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RESEARCH ARTICLE

A study of the mechanical and thermophysical properties of crystal matrices for the immobilization of high-level wastes

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Abstract

Objectives. The aim of the study was to confirm the compliance of the mechanical and thermophysical properties of titanate-zirconate mineral-like matrices intended for immobilization of the rare-earth-actinide fraction of high-level waste (HLW) with pyrochlore structures (Nd_2ZrTiO_7) and orthorhombic titanate of rare earth elements ($Nd_4Ti_9O_{24}$+TiO_2) with the Russian requirements for the final forms of radioactive waste sent for disposal. With regard to fractionated radioactive waste, this type of matrix is preferable when compared with conservative aluminophosphate and borosilicate glasses. This is due to larger capacity, and a better level of chemical, thermal, and radiation resistance.

Methods. The synthesis of mineral-like matrices was carried out by remelting a granular precursor consisting of mineral-forming metal oxides and a solution imitating the rare earth-actinide fraction of HLW in an induction furnace with a cold crucible. The thermal diffusivity was determined by the laser flash method. The heat capacity of the matrix samples was measured by differential scanning calorimetry. Ultimate flexural and compressive strengths were determined using universal test machines. The elastic moduli (Young's) were measured by the acoustic method. The temperature coefficients of linear expansion were determined using a high-temperature dilatometer.

Results. The ultimate strength of the matrices (Nd_2ZrTiO_7) and ($Nd_4Ti_9O_{24}$+TiO_2) was found to be 150–179 and 20.6–57.8 MPa in compression and bending respectively. Young's moduli vary from $3.7 \cdot 10^7$ to $2.15 \cdot 10^8$ kN/m^2. With an increase in temperature from 50 to 500°C, the values of thermal conductivity have a pronounced tendency to decrease from 1.71 to 0.91 W/(m·K). The temperature coefficients of linear expansion increase from $6.96 \cdot 10^{-6}$ to $1.01 \cdot 10^{-5}$ K^{-1} in the same temperature range.

Conclusions. Comprehensive studies of titanate-zirconate mineral-like matrices show that their mechanical and thermal properties in certain cases significantly exceed the minimum requirements of regulatory documentation for the final forms of HLW.

Keywords

high-level waste, pyrochlore, orthorhombic titanate, strength, thermal conductivity

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НАУЧНАЯ СТАТЬЯ

Исследование механических и теплофизических свойств кристаллических матриц для иммобилизации высокоактивных отходов

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Аннотация

Цели. Целью работы являлось подтверждение соответствия механических и теплофизических свойств титанатно-цирконатных минералоподобных матриц, предназначенных для иммобилизации редкоземельно-актинидной фракции высокоактивных отходов (ВАО) российским требованиям, предъявляемым к конечным формам радиоактивных отходов, направляемых на захоронение. Матрицы имеют структуры пирохлора (Nd_2ZrTiO_7) и орторомбического титаната редкоземельных элементов ($Nd_4Ti_9O_{24}$+TiO_2). Применительно к фракционированным радиоактивным отходам данный тип матриц более предпочтителен по сравнению с консервативными алюмофосфатными и боросиликатными стеклами благодаря большей емкости и лучшей химической, термической и радиационной устойчивости.

Методы. Синтез минералоподобных матриц осуществляли путем переплавки гранулированного прекурсора, состоящего из минералообразующих оксидов металлов и раствора, имитирующего редкоземельно-актинидную фракцию ВАО, в индукционном плавителе с холодным тиглем. Исследование температуропроводности проводили методом лазерной вспышки; теплоемкость образцов матриц измеряли методом дифференциальной сканирующей калориметрии; пределы прочности на изгиб и сжатие определяли с помощью универсальных испытательных машин; модули упругости (Юнга) измеряли акустическим методом. Температурные коэффициенты линейного расширения находили с помощью высокотемпературного дилатометра.

Результаты. Установлено, что пределы прочности матриц (Nd_2ZrTiO_7) и ($Nd_4Ti_9O_{24}$+TiO_2) составляют 150–179 и 20.6–57.8 МПа при сжатии и изгибе соответственно. Модули Юнга варьируются от $3.7 \cdot 10^7$ до $2.15 \cdot 10^8$ кН/м^2. Значения теплопроводности при повышении температуры от 50 до 500°C имеют выраженную тенденцию к уменьшению от 1.71 до 0.91 Вт/(м·К). Температурные коэффициенты линейного расширения увеличиваются от $6.96 \cdot 10^{-6}$ до $1.01 \cdot 10^{-5}$ К$^{-1}$ в том же температурном интервале.

Выводы. Комплексные исследования титанатно-цирконатных минералоподобных матриц показали, что их механические и теплофизические свойства в ряде случаев существенно превосходят минимальные требования нормативной документации, предъявляемые к конечным формам ВАО.

Ключевые слова

высокоактивные отходы, пирохлор, орторомбический титанат, прочность, теплопроводность

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INTRODUCTION

The generation of significant amounts of high-level waste (HLW) during spent nuclear fuel (SNF) reprocessing (variants of the PUREX process[1]) is an obstacle to the large-scale development of modern nuclear power [1, 2]. According to The International Atomic Energy Agency recommendations[2] and the current regulations of countries operating nuclear power plants, liquid HLW must be conditioned in order to reduce its volume. It then must be converted into a final form suitable for environmentally safe long-term storage and burial in geological formations at a depth of at least 500 m [1–5]. The resulting matrix must be chemically, thermally and radiation resistant and retain its insulating capacity for at least 1000 years.[3]

Two technological approaches to immobilization of liquid HLW into matrix materials are currently industrially implemented in the world. At *Mayak Production Association*, universal aluminophosphate glass matrix (AGM) is used. This is characterized by a relatively low synthesis temperature of 900–1050°C and a unique ability to include a wide range of elements and compounds in its composition. This includes significant amounts of molybdenum and aluminum oxide, the source of which is the fuel of mobile power plants [6–8]. On average, the Russian Federation annually generates up to 74 m^3/year of vitrified HLW sent for temporary storage.[4]

France and Great Britain use more specialized final forms as borosilicate glasses which have a slightly higher radionuclide capacity of up to 18.8 wt % [8–9]. Their matrix density is about 2.85 t/m^3. However, even when such relatively high values are achieved, the volume of HLW sent for storage and/or disposal is significant and amounts to 0.1–0.11 m^3/t of SNF [10].

Fractionation may perhaps be the only solution acceptable from the point of view of ensuring relative economic efficiency and environmental safety when handling liquid HLW [11–18]. The process implies maximum extraction of energy nuclides with the purpose of returning to the nuclear fuel cycle and afterburning in fast or liquid salt reactors. At the same time, unclaimed fission products can be divided into several fractions based on the principle of similarity of chemical properties, making it possible to select the optimal composition of the final form. Fractionation scenarios which are more realistic for industrial realization require the separation of HLW from solution:

- rare-earth actinide (REE-actinide) fraction, formed after extraction of uranium, plutonium and neptunium, containing mainly lanthanides, americium and curium (up to 3.5 and 0.44 wt % of the total amount of metals in solution), as well as traces of U, Pu, and Np [11–18];
- cesium-strontium fraction, saturated with active and stable isotopes of Cs and Sr, as well as Ba, the share of which can be up to 26% of the total mass of metals [11–19].

One of the promising forms for immobilization of REE-actinide fraction are crystalline matrices [11]. These have high radionuclide capacity, density, thermal, chemical, and radiation stability [20–22]. Their long-term stability is confirmed by the long-term existence of structurally identical minerals in the harsh conditions of the Earth's crust [23]. To date, there have been many fundamental studies confirming the above-mentioned advantages. However, a coherent, comprehensive and structured justification of their applicability is lacking. In this regard, there is no regulatory framework governing the quality of crystalline matrices. This also limits their industrial development.

Current regulations require cured HLW to be buried in geological formations up to several hundred meters deep [24]. The mechanical and thermophysical properties are the most important qualitative characteristics of the final forms. Strength limits in bending and compression determine the preservation of matrix integrity during transportation operations and the impact of pressure of the geological environment at the point of final disposal. Insufficient strength of matrix blocks can lead to the formation of cracks and fractures in the developed surface, thus reducing the materials resistance to leaching.

The thermal conductivity of the matrix determines its resistance to overheating as a result of to the decay of incorporated radionuclides. This also affects the maximum fraction of incorporated HLW, as well as the ingot dimensions which provide an acceptable level of matrix heating. Low thermal conductivity can lead to local matrix overheating, accompanied by mechanical stress and, can eventually lead to matrix failure.

The linear expansion temperature coefficient (LETC) affects the change in the dimensions of the matrix ingot in the process of its heat release drop. This is due to the decay of short-lived radionuclides. The indicator is critical at the stage of container selection. The values

[1] Plutonium-Uranium Recovery by Extraction.

[2] https://www.iaea.org/ru. Accessed March 25, 2023.

[3] Federal Standards and Rules in the Field of Atomic Energy. Criteria for Accepting Radioactive Waste for Disposal. NP-093-14. *Nuclear and Radiation Safety*. 2015;77:(3):59–82. https://docs.secnrs.ru/documents/nps/НП-093-14/НП-093-14.pdf. Accessed March 23, 2023.

[4] Semyonov M.A. Issues of preparation of class 2 radioactive waste for disposal. Proceedings of the Scientific and Technical Seminar “SNF and RW Management at ZNFC,” May 27, 2021; Moscow, Russia. A.A. Bochvar VNIINM; 2021. https://bochvar.ru/materialy-konferentsiy/06%20Семенов%20М.А.%20(ФГУП%20ПО%20Маяк)%20-%20Презентация.pdf. Accessed March 27, 2023.

of LETC of the final mold and its packaging material should not differ significantly.

The aim of the study was to confirm the compliance of the mechanical and thermophysical properties of titanate-zirconate mineral-like matrices intended for immobilization of REE-actinide fraction of HLW with the current Russian requirements for the final forms of radioactive wastes sent for disposal.

MATERIALS AND METHODS

The following types of crystalline matrices for immobilization of REE-actinide fraction were tested in the present study:

- structure of titanate-zirconate pyrochlore Ln_2ZrTiO_7 (up to 62 wt % of Ln_2O_3);
- phase of orthorhombic REE titanate and rutile $Ln_4Ti_9O_{24}$+TiO_2 (up to 33 wt % of Ln_2O_3).

The above crystal matrices were selected due to their versatility, expressed in the ability to include REE-actinide fraction with different An : Ln (actinides and lanthanides) ratios. This solution is convenient from a technological point of view, since it does not impose strict limitations on the fractionation process.

Matrix materials were synthesized using an original method which includes obtaining a granular precursor from inactive simulant of liquid HLW and solid mineral formers with subsequent cold crucible induction melting (CCIM). The resulting matrices were ingots with a diameter of 120 mm and a height of ~120 mm. Before testing, the material to be studied was inspected for compliance of phase and chemical compositions with those specified using the Vista PRO atomic emission spectrometer with inductively coupled plasma (*Varian*, Australia) and DRON-4M powder X-ray diffractometer *(Burevestnik*, USSR). X-ray diffraction data decoding and phase identification were performed using the Match! software package (*Crystalimpact GmbH*, Germany) and ICDD-2 database.[5]

Crystalline matrices with confirmed characteristics were cut into samples to study the mechanical and thermophysical properties. Material fragmentation and surface treatment were performed using the following precision machines: Mecatome T210 cutting machine (*Presi SAS*, France) and Mecatech 234 grinding and polishing machine (*Presi SAS*, France), respectively. The configurations of the specimens and references to the test methods according to the manufacturing requirements are presented in Table 1.

Figure 1 shows the appearance of the samples, Fig. 2 shows the diffraction pattern of matrices with pyrochlore (a) and orthorhombic titanate (b) structures.

The compressive and flexural strengths were determined using a universal testing machine LFM-50 (*Walter+Bai*, Switzerland). The final values of the parameters were calculated as arithmetic averages in a series of measurements. Young's moduli were measured acoustically by recording the time of passing through the sample ultrasonic signal with a frequency of 2.5 MHz. The propagation velocity (v_l) of ultrasonic longitudinal waves was determined by Eq. (1):

$$v_l = \frac{l}{t_2 - t_1}, \quad (1)$$

wherein l—sample length, m; t_1—travel time of ultrasonic waves with the sample, s; t_2—travel time of ultrasonic waves without sample, s.

Young's modulus (E) was calculated by Eq. (2):

$$E = v_l^2 \times \rho, \quad (2)$$

wherein ρ—density, kg/m^3.

Thermal conductivity was determined by calculation method on the basis of measured values of heat capacity and thermal diffusivity by the Eq. (3):

$$\lambda = 1000000 \times a \times c \times \rho, \quad (3)$$

wherein a—diffusivity, m^2/s; c—specific heat capacity, J/(g·K); λ—heat transfer coefficient, W/(m·K); ρ—material density, t/m^3.

Heat capacity was determined using a differential scanning calorimeter DSC 404 F1 (*Netzsch*, Germany). The temperature conductivity was determined using a LFA 457 (*Netzsch*, Germany) solid state thermophysical parameter meter by the laser flash principle [25, 26]. In both types of tests, three parallel measurements were performed for each matrix type in the range of 50–500°C with a step of 50°C at a furnace heating rate of 3°C/min for each type of matrix.

LETC was determined using a DIL 402 horizontal pusher dilatometer (*Netzsch*, Germany) at a temperature range of 20 to 500°C in 20°C increments, with a furnace heating rate of 3°C/min.

RESULTS AND DISCUSSION

As mentioned earlier, the only certified final form for immobilization of HLW in the Russian Federation is AGM. The requirements for its quality are given in state standard GOST R 50926-96[6]. The initial data for the drafting of this legislative document was based on the parameters of glass produced at *Mayak*. This was done during solidification of the collective flow of liquid HLW generated during reprocessing of SNF of different

[5] International Center for Diffraction Data. https://www.icdd.com/. Accessed March 10, 2023.

[6] GOST R 50926-96. State Standard of the Russian Federation. High level solidified waste. General technical requirements. Moscow: Gosstandart Rossii; 1997.

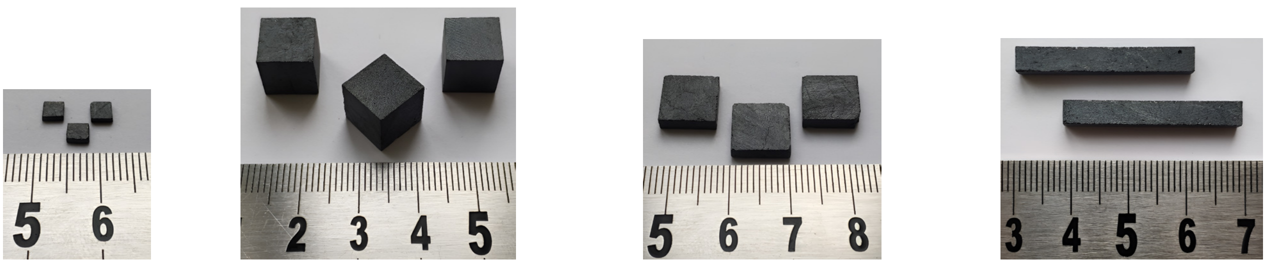
genesis. In the case of matrices with a crystalline structure, the above standard is applicable in only a very limited way. This is due to the fundamentally different nature of the materials under study. The assumption can be made that promising matrix materials will be introduced, if they achieve comparable or superior values to those presented in the standard document. In this regard, the indicators of physical properties given in state standard GOST R 50926-96 are chosen as reference values for comparison. The results of tests with reference values of the standard are presented in Table 2.

The data in Table 2 shows that Young's modulus values of crystalline matrices with pyrochlore and orthorhombic REE titanate structures reach values of $2.15 \cdot 10^8$ kN/m^2. This significantly exceeds the requirements for vitrified HLW ($5.4 \cdot 10^7$ kN/m^2). It also eliminates the issues of stacking during containerization, intermediate storage and disposal.

The compressive strengths fall within the range of common technical oxide ceramics: from 30 MPa for construction ceramics to 300 MPa for technical corundum. The tensile strengths for pyrochlore and

Table 1. Nomenclature of mineral-like matrices (MLMs) samples made for the mechanical and thermophysical properties study

Properties		Sample sizes, mm	Regulatory act	Number of prepared samples, pcs	
				Pyrochlore structure	Orthorhombic titanate structure
Flexural strength, flexural modulus		Square beam 4.5 × 4.5 × 35.0	GOST R 24409-80[7]	14	15
Compressive strength, Young's modulus in compression		Cube with a side of 10	GOST R 57606-2017 (ISO 20504:2006)[8]	21	19
Thermal conductivity	Thermal diffusivity	Rectangular parallelepiped 10.0 × 10.0 × 2.5	GOST R 24409-80 ASTM E1461-13[9]	3	4
	Heat capacity	Square plate 3.4 × 3.4 × 1.0	GOST R 24409-80 ASTM E1269-11[10]	3	3
Linear expansion temperature coefficient (LETC)		Square beam 4 × 4 × 30	GOST R 57743-2017 (ISO 17139:2014)[11]	5	3



(a) (b) (c) (d)

Fig. 1. Appearance of MLMs samples of various sizes: (a) heat capacity determining samples; (b) compressive strength determining samples; (c) thermal diffusivity determining samples; (d) ultimate strength in bending determining samples

7 GOST 24409-80. Interstate Standard. Ceramic electrotechnical materials. Methods of testing. Moscow: Standartinform; 2005.

8 GOST R 57606-2017 (ISO 20504:2006). National Standard of the Russian Federation. Fine ceramics. Test method for compressive behavior of continuous fiber-reinforced composites at room temperature, MOD. Moscow: Standartinform; 2017.

9 ASTM E1461-13. Standard Test Method for Thermal Diffusivity by the Flash Method. https://www.astm.org/e1461-13.html. Accessed January 15, 2023.

10 ASTM E1269-11. Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry.

11 GOST R 57743-2017 (ISO 17139:2014). National Standard of the Russian Federation. Fine ceramics. Thermophysical properties of ceramic composites — Determination of thermal expansion, MOD. Moscow: Standartinform; 2017.

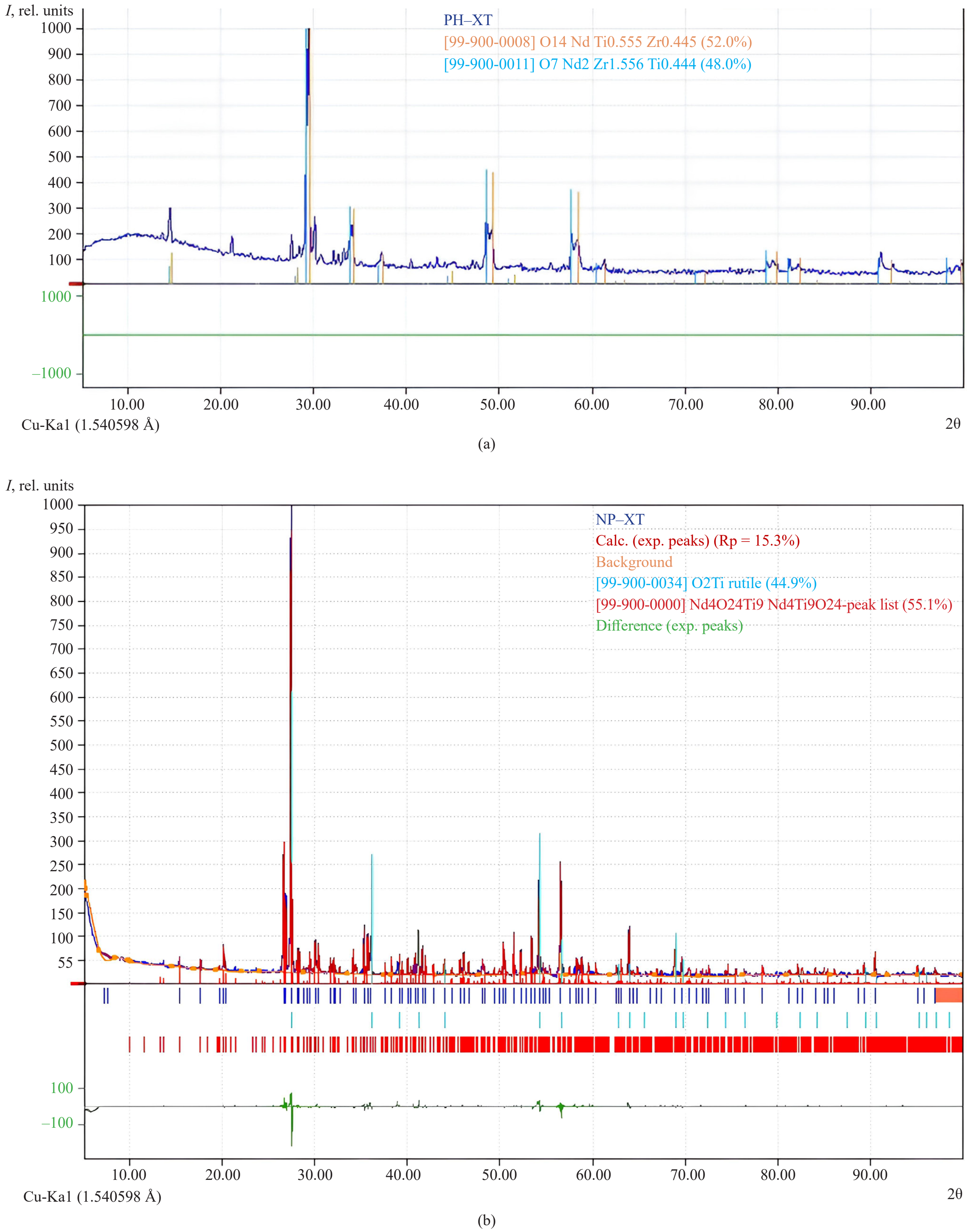


Fig. 2. Synthesized matrices X-ray diffraction patterns:
(a) titanate-zirconate matrix with the pyrochlore structure; (b) matrix with the structure of orothorhombic REE titanate

Table 2. Results of the study of mechanical and thermophysical properties

Type of test	GOST R 50926-96 requirements	Measured values	
		Pyrochlore Ln_2TiZrO_7	Pyrochlore Ln_2TiZrO_7
Thermal conductivity in the temperature range from 20 to 500°C, W/(m·K)	1–2	0.91–1.18	1.54–1.71
LETC in the temperature range from 20 to 500°C, $K^{-1} \cdot 10^{-6}$, no more than	9	9.12–10.10	6.96–7.88
Young's modulus in compression, kN/m^2, no less than	$5.4 \cdot 10^7$	$1.78 \cdot 10^8$	$2.15 \cdot 10^8$
Ultimate compressive strength, MPa, no less than	9	179 ± 26	150 ± 10
Bending strength, MPa, no less than	41	57.8 ± 3.9	20.6 ± 4.0

orthorhombic REE titanate were 179 and 150 MPa, respectively, while for AGM this index was 9 MPa.

The compressive strength of ceramic material and Young's modulus are usually characterized by values of the same order. The multiple differences observed in the case of the matrices studied is due to the presence of a certain number of pores in the samples. The averaged bending strength values amounted to 20.6 and 57.8 MPa for crystalline matrices with pyrochlore and orthorhombic REE titanate structures, respectively. The values obtained are 4–7 times lower than the values measured under compressive loads. This is typical for structural ceramics. Bending is a special case of simultaneous compression and tension [27]. The expectedly low index is also due to the crystalline structure which is comparatively poor in absorbing bending loads. At the stage of engineering barrier design this feature must be leveled. Package rigidity must be provided by the side wall of the non-returnable container used for intermediate storage and burial.

The results of heat capacity of the MLM samples in the temperature range of 50–500°C are presented in Fig. 3.

The diffusivity coefficients were obtained by comparing the experimental thermogram with the theoretical model. The determination results averaged over three parallel measurements of matrix samples of each type are presented in Fig. 4.

The dependencies of matrix thermal conductivities on temperature obtained by calculation are shown in Fig. 5.

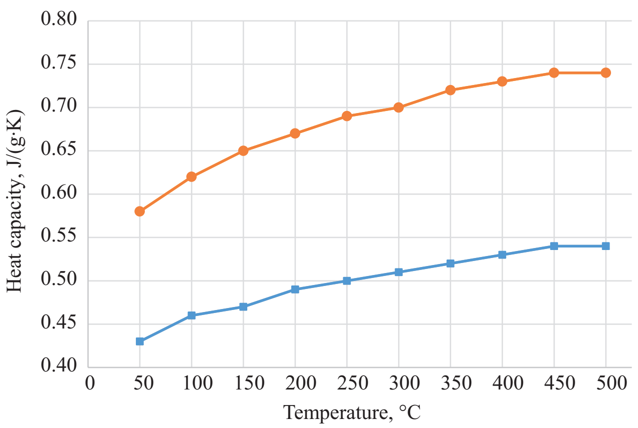


Fig. 3. Dependencies of the crystalline matrices heat capacity on temperature (squares, blue line—a matrix with a pyrochlore structure; circles, red line—a matrix with structure of an orthorhombic REE titanate)

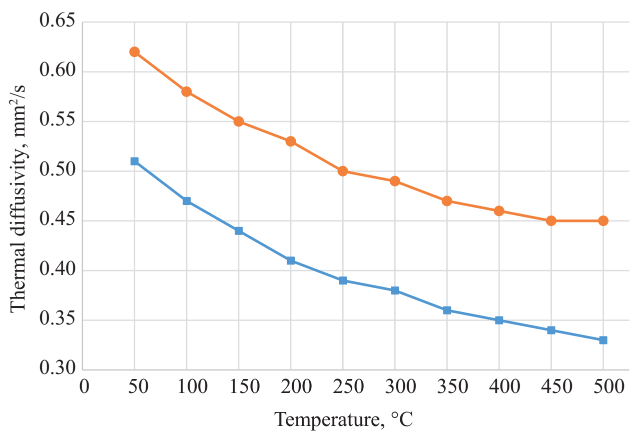


Fig. 4. Dependencies of the crystalline matrices thermal diffusivity on temperature (squares, blue line—a matrix with a pyrochlore structure; circles, red line—a matrix with structure of an orthorhombic REE titanate)

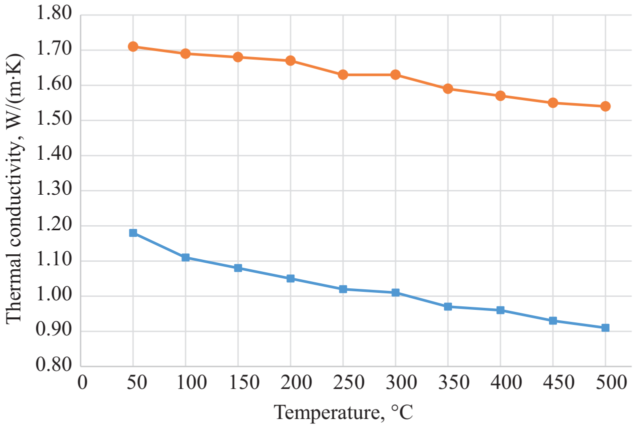


Fig. 5. Dependencies of the thermal conductivity coefficients of crystalline matrices on temperature (squares, blue line—a matrix with a pyrochlore structure; circles, red line—a matrix with structure of anorthorhombic REE titanate)

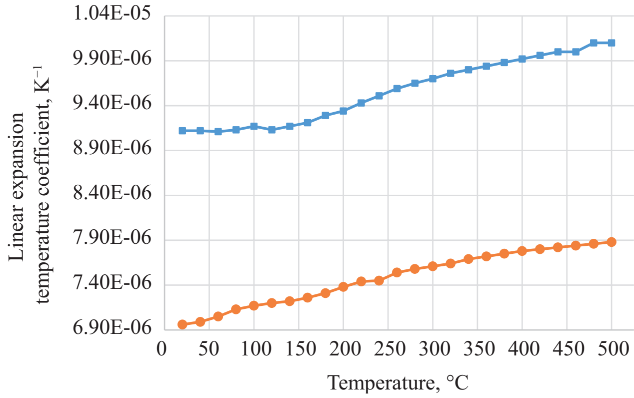


Fig. 6. Dependence of the matrices thermal expansion coefficient on temperature (squares, blue line—a matrix with a pyrochlore structure; circles, a red line—a matrix with an orthorhombic REE titanate structure)

As seen from the above dependencies, the thermal conductivity of the material is significantly affected by its chemical composition. The thermal conductivity of the matrix with orthorhombic REE titanate structure is higher in the whole range of investigated temperatures. This is apparently due to the contribution of the rutile phase with a very high intrinsic index, reaching reaches 5.3 W/(m·K) at 473 K [31]. The values of the matrices tested can vary from 0.9 to 1.7 W/(m·K) and almost completely fit into the range of 1–2 W/(m·K), as regulated by Russian state standard GOST R 50926-96. In general, the values of thermal conductivity of both matrices are close to the range of 0.8–1.5 W/(m·K). This is characteristic for oxide ceramics with variations caused by differences in chemical composition and porosity of the materials. Known exceptions include ceramics based on Al_2O_3, where the thermal conductivity in the range of 100–1000°C can range from 30 to 6 W/(m·K). The decrease in the level of thermal conductivity is also typical for non-metallic materials. An increase in the index can only be observed in the temperature region above 600°C for quartz glass and several types of translucent materials for thermal radiation.

The LETC was determined to be in the temperature range from 20 to 500°C in steps of 20°C at a heating rate of 3°C/min. The pyrochlore structure matrix was tested for five parallel samples, with the structure of orthorhombic REE titanate—for three samples. The measurement results are presented graphically in Fig. 6.

As seen from Fig. 6, there is a smooth growth of LETC in both cases with increasing temperature. This is typical for the vast majority of technical ceramics [29]. The increase of the index with increasing temperature is common for most solids. In the case of the materials being studied herein, this indicates the absence of allotropic transformations in the heating process where decrease in volume is possible. Fluctuations observed at 100 and 240°C on the curves of matrices with pyrochlore and orthorhombic REE titanate structures are apparently caused by measurement errors.

It should be noted that thermophysical and mechanical properties of the matrices being studied here are close to typical parameters of technical ceramics. They mainly correspond or exceed the characteristics of AGM used for immobilization of HLW.

Thus, we can tentatively conclude that crystalline matrices are able to fully perform the function of final forms for immobilization of the corresponding HLW fractions. The application of materials of this type will allow the long-term safety of intermediate storage sites and deep burial sites of conditioned wastes to be enhanced. The specific features of the matrices due to their crystalline structure are not a limitation to their application. Indeed, they are leveled out by the qualities of engineering barriers, especially primary packaging.

CONCLUSIONS

The study established the mechanical and thermophysical properties of crystalline matrices in the immobilization of REE-actinide fraction obtained on an enlarged scale using the method combining granular precursor and CCIM.

The compressive strength limits of matrices with pyrochlore and orthorhombic REE titanate structures were found to be 17–20 times higher than the index regulated for AGM. This fact indicates the possibility of safe handling of the final product during manipulations during *in situ* handling, transportation to the disposal site and during actual disposal.

The bending strength is lower than that of glass by up to 50%, due to the nature of the crystalline material which is poorly able to absorb tensile loads. However, this feature can be offset by the stiffness of the primary packaging.

The values of thermal conductivity coefficients in the temperature range of 50–500°C range from 0.91 to 1.71 W/(m·K) depending on the matrix composition. LETC is characterized by quite low values (7–10) · 10^{-6} K^{-1}. This is comparable to the values of corundum (8 · 10^{-6} K^{-1}) and quartz glass (8.5 · 10^{-6} K^{-1}).

In general, the mechanical and thermophysical properties of MLMs are comparable or superior to the regulated parameters of preserved final forms. This, in addition to other advantages, confirms the potential for their application in the immobilization of fractionated wastes.

When implementing the technology for the immobilization of fractionated HLW, attention must be paid to the development of specialized non-returnable containers taking into account the LETC of crystalline matrices and the relatively small bending strength limits.

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Authors' contributions

M.Yu. Kalenova, I.V. Kuznetsov, O.I. Stefanovskaya, V.A. Stepanov—study concept and methodological support.

M.Yu. Kalenova, I.V. Kuznetsov, A.Yu. Zobkova—draft manuscript preparation.

I.M. Melnikova, A.S. Shchepin, K.V. Klemazov—data collection.

O.N. Budin, V.A. Stepanov, K.V. Klemazov, I.M. Melnikova—analysis and interpretation of results.

A.S. Shchepin, O.N. Budin—specimen synthesis and sample preparation.

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