamond-like carbon coating under the effect of temperature cycling (3 cycles) in the range from -196 to 100 °C. It was also measured the optical (reflection coefficient in the wavelength range from 380 to 1700 nm), mechanical (hardness and elastic modulus) and geometric (roughness and shape deviation) characteristics of the mirror with a diamond-like carbon coating. The results of the investigation allow to conclude that mirrors with a diamond-like carbon coating are suitable to withstand a rapid cyclic change of ambient temperature and to maintain optical and mechanical properties.

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References
[1] Wernham D and Piegari A 2018 Optical coatings in the space environment Optical Thin Films and Coatings ed A Piegari and F Flory (Cambridge: Woodhead Publishing) chapter 22 pp 789–811
[2] Bewilogua K and Hofmann D 2014 Surf. Coat. Technol. 242 pp 214–225
[3] Macleod H 2010 Thin-Film Optical Filters (Boca Raton, Florida: CRC Press) p 782
[4] Osipkov A, Bashkov V, Belyaeva A, Stepanov R, Mironov Y and Galinovsky A 2015 Surface hardening of optic materials by deposition of diamond like carbon coatings from separated plasma of arc discharge Proc. IOP Conf. Ser.: Materials Science and Engineering (St. Asaph) vol 74 p 012013
[5] Batshev V, Kozlov A, Machikhin A, Makeev M, Osipkov A, Bulatov M, Kinhagulov I and Stepanova K 2019 Optics and Spectroscopy 127 pp 634–638
[6] Suasnavas C, Makeev M, Osipkov A, Solano N, Shupenev A and Mikhailov P 2019 Relationship between hardness and optical properties of diamond-like carbon coatings Proc. IOP Conf. Ser.: Materials Science and Engineering (Moscow) vol 675 p 012051
[7] Mironov Y, Stepanov R, Osipkov A, Mironova A, Makeev M, Mikhailov P and Sedih N 2015 Optical and mechanical properties of diamond-like carbon coatings deposited by filtered cathodic vacuum arc deposition Proc. 5th Int. Workshop on Computer Science and Engineering: Information Processing and Control Engineering (Moscow) pp 295–300
[8] Klyui N, Litovchenko V, Lukyanov A, Neselevska L, Osovsiky V, Yaroschuk O and Dolgov L 2006 Ukr. Phys. J. 51 pp 710–714
[9] Sizov F, Klyuj N, Lukyanov A, Savkina R, Smirnov A and Evmenova A 2008 Pis’ma v zhurnal tekhnicheskoy fiziki 34 pp 32–40
[10] Robertson J 2002 Materials Science and Engineering (Amsterdam: Elsevier Science B.V.) pp 129–281

[11] Makeev M, Zhukova E, Mikhailov P, Osipkov A and Mironov Y 2015 Physical chemical and protective properties of the diamond-like carbon coatings synthesized from separated plasma of electric arc Proc. 5th Int. Workshop on Computer Science and Engineering: Information Processing and Control Engineering (Moscow) pp 255–259

[12] IEC 2006 GOST 28209-89 Basic environmental testing procedures part 2: tests. Test N: change of temperature (Moscow: Standartinform)

[13] IEC 2006 GOST 28209-89 Basic environmental testing procedures. Part 2: Tests. Test B. Dry heat (Moscow: Standartinform)

[14] IEC 2006 GOST 28209-89 Basic environmental testing procedures. Part 2: Tests. Test A: Cold (Moscow: Standartinform)

[15] Osipkov A, Makeev M, Mikhailov P, Machikhin A, Batshev V, Shiriaev P and Shishov K 2020 Diamond-Like Carbon Coatings to Protect the Optical Surfaces of Orbital Telescopes from the Outer Space Factors Proc. Int. Conf. on Advances in Astronautical Sciences (Moscow) vol 170 (California: Univelt Inc.) pp 665–670

[16] Oliver W and Pharr W 1992 An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J. Mater. Res. 7 pp 1564–83

[17] 2019 GOST 8.296-2015 State system for ensuring the uniformity of measurements. State verification schedule for instruments measuring the surface roughness parameters Rmax, Rz in the range from 0,001 to 3000 μm and Ra in the range from 0,001 to 750 μm (Moscow: Standartinform)

[18] Malacara D 1985 Opticheskij proizvodstvennyj kontrol' (Moscow: Mashinostroenie) p 400