Structure formation and properties of corundum ceramics based on metastable aluminium oxide doped with stabilized zirconium dioxide

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ABSTRACT

The work presents a successful example of the use of YSZ binary systems for production of composite ceramic materials based on θ-Al₂O₃ with improved physical and mechanical characteristics which was previously considered an unpromising material. It was first obtained result of extreme nature of dependence of physical and mechanical properties of Al₂O₃ + YSZ on the concentration of YSZ (ZrO₂–3mol% of Y₂O₃) additive. The sintering temperature was decreased on 250 °C (from 1800 to 1550 °C). The phase composition of powders and the structure of ceramics of the Al₂O₃ + YSZ system were investigated depending on the amount of YSZ dopant, the structure-properties relationship was established. It is shown that maximum of physical and mechanical characteristics achieved at YSZ concentration equal to 10% wt.

1. Introduction

Ceramic materials take a leading place among materials used in many spheres of technology, industry and medicine. Ceramics based on aluminum oxides have become the most widespread among all ceramic materials. This material is used in construction and industry as heat insulation material that is resistant to corrosive media and does not release harmful substances during operation [1,2]. Corundum ceramics are widely used in mechanical engineering for the manufacturing of refractory and high-temperature parts of machines, and it is used in medicine as a material for creating dental implants [3–5].

Corundum ceramics has become widespread due to such physical and mechanical properties as high strength, hardness, wear resistance, refractory resistance, thermal conductivity, chemical resistance and high electrical strength. Sintering temperature of pure aluminum α-oxide powders is 1700–1800 °C, and in the presence of eutectic additives – 1550–1650 °C [6,7]. Relatively high sintering temperatures and polymorphism (there are eight metastable modifications of aluminum oxide) are a certain scientific and technological problems that initiate an interest in optimizing the producing technology of aluminum oxide ceramics while simultaneously increase in the level of their physical properties.

There are a number of independent hypotheses describing the causes of the appearance and nature of metastable polymorphic modifications of Al₂O₃ existing in the temperature range up to 1200 °C. According to Refs. [8,9], the primary reason for the formation of metastable phases is the presence of impurity ions: OH⁻, SO₄²⁻, CO₃²⁻, etc. in the structure of the material, the removal of which during thermal treatment “destabilizes” the metastable state.

In works [10,11,13,14] the influence on the process of emergence...
and existence of metastable phases of particle size (dimensional factor) was suggested. This assumption is supported by the fact of obtaining metastable phases when crushing large powder materials, as well as the existence of metastable phases in thin films [9].

However, the energy theory [10,11] does not describe enough the conditions for the formation of metastable phases. In particular [12], suggests a predominant role in the mechanism of the emergence of metastable phases of martensitic transition and micro domains. Scheme of phase transformation in the aluminum-oxygen system Al₂O₃ is shown in Fig. 1.

The aluminum oxide system has two stable crystalline states: Al(OH)₃ and α-Al₂O₃ (Fig. 1) and a number of metastable intermediate states formed during thermal evolution. The transition of crystalline gibbsite, nordstrandite and bayerite to aluminum oxide can be carried out in two ways:

1. Metastable states are: γ- and α-Al₂O₃, and η + γ-Al₂O₃.
2. An intermediate – boehmite (γ-AlO(OH), orthorhombic modification) and its metastable states are: γ, δ- and θ-Al₂O₃.

Structural water plays an essential role as a structure-forming factor. Sufficiently large sizes of aluminum hydroxide crystals are explained by the presence in the structure of water in the form of coordinated hydro groups OH⁻. Thermal treatment leads to dihydroxylation of Al(OH)₃, reduction of crystal size and formation of intermediate metastable phases of aluminum oxide [15].

Fig. 2 shows bar diagram of particle sizes (coherent scattering regions – CSR), in the process of thermal evolution. According to the X-ray structural analysis, the CSR value in the hydroxide Al(OH)₃ is \( d = 23 \) nm, in the γ-Al₂O₃: \( d = 8 \) nm, in the θ-Al₂O₃: \( d = 12 \) nm, and in the stable α-Al₂O₃: \( d = 55 \) nm respectively.

One of the ways to increase the competitiveness of corundum ceramics is to obtain binary and more complex systems of ceramic oxides from those previously considered unpromising.

Due to the polymorphism, low density and high porosity [16] in comparison to the thermodynamically stable α-Al₂O₃ phase [17,18], the θ-Al₂O₃ modification has been poorly studied as a raw material for the corundum ceramics production. Solid solutions based on YSZ due to the lower sintering temperature and higher density are of interest as an doping for Al₂O₃ systems. The phase diagram of the YSZ – Al₂O₃ system is not comprehensively studied at high concentrations of Al₂O₃, however, the existing experimental background of the authors in the study of zirconium solid solution of the YSZ system with an admixture of Al₂O₃ [19,20] suggests that the addition of YSZ in θ-Al₂O₃ will reduce the sintering temperature and increase the density of corundum ceramics.

The main goal of the present research is confirmation of this assumption experimentally by examining corundum ceramics based on the metastable θ-Al₂O₃ oxide doped with stabilized tetragonal zirconium dioxide (YSZ) additives structure and its physical and mechanical properties.

2. Materials and research methods

The powders for the study were prepared by chemical co-precipitation from a solution of aluminum chloride (AlCl₃ • 6H₂O), zirconium chloride (ZrOCl₂ • 8H₂O), and yttrium nitrate salts (Y(NO₃)₃) at room temperature. To obtain a homogeneous product, the method of reverse chemical precipitation was used: chemical mixing of salts was carried out before their introduction into the precipitator. The co-precipitation method is a more laborious process than mechanical mixing, but the degree of homogenization of the co-precipitated masses is incomparably higher than the degree of homogenization of the mixture obtained mechanically. The co-precipitation method provides a greater value of the dispersion and homogeneity of the nanopowder system, which plays an important role in obtaining ceramics with high physical and mechanical properties [21]. Crystallization and formation of powder particles were carried out in air by calcining hydroxides at temperatures of 1000, 1200 and 1300 °C for 2 h. The resulting oxides had the structure θ-Al₂O₃ + nYSZ, where \( n = 0, 1, 5, 10, 15 \) wt%.

The powders were compacted in steel molds by uniaxial pressing at a pressure of 20 MPa, followed by treatment of compacts with high hydrostatic pressure (400 MPa). Three series of samples – compacts in the form of beams with dimensions 4x4x40 mm were sintered in air at 1000, 1200 and 1300 °C for 2 h. The resulting oxides had the structure θ-Al₂O₃ + nYSZ, where \( n = 0, 1, 5, 10, 15 \) wt%.

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temperatures, respectively, 1450 (series 1), 1500 (series 2) and 1550 °C (series 3) for 2h. Polymorphic modifications of aluminum oxide have been little studied in as a raw material for the production of corundum ceramics, and the choice of the sintering temperature with a step of 50 °C will allow a detailed study of this system.

The analysis of the phase composition of powders and ceramics was carried out on an Empyrean diffractometer (PANalytical), in filtered copper radiation. Density was measured by hydrostatic weighing on a TP-7R-1 installation with a load of 5 kg, with a step of 2 kg in three times repetition. Strength was examined by 4-point bending on the T-Series Materials Testing Machine H50K-T (Tinius Olsen) five-fold repetition. The surface microstructure was examined on a Jeol scanning electron microscope JSM-6490LV.

3. Results and discussion

3.1. Powder systems

Phase composition of the Al₂O₃ powders and powder mixture Al₂O₃ + nYSZ annealed at a temperature 1000 °C and containing min (1%) and max (15%) of concentration of YSZ are given in Fig. 3.

X-ray structural analysis method shows that all powder composites of Al₂O₃ + nYSZ (n = 1, 5, 10, 15 wt%), after annealing at a temperature 1000 °C for 2h have three-phase structure: cubic modification γ-Al₂O₃, monoclinic modification θ-Al₂O₃ and tetragonal ZrO₂.

The presence of θ and γ phases Al₂O₃ is explained by the fact that during thermal treatment of Al(OH)₃, which is a mixture of bayerite (β-Al(OH)₃) and nordstrandite (γ-Al(OH)₃), the crystal lattices transform through two intermediate states. In the range 200–250 °C from Al(OH)₃, 0,5 amount of water molecules are removed and boehmite is partially formed, and with complete dehydration corresponding to the temperature range 300–500 °C, a low-temperature cubic γ-Al₂O₃ is formed (way 1).

In the range of 250–350 °C, 1,5 molecules of H₂O are removed from Al(OH)₃ to form the Al₂O₃-phase and 1 molecule of H₂O, and at complete dehydration corresponding to the temperature of 350–500 °C, a monoclinic θ-Al₂O₃ is formed (path 2). The presence of peaks of tetragonal zirconium dioxide even at its minimum amount (1%) is explained by its strong scattering ability, which is directly proportional to Z₄, where Z is an ordinal number in the periodic table of elements [22, 23].

Phase composition of co-precipitated powder mixtures of Al₂O₃ + nYSZ composition, where n = 0, 5, 15 wt%, obtained at temperature 1200 °C are given in Fig. 4.

It can be seen from the x-ray diffraction patterns that for Al₂O₃ + 0% YSZ powder the phase transition to stable α-Al₂O₃ phase occurs at 1200 °C, and for powder Al₂O₃ + nYSZ (n = 5, 15 wt%) the phase transition to stable α-Al₂O₃ state does not occur at the same temperature (Fig. 4).

Powder mixtures of the composition Al₂O₃ + 5% YSZ and Al₂O₃ + 15% YSZ annealed at 1200 °C and 1300 °C (Fig. 5) were investigated to determine the temperature range of the phase transition from the metastable of θ-Al₂O₃ to stable α-Al₂O₃ state.

Thus, based on the results of XRD analysis we can conclude that the addition of zirconium dioxide to aluminum oxide powders impedes the phase conversion process and increases the transition temperature of alumina powders from metastable to stable α-Al₂O₃. Phase transition of aluminum oxide to a stable state for the system Al₂O₃ + nYSZ, n = 1, 5, 10, 15% corresponds to the temperature 1300 °C, and in Al₂O₃ + 0%YSZ system the phase transition occurred at a temperature 1200 °C. The reason for the delay in the phase transformation is the effect of mutual protection against crystallization, which is characteristic of powder mixtures obtained by the method of chemical deposition: two co-precipitated substances heated to a certain temperature can remain amorphous, while each of them separately, is heated to the same temperature, completely crystallizes [24].

3.2. Ceramics

Based on powder mixtures annealed at a temperature 1000 °C, three sets of ceramic samples were prepared at different sintering temperatures. Fig. 6 shows XRD patterns of ceramic materials of following composition Al₂O₃ + 10% YSZ, obtained at 1450, 1500 and 1550 °C. As can be seen typical ceramic features of the system Al₂O₃ + nYSZ, where n = 1, 5, 10, 15 wt% are observed. Thus, is a composite system including matrix α-Al₂O₃ (corundum) with YSZ filler.

Physical and mechanical characteristics of aluminum-zirconium ceramics.

Fig. 7 shows the dependence of the density of aluminum-zirconium ceramics based on powders annealed at 1000 °C on the concentration of YSZ and sintering temperature.

The range of density change of obtained ceramics depending on concentration of the stabilized zirconium dioxide and agglomeration temperature varied from 3.42 to 4.04 g/cm³). The theoretical density of
the aluminum-zirconium composite for the Al2O3 + 10% Z3Y system is 4.19 g/cm³. The maximum density value is achieved at a sintering temperature of 1550 °C for 2 h and a zirconium dioxide concentration n = 10 wt%, which is 96.3% of the theoretical density of the aluminum-zirconium composite (Fig. 7). Comparison of physical parameters with similar control batches of two-phase powder based on α-Al2O3 + nYSZ where n = 0, 1, 5, 10, 15 wt% received under the same conditions (\(\rho_{\text{max}} = 2.05 \div 2.84 \text{ g/cm}^3\), porosity \(\varsigma = 35 \div 50\%\)) testifies the advantage to use metastable phases of aluminum oxide to obtain dense corundum ceramics at agglomeration temperatures of ~1450–1550 °C.

Variation of hardness of the analyzed ceramic samples on zirconium dioxide concentration and sintering temperatures is given in Fig. 8. Depending on concentration of zirconium dioxide the strength of the studied ceramics varied in the range from \(A = 240 \div 315 \text{ MPa}\). The maximum strength value corresponds to composites obtained at 1550 °C, containing 10 and 15% of YSZ and samples obtained at 1500 °C, containing 5% of YSZ. As can be seen from Fig. 10, if the concentration of YSZ increases up to 5%, the strength of the ceramic samples increases significantly. Further increase of the doping element concentration, leads to the strength saturation and practically does not change.

3.3. Microstructure

Fig. 10 shows the surface structure of the samples of compositions: a – Al2O3 5% YSZ and b – Al2O3 10% YSZ obtained in air at a temperature 1550 °C for 2h. SEM data represent the presence of zirconium dioxide at the grain boundaries of aluminum oxide (Fig. 10). Probably, at the stage of ceramic sintering, zirconium dioxide is distributed along the boundaries of aluminium grains, which led to the formation of a composite matrix with inclusions of zirconium dioxide in the intergranular space.
Based on the behavior of the physical characteristics of the studied ceramics (Figs. 7–9), with low additions of the doping element (n = 1–10%), zirconium dioxide uniformly fills the internal grain space, forming a damping interlayer, leading to increase in the resistance of the composite ceramics to mechanical effects, increased density and reduced porosity. Here, a special property of zirconium ceramics is used - the effect of super-plasticity, due to this effect it is possible to obtain the so-called “ceramic steels” [25,26]. With a zirconium dioxide concentration of n ≥ 10% agglomeration occurs (aggregate size up to 1,5 μm) which leads to a softening of the ceramic structure.

Thus, in aluminum oxide ceramics the average grain size of 3,5–4 μm and when an YSZ is added, the average grain size is halved. At the same time, the effective grain size of YSZ increases with its concentration in the aluminum dioxide matrix. In ceramics of the composition Al₂O₃ 5% YSZ, the average grain size of zirconium dioxide is 0,2–0,3 μm, and at a zirconium dioxide concentration of 10%, the average grain size is 0,5–0,75 μm (Fig. 10).

The above data show that when YSZ of 10 wt% is added to aluminum dioxide ceramic, the diffuseness of the ceramic composite material decreases and, as a result, its physical and mechanical properties are improved.

Fig. 11 shows the image of a surface of ceramics of structure Al₂O₃ + 10%YSZ at increase is submitted x2500.

When YSZ concentration increases to 10 wt%, a small amount of zirconium dioxide grain agglomerates in the material is forming (Fig. 11). Based on the physic-mechanical properties of ceramics (Figs. 7–9) and SEM data, it can be concluded that in Al₂O₃ + 10% YSZ ceramics, the maximum physical and mechanical properties are achieved, since this concentration of zirconia is optimal for the uniform distribution of YSZ over the entire volume of the alumina matrix, and a small amount of the formed agglomerates is insufficient to deteriorate the properties of the composite.

In alumocyrconium ceramics doped with 15% YSZ, a deterioration in physical and mechanical characteristics is observed. Fig. 12 shows the surface structure of ceramics of the composition Al₂O₃ + 15%YSZ.

When the concentration of the YSZ increases to 15%, a large number of agglomerates concentrated in the inter-grain space are formed which lead to softening of the material and, as a result, to deterioration of the physical and mechanical characteristics of the studied ceramics.

Samples containing 5% YSZ were examined for the effect of sintering temperature on the structure of composite ceramics. The effect of sintering temperature on the structure of composite ceramics of the

![Fig. 9](image)

**Fig. 9.** Dependence of strength of investigated ceramics obtained at different sintering temperatures on zirconium dioxide concentration: 1450 °C (curve 1), 1500 °C (curve 2), 1550 °C (curve 3).

![Fig. 10](image)

**Fig. 10.** SEM-images of surface of samples Al₂O₃ + 5%YSZ (a), Al₂O₃ + 10% YSZ (b); Filling the grain space by zirconium dioxide prevents the growth of aluminum oxide grains. This effect is evidenced by the average effective grain size of aluminum oxide. For Al₂O₃ 0% YSZ, the effective grain size is 3,5–4 μm [27], with an increase in the amount of zirconium dioxide to 5%, the effective grain size of aluminum oxide decreases to 0,8–2 μm, and at a concentration of YSZ of 10%, the effective grain size is from 1 to 1,8 μm. At YSZ concentration increase to 15% average grain size does not decrease and remains within 0,8–1,6 μm.

![Fig. 11](image)

**Fig. 11.** SEM-image of surface of sample Al₂O₃ + 10%YSZ.
composition Al$_2$O$_3$ 5%3 is shown in Fig. 13.

At sintering temperature of 1500°C there is observed a large number of surface pores of different size and shape (Fig. 13, a). The ceramic density of the composition Al$_2$O$_3$ 5% YSZ obtained at a sintering temperature of 1500°C is 3.78 g/cm$^3$. At a sintering temperature of 1550°C, a significant decrease in the number of surface pores (Fig. 13, b) is observed. The density of the composite ceramic obtained at the sintering temperature of 1550°C is 3.90 g/cm$^3$.

These data indicate that the sintering temperature plays an important role in the production of dense and non-porous aluminum oxide-based ceramics. The optimum sintering temperature of the metastable 0-aluminum oxide based composite ceramics is 1550°C.

4. Conclusions

1. Composite powder Al$_2$O$_3$ + nYSZ (n = 1, 5, 10, 15 wt%) based on metastable 0-Al$_2$O$_3$ is obtained by co-precipitation method. Obtained raw material is a binary composite material of the Al$_2$O$_3$ – YSZ system.

2. The evolution of the alumina system depending on the concentration of YSZ in the high temperature range (1000–1300°C) has been studied. Based on the analysis of the phase composition of the Al$_2$O$_3$ + YSZ powder system was recorded the fact of delay of the phase transition from 0-Al$_2$O$_3$ to α-Al$_2$O$_3$ in the presence of the YSZ phase.

3. Reduction of sintering temperature by 250°C in dense composite ceramics achieved due to use of reactive metastable phase 0-Al$_2$O$_3$.

4. The composition of ceramics based on powder of metastable 0-Al$_2$O$_3$ + 10 wt% YSZ (1000°C annealing) demonstrated max physical and mechanical characteristics (ρ = 4.04 g/cm$^3$, Hv = 19.5 GPa, σ = 315 MPa) after sintering at 1550°C.

5. The SEM analysis results showed the distribution of the YSZ phase along the grain boundaries of α-Al$_2$O$_3$ which is typical for aggregate-hardened structure of the ceramic composites.

6. The fact of the deterioration of physical and mechanical properties of alumina-zirconia ceramics of the composition Al$_2$O$_3$ + 15% YSZ caused by the formation of a large number of weakly bounded agglomerates of YSZ grains has been established.

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Fig. 12. SEM-image of surface of sample Al$_2$O$_3$ + 15%YSZ.

Fig. 13. SEM-images of surface of samples Al$_2$O$_3$ + 5%YSZ (1500°C) (a), Al$_2$O$_3$ + 5%YSZ (1550°C) (b).

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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