SECTION 4
DIAGNOSTICS AND METHODS OF RESEARCHES

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USING ION BEAM ANALYSIS AND COMPUTER TOMOGRAPHY FOR STUDY THE CONTROL RODS OF REACTOR PROTECTION SYSTEM

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The work is to develop methods for studying the Control Rods of Reactor Protection System (CR RPS). The isotopic and elemental content powder of boron carbide, the elemental content of alloy of shell of control rods, density and uniformity of the distribution of the absorber in the rod was studied. For investigate isotopic and elemental content was used PIGE and PIXE. A method has been developed and the isotopic composition of B_4C has been experimentally studied on the basis of the registration of γ-radiation excited in the proton reactions of the nuclei of isotopes ^{10}B, ^{11}B, ^{12}C. A method for analyzing the mass content in samples of boron carbide and 42HNM alloy was created for the range of elements from Al to W. The possibilities of research of integrity of a cover of control rods and density and uniformity of distribution in it of an absorber by a method of a computer tomography are studied.

INTRODUCTION

The share of nuclear power plants in ensuring energy balance remains in many countries at 50% and more. The safety of nuclear reactors is one of the most important factors in the further development of the industry. A special role in improving the safety of energy production at nuclear power plants is played by the control and protection systems of the reactor core – Reactor Protection System (RPS), in particular the neutron flux control system in the core, which is carried out by absorbing rods located in the reactor core together with fuel rods. To ensure long-term and reliable operation of the Control (neutron absorption) Rods of Reactor Protection System (CR RPS), it is very important to ensure the optimal choice of absorbent and structural materials for the manufacture of rod shells. One of the most efficient absorbers is ^{10}B, but the use of pure boron for the manufacture of aircraft is impractical: it does not have sufficient radiation resistance, mechanical strength and is incompatible with coolants. Instead, boron carbide – B_4C is one of the most chemically and mechanically stable substances, has high resistance to oxidation, insoluble in aqueous media and concentrated acids [1]. Does not react with nitrogen, phosphorus and sulphur up to temperatures above 1000 °C. An important factor is the ability of this compound to form a homogeneous mixture with a wide range of constituent chemical elements from B_4C to $B_{13}C_2$, which is important given the formation of neutron fluxes spatial density. The combination of all these factors led to the use of boron carbide as a filler CR RPS core of reactors for thermal neutrons.

Among the publications on the determination of the elemental and isotopic content of boron are the methods using inductively coupled plasma ICP-MS and ICP-AES and the nuclear-physical method PIGE (particle induced gamma-ray emission) [2–4]. The latter method combines the possibilities of non-destructive rapid analysis of elemental content in the range of elements from Li to U and isotopic composition for a large number of elements [5] and can be performed in parallel with the PIXE (particle induced X-ray emission) method [6], which allows to analyse elemental content from Al to U characteristic X-ray radiation excited by protons, and therefore, it is expedient to use them to study the elemental and isotopic content of the materials of the CR.

Recently, in many cases, non-destructive research methods have been used to solve the problems of production support and final product certification, the choice of which is rather limited. In particular, computed tomography (CT), which, depending on the energy of the radiation source, allows with satisfactory spatial separation to determine the defects of production in the volume of the sample, in a wide range of elements and sizes of the object under study [7].

The method of CT was chosen to study its suitability for the study of the quality of the manufacture of models of CR RPS, produced at the STC NFC NSC KIPT. The possibility of determining the integrity of the shell of the CR RPS and the density of the absorber, as well as possible irregularities in the distribution of the density of the absorber depending on the technology of it production.

The purpose of this work is to study the neutrons absorbing rods – with using methods PIGE and PIXE the isotopic and elemental content of powder boron carbide, the elemental content of 42HNM alloy intended for the manufacture of shells and method CT study density and uniformity of filling the rod with the absorber and integrity of shell.

SAMPLE PREPARATION AND EXPERIMENTAL EQUIPMENT

There were studied the stoichiometry and batch element content of four B_4C samples and two structural

steel samples in experiment. B_4C was a powder with different particle sizes. From this powder were compressed 8 tablets on an aluminium substrate framed in aluminium rings with an inner diameter of 9 mm and a width of 2 mm. The thickness of the B_4C layer in the target was about 0.03 cm. Pressing took place under pressure 10^6 Pa, and lasted 1…2 min. The chosen design of the target provided acceptable mechanical stability of the target, proper heat exchange between the target substance irradiated with a proton beam and the substrate, the ability to vary the diameter of the beam projection on the target in the range from 2 to 8.5 mm. Samples of alloys were metal plates measuring 9×11.5 mm, 1 mm of thick.·

The research was performed on an electrostatic accelerator, which is part of the analytical nuclear physic complex "Sokil" [8]. In Fig. 1 the scheme of geometrical conditions of measurements is given.

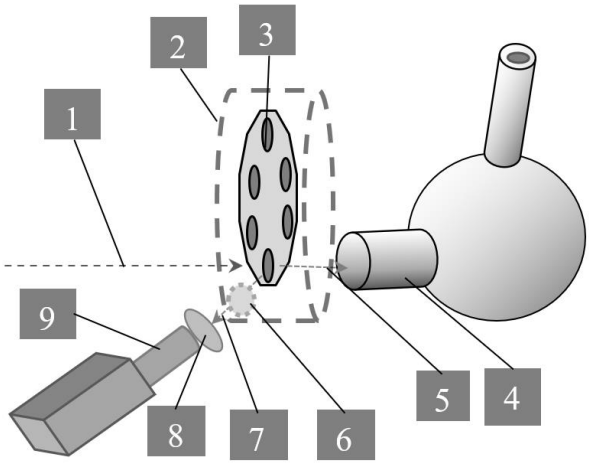

Fig. 1. Scheme of geometric conditions for X-ray and γ-radiation: 1 – beam of accelerated protons; 2 – vacuum chamber; 3 – cassette with samples; 4 – Ge (Li)-detector; 5 – γ-radiation from the sample; 6 – window camera with Be foil; 7 – characteristic X-ray emission from the sample; 8 – absorber; 9 – Si (PiN)-detector

The target cassette was in the irradiation chamber in vacuum at a pressure of 10^{-3} Pa. With the help of an automatic moving device, the analyzed objects were alternately placed in the irradiation position. Protons from the accelerator, passing a system of formation consisting of a set of diaphragms and collimators reached the surface of the target, moving along the normal to its surface. The projection of the proton beam on the target was a circle with a diameter of 3 mm

A Ge (Li)-detector with an efficiency of 18% of the efficiency of the NaI (Tl)-detector measuring 7.62×7.62 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 0° 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. A Si-pin detector with a crystal area of 2x3 mm and a thickness of 500 μm with a resolution of 160 eV along the 5.9 keV line was used to record X-rays. The detector was placed at a distance of 45 mm from the target at an angle of 135° to the direction of movement of the proton beam. In front of the detector was installed a collimator in the form of a cylinder 2 mm long with a hole diameter of 0.8 mm and one of a set of absorbers made of polyethylene with a thickness of 65, 100 or 150 μm, and aluminum – 10, 45, 50, 100 or 200 μm.

Standard spectrometry equipment, AI-4096 multi-channel pulse analyzer, and "Green Star" ADC boards were used to convert the pulses recorded by the detectors into X-ray and γ-ray spectra data storage and processing.

Samples of CR models were presented STC NFC NSC KIPT. A sample model is shown in Fig. 2. It was a metal tube in the main part with a diameter of about 9 mm with welded limbs, in which at different distances were placed fillers of boron carbide of different dispersion.

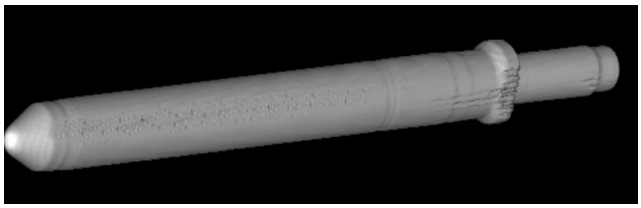

Fig 2. Model of CR manufactured by STC NFC NSC KIPT

CR models were studied on a multidetector CT SOMATOM Definition AS 64 (Siemens) (Fig. 3).

Fig. 3. CT scan SOMATOM Definition AS 64 (Siemens)

The measurement was performed using spiral X-ray tomography technology. The UFC (Ultra Fast Ceramics) CT scan detector allows you to get 64 slices in one revolution. A STRATON X-ray tube with two focal points was used. During the reconstruction of the image, z-Sharp technology was used, which allows to obtain a minimum isotropic voxel of 0.24 mm, regardless of the scanning field.

EXPERIMENTAL RESEARCH

Data on the content of boron and carbon in boron carbide, the natural isotopic content of these elements and the cross-sections of the absorption by the nuclei of their isotopes of thermal neutrons of the reactor spectrum are given in Table 1.

Table 1

Neutron absorption cross sections for boron and carbon isotopes [9]

Isotope	Content, at.%	Cross-section of thermal neutrons absorption, barn
^{10}B	19.97*	3837
^{11}B	80.17	–
^{12}C	98.93	1.27
^{13}C	1.07	0.6

*According to [10], the content of ^{10}B in the substance can vary within ± 2 at.%.

From the data given in Table 1, it follows that for the effective use of boron carbide in the CR RPS you need to have reliable data on stoichiometry and isotopic content of this substance.

The absorbent from the powdered material of boron carbide is placed in a shell of steel alloys. The material for the manufacture of the shell must meet a number of conditions, including: radiation resistance, ability to operate at temperatures up to 350 °C, high chemical resistance. Pressurized water reactors use steel alloys as material of CR shells: 06Cr18Ni10Ti (6% Cr, 18% Ni, 1…2% Mn, 0.7% Ti, Fe – base) and 42CrNiCu (42% Cr, 1% Mo, 0.01% C, Ni – basis) [11].

To determine the mass and isotopic composition of the main components of B_4C used the PIGE with the registration of γ-quanta excited by nuclear reactions, the characteristics of which are given in Table 2.

Since the intensity of γ-quanta with an energy of 429 keV from the reaction $^{10}B(p,\alpha\gamma)^7Be$ increases sharply with increasing proton energy, and the reaction $^{12}C(p,\gamma)^{13}N$ has a pronounced resonant character, the optimal range of proton energies for simultaneous determination of carbon and boron is 500…600 keV. In this energy range, the radiation yield of the 429 keV line does not create difficulties for the spectrometric path and the selection of the peaks of the analytical lines used to determine the content of ^{10}B and ^{12}C, and at the same time, fully realized resonance contribution at 459 and 163 keV and partially, resonance at an energy of 675 keV to determine ^{12}C and ^{11}B, respectively.

Table 2

Data on nuclear reactions on boron and carbon isotopes [12]

Reaction	Energy in keV of			Res. cross-sect., barn
	γ-ray	Res.	Res. widths	
$^{10}B(p,\alpha\gamma)^7Be$	429	Resonances are absent		
$^{11}B(p,\gamma)^{12}C$	4439	163 675 1390	7 322 0.053	0.157 0.050 1270
$^{12}C(p,\gamma)^{13}N$	2365	459	32.5	0.127

Measurement of γ-radiation spectra from nuclear reactions on the nuclei of B and C isotope atoms took place under the following conditions. The energy of protons during the measurements was 600 keV, current 500...800 nA, proton charge on the target 400 μC. γ-quanta were detected by a Ge (Li)-detector. A typical spectrum of γ-radiation of a sample of boron carbide is shown in Fig. 4.

As can be seen from Fig. 4 in addition to C and B, Al and Li are also present in the studied samples of B_4C. In the high-energy part of the spectrum, which is not included in the figure, there is also 6130 keV γ-emission from the reaction $^{19}F(p,\alpha\gamma)^{16}O$.

The method of external standard was used to determine the mass content of C and B and the isotopic content of B. As a standard, a sample was used, the content of C and B in which corresponded to the stoichiometry of B_4C, and the isotopic composition of boron – the natural content of isotopes ^{10}B and ^{11}B. For the processing of γ-radiation spectra we used the program Gamma, developed by NSC KIPT.

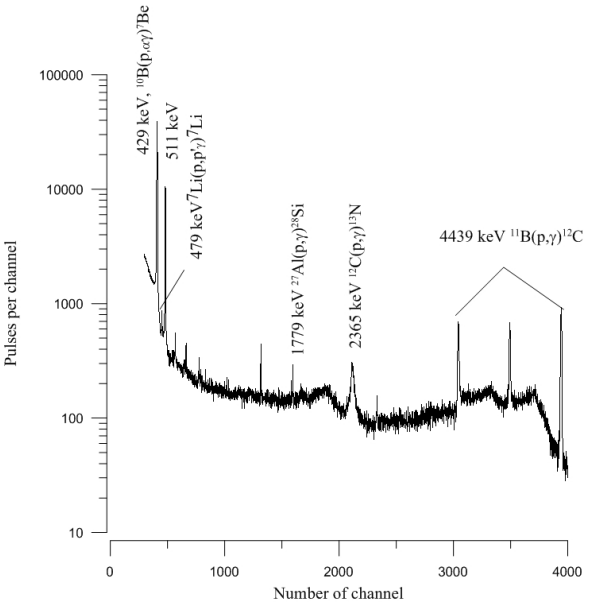

Fig. 4. The spectrum of γ-ray of a sample of B_4C. Proton energy 600 keV, beam current 500 nA

The PIXE was used to determine the content of impurities in the boron carbide samples. The spectra were measured at proton energy of 1500 keV, current of 50 nA, proton charge at the target of 10 μC. Spectrum of X-rays of a sample of boron carbide measured under the above conditions is shown in Fig. 5.

As can be seen from Fig. 5, in the studied sample of B_4C there are impurities of 12 elements from Al to Pb. This requires of study of the possible effect of these impurities on the absorbing properties of neutron absorption rods.

Experimental spectra of characteristic X-ray. The mass content of impurities was determined by the method of external standard. Emission was processed using WinQXas computer code.

PIGE and PIXE were used to determine the elemental composition of the 42CrNiMo alloy samples. The same detectors were used to register the quanta, in the same geometric configuration and the same procedure for measuring, processing and determining the result was used as for the study of boron carbide samples.

In the analysis of samples for the content of elements from Si to W used the method of PIXE. Tungsten was determined using characteristic X-ray emission L-series, and other elements – characteristic X-ray emission K-series. To determine the elemental composition used the method of external standard. As a standard sample used ISO standard based on steel 31H19N9MVBT with increased content of S and C.

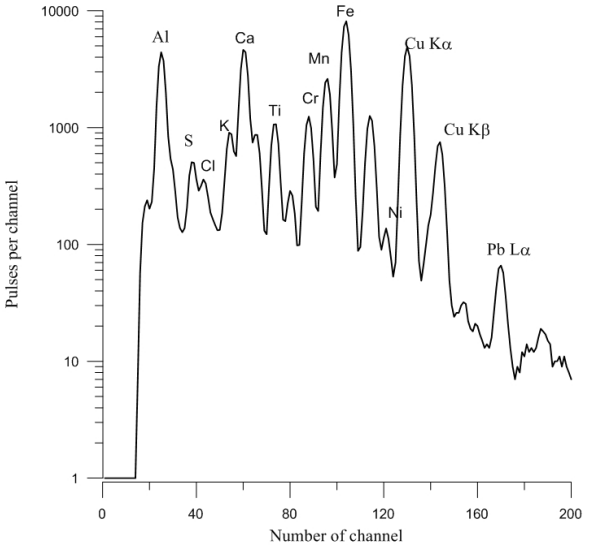

Fig. 5. The spectrum of X-ray of B_4C sample. Proton energy – 1500 keV, beam current – 50 nA

In Fig. 6 shows the spectrum of characteristic X-ray emission sample of steel alloy 42HNM in the low-energy region, under conditions of optimization for the determination of S and Si, and in Fig. 7 – spectrum of the same sample under optimal conditions for the determination of Mo and W.

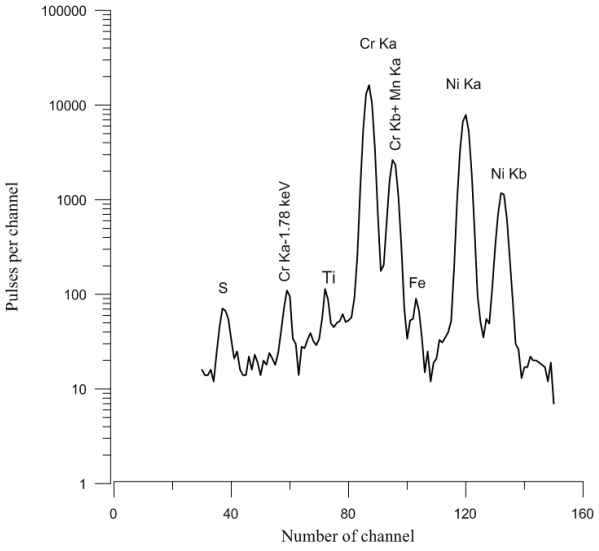

Fig. 6. Spectrum of characteristic X-ray emission of sample of steel alloy 42CrNiMo – low energy region. Proton energy 600 keV, current 20 nA

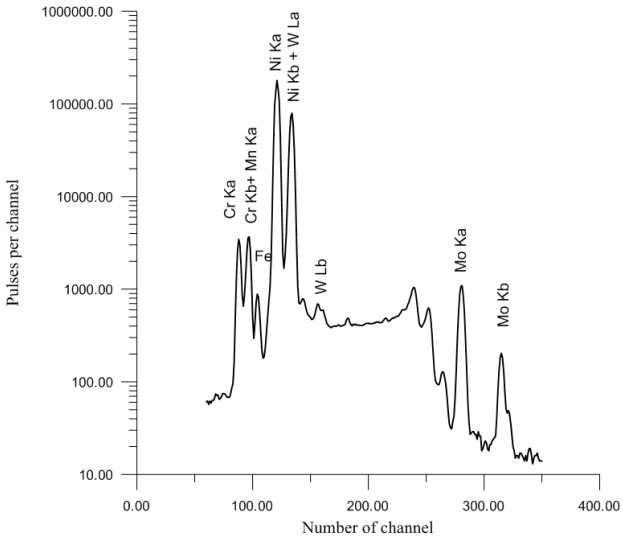

Fig. 7. Spectrum of X-ray of sample of steel alloy 42CrNiMo – high energy region. Proton energy 1500 keV, current 40 nA

RESULTS

1. USING ION BEAM ANALYSIS

The results of determination of stoichiometry, isotopic boron and elemental content of boron carbide by PIGE and PIXE are given in Tables 3 and 4.

Table 3

Isotopic composition of boron in samples of boron carbide, %

Isotope	#1634	#1650	#172	#1837
^{10}B	20.18	19.47	19.91	19.51
^{11}B	79.82	80.53	80.09	80.49

As can be seen from the data shown in Table 3, the isotopic content of boron in all analyzed samples did not show large differences, and was found to be stable within the measurement errors. This is evidence that the technology used to make powdered material from boron carbide does not lead to changes in the isotopic composition of the main absorbing element of boron.

Instead, the stoichiometry of the analyzed boron carbide samples, as can be seen from the data in Table 3, differs markedly. Yes, similar in stoichiometry to each other. There are samples from batches #1634, 1837, where the mass content of boron and carbon is correlated as 8:2, and from batches #1650, 172, where the corresponding values are correlated as 70:30. Given the same boron isotopic content in all batches (see Table 3), this means that the number of ^{10}B isotope atoms per unit mass of boron carbide powder will be different for these two samples from the batches and neutron absorption in CR made from this powder will be excellent. Table 4, in addition to macronutrients, also shows the content of impurities in the analyzed objects.

Table 4

Elemental composition of samples of boron carbide, wt.%

El.	# 1634	# 1650	# 172	# 1837
B	78.19	71.98	70.53	79.34
C	21.81	27.41	28.87	19.97
Si	0.18	0.20	0.16	0.16
Ca	0.049	0.127	0.089	0.136
Ti	0.014	0.010	0.013	0.026
Cr	0.005	0.009	0.006	0.004
Mn	0.003	0.005	0.003	0.007
Fe	0.28	0.25	0.32	0.36

The content of impurities in all batch's ranges from thousandths to tenths of a percent by weight. Si and Fe impurities have the highest content; differences for the content of these elements in individual batches reach 20%. It should be noted that the batch #1837 has the highest content for all impurities, except Si and Cr. And batches #1634 and 172 are very similar in quantitative content of impurities except for Ca. It should also be noted that the Ca content differs most from batch to batch of all impurities.

Table 5 shows the elemental content of samples of alloy 42HNM. As can be seen from the data in Table 5, the stoichiometry of the reference sample and the test sample are slightly different. In the test sample by 1…1.5 wt.% lower nickel content and correspondingly higher chromium content. If for the group of elements – C, Ti, Fe, Mo mass content is similar, then Al in the sample is significantly less, and S – more than twice as much. Since for each of the samples: the benchmark and the subject were determined by the content in two control samples, it is obvious that differences in elemental content are unlikely to indicate heterogeneity of alloy samples, and indicate differences in the substance of the benchmark and the subject.

Analysis by PIGE indicates the presence in the alloy samples of a group of light elements – Li, B, Na, F. Their content is not regulated by DSTU, but their presence in the substance CR RPS may in some way affect neutron absorption. The effect of this should be evaluated as well as the effect of the presence of a group of impurities in the boron carbide substance.

Table 5

Elemental composition of samples of alloy 42HNM, wt.%

El.	Accord	#41 6006/1 (3)		#50 60793
#	DSTU	#41-1	#41-2	#50-1
Al	≤0.40	0.24	0.23	0.15
C	≤0.030	0.0012	0.0024	0.0019
S	≤0.010	0.08	0.07	0.17
Ti	≤0.25	0.11	0.09	0.13
Cr	41.0…43.0	40.77	40.40	42.16
Mn	≤0.20	0.41	0.41	0.44
Fe	≤0.60	0.05	0.10	0.04
Ni	55…57	56.64	56.97	55.09
Mo	1.00…1.50	1.09	0.92	0.98
W	0.05…0.30	0.15	0.16	0.17

2. USING CT

The study revealed inconsistencies in the metal, which belong to the final structures of the neutron absorption rods and do not affect the metal of the main tube. Fig. 8 shows examples of discontinuities in finite structures.

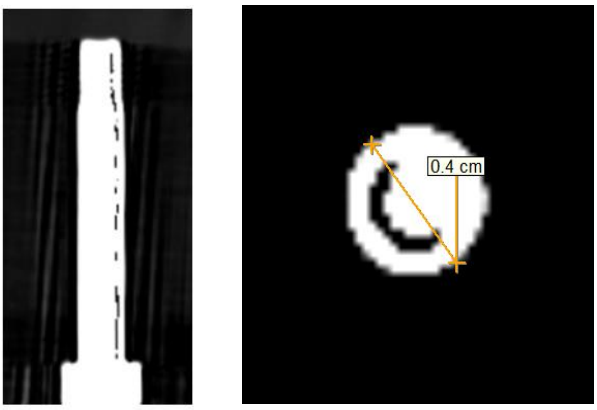

Fig. 8. The end parts of the CR

The use of different fillers in the manufacture of CR models has been recorded. Fig. 9 shows part of the CR with different fillers. It should be noted that the filler with a higher coefficient of absorption of X-rays is a more homogeneous structure.

The largest differences in the content of boron carbide among the studied objects were found in samples from batches #172 and 1837.

Based on the experimentally determined elemental content of these samples, the fraction of X-rays absorbed in them for an energy of 140 keV was calculated, at which computed tomography of neutron absorption rods with fillers from batches #172 and 1837 was performed.

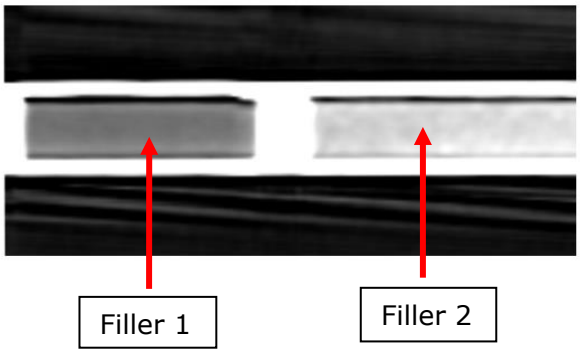

Fig. 9. Shows part of the CR with different fillers. It should be noted that the filler with a higher coefficient of absorption

The share of X-rays absorbed in the boron carbide layer with a thickness of 9 mm under the same packing density for two batches, due to the difference in content, differs by 0.2%, which can hardly be recorded by CT. On the other hand, as can be seen from the figures, the difference in the attenuation of X-rays is much more obvious, which can be explained only by the difference in the density of the package.

If we assume that the packing density for CR made of boron carbide batch #172 is 90% of the same for CR made of boron carbide batch #1837, the ratio of X-ray absorbed radiation absorbed in these CR differs by

almost 10%. From this we can conclude that CT provides a clear opportunity to qualitatively estimate the density of filling the neutron absorption rods absorber, and the difference in absorption coefficients, for the case when the density differs within 10%, contributes less than one and a half orders of magnitude

Thus, the obtained data show that CT makes it possible to control the integrity of the shell and the distribution of the density of the absorber in the studied samples of CR without their destruction with the possibility of further use for its intended purpose.

CONCLUSIONS

As a result of research, a method of analysis of isotopic and elemental composition was developed and stoichiometry and isotopic content of boron and carbon in the batch of powdered substances from boron carbide, intended for use in CR RPS. To determine these data, ɜ radiation from nuclear reactions excited by protons with an energy of 600 keV on the nuclei of atoms of ^{10}B, ^{11}B, and ^{12}C: $^{10}B(p,\alpha\gamma)^{7}Be$, $^{11}B(p,\gamma)^{12}C$, and $^{12}C(p,\gamma)^{13}N$. The analysis of the obtained data shows that the chosen methods of analysis of elemental and isotopic content make it possible to detect differences in stoichiometry and isotopic composition of boron carbide in a non-destructive manner, with sufficient accuracy without complex sample preparation, with high expressiveness. Using PIXE, a technique was developed and the mass content of Si, Ca, Ti, Cr, Mn, Fe impurities in the batch substance of boron carbide powders was determined.

Both of these methods were also used to create a method for analyzing the elemental content of samples of alloy 42HNM. On the basis of the conducted researches the mass content of elements in the range from Al to W in the samples of the alloy intended for production of a cover of CR RPS with filler on the basis of powdered boron carbide was determined.

The X-ray absorption coefficients for a series of CR fillers were calculated, the influence of differences in the composition of individual fillers and the density of CR filling on the results of the CT scan study was estimated.

The study of models of CR produced by STC NFC by computed tomography. It is shown that the use of CT scan in certain physical conditions, radiation energy – 140 keV, tube current more than 200 μA, is appropriate to determine the integrity of the shell, end structures, as well as density and its distribution for the filler (neutron absorber).

Based on the obtained results, preliminary study and certification of materials for the manufacture of nuclear power plant, as well as the study of changes in the isotopic and elemental content of boron carbide after work in the core zone can be performed.

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ВИКОРИСТАННЯ ІОННО-ПУЧКОВОГО АНАЛІЗУ ТА КОМП'ЮТЕРНОЇ ТОМОГРАФІЇ ДЛЯ ДОСЛІДЖЕННЯ СТРИЖНІВ КЕРУВАННЯ СИСТЕМИ ЗАХИСТУ РЕАКТОРА

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Розроблено методи дослідження стрижнів системи захисту реактора (СЗР). Досліджено ізотопний та елементний склади порошку карбіду бору, елементний склад сплаву оболонки керуючих стрижнів, щільність і рівномірність розподілу поглинача в стрижні. Для дослідження ізотопного та елементного вмісту використовували МІЯР та ХРВ. Розроблено метод та експериментально досліджено ізотопний склад B_4C на основі реєстрації γ-випромінювання, що збуджується в протонних реакціях ядер ізотопів ^{10}B, ^{11}B, ^{12}C. Створено метод аналізу масового вмісту в зразках карбіду бору та сплаву 42ХНМ для діапазону елементів від Al до W. Розглянуто можливості дослідження цілісності та щільності оболонки керуючих стрижнів та рівномірності розподілу в ній поглинача методом комп'ютерної томографії.