

Engineering Multicomposite Ceramic Systems with Improved Thermal Insulation and γ -Ray Shielding Performance

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Abstract. The article presents the results of complex studies of the assessment of alterations in thermal insulation and shielding characteristics in $1-x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO}_2 - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$ ceramics with variations in the ratio of components during their sintering, which leads to a change in the phase composition. It is determined that the addition of zirconium dioxide to the composition of the main $\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO}_2 - \text{Bi}_2\text{O}_3$ matrix leads to the formation of a monoclinic phase ZrCeO_2 , the increase in the contribution of which is directly proportional to the observed hardening effect, expressed in an elevation in hardness and bending strength. Thermal insulation properties of composite $1-x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO}_2 - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$ ceramics were studied depending on the variation of the zirconium dioxide component in the composition, the addition of which changes the strength and thermal insulation properties. During the experiments, it was established that a growth in the monoclinic ZrCeO_2 substitution phase in the composition of ceramics leads to a rise in the difference in temperatures on the front and back sides, which indicates an enhancement in thermal insulation properties, a growth in the temperature difference, which is due to the low thermal conductivity of zirconium dioxide. A general analysis of the shielding characteristics showed a positive effect of the formation of the ZrCeO_2 phase on the shielding efficiency, allowing the absorption capacity of ceramics to be increased by 10 – 15 %, while the resulting ceramics have high strength and thermal insulation properties

Keywords: composite ceramics, thermal insulation materials, protective shielding ceramics, absorption, shielding, radiation exposure.

Introduction

The expansion of the use of ionizing radiation for peaceful purposes, including in medicine, industry, energy and security, requires increased attention not only to the issues of use and application, but also protection from the negative impact of ionizing radiation on living organisms [1,2]. Also, significant attention should be paid not only to artificial sources of radiation that arise when using sources of ionizing radiation, but also to natural sources that can also have a negative impact. The main negative impact of ionizing radiation, especially gamma quanta, which have a high penetrating ability, is the possible damage to cells [3,4], which can cause mutations or diseases in the case of living organisms, and in the case of electronic devices, lead to destabilization of their performance or complete failure, which can be very critical, especially in

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cases where rapid replacement of failed equipment is not possible or is very difficult [5,6]. In this regard, in recent years, active research aimed at finding and creating new protective materials capable of containing the negative impact of ionizing radiation, as well as combining the possibilities of using shielding materials in various industries, including the possibility of operation in extreme conditions, has been carried out [7-10].

One of the potential solutions in this direction is the use of composite ceramics based on tellurium, cerium, zinc, tungsten and bismuth oxides, which have fairly high shielding efficiency for gamma and neutron radiation [11-14]. The consideration of the prospects for using these types of composite ceramics as materials for protection against the negative effects of ionizing radiation is primarily due to the content of heavy elements such as tungsten and bismuth, the use of which allows for an elevation in the effective atomic number (Z_{eff}) [15,16], which in turn allows for an increase in the value of the interaction (absorption) cross-section of gamma and neutron radiation. At the same time, the use of zinc or tellurium oxides allows obtaining optically active materials, opening up the possibility of using them as materials that have the ability to transmit light in a certain range while maintaining the stability of shielding [17,18]. The addition of bismuth, tungsten, or tellurium to the composition leads not only to an increase in absorption capacity due to large atomic numbers but also, in some cases, to an increase in strength due to the reinforcing properties of these elements.

An important role in the assessment of the applicability of composite ceramics and glass-like ceramics with good shielding efficiency indicators has recently been given to studies aimed at studying the possibility of combining high shielding indicators with mechanical strength and thermal barrier properties of ceramics, which play a very important role in the determining the applicability potential [19-21]. In this area, much attention is paid to the possibility of modification of composite ceramics with oxides that have low thermal conductivity and high strength and mechanical parameters, the addition of which will not only reduce thermal conductivity, but also increase the hardness and strength of composite ceramics. In this case, the most promising oxide for modification is zirconium dioxide, the chemical nature of which allows it to be combined with other types of materials, and its structural features provide high strength indicators and fairly low thermal conductivity values in comparison with other oxides of refractory elements used to modify materials [22-25].

The main aim of this work is to determine the effect of addition of zirconium dioxide in various concentrations (from 5 to 25 wt. %) to multicomponent ceramics based on $\text{WO}_3\text{-Bi}_2\text{O}_3\text{-ZnO-TeO}_2\text{-CeO}_2$, in order to determine the possibility of enhancement of the strength and thermal barrier characteristics of ceramics, the variation of which, in combination with changes in the parameters of the shielding characteristics, will make it possible to obtain a new class of materials that can be considered as one of the promising materials for protection against the negative effects of ionizing radiation. In this work, special attention is paid to the possibility of a combination of the strength, thermal insulation, and shielding properties of ceramics, with the aim of obtaining optimal compositions of multicomponent ceramics that have the potential for use in cases where a combination of thermal insulation properties, mechanical strength, and resistance to cracking, as well as shielding of ionizing radiation, is required.

Materials and methods

For the synthesis of multicomponent ceramics considered as heat-insulating and shielding materials, the method of solid-phase grinding with subsequent thermal sintering was used. The influence of stoichiometry variation due to the addition of a stabilizing component in the form of zirconium dioxide (ZrO_2) to the ceramic composition, selected for the purpose of enhancing strength characteristics, as well as elevation of thermal insulation, was determined by alteration of the stoichiometry of the components in the composition. The following refractory metal oxides were chosen as the basis (matrix): WO_3 ($T_{\text{melt}}=1473^\circ\text{C}$), Bi_2O_3 ($T_{\text{melt}}=817^\circ\text{C}$), ZnO ($T_{\text{melt}}=1975^\circ\text{C}$), TeO_2 ($T_{\text{melt}}=733^\circ\text{C}$), CeO_2 ($T_{\text{melt}}=2400^\circ\text{C}$); zirconium (IV) oxide ZrO_2 ($T_{\text{melt}}=2700^\circ\text{C}$) was chosen as the dopant. The dopant content in the total mass was 0%, 5%, 10%, 15%, 20%, 25%. Table 1 presents the stoichiometric compositions of the studied ceramics.

Table 1 – Stoichiometric compositions of the studied ceramics

Concentration of elements		Stoichiometric formula
WO ₃ -Bi ₂ O ₃ -ZnO-TeO ₂ -CeO ₂	ZrO ₂	
20 %	0 %	0.2WO ₃ -0.2Bi ₂ O ₃ -0.2ZnO-0.2TeO ₂ -0.2CeO ₂
19 %	5 %	0.19WO ₃ -0.19Bi ₂ O ₃ -0.19ZnO-0.19TeO ₂ -0.19CeO ₂ -5ZrO ₂
18 %	10 %	0.18WO ₃ -0.18Bi ₂ O ₃ -0.18ZnO-0.18TeO ₂ -0.18CeO ₂ -10ZrO ₂
17 %	15 %	0.17WO ₃ -0.17Bi ₂ O ₃ -0.17ZnO-0.17TeO ₂ -0.17CeO ₂ -0.15ZrO ₂
16 %	20 %	0.16WO ₃ -0.16Bi ₂ O ₃ -0.16ZnO-0.16TeO ₂ -0.16CeO ₂ -0.2ZrO ₂
15 %	25 %	0.15WO ₃ -0.15Bi ₂ O ₃ -0.15ZnO-0.15TeO ₂ -0.15CeO ₂ -0.25ZrO ₂

The main technological method for obtaining composite ceramic materials included the use of a mechanochemical solid-phase synthesis process, during which the initial components of refractory oxides were ground using a PULVERISETTE 6 classic line planetary mill (Fritsch, Berlin, Germany). Mechanochemical grinding was carried out in a tungsten carbide (WC) beaker with a capacity of 80 ml, using tungsten carbide grinding balls with a diameter of 10 mm. The ratio of the number of grinding bodies and the components to be ground was 2:1. The grinding rate was 250 rpm, and grinding was carried out for 30 minutes. Thermal annealing of the samples was performed by placing all the samples in special corundum crucibles placed in a SNOL muffle furnace (SNOL/Umega group, Utena, Lithuania), after which the furnace chamber was heated to a temperature of 1000 °C, at a heating rate of about 20 °C/min. Upon reaching the set temperature of 1000 °C, the temperature in the chamber was stabilized, followed by holding the samples inside the chamber for 5 hours, after which the heating was switched off and the samples were cooled together with the furnace until reaching a temperature that allowed the samples to be removed (room temperature).

The phase composition of the studied ceramics was determined depending on the variation of the components in the composition using the X-ray phase analysis method implemented on a D8 Advance ECO powder diffractometer (Bruker, Germany). X-ray diffraction patterns were recorded in the Bragg-Brentano geometry at an angular range of 2θ from 20 to 100 °, and the Rietveld method was used to assess the weight contributions and the presence of phases in the composition.

Determination of optical properties related to changes in optical transmittance over a wide wavelength range from 200 to 1000 nm was accomplished by obtaining transmittance spectra and their subsequent processing to calculate the transmittance at a wavelength of 500 nm. Measurements were performed using a SPECORD 200/210/250 PLUS spectrophotometer (Analytik Jena, Jena, Germany).

The strength properties were studied using the indentation method, and the Duroline M1 microhardness tester (Metkon, Bursa, Turkey) was used for measurements. The Vickers pyramid was used as an indenter; the load on the indenter was about 100 N. The bending strength was determined using the three-point bending method, implemented on the Duroline M1 testing machine (Metkon, Bursa, Turkey).

The thermal insulation properties of multicomponent ceramics were assessed by measuring the temperature difference of samples from both sides during 24 hours of sequential heating of one side of the samples on a heater. Based on the results of the temperature difference, the insulation temperature parameter was assessed, which determines the containment of temperature heating during operation. The measurements were carried out at a heating temperature of the front side of the sample to a temperature of 1000 °C, and the temperature change was monitored using thermocouples.

The determination of the efficiency of using the proposed ceramics as shielding protective materials to reduce the negative impact of gamma radiation was carried out in two stages. The first stage consisted of the simulation of the effects of gamma radiation interaction in a wide energy range with the ceramic material, with a variation in the ratio of

oxides used to obtain the samples. Simulation was carried out using the XCOM program code [26], freely available on the platform [27]. The calculations were performed using a code in which the materials are used as a mixture (Compound), the materials were oxides in a given ratio of components used for synthesis. The second stage of the efficiency assessment consisted of conducting experimental work related to determining changes in the intensity of gamma radiation created by the Cs^{137} source generating gamma quanta with an energy of 662 keV ($T_{1/2} = 30.08$ years). The experiments were carried out by placing the gamma-quantum source in a special container with a 3 mm diameter hole, into which the shielding sample was placed. A detection system was placed at a distance of 10 cm, allowing the intensity of gamma radiation to be estimated before and after using the shielding material. Using the Lambert-Beer law, the linear attenuation coefficient (LAC) was determined, on the basis of which such quantities as the mass attenuation coefficient (MAC), half-value layer (HVL) and mean free path (MFP) were calculated. These parameters made it possible to evaluate the efficiency of using the proposed ceramic compositions as shielding protective materials, as well as to determine the influence of composition variations due to the addition of zirconium dioxide to them on the change in absorption and shielding abilities.

Results and discussion

Figure 1 demonstrates the results of X-ray phase analysis of the studied ceramic samples depending on the variation of the component ratio in the ceramic composition with variation of the zirconium dioxide concentration. The general appearance of the presented X-ray diffraction patterns in the case of the initial sample without a dopant indicates that under the selected thermal annealing conditions, multicomponent ceramics are formed, represented by the presence of at least four main phases in the structure, with the dominance of the CeO_2 phase with a cubic type of crystal lattice and spatial syngony $\text{Fm-}3\text{m}(225)$ (PDF-00-064-0737), the contribution of which is more than 60 wt. % (see the data presented in Figure 1b). Also in the composition of ceramics, the formation of ZnTeO_3 phases with an orthorhombic type of crystal lattice and spatial syngony $\text{Pbca}(61)$ (PDF-01-072-1410), the weight contribution of which is about 20 wt.%, as well as two phases, one of which is the ZnWO_4 tungstate phase with a monoclinic type of crystal lattice and spatial syngony $\text{P2}/\text{c}(13)$ (PDF-01-088-0251) and the $\text{Bi}_2\text{Te}_4\text{O}_{11}$ phase with a monoclinic type of crystal lattice and spatial syngony $\text{P21}/\text{n}(14)$ (PDF-01-081-1330) is observed. Thus, the matrix of the initial ceramics is represented by a mixture of four phases, the distribution of which in the sample composition is equally probable and isotropic. When zirconium dioxide with a monoclinic crystal lattice type is added to the composition of ceramics, according to X-ray diffraction data, characteristic reflections appear (the presence of which was demonstrated by decomposition of reflections in the region of $2\theta = 25 - 35^\circ$ using the Rietveld method) for the presence of the monoclinic phase ZrCeO_2 (PDF-01-088-2391) in the composition, the formation of which occurs during mechanical mixing of powders with subsequent partial cationic substitution of zirconium by cerium. It should be noted that at the selected annealing temperatures of 1000 °C, as well as the presence of various components in the composition capable of initiating processes of polymorphic transformations in zirconium dioxide, these effects were not observed in the studied samples. For all selected compositions, the main diffraction reflections for all established phases are preserved, undergoing only changes in intensities characterizing the change in phase contributions in the samples (see data in Figure 1b).

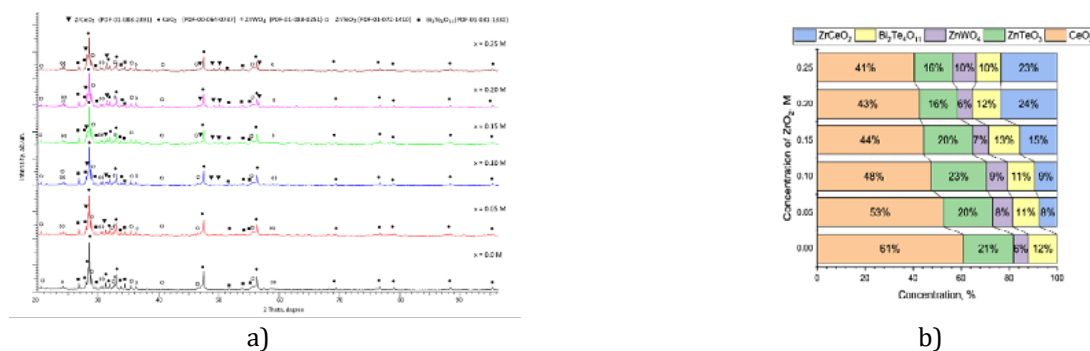


Figure 1. a) Results of X-ray diffraction of the studied samples of multicomponent ceramics depending on the addition of a stabilizing component in the form of ZrO_2 (The following symbols were used to designate the phases on the diffraction patterns): \blacktriangledown - ZrCeO_2 (PDF-01-088-2391), \bullet - CeO_2 (PDF-00-064-0737), \diamond - ZnWO_4 (PDF-01-088-0251), \star - ZnTeO_3 (PDF-01-072-1410), \blacksquare - $\text{Bi}_2\text{Te}_4\text{O}_{11}$ (PDF-01-081-1330)); b) Results of changes in the phase composition of multicomponent ceramics with variations in the ratio of components in the case of addition of ZrO_2

Figure 2 demonstrates the results of measurements of the optical properties of the studied multicomponent ceramics with a variation in the ratio of components in the case of addition of ZrO_2 to the composition, leading to the formation of the $ZrCeO_2$ phase, as well as, according to the data presented in Figure 1b, a change in the ratio of the main established phases while maintaining the dominance of the cubic phase CeO_2 in the composition. According to the assessment of the fundamental absorption edge values, which is determined by the dominant phase of CeO_2 , the change in the phase composition due to the formation of inclusions in the form of $ZrCeO_2$ leads to a slight shift, which is due to the formation of effects of local electronic states while maintaining the dominance of the cubic phase in the composition of ceramics.

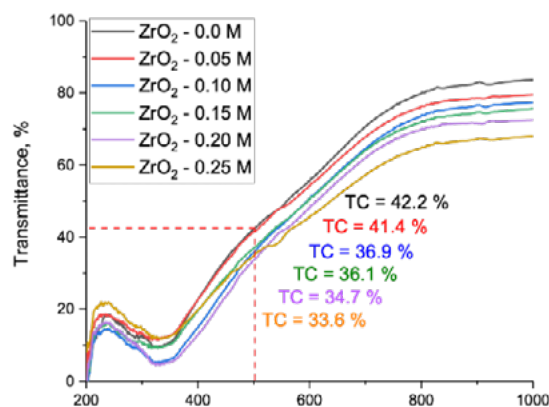


Figure 2. Evaluation results of optical properties of multicomposite ceramics with variation of concentration of zirconium dioxide in the composition

According to the obtained data, the addition of ZrO_2 to the composition of multicomposite ceramics leads to a decrease in the optical transmittance coefficient in the visible (380 – 780 nm) and near IR (above 780 nm) range by approximately 8 – 10 %. At the same time, in the case of the visible light wavelength range, the change in the optical transmittance coefficient depending on the concentration is non-linear, which indicates a direct effect of changes in the phase composition on the optical parameters of the ceramics. The observed decrease in the optical transmittance coefficient in this case can be explained by the effects of the emergence of additional scattering centers in the form of grain boundaries, the proportion of which increases due to a change in the phase ratio in the composition of the ceramics, while an increase in the weight contribution of the $ZrCeO_2$ phase in the composition of the ceramics can lead to an increase in stresses near the boundaries, which further enhances the scattering effect and, as a consequence, a decrease in the transparency of the ceramics in the visible and near IR range. The presence of a local maximum in the wavelength range from 200 to 310 nm is due to the presence of oxygen vacancies in the ceramic structure, the change in the concentration of which (by the change in the area and intensity of the peak) indicates the influence of changes in the phase composition on the structural effects associated with the formation of vacancy defects. In this case, the presence of oxygen vacancies leads to the creation of additional energy levels inside the band gap, which leads to an increase in absorption, which manifests itself as an absorption band in the UV region, which is quite clearly manifested at a zirconium dioxide concentration of 25 %. It should also be noted that the presence and subsequent change in the concentration of oxygen vacancies in the composition of ceramics can cause changes not only in optical properties, but also in thermophysical ones, since the presence of oxygen vacancies makes it possible to change the heat transfer rate by inhibiting the phonon mechanisms of heat exchange in ceramics, thereby increasing thermal insulation properties.

Figure 3 reveals the evaluation results of the strength characteristics of ceramics, presented in the form of dependencies of the change in the parameters of hardness and bending strength with a change in the concentration of ZrO_2 in the composition of ceramics. The general trend of changes in the values of hardness and bending strength indicates a positive effect of zirconium dioxide addition to the composition of WO_3 - Bi_2O_3 - ZnO - TeO_2 - CeO_2 ceramics, resulting in an increase in strength characteristics from 3 – 7 to 39 – 40 % compared to unmodified samples. At the same time, the trend of changes in strength characteristics has differences depending on the concentration of zirconium dioxide, from which it can be concluded that the change in strength characteristics is influenced by the change in the phase ratio, associated not only with the formation of the $ZrCeO_2$ phase in the composition of ceramics, but also by the general trend of changes in the weight contributions of the phases, the results of which are shown in Figure 1b.

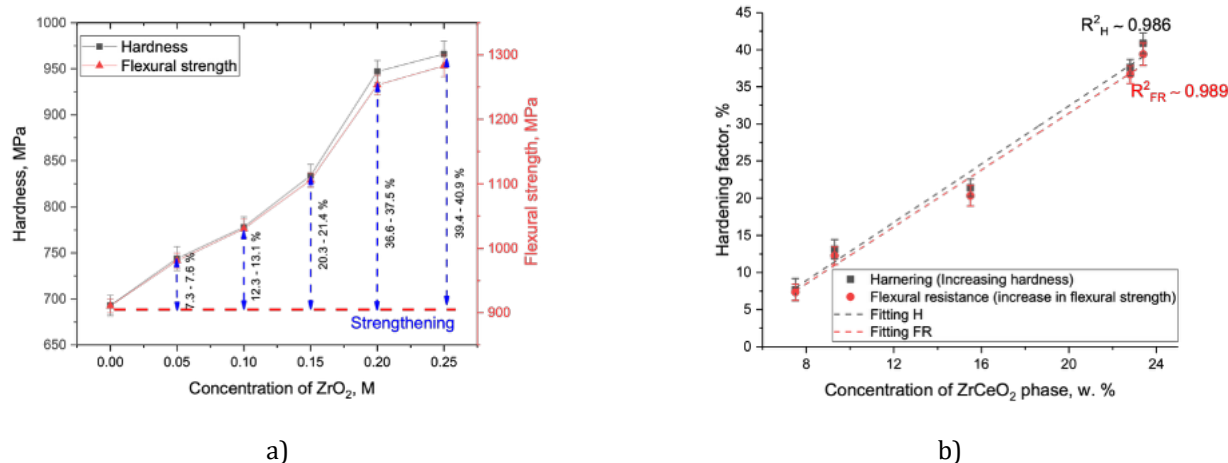


Figure 3. Results of the strength characteristics of the studied ceramics: a) Results of changes in the values of hardness and bending strength from variations in the concentration of components in the composition; b) Comparative analysis of the values of hardening and changes in the concentration of the $ZrCeO_2$ phase in the composition of ceramics

The dependence between the change in the weight contribution of the $ZrCeO_2$ phase in the composition of ceramics and the hardening factors (increase in hardness and bending strength) shown in Figure 3b reflects a direct relationship between these parameters, which in turn can be explained by changes in the structural features of ceramics associated with the formation of inclusions in the form of $ZrCeO_2$. Moreover, the observed changes in strength characteristics (hardness and bending strength) due to changes in the phase composition of ceramics are in good agreement with the results of a number of studies [28,29], in which the strengthening effect is associated with the formation of additional barrier obstacles caused by the presence of interphase boundaries and their contributions to restraining the propagation of microcracks and chips in the case of external mechanical loads [30,31]. The mechanism of microcrack containment in this case is due to the fact that the presence of interphase or intergranular boundaries leads to a change in the direction of microcrack growth or their complete inhibition in the case of a large number of barriers. At the same time, the presence of additional interphase boundaries leads to an increase in energy dissipation under mechanical loads and deformations, which in turn increases the resistance of the material, as well as an increase in crack resistance under external mechanical loads. Also, an important role in enhancement of resistance to softening under mechanical influences is played by local stresses near grain boundaries, arising due to a change in the weight contribution of the $ZrCeO_2$ phase in the main matrix, the presence of which can cause not only the formation of local stresses, but also the formation of the effect of transformation hardening, resulting from a change in the weight ratio of the cubic phase CeO_2 and the monoclinic phase $ZrCeO_2$, which is accompanied by an increase in volume, and as a consequence, the creation of compressive stresses, the presence of which can restrain the propagation of cracks under external mechanical loads (in the case of indentation or assessment of strength in three-point bending).

The effect of phase composition variation on the change in thermal insulation properties is one of the important criteria for the applicability of ceramics as thermal barrier materials used in microelectronics, allowing protection against overheating of the main elements during operation in extreme conditions, including exposure to high temperatures and radiation. The efficiency of thermal insulation during the operation of materials under high temperatures determines the resistance of not only the protective material to thermal expansion and oxidation processes, but also, in the case of protecting microelectronic devices, preventing or reducing to a minimum the effect of thermally induced deformations arising as a result of thermal expansion of the crystalline structure under prolonged thermal exposure.

Figure 4a-b shows the results of experiments reflecting the change in temperature on the front and back sides of ceramics during high-temperature heating (at a temperature of 1000 °C) over a long period of exposure, the difference between which reflects the temperature of the thermal insulation. As is known, in ceramics in the case of high-temperature exposure, the heat transfer mechanisms are associated with phonon processes consisting of changes in the vibrations of the crystal lattice, as a result of which energy is transferred from one area to another. In this case, the formation of heterogeneities in the structure, including those associated with changes in the concentration of point defects, grain

boundaries or interphase boundaries acting as dissipating centers, makes heat transfer difficult, resulting in a decrease in thermal conductivity.

As can be seen from the data presented, the change in phase composition due to the formation of the $ZrCeO_2$ phase in the ceramics leads to a growth in the difference between the temperatures on the front and back sides of the ceramics, which indicates an increase in the thermal insulation properties of the ceramics, which in this case can be due to the effects of inhibiting phonon heat transfer mechanisms. In this case, the formation of the monoclinic phase of $ZrCeO_2$ in the composition of ceramics leads to the creation of additional barrier boundaries (the presence of which, in the case of strength parameters, plays a key role in strengthening), as well as a change in the structural features associated with the volumes of crystal lattices with partial substitution of Ce^{4+} ions for Zr^{4+} ions in the composition of $ZrCeO_2$, as well as possible reverse substitution in the cubic phase of CeO_2 , in the case of differences in ionic radii (for Ce^{4+} , the value of $r_{Ce^{4+}} \sim 0.97 \text{ \AA}$, for Zr^{4+} , the value of $r_{Zr^{4+}} \sim 0.84 \text{ \AA}$), causing the formation of additional defects, including oxygen vacancies (a change in the concentration of which was established during the analysis of the optical transmission spectra). These defects lead to an increase in phonon scattering, which reduces the effective thermal conductivity and leads to an increase in thermal insulation, as evidenced by the results of the temperature difference (ΔT), as well as the data presented in Figure 4b. It should be noted that the formation of the $ZrCeO_2$ phase in the composition of ceramics leads not only to an increase in thermal insulation properties, but also to an increase in the stability of maintaining the temperature difference under prolonged exposure to high temperatures, which is clearly visible when comparing the curves of the back side of the ceramics after 20 hours of testing, for which in the case of unmodified ceramics there is an insignificant growth in temperature, indicating a loss of thermal insulation properties.

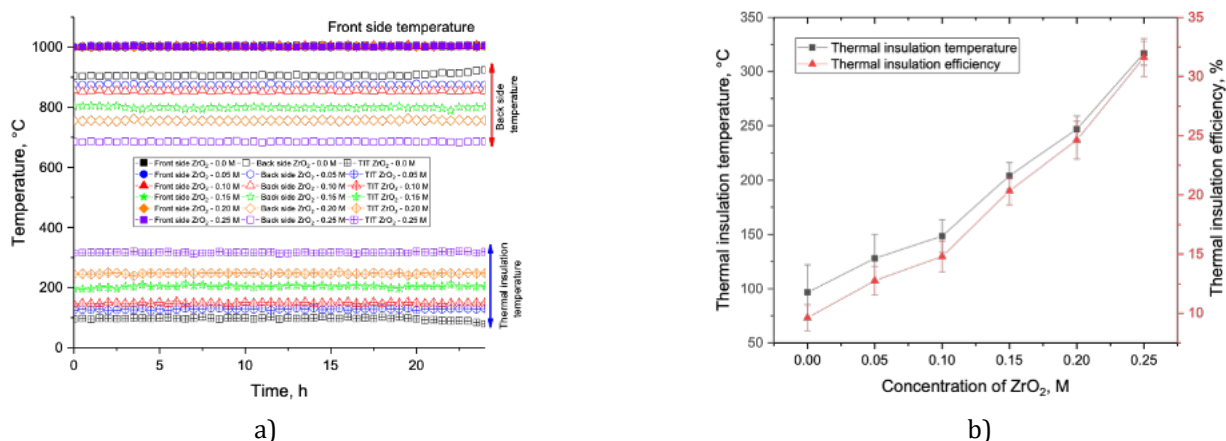


Figure 4. a) Results of experiments aimed at the identification of the thermal insulation properties of multicomponent ceramics; b) Results of a comparative analysis of changes in the ΔT value and thermal insulation efficiency with changes in the concentration of ZrO_2 in the composition of ceramics

The results of a comparative analysis of changes in the values characterizing the thermophysical parameters of ceramics when changing the components in the composition of ceramics are shown in Figure 5. The parameters chosen to determine the thermal physical properties were the thermal conductivity coefficient, which was measured using the longitudinal heat flow method, thermal conductivity losses, reflecting the change in the thermal conductivity coefficient with variations in the composition of the ceramics, as well as the efficiency of thermal insulation, calculated on the basis of changes in the temperature difference during heating tests of the samples. The general trend of observed changes in thermophysical parameters confirms the positive effect of changing the phase composition of ceramics due to the formation of the $ZrCeO_2$ phase in the structure, the presence of which leads not only to a reduction in thermal conductivity, but also to an increase in thermal insulation parameters, opening up the possibility of using these ceramics as thermal barrier coatings comparable in efficiency to zirconates [32,33].

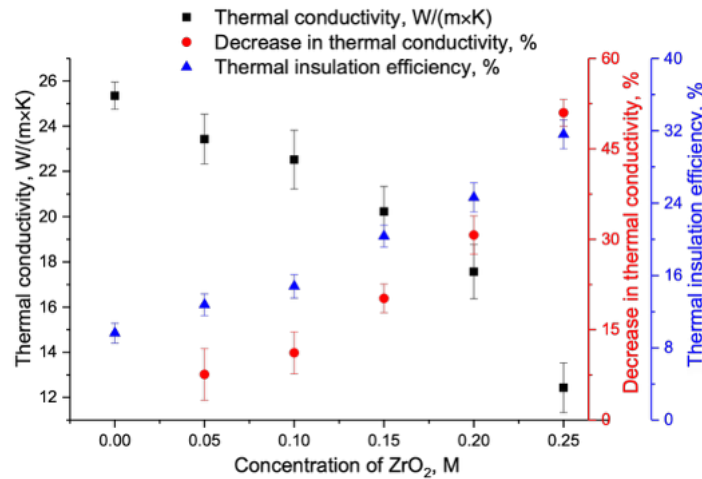


Figure 5. Results of a comparative analysis of changes in the values of the thermal conductivity coefficient, as well as changes in the decrease in thermal conductivity and increase in thermal insulation depending on the composition of ceramics with a change in the concentration of components

The possibility of creation of thermal barrier ceramics with high thermal insulation characteristics, which also combine high parameters of shielding gamma or neutron radiation, allows them to be considered not only as thermal barrier materials, but also as shielding materials, with the possibility of using them in extreme conditions. In this case, the combination of thermal insulation properties and shielding parameters opens up prospects for the use of these ceramics as protective materials for long-term storage of spent nuclear fuel, due to the need to keep it in conditions that do not allow overheating (high thermal insulation values), as well as shielding secondary radiation (electron, gamma) in combination with resistance to radiation damage. At the same time, as was shown in the works [34,35], the radiation damage accumulation leads to the destabilization of thermophysical parameters, causing a reduction in thermal conductivity, which in turn can be used to increase thermal insulation while maintaining a balance between the strength of the damaged layer during irradiation and the mechanisms of softening.

Figure 6 shows the results of the mass attenuation coefficient (MAC) estimation for all the ceramics under study, obtained by modeling the effects of the interaction of incident gamma quanta with energies in the range from 10^{-3} to 10^5 MeV. The results were obtained using the XCOM software code. The general form of the presented dependencies reflects small changes in the influence of variation in the ratio of oxide components in the composition of multicomponent ceramics. Based on the conducted simulation results, theoretical values of the shielding parameters were determined, which were compared with experimentally obtained data.

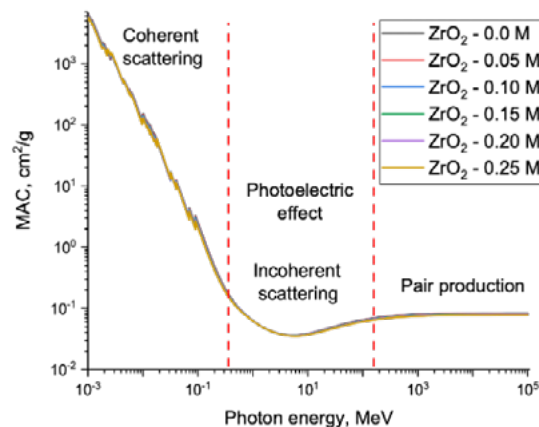


Figure 6. Evaluation results of the total cross-section value depending on the photon energy value obtained using the XCOM software code for multicomposite ceramics, depending on the concentration of the stabilizing component

Figures 7a-d reveal the evaluation results of the shielding efficiency of gamma quanta with an energy of 662 keV, reflecting the influence of the variation in the composition of multicomposite ceramics on the change in the shielding parameters. The data are presented in the form of comparative diagrams reflecting the comparison of the values obtained from the analysis of theoretical data obtained using the XCOM calculation code and experimentally obtained data that were obtained when evaluating the shielding of gamma quanta from the Cs^{137} source using the standard method for evaluating the change in intensity according to the Lambert-Beer law. The general trend of the observed changes between the theoretically obtained values of the shielding characteristics and the experimental data has a clearly expressed discrepancy, especially in the case of high concentrations of ZrO_2 in the composition, for which the magnitude of the difference in trends is about 10 – 12 % for cases of 20 – 25 wt. % ZrO_2 in the composition. This difference can be explained by the fact that in the case of simulation, the density values of the oxides used for calculations are taken into account, which, in the case of an increase in the proportion of ZrO_2 in the composition, results in total calculated density reduction from 6.9 g/cm^3 to 6.6 g/cm^2 , which is due to the fact that an elevation in the proportion of ZrO_2 with a density of 5.68 g/cm^3 leads to the substitution of weight fractions of oxides with higher density values (thus, the density of CeO_2 is about 7.22 g/cm^3 , and the densities of WO_3 and Bi_2O_3 are 7.16 and 8.9, respectively). In this case, the calculated data based on the density values reflect a slight decrease in the shielding values, which is about 2 – 3 %, which is quite acceptable for such compositions. However, the differences with the experimental data, especially in the case of large proportions of ZrO_2 in the composition, indicate a serious discrepancy between the theoretical and experimental data. The explanation for this difference may primarily be that the simulation did not consider the actual phase composition of the resulting ceramics, which has significant differences from the original oxides, which also causes changes in the density of the resulting ceramics. In this case, the shielding efficiency growth due to a change in the phase composition of ceramics indicates that in prediction and assessment of the applicability of multicomponent materials as protective shields, in the case where the composition of the obtained samples is a mixture of different phases, it is necessary to take into account not only the initial compositions, but also the phase composition, while for amorphous glass ceramics or glasses, the phase composition cannot be determined [36,37].

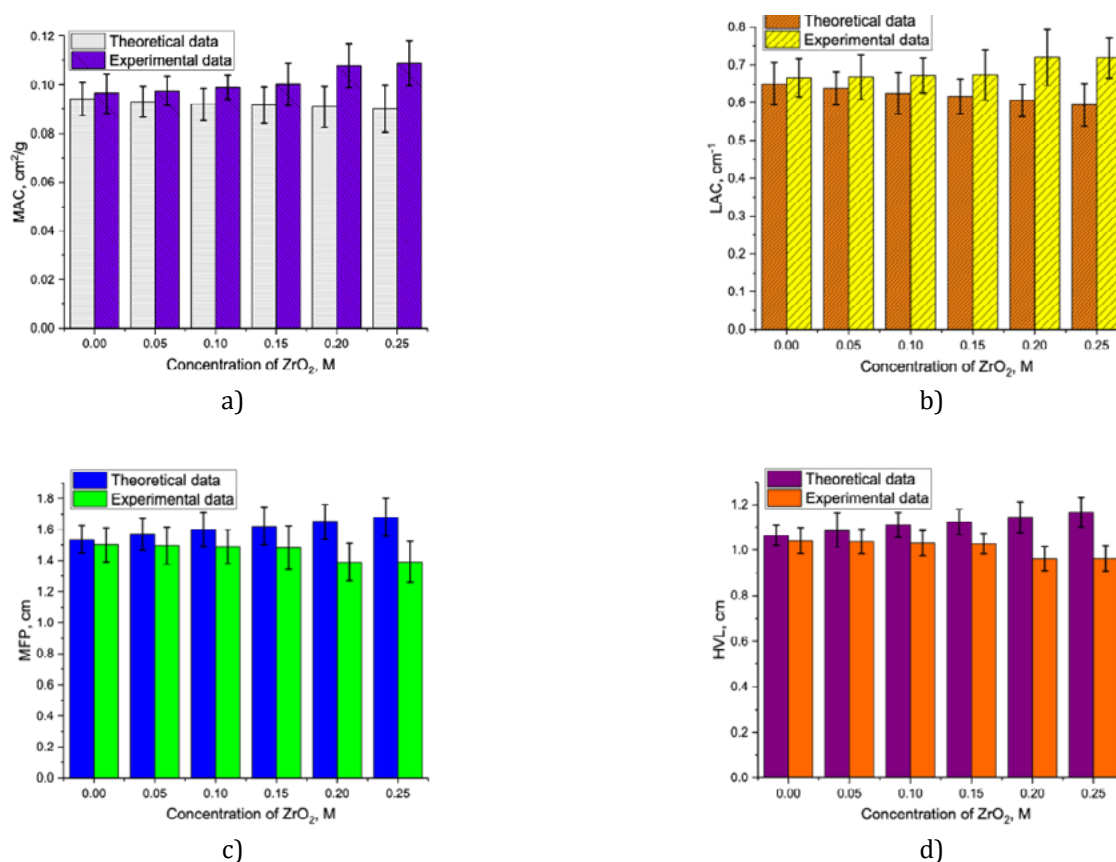


Figure 7. Evaluation results of the shielding characteristics of the studied ceramics depending on the variation in the concentration of the ZrO_2 component in the composition of the ceramics: a) MAC vs. concentration of ZrO_2 ; b) LAC vs. concentration of ZrO_2 ; c) MFP vs. concentration of ZrO_2 ; d) HVL vs. concentration of ZrO_2

It should be noted that the change in the phase composition of ceramics due to the formation of the $ZrCeO_2$ phase in the structure, as well as the general ratio of contributions, leads to an increase in the shielding efficiency, which is expressed in an increase in the MAC value, indicating an increase in the shielding efficiency. The results of the LAC value assessment for the studied ceramics show a trend towards a decrease in the value when the main components are replaced by zirconium dioxide, and the LAC value itself changes from 0.059 cm^{-1} to $0.63\text{-}0.65 \text{ cm}^{-1}$, which is a very good indicator, comparable to telluride glasses [38,39]. At the same time, the observed trend towards an increase in the LAC value indicates a positive effect of the influence of the formation of the $ZrCeO_2$ phase in the composition of ceramics on increasing the shielding efficiency. The obtained results indicate that changing the phase composition of ceramics allows not only the enhancement of the efficiency of thermal insulation and the strength of ceramics, but also the growth of shielding efficiency. Moreover, the inverse dependence of the LAC and MFP values shows that an increase in the zirconium dioxide concentration decreases the value from $\sim 1.6\text{-}1.7 \text{ cm}$ to $\sim 1.3\text{-}1.4 \text{ cm}$, which indicates that a change in the phase composition of ceramics leads to a reduction in the photon path length due to the presence of absorption centers, as well as additional structural features. The observed change in the HVL value from 1.2 cm to $1.0\text{-}1.1 \text{ cm}$ depending on the concentration of zirconium dioxide indicates that the addition of zirconium dioxide to the composition of ceramics, which leads to a change in the phase composition, leads to an increase in the ability of ceramics to absorb and scatter photon radiation, which leads to a decrease in the thickness of the material required to halve the intensity of gamma radiation, which can be used for practical applications.

Figure 8 shows the evaluation results of the shielding efficiency value determined on the basis of changes in the MAC value obtained experimentally during shielding of gamma quanta with an energy of 662 keV , when comparing it with the MAC value obtained for lead (Pb), which is a standard for shielding materials due to the highest shielding and absorption efficiency. Also shown for comparison are the results of the evaluation of the density values of multicomposite ceramics in comparison with the density value of lead. These parameters allow not only to evaluate the shielding efficiency of the proposed ceramic compositions in comparison with each other at the composition variation, but also to determine how promising the use of these ceramics for shielding is in comparison with the density values, which are one of the determining criteria in the assessment of the mass and dimensional parameters.

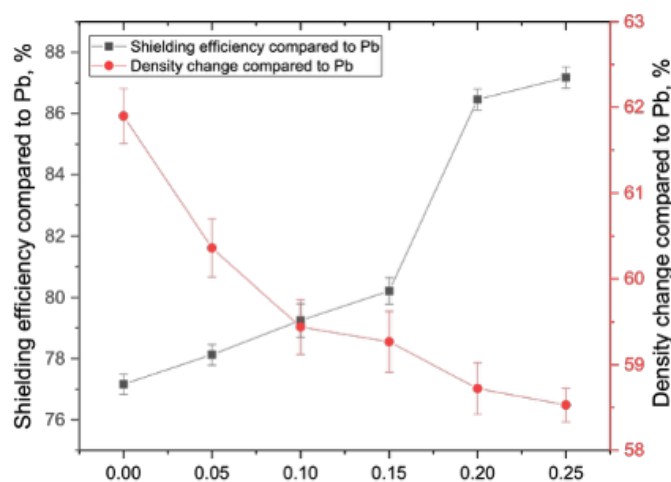


Figure 8. Results of comparative analysis of changes in the shielding efficiency parameter and density reduction when ZrO_2 is added to the composition of ceramics in comparison with the shielding parameters of the density of lead (reference sample for shielding)

As is evident from the data presented, alteration of the phase composition of ceramics by addition of zirconium dioxide to the composition allows the shielding efficiency growth from 77 to 86 % compared to the shielding efficiency of lead, while addition of more zirconium dioxide allows reduction of the density of ceramics from 62 % to 58.5 % compared to the density of lead at maintenance of shielding efficiency of more than 80 – 85 %.

Conclusion

The results of complex studies of the effect of the addition of zirconium dioxide to WO_3 - Bi_2O_3 - ZnO - TeO_2 - CeO_2 ceramics on the change in strength, thermal insulation and shielding characteristics were obtained.

According to the studies conducted, it was established that the formation of the $ZrCeO_2$ phase in the composition leads to an elevation in strength characteristics due to the formation of a hardening effect associated with the presence of interphase boundaries, the concentration of which increases with $ZrCeO_2$ phase growth in the composition.

During the studies conducted, it was determined that the addition of zirconium dioxide to the composition of multicomposite ceramics, which is accompanied by the formation of the $ZrCeO_2$ phase, an increase in the thermal insulation efficiency due to a decrease in the thermal conductivity of ceramics due to the thermal physical parameters of zirconium dioxide. The formation of this phase leads to an increase in scattering centers that prevent phonon heat transfer, which in turn leads to a slowdown in heat transfer.

Evaluation of the shielding efficiency with variations in the ratio of components in the composition of multicomposite ceramics in comparison with the reference values of shielding efficiency when using lead showed an increase from 77 to 86 % of shielding in comparison with the efficiency of lead at alteration of zirconium dioxide concentration in the composition, while substitution of heavy elements by zirconium dioxide makes it possible to reduce the density of ceramics to 58 – 60 % of the density of lead at maintenance of high shielding efficiency.

The general conclusion of the conducted research is that the addition of zirconium dioxide to the composition of multicomponent WO_3 - Bi_2O_3 - ZnO - TeO_2 - CeO_2 ceramics leads not only to an increase in strength and hardness, together with an increase in thermal insulation due to interphase boundaries and structural defects associated with oxygen vacancies, but also to an increase in the efficiency of gamma radiation absorption. The results of the comparative analysis indicate the prospects for using the proposed ceramic compositions as protective shielding materials combining high strength and thermal insulation indicators.

The contribution of the authors

Uglov V. and **Blynskiy P.** contributed equally to the research and preparation of the manuscript. The authors jointly developed the concept and design of the study, performed data collection, processing, and analysis, and carried out the discussion and interpretation of the results. Material synthesis and experimental investigations were also conducted jointly by the authors. Both authors have read and approved the final version of the manuscript.

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Жылу оқшаулау сипаттамалары жоғары және ү-сәулеленуден тиімді қорғанысты қамтамасыз ететін көпкомпонентті керамикалық композиттік жүйелерді жобалау

Аңдатпа. Мақалада 1- x(TeO₂ – CeO₂ – WO₃ – ZnO₂ – Bi₂O₃) – xZrO₂ құрамды керамикалардың компоненттер арақатынасын өзгерту арқылы спектеу кезінде фазалық құрамының өзгеруіне байланысты жылуоқшаулағыш және ү-сәулеленуден қорғаныш қасиеттерінің өзгерістерін бағалау бойынша кешенді зерттеулердің нәтижелері ұсынылған. Негізгі TeO₂ – CeO₂ – WO₃ – ZnO₂ – Bi₂O₃ матрицасына цирконий диоксидін енгізу нәтижесінде моноклиндік ZrCeO₂ фазасының түзілетіні анықталды. Осы фазаның үлесінің артуы материалдың қаттылығы мен иілу кезіндегі беріктігінің жоғарылауымен сипатталатын беріктену әсерімен тікелей байланысты. 1- x(TeO₂ – CeO₂ – WO₃ – ZnO₂ – Bi₂O₃) – xZrO₂ композиттік керамикаларының жылуоқшаулағыш қасиеттері құрамдағы цирконий диоксиді мөлшерінің өзгеруіне байланысты зерттелді. Цирконий диоксидін қосу материалдың беріктік және жылуоқшаулағыш сипаттамаларын өзгертетіні анықталды. Тәжірибелер барысында керамика құрамындағы моноклиндік ZrCeO₂ орынбасушы фазасының үлесі артқан сайын үлгінің алдыңғы және артқы беттері арасындағы температура айырмасы өсетіні анықталды, бұл жылуоқшаулағыш қасиеттердің жақсарғанын көрсетеді.

Температура айырмасының артуы цирконий диоксидінің төмен жылуөткізгіштігімен түсіндіріледі. Экранирлеу қасиеттерінің жалпы талдауы $ZrCeO_2$ фазасының түзілуі радиациялық қорғаныс тиімділігіне оң әсер ететінін көрсетті. Нәтижесінде керамиканың сәулені жұту қабілеті 10 – 15 % артады. Сонымен қатар алынған керамикалар жоғары беріктікке және жақсартылған жылуоқшаулағыш қасиеттерге ие.

Түйін сөздер: композиттік керамика, жылуоқшаулағыш материалдар, қорғаныш экрандаушы керамика, жұту, экранирлеу, сәулелену әсері.

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Проектирование многокомпонентных керамических композитных систем с повышенными теплоизоляционными характеристиками и эффективной защитой от γ -излучения

Аннотация. В статье представлены результаты комплексных исследований по оценке изменений теплоизоляционных и экранирующих характеристик керамики состава $1-x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO}_2 - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$ при варьировании соотношения компонентов в процессе спекания, что приводит к изменению фазового состава. Установлено, что введение диоксида циркония в состав основной матрицы $\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO}_2 - \text{Bi}_2\text{O}_3$ приводит к образованию моноклинной фазы $ZrCeO_2$, увеличение содержания которой прямо пропорционально наблюдаемому эффекту упрочнения, выражающемуся в повышении твёрдости и прочности при изгибе. Теплоизоляционные свойства композитной керамики $1-x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO}_2 - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$ были исследованы в зависимости от содержания диоксида циркония в составе, добавление которого изменяет прочностные и теплоизоляционные характеристики материала. В ходе экспериментов установлено, что увеличение доли моноклинной замещающей фазы $ZrCeO_2$ в составе керамики приводит к возрастанию разности температур между лицевой и тыльной сторонами образца, что свидетельствует об улучшении теплоизоляционных свойств. Рост температурного перепада обусловлен низкой теплопроводностью диоксида циркония. Общий анализ экранирующих характеристик показал положительное влияние образования фазы $ZrCeO_2$ на эффективность радиационной защиты, позволяя увеличить поглощающую способность керамики на 10 – 15 %. При этом полученная керамика характеризуется высокой прочностью и улучшенными теплоизоляционными свойствами.

Ключевые слова: композитная керамика, теплоизоляционные материалы, защитная экранирующая керамика, поглощение, экранирование, радиационное облучение.

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