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AFM ANALYSIS OF GLASSY CARBON COATINGS AFTER AN EXTENDED STAY ON THE INTERNATIONAL SPACE STATION (ISS)

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Abstract

In this paper we present the results of atomic force microscopy (AFM), which is applied to characterize the surface morphology of graphite samples with glassy carbon coatings is developed, stored for 28 months under different conditions. One sample was stored on Earth at room temperature and second one was mounted on the outer side of the International Space Station.

Introduction

In the course of the development of the technique, various methods and technologies are developed and used to prepare sensors for measuring direct (DC) and alternating (AC) electric fields in space plasma. The study and knowledge of the characteristics of materials for creating sensors, including materials for creating Legmuir sensors, presumption the use of technologies for creating materials with certain qualities of the working surfaces of sensitive elements of the sensors.

The main requirement for these materials is determined by the need to increase the accuracy and sensitivity of measurements. The accuracy of the electric field measurements, by the double probe method [1–6], strongly depends on variations, the separation work function for electrons on the working surfaces of the probes, as a result of the working environment characterized by radiation exposure, charged particle flows, sudden changes in temperature, high vacuum, the concentration of the ambient plasma, bombardment of their working surfaces by micrometeorites, vibration and shock loads, etc.

Glassy carbon is a preferred material for making sensors. It is a black glass-like material with a shiny surface and fragility, earning it the name vitreous or glassy carbon. GC is easy to process and can be produced in various shapes, sizes, and cross-sections [7–10]. Electron work function measurements on sample surfaces with glassy carbon (CG) coatings or monolithic glassy carbon coatings show the highest value, which suggests little photoemission and provides minimal point-to-point variation across the surface of the probes [11–13].

Materials and Methods

Sample Types

Graphite samples coated with glassy carbon stored in different environments for a period of two years and four months were analyzed. One of the samples was stored in Earth's environmental conditions, "reference", and the other sample was mounted outside of the International Space Station for a period of more than two years, "space" [14]. In outer space, the samples were exposed to radiation and a two-hour cyclic temperature change in the range of ~ 300°C. The purpose of the experiment was to obtain new data on the influence of space factors on the physico-chemical characteristics of materials used in space research and clarify the aging processes of these materials, as a result of their long stay in the conditions of outer space.

The method of coating graphite with glassy carbon is an original Bulgarian technology developed and implemented by a team of scientists of the Bulgarian Academy of Sciences (IKIT-BAS and IMCTH) and is protected by a copyright certificate [15].

For the studies presented below, samples with dimensions of 15 × 0.5 × 0.3 mm were cut, according to the requirements of the AFM apparatus. Measurements were made on three samples of "reference" R1, R2, and R3 and three samples of "space" S1, S2, and S3. The analyzed surfaces for both types of samples are carried out at five points: end, periphery, center, periphery and end, along the diagonal of the sample for the purpose of reproducibility of the results. The "space" samples were investigated from the front side of the sample - direct contact with outer space, and from the back side of the sample from the side of the block in which they were located [1].

Characterization methods

AFM imaging was performed on the Nano Scope V system (Bruker Ltd, Germany) operating in tapping mode in the air at a room temperature. We used silicon cantilevers (Tap 300Al-G, Budget Sensors, Innovative solutions Ltd. Bulgaria) with 30 nm thick aluminum reflex coatings. According to the producer's specifications, the cantilever spring constant and the resonance frequency are in the range of 1.5 to 15 N/m and 150 ± 75 kHz, respectively. The radius of tip curvature was less than 10 nm. The scanning rate was set at 1 Hz and the images were taken in the highest possible resolution mode of the AFM 512 × 512 pixels in JEPG format. The NanoScope software was used for the section analysis and roughness of all images.

Results and discussion

Atomic force microscopy (AFM) is widely used to study the topography of materials with great accuracy and precision. By measuring the surface of the material at the nano level, the roughness of the investigated sample is quantified.

The topography of the investigated samples “space” - face, “space” - back and “reference” are shown in Fig. 1 and Fig. 2 in 2D and 3D format at a single point of the performed analysis with a scan area of 5 µm × 5 µm. The morphology of the “space” – face, “space” – back samples have a smoother surface in comparison with the morphology of the “reference” samples with the same scan area (Fig. 1 and Fig. 2). In the AFM images of the “reference” samples, structures with different diameters of the order between 0.5 µm and 1 µm are observed. The surface of the three graphite samples coated with glassy carbon “space” samples – back side S1(*BS*), S2(*BS*), S3(*BS*) is smoother in comparison to the surface of “space” samples – front S1(*F*), S2(*F*), S3(*F*). The difference in R_q values for the two types of samples is 0.34 nm for the second S2, 1.56 nm for the first S1 and 1.84 nm for the third S3 samples, respectively. These differences in the roughness values of the material on the front and back sides are due to the influence of sudden temperature changes, radiation, meteors, etc. in space.

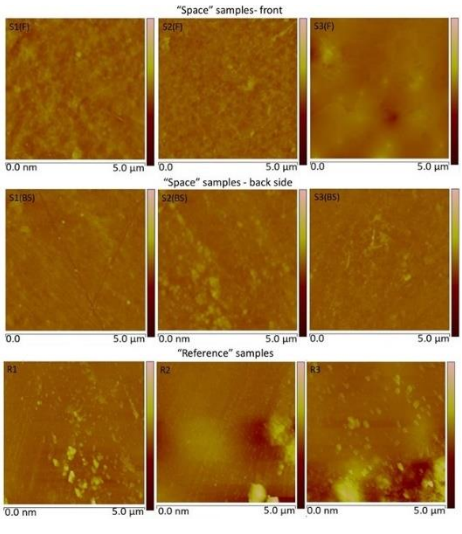

Fig. 1. 2D AFM images 5 µm × 5 µm of the surface of the three graphite samples coated with glassy carbon – “space” samples – front (S1(F), S2(F), S3(F)), “space” samples – back side (S1(BS), S2(BS), S3(BS)), and “reference” samples (R1, R2, R3)

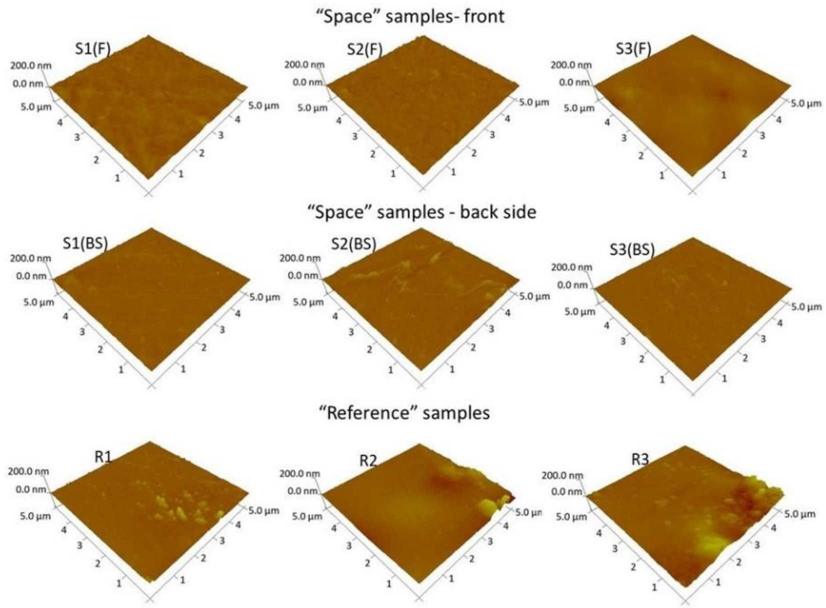

Fig. 2. 3D AFM images 5 µm × 5 µm of the surface of the three graphite samples coated with glassy carbon – "space" samples – front (S1(F), S2(F), S3(F)), "space" samples – back side (S1(BS), S2(BS), S3(BS)), and "reference" samples (R1, R2, R3)

The roughness analysis gives the value R_a which is the arithmetic mean of the absolute values Z_i of the surface height deviations measured from the mean plane, i.e.

(1) $$R_a = \frac{1}{N}\sum_{i=1}^{N}|Z_i|$$

while R_q is the root-mean-square value of the height deviations taken from the plane of the average images date [16].

(2) $$R_q = \sqrt{\frac{1}{N}\sum_{i=1}^{N} Z_i^2}$$

Fig. 3 presents the average values obtained from the five analysis points for each of the investigated samples for R_a and R_q.

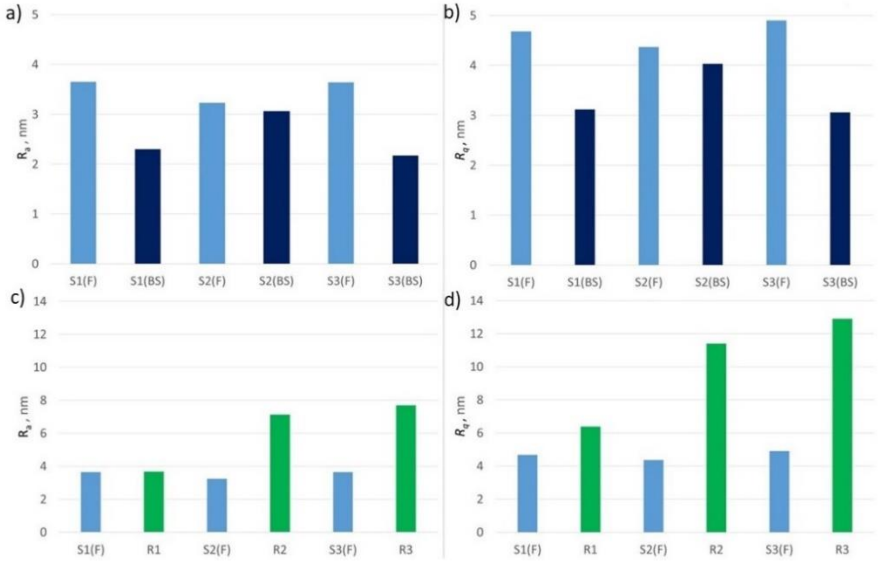

Fig. 3. Roughness analysis of AFM images 5 µm × 5 µm for the three graphite samples coated with glassy carbon – a) R_a for "space" samples – front and "space" samples – back side; b) R_q for "space" samples – front and "space" samples – back side; c) R_a for "space" samples – front and "reference" samples; d) R_q for "space" samples – front and "reference" samples

The morphology and roughness of graphite samples with glassy carbon coatings were compared and presented in Table 1 and Fig. 3. The "space" samples - face "space" samples - front (S1(F), S2(F), S3(F)) have a rougher surface for the values of R_a and R_q compared to the surface of "space" samples - back side (S1(BS), S2(BS), S3(BS)).

The differences in the roughness value for R_a of S1(F) versus S1(BS) is 3.65 nm to 2.3 nm and for the R_q value - 4.68 nm to 3.12 nm, respectively. For sample S2, the differences in the roughness value for R_a of S2(F) versus S2(BS) are smaller, 3.23 nm to 3.06 nm, respectively, and for the value for R_q – 4.37 nm to 4.03 nm, respectively. The difference in the roughness value for R_a of sample S3(F) is approximated as sample S1(F). The roughness value for R_a of S1(F) vs. S1(BS) is respectively 3.64 nm to 2.17 nm and for the value of R_q – 4.90 nm to 3.06 nm, respectively for "space" – face, "space" – back. The roughness values for R_a and R_q of "reference" samples R-1, R-2, and R-3 are higher than those of "space" samples - face and "space" samples - back side.

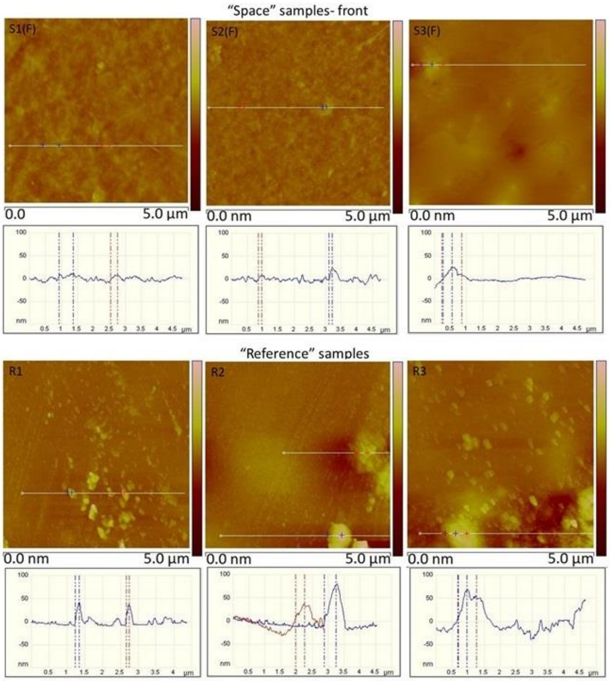

Fig. 4. 2D AFM images and section analysis of the surface of "space" samples – front and "reference" samples

Atomic force microscope data provide valuable information on the homogeneity of the glassy carbon layer for "space" samples. The AFM images of both types of "reference" and "space" samples were compared and presented in Fig. 4. The morphology of the "reference" samples with a scanning area of 5 × 5 μm^2 (R-1, R-2, R-3) is smooth with the presence of places with "spherical" structures in comparison with the morphology of the "space" samples –front (S1(F), S2(F), S3(F)). From the cross-section and surface roughness determined for both the "reference" and "space" samples, it was found that the "reference" samples have a rougher surface.

Conclusions

The surface topography of the glassy carbon coatings was studied using Atomic force microscopy. The surface morphology of the surface of graphite samples with glassy carbon coatings, stored for 28 months in Cosmos "space" - face, "space" - back is smooth and homogeneous. The surface morphology of graphite samples with glassy carbon coatings stored for 28 months on Earth at room temperature "reference" is rougher with the presence of structures with diameters from 0.5 μm to 1.5 μm. The roughness values for Ra and Rq of “reference” samples R-1, R-2, and R-3 are higher than those of “space” samples - face S1(F), S2(F), S3(F). The presence of “spherical” structures on the surface of the “reference” samples is a result of the storage conditions on Earth. The surface of the deposited glassy carbon coatings on a graphite substrate of both types of samples – “reference” and “space” is homogeneous with small differences in roughness values.

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АФМ АНАЛИЗ НА СТЪКЛОВЪГЛЕРОДНИ ПОКРИТИЯ СЛЕД ПРОДЪЛЖИТЕЛЕН ПРЕСТОЙ НА МЕЖДУНАРОДНАТА КОСМИЧЕСКА СТАНЦИЯ (МКС)

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Резюме

В тази статия представяме резултатите от охарактеризирането на повърхностна морфология с Атомно-силовата микроскопия (АФМ) на графитни проби със стъкловъглеродни покрития, съхранявани в продължение на 28 месеца при различни условия. Едни проби бяха съхранявани на Земята при стайна температура, а вторите бяха монтирани от външната страна на Международната космическа станция.