Influence of Hydrogen onto Work Surfaces of Discharge Chamber of SPT MAG

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Abstract: The investigation of the work surfaces of the discharge chamber insulators has been carried out for SPT MAG-A2 (belonging to Hall Thrusters family) under its operation at the pure xenon and under the "threshold" values of hydrogen content in the vacuum chamber. The "threshold" values of the hydrogen content in the vacuum chamber (under which the sharp performances degradation begins) have been determined under the additional H_2 (or butane) feeding into the vacuum chamber and carefully monitoring the change of SPT integral characteristics at the different hydrogen partial pressures accordingly. Using the method of X-ray photoelectron spectroscopy (XPS) it has been shown the near-surface layer of the channel insulator with the thickness no more than 10nm is responsible for the change in the plasma accelerator behavior. The change of the distribution of the carbon and boron atoms in valence states takes place in this layer under the influence of the hydrogen. Using the spectrometry of recoil nucleus outgoing forward (FRS method), it has been obtained, the hydrogen percentage for the so named "dark" zone of the insulator is three times as much than for the "light" one (where the energetic bombardment of the surface by the xenon ions took place) under the butane feeding. These

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facts are the direct confirmation of the influence of hydrogen onto work surfaces of SPT discharge chamber.

Nomenclature

U_d = discharge voltage
$\dot{m}_a$ = mass flow rate through the anode
$\dot{V}_a$ = volume flow rate through the anode
$\dot{m}_{cath.}$ = mass flow rate through the cathode
$\dot{V}_{cath.}$ = volume flow rate through the cathode
$\dot{V}$ = volume flow rate

I. Introduction

As a result of numerous investigations of Hall Thrusters at different stands [1-5] it has been revealed their integral parameters essentially depends on tests conditions, i.e. on dimensions of the vacuum chamber and its material, the vacuum composition, tests time duration, the method of the mode reaching, etc. This problem has revealed itself the most strikingly under the researching of Hall Thrusters of the second generation of ATON type [1], where, due to the flow separation from the channel walls, the ion bombardment of the acceleration channel walls is essentially weakened and consequently the cleaning of work surfaces by the accelerated ion beam is weakened too. Just this circumstance has allowed for the first time to stand a question about the adequacy of the tests conditions to the physical processes in SPT [2-5].

In order to explain the difference of ATON integral parameters, obtained for the different vacuum conditions, in 1993y. (MIREA) the assumption about the influence of the hydrogen onto the state of SPT work surfaces (due to the bad pumping of hydrogen by cryogenic pumps) has been expressed [3,6]. Later in MIREA it has been experimentally shown, small admixtures (4 volume percents) of hydrogen into xenon derange the modes of ATON operation with high integral characteristics [6].

After "FAKEL" cryostand was equipped by cryopanels, providing the reliable pumping of hydrogen, already in 2000-th years high integral characteristics of SPT MAG type, obtained under the diffusion pumping at MIREA stands, have been confirmed for cryovacuum conditions [7-9].

Being in the frames of the conception of the surface-dominant discharge in SPT [3] we have to assume, the state of work surfaces of SPT discharge chamber, properties of films arising on them and their composition are one of the essential factors under the formation of the physical processes in the plasma flow.

The direct confirmation of the influence of hydrogen onto work surfaces of SPT discharge chamber (and, as a result, onto its integral parameters) has been the purpose of the given work.

II. Statement of Experiment

Quadrupole gas mass-analyzer e-Vision$^+$ (RGA) has been used for the monitoring of the vacuum spectral composition in the chamber (i. e. for the determination of partial pressures of all its components, especially – of hydrogen) over all duration of SPT tests (before its start, during the start and during the operation in the stationary mode). The given device has allowed to work in the mass band (1÷200) a. m. u. (atomic mass units). The range of working pressures of the investigated vacuum has been equal to ($2\cdot10^{-11}\div5\cdot10^{-4}$) Torr (the band $1\cdot10^{-4}\div5\cdot10^{-4}$ is nonlinear part).

Providing the additional H_2 (or butane) feeding into the vacuum chamber and carefully monitoring the change of SPT integral characteristics at the different hydrogen partial pressures accordingly, so named "threshold" values of the hydrogen content in the vacuum chamber (under which the sharp performances degradation begins) have been determined [10]. The feeding of hydrogen and gaseous butane C_4H_8 into the vacuum chamber has been realized using the Digital Mass Flow Controller of MKS firm.

The investigation of the work surfaces of the discharge chamber insulators has been carried out for SPT MAG-A2 [11] under its operation at the pure xenon and under the "threshold" values of hydrogen content in the vacuum chamber [10]. The method of X-ray photoelectron spectroscopy (XPS) [12] and the Rutherford backscattering

spectrometry (RBS) [13] have been used for the researching of the work surfaces. The hydrogen content inside the near-surface layer has been investigated by the spectrometry of recoil nucleus outgoing forward (FRS method) [13] because all other methods are not sensitive to hydrogen.

In order to carry out the experiments on the study of the internal plasma receiving surfaces of the accelerator the cell (of the width 4mm and of the depth 1mm) has been made in the model external dielectric channel, in which the samples-substrates have been placed. All samples had the same dimensions 12mm x 4mm x 1mm. They have placed in the cell by such way in order to cover the field along the channel which is of the most interest from the point of view of taking place physical processes: (7÷19)mm from the anode.

All samples-substrates have been made from the same piece of ceramics BGP-10, having worked as SPT accelerative channel. Thus it went off through the total procedure of annealing in the plasma flow. Before installing into the vacuum chamber the ceramic surface has been subjected to the mechanical cleaning with the help of Waterproof Abrasive Paper (GRIT #240), as a result of which the surface layer of the width ~1mm has been removed.

The description of the samples tests conditions are represented in the Table 1.

Table 1

Sample (serial number)	Operation mode on xenon	Additional H_2 or butane feeding into the vacuum chamber	SPT operation time
№1, №9	-	-	-
№2, №8	$\dot{m}_a = 2.0mg/s$ ($\dot{V}_a = 0{,}38cm^3/s$) $\dot{m}_{cath.} = 0.4mg/s$ ($\dot{V}_{cath.} = 0{,}076cm^3/s$) U_d=350V	-	3 hours
№3	The same	-	10 hours
№4, №10	The same	Hydrogen, $\dot{V}_{H_2} = 0{,}25cm^3/s$	3 hours
№5	The same	Gaseous butane (C_4H_8), $\dot{V}_{C_4H_8} = 0{,}077cm^3/s$	3 hours
№6	The same	Hydrogen, $\dot{V}_{H_2} = 0{,}50cm^3/s$	3 hours

The following notations are used in the Table 1: $\dot{m}_a$ and $\dot{V}_a$ - the mass and volume flow rate of xenon through the SPT anode, $\dot{m}_{cath.}$ and $\dot{V}_{cath.}$ - the mass and volume flow rate of xenon through its cathode, U_d – the source discharge voltage.

The surface composition has been researched in the central part of the samples-substrates, i.e. at the distance ~13mm from the source anode (or ~11mm from the exit).

The investigation of the element composition of the samples surfaces by XPS method has been carried out with the help of ESCA-spectrometer in the laboratory of the sub-faculty of the solid-state physics of Moscow Engineering Physics Institute. Mg (line K_α) has been used as X-rays source. There has been used the energy analyzer of hemispherical type. The energy resolution has been equal to 1eV by the line Ag $3d_{5/2}$. The calibration has been carried out by the line Au (84 eV). At the given way the analyzed depth (the electron mean free path relative to inelastic collisions) is about 5nm. In order to obtain information about the element composition at the certain depth of near-surface layer, the samples surface etching by the argon ions Ar^+ with the energy 2keV has been carried out during 5 minutes (the layer about 10nm has been removed).

The research of the element composition of the samples surfaces by RBS method has been carried out in the department of physics of atomic nucleus of Lomonosov Moscow State University Skobeltsyn Institute of Nuclear Physics. Van de Graaff accelerator has been used in order to obtain the collimated beam of helium nucleuses ^4He with the energy 2MeV. In order to register the particles there have been used the semiconductor detectors, which form at the output the electrical impulse with the amplitude proportional to the energy of particles scattered by the sample in the direction of the detector. The analyzer of the impulses amplitude registers impulses of the different

amplitudes in the different channels (the alternative name derives from this: multichannel analyzer). The numeration of the channels is determined by the impulses amplitude and hence the one-to-one correspondence exists between the number of the channel and particle energy.

The backscattering of the ions ^{4}He with the energy 2,0MeV provides the resolution of the isotopes with masses up to 40 atomic mass units (a.m.u). The resolutions for the target from the elements with atomic masses of the order 200 a.m.u. is about 20, that leads to the impossibility to distinguish the atoms of elements being between ^{181}Ta and ^{201}Hg.

The research of the samples in the hydrogen content inside the near-surface layer by FRS method has been carried out in the same department of Skobeltsyn Institute of Nuclear Physics.

III. Experimental Results and Their Discussion

A. XPS method

The investigation of the element composition of the insulators work surfaces by XPS (samples №1, №2, №3, №4, №6) has shown, the elements being the part of the ceramics composition (B, N, Si, O, Zr, Y) and carbon (C) are present in their composition (including of course the sample №1) both for SPT operation at the pure xenon (samples №2, №3) and for its operation under the "threshold" values of the hydrogen content in the vacuum chamber (samples №4, №6). The detected elements percentage directly on the surface of the samples №№1÷4 (i.e. without the etching of Ar^+ ions) is given in the Fig.1. The detected elements percentage on the surface of the samples №№1÷4 after the etching by Ar^+ ions during five minutes is presented in the Fig.2.

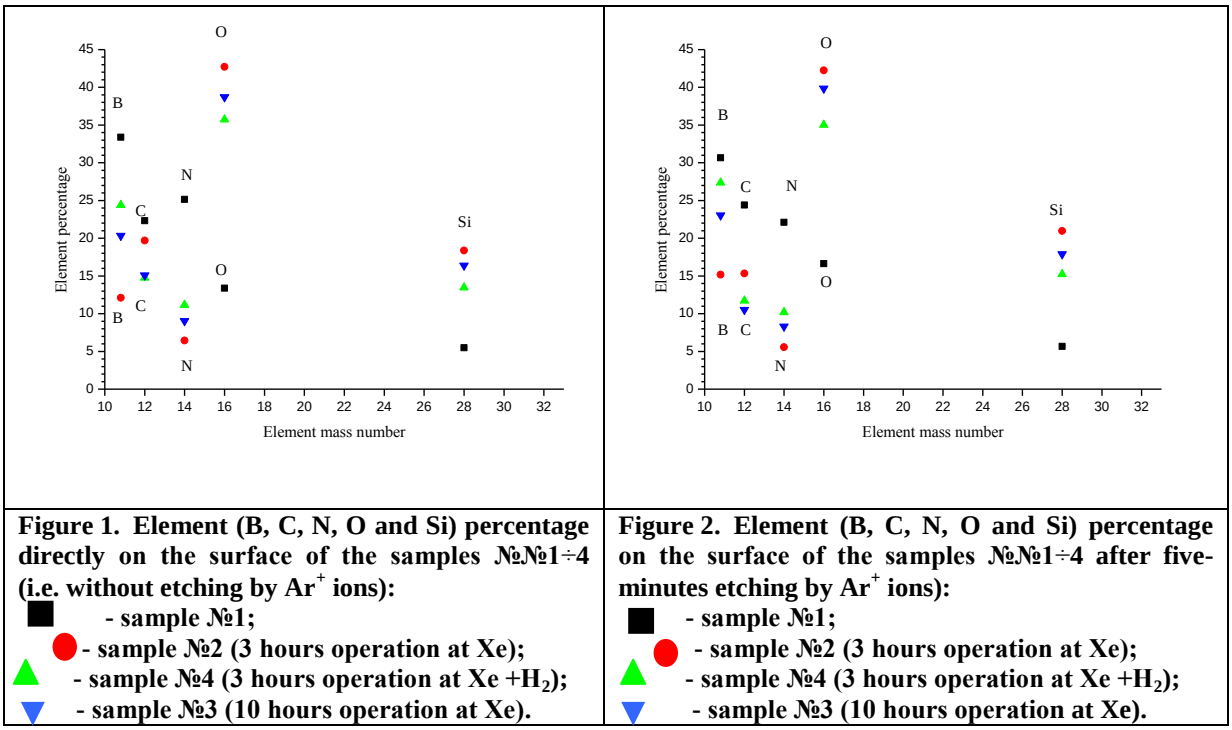

Figure 1. Element (B, C, N, O and Si) percentage directly on the surface of the samples №№1÷4 (i.e. without etching by Ar^+ ions):
- sample №1;
- sample №2 (3 hours operation at Xe);
- sample №4 (3 hours operation at Xe +H_2);
- sample №3 (10 hours operation at Xe).

Figure 2. Element (B, C, N, O and Si) percentage on the surface of the samples №№1÷4 after five-minutes etching by Ar^+ ions):
- sample №1;
- sample №2 (3 hours operation at Xe);
- sample №4 (3 hours operation at Xe +H_2);
- sample №3 (10 hours operation at Xe).

The analysis of ESCA spectra of elements B, N, C, O, Si, Zr has shown, the valence states of all listed elements on the surface and in the near-surface layer of the sample №1 (initial insulator) significantly differ from the valence state of these elements on the surface and in the near-surface layer of the samples №2÷4, having been in the plasma flow of the operating source. As an example, the normalized by the intensity distribution of boron 1s line obtained directly from the surface of samples №№1÷4 is given in the Fig.3 and the analogous curves obtained after five-minutes etching of the surfaces by argon ions are represented in the Fig.4.

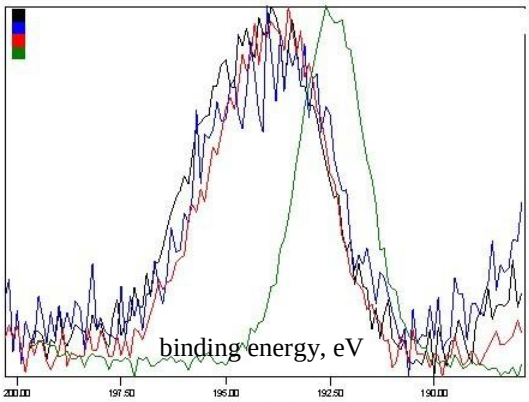

Figure 3. Distribution of the Boron line 1s (normalized by intensity), obtained directly from the surface of samples №№1÷4 (i.e. without etching by Ar ions):

- sample №1;
- sample №2;
- sample №3;
- sample №4.

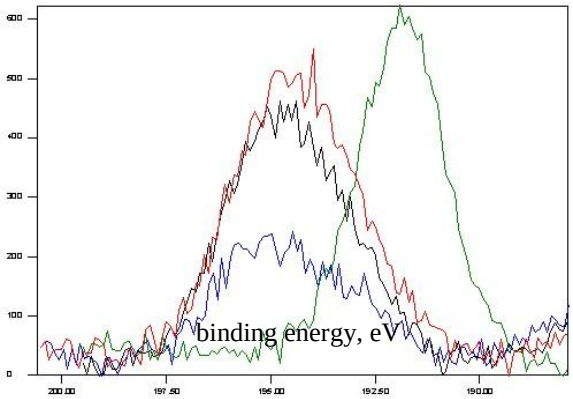

Figure 4. Distribution of the Boron line 1s from the surface of samples №№1÷4 obtained after Ar ions five-minutes etching:

- sample №1;
- sample №2;
- sample №3;
- sample №4.

As it follows from these observations, the source plasma flow has the determining influence upon the state of the channel insulator surface, and the influence of the hydrogen feeding into the chamber for the sample №4 under the given ESCA method has not been displayed in an explicit form.

In order to increase the hydrogen influence onto the insulator surface, the hydrogen additional feeding has been increased up to the value $\dot{V}_{H_2} = 0{,}50 cm^3/s$ (which is far above the threshold value for the given source operation mode), and the sample №6 has worked as the part of the external ceramic channel of the source during 3 hours under these conditions (see the Table 1).

ESCA spectra of elements B, N, C, O, Si, obtained directly from the surface of the samples №4 and №6 (i.e. without the etching) are represented in the Fig.5. The spectra of the same elements, obtained from the surface of the samples №4 and №6 after the argon ions five-minutes etching are represented in the Fig.6.

If one compares the spectra directly from the surface of the samples №4 and №6 (Fig.5), than one may see the valence states of the carbon and boron differ. The spectra of the rest elements (N, O, Si) are completely identical.

It seen from the Fig.6, that the spectra of all pointed out elements from the surface of the samples №4 and №6 after the etching are completely identical. Hence the near-surface layer of the channel insulator with the thickness no more than 10nm is responsible for the change in the plasma accelerator behavior.

The analysis of ESCA spectra of B obtained directly from the surface of the samples №4 and №6 shows the boron line maximum for the sample №4 surface is located at the binding energy ~193,8eV (the line half-width is equal to ~3eV), while the maximum of this element for the sample №6 surface is at the binding energy ~194,5eV (the line half-width is equal to ~4eV). After the etching both maxima are located at the binding energy ~194,5eV (with the line half-width of ~3eV). Thus the boron on the insulator surface is in two valence states: with the higher and smaller binding energy accordingly. The registered signal is summarized from the signals of the boron atoms being in different valence states. Depending on what kind of atoms prevails the maximum of the total (common) curve is shifted to one or another direction. Then the "critical" number of boron atoms in the valence state with the higher binding energy may exist for the surface, which breaks up the model work with the high integral characteristics under the presence of the hydrogen impurities.

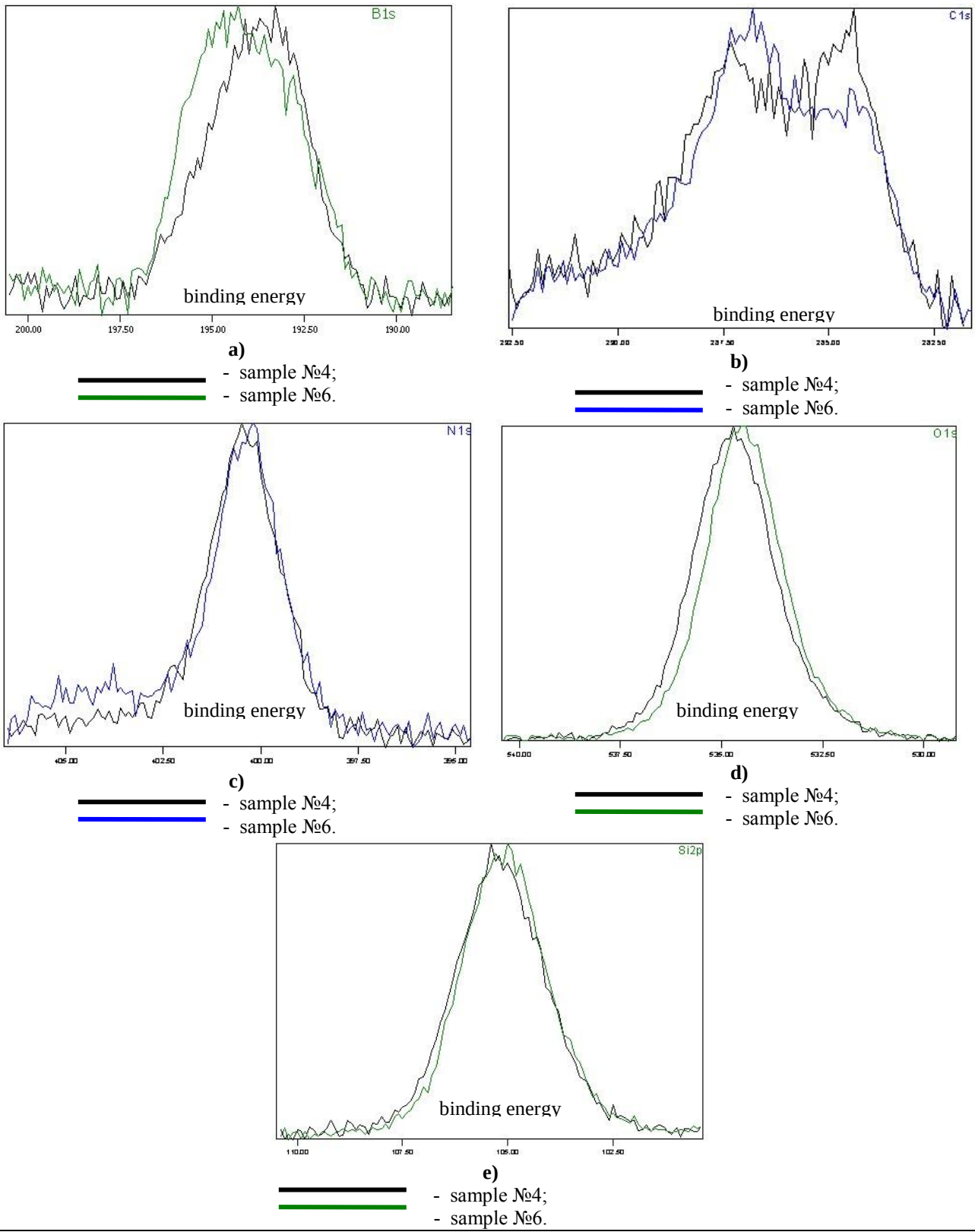

Figure 5. ESCA spectra of elements obtained directly from the surface of samples №4 and №6: a)– boron, line 1s; b) – carbon, line 1s; c) – nitrogen, line 1s; d) – oxygen, line 1s; e) – silicon, line 2p

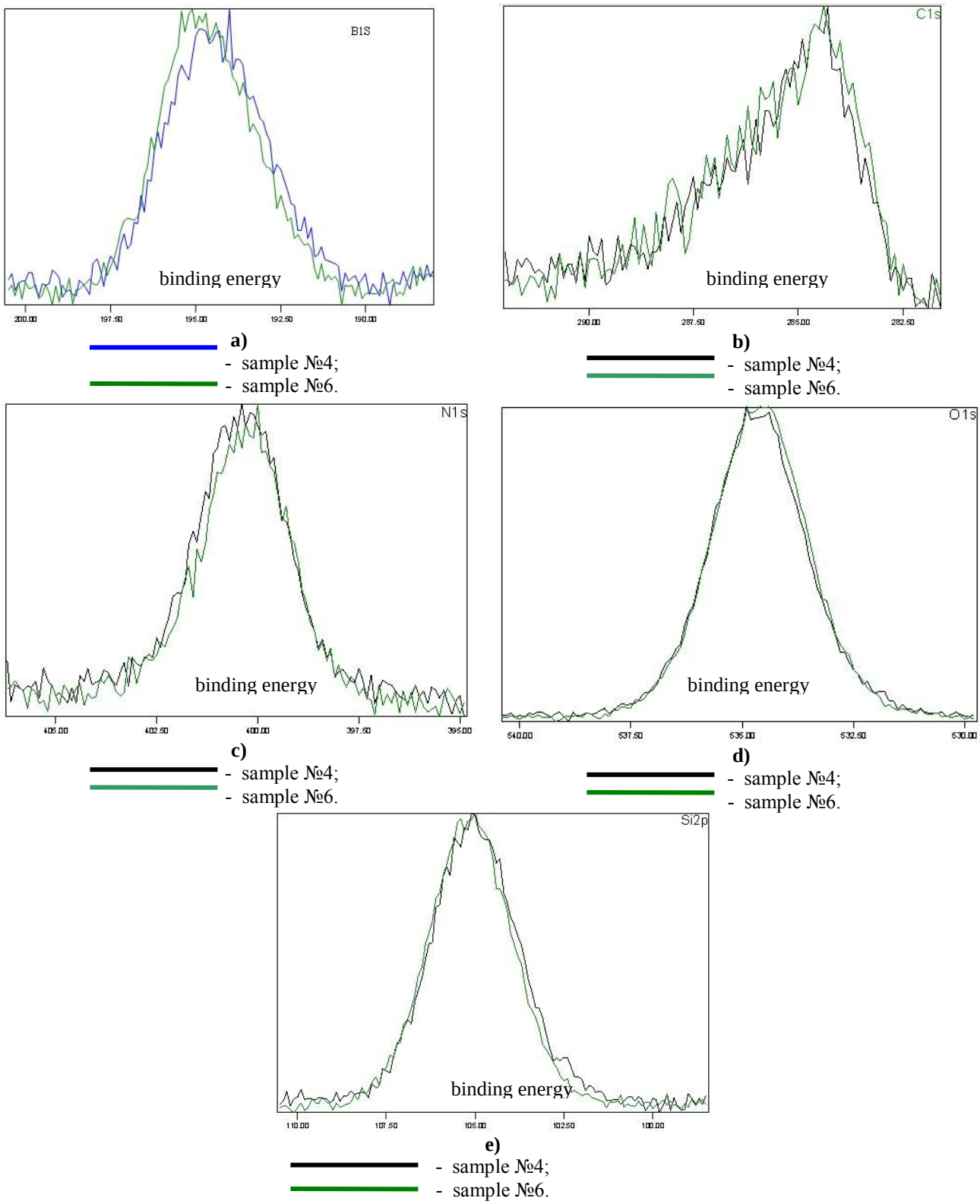

Figure 6. ESCA spectra of elements obtained from the surface of samples №4 and №6 after five-minutes etching by Argon ions :
a)– boron, line 1s; b) – carbon, line 1s; c) – nitrogen, line 1s; d) – oxygen, line 1s; e) – silicon, line 2p

B. RBS method

The surface of the samples №5, №8, №9, №10 has been investigated by RBS up to the depth ~2μm with the step 0.2μm.

Experimental curves obtained from the samples surfaces for the investigation of their element composition are represented in the Figs.7-10. Using these experimental curves and corresponding software the curves of the surface element composition (in atomic percents[10]) of the investigated samples at the different depth up to the 2mcm have been graphed. The corresponding curves are shown in the Figs.11-14. The elements B, N, Si and O, being the main ones in the composition of the ceramics BGP-10, are fixed on the surfaces of all samples.

The carbon is also found out on the surfaces of all samples. Only the sample №8 is the exception (or the carbon content in the sample №8 is too small in order to obtain the quantitative data about its percentage). It has worked as the part of the source channel when the SPT operated at the pure xenon.

The analysis of the curves represented in the Figs.11-14 shows the carbon percentage on the surface of the channel insulator at the distance ~13mm from the anode is practically the same both under the hydrogen feeding (sample №10) and under the butane feeding (sample №5) and is about the carbon percentage on the surface of the initial sample (№9), but, with an accuracy of the experimental error, the carbon does not deposit at this co-ordinate under the model operation at the pure xenon.

For all samples, in which the carbon has been found out, the carbon presence finishes at the depth ~0.4μm, excepting the sample №5 having been tested with the butane feeding. For the last case the carbon is observed up to the depth 2μm, and its percentage monotonically decreases from 8% on the surface down to 1% at the depth of 2μm.

Thus by the qualitative elements composition of the surface, the given results are in the correspondence with the results obtained for the element composition of the surface of the samples №№1, 2, 3, 4 and №6 with the help of XPS (ESCA) procedure. The discrepancy between quantitative values is due to the fact that the thickness of the layer, by which the averaging is carrying out (so named "the analyzed depth"), differs by more than the order for these methods.

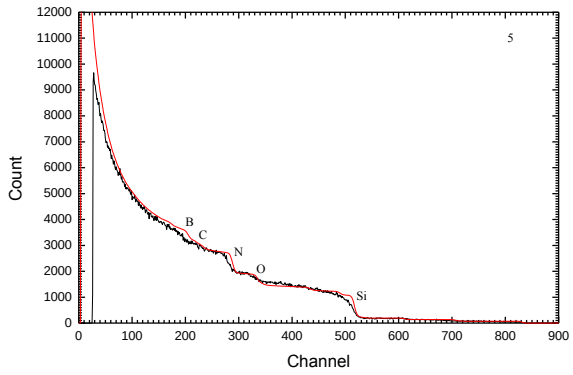

Figure 7. Backscattering spectrum from the surface of the sample №5.

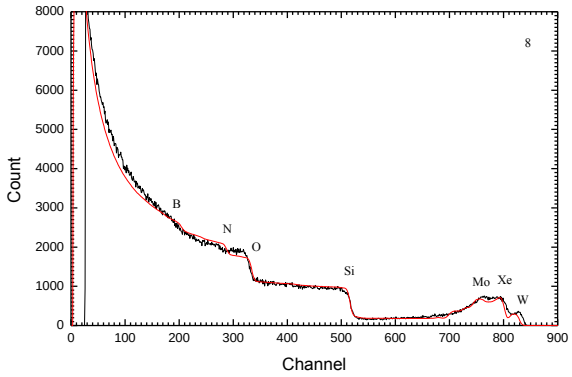

Figure 8. Backscattering spectrum from the surface of the sample №8.

[10] The atomic percent determines the percentage of the number of atoms of every element (or compound) in the material. The weight percent shows the content in weight of every element (of compound) in the material.

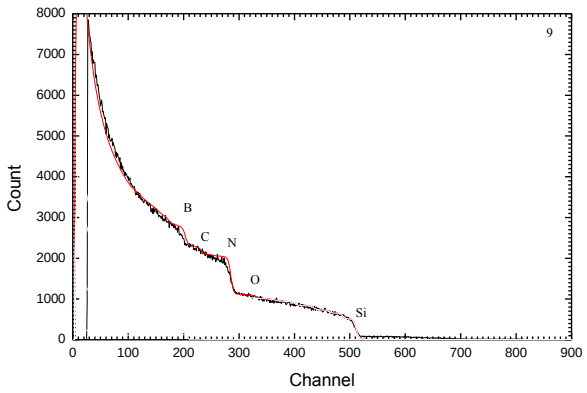

Figure 9. Backscattering spectrum from the surface of the sample №9.

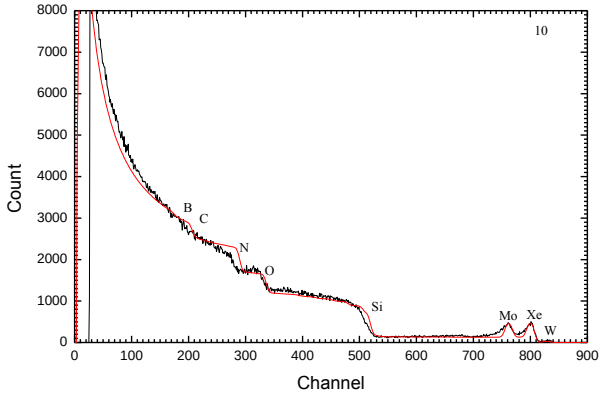

Figure 10. Backscattering spectrum from the surface of the sample №10.

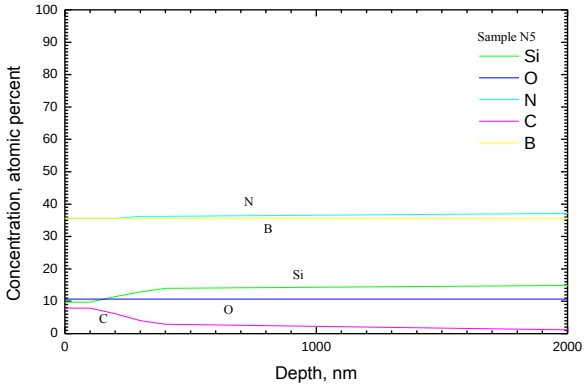

Figure 11. Element composition distribution in the depth on the surface of the sample №5.

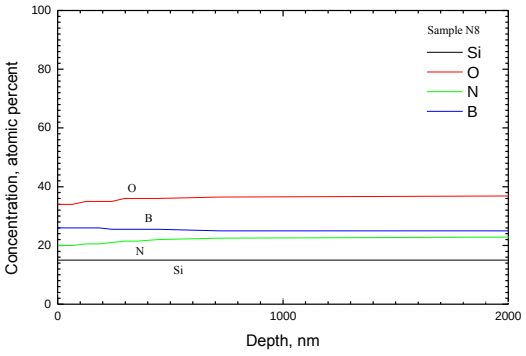

Figure 12. Element composition distribution in the depth on the surface of the sample №8.

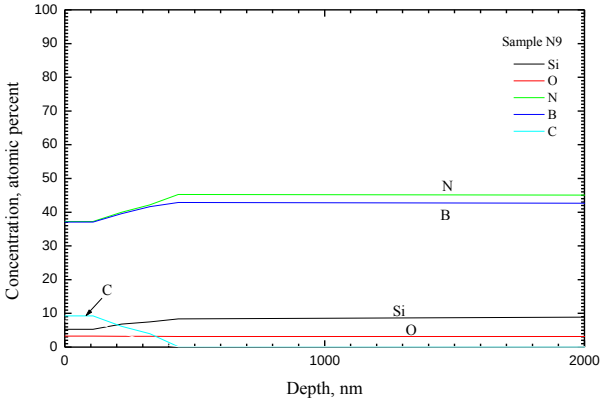

Figure 13. Element composition distribution in the depth on the surface of the sample №9.

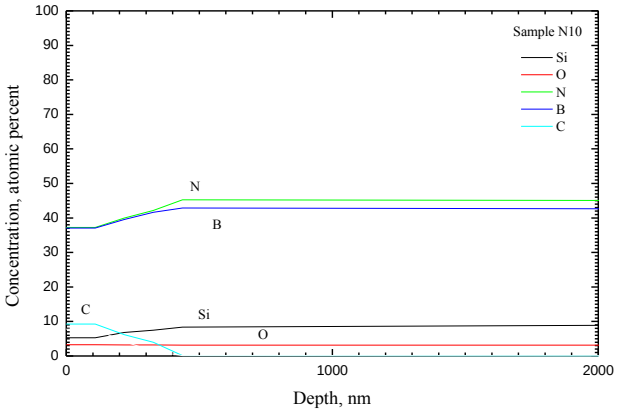

Figure 14. Element composition distribution in the depth on the surface of the sample №10.

C. Investigation of the hydrogen content in the near-surface layer of the insulators by FRS method

The samples №5, №8, №9, №10 have been investigated in the hydrogen content inside the near-surface layer by FRS method. Obtained experimental curves and hydrogen percentage in atomic percents are shown in the Figs. 15-19.

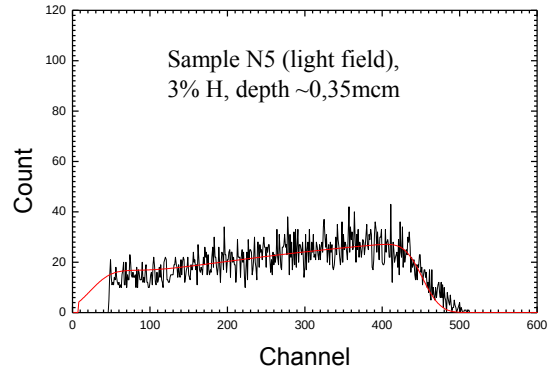

Figure 15. Spectrum of recoil atoms ^{1}H from the "light" field of the surface of the sample №5.

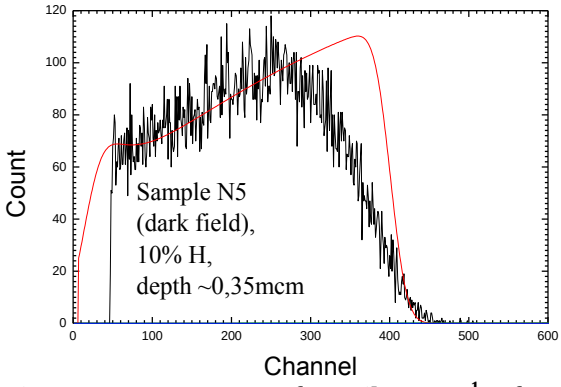

Figure 16. Spectrum of recoil atoms ^{1}H from the "dark" field of the surface of the sample №5.

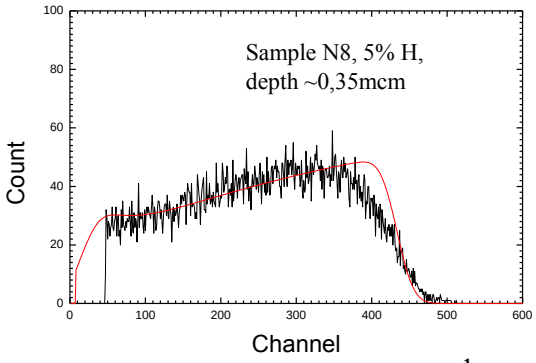

Figure 17. Spectrum of recoil atoms ^{1}H from the surface of the sample №8.

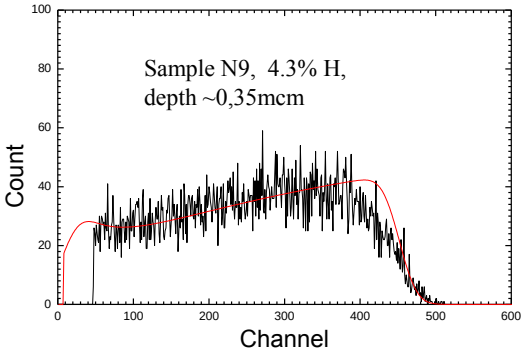

Figure 18. Spectrum of recoil atoms ^{1}H from the surface of the sample №9.

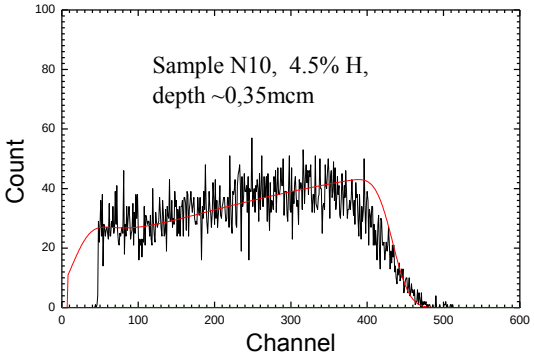

Figure 19. Spectrum of recoil atoms ^{1}H from the surface of the sample №10.

The influence of the vacuum composition onto the surface of SPT discharge chamber insulators has revealed the most strikingly under the butane feeding, i.e. for the sample №5. Its surface has been sharply divided into the "dark" and "light" (where the energetic bombardment of the surface by the xenon ions took place) zones. The measurements for the sample №5 have been carried out in two points: in the so named "light" field of the insulator and in the "dark" one. The hydrogen percentage for these fields is significantly different: it is equal to 3% in the light field (in the layer of the depth 0.35μm) and it increases by a factor of more than three in the dark field and reaches 10% (in the layer of the depth 0.35μm).

For all other samples the measurements have been carried out in the single point which is at the distance 13mm from the anode. The hydrogen percentage in these samples varies within (3÷5)% (in the layer of the depth 0.35μm).

IV. Conclusion

Using XPS method it has been shown the near-surface layer of the channel insulator with the thickness no more than 10nm is responsible for the change in the plasma accelerator behavior. The change of the distribution of the carbon and boron atoms in valence states takes place in this layer under the influence of the hydrogen. Using FRS method it has been obtained, the hydrogen percentage for the so named "dark" zone of the insulator is three times as much than for the "light" one (where the energetic bombardment of the surface by the xenon ions took place) under the butane feeding. These facts are the direct confirmation of the influence of hydrogen onto work surfaces of SPT discharge chamber.

It is known the insulator used as the wall of SPT channel has porous structure, and its surface is rough with the irregularities of the order of a few micrometers. This circumstance made very difficult the carrying out of investigations by the given methods. However this obstacle has not prevented to carry out the researches having the pioneer nature. We have succeeded not only in combining together the vacuum composition in the vacuum chamber under SPT tests, the integral parameters of the source and the properties of its work surfaces, but also in proving the determining influence of hydrogen onto the state of the work surfaces of SPT discharge chamber.

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