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Effect of dislocation density-associated strengthening factors on the thermal stability of composite ceramics

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Interest in composite ceramics based on oxide and nitride compounds is due to the combination of their structural, strength and thermophysical parameters. Moreover, in the case of composites based on $xSi_3N_4 – (1-x)ZrO_2$, the strength parameters, as well as resistance to thermal expansion, are determined by the characteristics of zirconium dioxide, while the thermophysical parameters are determined by silicon nitride, for which the thermal conductivity values are almost an order of magnitude higher than for zirconium dioxide. The main method for production of composite $xSi_3N_4 – (1-x)ZrO_2$ ceramics was the mechanochemical solid-phase synthesis method using high-speed grinding and thermal annealing, used to stabilize structural deformations caused by mechanical action. During the research, it was determined that a change in the ratio of ceramic components due to an increase in the contribution of Si_3N_4 in the composition leads to an increase in thermophysical parameters, the change of which is due to the higher thermal conductivity of silicon nitride. In turn, a change in thermophysical parameters, and as a consequence, an alteration in the rate of heat transfer due to phonon mechanisms, causes an elevation in resistance to external influences during thermal shocks. Experiments to determine resistance to thermal influences have shown that the presence of a high density of dislocations in the near-surface layer of ceramics contributes to a rise in resistance to temperature changes, alongside external mechanical influences, which is expressed in less pronounced trends in decreasing hardness indicators during heat resistance tests.

Key words: composite ceramics, thermal stability, thermophysical parameters, silicon nitride, zirconium dioxide, dislocation strengthening.
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1 Introduction

Over the past few years, in the field of structural materials, the main emphasis has been placed on increasing the resistance of new types of materials to external influences, alongside their ability to operate under extreme conditions, including elevated temperatures, high doses of radiation damage, exposure to aggressive media, etc. [1,2]. Among the most promising materials in this research area are high-entropy alloys based on refractory compounds, as well as composite ceramics, which are a combination of oxide, nitride and carbide compounds of aluminum, silicon, zirconium, niobium, tantalum or tungsten [3-5].

At the same time, the key limiting factor for the widespread use of composite ceramics based on oxide and nitride compounds is the low thermophysical parameters, in particular, thermal conductivity indicators, which in the case of ceramics are about 2-50 W/Km, while for steels and alloys these indicators are two orders of magnitude higher [6,7]. It should also be noted that in most cases, nitride ceramics have higher thermal conductivity than oxide ceramics, however, oxide ceramics have fairly high resistance to external influences, which determines their strength properties [8,9]. In this regard, one of the important areas of research in the field of creating composite ceramics is the search for new methodological approaches to improve the strength characteristics of ceramics, the main goal of which is to preserve the phase and elemental composition. In particular, one of the ways to increase resistance to external influences, including mechanical and thermal, is the use of ceramics with nano-sized grains, interest in which is due to their unique properties, which are caused by the presence of a large number of grain boundaries, as well as dislocations. In this case, changing the grain size allows you to vary the dislocation density, as well as the packing density of grains, which eliminates the presence of a large number of voids in the volume of ceramics, which also allows you to solve a number of problems associated with the porosity of ceramic materials.

The dislocation strengthening factor associated with size effects is considered as one of the ways to increase the resistance of materials, including composite ceramics, to external influences, such as thermal effects, high-temperature aging associated with degradation and embrittlement, and radiation damage [10-12]. In this case, the variation in the density of dislocations can be carried out both by crushing grains under external mechanical loads and by changing the variation in the concentration of components in the composition of composite ceramics. If in the first case, the change in dislocations is directly dependent on the grain sizes, then in the case of variations in the components, the change in dislocation density depends on many factors, including the phase composition of the composites, the change of which directly depends on the conditions for obtaining ceramics [13, 14]. The combination of two methods of changing the dislocation density makes it possible to obtain high-strength ceramics that can withstand large mechanical loads, as well as maintain the stability of strength properties during long-term exposure to external factors, such as high temperatures or thermal shocks. Moreover, earlier, in [15], it was shown that the most effective way to change the dislocation density due to mechanical influences is high-speed grinding in planetary mills with subsequent thermal stabilization of the phase composition by removing deformation distortions in the structure of composite ceramics arising under external mechanical influences.

The main aim of the presented study is to determine the influence of dislocation strengthening caused by size effects in $xSi_3N_4 – (1-x)ZrO_2$ on the change in resistance to thermal shocks resulting from extreme operating conditions [16-18]. At the same time, during the experiments, dependencies were obtained for changes not only in strength properties, i.e. changes in the hardness values of ceramics before and after external influences, but also in maintaining the stability of thermophysical parameters, the change of which is directly related to deformation distortions that occur during embrittlement and softening of ceramics. Interest in this type of ceramics is primarily due to the possibility of creating high-strength ceramics due to the strength properties of zirconium dioxide, which have high thermal conductivity, which is due to the presence of silicon nitride in the composition of composite ceramics, which has fairly high thermal conductivity in comparison with oxide materials [19, 20].

2 Methodology

To obtain samples of composite ceramics based on $xSi_3N_4 – (1-x)ZrO_2$ compounds, mechanochemical solid-phase synthesis combined with thermal sintering of the samples was used. The synthesis was carried out using chemical reagents of 99.95 % purity; the initial powders were purchased from Sigma Aldrich (Sigma Aldrich, USA). The grain sizes of the original powders were on the order of 5 – 10 µm. To give them nanosizes, a high-speed grinding method was used in a planetary mill PULVERISETTE 6 classic line (Fritsch, Berlin, Germany). The grinding speed was 600 rpm, the time of mechanical action of the grinding media was about 30 minutes. Thermal sintering in a muffle furnace at a temperature of 1500°C for 5 hours was chosen to stabilize the crystal structure, initiate phase transformation processes, and also reduce mechanically induced deformation distortions in the crystal structure of ceramics during grinding. At the same time, the selection of temperature and annealing time conditions was carried out a priori to prevent the occurrence of effects of thermal fusion and agglomeration of grains into large agglomerates, eliminating the occurrence of the effect of dislocation strengthening associated with the size factor.

The grain sizes of the studied ceramics were determined using the optical laser diffraction method implemented on an ANALYSETTE 22 NeXT Nano particle analyzer (Fritsch, Berlin, Germany).

Figure 1 reveals data on alterations in grain sizes depending on the concentration of components in the ceramic composition, obtained by analyzing grain size distribution diagrams in the form of average values with measurement error. The formation of dislocation strengthening associated with the size factor was carried out by varying the concentration of components in the ceramic composition in the range x from 0.1 to 0.5 M, changing which during high-speed grinding results in more intensive crushing of grains and their reduction by more than 2 times. At the same time, a reduction in grain size, as is evident from the data presented in Figure 1, leads to the effect of an elevation in dislocations and the volumetric contribution of grain boundaries. This effect is most pronounced at Si_3N_4 concentrations above 0.25 M in the ceramic composition. According to the presented data, a decline in grain size by more than 2 times leads to a growth in the dislocation density of the volume fraction of grain boundaries by more than 3.5 – 4 times, which indicates that even small changes in grain size allow one to vary the dislocation density, changes in which can be used to create strengthening effects [21-23].

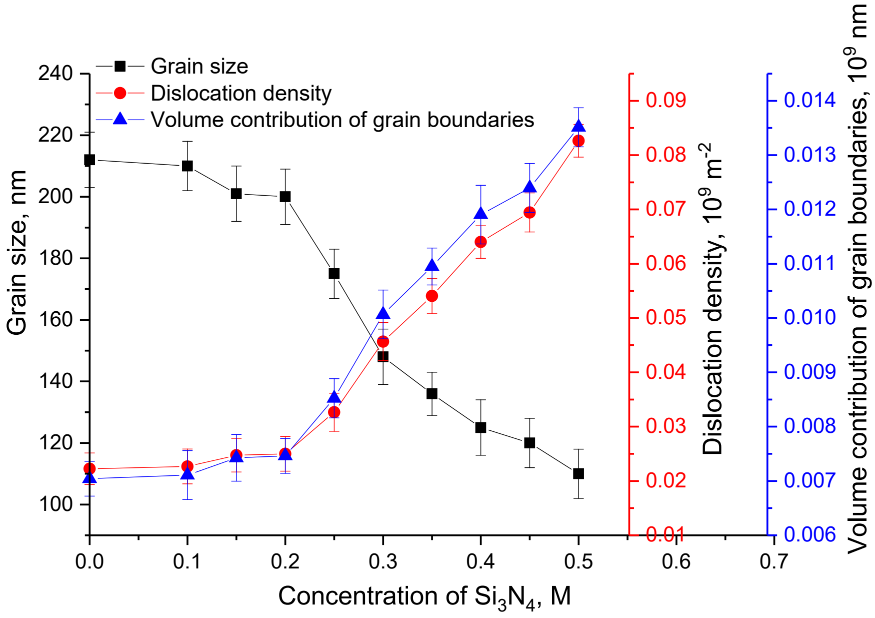

Figure 1 – Results of a comparative analysis of changes in grain sizes, dislocation density and volumetric contribution of grain boundaries

Determination of the influence of variations in the ratio of components in $xSi_3N_4-(1-x)ZrO_2$ ceramics on changes in thermophysical parameters, in particular, on changes in the thermal conductivity coefficient, was carried out using the method of measuring longitudinal thermal flow. The measurements were carried out on ceramic samples pressed into tablets with a diameter of 10 mm and a thickness of about 1 mm, which made it possible to eliminate the effects of uneven heat transfer and also reduce heat losses in a small volume. The measurements were carried out using a thermal conductivity meter KIT-800 (KB Teplofon, Russia).

The determination of strength properties, in particular, the hardness values of ceramics, alongside their changes depending on external influences, including thermal tests for heat resistance, was carried out by the indentation method, implemented using a Duroline M1 microhardness tester (Metkon, Bursa, Turkey).

The thermal stability of the studied xSi_3N_4 – (1-x)ZrO_2 ceramics, obtained by mechanochemical grinding, was assessed through experimental work, consisting of rapid thermal heating of samples to a temperature of 1000 °C, holding them at this temperature for 1 hour and subsequent rapid removal to air in order to create a sharp temperature gradient that can lead to the initiation of oxidation processes and destabilization of the strength properties of ceramics. The heating rate of the samples was about 50 °C/min. For testing, a muffle furnace was used with the ability to control the temperature inside the chamber with an accuracy of about ±5°C, which is an acceptable error parameter for high-temperature effects. Determination of the influence of hardening factors in this case was carried out by assessment of the change in the strength and thermophysical parameters of ceramics before and after heat resistance tests. Determination of the effect of dislocation strengthening on maintaining stability after thermal tests was carried out by comparative analysis of changes in the degree of hardness degradation after 5 successive test cycles with changes in dislocation density, presented in Figure 1. Calculation of the strength degradation degree was carried out by comparing the hardness value in the initial state and after 5 successive test cycles, which, in terms of percentage, made it possible to estimate the amount of softening caused by thermal effects.

3 Results and discussion

Figure 2 demonstrates the results of alterations in the thermal conductivity coefficient for xSi_3N_4 – (1-x)ZrO_2 ceramics with varying component concentrations. Changes in the thermal conductivity coefficient observed in the presented dependence reflect the direct influence of the ratio of components in the composition on the increase in thermal conductivity. In the case of low concentrations of Si_3N_4 (less than 0.2 M), the thermal conductivity coefficient is less than 2 W/m×K, a value characteristic of ZrO_2 ceramics, the thermal conductivity value of which is significantly lower than for nitride ceramics. In the case when the Si_3N_4 concentration is greater than 0.25 M, an increase in the thermal conductivity coefficient to 4 – 10 W/m×K is observed, depending on the Si_3N_4 concentration in the composition. Such changes in thermal conductivity are due to the effect associated with higher thermal conductivity of Si_3N_4, the contribution of which increases with increasing concentration in the ceramic composition. It should be noted that, according to the data presented in Figure 2, the mechanisms of changes in thermal conductivity at high concentrations of Si_3N_4 are not significantly affected by changes in grain sizes, since in the case of thermophysical parameters, a key role in determination of the heat transfer mechanisms is played by phonon mechanisms, which are the rescattering of phonons that carry heat, and in the case of a large number of small grains, the rescattering effect can be slowed down. However, the thermophysical parameters of Si_3N_4 themselves in this case suppress the size effect, which results in thermal conductivity growth when the ratio of components in the ceramic composition changes.

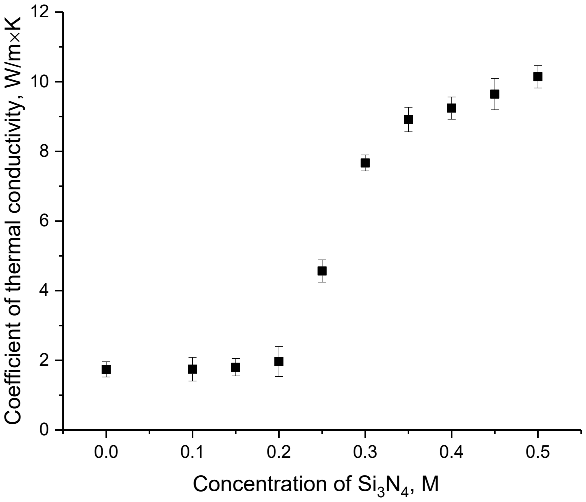

Figure 2 – Data on changes in the thermal conductivity coefficient for xSi_3N_4 – (1-x)ZrO_2 ceramics depending on variations in the concentration of components

Figure 3 illustrates the results of changes in the hardness of xSi_3N_4 – (1-x)ZrO_2 ceramics during heat resistance tests, which were carried out to establish the stability of the strength properties of ceramics under external thermal influences. Assessing the data on changes in hardness values for xSi_3N_4 – (1-x)ZrO_2 ceramics in the initial state, it can be concluded that a decline in grain size from 200 – 210 nm, characteristic of low Si_3N_4 concentrations in ceramics up to 110 – 140 nm, characteristic of Si_3N_4 concentrations of more than 0.2 M, leads to an elevation in hardness. Such changes are due to the effect of dislocation strengthening, which manifests itself for ceramics with small grain sizes and causes strengthening, as well as an increase in stability to mechanical stress, which can be assessed by the shape and imprints of the indenter, characterized by the absence of microcracks near the tops of the imprint pyramid. Moreover, according to the data presented, an alteration in grain size, caused by a variation in the ratio of components in the composition of ceramics, results in hardness growth by more than 1.4 – 1.5 times compared to ZrO_2

ceramics, without adding Si_3N_4 to them. From which it can be concluded that the variation in the ratio of the components determines the processes of grain crushing when the Si_3N_4 concentration in the ceramics reaches more than 0.2 M, which indicates that at low concentrations of Si_3N_4 in the ceramics, the dominant role in determining the strength parameters is played by ZrO_2. In the case when the Si_3N_4 concentration is more than 0.2 M, strengthening is due to the effects of grain crushing and boundary effects, leading to the emergence of additional barriers to the propagation of microcracks that arise under mechanical stress.

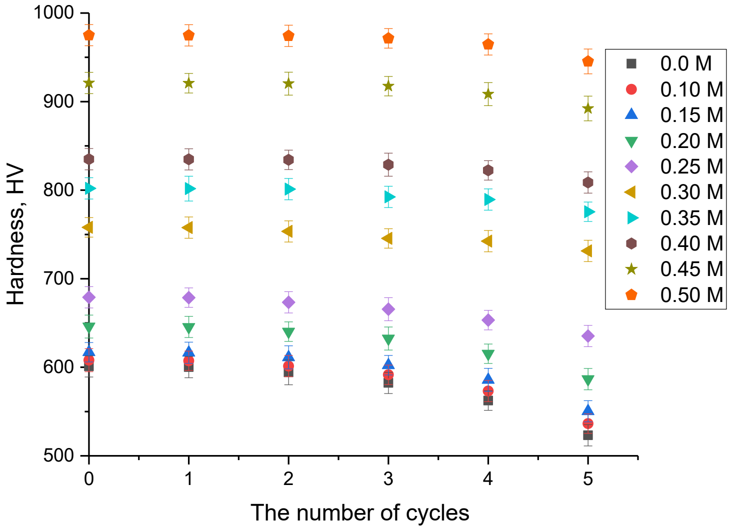

Figure 3 – Results of measurements of hardness values of the studied samples contingent upon the number of heat resistance test cycles

The overall view of the presented dependences of alterations in hardness values during heat resistance tests indicates that with a small number of thermal changes (1 – 2 consecutive tests), the decrease in hardness values is within the permissible error, and the deviation is no more than 0.1 %. In this case, a change in the ratio of components in $xSi_3N_4 – (1\text{-}x)ZrO_2$ ceramics results in a decrease in the hardness value deviation from the initial value. The main changes in hardness values are observed after 2-3 cycles, while the trend of changes in hardness is different for the ceramics under study. The observed reduction in the hardness values of ceramics after 2-3 successive cycles of heat resistance tests is due to degradation associated with deformation processes resulting from thermal expansion of the crystal lattice and subsequent sudden cooling. In the case where ZrO_2 dominates in the composition of $xSi_3N_4 – (1\text{-}x)ZrO_2$ ceramics, the trend of hardness deterioration as a result of successive tests is more pronounced, and the maximum deviation (ΔHardness) is more than 12 % compared to the initial value, which indicates a fairly pronounced softening of ceramics as a result of thermal tests for heat resistance. In the case of ceramics in which the Si_3N_4 concentration is more than 0.2 M, the value of ΔHardness is no more than 3.0 – 3.2 %, which is acceptable for such tests, and such a small change indicates a fairly high resistance of the ceramics to external influences. including thermal expansion and deformation distortion of the structure caused by a sharp change in the temperature of the sample.

Figure 4 reveals a comparison of variations in the softening value (decrease in hardness after 5 successive cycles of heat resistance tests) and dislocation density associated with changes in grain sizes in $xSi_3N_4 – (1\text{-}x)ZrO_2$ ceramics.

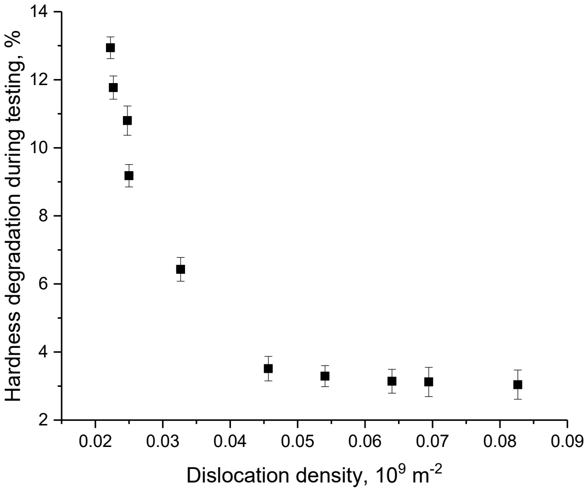

Figure 4 – Comparison of the softening value after 5 cycles and dislocation density in $xSi_3N_4 – (1\text{-}x)ZrO_2$ ceramics

The general view of the presented dependence of the variation in the softening value (decrease in hardness after 5 cycles of heat resistance tests) on the dislocation density indicates the positive effect of increasing the dislocation density on maintaining the stability of hardness during thermal tests. It is important to highlight that the most pronounced alterations are observed with an elevation in dislocation density from 0.02×10^9 m^{-2} to 0.04-0.05×10^9 m^{-2}, at which the softening resistance growth is more than fourfold (from 12 % to 3 %). In turn, a further increase in dislocation density does not lead to such a pronounced change in the resistance to degradation of strength properties, which can be explained by effects associated with dimensional factors, as well as the phase composition of ceramics. The influence of the phase composition in this case may be that with an increase in Si_3N_4 composition, which results in hardness growth in the initial state, as well as a decrease in grain size, in the case of long-term thermal tests, it does not contribute to higher stability under thermal influences, as well as mechanical loads.

Figure 5 demonstrates the comparison results of changes in the thermal conductivity coefficient of the studied $xSi_3N_4 – (1\text{-}x)ZrO_2$ ceramics after 5 successive cycles of heat resistance tests, reflecting the influence of dislocation strengthening factors on resistance to thermal influences.

The general appearance of the data presented reflects the direct influence of both the size factor, which determines dislocation strengthening, and the influence of variations in the ratio of components in the composition of ceramics. In the case when ZrO_2 dominates in the composition of ceramics, the degradation of strength and thermophysical parameters is maximum and amounts to more than 8 – 12 % after five test cycles. Such a strong decrease is due to heat transfer mechanisms, which in the case of low thermal conductivity of ZrO_2 result in thermal expansion coefficient growth, which in turn plays an important role in the volumetric change of the heated material due to an increase in the vibration amplitude of atoms in the crystal lattice. In this case, a sharp change in temperature (the so-called thermal shock) and low thermal conductivity lead to the occurrence of deformation structural distortions caused by thermal expansion and slow heat transfer. As a result, not only volumetric distortion of the crystalline structure occurs, but also the formation of highly disordered inclusions in the ceramic composition, a rise in the number of which as a result of successive thermal effects results in surface layer destabilization, and as a consequence, a decrease in hardness and resistance to external mechanical influences. A change in the thermophysical parameters of $xSi_3N_4 – (1\text{-}x)ZrO_2$ ceramics with an increase in the concentration of Si_3N_4 in the composition leads to an increase in the stability of the strength and thermophysical parameters, and their decrease after five cycles of successive tests is no more than 3 – 3.3 % of the initial value. Such small alterations are due to heat transfer mechanisms due to the contribution to the thermal conductivity of Si_3N_4, which leads to the fact that with sharp temperature changes, the influence of the disorder factor caused by changes in the amplitude of thermal vibrations is less pronounced than in the case of ZrO_2 dominance. Also, in this case, the stabilization of strength properties is influenced by the dislocation strengthening factor, the influence of which is expressed in the creation of additional dissipative barriers that restrain deformation distortion resulting from thermal effects. From the analysis it follows that the formation of composite ceramics in which the ratio of components is close to equal makes it possible to obtain high-strength ceramics with good resistance to thermal shock, as well as having fairly high thermal conductivity in comparison with ZrO_2. At the same time, the use of high-speed mechanical-chemical grinding makes it possible to obtain nano-sized composite ceramics, the grain size of which is about 100 – 120 nm, which causes the presence of dislocation strengthening associated with the size factor.

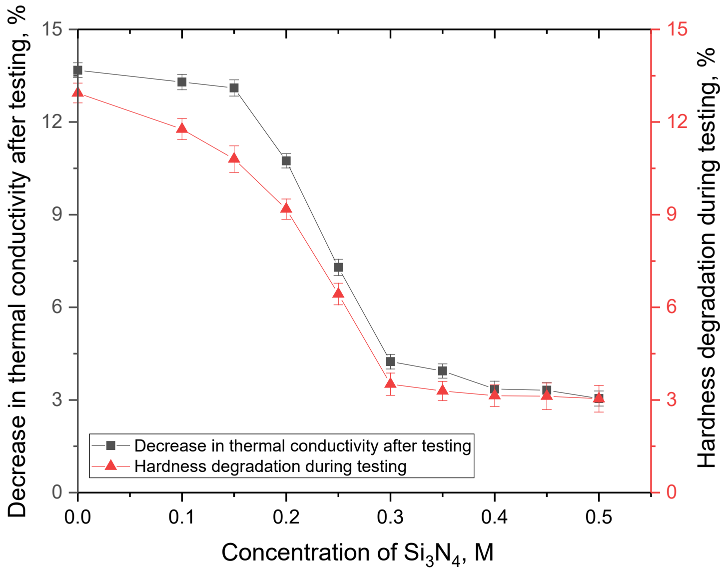

Figure 5 – Comparison of the results of changes in thermal conductivity coefficient and hardness degradation during heat resistance tests

4 Conclusions

The paper presents the assessment results of changes in the thermophysical and strength parameters of $xSi_3N_4 – (1-x)ZrO_2$ ceramics during heat resistance tests (i.e., under a sharp temperature change). According to the studies carried out, it was found that a variation in the ratio of components in ceramics, caused by a rise in Si_3N_4 in the composition, results in accelerated fragmentation of grains during mechanical grinding, which in turn determines a change in the dislocation density and volume fraction of grain boundaries in the composition of ceramics.

According to the assessment of changes in thermophysical parameters, it was determined that an increase in the composition of $xSi_3N_4 – (1-x)ZrO_2$ ceramics Si_3N_4 by more than 0.2 M leads to an increase in the thermal conductivity coefficient from 2 W/m×K to 9 – 10 W/m×K, which in turn results in stabilization of thermophysical parameters under external influences.

During determination of the main mechanisms of softening caused by thermal effects, it was determined that the most significant alterations in hardness are associated both with the size effect, which consists in the fact that at Si_3N_4 concentrations in $xSi_3N_4 – (1-x)ZrO_2$ ceramics above 0.2 M, a sharp decrease in grain size is observed, leading to the appearance of a large number of dislocations and grain boundaries. In this case, a variation in dislocation density causes the strengthening of ceramics, both in the case of initial values and during heat resistance tests.

According to the assessment of alterations in the hardness indicators of the studied $xSi_3N_4 – (1-x)ZrO_2$ ceramic samples after heat resistance tests, it was found that an increase in dislocation density from 0.02×10^9 m^{-2} to $0.04 – 0.08\times10^9$ m^{-2} leads to an increase in the stability of ceramics by more than 3.0 – 3.5 times. In this case, the most significant changes in stability indicators are observed when the dislocation density changes from 0.02×10^{9} m^{-2} to 0.04×10^{9} m^{-2}, while a further increase in dislocation density does not have a significant effect on the change in degradation resistance indicators.

Further research in this direction will be related to the study of the prospects for using the dislocation strengthening factor to maintain the stability of composite ceramics under external influences, including gas swelling and radiation damage caused by exposure to both low-energy and high-energy ions. The main goal of such research will be to expand opportunities in the search for alternative structural materials with high thermal conductivity, strength, and resistance to external influences.

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