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## EVALUATION OF THE APPLICATION EFFICIENCY OF MULTICOMPONENT CERAMICS AS PROTECTIVE SHIELDING AND THERMAL BARRIER MATERIALS

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**Abstract.** The paper presents the comparative analysis results of the shielding characteristics of multicomponent ceramics obtained by mixing  $\text{TeO}_2$ ,  $\text{CeO}_2$ ,  $\text{WO}_3$ ,  $\text{ZnO}$ ,  $\text{Bi}_2\text{O}_3$  and  $\text{ZrO}_2$  oxides in various molar ratios, enabling acquisition of ceramics with a variable phase composition. According to the results of X-ray phase analysis, the addition of  $\text{ZrO}_2$  to the composition of composite ceramics leads to the formation of a monoclinic substitution phase  $\text{ZrCeO}_2$ . The contribution growth of the latter results in strength properties growth due to a change in the concentration of interphase boundaries in the composition of ceramics. During the tests conducted for resistance to thermal effects capable of leading to destabilization of the crystalline structure, it was established that an increase in the contribution of  $\text{ZrO}_2$  in the composition of ceramics leads to an increase in resistance to thermally induced softening processes, and an increase in the stress resistance of ceramics during tests for thermal shock effects. During determination of the shielding characteristics of the studied ceramics, it was found that the formation of a stabilizing  $\text{ZrCeO}_2$  phase in the composition of multicomponent ceramics leads to an elevation in the shielding efficiency, as well as the stability of the preservation of shielding characteristics as a result of long-term thermal effects and thermal shock tests. Moreover, the greatest increase in the efficiency of the measured parameters is observed in the case of assessment of thermal insulation characteristics. The increase in the latter is more than 2.5 times compared to non-stabilized ceramics, which do not contain inclusions in the form of the  $\text{ZrCeO}_2$  phase.

**Keywords:** composite multicomponent ceramics, shielding materials, thermal shock exposure, stabilization, doping

### 1. Introduction

Interest in the creation of highly effective shielding materials is primarily due to the need to reduce the negative impact of ionizing radiation on living organisms and microelectronic devices, as well as the search for alternative materials to replace traditional lead and tungsten protective shields, which are highly effective in shielding, but have a number of disadvantages, including toxicity during prolonged contact (for lead) and high cost of manufacture (for tungsten). In recent years, much attention in this area of research has been paid to assessment of the possibilities of creation of shielding protective materials based on composite ceramics. Its concept includes combination of the properties of various oxide compounds, variation of the ratio of which makes it possible to obtain various materials that have high strength indicators, resistance to external influences [1-3], including aggressive environments, and have high shielding characteristics comparable to

those of lead. Interest in composite multicomponent ceramics and amorphous-like glasses is primarily due to the possibility of the material density reduction by variation of the components in the composition while maintaining high shielding efficiency indicators, with the possibility of bringing the parameters to values close to the shielding parameters of lead. In most cases, the density of composite multicomponent ceramics is about 3 – 5 g/cm<sup>3</sup>, while the density of lead is about 11.3 g/cm<sup>3</sup>. The reduction of density by more than 2 – 2.5 times compared to lead while maintaining high values of shielding characteristics, as well as the non-toxicity of ceramics in contrast to lead, allows their consideration as one of the key parameters in the field of shielding protective materials applicable in the medical sector, aerospace and energy industries [4,5]. It should also be noted that interest in composite ceramics in the field of protection of microelectronic devices is due to the need to reduce weight and overall dimensions, which play a very important role in spacecraft, for which any kilogram of weight when launched into orbit requires a large amount of fuel resources. In addition to shielding characteristics, recently much attention in the creation of composite multi-component shielding ceramics or glasses has been paid to their strength and heat-insulating parameters. These values determine their applicability in extreme operating conditions, including high mechanical loads, long-term mechanical friction, thermal effects and sudden changes in operating temperature conditions [6,7]. Elevation of strength characteristics, as well as resistance to external influences, along with high shielding characteristics, is one of the key areas of research, for the implementation of which methods of doping with high-strength components are used. The addition of these to the composition of ceramics or glasses can initiate the formation of inclusions that have a reinforcing effect, the occurrence of which increases resistance to external influences [8,9]. This effect is based on the possibility of filling the intergranular space with impurity inclusions in the form of new phases, which leads to the creation of interphase boundaries that prevent the spread of microcracks under external influences, as well as inhibition of oxidation processes caused by the diffusion of oxygen into the sample under high-temperature exposure. In this case, the choice of stabilizing components is usually based not only on the possibilities of alteration of the strength characteristics, but also enhancement of the shielding efficiency due to changes in the overall value of  $Z_{\text{eff}}$  [10-13], which plays a very important role in determination of the shielding characteristics, especially in the case of shielding gamma quanta with energies up to 1 MeV, which are the most common type of gamma quanta, and also have high penetrating power [14-16].

The main aim of this study is to determine the prospects for using composite 1-  $x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO} - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$  ceramics as shielding materials by comparative analysis of shielding efficiency depending on the variation of the ratio of components in the composition, as well as to determine the influence of external influences on maintenance of the stability of shielding and thermal insulation characteristics [17,18]. At the same time, much attention in the work is paid to assessment of the prospects for using these composite ceramics not only as shielding materials, but also as high-strength thermal barrier coatings, interest in which is due to the field of heat-resistant materials used in extreme conditions. The combination of the obtained results made it possible to evaluate both the prospects for using the proposed composite ceramics as protective materials and to expand the general understanding of the variation of phase composition in composite ceramics due to the observed phase transformations caused by changes in the ratio of components [19,20].

## 2. Material and Methods

The variation of components in the ceramics was carried out by addition of  $\text{ZrO}_2$  to the five-component mixture in a molar fraction from 0.05 M to 0.25 M. In this case, all the initial oxides were purchased from Sigma-Aldrich (St. Louis, Missouri, USA), the chemical purity of the oxide powders was about 99.95 %. The synthesis of  $x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO} - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$  ceramics was carried out by mechanochemical solid-phase milling of oxide components in different molar fractions, the variation of which is due to the addition of  $\text{ZrO}_2$  to the composite during mixing. Grinding for the purpose of obtaining a uniform distribution of all components in the volume was carried out using a PULVERISETTE 6 classic line planetary mill (Fritsch, Berlin, Germany), the grinding speed was about 250 rpm, the grinding time was 30 minutes. After grinding, the samples were removed from the grinding cup (tungsten carbide), divided into equal parts and annealed in a muffle furnace at a temperature of about 1000 °C (the heating rate until the final set temperature was reached was 20 °C/min). Annealing was carried out for 5 hours, after which the samples cooled together with the furnace for 24 hours until the furnace chamber had completely cooled.

After sintering, the obtained powders were subjected to a phase composition study, the changes of which in this case were due to a variation in the ratio of components in the composition of ceramics when  $\text{ZrO}_2$  was added to them. The study of phase changes that occurred as a result of thermal sintering was carried out using the X-ray phase analysis method, performed using a D8 Advance ECO X-ray diffractometer (Bruker, Germany). To assess the phase composition, the Diffrac EVA v.4.2 software was used, the application of which made it possible to determine with high accuracy (at least 0.1 wt. %) changes in the phase ratio in the composition, the establishment of which was carried out using the method of comparative analysis of the observed diffraction reflections with the data of the card values taken from the PDF-2 (2016) database. Tests of heat resistance (resistance of ceramics to thermal shock effects), including a sharp change in the temperature of impact on the samples studied, were carried out according to the standard method. It consisted of rapid heating (heating rate 50 °C/min) of the samples to a given temperature of 1000 °C, 1300 °C and 1500 °C, holding at this temperature for 30 minutes and then quickly removing it into the air, which leads to the creation of a sharp temperature gradient. To determine the effect of thermal shock effects on ceramic samples, as well as to establish the relationship between the addition of  $\text{ZrO}_2$  to the composition on resistance to high-temperature degradation, measurements of the hardness and bending strength of the samples under study were carried out before and after a specified number of cyclic tests. The number of test cycles was at least 10, which made it possible to evaluate the resistance of ceramics to high-temperature degradation, as well as to determine its effect on the softening degree of ceramics. Experimental work was also carried out to identify the effect of high-temperature degradation on the preservation of thermal insulation properties, which included testing samples when heated on one side and assessing the thermal insulation temperature parameters by calculating the temperature difference on both sides. These experiments were carried out at a test temperature of 1500 °C, and the temperature difference was controlled using thermocouples.

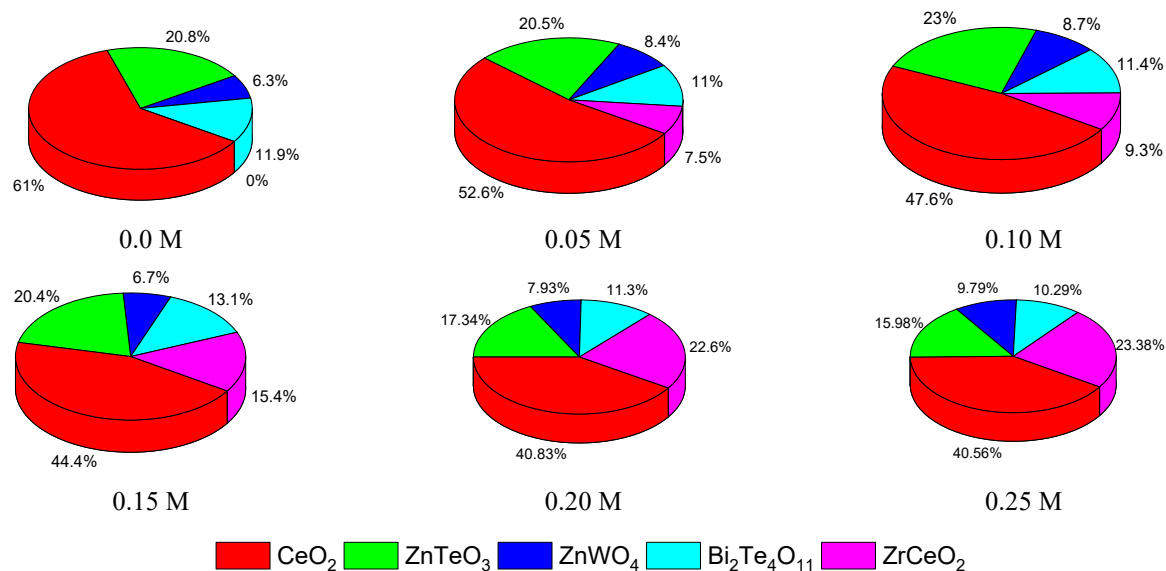
The shielding performance of the ceramic samples was evaluated using a  $\text{Cs}^{137}$  gamma source ( $E_\gamma = 662 \text{ keV}$ ) and a detector placed 10 cm from the source. From the measured intensity changes with and without the ceramic shielding, the linear and mass attenuation coefficients as well as the half-value layer thickness were determined.

### 3. Results and discussion

One of the important criteria playing a key role in determination of the applicability potential of composite ceramics is their phase composition, the change of which directly depends on the conditions of ceramic synthesis. In most cases, during production of telluride composite materials, the phase composition is represented by an amorphous nature, caused by the melt of tellurium oxide, the content of which plays a very important role in determination of the shielding parameters. Moreover, in the case of addition of cerium dioxide and tungsten oxide to the composition of composite glasses, the melting point of which is significantly higher, as a rule, the phase composition of composite telluride materials is represented by a mixture of different phases, the variation of the weight contributions of which determines the key role both in the shielding characteristics and in the strength parameters.

The circular diagrams (see Figure 1) show the X-ray phase analysis of the studied  $x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO} - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$  ceramics with varying component ratios, reflecting the role of adding  $\text{ZrO}_2$  in the phase formation processes in ceramics during thermal sintering. According to the presented X-ray phase analysis data, the dominant phase in the composition of composite ceramics is the cubic phase  $\text{CeO}_2$ , the weight contribution of which varies from 61 to 40.5 wt. % depending on the concentration of  $\text{ZrO}_2$ , the addition of which, as is evident from these changes in weight contributions, leads to the formation of the monoclinic  $\text{ZrCeO}_2$  phase with a subsequent increase in the share of its weight contribution. The samples also contain inclusions in the form of the orthorhombic  $\text{ZnTeO}_3$  phase, the monoclinic phase of zinc tungstate ( $\text{ZnWO}_4$ ), and the monoclinic  $\text{Bi}_2\text{Te}_4\text{O}_{11}$  phase, associated with phase transformations of oxides during their thermal sintering. The formation of the  $\text{ZrCeO}_2$  phase when  $\text{ZrO}_2$  is added to the composite ceramics is due to the processes of cationic substitution of zirconium by cerium, which leads to the formation of a substitution solid solution phase, while the selected conditions of thermal annealing at a temperature of 1000 °C do not allow the initiation of polymorphic transformation processes in  $\text{ZrO}_2$ , which also maintains the stability of the crystal structure. Thus, it is possible to make a general conclusion that the selected conditions of thermal sintering of ceramics at a temperature of 1000 °C do not lead to amorphization or vitrification of the crystalline structure. Also, the observed phase changes are associated with the processes

of structural transformations caused by the formation of the substitution  $\text{ZrCeO}_2$  phase, with the addition of  $\text{ZrO}_2$  with a monoclinic type of crystal lattice. Its change occurs due to the partial substitution of zirconium cations ( $\text{Zr}^{4+}$ ) by cerium cations ( $\text{Ce}^{4+}$ ), as a result of which there is an increase in this phase in the composition of ceramics, and a decrease in the weight contribution of the  $\text{CeO}_2$  phase due to its partial transformation.

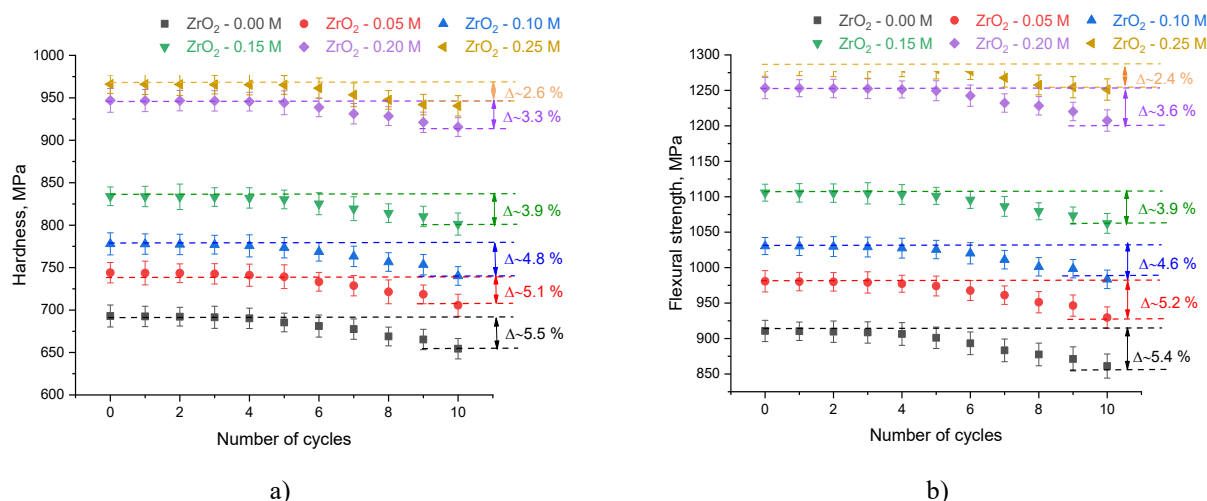


**Fig.1.** Results of X-ray phase analysis of the studied  $x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO} - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$  ceramics at varying  $\text{ZrO}_2$  concentration

At the same time, the weight contributions of  $\text{ZnTeO}_3$ ,  $\text{ZnWO}_4$  and  $\text{Bi}_2\text{Te}_4\text{O}_{11}$  in the composition of ceramics remain almost unchanged, and small variations in their weight contributions are due to the degree of manifestation of their diffraction reflexes on diffraction patterns, which affects the determination of the weight contribution. The formation of the zinc tungstate phase ( $\text{ZnWO}_4$ ) in the composition of composite ceramics can contribute to an increase in the stability of both strength parameters and the accumulation of radiation damage, since it is known that tungstates have fairly high resistance to radiation exposure. In turn, the content of this phase in the composition of composite ceramics is stable regardless of the concentration of the  $\text{ZrO}_2$  dopant, from which it can be concluded that the addition of a stabilizing dopant does not have a significant effect on its formation. Similar trends of small variations in weight contributions are also observed for the  $\text{ZnTeO}_3$  and  $\text{Bi}_2\text{Te}_4\text{O}_{11}$  phases, from which it can be concluded that the substitution processes occur in cerium dioxide. Some of the important parameters influencing the determination of the applicability potential of composite ceramics as protective or heat-insulating materials are their strength characteristics (hardness, bending strength), as well as maintenance of their stability under external influences, including a sharp change in temperature, the variation of which leads to the initialization of oxidation processes of the near-surface layer, leading to softening and destruction with subsequent embrittlement. In this case, determination of the resistance to external influences, including high-temperature ones, allows assessment of the possibilities of using composite ceramics during their operation in extreme conditions, the impact of which can result in destabilization of properties [21,22].

Figure 2 demonstrates the assessment results of changes in the values of hardness and bending strength determined during cyclic tests for heat resistance. The obtained dependences of changes in strength characteristics reflect the rate of degradation under high-temperature effects associated with temperature differences that initiate oxidation processes of samples due to a sharp change in the temperature gradient. The overall trend of the observed alterations in strength characteristics indicates a reduction in strength characteristics upon reaching a certain number of test cycles, from which it follows that oxidation processes have a cumulative effect associated with an increase in stress effects when the temperature of the sample changes. It should be noted that the addition of  $\text{ZrO}_2$  to the composition of composite ceramics slows down the processes of destruction of strength characteristics, which can be due to the following factors. In the case of a sharp change in temperature, the resulting temperature gradient between the surface and internal layers

that are heated due to the effect of heating, the formation of thermal stresses occurs. At the same time, the presence of interphase boundaries can lead to uneven heating of samples due to low thermal conductivity coefficients, as well as differences in thermal expansion coefficients. The occurrence of thermal stresses in the near-surface layer with a sharp change in the exposure temperature (with rapid cooling) can lead to the formation of intergranular cracks, as well as extended intragranular cracks that arise along weakly bonded boundaries or defective inclusions, the presence of which is due to phase transformation processes.

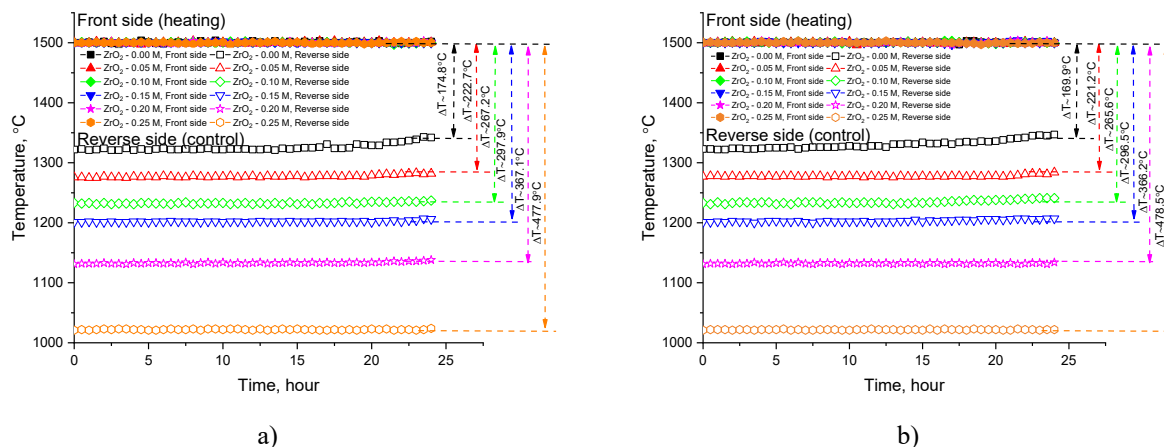


**Fig.2.** Results of cyclic tests of composite ceramics, reflecting changes in strength properties under thermal shock exposure: a) results of changes in hardness; b) results of changes in bending strength

In this case, the emerging local redistribution of deformation stresses, the presence of which is associated with the processes of phase formation during sintering of ceramic samples, together with thermal stresses formed during a sharp change in the temperature gradient can lead to destabilization of the phase composition of ceramics. This is associated both with a change in the density of dislocations and microcracks in the near-surface layer, and the emergence of a gradient in the density of defects between the near-surface layer and the deep layers, which were subjected to less heating due to the low thermal conductivity of the samples. The formation of such a defect density gradient in combination with the generation of microcracks in the near-surface layer leads to destabilization of the surface, which in turn leads to a decrease in hardness and bending strength. In this case, effects associated with the formation of cavities in the damaged layer can also be observed, the occurrence of which occurs due to the deformation effect on the crystalline structure, as well as partial expulsion or destruction of individual grains from the near-surface layer, which in turn leads to surface erosion during long-term heat resistance tests. In the case of the formation of the ZrCeO<sub>2</sub> substitution phase in the composition of composite ceramics, the increase in the contribution of which, according to the presented measurement results, leads to an increase in the strength characteristics of the samples in the initial state (the observed change in hardness and bending strength with an increase in the weight contribution of the ZrCeO<sub>2</sub> substitution phase in the composition varies from 10 to 50 % compared to undoped ceramics). Such changes in the strength characteristics indicate that the change in the phase composition due to a change in the concentration of the ZrO<sub>2</sub> dopant (and, consequently, the weight contribution of the ZrCeO<sub>2</sub> substitution phase) about the positive dynamics of hardening of ceramics to external mechanical effects. In this case, the hardening mechanism is associated with the effects of the interphase (intergrain) boundaries contribution growth, which leads to an increase in the concentration of grain boundaries that prevent the propagation of microcracks and chips under external influences. This is evidenced by the change in the values of the difference in hardness and bending strength obtained by comparing these values after a certain number of cycles with the initial value. Formation of inclusions in the form of ZrCeO<sub>2</sub> with an increase in the concentration of the stabilizing additive allows to significantly increase both the resistance to external influences and to increase the strength parameters, which is a fairly significant indicator in the field of expanding the potential for the use of composite ceramics. Moreover, the obtained results of the resistance enhancement are in good agreement with the results of a number of works [23-26], in which an increase in the strength parameters was achieved by formation of a size effect in the structure, as well as alteration of the phase composition of the ceramics. According to the data presented, in

the case of unmodified composite ceramics, the difference in hardness and bending strength values, reflecting the degree of softening (degradation of strength properties), is about 5.4 ~ 5.5 %. At the same time, the addition of  $\text{ZrO}_2$  to the ceramics, leading to the formation of the  $\text{ZrCeO}_2$  phase in a small amount (within 10 wt. %), leads to a rise in the initial values of hardness and bending strength, but the softening degree changes insignificantly compared to unmodified ceramics (the difference is less than 10 %). An elevation in the weight contribution of  $\text{ZrCeO}_2$  in the composition of composite ceramics above 10 % leads to a significant growth in the initial values of the strength parameters, as well as an increase in the degree of resistance to thermal shock effects, which consists in a decrease in the softening degree of less than 4.0 %, and in the case of a weight contribution of  $\text{ZrCeO}_2$  phase of about 20 – 23 %, the observed change is more than 2 times less than in the case of unmodified ceramics. At the same time, the values of hardness and bending strength for these ceramics are also quite high compared to unmodified ceramics. Thus, it can be concluded that the formation of the  $\text{ZrCeO}_2$  phase in the structure of ceramics results in emergence of additional interphase boundaries that restrain the effect of thermal expansion, and as a result, increases resistance to softening under thermal influence. It should also be noted that one of the factors restraining softening processes is the thermophysical properties, the change of which allows restraining high-temperature expansion processes. The decrease in the thermal conductivity of ceramics, caused by a change in the phase composition, expressed in the composite ceramics' thermal conductivity coefficient reduction from 26 W/(m×K) to 16 – 12 W/(m×K) leads to a slowdown in heat transfer processes, and therefore, an increase in thermal insulation due to the inhibition of phonon heat exchange mechanisms. As a result, the observed change in the softening degree can be explained by the fact that the restraint of heat exchange processes results in less pronounced destruction processes in the case of temperature differences.

Figure 3 shows the results of experimental work reflecting the change in temperature on the front and back side of ceramic samples and showing the thermal insulation properties of ceramics. These experiments were aimed at identifying the effect of variation in the phase composition of composite ceramics on the change in thermal insulation properties, as well as assessing the applicability of composite ceramics as thermal insulation materials capable of operating in extreme conditions.



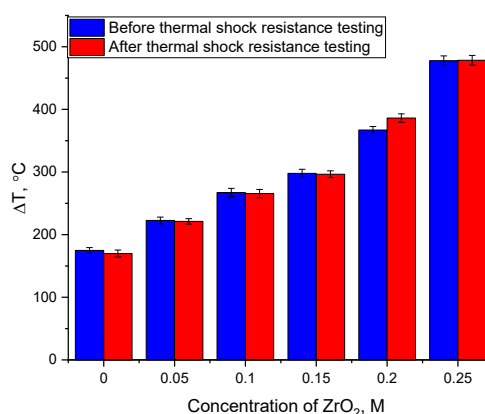
**Fig.3.** The assessment results of changes in temperature values on the front and back sides of the sample, reflecting the results of thermal insulation of ceramics depending on the holding time at high temperatures: a) ceramics in the initial state; b) ceramics after testing for thermal resistance to thermal shock exposure.

The measurements were carried out by heating one of the sides (designated as the front side) with recording the surface temperature using a thermocouple and measuring the temperature on the back side of the samples, the difference between which ( $\Delta T$ ) determines the value of thermal insulation. Moreover, in the case of heating, the temperature difference was measured after stabilization of the surface temperature in a given temperature mode and recording stable temperature values on the back side. The time for stabilization of the temperature difference during heating of the samples was about 30 – 60 minutes. The total time of testing for thermal insulation and maintaining thermal insulation properties was 24 hours, with control temperature measurements taken every half hour on the front and back sides of the samples. According to the data presented, it is evident that in the case of non-stabilized composite ceramics, the temperature difference  $\Delta T$  is about 175 °C, while after about 20 hours of cyclic testing, a decrease in the difference  $\Delta T$  between the



front and back sides is observed. This means that with prolonged thermal exposure, the thermal insulation properties of the ceramics are lost. According to the analysis of the obtained experimental data, the addition of  $\text{ZrO}_2$  to the composition leads to significant changes in the thermal insulation parameters, which are expressed both in the overall increase in the difference  $\Delta T$  and in the increase in the stability of maintaining the temperature difference during long-term thermal exposure. At the same time, the  $\Delta T$  value growth has a direct relationship with the change in the phase ratio in the composition of composite ceramics, due to the increase in the contribution of the monoclinic phase  $\text{ZrCeO}_2$ , formed as a result of the addition of  $\text{ZrO}_2$  to the composition. The  $\Delta T$  value growth in this case is due to a decrease in the thermal conductivity of ceramics, which in turn leads to a slowdown in the heat transfer mechanisms and, therefore, a reduction in heat transfer and heating of ceramic samples. The maximum  $\Delta T$  value for the studied ceramics in which the weight contribution of the  $\text{ZrCeO}_2$  phase is about 22 – 23 wt. % is equal to more than 475 °C, which is more than 2.5 times greater than the  $\Delta T$  value established for unmodified ceramics. Such changes make it possible to consider modified composite ceramics as high-strength heat-insulating materials capable of withstanding both mechanical loads and temperature changes, as well as long-term high-temperature exposure. Figure 3b also reveals the assessment results of the change in the heat-insulating properties of ceramics after testing for heat resistance under thermal shock effects, reflecting the effect of high-temperature degradation on the heat-insulating properties of ceramics. The general trend of changes in the values of temperature differences between the front and back sides of ceramics subjected to heat resistance tests does not differ significantly from the results of thermal insulation tests in the case of the original samples, from which it can be concluded that the impact of oxidation processes caused by sudden changes in temperature does not have a significant negative impact on the stability of ceramics and the preservation of the stability of thermal insulation properties.

Figure 4 demonstrates the results of a comparative analysis of the  $\Delta T$  values before and after testing the samples, reflecting the influence of high-temperature oxidation processes caused by thermal shock exposure on the preservation of thermal insulation characteristics. The obtained data showed that the thermal shock effect on the studied samples results in minor differences in the  $\Delta T$  values, from the analysis of which it can be concluded that the observed processes of thermal oxidation and softening do not have a significant effect on the loss of thermal insulation characteristics, and in some cases lead to a slight increase in the difference  $\Delta T$ , which may be due to a change in the thermophysical parameters of ceramics caused by oxidation.



**Fig.4.** Results of comparative analysis of  $\Delta T$  values before and after testing ceramic samples for thermal resistance to thermal shock exposure in the amount of 10 consecutive cycles

Determination of shielding characteristics reflecting the prospects of using the obtained multicomponent ceramics as protective shielding materials, allowing to reduce the negative impact of gamma radiation. As a source of gamma quanta, a  $\text{Cs}^{137}$  source, allowing the generation of gamma quanta with an energy of about 662 keV, which is characterized by the dominance of Compton scattering processes during the interaction of gamma quanta with materials, was used. The calculation was conducted by the assessment of changes in the intensity values of the registered gamma quanta without a protective shield and with a protective shield, on the basis of which the linear and mass absorption coefficients (LAC and MAC) and the thickness of the half-layer at which gamma quanta are absorbed (HVL) were estimated. The results of the experimentally determined values are given in Table 1.

According to the data presented, a change in the ratio of components in the composition of composite ceramics with the addition of  $\text{ZrO}_2$  to the composition, followed by the formation of the  $\text{ZrCeO}_2$  phase, leads to a growth in the shielding characteristics of LAC and MAC, an elevation in which indicates an increase in the shielding efficiency, and, as a consequence, a decrease in the negative effects of exposure to ionizing radiation. At the same time, the shielding efficiency growth due to the change in phase composition indicates a positive effect of modification not only on the strength and thermal insulation properties, but also on the shielding ability of ceramics. It is important to highlight that the order of magnitude of LAC, MAC and HVL itself indicates a fairly high shielding efficiency, since for most of the composite materials under consideration, the LAC value is usually about  $0.2 - 0.5 \text{ cm}^{-1}$ .

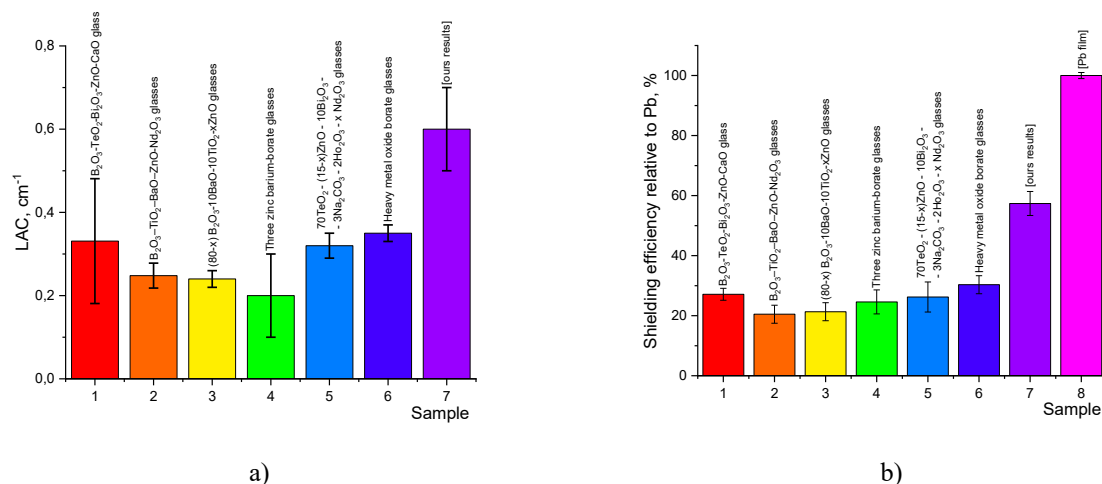
**Table 1.** Parameters of shielding characteristics depending on the ratio of components in the composition of ceramics

Shielding parameter	Concentration of $\text{ZrO}_2$ in ceramics, M					
	0.0	0.05	0.10	0.15	0.20	0.25
LAC, $\text{cm}^{-1}$	$0.65 \pm 0.04$	$0.66 \pm 0.07$	$0.67 \pm 0.05$	$0.68 \pm 0.03$	$0.71 \pm 0.03$	$0.72 \pm 0.05$
MAC, $\text{cm}^2/\text{g}$	$0.096 \pm 0.009$	$0.097 \pm 0.004$	$0.098 \pm 0.005$	$0.101 \pm 0.006$	$0.108 \pm 0.005$	$0.109 \pm 0.007$
HVL, cm	$1.041 \pm 0.014$	$1.037 \pm 0.012$	$1.032 \pm 0.016$	$1.029 \pm 0.017$	$0.965 \pm 0.015$	$0.0964 \pm 0.012$

A comparative analysis of the gamma radiation shielding efficiency was carried out using a number of literature data taken from works [27-31]. The choice of compositions of protective shielding materials is based on close ratios of components of the obtained samples, as well as their variations, which allowed the authors the estimation of the shielding efficiency. The results are shown in Figure 5; for comparison, the values of the LAC were selected in the case of shielding gamma quanta with energies of 662 keV, which are characterized by the dominance of Compton scattering in the mechanisms of interaction of gamma quanta with the shielding material. The comparison of the shielding efficiency was performed by comparison of the values of the LAC, which reflects the efficiency of gamma quanta absorption during shielding, while the choice of this value makes it possible to compare the efficiency without reference to density (the value of which, according to the results of these studies, varies within the range of  $3.5 - 4.5 \text{ g/cm}^2$ , which is significantly lower than the density of lead  $\sim 11.34 \text{ g/cm}^3$ ). The general appearance of the obtained comparison data shows that the shielding efficiency directly depends on the composition and quantity of components in the shielding glasses, in particular, the key role in increasing the efficiency is played by heavy oxide compounds ( $\text{Bi}_2\text{O}_3$ ,  $\text{WO}_3$ ,  $\text{Nd}_2\text{O}_3$  et.al.), alongside compounds with rare earth elements, the addition of which makes it possible to increase the absorption capacity of the shielding materials. Moreover, the observed values, as well as their dependences of the LAC on the composition of the shielding materials, are in good agreement with the results presented in the relatively recently published mini-review of Recep Kurtulus [32], which indicates that the use of heavy oxide components allows for an increase in shielding efficiency, while eliminating lead or its oxide in the composition of glass, the addition of which allows for an increase in the density of shielding materials above  $5 - 5.5 \text{ g/cm}^3$ . The systematization provided in the work of Recep Kurtulus allows us to identify the most promising composites in this area, as well as to determine the role and potential applicability of various composite glasses and ceramics as shielding protective materials. The results of the comparative analysis of the values of the LAC obtained for the studied  $x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO} - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$  ceramics showed a significant superiority over other types of composite glasses considered as shielding protective coatings, which in turn is due to the presence of both heavy oxides such as  $\text{Bi}_2\text{O}_3$ ,  $\text{WO}_3$  in the composition of the glasses, and the difference in the charge state of the selected elements, which makes it possible to obtain sufficiently high values of  $Z_{\text{eff}}$  determining the shielding efficiency for the selected energies (in this case, the addition of heavy components and  $\text{ZrO}_2$  to the composition leads to the value of  $Z_{\text{eff}} \sim 45 - 47$ ). In this case, the high value of  $Z_{\text{eff}}$  in the region of gamma quanta energies, characteristic of the dominance of interaction mechanisms of the photoelectric effect type, allows for the effective absorption of low-energy gamma quanta, which in turn allows for the use of sufficiently thin layers of protective materials for shielding, which in the case of protecting microelectronic devices plays a very important role in determination of the mass and overall dimensions. In the energy range from 0.1 to 1 MeV, for which the dominant effect in the interaction mechanisms is Compton scattering, the influence of  $Z_{\text{eff}}$  on the absorption capacity is most pronounced, since the absorption efficiency is directly



proportional to the value of  $Z_{\text{eff}}$ . Thus, a large value of  $Z_{\text{eff}}$  for the synthesized ceramics provides a high absorption capacity, significantly different from a number of borate and telluride glasses selected for comparison. In the case of high-energy gamma quanta, the greatest contribution to the shielding efficiency is made by the density of the material and its thickness, which, together with high  $Z_{\text{eff}}$  values, makes it possible to significantly increase the prospects for using composite ceramics.

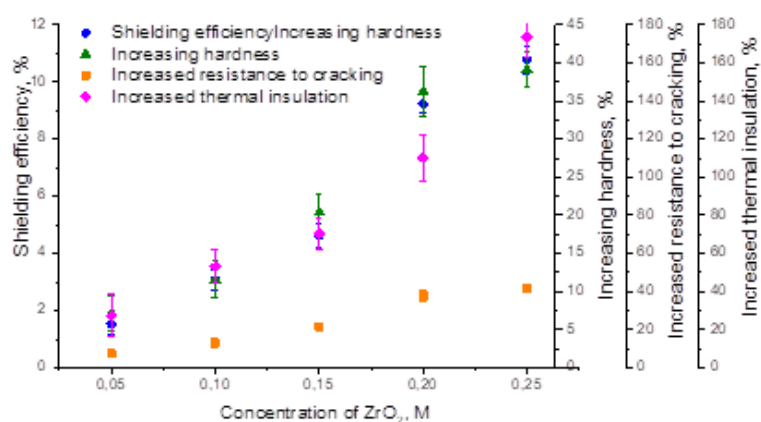


**Fig.5.** Comparative analysis of shielding properties

- a) results of a comparative analysis of the LAC taken from literature data with the results of the studies conducted;  
 b) results of the evaluation of the shielding efficiency in comparison with the LAC for lead

Figure 5b demonstrates the results of a comparative analysis of the shielding efficiency, determined on the basis of the measured LAC values in terms of the shielding efficiency of lead, which is a reference shielding material with the highest efficiency among the currently known protective shielding materials. In this case, the high efficiency of lead is primarily due to its high density, which leads to the maximum absorption of emitted gamma quanta. The results of the comparative analysis revealed that the efficiency of the obtained composite ceramics is about 60 – 65 % of the efficiency of lead, which is 1.5 – 2.0 times higher than the efficiency of the composite glasses and glass-like ceramics selected for comparison. This difference is primarily due to the difference in the density of the ceramics under consideration, as well as the  $Z_{\text{eff}}$  values, which play a very important role in determination of the absorption and shielding capacity.

Figure 6 shows the results of a comparative analysis reflecting changes in the strength (hardness and resistance to three-point bending), heat-insulating and shielding characteristics of the studied ceramics when modified with  $\text{ZrO}_2$ , the addition of which leads to a change in the phase composition of the ceramics due to the formation of the substitution phase  $\text{ZrCeO}_2$ .



**Fig.6.** Results of comparative analysis of changes in strength, heat-insulating and shielding characteristics of the studied composite ceramics with the addition of  $\text{ZrO}_2$ , reflecting the effectiveness of alteration of properties in comparison with non-stabilized ceramics

According to the data presented, the change in phase composition due to the addition of  $\text{ZrO}_2$  to the ceramics makes it possible to significantly increase the strength and mechanical properties, the increase of which is from 10 to 43 – 44 % depending on the concentration of the stabilizing additive in the composition. At the same time, the maximum achievement of efficiency is observed in thermal insulation characteristics, the growth of which is about 2.5 times compared to unmodified ceramics. In the case of assessment of the shielding characteristics, a change in the phase composition leads to an increase in the shielding efficiency, however, the increase in efficiency in the case of a change in the ratio of components in the composition is not as clearly expressed as in the case of strength and thermal parameters, and is no more than 10 – 12 % compared to unmodified ceramics. Such a difference between changes in the effectiveness of hardening and shielding is primarily due to the fact that hardening and thermal insulation growth is associated with the formation of impurity inclusions in the composition, which provide dispersion hardening due to size effects and the appearance of new inclusions, increasing the number of grain boundaries, restraining the spread of microcracks and oxidation processes.

Also, in the case of assessment of thermal insulation characteristics, the increase in efficiency is associated with a decrease in the thermal conductivity of ceramics due to impurity inclusions, which reduces the rate of heat exchange due to impurity inclusions, and therefore, inhibits heat transfer processes. In the case of shielding parameters, the main contribution is made by a change in the density of the material, which changes insignificantly with variations in the phase composition, which results in small changes in the shielding characteristics depending on the concentration of the component ratio. However, when compared with other samples of similar composite ceramics, as well as when recalculating the shielding characteristics in comparison with the parameters of lead, we can conclude that the absorption of gamma quanta and the containment of their negative impact are quite effective. Thus, it can be concluded that the proposed compositions of composite ceramics, as well as their modification due to the formation of inclusions in the form of the  $\text{ZrCeO}_2$  phase, can constitute significant competition in the field of shielding and heat-insulating materials, which have great potential for use in extreme conditions.

#### 4. Conclusion

In conclusion, we can summarize the main results of the experimental work carried out, reflecting the prospects for using composite multicomponent  $1-x(\text{TeO}_2 - \text{CeO}_2 - \text{WO}_3 - \text{ZnO} - \text{Bi}_2\text{O}_3) - x\text{ZrO}_2$  ceramics as shielding and heat-insulating materials. During examination of the X-ray phase analysis of the studied ceramic samples, it was found that the addition of a stabilizing additive in the form of  $\text{ZrO}_2$  leads to the formation of a substitution phase of the  $\text{ZrCeO}_2$  type with a monoclinic crystal lattice type, the appearance of which in the composition of ceramics leads to an elevation in strength characteristics, alongside an increase in resistance to thermal effects and efficiency during their use as heat-insulating and shielding materials. According to the conducted experimental works aimed at study of resistance to thermal effects, it was found that the addition of  $\text{ZrO}_2$  to the composition of composite ceramics leads to an increase not only in strength properties (hardness and bending strength), but also in resistance to high-temperature corrosion caused by cyclic tests.

In the case of tests to determine the effectiveness of thermal insulation characteristics, it was found that reduction in the thermal conductivity properties of ceramics during the formation of a new phase of  $\text{ZrCeO}_2$  in the composition of ceramics leads to an increase in thermal insulation characteristics, as well as an increase in the retention of resistance to long-term temperature effects.

During determination of the shielding characteristics of the studied ceramics, it was found that the obtained ceramic samples have a high shielding capacity, amounting to about 60 – 70 % of the shielding efficiency of lead, which is 1.5 – 2 times higher than similar composite ceramics.

#### Conflict of interest statement.

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

#### CRedit author statement

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