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NUCLEAR ENERGY MATERIALS RESEARCH

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The characteristics of the equipment used in the analytical nuclear physics complex for determining the elemental composition and distribution of elements (isotopes) in a solid are shown. Depending on the equipment used, different errors, ranges of elements, etc. are possible. Studies of materials developed or modified for use in nuclear power engineering are presented.

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INTRODUCTION

Science has witnessed the recognition of the important role of elemental composition analysis methods in solving some global problems, for example, nuclear safety, climate change, food security, etc. At the same time, the international community is characterized by the development of digital technologies, including for the use of analysis methods in various fields.

In view of this, requirements arise for the analysis methods used in the study of matter. They allow analyzing all elements and preferably their isotopes, ions. The analysis time is quite limited, it ranges from a fraction of a minute to several tens of minutes. These analysis methods should not require complex sample preparation. When analyzing, using them, the object should not be destroyed or accumulate something uncharacteristic for the object of study, for example, radioactivity. Surface analysis should correspond to the qualities of the whole body. The detection limit of the analytical method is as low as possible – 1 ppm and lower. Nanotechnology may also be involved, both in the analysis method itself and in the object.

These analysis methods currently used include mass spectrometry, neutron activation, chromatography, X-ray methods, microscopy, chemical analysis, etc.
There are no modern analysis methods that fully meet these requirements. But a set of non-destructive nuclear -physical methods of analysis on accelerators can satisfy almost all conditions. This primarily applies to the methods of analysis of matter PIXE, PIGE, NRA, RBS and others. Table 1 provides information on the application of these methods. We see that the most popular are materials and alloys.

Non-destructive methods based on a combination of two or more analytical technologies on an accelerator [1] allow to study materials of nuclear reactors [2, 3], as well as air filters of nuclear power plants [4], paintings of famous artists [5], food [6], etc.

The aim of the work is related to the description of the use of two physical analytical installations developed and created at the NSC KIPT and their use for the study of new and modified materials of nuclear power engineering created at the institute.

Table 1

Fields of application of leading analytical methods on ion beams

Scope	PIXE	NRA (PIGE)	RBS
Medical and biological	14.8	6.9	0.6
Ecological	18.3	0.3	1.2
Geology	3.7	1.4	0.6
Waste	8.4	12.3	6.0
Materials and alloys	53.5	61.7	85.4
Nuclear materials	1.5	14.8	6.3

METHODS

When developing a technology for producing materials with increased requirements for purity and composition, X-ray fluorescence analysis, which is performed on the ELEAN installation, is used at the first stages of research (Fig. 1).

It consists of the following main components:

1. A source of highly stable X-ray radiation with an energy of 60 keV. The nominal output power of the device is 3 kW.

2. An analytical chamber designed to implement the X-ray optical scheme, fix the X-ray tube, place and change the samples being analyzed.

3. An energy-dispersive spectrometer designed to perform X-ray diffraction.

4. A controller unit for controlling the X-ray source and the analytical chamber.

The X-ray optical scheme of the ELEAN installation is built on the basis of optics with a mutually perpendicular arrangement: the anode of the X-ray tube – secondary emitter – sample – X-ray detector. The detector is located in a plane different from the anode of the tube, the secondary emitter.

The use of such X-ray optics allows you to obtain the following metrological characteristics: the range of elements being analyzed from Al to U (depending on the excitation series of the elements of the sample and the secondary emitter); limit of determination – up to

10^{-5}% by mass; measurement time (for most tasks) – 15…20 min; number of samples – 15 pieces; type of samples – solid, powders, liquid.

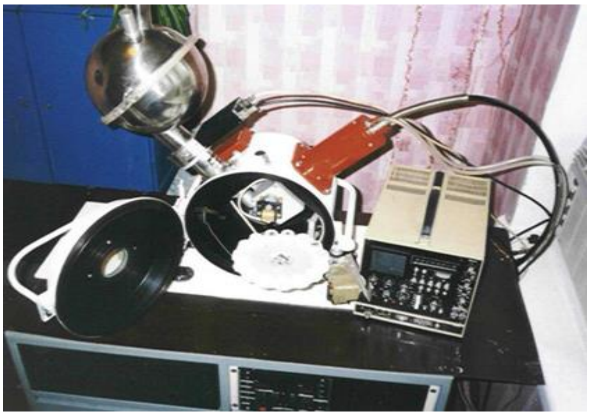

Fig. 1. X-ray fluorescence installation ELEAN

Analytical Nuclear Physics Complex (ANPC) SOKIL

The ANPC SOKIL (Fig. 2) consists of the following n systems:

- electrostatic accelerator (ESP);
- output devices;
- experimental channels for the use of ANPK;
- measuring and computing equipment.

Technical characteristics of the accelerator are given in Table 2.

Five output channels allow using all technologies on heavy ion beams. These are such as PIXE, PIGE and other analytical technologies, as well as releasing an accelerated beam into the atmosphere, using an X-ray filter, irradiating the sample at different temperatures, using a nuclear microprobe. This is partially described in [7].

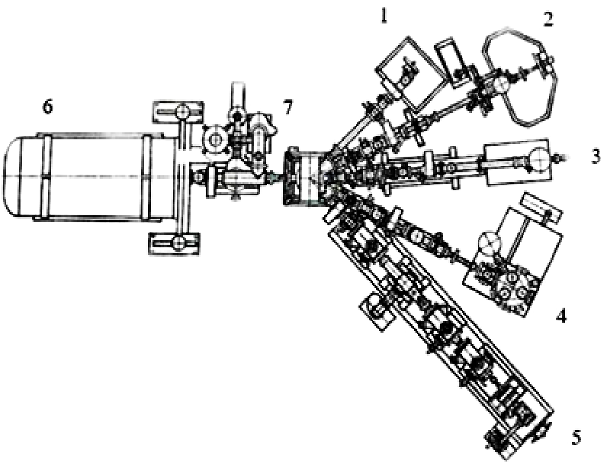

Fig. 2. ANPC SOKIL:
1–5 – experimental channels; 6 – electrostatic accelerator; 7 – output devices

Separate or complex use of nuclear physical analytical methods at ANPC SOKIL allows:

- to determine the elemental composition of the surface of a solid in the range of elements from H to U with concentrations of 10^{-5}…100%;
- to conduct multi-element analysis of samples or real products without destruction and to introduce final radioactivity;
- to determine the distribution of element concentrations over depth with a resolution of $\geq$ 50A;
- to determine the position of alloying (impurity) atoms in the crystal lattice;
- to determine the thickness and composition of coatings, etc.

Finally, these installations and software can be seen from the works [8, 9].

Table 2

Technical characteristics of the accelerator

The energy range of accelerated ions, MeV	0.2...2.0
The smoothness of energy regulation, keV	0.1
The maximum ion current at the direct output of the magnetic analyzer, μA	30
Beam current in experimental channels, μA	0.001…10
The largest diameter of the beam on the target, mm	5
The smallest diameter of the beam on the target, μm	3
Monoenergeticity and energy stability, %	0.1

MATERIALS

In the process of researching new or modified nuclear energy materials, all analytical capabilities were used. Some of the works will now be presented.

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First, a complex of nuclear physics techniques on accelerated ion beams is presented with the study of the elemental composition and structure of porous nanostructured materials of the V-N-He type on an ion beam. For which channel 4 was used Fig. 2.

It is shown that the helium content in them was less than 1 at.%, the total porosity can reach 27%, and the content of adsorbed hydrogen reaches 7 wt.%.

A significant part of the work is devoted to zirconium, steel, coatings. An important characteristic of the technology for producing zirconium-based alloys is the uniformity of the distribution of elements throughout the ingot. It affects the physical properties of the material and the subsequent process of manufacturing pipes, rods, etc.

A proton beam released into the atmosphere was used to study the distribution of the content of a group of elements in the volume of a cylindrical zirconium ingot. The length and diameter of the ingot were 2000 and 230 mm, respectively. During the analysis, the distribution of Ca, Ti, V, Fe, Ni, Nb, Hf along the length of the ingot, as well as in the transverse direction, was studied. The choice of locations for studying the element content is shown in Fig. 3.

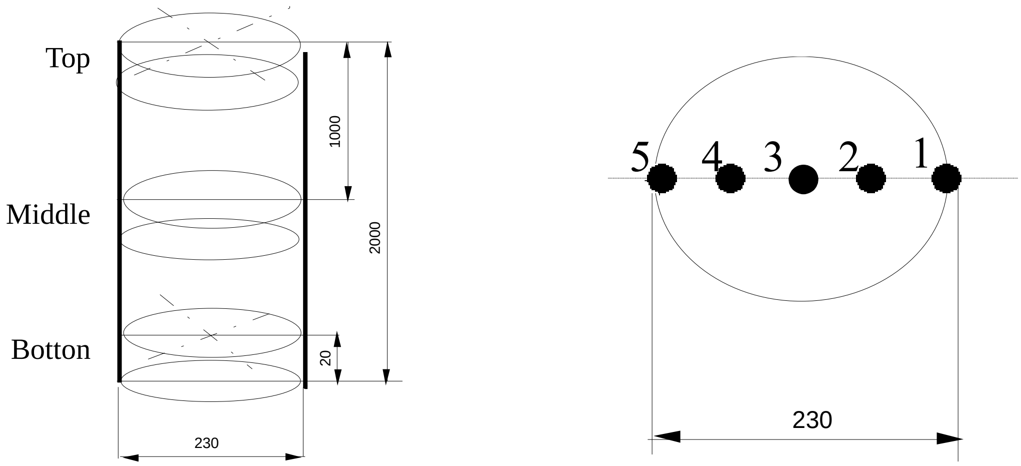

Fig. 3. Scheme of studying the distribution of element concentrations in the volume of a zirconium ingot

A fragment of the X-ray spectrum of a zirconium sample of the Zr1%Nb alloy, measured on the extracted beam, is shown in Fig. 4. As can be seen from the figures above, the developed method allows obtaining information about the content and distribution of elements in the sample without destroying the sample.

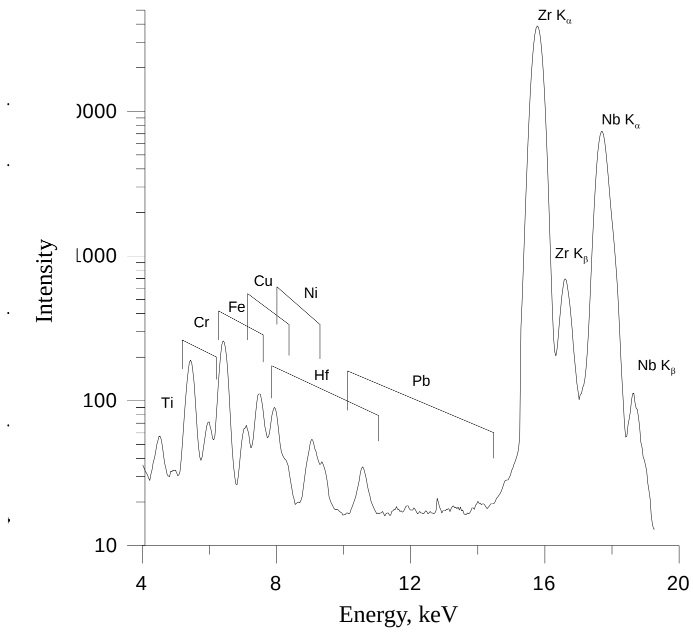

Fig. 4. The spectrum X-ray radiation of a zirconium alloy sample measured on a proton beam emitted into the atmosphere

The information obtained illustrates the uniformity of the distribution of the determined elements in the zirconium ingot in the radial and longitudinal directions. It was found that only the distribution of niobium, within the limits of the errors of the method, is uniform. The distribution of other elements is quite complex, both in the radial direction and along the ingot.

The distribution of elements in high-entropy coatings was determined depending on the composition of the cathode and the conditions for its production.

The creation and modification of coating technologies based on the use of simple materials have exhausted their development potential. It is believed that the most appropriate is the application of thin layers of coating from several tens of nanometers to several tens of micrometers from different materials with certain structural, physical-mechanical and chemical properties.

The cathodes from which the required coating composition was made were produced by remelting by the vacuum-arc melting method in a high-purity argon atmosphere and the mass content of the cathode substance was studied.

The mechanism of cathode substance transfer to the substrate during the deposition process and the elemental content of the cathodes at several points were studied. Some of the cathodes used and the distribution of beam interaction with them are shown in Fig. 5.

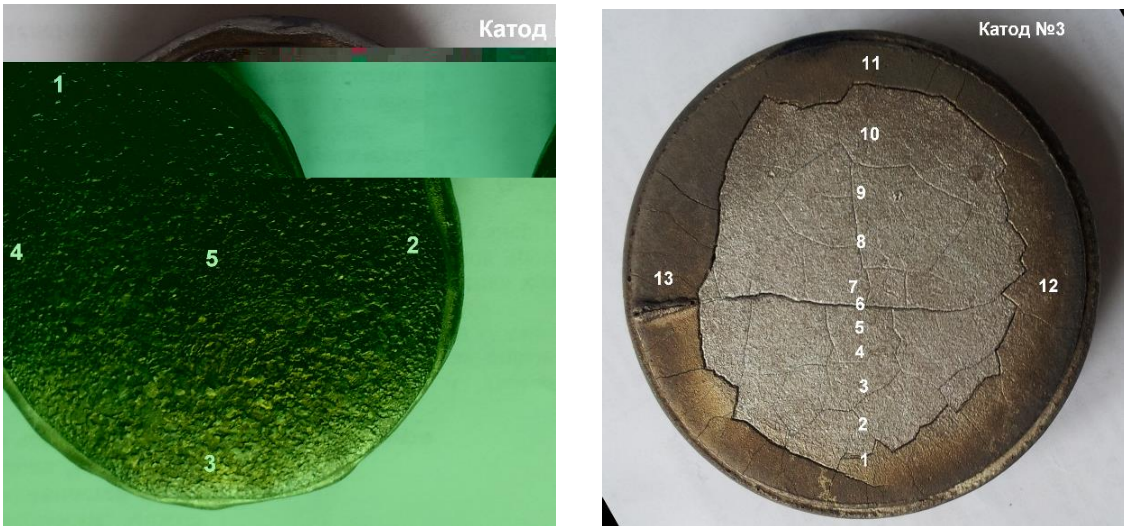

Fig. 5. Analysis point on cathodes 1 and 3

Distribution of elements in cathode No. 1 and 3 presented in Tables 3 and 4.

Table 3

Distribution of elements in cathode No. 1

Analysis point, No.	Content, mass.%				
	Ti	**Zr**	**Nb**	**Hf**	**Ta**
1	36.00	30.86	31.82	0.82	0.50
2	35.56	31.01	32.20	0.72	0.51
3	35.11	31.42	32.30	0.72	0.44
4	35.66	30.29	33.19	0.51	0.35
5	36.53	30.61	31.57	0.79	0.50
Average	35.77	30.84	32.22	0.71	0.46

As we can see from the data, the basic elements of the cathodes are fairly evenly distributed along the working surface in all directions. For the mass content ranges of 15…60 wt.%, the relative value of the average static deviation is 0.8…1.7%. With a decrease in the element content to 3 wt.%, this value increases to 4.3%, and in the range of 0.3…0.8 wt.% it is 10…15%, which is quite expected.

Table 4

Distribution of elements in cathode No. 3

Analysis point, No.	Content, mass.%		
	Ti	**Cr**	**Zr**
1	55.74	43.89	0.38
2	55.27	44.46	0.27
3	55.32	44.38	0.30
4	54.93	44.78	0.28
5	55.05	44.61	0.34
6	55.09	44.60	0.31
7	55.20	44.50	0.31
8	55.66	44.07	0.30
9	54.77	44.97	0.26
10	54.93	44.80	0.28
11	56.39	43.31	0.30
12	55.63	44.06	0.31
13	53.90	45.78	0.31
Average	55.22	44.48	0.30

Pure zirconium easily transmits neutrons (0.18 barn). The presence of hafnium in zirconium in an amount of only 0.02% reduces neutron transparency by 6–7 times (115 barn). Using a carbon-based X-ray filter, it is possible to determine the rather complex spectrum of the Zr alloy in one line of the Hf L series (Fig. 6).

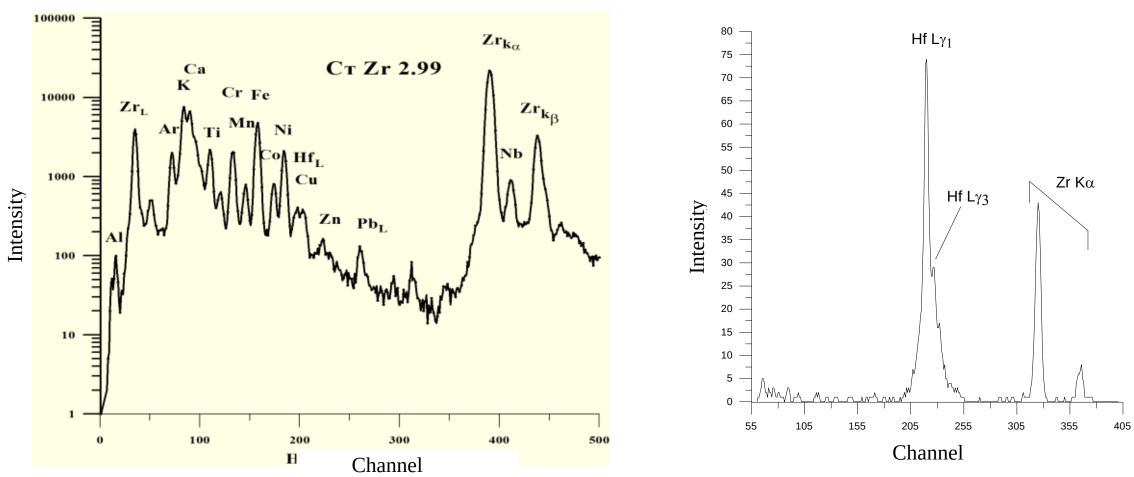

Fig. 6. X-ray spectra of the Zr standard and the Hf line

CONCLUSIONS

Large-scale studies of materials were carried out using installations developed and manufactured at the NSC KIPT.

The mathematical apparatus of probabilistic statistics was used to process the obtained data.

The conducted studies revealed that the activities of the NSC KIPT make a fairly significant contribution to the analytical studies of materials used in nuclear reactors and other areas.

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ДОСЛІДЖЕННЯ МАТЕРІАЛІВ ЯДЕРНОЇ ЕНЕРГЕТИКИ

Л.С. Глазунов, В.В. Левенець, О.Ю. Лонін, О.П. Омельник, А.О. Щур

Наведено характеристики обладнання, що використовується в аналітичному комплексі ядерної фізики для визначення елементного складу та розподілу елементів (ізотопів) у твердому тілі. Залежно від використовуваного обладнання можливі різні похибки, діапазони елементів тощо. Представлено дослідження матеріалів, розроблених або модифікованих для використання в ядерній енергетиці.