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The role of variation of the stabilizing magnesium oxide dopant on phase transformations in the composition of ZrO_2 – CeO_2 ceramics

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Abstract. The aim of this work is to determine the role of the addition of a stabilizing magnesium oxide dopant to the composition of composite ZrO_2 – CeO_2 ceramics in changing the kinetics of phase transformations that occur during high-temperature sintering of ceramics, depending on the variation in the concentration of the dopant. According to the assessment of the microstructural features of ZrO_2 – CeO_2 ceramics, it was found that the addition of MgO in low concentrations leads to the formation of finely dispersed grains of a globular shape that are tightly packed, while an increase in the concentration of MgO above 0.05 M leads to an enlargement of the grains and a change in their shape from globular to polyhedral with the formation of MgO inclusions, the presence of which is due to exceeding the solubility limit of magnesium oxide in the ZrO_2 – CeO_2 matrix. It has been established that variation in the MgO content allows for effective control of the morphology of ZrO_2 – CeO_2 ceramics from a loose porous structure to a dense fine-grained heterogeneous system with controlled grain size, high defectivity and developed intergranular boundaries. Using X-ray phase analysis methods, it was established that the introduction of MgO leads to a restructuring of the phase composition: from the initial two-phase state to a single-phase cubic structure at 0.01 M MgO and the subsequent formation of a mixture of two cubic and tetragonal phases at higher concentrations, with the dominance of the cubic phase in the composition.

Key words: stabilizing dopant, magnesium oxide, phase transformations, composite ceramics, zirconium dioxide

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Introduction

As is known, zirconium dioxide is one of the promising functional ceramic materials for solid oxide fuel cells, the interest in which is due to the combination of high thermal stability, chemical inertness and mixed type of conductivity, which makes it possible to use it in extreme high-temperature operating conditions [1-3]. Zirconium dioxide plays a key role in solid oxide fuel cells as a solid electrolyte, capable of providing transport of oxygen ions between the cathode and anode at operating temperatures of 700 – 1000 °C. At the cathode, molecular oxygen is reduced to form oxygen ions, which diffuse through the crystal lattice of the electrolyte. At the anode, oxygen ions interact with fuel - hydrogen, methane or synthesis gas, forming water or carbon dioxide with the release of electrons. This process results in the generation of electrical energy with a high efficiency factor. In this regard, much attention in solid oxide fuel cells is paid to oxygen-ion conductivity, which plays a key role in charge transfer mechanisms and electrochemical reactions [4,5]. The most commonly used ceramics are those based on zirconium dioxide stabilized with yttrium oxide, the addition of which leads to cationic substitution of the $Y^{3+} \rightarrow Zr^{4+}$ type, which is accompanied by the formation of oxygen vacancies, the appearance of which is necessary to maintain charge neutrality. The formation of oxygen vacancies plays a key role in ensuring high oxygen-ion conductivity of the material [6,7]. Vacancies serve as free positions for the migration of oxygen ions O^{2-} through the crystal lattice under the influence of a temperature gradient or electric potential. The higher the concentration of oxygen vacancies, the more intense the oxygen diffusion processes and the higher the electrical conductivity of the electrolyte. However, at high concentrations of oxygen vacancies in the structure of ceramics, resistance to external influences may decrease, including an increase in the embrittlement of ceramics and a tendency to fracture. This leads to the occurrence of internal stresses, the formation of microcracks and the destruction of ceramics. When exposed to high temperatures or cyclic thermal loads for a long time, such defects can initiate processes of local deformation and phase instability [8,9]. This is especially true for materials based on stabilized zirconium dioxide, in which an excess of oxygen vacancies contributes to the weakening of interatomic bonds and an increase in the sensitivity of the structure to thermal expansion. During the cooling process after sintering or operation, significant internal stress gradients arise due to the non-uniform distribution of phases and differences in thermal expansion coefficients [10,11]. In addition, a high concentration of oxygen vacancies can promote the agglomeration of defects and the formation of localized regions with increased defectiveness [12]. Such regions are characterized by reduced stability of the crystal lattice and can act as centers for the initiation of phase transformations, including the reverse transition of the tetragonal phase to the monoclinic phase [13,14]. This transition is accompanied by a volumetric expansion of the structure, which further increases internal stresses and accelerates the destruction of the material. Although the formation of oxygen vacancies is a key mechanism for enhancing the oxygen-ion conductivity of stabilized ceramics, their excessive concentration can have a negative impact on the mechanical and phase stability of the material [15,16]. Therefore, in the development of ceramic electrolytes, special attention is paid to the selection of the optimal stabilizing additive content, ensuring a balance between high ionic conductivity and sufficient mechanical strength of the ceramics [17,18].

The use of magnesium oxide as a stabilizing dopant for introduction into the composition of composite ZrO_2–CeO_2 ceramics is considered as one of the methods for controlling the kinetics of phase transformations during high-temperature sintering in order to purposefully change the ratio of phases in the composition, as well as the density of oxygen vacancies and the distribution of electron density. As is known, zirconium dioxide exhibits polymorphism and can exist in monoclinic, tetragonal and cubic modifications, the transition between which is accompanied by a change in the volume of the crystal lattice, which leads to changes in the properties of ceramics, as well as their resistance to external influences [19]. Cerium dioxide, in turn, due to its high oxygen mobility and the ability of cerium to change its oxidation state between the Ce^{3+}/Ce^{4+} states, promotes the formation of oxygen vacancies and increases the stability of high-temperature phases of zirconium dioxide due to partial cationic substitution. The introduction of CeO_2 into the composition of ZrO_2 leads to the formation of substitution solid solutions in which Ce^{4+} ions partially replace Zr^{4+} ions in the crystal lattice [19,20]. Moreover, the differences in the ionic radii of Ce^{4+} and Zr^{4+} lead to the fact that, during substitution, additional local distortions of the crystal structure, accompanied by an increase in the lattice parameters, are formed. An increase in the concentration of local structural distortions during cationic substitution promotes the stabilization of the tetragonal and cubic phases of zirconium dioxide at room temperature and prevents the formation of a monoclinic phase, which is characterized by volume expansion and can cause a decrease in resistance to external influences. Thus, the stabilization of zirconium dioxide and composite ceramics based on it is a fundamental mechanism for the formation of the functional properties of solid oxide electrolytes capable of operating under extreme conditions and withstanding high loads during operation. The study of the mechanisms of phase transformations with variations in the ratio of the main components and the stabilizing dopant

in the composition of composite ceramics makes it possible to better understand the properties of composite ceramics, as well as determine the optimal conditions for their modification.

Materials and methods

The synthesis of composite ceramics based on compounds of cerium dioxide, zirconium dioxide and magnesium oxide was carried out by mechanochemical grinding followed by thermal annealing of samples in a muffle furnace. A mixture of cerium and zirconium oxides in equal molar proportions was used as the base for the composite ceramics, and magnesium oxide was used as a stabilizer, which was added at the stage of weighing the samples before grinding. The concentration of magnesium oxide varied from 0.01 to 0.15 M. The choice of the concentration range was based on the possibility of changing the kinetics of phase transformations of composite ceramics during their thermal sintering. Grinding was carried out in a PULVERISETTE 6 planetary mill (Fritsch, Berlin, Germany). The grinding speed was 250 rpm, the grinding time was 30 minutes. Slow milling speeds were used to reduce the cold welding effect that occurs during vigorous mixing, which causes the powders being ground to stick together and become welded to the walls of the grinding bowl. This effect slows down the grinding process and deformation-induced crushing. After grinding, the resulting powders were removed from the grinding jar and subjected to thermal annealing in a Nabertherm muffle furnace (Nabertherm, Lilienthal, Germany). Annealing was carried out in air at a temperature of 1500 °C for 5 hours. The heating rate was 20 °C/min. After reaching the set temperature, the heating of the samples was maintained constantly; temperature fluctuations in the furnace were no more than ±10°. After the annealing time had elapsed, the furnace heating was switched off and the samples were cooled to room temperature without being exposed to air. The resulting powders, after thermal annealing, were placed in plastic containers and hermetically sealed to prevent oxidation processes or exposure to the atmosphere. Figure 1 shows a schematic representation of the main stages of the synthesis of composite ceramics.

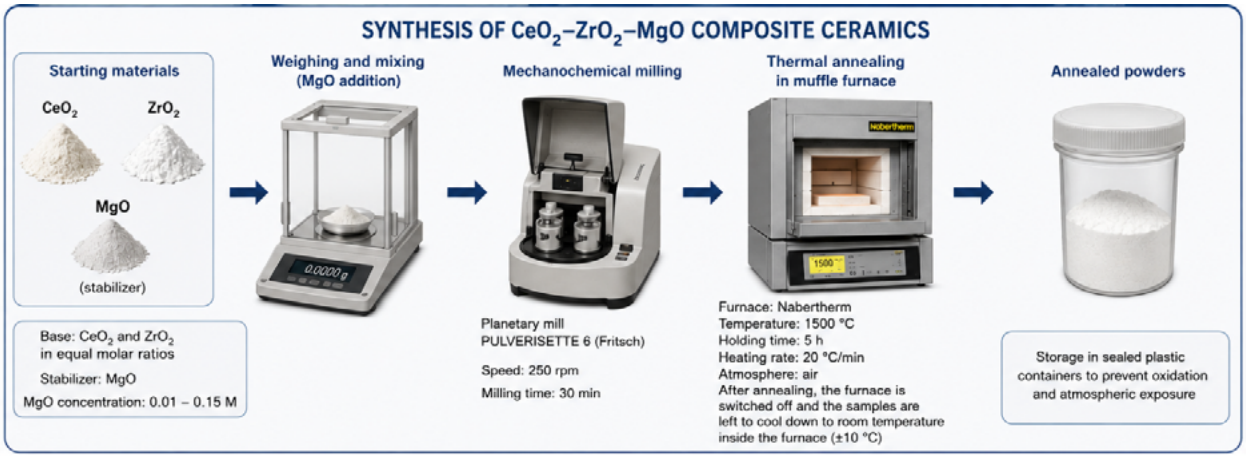

Figure 1. Schematic representation of the main stages of synthesis

Determination of the morphological features of composite ceramics depending on the concentration of the added stabilizing component in the form of magnesium oxide was carried out using the scanning electron microscopy method. Images were acquired on a Phenom™ ProX microscope (Thermo Fisher Scientific, Eindhoven, The Netherlands). All images were obtained using the same imaging modes, maintaining the image scale to enable determination of grain morphology and size.

The study of the kinetics of phase transformations depending on the ratio of components upon the addition of magnesium oxide was carried out using the X-ray structural analysis method. The diffraction patterns for subsequent processing were obtained on a D8 ADVANCE ECO powder diffractometer (Bruker, Karlsruhe, Germany). The survey was carried out in Bragg-Brentano geometry, the angular range was 2θ = 20 - 100°, the survey step was 0.03°, and the data acquisition time at a point was 1 sec. The assessment of structural parameters and changes in phase composition depending on the variation in the ratio of components in the composition was carried out using the DiffracEVA v.4.2 software code with standard processing procedures, including subtraction of kα2, smoothing of lines and their scaling for comparative analysis. The weight contributions of each phase in the samples were determined using a method for estimating the areas of diffraction reflections for each phase, followed by calculating the total area of the diffraction pattern. The weight contributions were estimated using corundum numbers determined from literature data for each identified phase. Structural

parameters were determined using a comparative analysis of peak positions obtained experimentally with the results of map values taken from the PDF-2 database. Parameter refinement was performed taking into account the cation sizes and their concentration dependences within the ceramics.

The optical properties of the studied ZrO2 – CeO2 ceramics were studied by a comprehensive analysis of the optical absorption spectra and calculation of the optical spectra of induced absorption depending on the concentration of magnesium oxide in the ceramics. Optical spectra were obtained on a SPECORD 200/210/250 PLUS spectrophotometer (Analytik Jena, Jena, Germany). Optical properties of ceramics were measured using an integral sphere, in which samples are placed in a special cuvette filled with barium sulfate, an optically transparent powder. Measurements are performed in a wavelength range from 190 to 10,000 nm with a 1 nm resolution.

Results and discussion

Figure 2 shows the results of the morphological features of the studied ZrO_2 – CeO_2 ceramics with the addition of magnesium oxide to the composition, a change in the concentration of which contributes, as can be seen from the data presented, to changes in the packing density of grains and crystallization processes during thermal sintering. In the case of ZrO_2 – CeO_2 ceramics without the addition of magnesium oxide (see data in Figure 2a), the microstructure is characterized by heterogeneous grains agglomerated into irregularly shaped dendrite-like processes with a highly developed surface and voids due to the loose structure formed by interconnected micron and submicron-sized particles. The presence of dendrite-like agglomerates consisting of spherical and elongated grains surrounded by a finely dispersed fraction indicates incomplete compaction of the ceramics during sintering of the particles, which in turn forms a developed porous surface in which the pores are unevenly distributed and formed by grain junctions and agglomerates. The presence of pores in the structure of ceramics is direct evidence of incomplete recrystallization processes, and the absence of faceted edges in agglomerates and the globular shape of grains, together with a high degree of aggregation of grains into dentritic inclusions, indicate that grain growth occurs by the mechanism of coalescence of small particles during sintering. This grain shape is typical for two-phase ceramics, which are represented by a solid solution of two phases, the presence of which leads to the formation of local structural stresses that prevent the formation of densely packed grains. When 0.01 – 0.03 M MgO is added to ZrO_2 – CeO_2 ceramics, the observed changes in the microstructure of the ceramics indicate an increase in the degree of compaction and structural uniformity due to the formation of finer grains with clearly defined intergranular contacts formed by rounded grains. Such changes in grain morphology are associated with the activation of sintering processes due to an increase in the mobility of grain boundaries, due to the dissolution of Mg^{2+} in the ZrO_2 – CeO_2 lattice with the formation of a large number of oxygen vacancies, the presence of which increases the diffusion mobility of oxygen and cations, which in turn accelerates mass transfer processes during high-temperature sintering. Acceleration of mass transfer processes leads to the formation of a more homogeneous structure, and intergranular contacts acquire a more pronounced continuous nature, indicating improved consolidation of ceramic grains during sintering. Moreover, the fine-grained fraction indicates that at low concentrations, MgO in the composition of ceramics does not cause intensive abnormal grain growth, and the absence of contrasting differences indicates that magnesium oxide is uniformly dissolved in the ZrO_2 – CeO_2 matrix, and the solubility limit is not exceeded. With an increase in the concentration of MgO in the composition of ZrO_2 – CeO_2 ceramics, the morphology of the grains is characterized by a transition to larger grains that have a polyhedral multifaceted shape with clearly defined boundaries, while having a densely packed structure. An assessment of the morphological features of the obtained ceramics indicates the presence of small grains having a clearly different phase from the main matrix, which, according to energy-dispersive analysis data, indicates the formation of inclusions in the form of MgO grains, the presence of which is due to exceeding the solubility limit of magnesium oxide in the ZrO_2 – CeO_2 matrix and the subsequent segregation of excess MgO in the intergranular space. With an increase in the concentration of magnesium oxide in the composition of ZrO_2 – CeO_2 ceramics, in addition to the process of dissolution of Mg^{2+} cations in the crystal lattice of ZrO_2 – CeO_2 and the formation of oxygen vacancies, the excess of magnesium oxide leads to the segregation of MgO grains into individual grains in the intergranular space in the form of a secondary phase, which affects the movement of grain boundaries and, as a consequence, the manifestation of heterogeneity in the sizes of polyhedral grains of the main ZrO_2 – CeO_2 matrix.

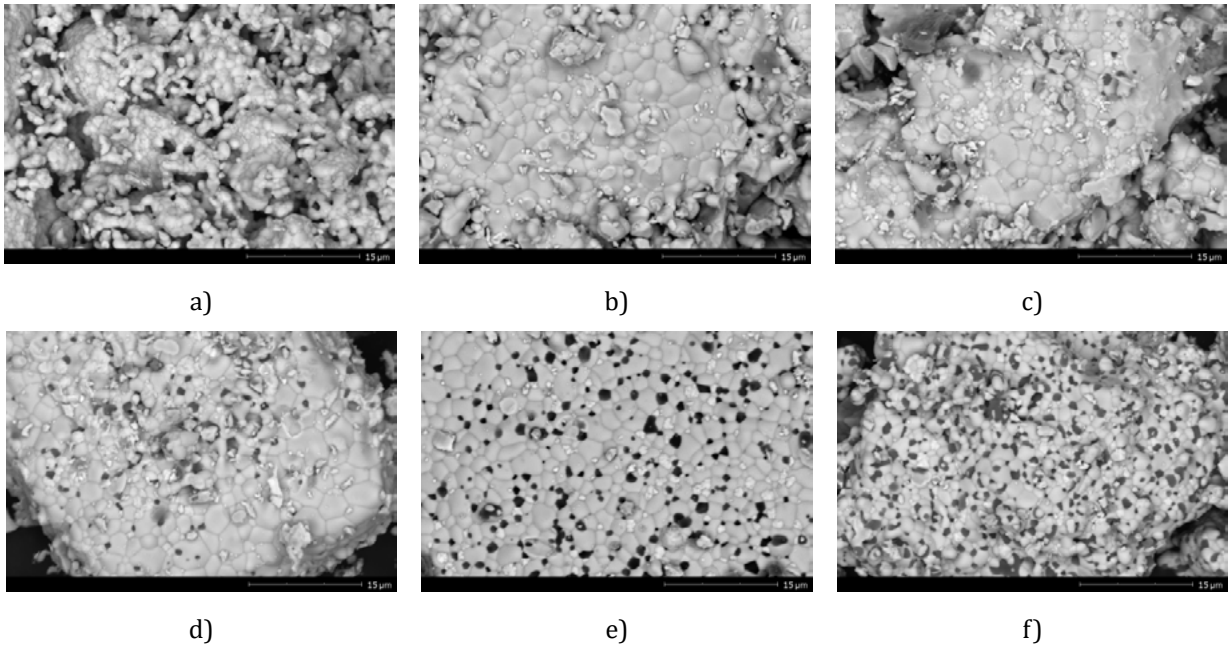

Figure 2. Results of morphological features of the studied ZrO_2 – CeO_2 ceramics depending on the variation of the MgO component in the composition: a) 0.0 M; b) 0.01 M; c) 0.03 M; d) 0.05 M; e) 0.10 M; f) 0.15 M

The growth of grains of the ZrO_2 – CeO_2 matrix, in turn, is caused by an increase in the concentration of oxygen vacancies, the formation of which is associated with cationic substitution and the need to maintain charge electroneutrality. The appearance of clearly defined grain boundaries, in turn, indicates an increase in the degree of recrystallization at high concentrations of MgO, which is accompanied by a restructuring of the morphology and compaction of ceramics. An increase in the MgO concentration to 0.10 M and above leads to an increase in the number of secondary inclusions and intergranular defects, as well as compaction of ceramics, which indicates an increase in the efficiency of sintering processes due to the intensive migration of intergranular boundaries, as well as the suppression of grain growth due to the pinning effect. Moreover, this pinning effect manifests itself more intensely at concentrations of 0.15 M MgO, which is expressed in the restraint of grain position, which leads to increased grain fragmentation, as well as a decrease in the average grain size with clearly defined smoothed boundaries.

Figure 3 shows a schematic representation illustrating the trend of changes in the shape of grains and intergranular boundaries in the composition of ZrO_2 – CeO_2 ceramics at the addition of MgO to the composition with different concentrations, the change of which leads to variations in the sintering mechanisms of grains, and also has a significant impact on the kinetics of recrystallization processes. Analyzing the presented data on the morphological features of ZrO_2 – CeO_2 ceramics, shown in Figures 2 and 3, it can be concluded that at low concentrations of MgO, the dominant role in the processes of recrystallization and grain formation is played by the processes of magnesium oxide dissolution in the ZrO_2 – CeO_2 matrix and cationic substitution, which is accompanied by the formation of oxygen vacancies, a change in the concentration of which can influence the processes of polymorphic transformations and the subsequent rearrangement of the phase composition of ceramics.

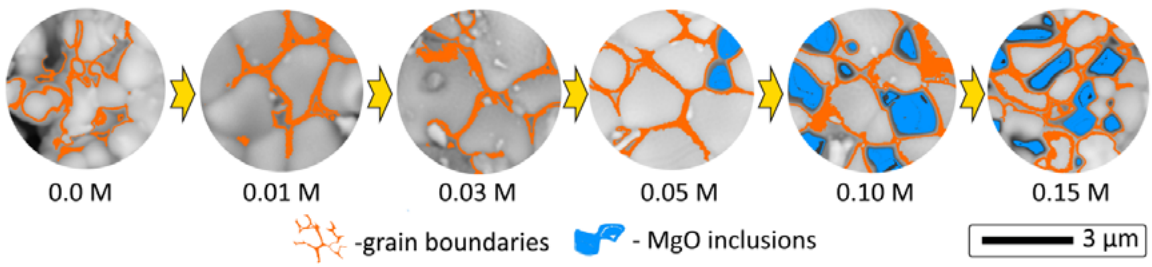

Figure 3. Results of the evaluation of the morphological features of the formation of grains in the composition of ZrO_2 – CeO_2 ceramics with the addition of MgO components with different ratios of components

When the solubility threshold of magnesium oxide is reached, which is achieved at concentrations above 0.05 M, segregation of secondary phases in the form of MgO inclusions formed in the intergranular space is observed in the composition of ceramics. An increase in the concentration of these inclusions leads to the initialization of grain growth inhibition processes due to the pinning effect, which leads to the formation of a heterogeneous structure consisting of polyhedral grains having an elongated or polyhedral shape, and the inclusions of the secondary phase themselves acquire a coalesced shape, which leads to increased fragmentation of intergranular boundaries.

Figure 4 demonstrates the results of X-ray phase analysis of the studied samples of ZrO_2 – CeO_2 ceramics depending on the variation of the concentration of MgO in the composition, the addition of which leads to an increase in the processes of phase transformations caused by the mechanisms of cation substitution, accompanied by the formation of oxygen vacancies.

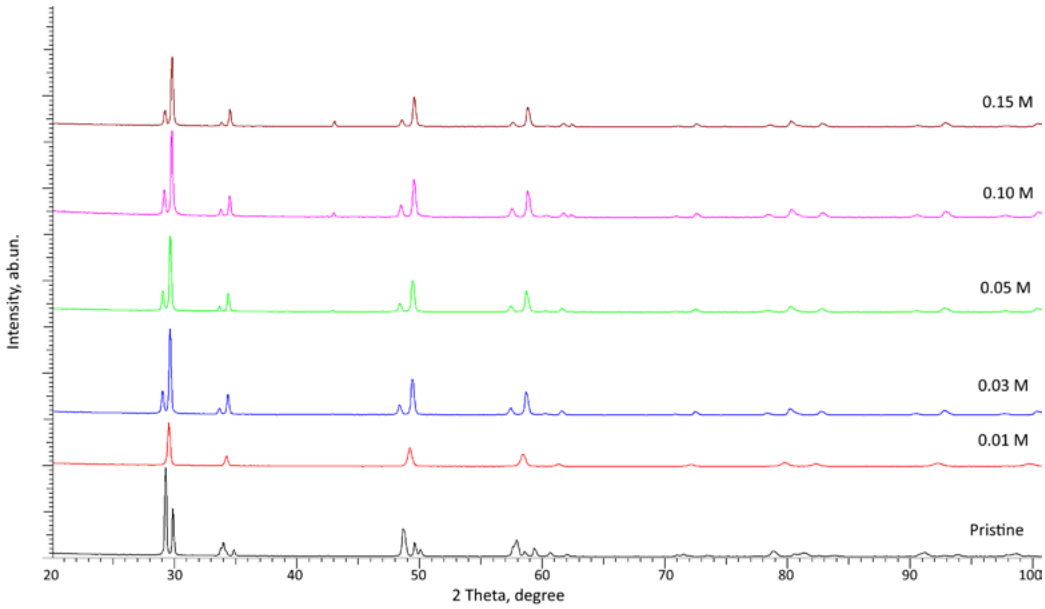

Figure 4. Results of X-ray diffraction of the studied ZrO_2 – CeO_2 ceramics with the addition of MgO in different concentrations

The initial composition of the ceramics is a mixture of a solid solution of two phases: tetragonal $Ce_{0.2}Zr_{0.8}O_2$ (PDF-01-080-6303) and cubic $Ce_{0.5}Zr_{0.5}O_2$ (PDF- 01-076-8752). When 0.01 M MgO is added to the composition, according to X-ray diffraction data, a phase transformation, expressed in the formation of the cubic phase $Ce_{0.33}Zr_{0.67}O_2$ (PDF-01-082-9829), which in turn indicates the homogenization of the structure due to the low content of the modifier and leads to the stabilization of the ZrO_2 – CeO_2 solid solution by forming a cubic phase, occurs. With an increase in the MgO concentration to 0.03 M and higher, according to X-ray diffraction data, destabilization of the cubic phase $Ce_{0.33}Zr_{0.67}O_2$ occurs, followed by displacement of the secondary tetragonal phase $Zr_{0.35}Ce_{0.65}O_2$ (PDF-01-074-8062) from it, the formation of which leads to the formation of a solid solution of two phases that differ in the cationic ratio Ce^{4+}/Zr^{4+}, which occurs as a result of cationic substitution, as well as a change in the thermodynamic stability of the phases. According to the analysis of the presented results of X-ray diffraction for samples of ZrO_2 – CeO_2 ceramics, the composition contains MgO from 0.05 M and higher, in the region of 2θ=43-44° and 2θ=62-63°, the appearance of diffraction reflections characteristic of the MgO phase, the identification of which is in good agreement with the results of the assessment of morphological features, in which it was established that when the solubility limit of MgO in the composition is reached, there is a segregation of MgO inclusions in the interboundary space, is observed. A comparison of the density of MgO inclusions and the change in the intensity of diffraction reflections characteristic of the MgO phase makes it possible to conclude that there is a good agreement between the data, indicating that an increase in the concentration of MgO above the solubility limit leads to an increase in the concentration of inclusions that initiate pinning, an effect that leads to inhibition of grain growth.

Figure 5 shows the assessment results of changes in the phase composition of the studied ZrO_2 – CeO_2 ceramics, reflecting the kinetics of phase transformations in the composition of ceramics with variations in the ratio of components in the composition. According to the assessment of the weight contributions in the initial ZrO_2 – CeO_2 ceramics, the ratio of the tetragonal $Ce_{0.2}Zr_{0.8}O_2$ and cubic $Ce_{0.5}Zr_{0.5}O_2$ phases is 46/54 wt. %, and the formation of a solid solution from two phases is due to the limited homogeneity of the distribution of Ce^{4+} and Zr^{4+} ions during the sintering process, which leads

to the emergence of a local separation of regions with different component ratios. The addition of 0.01 M MgO, according to X-ray diffraction data, leads to the formation of the cubic phase $Ce_{0.33}Zr_{0.67}O_2$ in the composition, with its subsequent dominance in the composition with an increase in the MgO concentration. The main mechanism of formation of this phase and polymorphic transformation of the t - $Ce_{0.2}Zr_{0.8}O_2$ →c - $Ce_{0.33}Zr_{0.67}O_2$ type is associated with cationic substitution $Mg^{2+} \rightarrow Zr^{4+}$ (Ce^{4+}) + V^{*}_{O}, which is accompanied by the formation of oxygen vacancies (V^{*}_{O}), the presence of which leads to an increase in the mobility of the anionic subsystem and acceleration of the diffusion of Ce^{4+} and Zr^{4+} cations, leading to a decrease in chemical heterogeneity, as well as a decrease in elastic stresses and stabilization of the cubic structure. With an increase in the concentration of MgO to 0.03 M and above, a displacement of the secondary tetragonal phase $Zr_{0.35}Ce_{0.65}O_2$ from the cubic phase is observed, the formation of which is due to reaching the solubility limit of Mg^{2+} in the composition of ceramics, which leads to the fact that charge compensation cannot occur only due to the formation of oxygen vacancies, the density of which increases, which leads to an increase in local distortions and energy destabilization of the homogeneous solid solution $Ce_{0.33}Zr_{0.67}O_2$, which leads to the release of the tetragonal phase $Zr_{0.35}Ce_{0.65}O_2$, the weight contribution of which is no more than 25 wt. %.

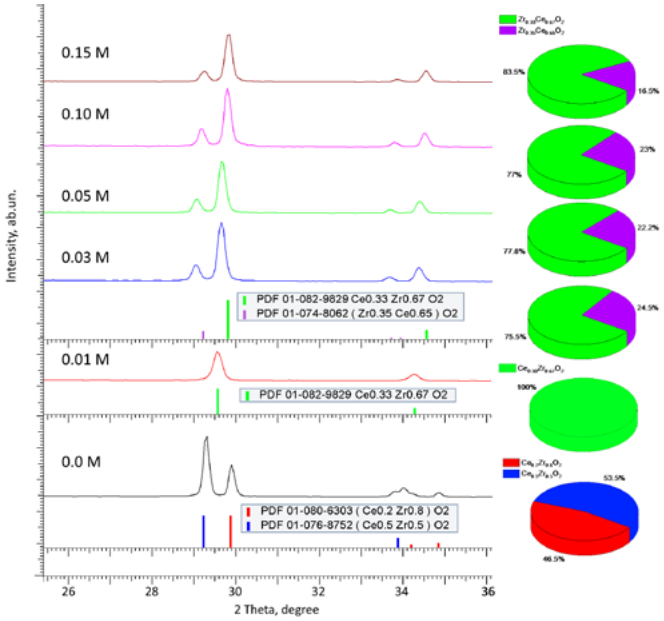

Figure 5. Results of the phase composition evaluation of composite ZrO_2 – CeO_2 ceramics, determined on the basis of the weight contributions of the phases in the composition with variations in the ratio of the MgO component

At the same time, the assessment of the shape of diffraction reflections for samples with a MgO content in the composition above 0.05 M indicates that during the formation of the tetragonal phase of $Zr_{0.35}Ce_{0.65}O_2$, a decrease in local structural distortions and stresses is observed, which indicates that such phase separation leads to a change in the differences in the binding energy of Ce – O, Zr – O, as well as stabilization of the structure. It should also be noted that in the case of high concentrations of MgO in the composition, at which the formation of MgO inclusions occurs in the form of individual well-structured grains, acting as barriers that inhibit grain growth, a decrease in the proportion of the tetragonal phase $Zr_{0.35}Ce_{0.65}O_2$ (at an MgO concentration of 0.15 M) is observed from 23 – 25 wt. % to 16 wt. %. Such a decrease indicates the inhibition of not only grain growth observed during the analysis of morphological features but also the initialization of phase transformations in the structure.

Table 1 presents the data on the structural parameters of the studied samples of ZrO_2 – CeO_2 ceramics, in the evaluation of which the ionic radii of Zr^{4+} ~ 0.84 Å, Ce^{4+} ~ 0.97 Å and Mg^{2+} ~ 0.72 Å were taken into account. According to the assessment of the crystal lattice parameters of the cubic phase **$Ce_{0.33}Zr_{0.67}O_2$** formed by the addition of MgO to the composition of ceramics, the observed decrease in the lattice parameters can be explained both by the effect of cationic substitution of the $Mg^{2+} \rightarrow Zr^{4+}$ or $Mg^{2+} \rightarrow Ce^{2+}$ type, which, due to differences in ionic radii, will lead to a decrease in the lattice size, and by a change in the Zr^{4+}/Ce^{4+} ratio in the lattice associated with an increase in the proportion of Zr^{4+}, which has a smaller ionic radius.

Table 1. Data on the structural parameters of ZrO_2 – CeO_2 ceramics with variations in the ratio of components in the composition

hase	Concentration of MgO, M					
	0.0	**0.01**	**0.03**	**0.05**	**0.10**	**0.15**
$Ce_{0.2}Zr_{0.8}O_2$ – Tetragonal P42/ nmc(137)	a=3.6375±0.0015 Å, c=5.2395±0.0019 Å	-	-	-	-	-
$Ce_{0.5}Zr_{0.5}O_2$ – Cubic Fm-3m(225)	a=5.2869±0.0021 Å	-	-	-	-	-
$Ce_{0.33}Zr_{0.67}O_2$ Cubic Fm-3m(225)	-	a=5.2276±0.0024 Å	a=5.2124±0.0016 Å	a=5.2145±0.0015 Å	a=5.1916±0.0024 Å	a=5.1854±0.0022 Å
$Zr_{0.35}Ce_{0.65}O_2$ Tetragonal P42/ nmc(137)	-	-	a=3.7594±0.0022 Å, c=5.3156±0.0019 Å	a=3.7616±0.0017 Å, c=5.3157±0.0022 Å	a=3.7371±0.0013 Å, c=5.3211±0.0019 Å	a=3.7319±0.0019 Å, c=5.3115±0.0015 Å
Degree of structural ordering, %	90.4	89.7	89.5	91.5	86.5	88.3

However, an assessment of the trend of changes in the crystal lattice parameters for the $Ce_{0.33}Zr_{0.67}O_2$ phase, especially at high concentrations of MgO in the composition, makes it possible to conclude that the dominant process is cationic substitution in the composition, which leads to an increase in the concentration of oxygen vacancies, since the substitution process is accompanied by the formation of oxygen vacancies in the structure to maintain charge electroneutrality. At the same time, the formation of oxygen vacancies leads to the facilitation of the processes of mutual diffusion of $Zr^{4+} \leftrightarrow Ce^{4+}$ ions, which makes it possible to reduce chemical heterogeneity and accelerate the processes of phase transformations, the result of which is an increase in the proportion of the cubic phase in the composition of ceramics.

Figure 6 shows the assessment results of changes in the optical properties of ceramics, expressed in absorption spectra, as well as calculated spectra of induced absorption, reflecting the kinetics of changes in structural defects and oxygen vacancies depending on the concentration of MgO in the composition, as well as the processes of phase transformations arising as a result of cationic substitution, accompanied by the formation of oxygen vacancies and structural distortions. According to the presented optical absorption spectra, in the case of the initial ZrO_2 – CeO_2 ceramics, a smooth increase in absorption is observed in the region of 3.5 – 5.3 eV and a subsequent sharp decline near 5.5 – 5.6 eV, and the optical spectrum itself is characteristic of two-phase ceramics, in which the optical response is formed by the contribution of both structural modifications, in this case a mixture of cubic and tetragonal phases. When MgO is added to the composition of ZrO_2 – CeO_2 ceramics, the change in the optical absorption spectra indicates a structural rearrangement associated with a variation in the phase composition of the ceramics, as well as changes in the local cationic environment of Zr^{4+}/Ce^{4+}. The increase in absorption intensity in the range of 4.4 – 5.6 eV upon the addition of 0.01 M MgO to the composition of ZrO_2 – CeO_2 ceramics is due to the phase transformation of the solid solution of two phases into a cubic phase, the stabilization of which is accompanied by a sharp increase in the concentration of oxygen vacancies, as well as changes in the valence-coordination state of cerium ions, which enhances optical absorption. The addition of 0.03 M MgO to the composition of ZrO_2 – CeO_2 ceramics leads to the appearance of a pronounced absorption band in the region of 3.9 – 4.1 eV, the presence of which is associated with the formation of defect states, as well as local transitions caused by the redistribution of Ce^{4+} and Zr^{4+} cations that arise during the formation of the tetragonal $Zr_{0.35}Ce_{0.65}O_2$ phase. Moreover, a decrease in the intensity of the absorption spectrum in the region of 4.5 – 5.4 eV indicates a change in the electronic structure caused by a change in the concentration of oxygen vacancies and a redistribution of the charge density. An increase in the MgO concentration above 0.05 M in the composition of ZrO_2 – CeO_2 ceramics leads to a decrease in the absorption intensity of the spectra, which is due to the stabilization of two phases with different cationic distribution Zr^{4+}/Ce^{4+}, which leads to a more heterogeneous distribution of cations, but does not cause strong absorption due to the stabilization of the structure and compensation of charge electroneutrality.

a) b)

Figure 6. Results of optical properties of ceramics:
a) Absorption spectra of the studied composite ceramics depending on the ratio of components in the composition; b) Induced absorption spectra calculated for modified ceramics relative to the initial ZrO_2 – CeO_2 ceramics

Figure 6b shows the results of a comparative analysis of the effect of the addition of MgO to the composition of ZrO_2 – CeO_2 ceramics on the change in the intensity of induced absorption spectra depending on the concentration of magnesium oxide in the composition, according to which a change in the MgO content in the composition leads to a change not only in the number and concentration of defects, but also in their type. The optical absorption spectrum of the original sample without the addition of magnesium oxide was used as a comparison spectrum. A general analysis of the obtained induced absorption spectra made it possible to identify three characteristic regions characterizing different types of defects: the region of 3.1 – 3.5 eV is typical for defect levels associated with oxygen vacancies and Ce^{3+} centers; the region of 3.6 – 4.3 eV is typical for complex defects of the Ce^{3+} - V^*_O, Mg - V^*_O type and Ce – O – Zr disorder; the region of 5.4 – 5.7 eV is typical for interband transitions and changes in the band gap. When 0.01 M MgO is added to the composition of ZrO_2 – CeO_2 ceramics, the observed changes in the region of 3.3 – 4.2 eV indicate a low concentration of defect centers, as well as the formation of oxygen vacancies arising from the cationic substitution $Mg^{2+} \rightarrow Zr^{4+}$ or $Mg^{2+} \rightarrow Ce^{4+}$. An increase in the MgO concentration to 0.03 M leads to an increase in oscillations in the range of 3.3 – 4.1 eV, indicating an increase in the concentration of defects associated with oxygen vacancies, as well as partial reduction of $Ce^{4+} \rightarrow Ce^{3+}$. The addition of 0.05 M MgO leads to the appearance of spectral absorption lines in the region of 3.5 – 4.2 eV, associated with the formation of MgO inclusions, which leads to a decrease in the concentration of optically active vacancies in the structure. At a MgO concentration of 0.10 M, defect bands are preserved in the optical spectra, but their intensity does not increase in direct proportion to the content of MgO inclusions in the ceramics, which indicates that excess MgO forms individual inclusions rather than new vacancies in the structure of the main phase. At a MgO concentration of 0.15 M, the main changes in the spectrum are associated with changes in the high-energy region (5.5 – 5.7 eV), which are associated not only with defects, but also with an increased role of MgO inclusions in the composition, which inhibit grain growth and also affect the change in the ratio of tetragonal and cubic phases.

Conclusion

The paper presents the results of the effect of the addition of MgO to the composition of ZrO_2 – CeO_2 ceramics on the change in the morphological features and kinetics of phase transformations of ceramics with a change in the ratio of components during thermal sintering. According to the data from the assessment of morphological features, it was established that the addition of a low concentration of MgO to the composition of ceramics leads to a compaction of the structure, a decrease in porosity, and the formation of a homogeneous fine-grained fraction, the formation of which is due to the activation of diffusion processes due to the formation of oxygen vacancies and an increase in the consolidation of ceramics. At low concentrations, MgO acts as a structural modifier, leading to the stabilization of the ZrO_2 – CeO_2 solid solution and increasing the mobility of grain boundaries. An increase in the MgO concentration above 0.05 M leads to a transition to a denser polyhedral structure of grains with clearly defined intergranular boundaries, as well as the formation of secondary MgO inclusions, the appearance of which indicates that the solubility limit has been exceeded and two processes are competing: acceleration of grain growth due to high defectiveness and partial fixation of grain boundaries with secondary inclusions in the form of MgO grains. At high MgO concentrations, a stabilized fine-grained structure with a high density of grain boundaries and a significant number of defective areas is formed. Moreover, it was established that

the evolution of the morphological features of ZrO_2 – CeO_2 ceramics is determined by the competition of two mechanisms: MgO – induced activation of diffusion processes associated with the formation of oxygen vacancies and the fixation of intergranular boundaries by MgO – inclusions, which inhibit grain growth.

Analysis of X-ray diffraction data revealed that the addition of MgO leads to a restructuring of the phase composition, accompanied by a transition from the initial two-phase state to a single-phase cubic structure at 0.01 M MgO and the subsequent formation of a mixture of two phases at higher concentrations. It is shown that the introduction of MgO promotes the formation of oxygen vacancies and defect complexes, which determine the change in the optical and induced absorption spectra. According to the assessment of the optical properties of ceramics, it was found that at low concentrations of MgO equal to 0.01–0.03 M, Mg^{2+} actively enters the ZrO_2 – CeO_2 lattice, which leads to the formation of oxygen vacancies initiating phase transformation processes. At concentrations of 0.05 M and higher, the release of MgO inclusions begins, which leads to a disproportionate increase in defects in the matrix and a change in the optical spectra. It was established that the evolution of the microstructure is determined by the competition between the activation of diffusion processes due to oxygen vacancies and the pinning effect caused by the release of MgO inclusions. The obtained results demonstrate the possibility of effective control of the phase state, defect structure and optical characteristics of ZrO_2 – CeO_2 ceramics by variation of the concentration of MgO.

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The contribution of the authors

A.Z. Zikirina, I. Kenzhina, Z. Zaurbekova, K. Ilyasheva, G. Seitmaganbet and **A. Kozlovskiy** contributed to the synthesis, characterization, and analysis of composite ZrO2 – CeO2 – MgO ceramics.

A.Z. Zikirina and **I. Kenzhina** performed the synthesis of composite ceramics by mechanochemical grinding and thermal annealing, including preparation of samples, optimization of MgO concentration, and experimental procedures.

Z. Zaurbekova and **G. Seitmaganbet** conducted scanning electron microscopy investigations and analyzed the morphological features and grain structure of the obtained ceramics.

I. Kenzhina and **A. Kozlovskiy** performed X-ray diffraction measurements, phase composition analysis, and interpretation of structural parameters.

K. Ilyasheva contributed to the study of optical properties, including the acquisition and analysis of optical absorption spectra. All authors participated in data interpretation, discussion of results, and preparation and revision of the manuscript. All authors read and approved the final version of the manuscript.

Statement on the use of generative AI and AI-enabled technologies in the manuscript preparation process

In preparing this paper, the author(s) used ChatGPT to construct general experimental flow charts, reflecting the sequence of steps in ceramic synthesis and illustrating the main technological processes. After using this ChatGPT, the author(s) reviewed and edited the content as necessary and bear full responsibility for the content of the published article.

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ZrO_2 – CeO_2 керамикасының құрамындағы фазалық түрленулерге тұрақтандырушы магний оксиді қоспасының мөлшерінің әсері

Аңдатпа. Бұл жұмыстың мақсаты композиттік ZrO_2 – CeO_2 керамикасының құрамына енгізілетін тұрақтандырушы магний оксиді қоспасының жоғары температуралық күйдіру кезінде жүретін фазалық түрленулер кинетикасының өзгеруіндегі рөлін қоспа концентрациясына байланысты анықтау болып табылады. ZrO_2 – CeO_2 керамикасының микроструктуралық ерекшеліктерін зерттеу нәтижесінде MgO қоспасының төмен концентрацияларда енгізілуі тығыз орналасқан ұсақ дисперсті глобулалық пішінді түйіршіктердің түзілуіне әкелетіні анықталды. Ал MgO концентрациясы 0,05 М-ден жоғары болған жағдайда түйіршіктердің іріленуі және олардың пішінінің глобулалықтан полигональды түрге өзгеруі байқалады, сондай-ақ MgO қосындылары түзіледі. Бұл құбылыс магний оксидінің ZrO_2 – CeO_2 матрицасындағы ерігіштік шегінен асып кетуімен түсіндіріледі. MgO мөлшерін өзгерту ZrO_2 – CeO_2 керамикасының морфологиясын тиімді басқаруға мүмкіндік беретіні анықталды: борпылдақ кеуекті құрылымнан бастап, бақыланатын түйіршік өлшемі, жоғары ақаулылық және дамыған түйіршік шекаралары бар тығыз ұсақ түйіршікті гетерогенді жүйеге дейін өзгертуге болады. Рентгендік фазалық талдау әдістері арқылы MgO енгізу фазалық құрамның қайта құрылуына әкелетіні анықталды: бастапқы екі фазалы күйден 0,01 М MgO кезінде бір фазалы кубтық құрылым түзіледі, ал жоғары концентрацияларда кубтық және тетрагональды екі фазаның қоспасы қалыптасады, мұнда құрамда кубтық фаза басым болады.

Түйін сөздер: тұрақтандырушы қоспа, магний оксиді, фазалық түрленулер, композиттік керамика, цирконий диоксиді

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Роль изменения содержания стабилизирующей добавки оксида магния в фазовых превращениях состава керамики ZrO_2 – CeO_2

Аннотация. Целью данной работы является определение роли введения стабилизирующей добавки оксида магния в состав композитной керамики ZrO_2 – CeO_2 в изменении кинетики фазовых превращений, происходящих при высокотемпературном спекании керамики, в зависимости от концентрации добавки. В результате оценки микроструктурных особенностей керамики ZrO_2 – CeO_2 установлено, что добавление MgO в низких концентрациях приводит к формированию мелкодисперсных зерен глобулярной формы с плотной упаковкой. При увеличении концентрации MgO выше 0,05 М наблюдается укрупнение зерен и изменение их формы от глобулярной к полигональной с образованием включений MgO, наличие которых обусловлено превышением предела растворимости оксида магния в матрице ZrO_2 – CeO_2. Установлено, что изменение содержания MgO позволяет эффективно контролировать морфологию керамики ZrO_2 – CeO_2 от рыхлой пористой структуры до плотной мелкозернистой гетерогенной системы с регулируемым размером зерен, высокой дефектностью и развитой межзеренной структурой. Методами рентгенофазового анализа установлено, что введение MgO приводит к перестройке фазового состава: от исходного двухфазного состояния к однофазной кубической структуре при содержании 0,01 М MgO и последующему формированию смеси двух фаз – кубической и тетрагональной – при более высоких концентрациях, с преобладанием кубической фазы в составе.

Ключевые слова: стабилизирующая добавка, оксид магния, фазовые превращения, композитная керамика, диоксид циркония

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