

XPS INVESTIGATION OF THE WORK FUNCTION OF GLASSY CARBON COATINGS AFTER EXTENDED STAY ON THE INTERNATIONAL SPACE STATION (ISS)

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Abstract

Using XPS, the electron work function values on the surface of glassy carbon coatings of graphite samples, after an extended stay on a board of the ISS in open space conditions, were investigated. The results were compared with the characteristics of glassy carbon coatings of samples left on the ground for the same period. It was found that the electron work function does not change significantly, and this proves the possibilities for the successful application of these coatings, obtained by original Bulgarian technology, for space experiments on the board of satellites for measuring electric fields in the ionospheric-magnetospheric plasma. The minimal observed variations in the values of the electron work function are explained by small differences in the content of traces of different chemical elements on the surface of the coatings. It has been established that glassy carbon coatings have stable characteristics after a long stay in space, despite the small fluctuations in the values of the electron work function. The results show that glassy carbon coatings are chemically and mechanically stable. The results from this original technological experiment are unique for the development of sensitive elements, such as sensors for measuring weak electric fields in cosmic plasma.

1. Introduction

From the beginning of the space age until today, the double probe method [1–3] has been established as a method that measures the electrical potential difference between two points in space. Variations of the electron work function on

the surfaces of each probe affect the current value in a complex way, especially in the case of inhomogeneous plasmas such as ionospheric-magnetospheric plasmas, and are particularly important for experiments aboard rotating satellites. Differences in the floating potentials for the individual probes, relative to the surrounding plasma, can lead to additional electrical signals, many times exceeding the investigated ones. This fact determines the need for maximum identity of the physico-chemical and geometrical characteristics of the Sensitive Elements of the Probes (SPE). Obviously, a central factor that determines the stable performance of the SPE is the material choice and its surface properties.

During the development of the signal acquisition methods of constant and variable fields, various probe materials have been considered in order to gain better sensitivity and higher signal accuracy registration. The accuracy of electric field measurements, by the double probe method, strongly depends on the variations of the Work Function (WF) of the electrons on the probe surface due to the solar wind (irradiation) in outer space. The electron WF, on the surfaces of samples with Glassy-Carbon (GC) coatings, shows the highest value, about 5.05eV, which suggests lower photoemission compared to the other used materials such as silver, gold, aquadag, etc. Due to these properties of GC materials, spherical probes (Langmuir probes) made of GC have been used in the last three decades in practically all experiments for measuring electric fields. Spherical sensors made of monolithic GC have also been used in other satellite experiments such as GEOS, S3-3, ISEE-1,2, WIND, POLAR, CLUSTER – 1,2,3, 4, FREJA, THEMIS, CRRES, GEOS, Viking, and GEOTAIL [4-9].

An original method has been established [10] for GC synthesis on pyrolytic graphite, a technology that meets severe test requirements, and double SPE, has been installed on satellites, such as IC-Bulgaria 1300, IC-24 Active, IC-25 APEX, INTERBALL-2, the Magion-2-5 sub satellites, as well as the International Space Station (ISS) [10], for the period from 1981 to 2023. In order to study the influence of outer space on the physico-chemical parameters of GC coatings, in the period 2013–2015, an international project “Obstanovka 1-step” was planned and successfully implemented, which included the implementation of a technological experiment block “DP-PM” aboard the International Space Station (ISS) [10].

2. Materials and Methods

Sample Types

The analysed samples with dimensions of $30 \times 15 \times 5$ mm of spectrally pure graphite, compacted and covered with GC, were analyzed and they stayed at different conditions for 28 months. Some samples have been in open space mounted on the Russian module of the ISS [10] and conventionally were called “space”, while the others have been stored in terrestrial conditions and named as “reference”. To achieve high reliability in the analysis of the obtained data for the “space” samples,

they are compared with “reference”. The purpose of the experiment is to investigate the influence of outer space on the physico-chemical and structural characteristics of the GC coatings, after their long stay in the space. The most important characteristic for the stability and durability requirements of these coatings, we used the value and WF variations of the electrons on its surface. In open space, samples were directly exposed to various impacts on the front surface of the sample, which we conventionally call *S-front*, and those that were not exposed to direct contact, *S-back side*.

All samples were analyzed by surface sensitive technique – X-Ray Photoelectron Spectroscopy (XPS). Later the surface of as prepared samples is labeled as “ras”, the samples cleaned with isopropyl alcohol are labeled as “rclean”, as well as scribed surface, using a P2500 silicon carbide sandpaper, in order to get results more related to the bulk, labelled “rscribed”. The cleaning of the samples was done using an ultrasonic bath for 5 min in order to remove the fats and contamination residuals after sample preparation.

Characterization methods

X-ray photoelectron measurements have been carried out on the ESCALAB MkII (VG Scientific, now Thermo-Scientific) electron spectrometer with a base pressure in the analysis chamber of 5.10^{-10} mbar (9.10^{-8} mbar during the measurements), equipped with twin anode MgK α /AlK α non-monochromatic X-ray source used excitation energies of 1253.6 and 1486.6 eV, respectively. The measurements are provided only with an AlK α non-monochromatic X-ray source (1486.6 eV). The instrumental resolution is measured as the full width at a half maximum (FWHM) of the Ag3d $_{5/2}$, photoelectron peak is about 1 eV. The data was analyzed using SpecsLab2, Casa-XPS (Casa Software Ltd), and Origin 11 software. The processing of the measured spectra includes subtraction of X-ray satellites and a Shirley-type background [11]. The peak positions and areas are evaluated by a symmetrical Gaussian-Lorentzian curve fitting [11]. The relative concentrations of the different chemical species are determined based on the normalization of the peak areas to their photoionization cross-sections, calculated by Scofield [12].

Work function determination using the XPS technique

One of the most reliable and widely used spectroscopic techniques for surface analysis is X-ray Photoelectron Spectroscopy (XPS). It is a surface-sensitive, non-destructive, and element-specific technique that provides quantitative information on the chemical state of the elements of the top 10 atomic layers of the surface of the analyzed sample. Moreover, it gives information about the chemical state of the analyzed elements, which is based on the change of the binding energy and the line shape. Furthermore, XPS can easily be adapted to measure the electron

work function of the surfaces without any additional equipment modification or additional sampling procedure. That is because the equipment possesses a well-developed high-resolution electron analyzer, usually with a hemispherical shape and a well-defined X-ray source. However, there are some requirements that have to be taken into account. Samples need to be stable inorganic materials that are resistant to X-rays. It is generally necessary to bias the sample with a small potential voltage (e.g. - 5 V) in order to avoid effects that are a result of scattered electrons in the spectrometer's detection system. Therefore, the WF of insulating/organic samples is difficult to measure by XPS . The electron Work-Function (WF) is dependent very much on the surface condition; even minor modification to the surface brings a dramatic change in its value. The measured value strongly depends on the surface impurities and composition homogeneity. Even after cleaning the surface under ultrahigh vacuum (UHV) condition, the WF changes are often observed over time due to the contamination from residual gases within the chamber. Figure 1 illustrates the procedure of WF measurement for GC probes. The first calibration of the spectrometer is performed by the C1s element at 284.5 eV, and hence, the Fermi level (E_F) could be established. At that point, we only need to define the secondary electron cut-off position. Then, the WF is defined as the difference between X-ray excitation energy and secondary electron cut-off energy.

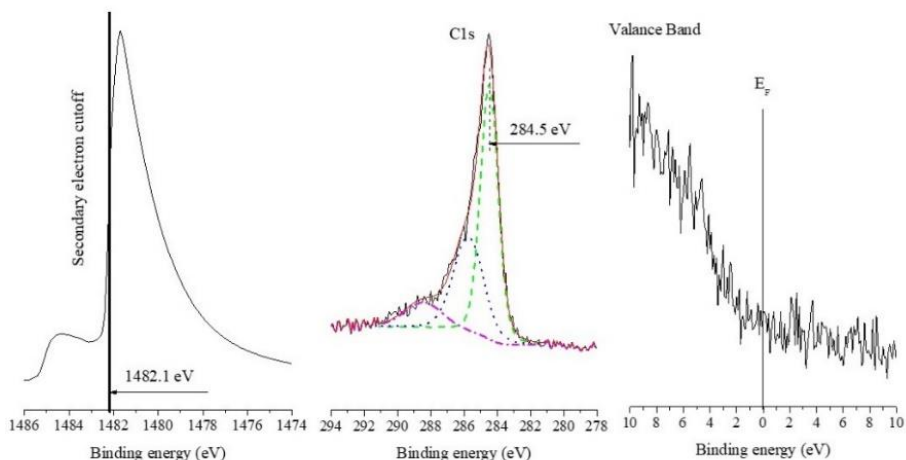


Fig. 1. WF measurement of GC material using spectrometer by XPS

3. Results and Discussion

In our approach, we use the method of WF measurement, generally explained by Kim & Kim [13], where the total photoelectron spectrum is displayed in the range between X-ray energy and the sample value of WF. Practically, two

values have to be obtained in order to calculate WF, namely, secondary electron cut-off and the maximum kinetic energy of photoelectrons emitted from a metal, defined as Fermi level (EF), which appears at 0 eV binding energy in the XP spectrum (see Fig. 1). Thus, the WF is obtained by the difference between the of X-ray photons energy (1486.6 eV) and the secondary electron cut-off energy. One can conclude that these energy values, secondary electron cut-off, and Fermi level EF at 0 eV, have to be measured very precisely. Therefore, calibration of the spectrometer by measuring noble metal surface after its cleaning, e.g. palladium, is crucial. The performed measurement of the well-defined surface of the Pd-metal gave a result of 5.2 eV, as expected, equal to the book values (5.22 eV) of Pd-metal WF. In this way, our measurements of glassy carbon surfaces can be considered reliable. Moreover, we have used additional parameter which has been followed to assure the calibration of the binding energy scale of spectra, namely C1s core level. Regardless of the presence of impurities and contaminations, we are investigating surfaces that consist basically of glassy carbon. Therefore, we can assume that the carbon 1s core level will have predominantly binding energy at 284.5 eV [14]. This gave us a chance to lower the error by defining EF = 0 eV of the investigated surface, which is challenging because of the low intensity and noisy spectrum of the valence band. Additionally, to determine it more precisely, the slope of the valence band was modeled using a high-degree polynomial and then the model curve was differentiated. The center of the resulting peak coincides with the Fermi level. Figure 1 demonstrates the shapes of the corresponding measured peaks of Secondary electron cutoff, C1s glassy carbon core level with additional sub-peaks resulting from C - O and C = O bonds impurities and contaminations, as well as the valence band slope and Fermi level.

Several surfaces of the sample were measured: various contamination has been found on the as prepared surface, labeled as (ras); surfaced cleaned with isopropyl alcohol ultrasonic bath for 5 min. in order to remove fats and contamination residuals after sample preparation (rclean); as well as scribed surface, using a P2500 silicon carbide sandpaper, in order to get results more related to the bulk (rscribed). The ras surface shows various discolorations on different points. Therefore, we performed XPS measurements on several points on the surface to study this discoloration.

The results summarized in Table 3 show differences in the concentration of elements presented on the surface as well as variations in the composition. As a consequence, we can expect different WF values for these surfaces. Indeed, values between 3.3 - 4.8 eV of WF have been obtained for the sample ras and rclean for all studied points. For the scribed GC sample, we have observed a relatively homogeneous surface with one discoloration and fewer impurities. Impurities on the glassy carbon surfaces like silicone, oxide, nitrogen, chlorine, and others have also been reported by other authors [15–17]. Close or even the same values are obtained by other authors published in scientific literature, for instance, by Ivey [18].

As expected, we obtained WF with close values for all the measured points from 4.3 to 4.6 eV. Small deviations of the WF values are observed for the exposed samples (S1, S2), rather than for the “reference” one (R). These results are summarized in Table 1, where S1-2 and S2-2 are noted as repeated measurements on samples S1 and S2. As expected, we obtained close WF values of 4.8 eV and 5.0 eV, respectively. These values are equal within the error bars and fully consistent with the close predominant carbon and oxygen atom concentrations of both surfaces.

For the “space” sample S2, the electron work function value for the face is 4.8 eV and the work function value for the back is 5.0 eV. These values are again very close and fully consistent with the close oxygen concentrations on the two surfaces. All discussed results are graphically expressed in Fig. 2 and Fig. 3, and the results are shown in Table 1.

Table 1. Fermi level and work-function of referent and exposed (space) samples

Sample:	Side	Fermi Level [eV]	HBE* [eV]	Work Function [eV]
R	face	-28.2	1454.0	4.8
	back	-29.6	1452.6	4.8
S1	face	-31.1	1451.1	4.4
	back	-30.4	1451.4	4.8
S2	face	-30.9	1450.3	5.4
	back	-30.2	1450.8	5.6
S1-2	face	-30.0	1451.4	5.2
	back	-29.5	1451.9	5.2
S2-2	face	-30.9	1450.9	4.8
	back	-30.8	1450.8	5.0

A standard sample of monolithic glassy carbon, protected by copyright [20], shows a very homogeneous surface with only one surface colour - dark grey. Nevertheless, the XPS study also detects some impurities on the surface of this sample. The surface atomic concentrations of the monolithic glassy carbon sample [20] are summarized in Table 2. The solutions used for the preparation of glassy carbon can be the origin of these impurities. The obtained WF of this sample is 4.5 eV, which is equal, within the error bar, to the value reported in the scientific literature [16–18].

Table 2. Surface atomic concentration of homogeneous monolithic glassy carbon [20]

Sample	C	O	N	Si	S	Ca	Cl	Al
	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]
<i>f</i>	79.10	14.03	2.57	0.78	0.82	0.85	0.31	1.55

Table 3. Surface atomic concentrations obtained for “ras”, “rclean”, and “rscribed” surfaces of the “reference” GC samples

Sample	C	O	N	Na	Zn	Si	S	Ca	Cl	F	Al	Mg
	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]	[at.%]
<i>ras</i> Point 1	85.40	11.20	2.70	0.80	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
<i>ras</i> Point 2	80.70	13.30	2.80	0.70	0.20	1.50	0.80	0.00	0.00	0.00	0.00	0.00
<i>rclean</i> Point 1	71.25	19.15	2.94	0.22	0.00	2.87	0.00	0.95	0.36	0.66	1.60	0.00
<i>rclean</i> Point 2	69.41	18.61	3.39	0.34	0.16	3.76	0.00	0.99	0.00	0.61	2.51	0.23
<i>rclean</i> Point 3	67.36	18.79	2.92	0.63	0.49	4.46	0.00	1.78	0.00	1.17	2.16	0.23
<i>rclean</i> Point 4	79.52	14.38	2.17	0.00	0.00	2.30	0.00	1.34	0.00	0.00	0.29	0.00
<i>rclean</i> Point 5	89.68	7.98	1.70	0.00	0.00	0.00	0.00	0.63	0.00	0.00	0.00	0.00
<i>rscribed</i> Point 1	93.20	4.51	0.00	0.00	0.00	1.90	0.00	0.00	0.00	0.39	0.00	0.00
<i>rscribed</i> Point 2	92.04	6.23	0.00	0.00	0.00	1.42	0.00	0.00	0.00	0.30	0.00	0.00
<i>rscribed</i> Point 3	93.15	4.71	0.00	0.00	0.00	1.70	0.00	0.00	0.00	0.45	0.00	0.00

Furthermore, we adopt the following notations for the studied samples: for the “reference” samples, face and back – R-front and R-back side and for the “space” samples, S1-front and S1-back side; S2-front and S2-back side, respectively. One can expect that there will be differences between both sides of the sample, namely, the side that is exposed directly to cosmic radiation (front) and the sample backside (back side). Therefore, both sides are studied by means of the XPS technique. The calculated WF values for sample S1-front and S1-back side are 4.4 eV and 4.8 eV, respectively. The small variation of these WF values can be explained by a significant difference measured for oxygen from 13 at. % to 7.6 at. % and presence of silicon, sulfur, and calcium atoms on the surface only on the S1-front. The sample S2 shows similar results with some minor variation in the surface atomic concentrations compared to both the S2-front and the S2-back side surfaces. The surface atomic concentrations of both samples – “reference” and “space” are displayed in Table 3.

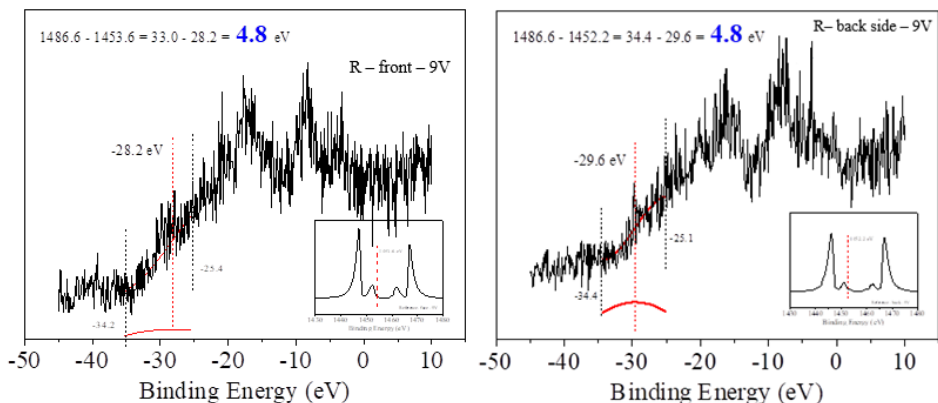


Fig. 2. XPS valence band spectrum for the “reference” samples R-front (a) and R-back side (b)

Summarizing and comparing the results for the WF regarding the “reference” samples R- front and R-back side of the “space” S1 and S2, we can conclude that the values between 4.4 and 4.8 are close-range values, and their insignificant difference lies in the impurities and contaminations of the surfaces. In this sense, and due to the importance of material WF used as a major parameter in the design of devices, we can conclude that the investigated glassy carbon can be used as a stable, sensitive element in electronic detectors.

In Table 3, one of the main components of the near-surface layers is the oxygen, whose concentration ranges over a wide range from approximately 8 at. % to about 15 at.%. Many other chemical elements are present on the surface, among which, the main ones, are nitrogen, sodium, silicon, fluorine, and aluminum. In the reference sample, there is also, although insignificant, a difference between the amount of oxygen on the face and the back of the sample - on the face, it is 11.2 at. %, while on the back, it is 13.3 at. %, i.e., the difference is about 2 at. %. At the same time, the concentration of nitrogen and sodium is approximately equivalent on both sides. Unlike the face, silicon and minor amounts of sulfur were found on the back of this specimen. Much more significant is the difference in oxygen content between the face and the back of sample *S1*. On the face of the sample, the oxygen coverage of this sample is 13 at. %, while on the back, it is 7.6 at.%.

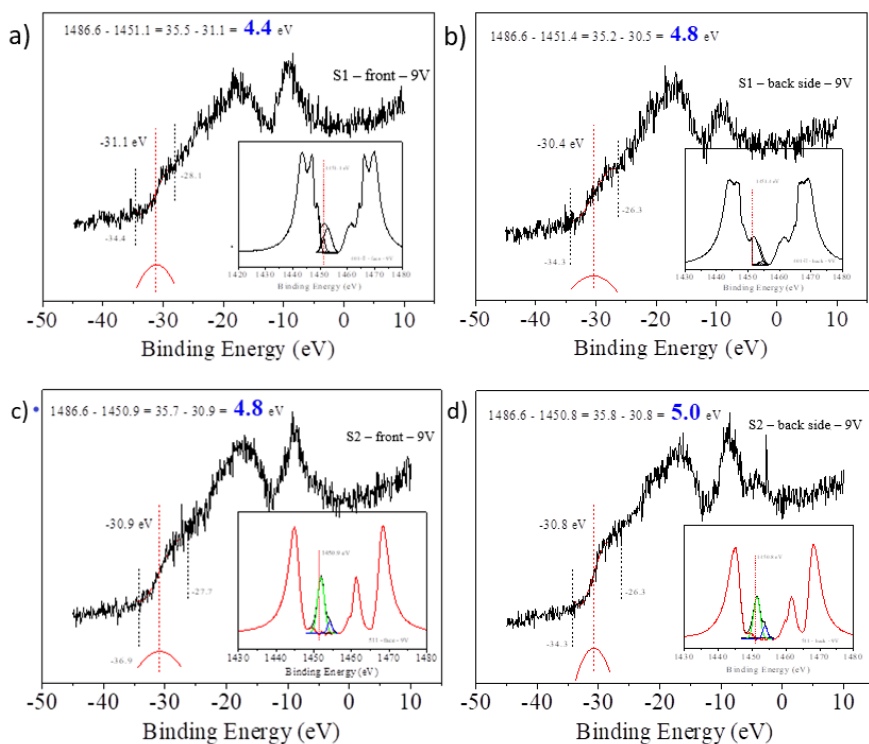


Fig. 3. XPS valence band spectrum for samples S1-front (a) and S1-back side (b) and S2-front (c) and S2-back side (d)

To verify the reproducibility of the data from the experiments, a second measurement of the chemical element concentration of the GC coating on the face and back was made for this sample. The results displayed in Table 3 show that the reproducibility of the measurements is very good. The oxygen concentration in sample S2 is also a bit different. On the face, the oxygen is 9.7 at. %, while on the back, it is significantly less – 10.8 at.%. What is interesting about this specimen is that significant amounts of silicon (Si) were also observed on the face, more so than the other specimens. On this sample, XPS analysis was performed at various points, on the face and back, with the size of each analyzed spot being $700 \mu\text{m} \times 300 \mu\text{m}$.

The analyses that were conducted show that the chemical elements do not significantly differ at different points of measurement. Most of the detected chemical elements on the sample surfaces on both sides have an accidental origin and lead to less to slow influence on the required values of the electron work function. The latter is also due to the spherical forms of the sensor element.

Table 3. Surface atomic concentrations obtained by XPS for samples R, S1, and S2

Sample	C [at.%]	O [at.%]	N [at.%]	Na [at.%]	Zn [at.%]	Si [at.%]	S [at.%]	Ca [at.%]	Al [at.%]	Ni [at.%]
<i>R-front</i>	85.4	11.2	2.7	0.8	0.0	0.0	0.0	0.0	0.0	0.0
<i>R-back</i>	80.7	13.3	2.8	0.7	0.2	1.5	0.8	0.0	0.0	0.0
<i>S1-front</i>	84.30	13.0	0.80	0.20	0.10	0.90	0.70	0.10	0.00	0.00
<i>S1-back</i>	90.70	7.60	1.00	0.60	0.20	0.00	0.00	0.00	0.00	0.00
<i>S2-front</i>	87.60	9.70	1.40	0.20	0.10	0.40	0.00	0.60	0.00	0.00
<i>S2-back</i>	85.70	10.8	0.80	0.30	0.10	0.00	0.00	0.50	1.20	0.20

Summarizing and comparing the results for the WF (table 1) regarding the “reference” samples R- front and R-back side of the “space” S1 and S2, we can conclude that the values between 4.4 and 4.8 are close-range values and their insignificant difference lays on the impurities and contaminations of the surfaces. In this sense, and due to the importance of material WF used as a major parameter in the design of devices, we can conclude that the investigated glassy carbon can be used as a stable, sensitive element in electronic detectors.

4. Conclusions

Detailed investigations on the electron work-function variations due to ionospheric-magnetospheric plasmas field have been carried out. The tested materials were glassy-carbon coatings obtained by the original method. A specific approach has been used to calculate these values for exposed and non-exposed samples. All measurements state similar values of the WF being between 4.4 eV and 4.8 eV. The discrepancy suggests that the GC coatings are stable against aging processes induced by energetic irradiations, which generally lead to severe crystalline defects. Moreover, the surface contaminations do not lead to noticeable WF fluctuations. Probes of glassy-carbon performed as chemically and mechanically stable and sensitive elements are used as double probes for satellite electric field measurements. The higher native values of the WF up to some 5 eV are a prerequisite for lower photoemission. The latter phenomenon increases the coating's electrical conductivity, which results in higher sensitivity to low-intensity electromagnetic fields.

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XPS ИЗСЛЕДВАНЕ НА ОТДЕЛИТЕЛНАТА РАБОТА НА СЪГЛОВОГЛЕРОДНИ ПОКРИТИЯ СЛЕД ПРОДЪЛЖИТЕЛЕН ПРЕСТОЙ НА МЕЖДУНАРОДНАТА КОСМИЧЕСКА СТАНЦИЯ (МКС)

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Резюме

С помощта на XPS са изследвани стойностите на отделителната работа на повърхността на съгловоглеродни покрития върху образци след продължителен престой на борда на МКС в условията на открития Космос. Резултатите са сравнени с характеристиките на съгловоглеродни покрития върху образци, престояли на Земята за същия период. Установено е, че отделителната работа не се променя съществено, и това доказва и потвърждава възможностите за успешно прилагане на тези съгловоглеродни покрития, получени по оригинална българска технология, за космически експерименти на борда на спътници за измерване на електрични полета в йоносферно-магнитосферната плазма. Минималните вариации на стойностите на отделителната работа се обяснява с малка разлика в съдържанието на следи от различни химични елементи на повърхността на покритията. Установено е, че съгловоглеродните покритията са със стабилни характеристики след дълъг престой в Космоса, въпреки малките колебания в стойностите на отделителната работа на електрона. Резултатите показват, че съгловоглеродните покрития са химически и механически стабилни. Получените резултати на този оригинален технологичен експеримент са уникални за създаване на чувствителни елементи – сензори за измерване на слаби електрични полета в околземната плазма.