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USING ION BEAM ANALYSIS AND COMPUTER TOMOGRAPHY FOR STUDY PRODUCTS MADE FROM CARBON MATERIALS

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Two analytical technologies were used in the study of products made from carbon materials: ion beam analysis and computer tomography. They are shown to agree well with each other – providing additional information. Products made from carbon-carbon materials were studied: their composition and its changes as a result of testing, and the influence of nature studies on the products.

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INTRODUCTION

Analytical studies of materials play an important role in the creation and study of the properties of products made from them. They help to understand their elemental (isotopic) composition, structure and functions of materials. About 25% of all analyzes performed are devoted to the study of inorganic materials and half of them are registration of radiation from parts of complex devices. Spectroscopy of electromagnetic radiation in different radiation ranges is one of the most widespread and used methods for studying materials and products.

In recent years, a huge variety of methods for studying objects has emerged, with computed tomography (CT) attracting special attention. In most cases, the general scheme for studying objects is quite traditional. It includes several elements that are characteristic of most computed tomography methods.

However, practice forces further improvements. In most cases, the non-destructive nature of research on real and very expensive products plays a major role. We present the results of a combination of technologies using ion beam analysis with the computer tomography method.

METHODS AND EQUIPMENTS

ION BEAM ANALYSIS (IBA)

Analytical technologies based on the combined use of PIXE and PIGE methods are being developed and improved. While not having the amazing detection limits of other analytical technologies such as mass spectrometry, these techniques nevertheless have a number of advantages that allow them to be widely used. The history of these methods dates back to some publications [1].

The research method technique is quite simple. The sample is irradiated with protons accelerated to an energy of several MeV, and the excited X-ray and γ-rays are recorded.

NSC KIPT was one of the first to create an accelerator that could become the basis for ion beam analysis of substances. The use of two or more methods of research on ion beam made it possible to expand the range of identified elements (isotopes) [2].

The analytical nuclear-physics complex Sokil (ANPC) (Fig. 1) consists of the following main systems: electrostatic accelerator; output devices; experimental channels for the use of analytical nuclear-physical methods of substance; measuring and computing equipment [3]. Analytical methods of PIXE and PIGE were jointly carried out in channel 1.

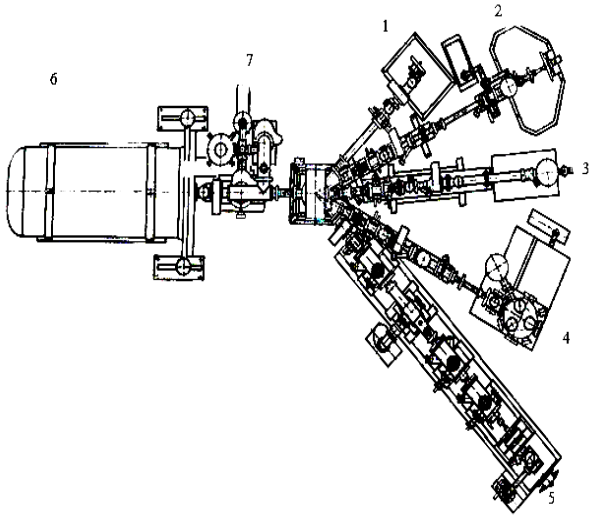


Fig. 1. ANPC Sokil:
1–5 – experimental channels;
6 – electrostatic accelerator; 7 – output devices

Registration of the excited characteristic X-ray emission of atoms was carried out using detector XR-100CR Si-pin. The detector was installed at an angle of 135º to a beam of protons, before beryllium foil windows. The detector with a resolution of 155 eV along line 6.4 keV was located at a distance of 7 cm from the target. X-ray radiation was collimated by a diaphragm Ø 1.5 mm. This detector and its spectrometry were used to implement the analytical technology of PIXE

A Ge(Li) detector with an efficiency of 18% of the efficiency of the NaI(Tl) detector measuring 7.62x7.62 cm

was used to record γ-radiation. The energy separation of the detector was 2.5 keV for the energy of γ-quanta of 1332 keV. The axis of the detector crystal was oriented at an angle of 0º relative to the direction of motion of the proton beam. The distance from the target to the surface of the detector crystal was 1.5 cm. This detector with its spectrometry over the entire energy range of the proton beam was used to release the method of PIGE.

For measuring the charge of protons, the chamber was used as a Faraday cup. The irradiation of the samples was carried out in a vacuum under pressure 10^{-4} Pa.

COMPUTER TOMOGRAPHY

The concept of CT method is to irradiate the object under study by X-Ray beam in different directions on the object and determine the decrease of their intensity linear trajectories. This decrease is determined by the Lambert-Baer law. In the case of the intensity of the monochromatic X-ray radiation from the source I_0 and the recorded radiation I, as well as the object under study, consisting of several materials, this law is written in the following way:

$$I = I_0 \cdot exp[\sum_i(-\mu_i x_i)],$$

where μ_i – attenuation factor of irradiation for each i material; x_i – path of irradiation transport.

Samples were tested on SOMATOM Definition AS 64 multi-detector computer tomograph (Siemens). Measurement was performed using spiral X-Ray tomography. UFC (Ultra Fast Ceramics) detector of CT allows to get 64 cuts in one rotation. STRATON X-Ray tube was used. Frequency of position change between points is 4608 Hz. Tube provides direct oil cooling of anode at rate 7.3 MHU/min with maximum rotation rate of anode 0.33 s. z-Sharp technology was used during the image reconstruction; this technology allows to get minimum isotropic voxel with size of 0.24 mm regardless of scanning field. Gantry aperture of tomograph was 78 cm. Computerized tomograph allows scanning along Z axis up to 200 cm with rate up to 98 mm/s with time separation 165 ms. Main data on scanning of products made from carbon materials were obtained at voltage of X-ray tube of tomograph 140 keV.

OBJECTS

Products made from carbon materials were subjected to two studies. First, they were examined in the form of three small-sized samples using computed tomography using the device described above.

Then, tissue samples were taken from each object according to Fig. 2. Fig. 3 shows one of the samples from position G1 or G2. They are located symmetrically in the product but may differ in their physical qualities. They were examined for elemental composition at the ANPC Sokil – surface and distribution of elements.

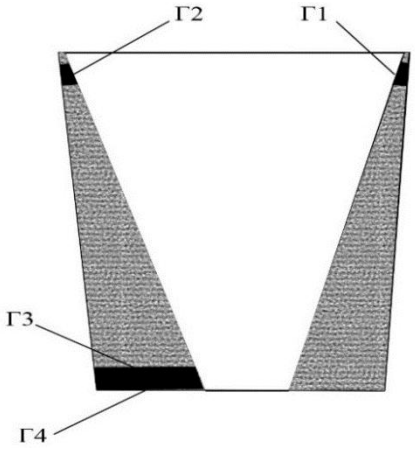


Fig. 2. Sampling on each of the nozzles. The numbers show the surface of the sample on which irradiation was carried out

Fig. 3. One of the samples

RESULTS

Analytical research of products is the most important condition for their safe and reliable operation. At the present stage of the development of analytical methods, their combination with other certification technologies, it is necessary to take into account a number of factors. This applies primarily to metrological indicators when conducting analytical studies.

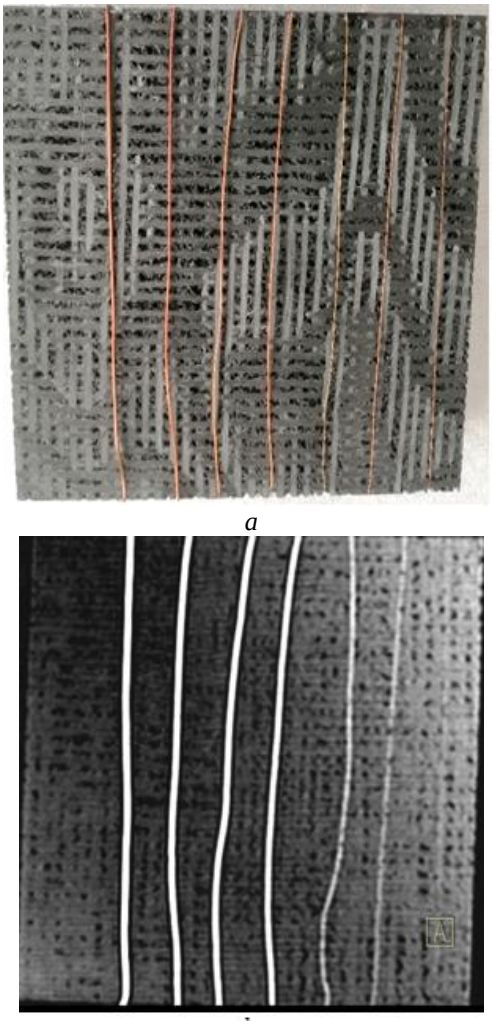
Therefore, before each measurement of IBA or CT, studies were carried out on standard samples. For the IBA technology, standard samples of carbon composition were used. Standard samples of spatial resolution were made for CT.

Since a lot has been written about standard composition samples for PIXE, further discussion will only affect samples for CT. Tomographic studies were carried out with a fairly good spatial resolution – hundreds of micrometers.

To prevent intense background and achieve better spatial resolution during scanning the maximum possible X-ray energy for the specified tomograph was selected. It was equal to 140 keV, with a current of about 200 mA.

Fig. 4 shows a standard object in the form of a parallelepiped with dimensions of 100x100x40 mm, made of carbon material, into which copper wires of different diameters are introduced. The wires are designed to actually determine the spatial resolution of the tomograph, combining its physical equipment and measurement technique with the software used.

After installation and gluing on the side surface of the sample, the wires were covered with another layer of material to more correctly simulate the presence of metal objects in the body of the carbon material.



a

b

Fig. 4. X-ray image and photograph of a carbon material specimen with wires:
a – photograph of a sample with a set of wires with diameters (from left to right) 0.56, 0.4, 0.36, 0.29, 0.18, 0.1, 0.05 mm; b – X-ray image

When performing CT scans, it was found that with different projections of the object of study, the sensitivity of the method and the tomograph may change. Thus, when studying a standard sample with wires, it was found that maximum sensitivity manifests itself in the axial projection. It is noteworthy that the effect of the material of the tomograph table on the results of the reconstruction of the sample image can be ignored.

The limit of detection (LOD) in the PIXE method depends on the energy of the incident ions and the composition of the matrix. For a carbon matrix, the LOD varies smoothly with the atomic number of the element from Al to Sn, with a minimum in the Fe region. For proton energy equal to 1.6 MeV, the minimum LOD is 1 ppm.

Limits detection for a number of isotopes from nuclear reactions for a carbon matrix are given in Table 1.

Table 1

Detection limits for some nuclear reactions [4]

Reactions	Registered γ-radiation, keV	Detection limits, ppm
^{7}Li (p, p'γ)^{7}Li	468	0.1
^{9}Be(p, αγ)^{6}Li	415	900
^{10}B(p, αγ)^{7}Be	429	1
^{12}C(p, γ)^{13}N	2365	1%
^{14}N(p, p'γ)^{14}N	2313	100
^{16}O(p, γ)^{17}F	495	1%
^{19}F(p, αγ)^{16}O	197	0.1
^{23}Na(p,p'γ)^{23}Na	440	1
^{24}Mg(p,p'γ)^{24}Mg	1368	20
^{27}Al(p, p'γ)^{27}Al	1014	6

After full-scale testing, product samples were examined on a computer tomograph in case of the presence of metal inclusions, delaminations or non-gluing. Since samples are very expensive, methods for their examination should be as non-destructive as possible. The results of computed tomography are presented in Fig. 5.

After computed tomography, the samples were examined at the ANPC Sokil. The composition of the secretions was studied, as well as the distribution of isotopes in the surface layers of the samples

The X-ray and gamma spectra of one of the objects recorded simultaneously at a proton energy of 1.6 MeV are shown in Fig. 6. From these spectra, the content of elements from Li to Zr is well identified. From this information, using a standard sample of the composition of the substance, it is possible to conduct a quantitative analysis of the substance of the object under study.

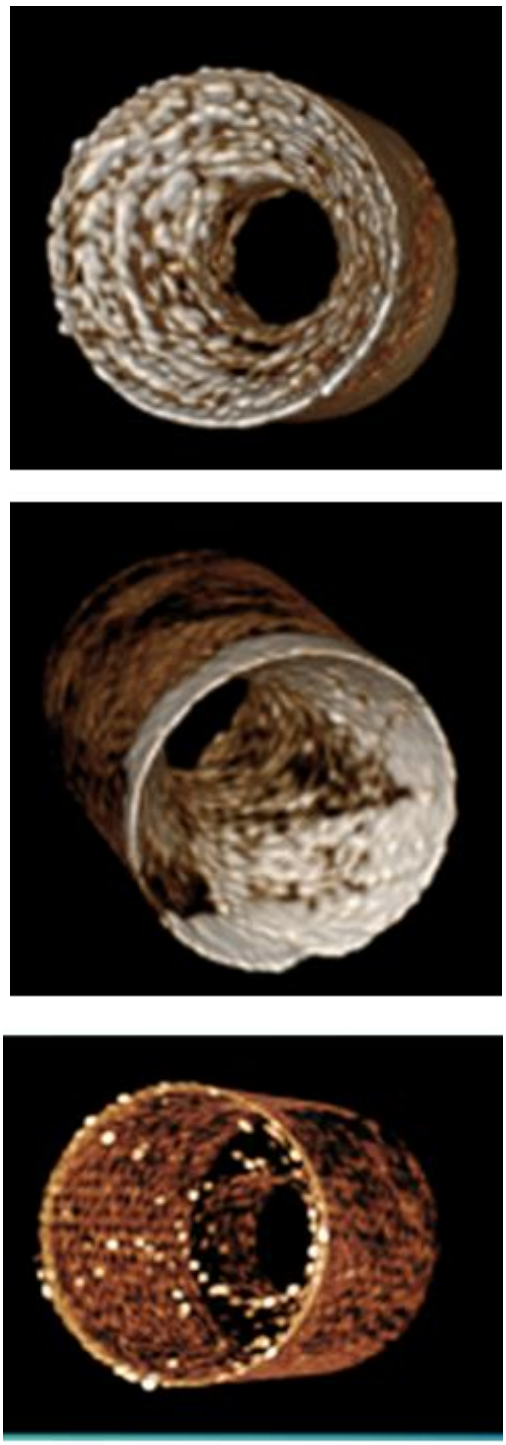
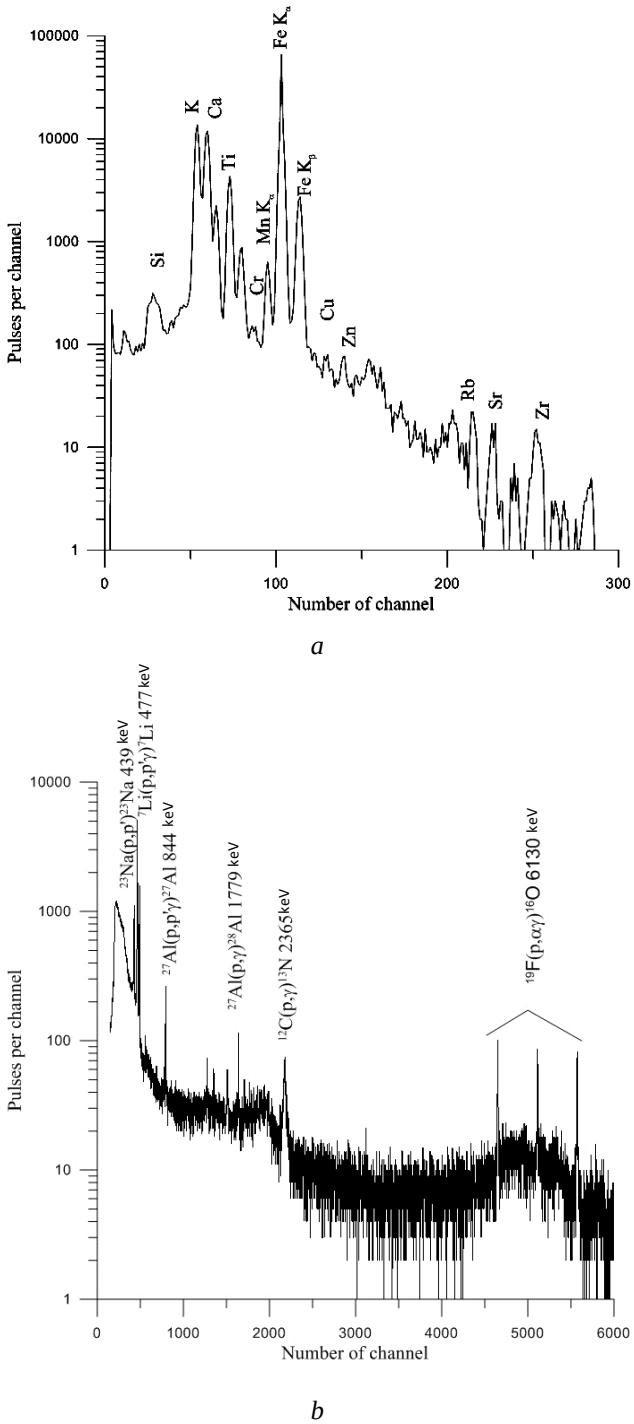


Fig. 5. Samples of original products after tests investigated on computer tomography



a

b

Fig. 6. The X-ray and gamma spectra of one of the objects when using the method IBA: a – PIXE; b – PIGE

The results of elemental analysis using a beam of accelerated protons up to an energy of 1.6 MeV of the samples under study are presented in Table 2. Samples of products are designated by numbers 1–3. From the data in the table, it can be seen that the composition of the samples varies quite a lot.

For most samples, a study of the distribution of isotopes (elements) in the depth of the surface layer was carried out. As an example of these studies, the distribution of lithium in sample number 3 is shown (Fig. 7).

Table 2

Mass content of elements

Element	Samples		
	1	2	3
Li	0.0002±0.0003	0.0002±0.0002	0.0015±0.0002
F	0.006±0.003	0.015±0.002	0.004±0.001
Na	0.026±0.007	0.008±0.002	0.033±0.005
Al	2.34±0.11	0.630±0.07	0.360±0.004
Si	0.302±0.02	0.373±0.02	0.324±0.02
S	0.079±0.002	0.117±0.003	0.098±0.004
Cl	0.380±0.04	0.381±0.04	1.342±0.06
K	0.077±0.002	0.054±0.002	0.166±0.031
Ca	0.190±0.014	0.269±0.015	0.161±0.006
Ti	0.036±0.003	0.037±0.003	1.062±0.036
Cr	0.007±0.003	0.025±0.008	0.023±0.006
Mn	0.002±0.009	0.006±0.002	–
Fe	3.27±0.25	4.56±0.35	0.91±0.07
Ni	0.016±0.003	–	–
Cu	–	–	0.005±0.005

CONCLUSIONS

Experimental studies were carried out using analytical methods IBA and CT.

The elemental composition of samples and secretions, as well as the depth distribution of isotopes (elements) in the surface layer, were studied.

After testing the samples, they were studied using computed tomography. At the same time, they were examined for the presence of inclusions, as well as discharges resulting from the tests.

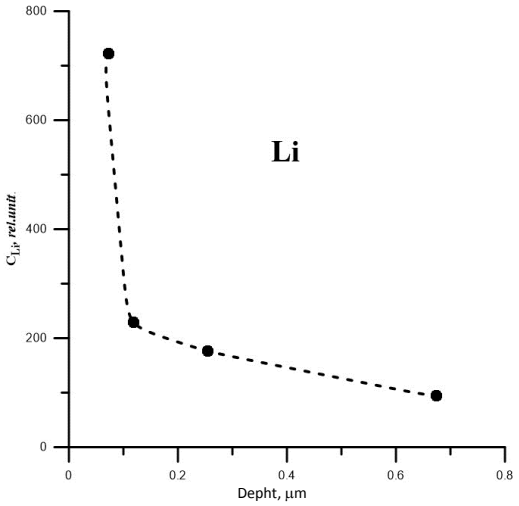


Fig. 7. Lithium distribution in the surface layer of sample

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ВИКОРИСТАННЯ АНАЛІЗУ НА ПУЧКАХ ПРИСКОРЕНИХ ПРОТОНІВ З КОМП'ЮТЕРНОЮ ТОМОГРАФІЄЮ ДЛЯ ДОСЛІДЖЕННЯ ВИРОБІВ З ВУГЛЕЦЕВИХ МАТЕРІАЛІВ

В. Левенець, І. Гурін, А. Щур

При дослідженні виробів з вуглецевих матеріалів використовувалися дві аналітичні технології: аналіз на пучку прискорених протонів та комп'ютерна томографія. Показано, що вони добре узгоджуються один з одним і надають додаткову інформацію. Вивчено вироби з вуглецевих матеріалів, їх склад і зміни у результаті випробувань, вплив натурних досліджень на вироби.