NEW SHIELDING MATERIALS WITH ADVANCED GAMMA ABSORPTION

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Abstract

This report is given up to the development and study of new shielding materials with higher ability to absorb a gamma radiation. As a result of the work we have produced two new materials, in which high ability to absorb the gamma radiation is provided due to the presence of the uranium dioxide particles in their composition.

We have developed formulation and production technology for high-density concrete (aggregated depleted uranium oxide and cement) with density of 6.5 g/cm^3 and high-density composition of depleted uranium and stainless steel (CERMET) with density of 8.3 g/cm^3 in collaboration with Oak Ridge National Laboratory (USA).

1. Introduction

General practice to ensure the required gamma absorption and strength of casks, intended for transportation, storage and disposal of spent nuclear fuel and high level radioactive waste, is based on the increasing of shielding material density. For industrial scale metal-concrete casks this parameter is about 4.0 – 4.2 g/cm^3. For all-metal casks the density of shielding material does not exceed 7.8 g/cm^3. The density of the concrete and stainless steel, which are the traditional materials for shielding containers, can be increased due to the addition into their composition of the materials with advanced gamma absorption - for example, uranium dioxide (UO_2).

The usage of depleted uranium dioxide in metal-concrete or metal casks, along with the ensuring of required degree of gamma absorption, can also provide slowdown of fast neutrons due to higher oxygen content in depleted uranium dioxide (1.3 g/cm^3) and then the capture of neutrons by thermal neutron absorption. This property is unique for such high-density shielding materials.

However, there is one more important circumstance, which we have taken into account, when we have being solved the problem of the increasing of concrete and stainless steel protective characteristics. USA and Russia have accumulated enormous amount of depleted uranium waste (~10^6 tons) in the forms of uranium hexafluoride (UF_6) and uranium tetra fluoride (UF_4). These chemical combinations are the waste of the process of the uranium enrichment and have no wide practical application up to now. Moreover, their reserves increase which, in its turn, leads to serious ecological problem and requires more expenses for its storage, monitoring and so on. The production of the depleted uranium dioxide from UF_6 and UF_4 and their usage as additions to concrete or stainless steel is the possible way for the decision of ecological problem.

2. The development and study of radiation shielding composition

Application of high-density concrete as structural and radiation shielding material in the casks, intended for transportation, storage or disposal of spent nuclear fuel or high level radioactive waste, invokes a number of contradictory requirements. On the one hand, its components should not be expensive and commercially available, on the other hand, the material should provide high strength and density (this indicates its absorption degree of ionizing radiation), tolerable thermal conduction, thermal, radiation and corrosion resistance, service life and water resistance. Specific characteristics of UO_2 (chemical activity, small size of particles and, thus, large specific surface area) prevent the use of traditional methods of UO_2 introduction into concrete composition. Therefore, the UO_2 particles must be preliminary coarsened (aggregated) and UO_2 chemical resistance must be improved due to additives.

Initially, the experiments on aggregation of powdered uranium dioxide for the use as concrete filler and the experiments on concrete production were carried out in INEEL (USA) by Paul Lessing and William Quapp according to the INEEL program for the USA DOE on the use of depleted uranium [1]. They have developed the UO_2 sintering technology with additives. These additives interact with uranium and with each other under the heating and generate liquid phase. During the sintering process glassy phase covers the oxide grains and fills the space between the grains forming a strong bond. At the same time UO_2 chemical resistance is increased and the aggregates of the required size are produced. The production technology of UO_2 ceramics aggregating and concrete manufacturing (DUAGG and DUCRETE) has been patented by INEEL in the TETON TECHNOLOGIES (TTI) Company in 1996 [2]. Then TTI cooperated with STARMET Company on commercialization of the technology.

UO_3 was used as a raw material for the INEEL and STARMET experiments. UO_3 has been transformed into uranium protoxide-oxide U_3O_8, and then uranium dioxide UO_2 was produced by low-temperature reduction. UO_2 which was the raw material to produce aggregated UO_2 ceramics (DUAGG), tested by INEEL and STARMET, is available in the amount of ~5 % of the total volume of UO_2. It is expected the other 95 % of UO_2 will be obtained using the “high-temperature” technology. First time the experiments with aggregated ceramics manufactured from “high-temperature” UO_2 were carried out. At the first step we investigated the properties of ceramics based on “high-temperature” depleted uranium dioxide and mineral additives. At the second step we investigated the characteristics of concrete with ceramic filler based on aggregated depleted uranium dioxide. The analysis of formula, characteristics and technology of DUAGG - INEEL [2] has shown that there are the following possibilities:

- To reduce the number of components used and, consequently, to reduce the cost of the ceramics;
- To optimize the conditions of the glass forming and sintering;
- To increase glass adhesion to UO_2 due to higher alkalinity of new glass;
- To increase the uranium content in ceramics.

2.1. Study of ceramics

Analysis of chemical composition and possible reactions between the components of DUAGG shows the running of two processes: glass-formation (sodium-boron-silicon glass with homogeneous dissolved uranium) and solid-phase synthesis of polyphase ceramics.

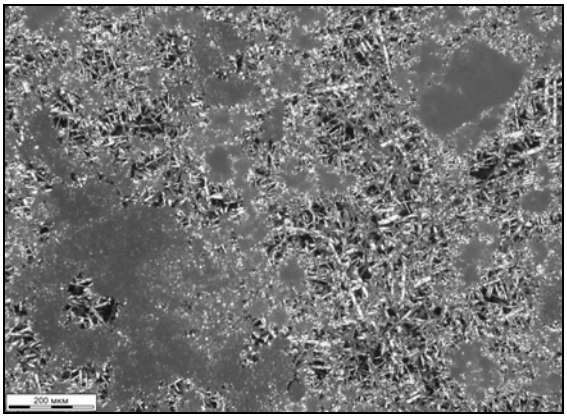
The high content of the uranium oxide in DUAGG is the principal difference of DUAGG from the sodium-boron-silicon glasses and polyphase ceramics. Boron-silicon glass forming systems, investigated in detail, are used now in industrial scale for immobilization of liquid high-level radioactive waste [3-12]. Some studies [13-18] show the possibility of the use of crystal glass matrixes for vitrification of radioactive waste, containing oxides of the fissile products up to 26 % mass. Mass content of uranium dioxide in composition of DUAGG [2] is more than 90 %. The new chemical composition of aggregated UO_2 ceramics and its production technology have been developed using our experience on vitrification of high-level radioactive waste. New aggregated uranium ceramics became filler in the new radiation shielding composition named RSC-VNIINM. Chemical compositions of DUAGG-INNEL and filler of RSC-VNIINM are shown in table 1.

Table 1 Chemical compositions of DUAGG – INEEL and filler of RSC-VNIINM

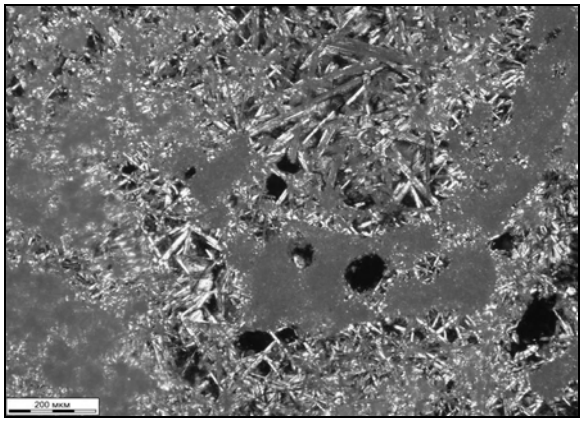
Component	Compositions, % mass	
	DUAGG – INEEL	Filler of RSC-VNIINM
UO_2	91.69	92.58
Bentonite	3.09	3.17
Pumice	1.09	-
Talc	0.13	-
$Ca(OH)_2$	0.75	-
Na_2CO_3	0.05	0.33
K_2CO_3	0.24	-
TiO_2	1.56	1.42
ZrO_2	0.81	0.67
H_3BO_3	0.59	-
Datolite	-	1.83
Total	100.01	100.0

Samples of the glass forming binder (all components excepting UO_2) were made according to INEEL and VNIINM formulas. For manufacturing of the glass forming binder samples of the INEEL composition we have used STARMET technology: compacting by pressure, drying, sintering during an hour at the temperature 1250 ^oC and an hour at the temperature 1300 ^oC in Ar + 7 % H_2 atmosphere. For manufacturing of the VNIINM's samples of the glass forming binder we used the following technology developed: compacting by pressure, drying, sintering during an hour at the temperature 1250 ^oC in Ar + 7 % H_2 atmosphere. All samples looked like brownish-beige glass. The studies of the samples were carried out by the methods of X-ray phase analysis, optical microscopy and scanning electronic microscopy.

According to the obtained data the INEEL and VNIINM samples of the glass forming binder have the similar structure. The INEEL binder consists of glass, titanium oxide, zirconium titanate, and zirconium oxide. The VNIINM binder includes zirconium silicate also. In both cases titanium oxide has two morphological varieties (see figure 1). It is easy to identify glass (black) and phase of rutile - titanium oxide (elongated grains with high level of interference color).



Glass forming binder of INEEL



Glass forming binder of VNIINM

Figure 1 Structure of the glass forming binder samples

2.2. Study of DUAGG and filler of RSC-VNIINM

Complex comparative studies of DUAGG and filler of RSC-VNIINM were carried out. Samples of DUAGG were made using STARMET technology. The technological regimes were varied during fabrication of the samples of RSC-VNIINM filler for determination of the optimal fabrication parameters. The sequence of operations and some technological parameters of the aggregated ceramics fabrication process according to STARMET and VNIINM technologies are shown in table 2.

Table 2 Technological regimes of aggregated ceramics fabrication

Technological regimes	DUAGG – INEEL (STARMET technology)	Filler of RSC-VNIINM
1	Mixing of DUAGG components	Mixing of filler components
2	Pressing of DUAGG half-finished product	Pressing of RSC-VNIINM filler half-finished product
3	Drying 1 h at 900^{o}C	Crushing of RSC-VNIINM filler half-finished product to obtain the required fraction composition
4	Sintering of DUAGG half-finished product 1 h at 1250^{o}C + 1 h at 1300^{o}C and further cooling	Drying 1 h at 700^{o}C
5	Crushing of DUAGG half-finished product to obtain the required fraction composition	Sintering of crushed RSC-VNIINM filler half-finished product 1 h at 1250^{o}C and further cooling

The filler of RSC-VNIINM is very competitive to DUAGG on its main physical parameters, and in some cases its parameters exceed ones of DUAGG. In addition, VNIINM ceramics fabrication requires minimal number of components that reduces its cost. This technological regime offers advantages in comparison with STARMET production technology, as it does not include the

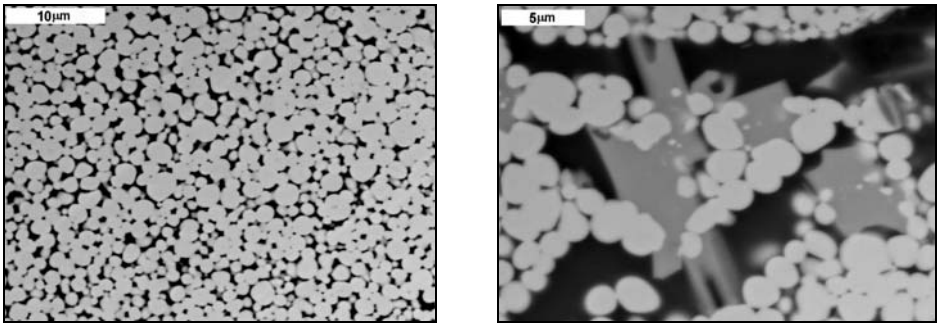
operation of sintered briquette crushing. Excluding of this procedure allows reducing the energy consumption and avoiding of dust containing UO_2.

The samples of aggregated ceramics based on UO_2 (DUAGG and RSC-VNIINM filler) were investigated. The results obtained prove the following:

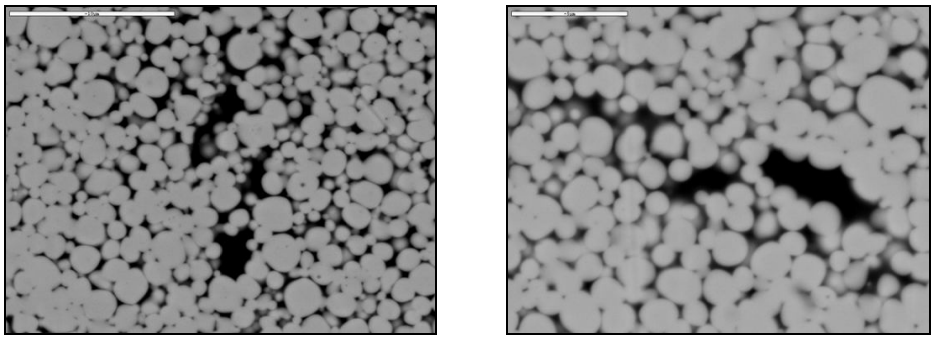
- Preliminary mixing of the components of glass forming additive does not affect the density of the finished samples actually;
- Increase of compacting pressure from 50 up to 350 MPa stimulates an increase of ceramics density after sintering; the further increase of compacting pressure causes density reduction. Optimum value of compacting pressure has been determined (~170 MPa);
- Increasing of the heating rate during the sintering process stimulates reduction of the density;
- Sintering provides the compacting of ceramic aggregate due to capillary forces under the wetting of uranium dioxide grains by amorphous glass-metal generated from the binder components;
- The DUAGG samples, manufactured from Russian components and "high-temperature" UO_2, have similar characteristics (microstructure, phase composition, structural distribution of phases) with DUAGG – INEEL fabricated in the USA with use of low-temperature UO_2;
- Density of DUAGG-INEEL ceramics manufactured under the technology similar to STARMET is of 7.85-7.90 g/cm^3; average density of the filler of RSC-VNIINM is of 7.85-7.90 g/cm^3 and its real density is 9.12 g/cm^3.

Material science testing of the aggregated UO_2 ceramics was carried out. The samples of DUAGG and filler of RSC-VNIINM have porous structure. The pore size varies in a large degree, but generally we could identify two size groups: 100-400 μm and 5-20 μm. Agglomerations of uranium dioxide grains form isometric isolations of 50-200 μm surrounded by glass. The identity of phases of the sintered UO_2 and aggregated ceramics based on UO_2 and additives proves that glass-formation processes at synthesis and formation of the glass protective phase of ceramics do not affect the structure of UO_2. The size of raw UO_2 cell is not significantly changed after simultaneous processes of glass-formation and solid-phase synthesis at aggregated ceramics manufacturing. Thus, the ratio U(4+)/U(6+), i.e. UO_2 oxygen coefficient of the synthesized ceramics is close to oxygen coefficient of the raw UO_2. According to measurements the oxygen coefficient of DUAGG – INEEL is 2.06 ± 0.08 and for RSC-VNIINM filler it is 1.96 ± 0.08. Such values of the oxygen coefficient mean that during the synthesis of aggregated ceramics the raw UO_2 (oxygen coefficient 2.16 ± 0.08) is reduced up to composition which is very close to stoichiometric one. The structures of DUAGG-INEEL and RSC-VNIINM filler investigated by the methods of scanning electronic microscopy (SEM) are shown in the figure 2.

The main phase of DUAGG-INEEL and filler of RSC-VNIINM is uranium dioxide with fluorite lattice. The composition of glass of DUAGG-INEEL and filler of RSC-VNIINM is constant in any points. Average grain size of UO_2 in RSC-VNIINM filler is less than 1 μm, maximum grain size is ~2 μm that less than grain size of UO_2 in DUAGG-INEEL. More than 10 % of uranium is dissolved in a glass due to high alkality of the glass-metal. In the glass phase of DUAGG the extended crystals of uranium titanate (brannerite - UTi_2O_6) have size up to 5 μm. Brannerite was also found in RSC-VNIINM filler, but it was inside of rare and small size areas enriched by titanium.



DUAGG–INEEL



Filler of RSC-VNIINM

Figure 2 SEM-microstructure of ceramics
(gray – uranium dioxide, dark-gray – uranium titanate, black - glass)

Cylindrical samples of 10-mm diameter and 10-15mm height were used for the test of strength characteristics of DUAGG–INEEL and filler of RSC-VNIINM. Compression strength of RSC-VNIINM filler is 265 MPa that is 1.2 times more than for DUAGG.

2.3. Study of RSC-VNIINM

The chosen materials and corresponding production technology would provide the following characteristics of concrete containing depleted uranium:

- Average density – no less than 6 g/cm^3;
- Workability of concrete mixture should satisfy standard requirements;
- Concrete mixture should not segregate under technological procedures (fabrication and compression).

The theoretical analysis of formation of concrete structure, its properties and destruction gave us a possibility to formulate the main rules for manufacturing of high-gravity concrete with high strength. There are the following rules:

- Aggregates which are used as a filler should have the various sizes: from small to large ones; the strength of any aggregate should exceed tension of the structure under the maximum loading;
- Reduction of discontinuity flaws (pores, cracks, hollows, etc.) as compared with traditional and high-strength concrete should be provided;

- High adhesion of cement with filler (uranium aggregate) and minimal water-cement ratio would be provided for the manufacturing of high strength concrete and for the decreasing (or eliminating) of concrete shrinkage.

A lot of experiments were carried out to realize the ideas mentioned above. Finally authors were success in the development of the high-gravity concrete and its production technology. This concrete, having the unique characteristics, was named "Radiation Shielding Composition RSC-VNIINM". RSC-VNIINM, filler of RCS-VNIINM and their production technologies were patented [19]. The composition and some properties of RCS-VNIINM are given in table 3.

Table 3 Composition and some properties of RSC-VNIINM

Composition and properties of RSC-VNIINM	Value
Portland cement M500-D0, kg/m^3	450
Water, l/m^3	165
Plasticizer C-3, kg/m^3	5
Filler of RSC-VNIINM: • Fraction 5…10 mm, kg/m^3 • Fraction 1.25…5 mm, kg/m^3 • Fraction 0.63…1.25 mm, kg/m^3	 2500 1230 1240
Cast-iron shot № 5, kg/m^3	1050
Volume mass (calc.), kg/m^3	~6640
Density, kg/l	6.5*
Strength, MPa	67.2
Tensile strength, MPa	6.5
Deformation limit, mm/m	$2.62 \cdot 10^{-3}$
Module of elasticity, MPa	55500
Poisson's ratio	0.22

*Final density is a little bit less than computed mass of 1 m^3 of RCS-VNIINM due to water evaporation during hardening of RSC-VNIINM.

Before filling of the form it is recommended to carry out the preparation of RSC-VNIINM in the following sequence:

- Mix up all filler's fractions with adding of plasticizer and water (excluding cement);
- Mix up with cement and water.

Mechanical tests were carried out on RSC-VNIINM samples of two sizes (40×40×160 mm and 70×70×70 mm) after their exposure in wet sawdust during 28 days (see figure 3). Basic

characteristics of RSC-VNIINM have values close to DUCRETE characteristics, and some RSC-VNIINM characteristics surpass DUCRETE ones.

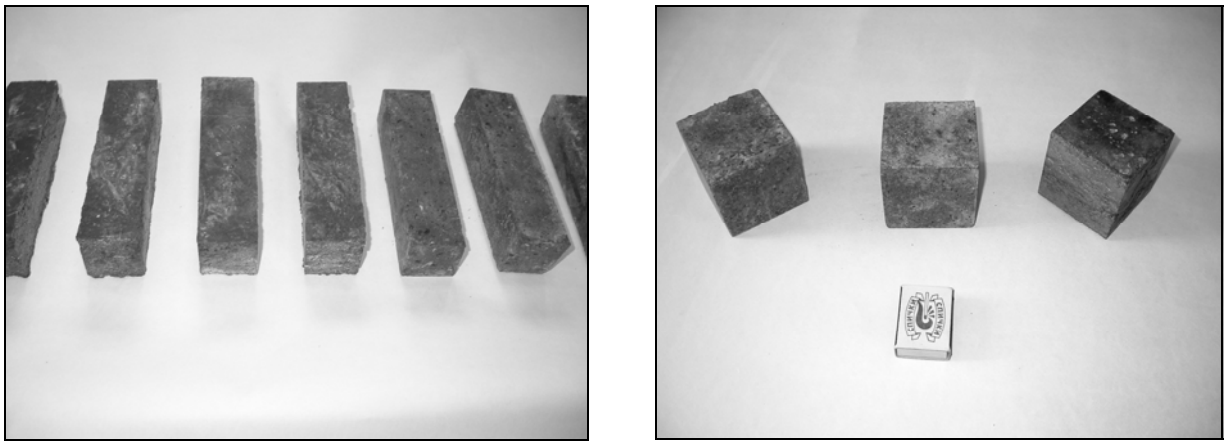


Figure 3 Samples of RSC-VNIINM

3. Development and study of CERMET

It was determined [20], that the way of cast uranium CERMET manufacturing with the use of introduction of uranium dioxide powder directly into the melt of stainless steel is not right, because density of uranium CERMET (~6.6 g/cm^3) produced is less than stainless steel density. Uranium CERMET has the low density and strength because of irregular distribution of uranium dioxide powder in steel matrix.

Then the manufacturing of uranium CERMET directly in a cold crucible of IPHT-100M furnace with use of impregnation of "melted uranium dioxide" by stainless steel was investigated [21]. There was ~50 % vol. of uranium dioxide in CERMET produced according to such technology. The structure of the ingot was comparatively uniform, however, separate pores with diameter up to 5 mm have been found. Tensile strength of CERMET was about 50 kg/mm^2; the samples had brittle type of fracture. Separate fragments had density from 7.8 up to 8.2 g/cm^3. CERMET produced by such way was not deformed plastically, and the fabrication of the ring elements of biological shield by rolling with the following welding was impossible. The results of this stage of investigations were both the demonstration of the principal possibility of uranium CERMET manufacturing and possibility of uniform distribution of uranium dioxide in stainless steel matrix.

Main result of the second stage of investigations was admission of inefficiency of cast uranium CERMET manufacturing with the use of cool crucible. This method does not provide the casting of overheated melt in beforehand prepared and heated up foundry. Besides, the possibility of preliminary electromagnetic heating of CERMET ceramics, required for the qualitative impregnation of the ceramics granules, is absent in the furnace with cool crucible (heating is carrying out due to heat conductivity only, but not by Fuko current). So, the further use of furnace with cool crucible for the production of the cast CERMET (ring elements) became unacceptable.

The method of melting and casting with the use of vacuum induction furnace, which is widely used in world practice of uranium materials melting, is perspective for the production of the ring castings and from the point of view of fabrication in future of castings with larger size and mass. However, the fabrication of cast uranium CERMET (stainless steel + depleted uranium dioxide) in induction furnace is connected with the melt heating up to temperature above 1700^{o}C. This is the certain difficulty in the choice of materials for melting equipment from the point of view of interaction of equipment and melted material.

For optimization of the cast CERMET production technology it was necessary to find the possibility of decreasing of the melting temperature and increasing of castability of the steel for best impregnation of granulated uranium dioxide and exception of CERMET porosity. Fabrication of the necessary amount of granulated uranium dioxide was the other problem in laboratory.

3.1. Development of matrix alloy and fabrication of granulated uranium dioxide

The composition of stainless steel with melting point less than 1200^{o}C was chosen on the base of literary and experimental data. This steel has the best technological properties amongst all compositions which were analyzed.

For the carrying out of experiments the granulated depleted uranium dioxide was manufactured. Production technology was traditional, accepted at fabrication of ceramic nuclear fuel. The size of granules was of 5-10 mm. Density of the granules was 10.2-10.55 g/cm^3, i.e. 94-96% of theoretical uranium dioxide density. The exterior view of the depleted uranium dioxide granules and their microstructure are shown in the figure 4. The granules manufactured did not have cracks, voids and pores.

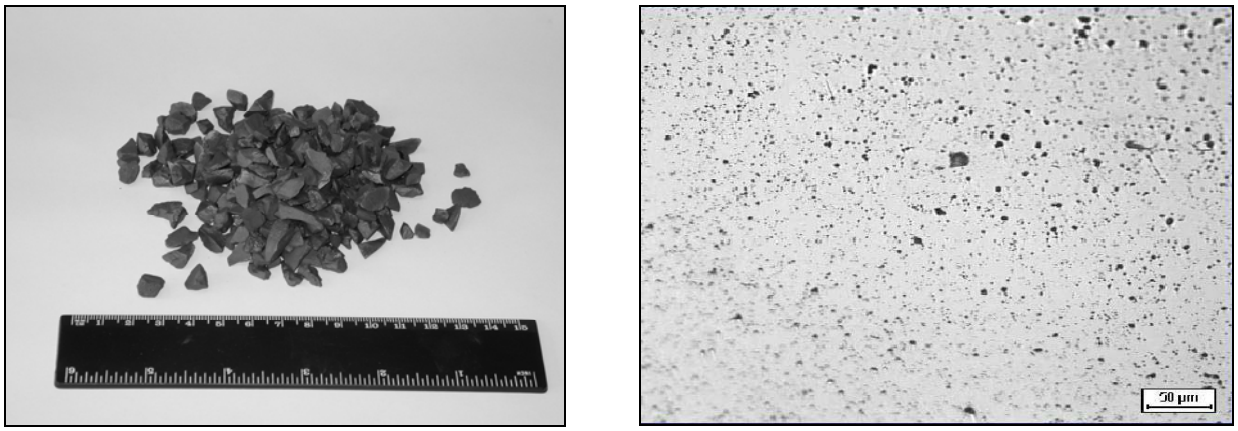


Figure 4 View of the depleted uranium dioxide granules and typical microstructure

3.2. Fabrication and study of cast uranium CERMET

The experiments on manufacturing of cast uranium CERMET were carried out with the use of induction vacuum furnace VVP-3. The graphite foundry assembly has been designed and manufactured (figure 5) for manufacturing of the CERMET.

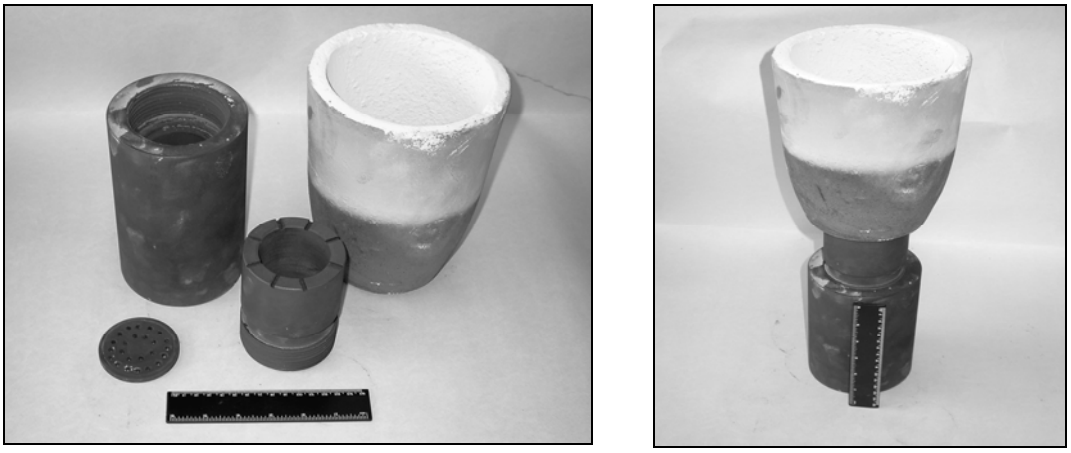


Figure 5 Elements of the cast assembly and the assembly prepared for melting

The alloy of stainless steel developed was loaded in crucible located on cast assembly and heated on ~200^{o}C above the melting temperature. Then melted metal was poured out into the foundry form with granulated depleted uranium dioxide. Assembly with uranium CERMET was held some minutes for impregnation of uranium dioxide by stainless steel and then it was cooled down with the furnace. Volumes of steel (matrix of CERMET) and uranium dioxide (filler of CERMET) were equal to provide equality of the volumes of ceramic and metallic phases. CERMET density in different parts of ingot was 8.49-8.51 g/cm^3. The exterior view of uranium CERMET ingot and its macrostructure are shown in the figure 6.

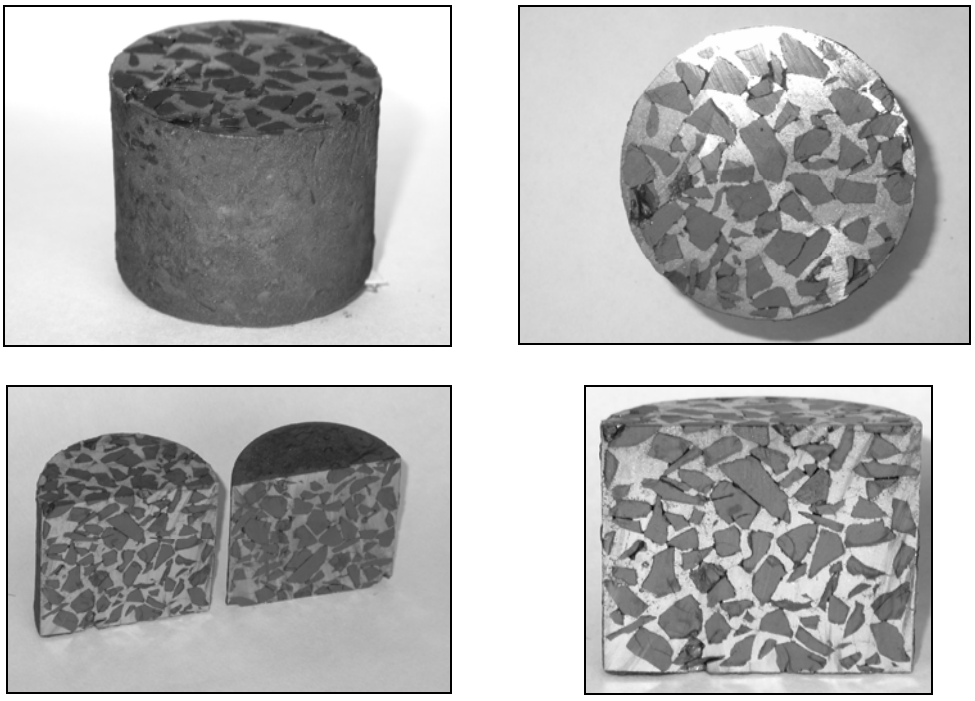


Figure 6 View of uranium CERMET ingot (Ø75 mm) and its macrostructure

4. Conclusion

1. The high-density radiation shielding composition (RSC-VNIINM) on the base of granulated "high-temperature" depleted uranium dioxide (filler of RSC-VNIINM) and production technologies have been developed, studied and patented. Density of the RSC-VNIINM developed is 6.5 g/cm^3, which is 1.5 times more than density of metal-concrete traditionally used as gamma shield material.

2. The cast uranium CERMET with density ~8.5 g/cm^3 has been produced with the use of ordinary vacuum induction furnace, intended for the melting and casting of uranium materials.

5. Acknowledgement

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