NUCET NUCLEAR ENERGY AND TECHNOLOGY

Nuclear Energy and Technology 4(2): 127–133
DOI 10.3897/nucet.4.30771

Research Article

Potential of application of IRT-T research reactor as the solution of the problem of graphite radwaste disposal*

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Academic editor: *Elena Artyushkova* ♦ **Received** 23 October 2018 ♦ **Accepted** 12 November 2018 ♦ **Published** 26 November 2018

Citation: Pavlyuk AO, Kotlyarevsky SG, Bespala EV, Bespala YR (2018) Potential of application of IRT-T research reactor as the solution of the problem of graphite radwaste disposal. Nuclear Energy and Technology 4(2): 127–133. https://doi.org/10.3897/nucet.4.30771

Abstract

Aspects of handling irradiated graphite during decommissioning uranium-graphite reactors (UGR) of different types were investigated. It was demonstrated that handling reactor graphite is complicated by the presence in the composition of graphite of long-lived radionuclides, especially ^{14}C, which may get entrained in biological cycles since carbon constitutes one of the main components of biological chains. Practical implementation of the process of selective separation of ^{14}C can significantly reduce potential danger represented by graphite radioactive wastes due to the reduction of graphite activity as related to the isotope in question, as well as due to the reduction of the leaching rate by separating ^{14}C isotope which is the most weakly bound within the graphite structure. Conclusion was formulated that analytical measurement methodologies and calculation methods allow reliably estimating only the total quantity of ^{14}C accumulated in graphite, the contribution of ^{14}C accumulation channel from $^{13}C(n, \gamma)^{14}C$ reaction, as well as the total contribution of $^{14}N(n, p)^{14}C$ reaction on nitrogen impurities and on nitrogen contained in purge gas. Method was suggested for estimating the values of contributions of different channels of accumulation on nitrogen impurities and nitrogen contained in purge gas using IRT-T research reactor (Tomsk, Tomsk Region). Parallel irradiation of batches of samples of non-irradiated (fresh) reactor-grade graphite contained in different gaseous media constitutes the basis of the study. Algorithm was suggested for calculating contributions of all channels of ^{14}C accumulation according to the results of measurements to be obtained in the proposed studies. Recommendations were formulated on the use of all brands of graphite applied for manufacturing elements of graphite stacks of uranium-graphite reactors designed in Russia for determining selectively separated fraction of ^{14}C for all types of graphite radioactive wastes by the companies in the RF which operated (are operating) the uranium-graphite reactors. Time of exposure of samples of irradiated graphite in the GEK-4 horizontal experimental channel of the IRT-T reactor was calculated and was found to be equal to ~ 10 days. Methodology was suggested for conducting a series of experiments for determining the values of contributions of ^{14}C accumulation channels in the irradiated reactor graphite. The methodology suggested can be applied for determining fraction of selectively separated ^{14}C in irradiated graphite elements of practically all uranium-graphite nuclear reactors, including reactors operated abroad Russia, under the condition of maintaining carbon dioxide gas atmosphere in one of the irradiated containers.

* Russian text published: Izvestiya vuzov. Yadernaya Energetika (ISSN 0204-3327), 2018, n.1, pp. 87-98.

Keywords

Irradiated reactor-grade graphite; uranium-graphite reactor; research reactor; IRT-T

Introduction

Significant number of nuclear installations equipped with uranium-graphite reactors (UGR) of different types and intended for different purposes were put into operation on the territory of the Russian Federation during the period of establishment and development of nuclear industry and power generation including the following:

– 13 production uranium-graphite reactors (PUGR);
– AM-1 uranium-graphite reactor of the Obninsk NPP;
– two UGR AMB-100 and AMB-200 of the Beloyarsk NPP АЭС;
– four UGR of EGP type of the Bilibino NPP;
– eleven RBMK reactors of the Leningrad, Kursk and Smolensk NPPs.

Preparation is ongoing at present to decommissioning several already shut down reactors including PUGRs, reactors of the Beloyarsk and Obninsk NPPs. Operations were completed in 2015 in accordance with the project on decommissioning EI-2 PUGR (Pavliuk et al. 2018). Service life of the majority of NPP power units including the extension will expire in the nearest future during the period of about 10 – 15 years. Shutting down power units of the Leningrad NPP is tentatively planned to be implemented during the next few years as follows: Unit 1 – 2018, Unit 2 – 2020, Unit 3 – 2024, Unit 4 – 2026.

As applied to the operation of UGR, especially power units of nuclear power plants, handling irradiated reactor-grade graphite is one of the main tasks. Dynamics of accumulation of irradiated graphite with final decommissioning of UGR in the RF is presented in Fig. 1 (Romenkov 2011).

Resolution of the problem of handling reactor graphite is aggravated by the presence in the composition of graphite parts of long-lived radionuclides (^{14}C, ^{36}Cl, actinides). Notably, overall quantity of irradiated reactor graphite in Russia reaches ~60 thousand tons.

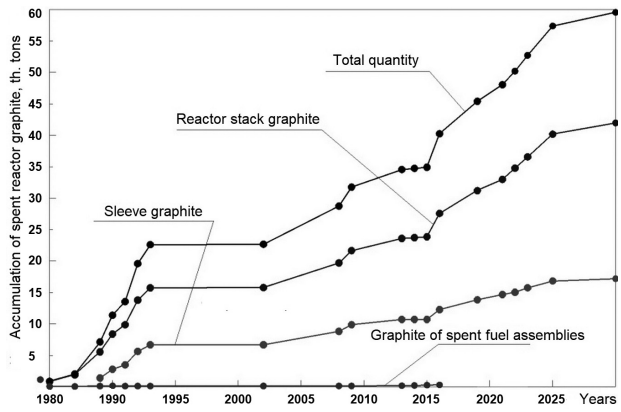

Figure 1. Forecast dynamics of accumulation of spent graphite in Russia. Sources: data from NIKIET (Romenkov 2011);

It has to be taken into consideration that the problem of spent reactor graphite is important for a number of other countries. About 250 UGR are built around the world and significant quantities of irradiated graphite were accumulated equal to around 230 – 250 thousand tons. Apart from Russia the problem of handling irradiated reactor graphite is important, in the first place, for the UK (more than 77 thousand tons), for the USA (more than 50 thousand tons) and for France (more than 23 thousand tons) (Dunzik-Gougar and Smith 2014, LaBrier and Dunzik-Gougar 2015, Liu et al. 2016). At present there is no coordinated approach to the solution of the problem of irradiated graphite disposal developed in the world (LaBrier and Dunzik-Gougar 2015).

The key problem determining complexity of handling irradiated graphite is the presence in graphite radioactive wastes of long-lived ^{14}C isotope ($T_{1/2}$ = 5730 years) which can be entrained in the biological processes since carbon is on the main components of biological chains. ^{14}C activity amounts to ~95 % of total activity of irradiated graphite and can reach the value in excess of 10^6 Bq/g for the most energy loaded areas of graphite stacks.

Specific features of accumulation of ^{14}C in reactor graphite

At first sight, the most "attractive" method for resolving problems associated with any radioactive wastes is the selective separation of radionuclides contained in them with minimum interference in the structure and chemical composition of material of radioactive wastes. Due to the fact that ^{14}C and ^{12}C isotopes represent isotopes of the same chemical element of which graphite is composed, separation of the "problematic" carbon isotope represents a technically and financially formidable task, similarly to all other processes associated with isotope separation. Methods of deep re-processing based on the transformation of graphite (including ^{14}C) in gaseous form with subsequent alkaline washing result in significant (up to 10 times) increase of volume of radioactive wastes. Other methods were investigated as well including the following: OMS method (oxidation in molten salts), plasma chemical processing and others, but, however, neither of these methods offers resolution of the main problem, namely the problem of ^{14}C.

However, there exist physical and chemical bases of selective separation of significant fractions of ^{14}C from

Table 1. Main reactions of ^{14}C formation in reactor graphite under neutron irradiation in UGR. Sources: data from (EPRI 2006).

Reaction	Cross-section σ, barn	Presence of isotope in natural element, %
$^{14}N(n, p)^{14}C$	1.81 ± 0.05	99.63 in natural N
$^{13}C(n, \gamma)^{14}C$	$(0.9 \pm 0.2)\cdot 10^{-3}$	1.07 in natural C

irradiated graphite without significant loss of mass of the main matrix material. Practical implementation of the process in question can significantly decrease the potential danger of graphite radioactive wastes due to the reduction of graphite activity with respect to the isotope under discussion and the rate of leaching by extraction of the weakest bound ^{14}C.

The process of generation of ^{14}C in graphite and its distribution in the structure of the material during UGR operation must be examined for estimating the possibility of implementation of the above approach.

Generation of ^{14}C nuclide in reactor graphite during reactor operation is determined by neutron activation of ^{14}N and ^{13}C isotopes (Table 1) (EPRI 2006).

Mechanisms of ^{14}C formation in graphite during UGR operation are following:

1. $^{13}C(n, \gamma)^{14}C$. ^{13}C is the carbon isotope contained in natural mixture of isotopes and is uniformly distributed in the graphite crystalline structure.
2. $^{14}N(n, p)^{14}C$. ^{14}N impurity (concentration in non-irradiated graphite is estimated to be within the range from 30 to 100 ppm) is contained in fresh graphite fractionally.

– In the form of solid substitution solution (uniformly distributed over the crystalline structure);
– In closed pores filled with air, as well as in the form of nitrogen adsorbed from air in the subsurface layer of pores and over crystallite boundaries.

Typical shape of distribution of impurity nitrogen in graphite in the subsurface layer is shown in Fig. 2 (IAEA 2007).

3. $^{14}N(n, p)^{14}C$. ^{14}N is contained in the composition of gaseous mixture blown through the graphite stack in the process of UGR operation and fills the whole volume of graphite pores available for gas. On the one hand, this leads to the adsorption of molecular nitrogen on the whole surface of graphite (including the external surface of pores, crystallites and microcrystallites), which may cause its subsequent activation with formation of ^{14}C. On the other hand, implantation of reaction products in the crystalline lattice occurs as the result of nuclear reaction with emission of proton and ^{14}C with recoil energy equal to 40 keV. Depth of implantation can reach ~ 60 nm (Fig. 3 (Vulpius et al. 2013)). Areas of ^{14}C localization will be concentrated within the thin (up to ~ 60 nm) subsurface layer of graphite products, pore surfaces accessible for gas, as well as graphite crystallites and microcrystallites.

The mechanism in question predetermines heterogeneous distribution in the graphite structure of ^{14}C isotope formed from blowdown nitrogen and from part of impurity nitrogen concentrated inside the same subsurface layers of the graphite structure (see Figs. 2 and 3). Such distribution of ^{14}C is observed in subsurface layer of parts of graphite cladding of uranium-graphite reactors operated in nitrogen or nitrogen-helium environment. Example of such distribution obtained by the authors by the method of layer-by-layer removal of surface layer is presented in Fig. 4 for graphite blocks of RBMK-1000 reactor of Unit 2, Leningrad NPP.

Thus, presence of heterogeneously distributed ^{14}C offers the possibility, in principle, of selective separation of ^{14}C with insignificant yield of ^{12}C constituting the main mass of irradiated graphite.

For assessing advisability of practical implementation of the above described process for resolving the problem of graphite radioactive wastes, as well as for optimization of parameters of the suggested process it is necessary to first of all determine the fraction of heterogeneously dis-

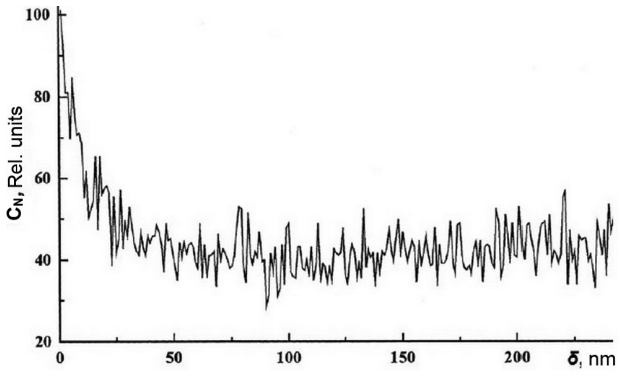

Figure 2. . Relative distribution of nitrogen impurities in graphite sample in the subsurface layer. Sources: data were published by Takahashi R (IAEA 2007).

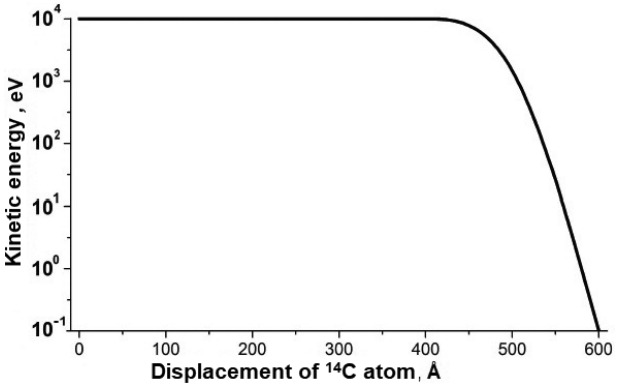

Figure 3. Displacement and loss of energy by ^{14}C atom in perfect graphite crystalline structure (lattice). Sources: data from (Vulpius et al. 2013).

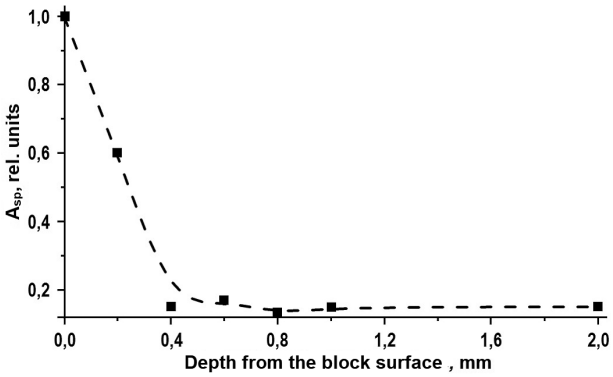

Figure 4. Typical distribution of ^{14}C activity in subsurface layer of graphite blocks of RBMK-1000 reactor, Unit 2, Leningrad NPP. The distribution was defined by the authors. Initial data were experimental data of radionuclide measurements of graphite fragments from graphite blocks of RBMK-1000 reactor, Unit 2, Leningrad NPP.

tributed ^{14}C for which organization of the process of selective separation is possible. This fraction is determined to a significant extent by the fraction of ^{14}Cgenerated from nitrogen contained in blowdown gaseous mixture (nitrogen or nitrogen-helium).

A number of uncertainties are present in the calculation estimations of contributions of different channels of ^{14}C accumulation in the total amount of accumulated ^{14}C. Calculation estimation can be used with acceptable accuracy only for estimating contribution of $^{13}C(n, \gamma)^{14}C$ reaction, where ^{13}C is the carbon isotope contained in the natural mixture of carbon isotopes (fraction is equal to 1.07%) and uniformly distributed in the graphite crystalline lattice. Measurements of specific activity of ^{14}C demonstrate that contribution of this reaction in ^{14}C accumulation in graphite of UGR amounts to not more than 5%. Uncertainties of calculation estimation emerge in the evaluation of contribution of $^{14}N(n, p)^{14}C$ reaction in total ^{14}C activity because of the following:

Significant variation of nitrogen impurities in fresh graphite between different products (from 30 to 100 ppm), as well as variation of parameters of distribution of atoms of impurity nitrogen in the material structure;

– Uncertainties associated with correctness of determination of all factors determining the fraction of ^{14}C formed in $^{14}N(n,p)^{14}C$ reaction from blowdown gas mixture remaining in the structure of irradiated graphite in the conditions of operational regimes of the reactor. It is specifically this part of ^{14}C which determines the fraction of total amount of the isotope for which organization of the process of selective separation is potentially possible.
– Thus, analytical measurement methodologies and calculation methods allow reliably estimating only the total amount of ^{14}C accumulated in graphite, contribution of ^{14}C accumulation channel according to $^{13}C(n, \gamma)^{14}C$ reaction, as well as the total contribution of $^{14}N(n, p)^{14}C$ reaction on impurity nitrogen and on nitrogen of blowdown gas mixture (as the difference between the total amount of ^{14}C and the fraction of ^{14}C accumulated in $^{13}C(n, \gamma)^{14}C$ reaction). Here the problem of estimating the values of contributions of accumulation channels on impurity nitrogen and on blowdown gas remains to be unresolved.

Application of IRT-T reactor for resolving problems of graphite radioactive wastes

Capabilities of such IRT-T reactor (IRT MEPhI) were successfully used before within the framework of investigation of the problem of irradiated graphite of shutdown PUGR (investigation of impurity elements in graphite; determination of isotopic composition of certain actinides contained in graphite by neutron activation analysis methods).

Parallel irradiation of batches of non-irradiated graphite contained in different gaseous environments with thermal neutron flux constitutes the basis of the suggested studies. It is suggested to qualitatively plan the procedure of the suggested experiments as follows:

1. Design and manufacturing of containers for irradiation of samples in one of measurement channels of the IRT-T reactor. Design of containers and layout of organization of irradiation in the reactor provide for creating inside the container of gaseous atmosphere (nitrogen, argon, helium, nitrogen-helium mixture, carbon dioxide) and for maintaining its parameters during the whole period of irradiation.
2. Preparation of two batches of samples from one graphite product (for reducing variation of nitrogen impurities) consisting of five – ten samples each. Mass and dimensional characteristics of the samples are determined by the dimensions and design of containers for irradiation.
3. Arrangement of batches of samples in two different containers. Creation of nitrogen (nitrogen-helium) atmosphere in one of the containers and atmosphere consisting of inert gas (argon, helium) in the second container.
4. Irradiation of containers in the measurement channel of the IRT-T reactor. The main irradiation conditions are as follows:

– batches of samples must be irradiated by thermal neutron flux of the same intensity;
– gaseous atmosphere and its parameters within the volume of containers must be maintained during the whole duration of irradiation.

5. Extraction of containers upon expiration of the preset time of irradiation. Interim cooling down of containers (if necessary).
6. Determination of activity of ^{14}C isotope in irradiated graphite samples by taking samples for assay from ir-

radiated graphite samples in the quantities sufficient for analysis.

7. Repetition of the above operations for the same sample according to paragraphs 3-6 above for the purpose of determination of dynamics of ^{14}C accumulation.

Data on thermal neutron flux in the GEK-4 horizontal measurement channel of the IRT-T (IRT TPU) reactor used for nuclear doping of silicon nuggets can be applied for preliminary estimation of duration of irradiation of samples. Typical values of thermal neutron flux on the measurement section of GEK-4 is ~ $(3–5)\cdot10^{12}$ $cm^{-2}s^{-1}$. The methodology for determining concentration of ^{14}C in irradiated graphite based on the transformation of graphite sample into CO_2 with subsequent trapping and measurement of the obtained solution using liquid scintillation spectrometers allows determining activity of ^{14}C with minimal detectable activity equal to 0.01 Bq per sample. For achieving optimal accuracy of measurements, it is advisable to collect during irradiation activity of graphite induced by ^{14}C equal to not less than ~ 10^2 Bq/g. Characteristic time of irradiation of reactor graphite to collect such activity levels from ^{14}C will amount to about ten days.

Dynamics of ^{14}C accumulation during irradiation in GEK-4 of the IRT-T reactor will have linear character because the following processes do not produce effect on the accumulation curve:

– Burnup of parent isotopes (13C and 14N) and bombardment of generated 14C because of low values of neutron absorption cross-sections (see Table 1);
– Decay of ^{14}C because period of irradiation is negligibly small as compared with half-life for the radionuclide.

The dynamics of ^{14}C accumulation generated in graphite samples irradiated in inert and nitrogen atmosphere is qualitatively represented in Fig. 5.

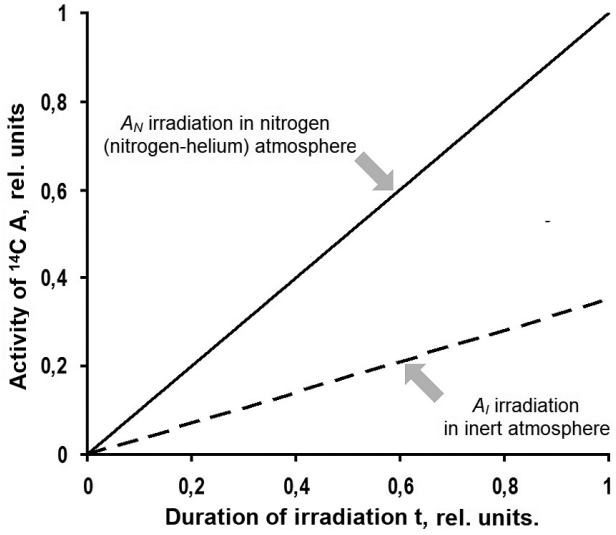

Figure 5. Qualitative dynamics of accumulation of ^{14}C generated in graphite samples under irradiation in inert and nitrogen (nitrogen-helium) atmosphere. The dynamics was defined by the authors.

Different slope of ^{14}C accumulation curves for graphite samples is explained by the presence of additional channel of generation of the isotope in question according to $^{14}N(n, p)^{14}C$ reaction on gaseous nitrogen in the container filled with nitrogen (nitrogen-helium) atmosphere. Accumulation of ^{14}C in the container with inert atmosphere is determined only by $^{13}C(n, \gamma)^{14}C$ reactions on ^{13}C isotope in the natural mixture of carbon isotopes and by $^{14}N(n, p)^{14}C$ reactions on nitrogen contained in the form of impurity in the fresh graphite. Thus, at any time moment t fraction of ^{14}C accumulated in graphite according to $^{14}N(n, p)^{14}C$ reaction from nitrogen containing gaseous environment and shaping the component of heterogeneously distributed ^{14}C will be determined by the following expression:

$$\nu_{gas} = (A_N(t) - A_I(t))/ A_N(t), \quad (1)$$

where ν_{gas} is the fraction of ^{14}C accumulated in graphite according to $^{14}N(n, p)^{14}C$ reaction from nitrogen containing environment; $A_N(t)$ is the specific activity of ^{14}C in graphite samples irradiated in nitrogen containing environment at time moment t; $A_I(t)$ is the specific activity of ^{14}C in graphite samples irradiated in inert gaseous environment at time moment t.

Fraction of ^{14}C accumulation of which is determined by $^{14}N(n, p)^{14}C$ reaction on nitrogen contained in the form of impurity in fresh graphite will be determined by the following expression:

$$\nu_{imp} = 1 - \nu_{gas} - \nu_C, \quad (2)$$

where ν_{imp} is the fraction of ^{14}C accumulation of which is determined by $^{14}N(n, p)^{14}C$ reaction on nitrogen contained in the form of impurity in fresh graphite; ν_C is the fraction of ^{14}C accumulation of which is determined by $^{13}C(n, \gamma)^{14}C$ reaction on ^{13}C isotope in the natural mixture of carbon isotopes.

Fraction ν_C is determined by calculation using the values of thermal neutron fluence during the period of irradiation, the data on ^{13}C content in the natural mixture of carbon isotopes and the neutron radiative capture cross-sections for ^{13}C.

Studies are conducted during recent period both in Russia and abroad on selective separation of ^{14}C from samples of irradiated graphite (Rublevsky et al. 2004, Pageot et al. 2016, Smith et al. 2012, Dunzik-Gougar and Smith 2014, Fachinger et al. 2008, Dirk et al. 2013, Von Lensa et al. 2011, Sibermann et al. 2014). Analysis of experience of such studies demonstrates that the most efficient are the methods based on thermal processing of irradiated graphite in gas or vapor-gas environment (in inert and/or weakly oxidizing environment) (Sach and Williams 1974, Kane et al. 2013, Poluektov et al. 2014). In some cases, the value of selective yield of ^{14}C reaches 70% with relatively low loss of mass of graphite (within the limits of 5%). Practical implementation of the process in question can significantly reduce potential danger of graphite radioactive wastes due to the reduction of both the graphite

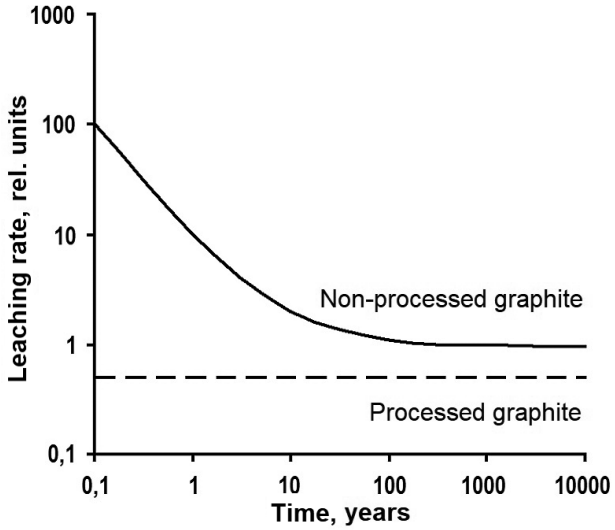

Figure 6. Qualitative dynamics of ^{14}C leaching for non-processed irradiated graphite and for irradiated graphite after separation of heterogeneously distributed ^{14}C by thermal processing in gas environments. Forecast dynamics was defined by the authors.

activity for the given isotope, and the rate of its leaching (LaBrier and Dunzik-Gougar 2015, Wickham et al. 2017) by the removal in the processing of the weakest bound and heterogeneously distributed ^{14}C isotope (Fig. 6).

One of the criteria of advisability of implementation of such processing is the fraction of heterogeneously distributed ^{14}C which is determined in the process of suggested experimental studies using nuclear reactor of IRT-T type. For commercial Russian-designed UGR this fraction is estimated to be at the level of 30 – 90% of total concentration of the isotope in question in graphite (Rublevsky et al. 2004). Such spread of estimates is determined by the dispersion of parameters of blowdown gas mixture, by characteristics of crystalline and porous structure for different graphite brands, as well as by the dispersion of concentrations and parameters of volume distribution of nitrogen impurities in fresh graphite.

Conclusion

Thus, in the conduct of experiments on research IRT-T nuclear reactor for the purpose of determination of selectively removed fraction of ^{14}C for all types of graphite radioactive wastes stored by different entities in the RF which are operating (operated before) UGR it is advisable to use as the material for the studies all brands of graphite applied for manufacturing parts of graphite stacks of indigenous UGRs. It has to be taken into consideration here that during irradiation of samples of stacks of RBMK-1000 reactors it is necessary to use not pure nitrogen but, instead, nitrogen-helium mixture with parameters typical for gaseous environment created in the graphite stack of reactors of this type. Implementation of similar studies of specific features of ^{14}C accumulation in graphite of foreign-designed reactors is also possible, in principle, under the condition of maintaining in one of the irradiated containers of carbon dioxide environment.

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