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Research article
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Effect of magnesium oxide on the microstructure and mechanical properties of yttria-stabilized zirconia-based ceramics

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Abstract. In the present work, sintering and investigation of composite ceramic materials based on nanostructured MgO–ZrO_2 powders were carried out. Zirconium dioxide was additionally stabilized with 3 mol. % yttrium oxide. The nanopowders were pre-treated by mechanical activation using a planetary ball mill at a rotation frequency of 10 Hz. Zirconium dioxide balls were used as the grinding media. The prepared powders were compacted at pressing pressures of 50, 100, 200, and 300 MPa. The compacts were sintered in a high-temperature furnace at 1700 °C. Microstructural studies were performed on the polished surfaces of the sintered samples using scanning electron microscopy (SEM). EDX mapping was conducted to determine the elemental distribution, confirming the presence of two phases in all samples. To evaluate the effectiveness of stabilizing additives on the polymorphic transformation of zirconium dioxide, *X*-ray diffraction (XRD) analysis was performed. The porosity of the materials and its dependence on the pressing pressure and magnesium oxide content were also assessed. Mechanical properties such as Martens hardness and elastic modulus were measured using a NanoIndenter G200, while flexural strength was evaluated by scratch testing on the same device. Fracture toughness was determined by the indentation method using the Marshall–Evans approach. The influence of magnesium oxide additives on the physical and mechanical properties of the MgO–ZrO_2 composite ceramics was established.

Keywords: zirconium dioxide, magnesium oxide, nanostructured powders, activated sintering, ceramics, nanoindentation, EDX mapping

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Влияние оксида магния на микроструктуру и механические свойства керамики на основе диоксида циркония, стабилизированного оксидом иттрия

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Аннотация. Проведены спекание и исследование композиционных керамических материалов на основе наноструктурированных порошков $MgO–ZrO_2$. Диоксид циркония был дополнительно стабилизирован 3 мол. % оксидом иттрия. Применяемые нанопорошки предварительно обрабатывались методом механоактивации с помощью планетарной шаровой мельницы при частоте вращения размольных сосудов 10 Гц. В качестве мелющих тел использованы шары из диоксида циркония. Подготовленные порошки были спрессованы при давлении прессования 50, 100, 200 и 300 МПа. Полученные прессовки спекались в высокотемпературной печи при температуре 1700 °С. На подготовленной полированной поверхности спеченных образцов проведены микроструктурные исследования методом растровой электронной микроскопии. Выполнено EDX-картирование для выявления распределения элементов, установлено наличие двух фаз во всех изученных образцах. Для оценки эффективности влияния стабилизирующих добавок на полиморфное превращение диоксида циркония осуществлен рентгенофазовый анализ. В ходе исследования определены пористость материалов и ее зависимость от давления прессования и содержания оксида магния. При проведении индентирования на приборе «NanoIndenter G200» изучены механические свойства образцов – твердость по Мартенсу и модуль упругости, а в ходе Scratch-тестирования на данном оборудовании – их предел прочности на изгиб. По методу индентирования с использованием зависимости Маршала–Эванса определена трещиностойкость образцов. В ходе исследования установлено влияние добавок оксида магния на физико-механические свойства композитной керамики $MgO–ZrO_2$.

Ключевые слова: диоксид циркония, оксид магния, наноструктурированные порошки, активированное спекание, керамика, наноиндентирование, EDX-картирование

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Introduction

Zirconium dioxide-based ceramics have found wide application in various fields of science and technology due to their outstanding properties. This material exhibits high fracture toughness [1], low coefficient of friction [2], and significant wear resistance and strength [3]. For this reason, zirconium dioxide-based ceramic materials are widely used in dentistry [4], as well as in the production of hip joint head implants, cutting tools, bearing rolling elements, heat-resistant components, and many other applications.

However, due to the phase transformation of zirconium dioxide into the monoclinic phase and the associated volumetric changes, a number of limitations arise in the manufacturing of components from this material [5]. In particular, the introduction of stabilizing additives is required – most commonly yttrium, calcium, or cerium oxides. The addition of stabilizers makes it possible to prevent the phase transformation by forming a substitutional solid solution based on zirconium dioxide and the introduced additive. The ionic radius of the substituting elements is close to that of (Zr^{4+}), but slightly larger [6].

The prevention of the phase transformation can also be achieved by other means, in cases where the components do not form solid solutions. One such method is the stabilization by creating a composite material based on $Al_2O_3–ZrO_2$. Due to the high elastic modulus of aluminum oxide and its lower thermal expansion, sintering results in a rigid matrix in which zirconium dioxide particles are uniformly distributed and subjected to a compressive stress field. As a result, zirconium dioxide does not undergo polymorphic transfor-

mation, and the composite structure enables improved mechanical properties [7]. Magnesium oxide, silicon nitride, and other high-modulus inclusions can also serve as the matrix material [8].

Zirconium dioxide stabilization can be achieved by the combined effect of several factors. However, complex oxide systems consisting of three or more ceramic oxide components have not yet been sufficiently studied. For example, in [9], the effect of small additions (up to 2 wt. %) of MgO on ZTA–CeO_2 ceramics was evaluated. It was found that the relationship between the additive and the resulting mechanical properties is nonlinear, with optimal values observed at 0.5 wt. % MgO. This was explained by the formation of two new phases – $MgAl_{11}CeO_{19}$ and $MgAl_2O_4$. In [10], it was noted that increasing the magnesium oxide content up to 8 mol. % enhances fracture toughness while reducing hardness. In [11], the addition of yttrium oxide to Mg–PSZ ceramics resulted in a noticeable increase in hardness with only a slight decrease in fracture toughness. These studies indicate significant potential for developing composite ceramics based on zirconium dioxide with magnesium and yttrium oxide additives.

The objective of this study was to determine the effect of magnesium oxide content on the microstructure and mechanical properties of ceramics in a complex oxide system: $MgO–ZrO_2–Y_2O_3$.

Materials and methods

In this study, nanostructured zirconium dioxide powders of grade UDPO VTU 4-25-90 produced by plasma chemical synthesis (with an average particle size of 500 nm) and industrial-grade micron-sized magnesium oxide powders of grade MRTU 6-09-3391-67 (particle size <40 μm) were used. The purity of both powders was 99 %.

A 3 mol. % yttrium oxide additive was introduced into the zirconium dioxide powder. Based on these powders, mixtures with the following compositions (mol. %) were prepared: $2MgO–98ZrO_2$; $4MgO–96ZrO_2$; $8MgO–92ZrO_2$; $16MgO–84ZrO_2$. The compositions of these mixtures in molar and mass percentages are presented in Table.

Composition of powder mixtures

Соотношение компонентов в порошковых смесях

Mixture composition	
mol. %	wt. %
$2MgO–98ZrO_2$	$0.65MgO–99.35ZrO_2$
$4MgO–96ZrO_2$	$1.33MgO–98.67ZrO_2$
$8MgO–92ZrO_2$	$2.75MgO–97.25ZrO_2$
$16MgO–84ZrO_2$	$5.83MgO–94.17ZrO_2$

The powders under investigation were pre-treated by mechanical activation. Mechanical activation was carried out in an Activator-2SL planetary ball mill (Activator Machine-Building Plant, Novosibirsk, Russia) under the following conditions: grinding vessel rotation frequency – 10 Hz, treatment time – 10 min, and a grinding media to powder mass ratio of 3:1. Zirconium dioxide balls were used as grinding media.

The prepared powder mixtures were compacted using carboxymethyl cellulose as a plasticizer at uniaxial pressing pressures of 50, 100, 200, and 300 MPa. The green compacts were then sintered in a high-temperature furnace at 1700 °C with a 1 h dwell time at the target temperature. The densities of the sintered samples were determined using the hydrostatic weighing method. Since the formation of a composite structure with possible new solid solutions complicates the determination of theoretical density, porosity was evaluated based on micrographs of the sample surfaces obtained by scanning electron microscopy (SEM) at low magnification ($200^{\times}$), following the procedure described in [12; 13].

SEM investigations of the sample surfaces and elemental analysis (EDX mapping) were carried out using a Zeiss EVO 50 scanning electron microscope (Carl Zeiss, Germany).

The phase composition of the materials was studied by *X*-ray diffraction (XRD) using a Shimadzu XRD-7000 diffractometer (Japan) with $CuK_{\alpha 1}$ radiation (λ = 1.5406 Å) and step scanning in the 2θ range of 10–90°. Diffraction peak identification was performed using the Crystallographica Search-Match software and the PDF4+ structural database. The structural analysis was conducted using the PowderCell 2.4 program and the same database.

The mechanical properties of the sintered samples were evaluated using a NanoIndenter G200 (KLA-Tencor, USA) equipped with a Berkovich diamond tip under a 500 mN load. Martens hardness and elastic modulus were determined from the loading curves. Scratch testing was used to determine the flexural strength of the samples under indentation. This method involves scratching the sample surface under a linearly increasing load up to 10 mN, followed by measurement of the crack depth and width. The nanoindentation procedure is described in detail in [14; 15]. Fracture toughness was determined using a Vickers microhardness tester PMT-3 (LOMO JSC, St. Petersburg, Russia) by the indentation method [16]. Cracks were induced under a 5 N load.

Results and discussion

After sintering under the specified conditions, high-density samples were obtained (Fig. 1). Among the studied compositions, the samples with 8MgO–92ZrO_2 exhibited the highest density. It was found that the dependence of the relative density of MgO–ZrO_2 composite ceramics on composition is nonlinear. At all investigated compaction pressures, the relative densities of the sintered samples decreased in the following order (mol. %): 8MgO–92ZrO_2, 4MgO–96ZrO_2, 16MgO–84ZrO_2, 2MgO–98ZrO_2, which is consistent with the findings reported in [17; 18]. It was shown in [17] that increasing the sintering time for the 8MgO–92ZrO_2 ceramic composition leads to a further increase in density, continuing up to 20 h of treatment. In [18], it was noted that the porosity of MgO–ZrO_2-based composites varies depending on the presence of magnesium oxide; however, this dependence differs across temperature ranges, exhibiting both an increase and a decrease in material porosity. Moreover, a linear trend was observed only within specific temperature intervals. The study in [18] was conducted at higher temperatures than the present work and reported elevated porosity levels in the range of 24 to 32 %. These findings highlight the effectiveness of the mechanical activation parameters applied in this study and suggest its further use when working with materials of this composition.

Using EDX mapping to determine the elemental composition of ceramic samples compacted at a pressure of 300 MPa, images of the polished cross-sectional surfaces were obtained. Fig. 2 shows the elemental distribution and microstructure images for the 2MgO–98ZrO_2 composition.

Based on the results of EDX mapping of MgO–ZrO_2-based samples, the presence of two distinct phases –

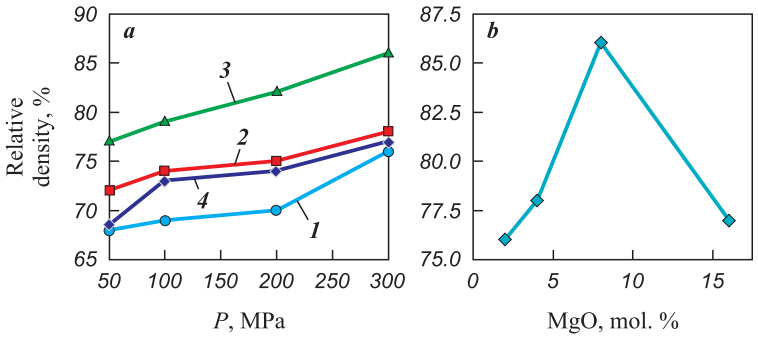

Fig. 1. Dependence of the relative density of sintered samples on pressing pressure (***a***) and on magnesium oxide content for samples compacted at P = 300 MPa (***b***)
Samples, mol. %: ***1*** – 2MgO–98ZrO_2, ***2*** – 4MgO–96ZrO_2, ***3*** – 8MgO–92ZrO_2, ***4*** – 16MgO–84ZrO_2

Рис. 1. Зависимость относительной плотности спеченных образцов от давления прессования (***a***) и содержания оксида магния для образцов, полученных при P = 300 МПа (***b***)
Образцы, мол. %: ***1*** – 2MgO–98ZrO_2, ***2*** – 4MgO–96ZrO_2, ***3*** – 8MgO–92ZrO_2, ***4*** – 16MgO–84ZrO_2

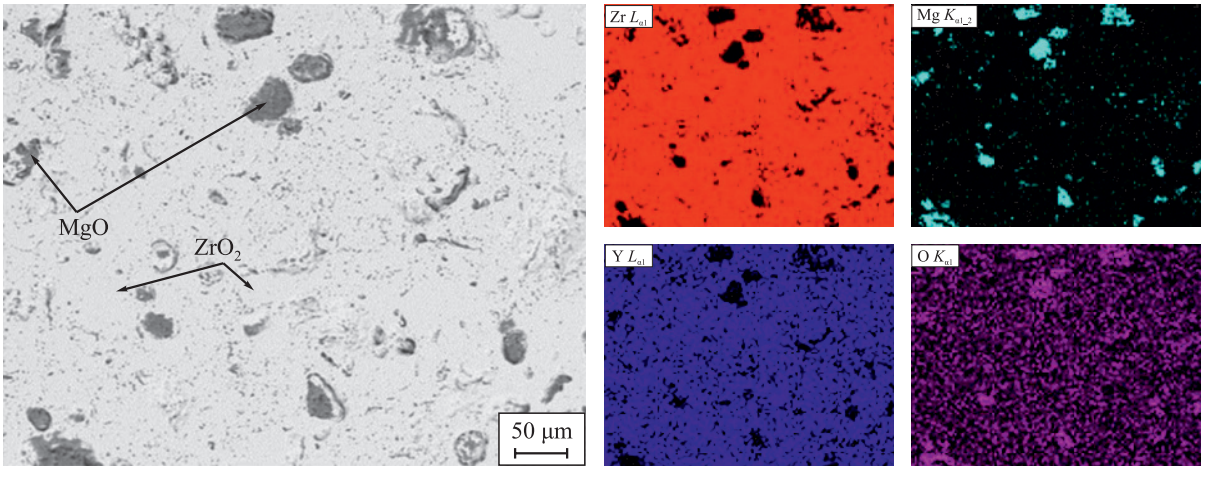

Fig. 2. Elemental analysis of a sintered sample with the composition 2MgO–98ZrO_2

Рис. 2. Элементный анализ спеченного образца состава 2MgO–98ZrO_2

MgO and ZrO_2 – was established, which is consistent with the observations reported in [19]. However, according to [10; 11; 20], the formation of a solid solution based on ZrO_2–MgO should occur. The two-phase composite structure obtained in this study indicates that no interaction between MgO and ZrO_2 takes place during sintering. According to the EDX mapping results, magnesium does not enter the crystalline structure of ZrO_2 and does not form a solid solution based on ZrO_2–MgO, which is attributed to the stabilizing effect of Y_2O_3.

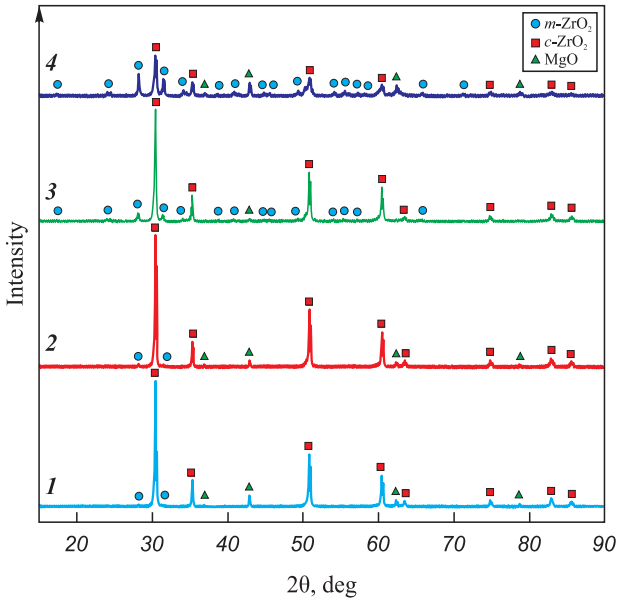

Fig. 3. *X*-ray diffraction (XRD) analysis
Samples, mol. %: ***1*** – 2MgO–98ZrO_2, ***2*** – 4MgO–96ZrO_2, ***3*** – 8MgO–92ZrO_2, ***4*** – 16MgO–84ZrO_2

Рис. 3. Рентгенофазовый анализ
Образцы, мол. %: ***1*** – 2MgO–98ZrO_2, ***2*** – 4MgO–96ZrO_2, ***3*** – 8MgO–92ZrO_2, ***4*** – 16MgO–84ZrO_2

To assess the effect of stabilizing additives, XRD analysis was performed (Fig. 3). It was found that zirconium dioxide in the studied samples exists in both the cubic and monoclinic phases, indicating an incomplete stabilization process of zirconium dioxide [10; 21; 22]. According to [23], increasing the sintering temperature should have a positive effect on the stabilization process, which represents a relevant direction for further research.

Mechanical tests showed that the hardness values of the investigated samples varied over a wide range. The highest Martens hardness (8.65 GPa) was recorded for the ceramic with the composition 16MgO–84ZrO_2, fabricated under a pressing pressure of 300 MPa. At this pressure, all samples exhibited their maximum hardness. An increase in hardness was generally observed with increasing pressing pressure; however, for the 4MgO–96ZrO_2 composition, values deviating from this positive trend were identified.

For the MgO–ZrO_2 ceramic composite materials, it was found that at a pressing pressure of 50 MPa, the hardness values of the investigated samples were approximately the same across all compositions, around 5 GPa. As the pressing pressure increased, the hardness also increased; however, a distinct contribution of magnesium oxide to the hardness enhancement became apparent only at pressures above 200 MPa. This effect is attributed to the reduced influence of porosity at a pressing pressure of ≥200 MPa, a linearly increasing dependence of hardness on the magnesium oxide content was observed (Fig. 4), which is associated with changes in the crystallochemical structure. A similar trend of increasing hardness with higher MgO content was also reported in [18]. Although the authors of [18] used higher sintering temperatures (from 1570 to 1970 K), our results demonstrate that even at lower

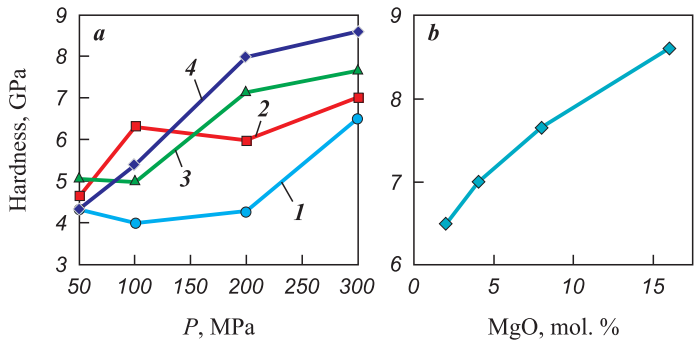

Fig. 4. Dependence of Martens hardness of sintered samples on pressing pressure (***a***) and magnesium oxide content for samples produced at *P* =300 MPa (***b***)
Samples, mol. %: ***1*** – 2MgO–98ZrO_2, ***2*** – 4MgO–96ZrO_2, ***3*** – 8MgO–92ZrO_2, ***4*** – 16MgO–84ZrO_2

Рис. 4. Зависимость твердости по Мартенсу спеченных образцов от давления прессования (***a***) и от содержания оксида магния для образцов, полученных при *P* = 300 МПа (***b***)
Образцы, мол. %: ***1*** – 2MgO–98ZrO_2, ***2*** – 4MgO–96ZrO_2, ***3*** – 8MgO–92ZrO_2, ***4*** – 16MgO–84ZrO_2

sintering temperatures, magnesium oxide retains its positive effect on material hardness, which highlights the potential of this ceramic composite material for further study at reduced sintering temperatures. At the same time, according to [10; 11], the hardness values of the investigated $MgO–ZrO_2–Y_2O_3$ materials exceed those of $MgO–ZrO_2$, $Y_2O_3–ZrO_2$, and MgO ceramics. For example, the Vickers hardness of $MgO–ZrO_2–Y_2O_3$ ceramics can reach 14.8 GPa, compared to the respective values of 10.9, 12.0–12.5, and 10–11 GPa reported for the above-mentioned materials.

During the study of the elastic modulus of the materials, the highest value – 330.3 GPa – was recorded for the ceramic with the composition $16MgO–84ZrO_2$ at a pressing pressure of 300 MPa (Fig. 5). For the $MgO–ZrO_2$ ceramics, the elastic modulus values deviated from the previously observed trend for hardness. For the $4MgO–96ZrO_2$ and $8MgO–92ZrO_2$ samples, a nonlinear dependence of the elastic modulus on the applied pressure was observed; however, this property was nearly identical at both the maximum and minimum pressures. Across all applied pressures, the highest elastic modulus was found for the $16MgO–84ZrO_2$ composition. At a pressing pressure of 50 MPa, it was observed that the elastic modulus increased with increasing magnesium oxide content. However, with further increases in pressing pressure, this dependence broke down and became nonlinear, no longer associated with porosity levels. It was found that at the highest pressing pressure and maximum material density, the ceramics with compositions $16MgO–84ZrO_2$ and $2MgO–98ZrO_2$ exhibited the highest elastic moduli, while the compositions $4MgO–96ZrO_2$ and $8MgO–92ZrO_2$ showed lower values. In other words, a parabolic dependence of the elastic modulus on the magnesium oxide content is formed, with a minimum at 4 mol. % MgO. Study [25] presents the dependence of the elastic modulus on the MgO content in $MgO–ZrO_2$ composites and shows an increase in modulus up to 20 mol. % MgO. At higher MgO contents, the elastic modulus decreases. However, since the increments of magnesium oxide addition in that study were large (about 20 %), they did not provide sufficient resolution to describe the influence of MgO on the elastic modulus in the 0–20 % range. Therefore, the present study is of particular relevance, as it reveals the behavior of the elastic modulus within this critical composition interval.

In this study, the strength parameters of ceramic samples obtained at a pressing pressure of 300 MPa were determined using the scratch test method, along with the critical stress intensity factors (fracture toughness) (Fig. 6). It was found that the lowest strength (467.17 MPa) was exhibited by the sample with the composition $2MgO–98ZrO_2$. An increase in strength was observed with rising magnesium oxide content, reaching up to 791.15 MPa, with this trend following a hyperbolic pattern. According to [25], the behavior of the strength parameter, similar to that of the elastic modulus, reaches a maximum upon the addition of 20 mol. % MgO, followed by a decline when this content is exceeded, assuming the same fixed intervals of magnesium oxide addition.

In the present study, it was found that the fracture toughness (critical stress intensity factor) varies nonlinearly with increasing magnesium oxide content, reaching a maximum value of 10.53 $MPa·m^{1/2}$ with the composition 16 mol. % MgO. Publications [9; 10] report that the introduction of magnesium oxide in various molar fractions increases the fracture toughness of zirconia-based ceramics; however, the dependence is nonlinear. According to [9], the addition of a small

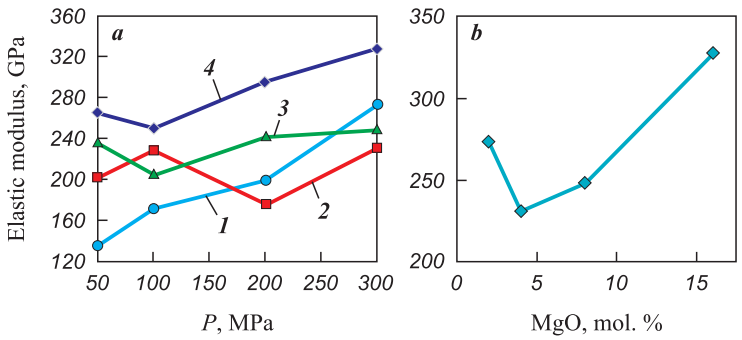

Fig. 5. Dependence of elastic modulus of sintered samples on pressing pressure (***a***) and magnesium oxide content for samples obtained at P = 300 MPa (***b***)
Samples, mol. %: ***1*** – $2MgO–98ZrO_2$, ***2*** – $4MgO–96ZrO_2$, ***3*** – $8MgO–92ZrO_2$, ***4*** – $16MgO–84ZrO_2$

Рис. 5. Зависимость модуля упругости спеченных образцов от давления прессования (***a***) и от содержания оксида магния для образцов, полученных при P = 300 МПа (***b***)
Образцы, мол. %: ***1*** – $2MgO–98ZrO_2$, ***2*** – $4MgO–96ZrO_2$, ***3*** – $8MgO–92ZrO_2$, ***4*** – $16MgO–84ZrO_2$

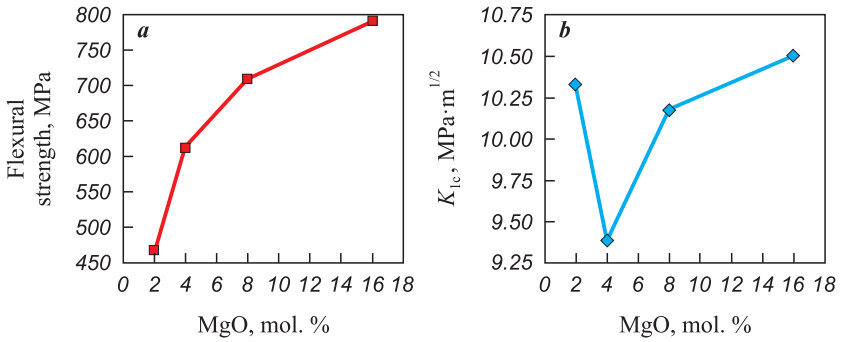

Fig. 6. Dependence of flexural strength (***a***) and fracture toughness (***b***) of sintered samples on the magnesium oxide content

Рис. 6. Зависимость предела прочности на изгиб (***a***) и трещиностойкости (***b***) спеченных образцов от содержания оксида магния

amount of magnesium oxide – 0.5 mol. % MgO – raises the fracture toughness to 9.14 MPa·m$^{1/2}$. In publication [10], zirconia samples stabilized with 8 mol. % MgO were studied. The authors [10] found that increasing the sintering temperature from 1450 to 1500 °C led to an increase in the critical stress intensity factor from 7.59 to 8.5 MPa·m$^{1/2}$. In the present work, raising the sintering temperature to 1700 °C and introducing an additional stabilizing additive – yttrium oxide – resulted in an increase in fracture toughness to 10.14 MPa·m$^{1/2}$ for the sample containing 8 mol. % MgO, which indicates the effectiveness of sintering temperature enhancement for improving this parameter.

Conclusions

1. The conducted study established that the consolidation of MgO–ZrO_2 ceramic powder mixtures with additional stabilization of zirconium dioxide using yttrium oxide enables the formation of a composite structure. It was shown that magnesium does not enter the crystal structure of ZrO_2 and does not form a ZrO_2–MgO-based solid solution due to the stabilizing effect of Y_2O_3.

2. Mechanical activation of the ceramic batches at a rotation frequency of 10 Hz for 10 min resulted in a reduction in the porosity of the sintered materials compared to previously reported data.

3. It was found that increasing the pressing pressure to 300 MPa has a positive effect on the mechanical properties of the materials.

4. Among the investigated samples, the highest Martens hardness (8.65 GPa) was observed for the ceramic with a composition of 16MgO–84ZrO_2 obtained at a pressing pressure of 300 MPa. An increase in the magnesium oxide content has a positive effect on the hardness of the material; however, a significant contribution of MgO to the improvement in hardness is observed only at $P > 200$ MPa, which is associated with a reduced contribution of porosity to the resulting hardness.

5. The sample with a composition of 16MgO–84ZrO_2 obtained at a pressing pressure of 300 MPa exhibited the highest elastic modulus among all tested materials – 330.3 GPa. This composition demonstrated the highest elastic modulus values at all applied pressing pressures. For the samples obtained at $P = 300$ MPa, a parabolic dependence of the elastic modulus on magnesium oxide content was revealed, with a minimum at 4 mol. % MgO.

6. It was found that increasing the magnesium oxide content leads to higher tensile strength, reaching 791.15 MPa at 16 mol. % MgO.

7. It was shown that the dependence of the critical stress intensity factor on magnesium oxide content is nonlinear, with a maximum value of 10.53 MPa·m$^{1/2}$ at 16 mol. % MgO.

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