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Study of the influence of structural modification of ZrO₂ ceramics on resistance to hydrogen absorption and gas swelling processes

Abstract. The main objective of this work is to study the influence of structural changes in ZrO₂ ceramics on the resistance to hydrogenation processes arising from testing the performance of ceramics as materials for solid oxide fuel cells. During the conducted studies, it was established that the use of stabilizing additives MgO and CaCO₃ to initiate processes of polymorphic transformations in ZrO₂ ceramics makes it possible to increase the resistance of ceramics to hydrogen swelling due to the stabilization of the crystal structure, the change of which is associated with polymorphic transformations. Suppression of volumetric swelling processes due to polymorphic transformations caused by the addition of stabilizing additives made it possible to elevate resistance to hydrogen absorption and destabilization of the near-surface layer by preventing hydrogen agglomeration processes in the near-surface layer. It has been determined that the modification of ZrO₂ ceramics by initiating processes of polymorphic transformations of the type $m\text{-ZrO}_2 \rightarrow t\text{-ZrO}_2$ and $m\text{-ZrO}_2 \rightarrow c\text{-ZrO}_2$ leads not only to an increase in strength indicators, but also to an increase in resistance to embrittlement processes during hydrogenation and high-temperature degradation.

Key words: ZrO₂ ceramics, gas swelling processes, modification, mechanochemical solid-phase synthesis, polymorphic transformations.

Introduction

Today, the share of renewable energy generated using alternative energy methods in the total energy generation in Kazakhstan is approximately 6.67%. At the same time, Kazakhstan's participation in the COP28, IAEA Atom4NetZero international programs, aimed at reduction of the amount of harmful emissions into the atmosphere and elevation of the share of alternative energy sources in the energy sector, opens up great opportunities for the development of scientific research in this area, the main goal of which is the transformation of fundamental theoretical knowledge into practical applications capable of increasing the energy efficiency of energy production, as well as increase the profitability of the production of alternative energy sources and their operation [1-3].

One of the promising directions in this field is the investigation of solid oxide fuel cells (SOFC) for energy generation. The use of SOFCs offers significant potential for reducing harmful emissions, thus

addressing environmental challenges, and provides versatility in fuel choices, including hydrogen, methane, ammonia, biogas, and others [4-7]. In the last few years, SOFC elements have been considered not only from the point of view of backup power sources used in emergency situations or at enterprises where it is necessary to maintain a constant supply of electricity, but also for the creation of local power plants, the use of which allows providing electricity to remote areas, as well as, if necessary, to provide an uninterrupted supply of energy to local areas or residential buildings [8,9].

The expansion of the range of ceramic materials used as the basis for solid oxide fuel cells is primarily driven by the need to address several issues, including the reduction of operating temperatures of SOFC components, enhancing their resistance to high-temperature degradation processes occurring during operation, and mitigating the accumulation of structural distortions. [10-12]. Degradation processes in SOFCs are commonly related to the development of gas-filled inclusions in the near-surface layers.

The accumulation of hydrogen in the near-surface layers causes destabilization of fuel cell performance, resulting in a decline in electrochemical properties, which ultimately leads to a loss of energy efficiency. Also, enhancement of the degradation resistance of SOFC elements by stabilization of the crystalline structure of ceramic materials will expand their application and reduce the cost of energy produced [13-15].

The primary objective of this study is to evaluate the impact of changes in the type and concentration of the stabilizing additive on the polymorphic transformation processes in ZrO₂ ceramics, and to inhibit the hydrogenation processes resulting in destabilization of the near-surface layer and its softening. The use of ZrO₂ ceramics as a basis for the creation of anode materials in solid oxide fuel cells is attributed to its mixed conductivity properties, as well as the possibility of operation under high temperature conditions, due to the small value of volumetric thermal expansion of the crystal lattice at high temperatures, which allows for the possibility to increase the stability of the crystal structure under prolonged exposure to high temperatures [16-19]. The choice of modified ceramics based on zirconium dioxide as the studied samples is due to the possibility of using them as materials for solid oxide fuel cells with a mixed type of conductivity, as well as high resistance to high-temperature degradation and corrosion resistance to most acids and alkalis. At the same time, simulation of hydrogenation processes in ceramics makes it possible to evaluate the kinetics of changes in structural and strength properties depending on the concentration of accumulated hydrogen, which is controlled by the hydrogenation time, which can subsequently make it possible to establish the potential applicability of these types of ceramics as materials for solid oxide fuel cells, alongside to determine further ways of targeted modification of ZrO₂ ceramics in order to enhance the efficiency of their use.

Materials and methods

The ceramics were obtained using the mechanochemical solid-phase synthesis method. A PULVERISETTE 6 planetary mill (Fritsch, Berlin, Germany) was used to grind the samples. Grinding was carried out in an 80 ml tungsten carbide beaker, and 10 mm diameter tungsten carbide balls were

used as grinding balls. To prepare the samples, ZrO₂, MgO and CaCO₃ chemical reagents were obtained as powders from Sigma Aldrich (Sigma Aldrich, USA), the chemical purity of which was approximately 99.95 %. Thermal sintering of the samples after grinding was carried out in a muffle furnace at a temperature of 1200 °C for 5 hours, after which the samples were cooled together with the furnace for 20–24 hours until reaching room temperature.

The samples, the data on which are presented in Table 1, were chosen as objects of study in order to establish the effect of doping with stabilizing components MgO and CaCO₃ on the resistance of ZrO₂ ceramics to gas swelling effects resulting from hydrogenation of the near-surface layers of ceramics, along with the related processes of embrittlement and softening of these layers due to deformation. The X-ray diffraction results are shown in Figure 1 The phase composition analysis. The structural characteristics were determined using a program code based on the Rietveld method. The structural parameters were refined by comparing the experimental data with the reference values from the PDF-2 database. Deformation factors were taken into account for modified ceramics, for which the change in parameters can be caused by both structural changes associated with substitution effects, as well as with thermally stimulated processes of phase transformations.

According to the data presented, thermal annealing of ZrO₂ ceramics at a temperature of 1200 °C, in the absence of dopants in the composition, does not lead to the initialization of the polymorphic transformation processes that occur in the composition of ceramics when the concentration of the stabilizing components MgO and CaCO₃ changes. It should be noted that in the case of the addition of MgO dopant with a concentration of 0.05 M to the composition of ZrO₂ ceramics, according to X-ray phase analysis data, a transformation monoclinic – ZrO₂ → tetragonal – ZrO₂ is observed, and in the case of MgO dopant concentration growth to 0.10 M, the structure of the ceramics is represented by a cubic phase c – ZrO₂, the formation of which is due to the processes of transformations monoclinic – ZrO₂ → (tetragonal-ZrO₂) → cubic-ZrO₂. In the case of using the CaCO₃ dopant, its addition at a concentration of 0.05 M leads to the initialization of polymorphic transformations of the m – ZrO₂ → t-ZrO₂ type, with the preservation of the monoclinic phase ZrO₂ in the inclusions, the

content of which is no more than 10 wt. %. Elevation of the CaCO_3 dopant concentration to 0.10 M leads to the formation of ceramics of the cubic phase ZrO_2 , while the content of inclusions in the form of the tetragonal phase is about 2 – 3 wt.%. The structural ordering degree of ceramics is 93 – 95 %, which

indicates a fairly good ordering degree of the crystal lattice. The analysis of the crystal lattice parameters presented in Table 1 also confirms this statement, and a small deviation from the reference values indicates the formation of a structure with a low content of structural distortions and vacancy defects.

Table 1 – Structural parameter data.

№	Sample name, method of its production	Structure type	Parameters and volume of the crystal lattice, Å
Z-1	ZrO_2 ceramics, without addition of stabilizing components	Monoclinic	$a=5.2782\pm 0.0013$ Å, $b=5.1869\pm 0.0015$ Å, $c=5.1320\pm 0.0012$ Å, $\beta=99.510^\circ$, $V=138.58$ Å ³
Z-2	ZrO_2 ceramics stabilized with 0.05 M MgO	Tetragonal	$a=3.5379\pm 0.0011$ Å, $c=5.1030\pm 0.0017$ Å, $V=63.88$ Å ³
Z-3	ZrO_2 ceramics stabilized with 0.10 M MgO	Cubic with inclusions of MgO phase	$a=5.0099\pm 0.0015$ Å, $V=125.75$ Å ³
Z-4	ZrO_2 ceramics stabilized with 0.05 M CaCO_3	Tetragonal with inclusions of monoclinic phase	ZrO_2 – Tetragonal: $a=3.5846\pm 0.0016$ Å, $c=5.1718\pm 0.0017$ Å, $V=66.45$ Å ³ ZrO_2 – Monoclinic: $a=5.3066\pm 0.0012$ Å, $b=5.2047\pm 0.0013$ Å, $c=5.1368\pm 0.0016$ Å, $\beta=99.061^\circ$, $V=140.10$ Å ³
Z-5	ZrO_2 ceramics stabilized with 0.10 M CaCO_3	Cubic with a small proportion of inclusions of the tetragonal phase	$a=5.0829\pm 0.0014$ Å, $V=131.33$ Å ³

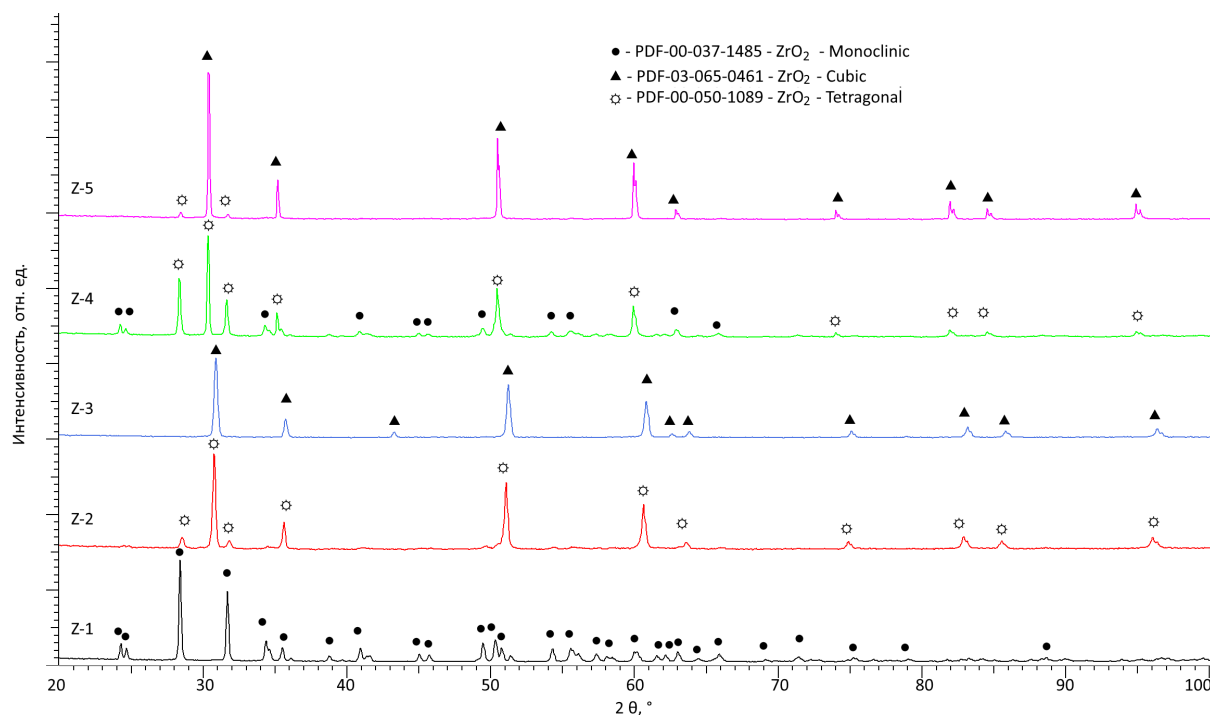


Figure 1 – Results of X-ray diffraction of the studied samples of ZrO_2 ceramics with different types of stabilizing additives used for modification.

Experiments to determine the resistance of ceramics to high-temperature degradation processes and gas swelling processes were carried out according to the following scheme: the studied ceramic samples were subjected to hydrogenation, then the samples were thermally annealed for a long time. Hydrogenation was carried out by electrochemical charging (current density 20 mA/cm²) in an aqueous solution of 3% sodium hydroxide and 3 g/l CH₄N₂S at a temperature of 80°C for 5, 10, 24 and 48 hours, which made it possible to obtain different concentrations of hydrogen in the near-surface layer of ceramics. Determination of the structural degradation degree of ceramics, as well as the influence of various stabilizing additives on the resistance to hydrogen absorption processes, was carried out by determination of the volumetric swelling value of the crystalline structure, the values of which were obtained from the analysis of X-ray diffraction data. The determination of the volume expansion value was assessed based on changes in the values of the structural parameters of the

ceramics under study after testing, followed by their comparison with the initial values for each sample. This method made it possible to determine changes associated with swelling caused by the accumulation of structural distortions of the crystal lattice in the samples under external influences.

The determination of strength properties in order to identify the resistance of ceramics to hydrogenation processes was carried out by measuring the hardness of ceramics using the Vickers indentation method. For measurements, an LM 700 microhardness tester (LECO, St. Joseph, USA) was used.

Results and discussions

The volumetric swelling value ΔV was determined by comparing the volumes of the crystal lattice in the initial state and after exposure to the model environment used for hydrogenation. The assessment results of the change in the ΔV value from the hydrogenation time is shown in Figure 2 in the form of dependencies for all the ceramic samples under study.

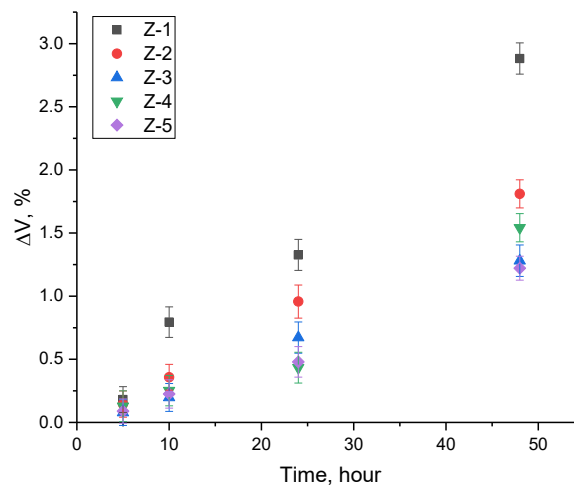


Figure 2 – Assessment results of the change in the ΔV value depending on the hydrogenation time.

The general appearance of the presented ΔV dependencies indicates a positive effect of addition of stabilizing components to the composition of ZrO₂ ceramics, the use of which, as can be seen from the $\Delta V(t)$ dependency data, leads to a reduction in the effect of volumetric swelling of the crystal structure, which in turn is associated with the effects of hydrogen agglomeration in pores, as well as the

formation of complex defects such as H-V_O, resulting in structural degradation. In the case of ZrO₂ ceramics not modified by addition of stabilizing dopants, an increase in the volumetric swelling value is observed after 10 hours of hydrogenation, and the maximum value after 48 hours is more than 2.5 %, which indicates a clearly expressed structural degradation of the ceramics associated with the accumulation

of deformation distortions in the structure. In the case of using stabilizing dopants, a decrease in the trend of changes in the $\Delta V(t)$ values is observed, which indicates the inhibition of volumetric swelling mechanisms, which can be explained by the following factors. In the case of polymorphic transformations of the monoclinic- $ZrO_2 \rightarrow$ tetragonal $-ZrO_2$ and monoclinic- $ZrO_2 \rightarrow$ cubic- ZrO_2 type, a decrease in the crystal lattice volume occurs due to rearrangement, which leads to its compaction and, as a consequence, a decrease in porosity, which limits the ability of hydrogen to fill voids. Also, polymorphic transformations lead to a decrease in the concentration of oxygen vacancies in the composition of ceramics, which also helps to restrain the mechanisms of formation of complex defects in the structure of ceramics. The third factor is due to the formation of interphase boundaries for ceramics with incomplete polymorphic transformation (for samples modified by adding $CaCO_3$), which in turn leads to the restraint of migration processes, leading to a decrease in the rate of degradation and disordering of the crystalline structure.

Figure 3 shows the assessment results of the change in the value of ΔV after 100, 300 and 500 hours of thermal annealing at a temperature of 700 °C, the choice of which is due to the possibilities of simulation of the operating processes of this type of ceramics when used as a basis for anode materials of solid oxide fuel cells. Experiments to determine the thermal impact on the degree of degradation of the studied samples associated with high-temperature corrosion processes were carried out in a muffle furnace in an oxygen-containing environment (air atmosphere); the holding time of the samples corresponded to the classical operating times of fuel cells. To determine the effect of hydrogen concentration in the ceramics on the rate of high-temperature degradation, comparative experiments were conducted using ceramic samples that were not subjected to hydrogenation processes and were annealed for similar time periods. Also, for all experiments, assessments of changes in the volumetric swelling of ceramics in the initial state, reflecting changes in the effect of thermal action on the structural degradation processes, in order to determine the effect of hydrogenation on changes in structural parameters, were made.

Tests of ceramics resistance to high-temperature degradation have shown that the most resistant to

volumetric swelling caused by thermal action are ceramics in which the cubic phase dominates, which is the most stable to high-temperature degradation processes. At the same time, the results of assessing the change in the value of $\Delta V(t)$ depending on the time of thermal action indicate a negative effect of accumulated hydrogen in the structure of ceramics on the resistance to disordering and deformation distortion of the structure. In this case, an increase in the concentration of accumulated hydrogen in the composition of ceramics leads to an acceleration of the processes of destabilization of the crystalline structure of ceramics due to the migration of point defects, as well as hydrogen, the agglomeration of which leads to an increase in deformation distortions. In this case, a change in the phase composition of ceramics due to polymorphic transformations leads to an increase in resistance to high-temperature degradation, which in turn allows for an increase in the service life of these materials as fuel cells.

Figure 4 shows the assessment results of the change in the hardness of the studied ceramics depending on the time of the hydrogenation experiments, reflecting the degradation of strength characteristics depending on their composition, as well as the concentration of accumulated hydrogen in the composition (with a change in the time of the experiment). The presented ΔHV data reflect the degree of hardness degradation after testing in comparison with the initial values, which makes it possible to determine both the maximum softening and the effect of stabilizing additives on resistance to external influences caused by hydrogenation processes. According to the general data presented, it is evident that the modification of ZrO_2 ceramics by adding stabilizing dopants leads to an increase in hardness, which indicates a positive effect associated with both the formation of impurity inclusions in the form of differences in the phase composition when adding the stabilizing dopant $CaCO_3$, and polymorphic transformations caused by the addition of MgO to the composition of the ceramics. Moreover, according to the assessment of changes in the hardness of ceramics in the initial state when doped with various stabilizing additives, it was established that the initialization of polymorphic transformation processes of the $m-ZrO_2 \rightarrow t-ZrO_2$ and $m-ZrO_2 \rightarrow c-ZrO_2$ type when adding MgO to the composition results in hardness growth by 10 % and 30 %, respectively. During addition of $CaCO_3$ to the composition of ceramics, with a change in the

concentration of which, in addition to polymorphic transformations of the $m\text{-ZrO}_2 \rightarrow t\text{-ZrO}_2$ and $m\text{-ZrO}_2 \rightarrow c\text{-ZrO}_2$ type, the presence of impurity inclusions in the monoclinic and tetragonal phases is observed in the composition of ceramics, the increase in hardness is 14 and 32 %, respectively. These differences in hardness values are caused by the effects of not

only polymorphic transformations, during which the volume of the crystal lattice changes, leading to its compaction and a decrease in porosity, but also by the effect of dispersion hardening, caused by the presence of impurity inclusions in the composition, which creates interphase boundaries that provide additional hardening under external influences on ceramics.

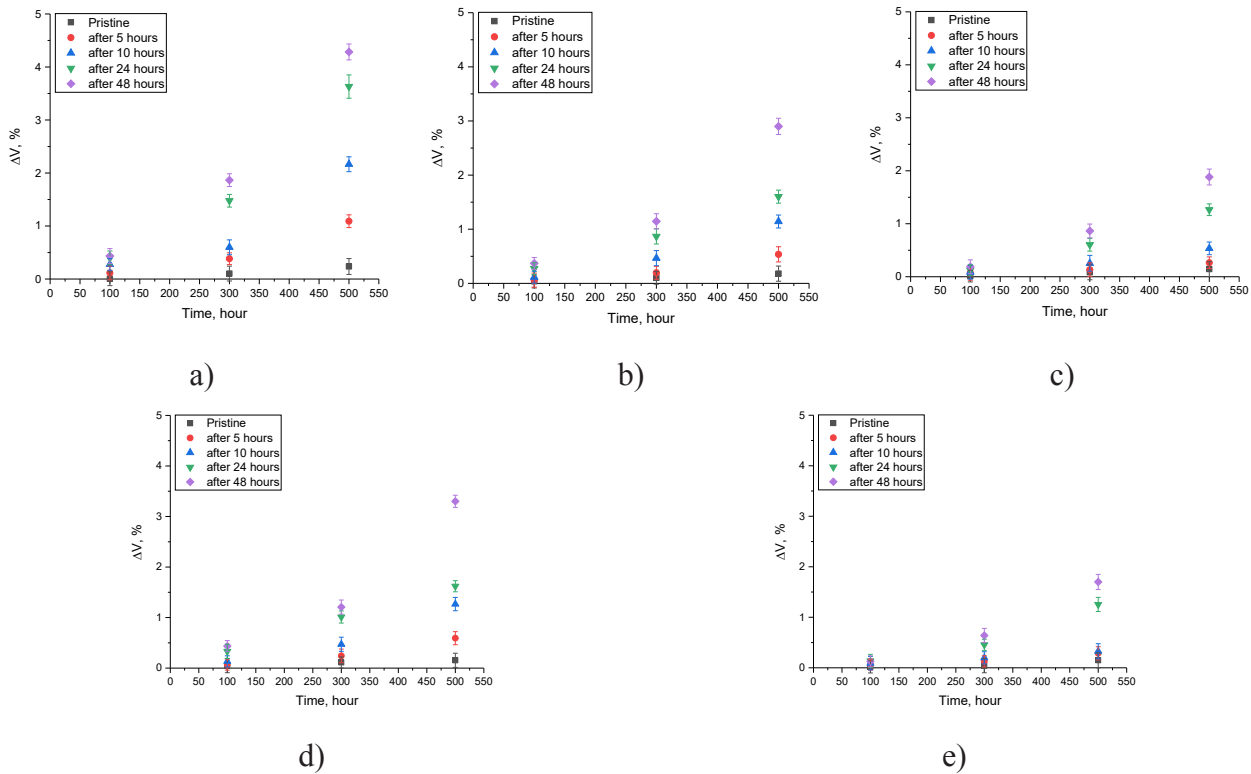


Figure 3 – Results of experiments on high-temperature degradation of the studied ceramic samples: a) Z-1; b) Z-2; c) Z-3; d) Z-4; e) Z-5.

For samples subjected to hydrogenation processes, a decrease in hardness indicates processes of structural degradation and weakening of ceramics, associated with both a change in the magnitude of volumetric swelling due to the accumulation of deformation distortions, and structural changes caused by the accumulation of complex defects in the structure. At the same time, the analysis of the observed changes in hardness indicates a positive effect of increasing resistance to softening, which is more than 1.5 – 2.5 times the increase in resistance to degradation caused by hydrogenation at the maximum time of hydrogenation experiments.

Figure 5 shows the results of the assessment of changes in hardness, indicating softening associated

with the effect of high-temperature degradation after 500 hours, as well as the influence of accumulated hydrogen on the rate of softening associated with diffusion effects that cause embrittlement and a decrease in resistance to external influences. The choice of conditions for conducting tests on the resistance of ceramics to degradation processes is determined by the potential operating modes of them as anode materials for solid oxide fuel cells. The results of the assessment of the stability of the strength properties of ceramics were carried out by comparative analysis of changes in hardness values in the initial state and after thermal tests simulating the processes of evolution of hydrogen migration in the damaged layer under prolonged thermal exposure.

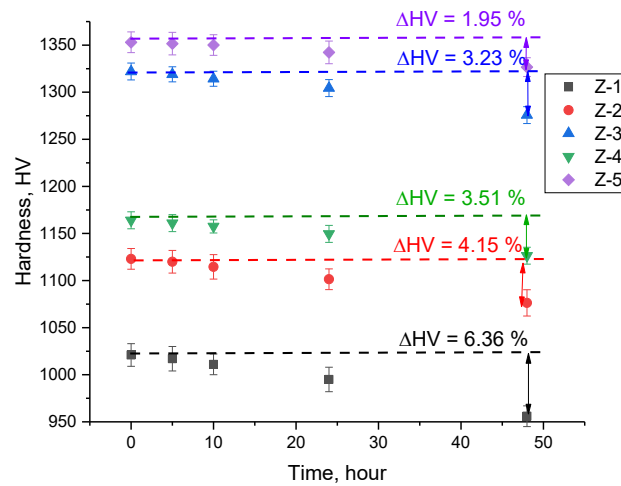


Figure 4 – Assessment results of the change in hardness of the studied ceramics depending on the hydrogenation time (the dotted lines show the maximum values of the degree of reduction in hardness after 48 hours of hydrogenation).

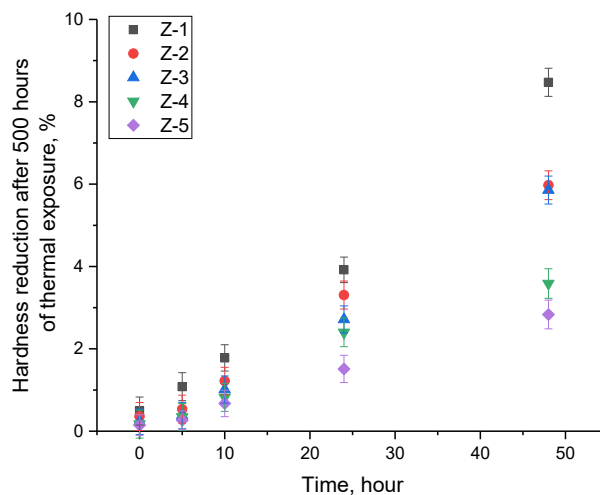


Figure 5 – Results of changes in the hardness values of the studied ceramics subjected to hydrogenation at different times, as well as subsequent thermal aging by holding the samples for 500 hours at a temperature of 700 °C in a muffle furnace.

The results of experiments to assess the softening of ceramics associated with the modeling of high-temperature degradation processes under thermal influence showed that the greatest changes in hardness associated with its decrease are observed for ceramic samples without dopants. It should be noted that the most pronounced degradation processes of strength characteristics occur for samples subjected to hydrogenation for 24 and 48 hours, for which the decrease in hardness is more than 4 and 10 %, respectively, with an increase in

the time of hydrogenation experiments. In the case of Z-2 and Z-3 ceramics, a decrease in hardness of about 6 % is observed, which indicates that the initialization of polymorphic transformation processes leads to an increase in resistance to high-temperature degradation. For samples Z-4 and Z-5, the maximum decrease in hardness is less than 4 %, which indicates a positive effect of dispersion hardening and the presence of interphase boundaries on resistance to high-temperature degradation, which is associated with the formation of additional barriers

to the propagation of microcracks under external influences. It should also be noted that for short hydrogenation experiment times, the change in the ΔHV value is less than 1.5 – 2.0 %, which indicates the high resistance of ceramics to hydrogenation processes and the associated structural embrittlement and swelling.

Conclusion

The results of experiments on hydrogenation of ZrO₂ ceramics, simulating the processes of structural degradation, comparable with operating modes, showed that the use of stabilizing components MgO and CaCO₃ makes it possible to increase the stability to volumetric swelling and high-temperature degradation. The hardening effects in this case are caused by the initialization of processes of polymorphic transformations of the m-ZrO₂ → t-ZrO₂ and m-ZrO₂ → c-ZrO₂ type caused in the composition of ceramics when stabilizing additives with different concentrations are added to their composition.

According to the assessment of the strength characteristics of ZrO₂ ceramics depending on the type of stabilizing dopant used for modification, it was found that the initialization of the polymorphic transformation processes of the m-ZrO₂ → t-ZrO₂ and m-ZrO₂ → c-ZrO₂ type leads to an increase in hardness by 10 and 30 %, respectively, while the formation of impurity inclusions in the structure, the presence of which is due to an incomplete process of polymorphic transformations, increases the hardness by 14 and 32 %, respectively. The observed growth in hardening due to impurity inclusions in this case is due to the effects of dispersion hardening, as well as the presence of interphase boundaries, the formation of which in the composition of ceramics leads to an increase in resistance to external influences.

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