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## EFFECTS OF THE YTTRIA CONTENT AND SINTERING TEMPERATURE ON THE PHASE EVOLUTION IN YTTRIA-STABILIZED ZIRCONIA

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**Abstract.** The microstructure of YSZ ceramics stabilized by the various amount of yttria, namely 3 mol %  $Y_2O_3$  (3YSZ), 4 mol%  $Y_2O_3$  (4YSZ) and 5 mol %  $Y_2O_3$  (5YSZ) has been studied. Three sintering temperatures, namely 1450 °C, 1500 °C and 1550 °C were used for each series of samples (3YSZ, 4YSZ, 5YSZ). The total area of the monoclinic and cubic zirconia phases in the microstructure of ceramics and the regularities of distribution of these phases were determined by ImageJ. Peculiarities of changes in volume percentage of the monoclinic and cubic phases with an increase in sintering temperature of ceramics were found. Quantitative analysis of these phases was carried out. The total distribution of the monoclinic and cubic phases by ranges of their areas was presented. Correlations between the yttria content, the sintering temperature and changes in the microstructure and phase balance of the studied ceramics were found.

**Keywords:** YSZ ceramics, microstructure, agglomerates of (cubic+monoclinic)-phases, sintering temperature.

### Introduction and problem statement

Due to the unique combination of properties, zirconia ceramics are widely used in mechanical engineering, electronics, energy, chemistry, medicine, etc. These ceramics are used in gas turbine engines (thermal barrier coating of turbine blades, coating of the inner surface of the boiler chamber walls, coating of the gas turbine combustion chamber) [1–3], for heat-resistant lining of furnaces [4, 5], for fuel cells [6–8], electrodes [9], gas sensors [10], as a protective coating for alloys [11, 12], as a material for dental implants, dental prostheses, artificial bone endoprostheses [13–18]. Widespread use of zirconia ceramics is due to their low thermal conductivity, high strength, wear resistance, high fracture toughness, high corrosion resistance, biological inertness, and high biocompatibility. Properties of zirconia ceramics are determined by their microstructure and phase composition.

The main structural component of zirconia ceramic is zirconium dioxide. According to the phase diagram, zirconium dioxide exists in three crystallographic phases: monoclinic, tetragonal and cubic. There is a monoclinic phase in the structure of zirconia ceramic at 20 °C and atmospheric pressure. The monoclinic phase exists below 1127 °C, tetragonal between 1127 °C and 2370 °C, and cubic above 2370 °C [1]. The monoclinic phase is stable and the tetragonal phase is metastable, due to the phase transition of the tetragonal phase to the monoclinic phase and an increase in volume by 3–5 %. Doping of zirconia ceramic causes changes in the phase transitions temperature, which leads to stabilization at 20 °C not only monoclinic, but also tetragonal and cubic phases. Tetragonal and cubic phases stabilization in the structure of zirconia ceramic was carried out by its doping with oxides of yttrium [1, 19–23], aluminum [24], calcium [25], magnesium [5, 26], cerium [27–30], and titanium [2].

Doping zirconia ceramic allows to obtain and to stabilize the tetragonal phase at room temperature, the presence of which provides the unique properties of this ceramic. Doping additives, namely, their nature and concentration, as well as the grain size significantly affect the microstructure of ceramic and determine its stability [19].

Zirconia ceramic stabilized by 3 mol%  $Y_2O_3$  characterized by high strength (about 700 MPa) and fracture toughness ( $K_c = 6-9 \text{ MN}\cdot\text{m}^{-3/2}$ ) due to the presence in its structure of fine-grained metastable tetragonal phase [20]. The reason for the high fracture toughness of this material was the martensitic transformation of the tetragonal phase grains into monoclinic (t→m) during deformation. This transformation accompanied by the absorption of deformation energy. In the structure of zirconia ceramic stabilized by 3 mol %  $Y_2O_3$  there were three phases, namely tetragonal, monoclinic and cubic [22]. The relatively low strength and fracture toughness were due to the high content of the monoclinic phase (37.8 %). The disadvantage of this ceramic was the phase transformation (t→m), which accompanied by an expansion of the volume to 5 % and caused the formation of cracks during cooling.

Properties degradation of zirconia ceramic stabilized by 3 mol %  $Y_2O_3$  under aging in water vapor conditions occurred due to the transformation of the tetragonal phase into monoclinic one [21]. It was concluded that the optimal sintering temperature for this ceramic is 1550 °C, at which the maximum microhardness (11.135 GPa) and high relative density (96.64 %) were achieved [23].

Doping additives and the grain size influenced the stability of the tetragonal phase under conditions of low-temperature degradation. The grain size of zirconia ceramic stabilized by 3 mol %  $Y_2O_3$  after sintering at temperatures of 1350 °C and 1400 °C was less than 3  $\mu\text{m}$ . Aluminum oxide and cerium oxide stabilized the grain size and increased the tetragonal phase stability. Therefore, no tetragonal to monoclinic phase transformation under aging in water vapor conditions occurred [19].

Zirconia ceramics microstructure and properties largely depend on the sintering temperature [19]. Therefore, ensuring the choice of optimal sintering mode of ceramics will allow obtaining a material with improved microstructure and, accordingly, properties.

Additional  $TiO_2$  doping of zirconia ceramic stabilized by 7.6 %  $\pm 1$  %  $YO_{1.5}$  significantly increased toughness by increasing the tetragonal phase stability [2]. This ceramic can be a promising material for thermal barrier coatings in gas turbine engines, because such growth of the toughness and phase stability significantly influence its strain tolerance, cyclic durability and erosion resistance. The microstructure and phase composition of zirconia ceramics are decisive in the formation of these properties.

Zirconia ceramic stabilized by 3 mol %  $Y_2O_3$  was further doped with  $Al_2O_3$  in order to increase the corrosion resistance in a humid environment at 150–250 °C. Even a small content of alloying additive (0.25 wt %  $Al_2O_3$ ) increased the resistance to degradation of the ceramic surface, especially under water or water vapor conditions [24].

Zirconia ceramic doped by 8–10 mol % MgO contains tetragonal (t- $ZrO_2$ ), monoclinic (m- $ZrO_2$ ) and cubic phases (c- $ZrO_2$ ). This ceramic is characterized by high fracture toughness (8-15  $\text{MPa}\cdot\text{m}^{1/2}$ ), high bending strength (700 MPa) and high thermal shock resistance [26].

When co-alloying zirconia ceramic by 2.5 wt %  $Y_2O_3$  and 25 wt %  $CeO_2$ , the cubic phase was observed in the structure in addition to the fully stabilized tetragonal phase. The presence of the cubic phase was explained by additional doping with cerium oxide [29]. Due to this phase composition, the thermophysical characteristics of such ceramic significantly improved, in particular high thermal shock resistance.

Doping and the optimal sintering modes of zirconia ceramic are determining factors for obtaining the desired structure and phase composition and, thus, improving the properties of such ceramic.

### **Main Material Presentation**

$ZrO_2$  ceramics partially stabilized by 3, 4 and 5 mol %  $Y_2O_3$  (3YSZ, 4YSZ, 5YSZ) have been studied.  $ZrO_2$  powders with initial particle sizes of 100–150 nm and  $Y_2O_3$  powders with initial particle sizes of 10–30 nm were used as raw materials for the production of such ceramics. The powders were mixed in appropriate proportions in a drum mill with the addition of isopropyl alcohol. From the resulting mixture of  $ZrO_2$  and  $Y_2O_3$  powders, a suspension by adding ethyl cellulose (polymer bond), dibutyl phthalate (plasticizer), isopropyl alcohol (solvent) was made. Then, until cessation of weight loss, the powder mixture was dried in an oven at 120 °C to remove residual liquid. The procedure of calcination of powders at 700 °C to obtain a specific distribution of particles size and phase composition was performed. To increase the density of the resulting mixture of powders, 5 % wt % polyvinyl butyral (5 % alcohol solution) was added [31–33]. Prismatic samples with a size of 4×4×50 mm were formed by the method of

bilateral densification in metal form under a pressure of 50 MPa. The obtained prismatic samples were sintered in an electric resistance furnace for 2 h in argon, using conventional sintering techniques. Three sintering temperatures, namely 1450 °C, 1500 °C and 1550 °C were used for each series of samples (3YSZ, 4YSZ, 5YSZ) [21].

The microstructure was examined using an optical microscope MICROTECH MMT-14C at magnifications of 100 and 400 times. To conduct microstructural studies, the surface of the samples was ground, polished and etched in hydrofluoric acid for 15 min.

Analysis of microstructure was performed using specialized ImageJ software. The software was used to scale the image, manipulate the contrast of the image, sharpen, smooth, and detect boundaries. The total area of monoclinic and cubic phases in the microstructure and the area of the microstructural (c+m)-phase agglomerates have been determined by ImageJ. Firstly, we have set the spatial scale of the photo, selected the working area and analyzed the working area according to the set parameter, i.e. cross-sectional area. When using the function “Threshold”, microstructural components displayed in dark gray and the background displayed in white. This function set the lower and upper thresholds to segment the desired area and image background. Using the function “Set Measurements”, it was selected the parameters (data) to be analyzed by the software, particularly the cross-sectional area of the particles. Then, using the function “Analyze particles”, the required parameters were selected, namely

- in the field “Circularity”, the range from 0 (straight line) to 1.00 (circle) was set, in order to the software considered particles of any shape;
- the options were set for displaying the results (Display results) and summing the cross-sectional areas of all particles (Summarise).

As a result of the analysis by ImageJ software, the values of the total area of analyzed particles, the cross-sectional area of each particle, and the number of particles in the set ranges of their areas (0–20, 20–40, 40–60, 60–80, 80–100, 100–120, 120–140, 140–160, 160–180, 180–200, 200–220, 120–140, and 240–260  $\mu\text{m}^2$ ) were obtained.

## Results and Discussion

Studies of the microstructure of 3YSZ, 4YSZ, and 5YSZ ceramics [21] have revealed the presence of the tetragonal, monoclinic and cubic phases in them. Monoclinic and cubic phases were of dark-gray color; therefore, they were easily identified in the images (Fig. 1). The tetragonal phase was of light-gray color; therefore, it was not identified in the images.

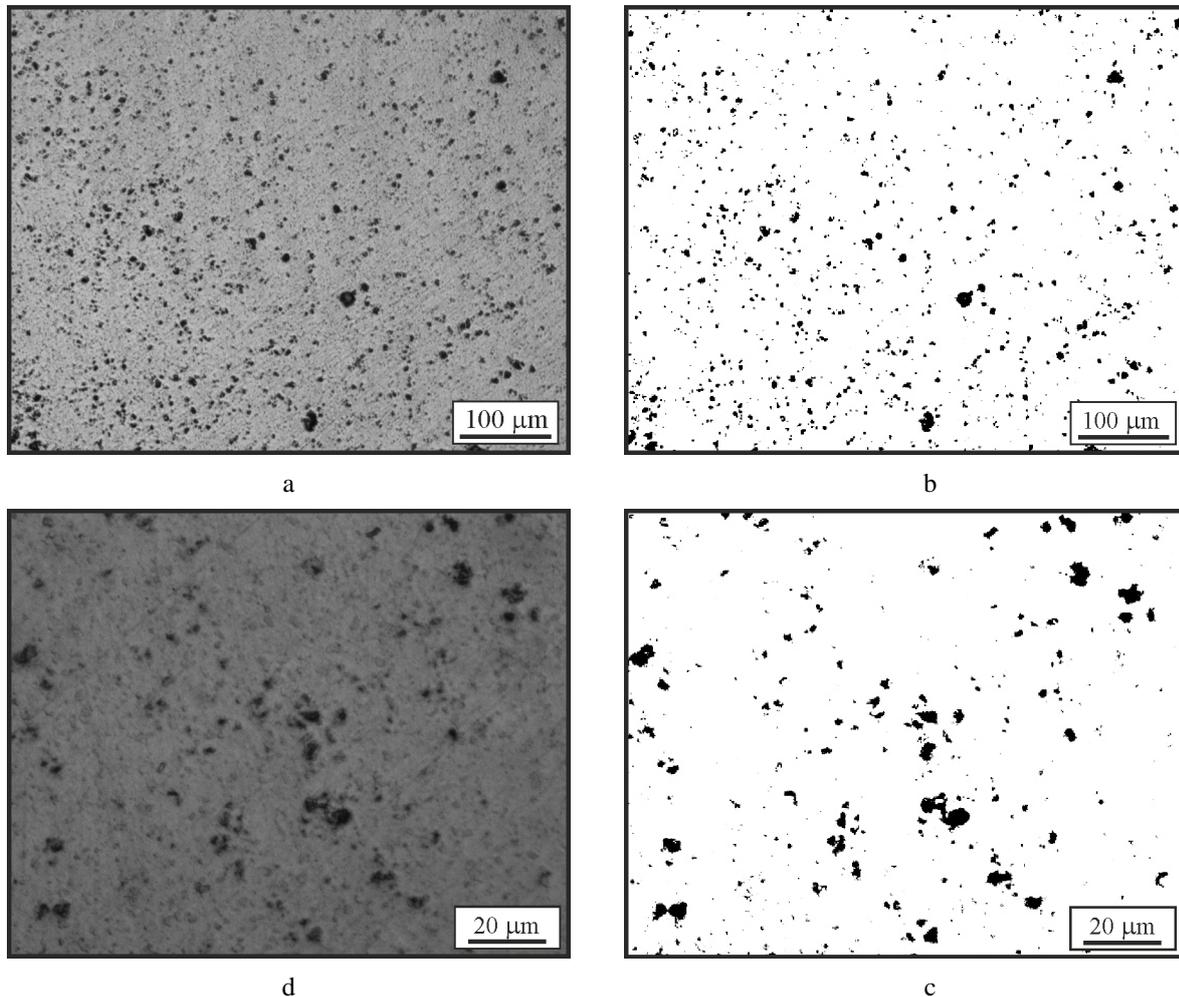
The measurement results are presented in graphs (Fig. 2), which exhibit the evolution of volume percentage (vol %) of the monoclinic and cubic phases in total for studied materials depending on the sintering temperature. The main peculiarities of the volume percentage changes of monoclinic and cubic phases with increase in sintering temperature of ceramics are clearly demonstrated at a high magnification (x400, Fig. 2, *a*), whereas quantitative analysis is better demonstrated at a low magnification (x100, Fig. 2, *b*).

It was found that with a more intensive increase of the monoclinic phase weight percentage (wt %), the volume percentage (vol %) growth of the monoclinic and cubic phases in total is suppressed. Conversely, when an increase in the monoclinic phase weight percentage decelerates, this process is intensified (Fig. 2, *a* and Fig. 3).

Besides, the evolution of volume percentage of the monoclinic and cubic phases depends on: 1) the yttria content (an increase in the volume percentage of monoclinic and cubic phases is intensified due to the yttria content growth); 2) the sintering temperature (an increase in the volume percentage of monoclinic and cubic phases is suppressed due to the sintering temperature growth). This is observed from the correlation between the (c+m)-phase weight percentage and the volume percentage of the monoclinic and cubic phases in total (Fig. 4).

For each material series (3YSZ, 4YSZ, and 5YSZ), the point in Fig. 4 was chosen that corresponds to the highest fracture toughness [21]. Then, mathematical function, that has the best fit to this point series, was obtained (dashed green line in Fig. 4) using Microsoft Excel curve fitting tools. The corresponding equation is as follows:  $[(c+m)(\text{vol } \%)] = 8 \cdot 10^{-9} [(c+m)(\text{wt } \%)]^{5.9717}$ . Thus, the volume percentage of (c+m)-phases must change according to this nonlinear law in order to achieve the maximum fracture toughness of

the studied ceramic compositions. Positions of the points corresponding to the fitted curve are shown in Fig. 4: 3YSZ–1550, 4YSZ–1500, and 5YSZ–1450.

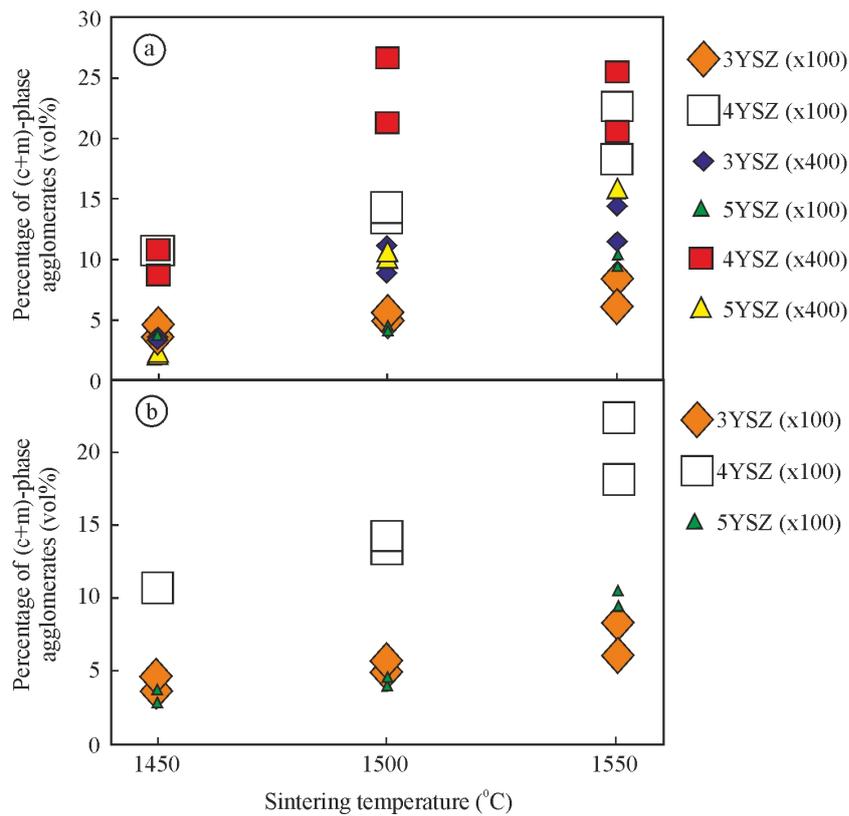


**Fig. 1.** Optical microstructure of 3YSZ ceramics sintered at 1450 °C (a, c) and after processing by the software ImageJ (b, d): magnification x100 (a, b); magnification x400 (c, d).

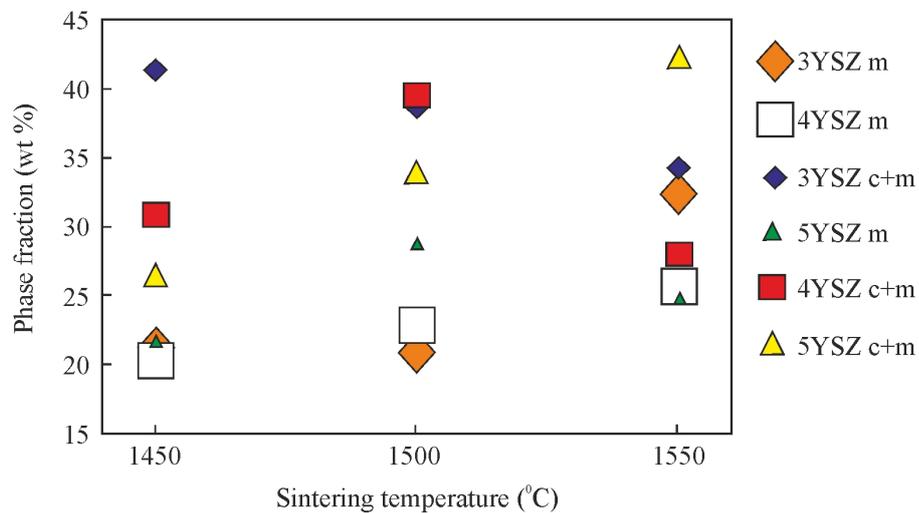
The distribution of the measured areas of the monoclinic and cubic phases (in total) by the ranges of their areas at magnifications x100 (Fig. 5, a) and x400 (Fig. 5, b) was also presented. It was found that the materials of 4YSZ series are characterized by the maximum number of (c+m)-phase agglomerates in the middle ranges of their areas (0–20, 20–40, 40–60, 60–80, 80–100 and 100–120  $\mu\text{m}^2$ ) as compared to other material variants.

This difference is especially noticeable for variant 4YSZ–1550, which correlates with the atypical ratio between the weight percentages of the monoclinic phase and the total weight percentages of the monoclinic and cubic phases as compared to other material variants (Fig. 3). In particular, at magnification x100 (Fig. 5, a), the following numbers of the (c+m)-phase agglomerates were recorded: 12253 agglomerates corresponding the range of area distribution of 0–20  $\mu\text{m}^2$ , 344 agglomerates for the range 20–40  $\mu\text{m}^2$ , 94 agglomerates for the range 40–60  $\mu\text{m}^2$ , 45 agglomerates for the range 60–80  $\mu\text{m}^2$ , 29 agglomerates for the range 80–100  $\mu\text{m}^2$ , and 12 agglomerates for the range 100–120  $\mu\text{m}^2$ . At magnification x400 (Fig. 5, b), a slightly different quantitative distribution was recorded, namely, 273328 agglomerates for the range 0–20  $\mu\text{m}^2$ , 432 agglomerates for the range 20–40  $\mu\text{m}^2$ , 64 agglomerates for the range 40–60  $\mu\text{m}^2$ , and 16 agglomerates for the range 60–80  $\mu\text{m}^2$ .

For this variant, the numbers of (c+m)-phase agglomerates are also consistent with the atypical deviation of the dependence between the weight percentage of (c+m)-phases and the volume percentage of monoclinic and cubic phases in total from the general tendency found for other studied material variants (Fig. 4).

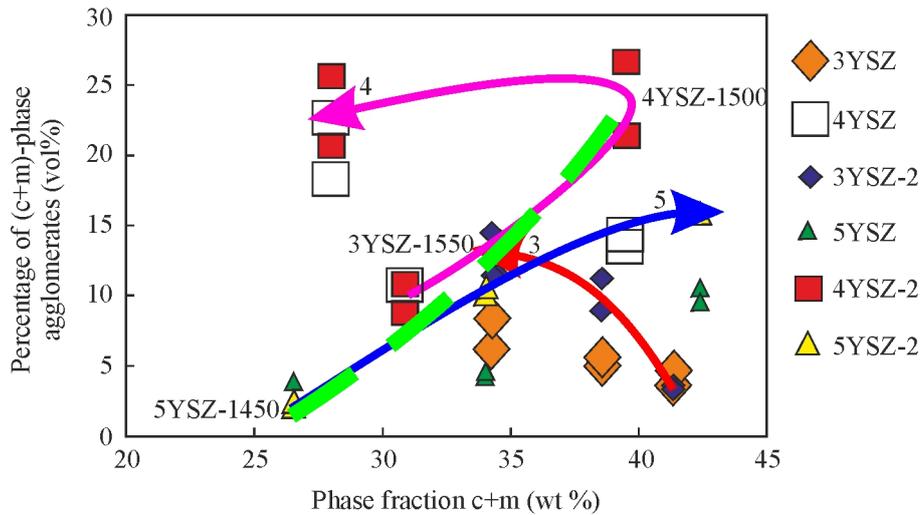


**Fig. 2.** Dependences of volume percentage of (c+m)-phase agglomerates on sintering temperature for studied materials. The number of agglomerates of (c+m)-phases was obtained after processing in the ImageJ software at magnifications x100 and x400 (a) and x100 (b)

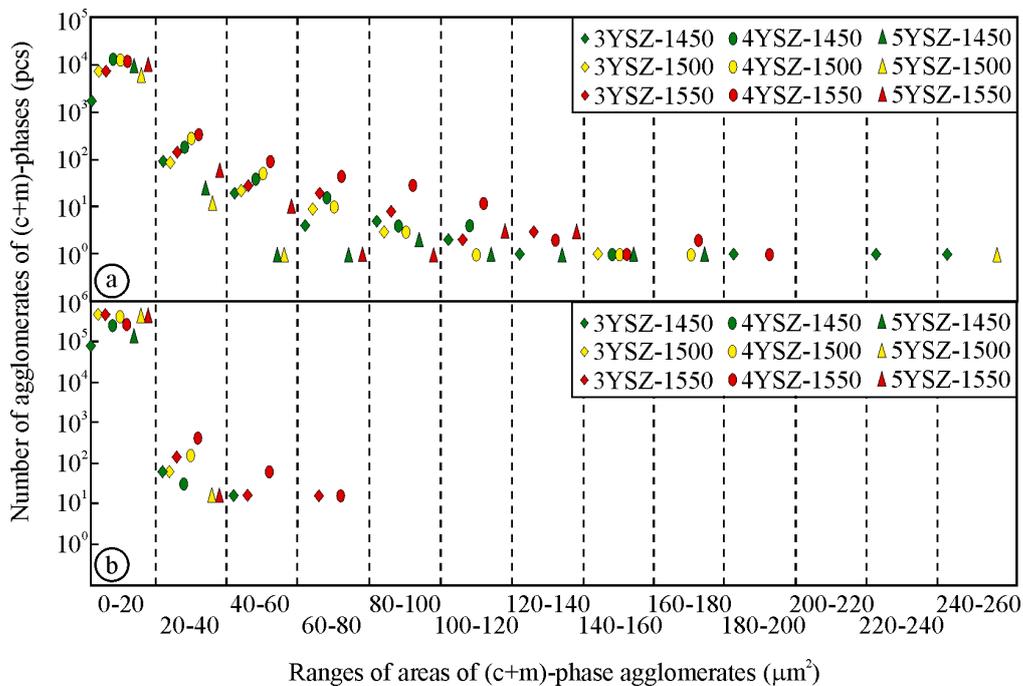


**Fig. 3.** Dependences of the monoclinic phase weight percentage and weight percentage of (c+m)-phase agglomerates on sintering temperature for studied materials.

The lowest number of (c+m)-phase agglomerates in the middle ranges of their areas (20–40, 40–60, 60–80 and 80–100  $\mu\text{m}^2$ ) was found for the 5YSZ series, namely, variants 5YSZ–1450 and 5YSZ–1500 (Fig. 5, a, Fig. 5, b). This characteristic area distribution of the monoclinic and cubic phases is consistent with the high fracture toughness of variant 5YSZ–1450 [21], and variant 5YSZ–1500 is adjacent to it.



**Fig. 4.** Dependences of volume percentage of (c+m)-phase agglomerates on weight percentage of (c+m)-phases for studied materials. The arrows indicate directions of data evolution with increasing the sintering temperature.



**Fig. 5.** Dependences of numbers of (c+m)-phase agglomerates on ranges of their areas, obtained after processing by ImageJ software at magnifications  $\times 100$  (a) and  $\times 400$  (b).

Quite low number of (c+m)-phase agglomerates in the middle ranges of their areas ( $0-20$ ,  $20-40$ ,  $40-60$  and  $60-80 \mu\text{m}^2$ ) was also found for variant 3YSZ-1450, that can be explained by the insufficient sintering ability of 3YSZ ceramic and low intensity of phase transformations at  $1450 \text{ }^\circ\text{C}$ .

In general, material variants exhibit the maximum number of (c+m)-phase agglomerates in the range of the smallest areas ( $0-20 \mu\text{m}^2$ ), but in the middle ranges of their areas ( $20-40$ ,  $40-60$ ,  $60-80$ ,  $80-100$  and  $100-120 \mu\text{m}^2$ ) the number of (c+m)-phase agglomerates is much smaller.

### Conclusions

It was found that with a more intensive increase of the monoclinic phase weight percentage, the volume percentage growth of the monoclinic and cubic phases in total is suppressed. Conversely, when an increase in the monoclinic phase weight percentage decelerates, this process is intensified.

It was shown that the evolution of volume percentage of the monoclinic and cubic phases depends on the yttria content and sintering temperature. An increase in the volume percentage of monoclinic and cubic phases is intensified due to the yttria content growth, whereas it is suppressed due to the sintering temperature growth.

It was found that the materials of zirconia ceramic series stabilized by 4 mol%  $Y_2O_3$  are characterized by the maximum number of (c+m)-phase agglomerates in the middle ranges of their areas (0–20, 20–40, 40–60, 60–80, 80–100 and 100–120  $\mu m^2$ ) as compared to other material variants. The lowest number of (c+m)-phase agglomerates in the middle ranges of their areas (20–40, 40–60, 60–80 and 80–100  $\mu m^2$ ) was found for zirconia ceramic series stabilized by 5 mol%  $Y_2O_3$ , obtained at sintering temperatures of 1450 °C (5YSZ–1450) and 1500 °C (5YSZ–1500). This characteristic area distribution of the monoclinic and cubic phases is consistent with the high fracture toughness of variant 5YSZ–1450.

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