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ORGANIZATION OF EXPERIMENTAL TECHNOLOGICAL COMPLEX FOR SERIAL MANUFACTURING AND TESTING OF PRODUCTS AND SEMI-FINISHED GOODS MADE OF DIAMOND-LIKE AND LAMINATED COMPOSITES FOR LEADING BRANCHES OF MECHANICAL ENGINEERING

An experimental technological complex has been created. It consists of the sections for coating with diamond-like nanostructured composites; for manufacturing the radio-protective and other types of laminated metal composites; and for testing the radio-protective characteristics of materials. The technological equipment and the tester of radio-protective properties have been upgraded. On the basis of the developed experimental methods the batch of diamond-like coated ring seals of large diameter and samples of Al-Pb laminated composites have been made. The radio-protective effectiveness of composites has been experimentally established to be 30—40 % higher than that of aluminum.

Key words: vacuum-arc and vacuum rolling methods, accelerator and radioisotope techniques, diamond-like coating, and laminated composites.

One of key priorities of Ukraine's socio-economic development is to raise the technological level of engineering, aerospace, and other domestic industries. To address this problem in a dynamic and effective way the domestic machine-building enterprises should apply new technologies, equipment, and materials created in the course of R&D works within the framework of fundamental and applied research. An important part of this process is the practical demonstration of capacity and advantages of developments through the creation of research and production enterprises for manufacturing a relatively limited amount of specific products and functional materials.

This project is targeted towards implementing these objectives. Its goal is to create conditions for applying the newest types of nanostructured materials at manufacturing enterprises in Ukraine by means of creating an experimental technological complex for mass production and testing of products and semi-finished goods made of diamond-like and laminated composites for mechanical en-

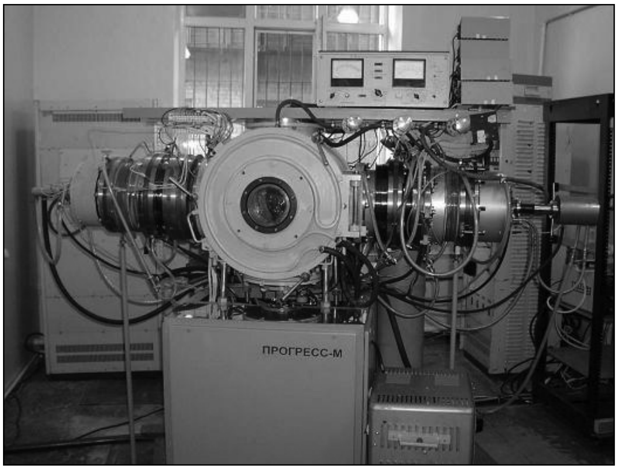


Fig. 1. Vacuum arc assembly for applying protective antifriction diamond-like coatings

gineering industry, at the National Research Center of Kharkiv Institute for Physics and Technology (KhIPT NSC).

In the course of the project implementation the following sections of experimental technological complex have been created:

+ Section for coating with diamond-like nanostructured composites;
+ Section for preparing the radio-protective and other laminated metalliic composites;

Section for testing the radiation-protective characteristics of metallic and non-metallic composites.

These sections are based on the following developments of KhIPT researchers:

+ Synthesis of diamond-like carbon phases from the vacuum arc carbon plasma flux purified from particulates;
+ Solid phase welding of metals of different composition by hot vacuum rolling (HVR) technique;
+ Means of artificial irradiation of materials and measurement of its parameters.

Within the framework of partnership cooperation agreements the project partners (contracting authorities and potential customers of products and services) are *Grace Engineering* (Sumy) and *Dniprotechservice* (Dnipropetrovsk) R&P corporations.

1. SECTION FOR COATING WITH DIAMOND-LIKE NANOSTRUCTURED COMPOSITES

Since the 1980s, the techniques for synthesis of widely used diamond-like films have been developed intensively because of unique combination of their properties: high hardness, low friction coefficient, chemical inertness, biocompatibility, thermal and radiation stability, high transparency, heat conductivity, etc. One of the important areas of application of diamond-like films is the creation of protective anti-friction coatings for high-precision friction units that are effectively used in the reconstruction of existing and the creation of new equipment for many industries [1, 2].

A modern global trend in the compressor equipment upgrade is refusal from the traditional oil seals for the sake of the dry running gas seals (DRGS). This type of seals ensures a significant increase in equipment service life, a decrease in energy consumption and operating costs, a significant growth in production safety; prevents oil pollution of gas or liquid pumped by charge compressor; and reduces their losses.

The KhIPT researchers have created equipment for the deposition of hydrogenated diamond-like antifriction coatings in glow discharge plasma in hydrocarbons environment. Today, this technique has applied in Ukraine for coating the DRGS parts. Over the last seven years, the DRGS modules with hydrogenated coatings obtained in KhIPT were used by *Grace Engineering* R&P Corporation, the leading Ukrainian manufacturer of DRGS for compressors and pumps. These sealing systems have proved themselves to be effective at industrial sites in Ukraine, Russia, and Iran. They show good performance in various environments for pressures in compactible systems up to 80—100 kg/cm^2.

However, at the pressure of more than 100 kg/cm^2 the performance of hydrogenated diamond-like coatings is unsatisfactory. To solve this problem, it is advisable to use a different method of producing diamond-like coatings that is the deposition from filtered flux of carbon vacuum arc plasma. Such coatings have 1.5—2 times higher hardness, heat resistance, and several times higher wear resistance as compared with the hydrogenated surfaces, which can significantly improve the performance of compressor equipment and make it possible to operate it at the pressure higher than 100 kg/cm^2 [1—3].

The *Progress-M* vacuum-arc assembly for the application of diamond-like carbon coatings was created on the basis of *Bulat-6* assembly discharge unit.

The appearance of upgraded equipment is showed in Fig. 1. High-quality diamond-like coatings are synthesized of separated carbon plasma fluxes generated in vacuum-arc discharge with graphite cathode.

It is known that the supply of pulsed high-voltage bias potential to the substrate leads to a significant reduction of residual stresses in coatings, which improves adhesion and makes it possible to get coatings of larger thickness as compared with the supply of constant potential. To utilize this opportunity an assembly has been developed and equipped with original generator of high-voltage pulses, which makes it possible to supply pulsed bias potential having several kilovolts amplitude to the substrate (Fig. 2).

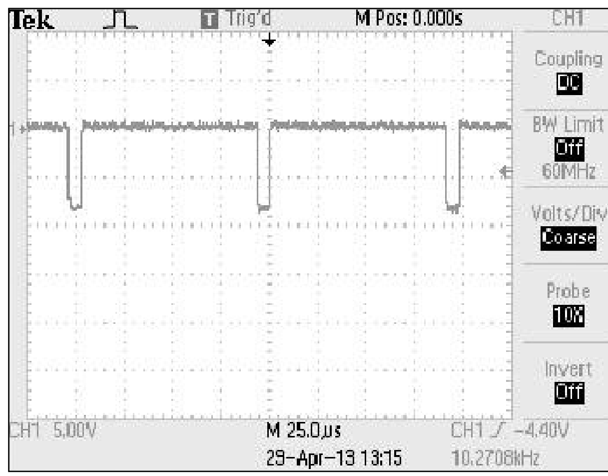
Basic technical parameters of the generator:

Line voltage, V	220
Power supply current, A	5
Voltage pulse amplitude, kV	up to 2,5
Minimum load resistance (of plasma), Ohm	200
Pulse duration, μs	6, 10 and 20
Pulse repetition frequency, kHz	0,5—12

Two improved plasma vacuum arc sources with linear filter were mounted on the assembly. Recently, they were developed at the KhIPT National Research Center.

A new type of source has been protected by patents of Ukraine and Russia; the procedure of patenting in Europe and Southeast Asia has been launched [4—6]. The main advantages of this source are as follows: high performance, stable parameters regardless of cathode burnout; uniformity of coating thickness over a large area with high quality of plasma purification from microparticles; structural simplicity; low cost of production as compared with the known sources with curved filters. Improved vacuum arc evaporator, special design of screens that intercept macroparticles, and original configuration of magnetic field transporting the plasma flux provide high filter transmission factor, up to 50%.

Vacuum arc plasma (Fig. 3) is created and transported in plasma optic system with linear filter. The «systemic factor» ε of this source was estimated by the ratio of the ion current I_i on the probe (a flat disk having a radius of 80 mm) located at the filter outlet to the discharge current (arc current I_{arc}), i.e. $\varepsilon = I_i/I_{arc}$. The ratio makes



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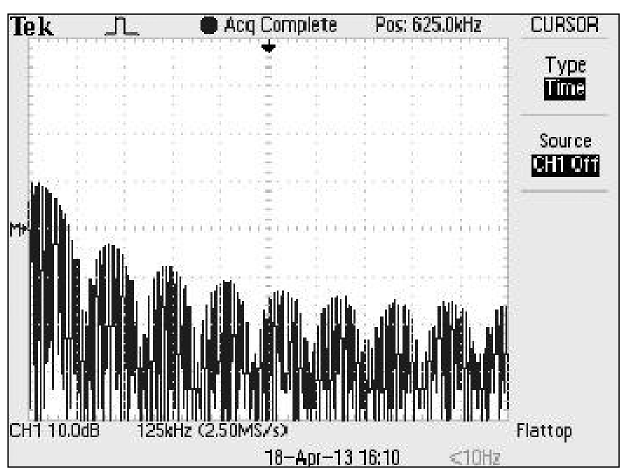

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Fig. 2. Parameters of high-voltage pulse generator output signal: *a)* signal shape; *b)* frequency spectrum

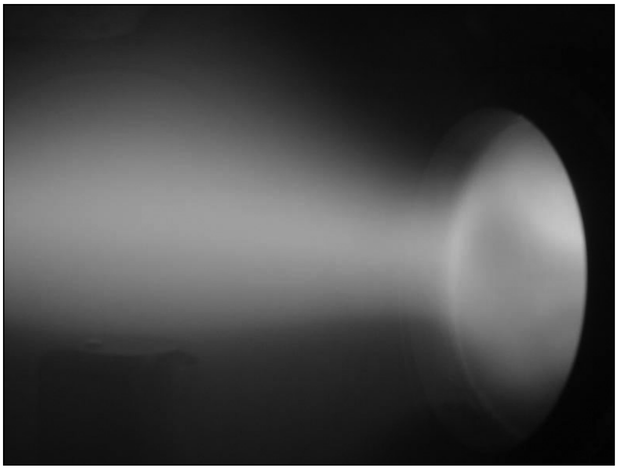

Fig. 3. Carbon plasma flux in vacuum-arc plasma source with linear filter of microparticles

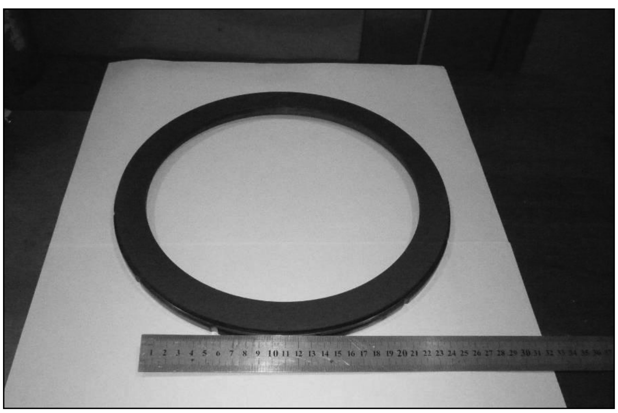

Fig. 4. Ring seals of DRGS system after being coated with diamond-like material

up 4% which is 1.5—2 times higher than that of the world counterparts.

The plasma source provides a sufficiently high deposition rate, up to 5 mm/h for diamond-like coatings and up to 20 mm/h for titanium layer.

In order to apply diamond-like coatings on silicon carbide a research process was designed on the basis of the results of previous studies. The stepwise coating synthesis includes the following steps:

- To clean the ring surface by pulse glow discharge in argon;
- To bombard the surface with titanium ions to create a mixed transition layer;
- To apply a layer of titanium;
- To bombard the surface with carbon ions to create a mixed transition layer;
- To apply a layer of diamond-like carbon.

The coating process research was carried out on silicon carbide samples with a diameter of 15 mm and a thickness of 6 mm. The diamond-like layers were deposited at a graphite cathode arc current of 70 A and an argon pressure of $1 \cdot 10^{-2}$ Pa. Maximum coating hardness (about 50 GPa) was established to be reached at a pulse duration of 6 μs, a pulse repetition frequency of 1.3 kHz, and an amplitude of 0.5—1 kV.

After determining the optimal modes of deposition a diamond-like coating was applied to the surface of DRGS friction rings made of silicon carbide. Coated rings (Fig. 4) have successfully passed the bench tests and demonstrated high performance at the pressure above 100 kg/cm^2. They will be used by *Grace Engineering* R&P Corporation in the experimental samples of DRGS for field tests.

2. SECTION FOR PRODUCTION OF LAMINATED COMPOSITES

The implementation of section for production of laminated composites within the KhIPT experimental and technological complex is a remarkable milestone in the long history of development of hot vacuum rolling technique (HVR). The KhPTI has been carrying out systematic research in this area since the early 1950s, when for the first time in the world practice of metal shaping the KhIPT NRC created pilot samples of vacuum rolling mills [7—9].

As of today, dozens of types and kinds of bimetal and laminated composites with different functions have been created. Some of them have found practical application [10—15]. Figure 5 shows some typical examples of laminated composites.

The HVR technique for welding of various combinations of layers of almost any metal in the solid phase is implemented in such a way as the original stack of plates of welded metal is heated and rolled in vacuum with a certain compression.

The weld is formed during collective stack deformation in the strain center. To produce bimetal and laminated composites the vacuum rolling mills and highly effective special vacuum roll-welding plants are used. Figure 6 shows a vacuum roll-welding plant (SVAPR) designed by the KhIPT, which produce 200 tons of bimetal and laminated composites annually.

One of the promising areas of application of HVR technique for creating new functional materials is the development and study of radio-protective (RP) laminated composites. The importance of this trend is caused by exhausted potential of traditional materials for creating on their basis the RP structures whose effectiveness would provide the opportunity to develop improved and new products to be widely used in various fields of human activity.

In particular, new RP materials that are more effective than the traditional ones are necessary for aerospace systems (protection of space stations, communication and navigation satellites, and cockpits of high-altitude aircrafts from cosmic radiation, etc.); mobile nuclear energy sources; personnel engaged in medical and engineering works associated with the use or treatment with a variety of ionizing radiation (IR) sources and so on. The absence of such materials that would fully meet the present-day and projected requirements for RP characteristics hinders the mankind advancement to the outer space, the search of new energy sources, and the protection from the IR harmful effects, etc.

There are two general approaches to addressing the problem of counteracting the IR impact on biological and semiconductor objects. The *first* approach is aimed at finding ways to utilize their natural ability to withstand the impact of IR, to improve this ability through the use of radio-protective substances, and to create new radiation-resistant semiconductor materials [16, 17]. The *second* one is directed towards developing RP structures based on special materials that can radically reduce the intensity of IR falling on the objects protected. This approach is not an alternative to the first one; it is more general in nature, insofar as it makes it possible to reduce radiation burden on both the electronic and the biological objects. It is very important that RP materials integrated in certain design can be used as structural materials.

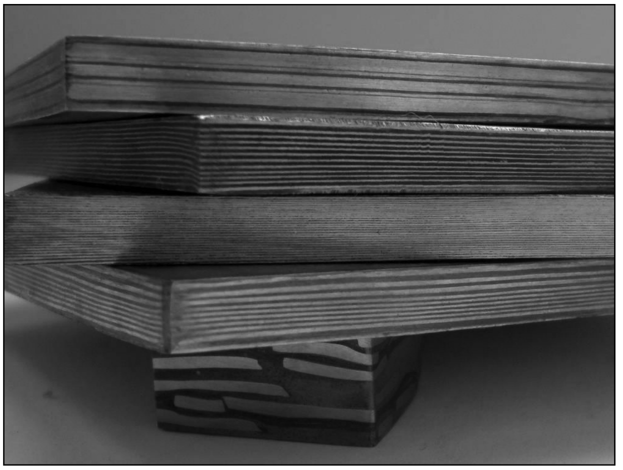

Fig. 5. Typical samples of laminated composites

Fig. 6. Vacuum roll-welding plant (SVAPR)

The main drawbacks of RP structures that have been used so far in nuclear power plants and radioactive waste deposits are their significant mass and size [18, 19]. They make it difficult or even impossible, in many cases, to use known RP structures, for example, in spacecraft, airplanes, mobile power plants, and so on. The above shortcomings are caused by low effectiveness of traditional homogeneous RP substances and materials. Therefore, to overcome this problem it is necessary to create new, innovative RP materials.

One of the promising directions of development of new highly effective RP materials is the creation of laminated spatial structures consisting of interconnected layers of two or more homogeneous metals with different atomic number *Z*. The heavy metals (elements with large *Z*) as compared with the light metals (elements with small *Z*) have not only better RP properties, but also much higher specific density. This correlation of natural characteristics of metals from these two groups does not allow the researchers to make both lightweight and highly effective radio- protective structures either of the heavy or of the light metals.

Combining the matters with small and large *Z* in one heterogeneous structure creates conditions for the multiple reflection of ionizing particles and gamma rays from the interface of these substances and for return of photons to the layers filled with substance having a high absorptive capacity.

Due to its specific design, this structure is a kind of trap for photons and therefore has a higher safety in comparison with the homogeneous substances of which it consists. In other words, the idea of this approach is that in heterogeneous materials to those mechanisms of scattering at the atomic level, which are inherent in the homogeneous substances, such mechanisms that can be considered structurally determined are added.

The results of calculations and analytical study of X-ray and γ-radiation photon [20] and high-energy electron [21] flux propagation shows that the criterion for the best choice of materials for laminated structures is the biggest difference in reflection factors (*albedos*) of adjacent layers. This criterion is met by light and heavy metals with low and high *Z*, respectively.

In general, it should be recognized that the absence of reliable data on interface albedo of different combinations of metals that can be composite constituents leads to the difficulties in computing the effectiveness of particular RP composite depending not only on its composition, but also on the thickness and the number of layers.

In this context, given the KhIPT opportunities for obtaining various laminated composites and the capacity of station for testing of RP characteristics of composites and other materials, which was organized as part of the experimental-technological complex, we have performed a series of experimental works to carry out a comprehensive study of the RP characteristics of laminated composites made of light and heavy metals.

The implementation of this project was preceded by experiments on the transmission of monochromatic electron beam with energy of 2.5 MeV through the samples of laminated composites «*light metal — heavy metal*». The components of these composites were aluminum (Z_{Al} = 13) and lead (Z_{Pb} = 82). To provide the experiment with materials to be studied a special technique of alternate rolling in vacuum and rolling at normal atmospheric pressure was developed. Using this technique the samples of Al-Pb composites with different internal architecture were made. Their characteristics are given in Table 1.

It is necessary to obtain composite strips of various thickness and to fit the thickness of aluminum strip insofar as correct comparison of RP effectiveness of materials having different composition and structure requires the samples of the same surface density χ which is the product of bulk density ρ of particular strip and its thickness *h*. Thus, to vary χ of plate made of certain material with density ρ it is necessary to change its thickness *h*. All the samples used in the previous experiments with irradiation had $\chi = 0.5$ g/cm^2.

In the previous experiments the source of high-energy electrons was *ELIAS* electrostatic electron accelerator (manufactured by *High Voltage Engineering Corporation*, model KS/3000) which allowed us to obtain electron beams with energy from 0.5 to 3 MeV at current from 1 to 500 mA.

A beam of accelerated electrons defocuses to a diameter of about 10 cm with the help of magnetic lenses (not showed in the chart). The copper collimator located on its way cuts from the central part of defocused beam a bundle having a section of approximately 1.5 × 1.5 mm^2. The bundle goes through the exit window of electron flux guide and falls on the sample behind which the detector is located. In order to reduce the influence of gamma background generated by accelerator on the measurements, the sample and the detector are protected with a wall of lead blocks. The energy of electrons on the sample surface is 2.5 MeV.

The energy of the electrons that passed through the test material and of the braking gamma radiation generated by the electron beam was gauged using detectors based on semiconductor wide-band CdZnTe compound developed and manufactured at the NRC of KhIPT. These detectors can operate in the current mode at room temperature.

The total energy ^{pen}E of electrons and γ-rays that penetrated through the sample was measured during the experiments. The radio-protective effectiveness was evaluated by energy ^{abs}E absorbed and reflected by the sample. To compare the properties of composites and aluminum the effectiveness ratio $K_{eff} = {}^{abs}E_{comp}/{}^{abs}E_{Al}$ was used. It is equal to 1 for aluminum. The experiment results are given in Table 1.

To facilitate the analysis of experimental data and the conclusions let us conditionally divide the investigated composite samples into two groups: the first group is the samples no. 2 and no.3 both having a similar asymmetric structure; the second group consists of the samples no. 4 and no. 5 having a symmetric structure.

The first group samples are cut from the same composite strip, but face the incident beam with light metal layer (sample no. 2, «*light metal — heavy metal*») and with heavy metal layer (sample no. 3, «*heavy metal — light metal*»). They demonstrated different RP effectiveness, with the difference of ^{abs}E values (0.04 MeV) being slightly larger than the measurement error. The «*light-heavy*» scheme has been experimentally established to have better RP properties as compared with the «*heavy-light*» one. This result is consistent with the calculation and analytical studies presented in [20] and implies that increasing the number of composite layers (hence, the number of *light-heavy* interfaces) leads to growing the RP effectiveness of composites at a constant volumetric share of light metal in the sample.

No similar comparison of data for the second group of samples is possible because the volumetric shares of aluminum in the samples are significantly different. However, it should be noted that as the share of aluminum in the composites decreases their radio-protective effectiveness increases.

To comprehensively assess the second group samples it is necessary to point out that despite a lower value of ^{abs}E (as compared with the sample no. 5) for the sample no. 4 the composite of this

Table 1

Parameters of Internal Architecture and RP Properties of Samples Used in the Previous Research

Parameters	Value of parameter				
Number of sample	1	2	3	4	5
Composition of sample	Al	Al—Pb	Pb—Al	Al—Pb—Al—Pb—Al	Pb—Al—Pb—Al—Pb
Thickness of sample, mm	1.85	1.15	1.15	1.00	0.78
Share of aluminum in sample, α_{Al}	1.00	0.81	0.81	0.73	0.56
Bulk density of sample ρ, g/cm^3	2.7	4.36	4.36	5.0	6.47
Total energy ^{pen}E of γ quants and electrons which penetrated through the sample, MeV	1.35	0.80	0.84	0.78	0.68
Total energy ^{abs}E of γ quants and electrons absorbed by the sample, MeV	1.15	1.70	1.66	1.72	1.82
Effectiveness, K_{eff}	1.00	1.48	1.44	1.50	1.58

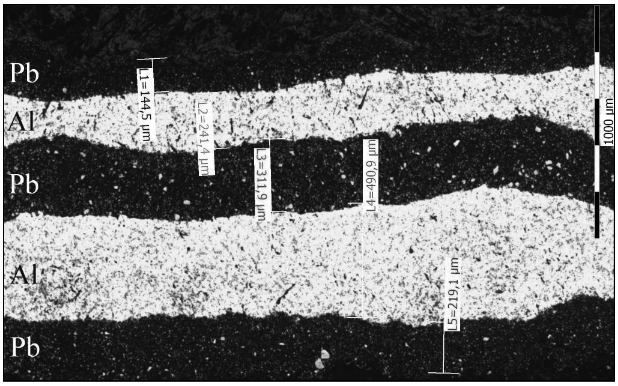

Fig. 7. Typical microstructure of 5-layered Al-Pb composite

type has an advantage over the other composites investigated.

Firstly, the outer layers of aluminum provide the composite with better corrosion resistance and prevent the toxic lead from contacting with environment. *Secondly*, one can predict that the mechanical properties of this composite will be better than the properties of composite that has a higher RP effectiveness, but contains a large volumetric share of lead which possesses lower strength as compared with aluminum (sample no. 5).

Thus, the results of previous studies confirmed the qualitative theoretical predictions about the RP effectiveness of composites «*light metal — heavy metal*»: the composites are 1.4—1.5 times more effective than aluminum. Proceeding from the analysis of these results and taking into account the existing experience of development of research and engineering technologies of many different types of laminated composites the symmetrical composition Al-Pb-Al-Pb-Al was selected as base type of RP composite internal architecture.

It is impossible to determine on the basis of data on experimental models used in the previous studies (see Table 1) which share of lead α_{Pb} the composites should contain to ensure an acceptable level of RP properties. This is caused by the fact that these samples have not only significantly different α_{Pb}, but also dissimilar internal architecture. Therefore, at the initial stage, it was decided to produce a series of composite strips with $\alpha_{Pb} \approx 0.15 + 0.5$ using initial packages with different α_{Pb}.

The general scheme of manufacture of this series of strips was the same as for the manufacture of samples in the previous studies described above. These composites were used for metallographic purposes, for determination of α_{Pb}, and for assessment of their RP properties.

The metallographic studies were carried out in accordance with standard optical metallography techniques using a *GX-51* toolkit. Neither structural defects in the form of local discontinuities of Al and Pb interfaces, nor third phases in Al-Pb interfaces were revealed. However, these interfaces were found to be largely undulated and to have uneven thickness of Al and Pb layers, which is a characteristic feature of laminated composites consisting of metals having significantly different strain resistances (see Fig. 7). Therefore, the determination of total thickness of Pb layers in composites and α_{Pb} by metallographic methods is considered impossible.

In this regard, it was decided to determine α_{Pb} by calculating on the basis of mixture principle proceeding from experimental data on bulk density ρ_{comp} obtained by the hydrostatic weighing technique [22]. The data on structure, size, and density of composite samples selected for the further study of PR properties are showed in Table 2.

The initial package of similar design composed of three Al plates each having a thickness of 0.67 mm and two Pb strips each having a thickness of 1.0 mm (i.e. the total thickness of package was 4 mm were used to produce a pilot batch of composite strips. The pilot batch of PR composites consisted of strips of two sizes. The data on structure, size, and density are given in Table 3. The visual appearance of several strips of the pilot batch is showed in Fig. 8.

3. SECTION FOR TESTING OF RP CHARACTERISTICS OF METALLIC AND NONMETALLIC COMPOSITES

The feasibility of inclusion of section for testing of RP characteristics to the experimental and technological complex at the NRC of KhIPT is caused by the following reasons. *Firstly*, as of today, several applications from research and industrial organizations of aerospace industry (including *Dniprotechservice* R&P Corporation) have made requests on conducting tests of RP characteristics of materials used in the products of these organizations. In addition, representatives of many organizations have repeatedly expressed their interest in such tests during their visits to KhIPT. *Secondly*, the organization of section for production of RP laminated composites within the framework of experimental and technological complex at the NRC of KhIPT requires monitoring and comprehensive certification of working characteristics of these materials, primarily, their RP properties. *Thirdly*, researchers of KhIPT NRC have developed a series of elementary particle accelerators and accumulated an extensive experience in conducting studies of IR impact on various materials, i.e. there are favorable preconditions for the organization and successful operation of station for testing of RP characteristics.

It should be noted that all the techniques for studying the RP characteristics and the gradual radiation-stimulated modification of properties of substances and materials under the action of IR can be divided into two types on the basis of the type of IR source: a) those that use natural sources (radioactive substances) and b) those that use various devices for controlled acceleration of charged particles and generation of neutrons.

Fig. 8. Strips of PR Al—Pb composites included into pilot batch

Table 2

Structure, Size, and Density of Samples Used in Further Study of RP Effectiveness of Composites

No. of sample	Thickness, mm	Bulk density ρ, g/cm^3	Volumetric share, α		Surface density χ, g/cm^2		
			Pb	Al	Pb	Al	Σ
1	0.51	4.0	0.16	0.84	0.09	0.12	0.21
2	0.69	4.0	0.16	0.84	0.12	0.16	0.28
3	0.90	4.0	0.16	0.84	0.16	0.20	0.37
4	1.13	4.0	0.16	0.84	0.20	0.26	0.46
5	1.29	4.0	0.16	0.84	0.23	0.29	0.53
6	0.33	5.3	0.30	0.70	0.11	0.06	0.18
7	0.48	5.3	0.30	0.70	0.16	0.09	0.25
8	0.61	5.3	0.30	0.70	0.21	0.12	0.32
9	0.73	5.3	0.30	0.70	0.25	0.14	0.39
10	0.88	5.3	0.30	0.70	0.30	0.17	0.47
11	0.28	6.6	0.45	0.55	0.14	0.04	0.18
12	0.41	6.6	0.45	0.55	0.21	0.06	0.27
13	0.51	6.6	0.45	0.55	0.26	0.08	0.34
14	0.59	6.6	0.45	0.55	0.30	0.09	0.39
15	0.73	6.6	0.45	0.55	0.37	0.11	0.48

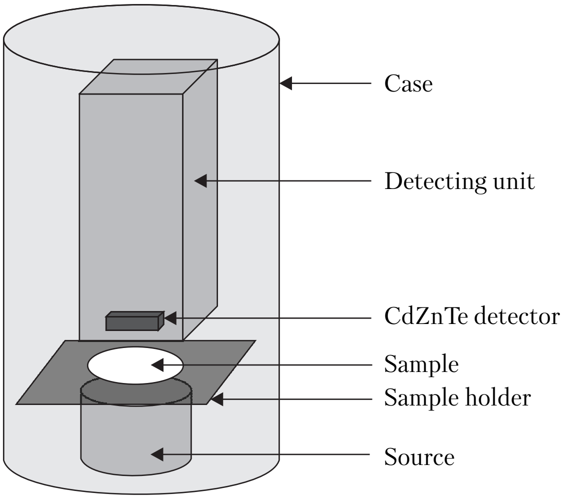

Fig. 9. Diagram of test unit of bench for testing of RP properties

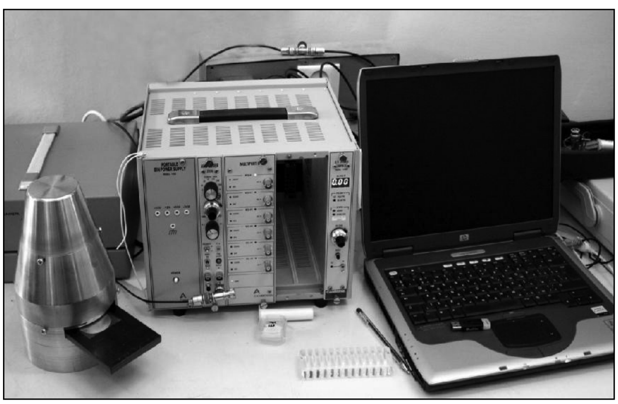

Fig. 10. Test bench for testing of RP properties of materials

To test the RP properties, namely, the ability of laminated composites of different structure to attenuate the energy of electron flux we used both types of IR sources and, accordingly, two methods: one of them involves the use of electron accelerator and the other practices the use of radionuclide β-emitter as source of electrons.

The section for testing of radio-protective characteristics is based on the radioisotope technique. The reasons for this are as follows:

- Possibility to create more realistic conditions of radiation load on the samples due to a continuous spectrum of the electron energy from radioactive source within the range from several dozens of keV to several MeV in contrast to the energy of monochromatic flux of accelerated electrons having an energy of several MeV;
- Less sophistication, size, and power consumption of hardware solution for the radionuclide technique as compared with the accelerator method;
- Lower total cost of preparation and testing per one sample, i.e. higher productivity of the radionuclide technique as compared with the accelerator one.

The KhIPT research and technical expertise in the sphere of radiation materials, as well as available hardware and auxiliary equipment were used as basis for the organization of section for testing of RP properties and for the measurement of different types of IR. Semiconductor CdZnTe-detectors and electron detecting units have been designed and manufactured.

The test bench consists of test unit and spectrometric path for processing, visualization, and archiving of detector signals. The general diagram of the test unit which is a key element of equipment of the section for testing of RP properties is showed in Fig. 9. The general view of the test bench is given in Fig. 10.

The main feature of the test bench is unequivocal geometric arrangement of its elements (*radiation source – material tested – radiation detector*) with a minimum distance between them, which is kept when changing the samples. This allows the researchers to obtain reproducible results for the determination of material ability to attenuate radiation depending on the sample thickness. The test unit consists of a) metallic case; b) detecting unit with a CdZnTe-detector, a thin (50 micron) opaque beryllium input window, and a charge-sensitive pre-amplifier; c) sample, and d) source of electrons. The spectrometric path includes:

1) *Canberra 2026* amplifier-shaper;

2) *Canberra Multiport II* multichannel analyzer;

3) Analog-to-digital converter;

4) Personal computer with *Genie 2000* software; and

5) *Canberra 3106D* voltage source.

The developed radionuclide method and the design of the test bench meet the radiation safety requirements. The bench was certified at the KhIPT Radiation and Environment Research Laboratory.

Radionuclide source ^{90}Sr—^{90}Y is a pure β-emitter with the following chain of decay [23]:

$$^{90}\mathrm{Sr}\ (T_{1/2} = 28.74\ \text{years}) \rightarrow \rightarrow {}^{90}\mathrm{Y}\ (T_{1/2} = 64.1\ \text{years}) \rightarrow {}^{90}\mathrm{Zr}. \quad (1)$$

The aluminum and lead samples of various thickness, as well as samples of Al-Pb laminated composites obtained in the course of development technique for manufacturing RP composites were tested on the bench.

The spectral amplitude distributions were obtained for all samples. On the basis of the measurement results the electron attenuation coefficients were calculated for different materials. The attenuating properties were calculated for the energy flux measured by detector and transferred through it by electrons and braking γ-quanta after passing through the materials tested as:

$$W = E_{adc} \sum_i i \times N_i, \quad (2)$$

where i is number of ADC channel, N_i is number of pulses in the channel, E_{adc} is ADC channel division in units of energy. The coefficient of attenuation of electron flux in the material as compared with that in the air is taken as $k = 1 - W_M/W_P$.

The results, their detailed analysis and discussion are given in Section 4. The RP characteristics of samples of Al-Pb composite strips from the pilot batch have been tested.

In addition to the experimental studies, the calculative and analytical study of electron flux passage through the layers of Al, Pb and Al-Pb composites has been carried out with the use of Monte Carlo method. The comparison of the experimental results with the calculated ones has showed their satisfactory correlation.

4. RESULTS OF TESTING OF RP COMPOSITE FUNCTIONAL CHARACTERISTICS

The RP characteristics of samples of Al-Pb laminated composite system and samples of aluminum and lead strips were tested by two different methods, *the radioisotope* and *the accelerator* ones. They differ by types of IR sources with different energy IR spectrum, as well as by methods of record of IR flux intensity, data analysis, and interpretation.

The following array of data on the RP effectiveness of composites was obtained using the radioisotope technique, while the second method, the accelerator technique, was used only for sampling verification of data and dependencies.

The comparison of dependencies of RP composite effectiveness on internal architecture parameters obtained by the first and the second methods has demonstrated their full qualitative correlation. The quantitative comparison of data obtained by these methods is impossible due to the above mentioned differences.

Before analysis and discussion of results on the composite RP properties it is necessary to interpret clearly the definition of *specific density*. As mentioned above, it is possible to compare the RP effectiveness of only those dissimilar materials that have the same surface density χ which is the product of bulk density ρ of particular strip on its thickness h and is measured in units of g/cm^2. This abstract value has a quite simple and practical interpretation: χ is a weight of plate made of material with a density of ρ g/cm^3 and having a thickness of h cm and an area of 1 cm^2. In other words,

Table 3

Structure, Size, and Density of Strips from Pilot Batch

No. of parameter	Controlled parameter	Value of parameter for strip with a thickness of	
		0.9 mm	1.5 mm
1	Number of aluminum layers	3	3
2	Number of lead layers	2	2
3	Thickness of strip, mm	0.9	1.52
4	Width of strip, mm	80	100
5	Length of strip, mm	200	300
6	Specific density of composite, g/cm^3	6.5	6.5

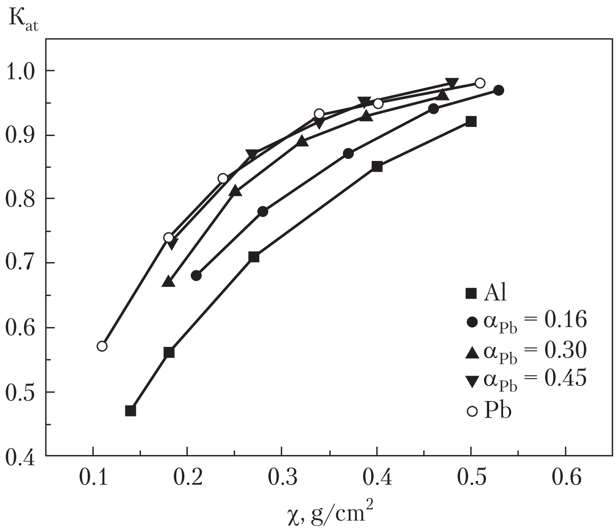

Fig. 11. Surface density dependence of attenuation factor K_{at} for composites with different volumetric share of lead α_{Pb}, as well as for Al and Pb

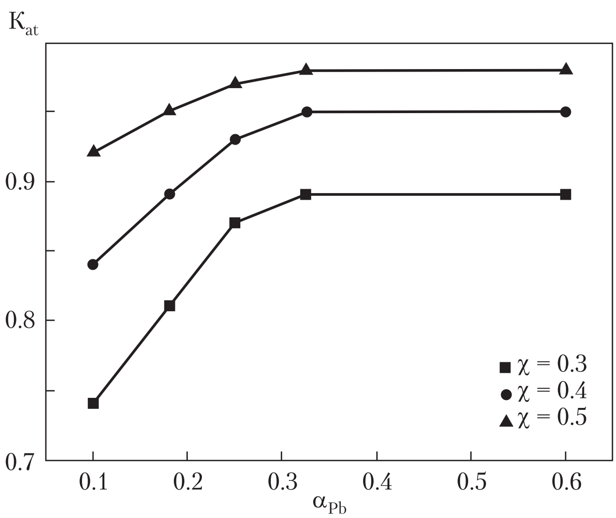

Fig. 12. Dependence of attenuation factor K_{at} for composites with different surface density χ on volumetric share of lead α_{Pb} in them

χ describes the number of substance atoms met by the IR stream on its way through this substance.

Among RP materials with the same value of χ the most effective is that material which has the largest attenuation factor K_{at}. Among the materials with the same value of K_{at} the most effective is the one which has the smallest value of χ. Thus, the χ parameter is a very important characteristic of the material in terms of its RP properties.

In addition to the data contained in Table 2, Table 4 shows the attenuation and transmission coefficients of composite samples, K_{at} and K_{trans}. The attenuation coefficient K_{at} increases as surface density χ and volumetric share of lead α_{Pb} grow. For a visual presentation of K_{at} dependencies on χ and α_{Pb} and their detailed analysis the respective graphs are given in Fig. 11 and 12 built on the basis of data contained in Table. 4.

Obviously, all these dependencies are nonlinear. Let us analyze the data on the effect of χ on K_{at}. It is a well-known fact that the RP ability of lead is higher than that of aluminum [18, 19] (see Fig. 11: the curve $K_{at} = f(\chi)$ for lead is above the curve for aluminum within the whole range of χ under review).

When comparing the RP effectiveness of composites with that of metals that are the components of composite materials (aluminum and lead), usually, aluminum is chosen as a reference metal, inasmuch as aluminum is a widely used structural material in aerospace industry that is the most interested in unconventional highly effective RP materials ensuring high protection from IR and reduction of mass of aircrafts and space vehicles.

As for lead, this metal does not belong to widely used construction materials because of its high toxicity, poor mechanical properties, and low corrosion resistance. Therefore, for estimating the RP effectiveness of composites we chose aluminum as an alternative material to the composites.

Unlike the previous estimates of composite RP effectiveness the algorithm for evaluating it in this project was as follows. For three selected values of attenuation factor K_{at} (i.e. $K_{at}1 = 0.75$, $K_{at}2 = 0.85$, and $K_{at}3 = 0.95$) the values of surface density of χ_i of aluminum and composites with different share of lead α_{Pb} corresponding to the attenuation coefficient K_{at} were determined (see Fig. 11). Thereafter, the ratio χ_i/χ_{Al} and the relative difference $(\chi_{Al} - \chi_i)/\chi_A$ were calculated. The data presented in Table 5 show that for the same at-

Table 4

Structure, Size, Density, and RP Properties of Composite Samples Studied by Radioisotope Technique

No. of sample	Thickness, mm	Bulk density ρ, g/cm³	Volumetric share of lead, α_{Pb}	Surface density χ, g/cm²			Coefficient	
				Pb	Al	Σ	Transmission, K_{trans}	Attenuation, K_{at}
1	0.51	4.0	0.16	0.09	0.12	0.21	0.32	0.68
2	0.69	4.0	0.16	0.12	0.16	0.28	0.22	0.78
3	0.90	4.0	0.16	0.16	0.20	0.37	0.13	0.87
4	1.13	4.0	0.16	0.20	0.26	0.46	0.06	0.94
5	1.29	4.0	0.16	0.23	0.29	0.53	0.03	0.97
6	0.33	5.3	0.30	0.11	0.06	0.18	0.33	0.67
7	0.48	5.3	0.30	0.16	0.09	0.25	0.19	0.81
8	0.61	5.3	0.30	0.21	0.12	0.32	0.11	0.89
9	0.73	5.3	0.30	0.25	0.14	0.39	0.07	0.93
10	0.88	5.3	0.30	0.30	0.17	0.47	0.04	0.96
11	0.28	6.6	0.45	0.14	0.04	0.18	0.27	0.73
12	0.41	6.6	0.45	0.21	0.06	0.27	0.13	0.87
13	0.51	6.6	0.45	0.26	0.08	0.34	0.08	0.92
14	0.59	6.6	0.45	0.30	0.09	0.39	0.05	0.95
15	0.73	6.6	0.45	0.37	0.11	0.48	0.02	0.98

Table 5

Comparison of Surface Density χ_i of Composites with Different Share of Lead α_{Pbi} Corresponding to Selected Values of Attenuation Factor K_{ati}, and Surface Density of Aluminum χ_{Al}

K_{ati}	χ_i	α_{Pbi}			
		α_{Al}	$\alpha_1 = 0.16$	$\alpha_2 = 0.30$	$\alpha_3 = 0.45$
0.75	χ_i	0.31	0.26	0.22	0.18
	χ_i / χ_{Al}	1.00	0.84	0.71	0.58
	Relative difference $(\chi_{Al} - \chi_i) / \chi_{Al}$, %	0	16	29	42
0.85	χ_i	0.40	0.35	0.29	0.26
	χ_i / χ_{Al}	1.00	0.88	0.73	0.65
	Relative difference $(\chi_{Al} - \chi_i) / \chi_{Al}$, %	0	13	28	35
0.95	χ_i	0.55	0.47	0.44	0.39
	χ_i / χ_{Al}	1.00	0.85	0.80	0.71
	Relative difference $(\chi_{Al} - \chi_i) / \chi_{Al}$, %	0	15	20	29

tenuation factor the mass of composites can be by 30—40% less than the mass of aluminum with equivalent protective characteristics. It should be noted that this algorithm makes it possible to determine the basic parameters of Al-Pb RP composite internal architecture (χ and α_{Pb}), which ensure the given value K_{at} or to predict the RP properties of composite with given value of χ.

CONCLUSIONS

1. The main outcome of the project is the creation of experimental and technological complex for mass production and testing of products and semi-finished goods made of diamond-like and laminated composites.

2. The results of activities carried out in the course of creating the experimental technological complex are as follows:

- An assembly for deposition of diamond-like coatings from filtered flux of vacuum arc carbon plasma on the samples with a diameter of 200 mm; a pilot process of application of diamond-like coating on the sealing elements of compressor equipment; a pilot batch of coated elements to be transferred to partner organization *Grace Engineering* R&P Corporation (according to the bench tests the pilot ring seals have showed high effectiveness at the pressure above 100 kg/cm^2);
- A pilot technique for the manufacture of radio-protective composites; a pilot batch of composite strips to be given to partner organization *Dniprotechservice* R&P Corporation;
- A test bench for experimental testing and certification of radio-protective characteristics of various composites and other materials; a method for testing of radio-protective characteristics using a radionuclide source ^{90}Sr + ^{90}Y.

3. The scientific results of the project are as follows:

- The distribution of diamond-like coating thickness in the plane perpendicular to the axis of outgoing plasma flux has been studied. The coating deposited at a distance of 220 mm from the filter outlet on diameter of 180—200 mm has uniform thickness; the deposition rate is 5 mm/hour;
- The dependence of IR attenuation factor K_{at} of composites on the parameters of their internal architecture, i.e. surface density and volumetric share of lead, has been established;
- The radio-protective effectiveness of composites has been established to be higher by 30—40 % than that of aluminum.

REFERENCES

1. Strelnitskii, V.E. and Aksenov, I.I. (2006). Films of Diamond-Like Carbon. Kharkiv: IPP Contrast (in Russian).
2. Aksenov, I.I., Andreev, A.A., Belous, V.A. et. al. (2012). Vacuum Arc: Plasma Sources, Deposition of Coatings, Surface Modification. Kyiv: Naukova Dumka (in Russian).
3. Aksenov, D.S., Aksenov, I.I., and Strelnitskii, V.E.: Suppression of Emission of Macro-particles in Vacuum Arc Plasma Sources. *Problems of Atomic Science and Technology. Physics of Radiactive Damages and Radiactive Materials Science,* 6, 106—115 (2007) (in Russian).
4. Vasiliev, V.V. and Strelnitskii, V.E.: Method for Transport of Vacuum Arc Cathode Plasma with Filtration from Micro-particles and Device for Its Realization. Patent of Ukraine no. 97584. *Bulletin*, 4 (2012) (in Ukrainian).
5. Vasiliev, V.V. and Strelnitskii, V.E.: Anode Node of Vacuum arc Source of Cathode Plasma. Patent of Ukraine no. 101443. *Bulletin*, 6 (2013) (in Ukrainian).
6. Vasiliev, V.V. and Strelnitskii, V.E.: Vacuum Arc Evaporator for Generation of Cathode Plasma. Patent of Ukraine no. 101678. *Bulletin*, 8 (2013) (in Ukrainian).
7. 50 Years of Kharkiv Institute for Physics and Technology of AS of USSR. Kyiv: Naukova Dumka (1978) (in Russian).
8. Krupin, A.V., Linetskii, B.L., Zarapin, Yu.A. et.al. (1972). Vacuum Rolling Mills. Moscow: Machine-Building (in Russian).
9. Ivanov, V.E. and Kovtun, S.F.: Vacuum Rolling of Chemically Active Metals. *Nonferrous Metals*, 11,. 15—17 (1962) (in Russian).
10. Krupin, A.V. (1974). Metal Rolling in Vacuum. Moscow: Metallurgy (in Russian).
11. Nekliudov, I.M., Didenko, S.Yu., Ilchenko, N.I. et. al.: Creation of Laminated Composites by Hot Rolling in Vacuum. *Works of Research and Practical Symposium «Equipment and Technologies, Heat Treatment of Metals in Machine-Building»*, 75—80 (2000) (in Russian).
12. Ilchenko, N.I., Didenko, S.Yu., Nekliudov, I.M., et. al.: Obtaining Laminated Materials and Composites of Copper-Steel Type by Hot Rolling in Vacuum and Studying Their Properties. *Problems of Atomic Science and Technology*, 3, 158—160 (2003) (in Russian).
13. Voevodin, V.N., Didenko, S.Yu., Ilchenko, N.I., et. al.: Thermo-activated Evolution of Structural Phase Condition of Transition Zones in Bimetals St3-12X18 H10T. *Problems of Atomic Science and Technology*, 2, 164—168 (2008) (in Russian).
14. Nekliudov, I.M., Voevodin, V.N., Didenko, S.Yu., et. al.: Prospects for Production and Use of Metallic Microlaminates Obtained by Vacuum Rolling. *Problems of Atomic Science and Technology*, 5, 89—94 (2010) (in Russian).
15. Voevodin, V.N., Didenko, S.Yu., Ilchenko, N.I., et. al.: Mechanical Properties and Structure of Copper-Steel

Microlaminates. *Problems of Atomic Science and Technology,* 5, 95-101 (2010) (in Russian).
16. Kudriashov, Yu.B. (2004). Radiation Biophysics (Ionizing Radiation). Moscow: Fizmatlit (in Russian).
17. Korshunov, F.P., Bogatyrev, Yu.V., and Vavilov V.A. (1996). Influence of Radiation on IC Chips. Minsk: Nauka i Tekhnika (in Russian).
18. Aleshin, V.S. and Sarkisov, A.A. (1961). Energy Nuclear Reactors. Leningrad: Sudpromgiz (in Russian).
19. Mashkovich, V.P. (1982). Protection from Ionizing Radiation. Moscow: Energoatomizdat (in Russian).
20. Belous, V.A., Komarov, A.O., and Shiliaev, B.A.: Thin-Film Laminated Screens to Protect from X-rays and Gamma Radiation. *Problems of Atomic Science and Technology. Vacuum, Pure Materials, and Semiconductors*, 1, 202—204 (2008) (in Russian).
21. Borts, B.V., Marchenko, I.G., and Bezdvernyi, P.N.: Modeling of Electron Passage through Laminated Composite. *Problems of Atomic Science and Technology. Physics of Radiactive Damages and Radioactive Materials Science*, 4, 2, 175—177 (2009) (in Russian).
22. Gauzner, S.I., Kivilis, S.S., and Osokina, A.P. (1972). Measurement of Mass, Volume, and Density. Moscow: Standard Publication (in Russian).
23. LBNL Isotopes Project — LUNDS Universitet. http://ie.lbl.gov/toi/index.asp.

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ОРГАНИЗАЦИЯ ЭКСПЕРИМЕНТАЛЬНО-ТЕХНОЛОГИЧЕСКОГО КОМПЛЕКСА ДЛЯ СЕРИЙНОГО ИЗГОТОВЛЕНИЯ И ТЕСТИРОВАНИЯ ИЗДЕЛИЙ И ПОЛУФАБРИКАТОВ ИЗ АЛМАЗОПОДОБНЫХ И МЕТАЛЛИЧЕСКИХ СЛОИСТЫХ КОМПОЗИТОВ ДЛЯ ВЕДУЩИХ ОТРАСЛЕЙ МАШИНОСТРОЕНИЯ

Создан экспериментально-технологический комплекс, в состав которого входят следующие участки: нанесения покрытий из алмазоподобных наноструктурированных композитов; изготовления радиационно-защитных и других типов слоистых металлических композитов; тестирования радиационно-защитных характеристик материалов. Модернизировано технологическое оборудование и стенд для тестирования радиационно-защитных характеристик материалов. По разработанным опытно-промышленным методикам изготовлены партии кольцевых уплотнений большого диаметра с алмазоподобным покрытием из многослойных композитов Al—Pb. Экспериментально доказано превышение радиационно-защитной эффективности композитов на 30—40 % в сравнении с алюминием.

Ключевые слова: методы вакуумно-дуговой и вакуумной прокатки, ускорительная и радиоизотопная методики, алмазоподобное покрытие, многослойные композиты.

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ОРГАНІЗАЦІЯ ЕКСПЕРИМЕНТАЛЬНО-ТЕХНОЛОГІЧНОГО КОМПЛЕКСУ ДЛЯ СЕРІЙНОГО ВИГОТОВЛЕННЯ ТА ТЕСТУВАННЯ ВИРОБІВ І НАПІВФАБРИКАТІВ ІЗ АЛМАЗОПОДІБНИХ ТА МЕТАЛЕВИХ ШАРУВАТИХ КОМПОЗИТІВ ДЛЯ ПРОВІДНИХ ГАЛУЗЕЙ МАШИНОБУДУВАННЯ

Створено експериментально-технологічний комплекс, до складу якого входять такі дільниці: нанесення покриттів із алмазоподібних наноструктурованих композитів; виготовлення радіаційно-захисних та інших типів шаруватих металевих композитів; тестування радіаційно-захисних характеристик матеріалів. Модернізовано технологічне обладнання та стенд для тестування радіаційно-захисних характеристик матеріалів. За розробленими дослідно-промисловими методиками виготовлено партії кільцевих ущільнювачів великого діаметру з алмазоподібним покриттям із багатошарових композитів Al–Pb. Експериментально доведено перевищення радіаційно-захисної ефективності композитів на 30—40 % порівняно з алюмінієм.

Ключові слова: вакуумно-дуговий метод, метод вакуумної прокатки, прискорювальна та радіоізотопна методики, алмазоподібне покриття, багатошарові композити.

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