

## APPLICATION OF ELECTROCONSOLIDATION OF POWDER COMPONENTS FOR PRODUCTION OF ULTRADENCED CERAMICS $Al_2O_3$ AND $ZrO_2$ (3% $Y_2O_3$ )

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The possibility of application of electroconsolidation for production of ultradenced ceramics based on  $Al_2O_3$  and  $ZrO_2$  (3%  $Y_2O_3$ ) compounds is studied. As a result of the conducted researches optimum values of process temperature at which the best parameters of  $Al_2O_3$  and  $ZrO_2$  (3%  $Y_2O_3$ ) ceramic properties are reached: the apparent density – 3.98 and 6.08 g/cm<sup>3</sup>, bending strength – 350 and 800 MPa, fracture toughness – 4.0 and 8.0 MPa·m<sup>0.5</sup> respectively are established. The researches of a ceramics microstructure are found that high parameters of  $Al_2O_3$  and  $ZrO_2$  (3%  $Y_2O_3$ ) properties are defined by dense and fine-crystalline structure.

**Keywords:** electroconsolidation, ceramic materials, mechanical properties, thermal shock resistance, microstructure, SEM analysis.

### ПРИМЕНЕНИЕ ЭЛЕКТРОКОНСОЛИДАЦИИ ПОРОШКОВЫХ КОМПОНЕНТОВ ДЛЯ ПОЛУЧЕНИЯ ОСОБОПЛОТНОЙ КЕРАМИКИ

$Al_2O_3$  и  $ZrO_2$  (3%  $Y_2O_3$ )

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Изучена возможность применения электроконсолидации для получения особоплотной керамики на основе  $Al_2O_3$  и  $ZrO_2$  (3%  $Y_2O_3$ ). В результате проведенных исследований установлены оптимальные значения температуры процесса, при которых достигаются наибольшие показатели свойств керамики на основе  $Al_2O_3$  и  $ZrO_2$  (3%  $Y_2O_3$ ) кажущаяся плотность 3.98 и 6.08 г/см<sup>3</sup>, предел прочности при изгибе 350 и 800 МПа, коэффициент интенсивности напряжений 4.0 и 8.0 МПа·м<sup>0.5</sup> соответственно. Исследованиями микроструктуры керамики установлено, что высокие показатели свойств  $Al_2O_3$  и  $ZrO_2$  (3%  $Y_2O_3$ ) определяются плотной и мелкокристаллической структурой.

**Ключевые слова:** электроконсолидация, керамические материалы, механические свойства, термостойкость, микроструктура, SEM анализа.

### ЗАСТУСОВАННЯ ЕЛЕКТРОКОНСОЛІДАЦІЇ ПОРОШКОВИХ КОМПОНЕНТІВ ДЛЯ ОТРИМАННЯ ОСОБЛИВОЩІЛЬНОЇ КЕРАМІКИ $Al_2O_3$ та $ZrO_2$ (3% $Y_2O_3$ )

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Вивчена можливість щодо використання електроконсолидації для отримання особливощільної кераміки на основі  $Al_2O_3$  та  $ZrO_2$  (3%  $Y_2O_3$ ). За результатами наведених досліджень встановлено оптимальні значення температури процесу, при яких досягаються найбільші показники властивостей кераміки  $Al_2O_3$  та  $ZrO_2$  (3%  $Y_2O_3$ ): уявна щільність 3.98 та 6.08 г/см<sup>3</sup>, міцність на вигин 350 та 800 МПа, коефіцієнт інтенсивності напруг 4.0 та 8.0 МПа·м<sup>0.5</sup> відповідно. Дослідженнями микроструктури було доведено, що високі показники властивостей  $Al_2O_3$  та  $ZrO_2$  (3%  $Y_2O_3$ ) визначаються щільною та дрібнокристалічною структурою кераміки.

**Ключові слова:** електроконсолидація, керамічні матеріали, механічні властивості, термостійкість, микроструктура, SEM аналізу.

### INTRODUCTION

Recently the technologies of constructional ceramics production for the solution of various technical tasks are widely developed all over the world. One of leading position among studied materials possess ceramic materials based on  $Al_2O_3$  and  $ZrO_2$  (3%  $Y_2O_3$ ) compounds [1 – 4].

Traditionally ceramic materials are produced from powder materials by various methods using

such as formation and sintering. Depending on a form and the size of products, their purpose and the set properties apply different types of pressing, slip casting in plaster molds, extrusion, casting under pressure from thermoplastic slips, etc. [5].

The last operation of ceramics production is high-temperature sintering at which there is a process of consolidation of the formed samples. This process is accompanied by density increase, and also shrin-

kage of a green body. The kinetics of sintering and final properties of the solid body significantly depends on both properties of original powder from which the product was formed and technology factors such as temperature of sintering, speed of temperature raising, exposure time at the maximum temperature [6].

Last time for production ceramics with high physicomechanical characteristics are using high-disperse powders and ways of high-speed heating which allow to optimize a combination of processes of the maximum consolidation and the minimum growth of grain during sintering process. There are new methods of consolidation of powder materials: the activated sintering under the influence of an external field, high-speed hot isostatic pressing, electropulse sintering under pressure, etc. [7, 8].

Among progressive methods of producing ceramic materials one of perspective is electroconsolidation process. At electroconsolidation heating is carried out by a direct transmission of an electric current through the graphite elastic squeezed medium in which one or several samples are placed. Purpose of the medium is transfer of pressure created by punches, and ensuring heating of preparations due to heat allocated at passing of current. The speed of heating of preparations can reach 200 °C/min. Thus this process allows to carry out quasiisostatic hot pressing of powder compositions for minimum short terms with necessary isothermal endurance at the maximum temperature up to 3000 °C. Means of effective control of process are necessary for the technology using.

In National science center Kharkov Institute of Physics & Technology the equipment for realization of process of electroconsolidation is developed. The equipment provides opportunity to realize technological process of sintering of powder materials of different structure, in the medium of inert gases and in vacuum [9].

The purpose of this work is studying the possibility of application of electroconsolidation for production of ultradenced ceramics on the basis of  $Al_2O_3$  and  $ZrO_2$  (3%  $Y_2O_3$ ).

## EXPERIMENTAL PROCEDURE

As the main raw materials were used powder of alumina (3000 SDP, "Almatis", Germany) with a size of particles of 0.5 microns; and the powder  $ZrO_2$  (3%  $Y_2O_3$ ) (PSZ-5.2 YB, Stanford Materials Cor-

poration, USA), with a size of particles of 0.04 – 0.07 microns.

For research carrying out the powders were filled up in forms and formed samples by a method of uniaxial cold pressing under pressure of 100 MPa. After formation alumina samples were sintered (electroconsolidation) at a temperature 1500 and 1600 °C, with a speed of heating 100 °C/min and endurance 30 minutes, and zirconia samples were sintered at a temperature 1400 and 1500 °C with similar values of speed of heating and exposure time.

Open porosity and apparent density of samples were defined according to GOST 2409-95.

Bending strength was determined by a standard method in compliance with DSTU 3716-98. Determination of fracture toughness  $k_{Ic}$  was carried out according to ASTM Standard C 1421-99.

For determination of thermal shock resistance the EN 820-3:2004 standard was used, according to which thermal stability characterized by difference of temperatures  $\Delta T$ , at which there was an emergence of cracks in samples.

The researches of microstructure were conducted on an electronic microscope of the translucent type.

## RESULTS AND DISCUSSION

Properties of ceramic samples  $Al_2O_3$  and  $ZrO_2$  (3%  $Y_2O_3$ ) produced at various temperatures, in comparison with analogous properties of import analogs are given in the table. From the provided data it is clear that the samples of  $Al_2O_3$  produced at a temperature 1500 °C, possess open porosity – 3 – 5%, their density makes 3.78 – 3.80 g/cm<sup>3</sup>. The samples of  $Al_2O_3$  produced at a temperature 1600 °C, are characterized by smaller porosity and higher values of the density – 3.96 – 3.98 g/cm<sup>3</sup> that conforms to requirements for ultradenced ceramics.

The  $Al_2O_3$  samples produced at a temperature of 1500 °C, possess rather high parameters of the main properties:  $\sigma = 260 - 290$  MPa,  $k_{Ic} = 3.0$  MPa·m<sup>0.5</sup>,  $\Delta T = 300$  °C, however is not compared well with parameters of the samples produced at a temperature 1600 °C. The  $Al_2O_3$  samples produced at a temperature 1600 °C, possess properties which are compared well with the parameters of the samples of import ceramics:  $\sigma = 350$  MPa,  $k_{Ic} = 3.5 - 4.0$  MPa·m<sup>0.5</sup>,  $\Delta T = 300$  °C.

Table  
Properties of ceramic samples  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$   
(3%  $\text{Y}_2\text{O}_3$ ) in comparison with import analogs

| Ceramic brand  | Properties of ceramic |                                   |                       |   |  |
|--|-----------------------|-----------------------------------|-----------------------|---|--|
|  | Open porosity, %      | Apparent density, $\text{g/cm}^3$ | Bending strength, MPa | Fracture toughness $k_{1c}$ , $\text{MPa}\cdot\text{m}^{0.5}$ | Thermal shock resistance $\Delta T$ , $^\circ\text{C}$ |
| $\text{Al}_2\text{O}_3$<br>(1500 $^\circ\text{C}$ )<br>NSC<br>Kharkov                        | 3–5                   | 3.78–3.80                         | 260–290               | 3.0   | 300  |
| $\text{Al}_2\text{O}_3$<br>(1600 $^\circ\text{C}$ )<br>NSC<br>Kharkov                        | 0                     | 3.96–3.98                         | 350                   | 3.6–4.0   | 300  |
| $\text{Al}_2\text{O}_3$<br>Dynamic ceramic<br>(Eng.)   | 0                     | 3.95                              | 350                   | 4.0   | –  |
| NSC<br>Kharkov<br>$\text{ZrO}_2$<br>(3% $\text{Y}_2\text{O}_3$ )<br>(1400 $^\circ\text{C}$ ) | 2–5                   | 5.80–5.86                         | 680                   | 6.0–6.4   | 400  |
| NSC<br>Kharkov<br>$\text{ZrO}_2$<br>(3% $\text{Y}_2\text{O}_3$ )<br>(1500 $^\circ\text{C}$ ) | 0                     | 6.04–6.08                         | 800                   | 7.6–8.0   | 400  |
| $\text{ZrO}_2$<br>(3% $\text{Y}_2\text{O}_3$ )<br>Kyocera<br>(Japan)                         | 0                     | 6.00                              | 750                   | 7.0–8.0   | 400  |

Similarly properties of  $\text{ZrO}_2$  (3%  $\text{Y}_2\text{O}_3$ ) samples are changed. With increase in temperature of electroconsolidation from 1400 to 1500  $^\circ\text{C}$ , open porosity of samples disappears, their density increases to 6.04 – 6.08  $\text{g/cm}^3$ , and the main parameters of properties correspond to level of import ceramics:  $\sigma = 800$  MPa,  $k_{1c} = 7.6 – 8.0$   $\text{MPa}\cdot\text{m}^{0.5}$ ,  $\Delta T = 400$   $^\circ\text{C}$ .

As properties of ceramics, substantially are defined by its structure, for an explanation of the received results the corresponding researches were carried out.

In fig. 1 and fig. 2 the microstructure of ultradensified ceramic  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  (3%  $\text{Y}_2\text{O}_3$ ) is respectively shown.

From fig. 1 it is visible that the ceramics  $\text{Al}_2\text{O}_3$  represents very fine-crystalline structure, with a prevailing size of grains of 1 – 3  $\mu\text{m}$ . Thus the minimum size of grains of corundum makes 0.5  $\mu\text{m}$  and maximum – 6  $\mu\text{m}$ . Grains have a crystallographic facet that testifies about completion of process of crystallization at a temperature of 1600  $^\circ\text{C}$ . Borders

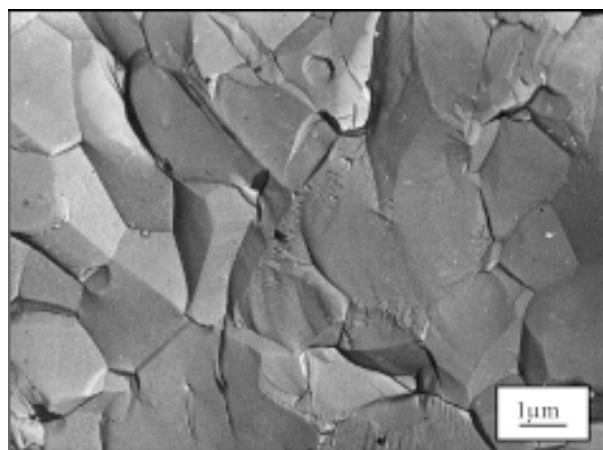


Fig. 1. Microstructure of ultradensified ceramic  $\text{Al}_2\text{O}_3$ .

of grains of corundum are very dense, thus on all volume of a ceramic sample are noticed fine-crystalline grains of spinel ( $\text{MgO}\cdot\text{Al}_2\text{O}_3$ ) with size  $\leq 0,5$   $\mu\text{m}$ .

From fig. 2 it is visible that the ceramic sample of  $\text{ZrO}_2$  (3%  $\text{Y}_2\text{O}_3$ ) also has fine-crystalline structure which consists of well crystal grains of 0.2 – 2.6  $\mu\text{m}$  in size, with a prevailing size of grains of 1.5 microns.

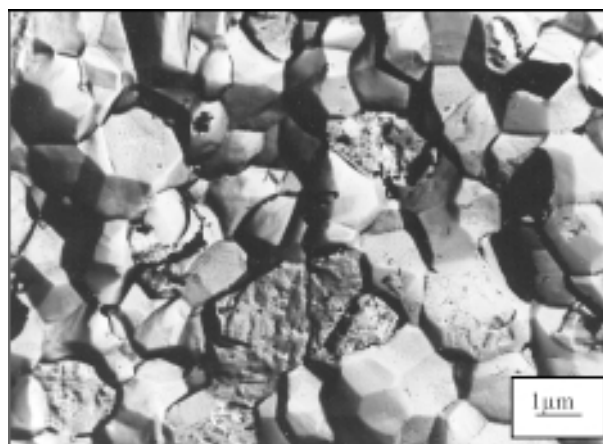


Fig. 2. Microstructure of ultradensified ceramic  $\text{ZrO}_2$  (3%  $\text{Y}_2\text{O}_3$ ).

As a result of the carried out researches, the possibility of application of electroconsolidation for production ultradensified ceramics  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  (3%  $\text{Y}_2\text{O}_3$ ) is established.

## CONCLUSIONS

The possibility of application of electroconsolidation for production of ultradensified ceramics on the basis of  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  (3%  $\text{Y}_2\text{O}_3$ ) is studied. As a result of the conducted researches optimum values of process temperature at which the best parameters of properties of  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  (3%  $\text{Y}_2\text{O}_3$ ) ceramics are reached: the apparent density – 3.98 and 6.08  $\text{g/cm}^3$ , bending strength – 350 and 800 MPa,

fracture toughness – 4.0 and 8.0 MPa·m<sup>0.5</sup> respectively are established. The researches of a microstructure of ceramics demonstrate that high parameters of properties  $Al_2O_3$  and  $ZrO_2$  (3%  $Y_2O_3$ ) ceramics are defined by dense and fine-crystalline structure.

Developed ceramics are perspective for application as constructional materials for various spheres of science and engineering.

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