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NEW TECHNOLOGIES OF PREPARATION AND TREATMENT OF MATERIALS

Influence of Mechanical Treatment on Consolidation Processes of Ultradisperse Powders of Stabilized Zirconium Oxide

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Abstract—The effect of preliminary machining of ultradisperse powders of stabilized zirconium dioxide and its composite on consolidation in compacts under uniaxial static pressing and subsequent sintering is studied. The investigations were carried out with powders of compositions (in mol %) $97ZrO_2$–$3Y_2O_3$ and $80Al_2O_3$–$20(ZrO_2$–Y), which were obtained by the sol-gel and the plasma-chemical method, respectively. Mechanical processing of powders was carried out in two ways. The first method consisted in preliminary static pressing of powders at elevated pressure of 900 MPa and their subsequent grinding in a ball mill. The second method consisted in grinding the initial powders in an Activator-2SL planetary mill with drums and grinding balls of zirconia. It is established that mechanical treatment significantly affects the density of compacts. In this case, there is no strict correlation between the density of the sintered ceramic and the density of compacts. With increasing density of compacts, their expansion can be observed at the isothermal holding stage, which leads to a decrease in density of the ceramic. It is shown that, in dry grinding to improve the technological properties of ultradisperse powders obtained by the sol-gel and plasma-chemical methods, the most suitable is the method of mechanical treatment, which consists in pre-pressing the powders at elevated pressure and then grinding them in a ball mill.

Keywords: ultrafine powders, zirconia, static pressing, sintering

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INTRODUCTION

Ceramic based on zirconia possesses some unique advantages, which determine its broad application as a functional and construction ceramic [1–4]. The preparation of each ceramic involves pressing processes of powder compacts [5] and their sintering. In this case, the quality of the ceramic is mainly determined by the features of the starting powder, which depend on the method for its preparation, dispersion, and shape and size of particles. In this context, the employment of ultradisperse powders (UDPs) is promising. The methods for the preparation of UDPs of zirconia and its composites are numerous. Among them, sol-gel [6–8] and plasma-chemical methods [8] are promising for industrial application, because the technologies providing large-scale production are realized on their basis. However, UDPs obtained using these methods are characterized by weak moldability [9]. This is related to the inhomogeneous morphology of the synthesized oxide particles. This fact complicates the preparation of a thick nonporous ceramic. For this reason, the problems of modification of properties of UDPs and ceramic [10] and determination of the principles of consolidation of particles depending on the method of compacting, the compacting conditions [11, 12], and the type and conditions of thermal heating [13–15] become relevant.

The aim of this work is to investigate the effect of preliminary machining of UDP of stabilized zirconia and its composite on the consolidation in compacts at pressing and further sintering.

PROCEDURE OF EXPERIMENT

The following UDPs were studied (in mol %): $97ZrO_2$–$3Y_2O_3$ (powder P1) and $80Al_2O_3$–$20(ZrO_2$–Y) (powder P2), which were prepared using the sol-gel and plasma-chemical methods, respectively. The powders were exposed to mechanical treatment using following methods. The first method involved preliminary static pressing of the powders P1 and P2 under elevated pressure $P = 900$ MPa and their further grinding in a ball mill [16]. The second approach involved grinding of starting powders P1 and P2 in an Activator-2SL planetary mill (PM) with drums and grinding balls made from zirconia. The ratio of mass of balls to the mass of powder was 1.5. The rotation speed of the drums was 1500 rpm. The grinding period was varied in the range from 7.5 to 30 min. It should be noted that the described procedures for treatment of powders did not significantly affect their phase state. The third

Table 1. Influence of the method of processing of initial ultrafine powders of zirconium dioxide on the density of samples compacted in the static pressing mode and ceramic samples sintered from them

Method of powder processing	Density of compact, ρ, g/cm^3	Relative density of compact, ρ_{rel}	Density of ceramic, ρ_c, g/cm^3	
			$T_{sintering}$ = 1550°C, 1 h	$T_{sintering}$ = 1600°C, 1 h
Pressing at P = 920 MPa and grinding	2.65	0.45	5.6	5.67
Grinding in PM for 7.5 min	2.64	0.45	5.47	5.5
15 min	3.1	0.53	5.51	5.52
30 min	3.12	0.54	5.21	5.12

Theoretical density of zirconium ceramic ρ_{theor} = 5.85 g/cm^3.

Table 2. Influence of the method of processing initial corundum-zirconium ultrafine powders on the density of samples compacted in the static pressing mode and ceramic samples sintered from them

Method of powder processing	Density of compact, ρ, g/cm^3	Relative density of compact, ρ_{rel}	Density of ceramic, ρ_c, g/cm^3	
			$T_{sintering}$ = 1550°C, 1 h	$T_{sintering}$ = 1600°C, 1 h
Pressing at P = 920 MPa and grinding	2.57	–	5.14	5.25
Grinding in PM for 7.5 min	2.43	–	5.01	5.14
15 min	2.69	–	5.03	5.09
30 min	2.7	–	5.04	5.15
Grinding in PM for 15 min, thermal sintering at T = 1300°C for 1 h, pressing at P = 920 MPa, and grinding	3.26	0.59	5.14	5.26

Theoretical density of the powder composite after thermal sintering is ρ_{theor} = 5.47 g/cm^3; theoretical density of ceramic is 5.47 g/cm^3.

approach was different from the first two in that powders P2 after treatment in planetary mill were sintered at T = 1300°C for 1 h. This was done in order to transfer amorphous alumina into α-Al_2O_3. In this case, the phase composition of the powder differed significantly from the initial one and was the following (wt %): 82 (t-ZrO_2)–1 (m-ZrO_2)–17 (α-Al_2O_3).

The specimens under study were prepared in the form of disks with the diameter of 1 cm and the thickness of 0.23–0.27 cm. The specimens were compacted under uniaxial static pressing at P = 150 MPa.

The kinetics of compacting of the specimens during their heating at a constant rate and further isothermal holding was investigated using a DIL 402 C high-sensitivity dilatometer (NETZSCH, Germany).

RESULTS AND DISCUSSION

The results of the effect of the method of processing of initial UDPs P1 and P2 on the density of specimens compacted in the static pressing mode and the ceramic sintered from them are given in Tables 1 and 2.

The theoretical density of the ceramic prepared from the ZrO_2–Al_2O_3 batch under the assumption of zero porosity was calculated using the following equation:

$$\rho_t = \frac{\rho_{Al_2O_3}\rho_{ZrO_2}}{M_1\rho_{ZrO_2} + M_2\rho_{Al_2O_3}}, \quad (1)$$

where $\rho_{Al_2O_3}$ and ρ_{ZrO_2} are the densities of alumina and stabilized zirconia, respectively, and M_1 and M_2 are the mass fractions of alumina and stabilized zirconia, respectively.

In this case, the density ρ_{ZrO_2} for the stabilized state of zirconia was determined according to the following equation:

$$\rho_{ZrO_2} = (A_1 + 2A_2)\frac{4A_0}{a^2}c, \quad (2)$$

where A_1 and A_2 are the relative atomic weight of zirconium and oxygen, respectively; A_0 = 1.66 × 10^{-24} g is the atomic mass number; and a and c are the lattice parameters of tetragonal phase of stabilized zirconia, which were measured experimentally.

It is clear from the analysis of the data given in Tables 1 and 2 that there is no unambiguous correlation between the density of the compact and the den-

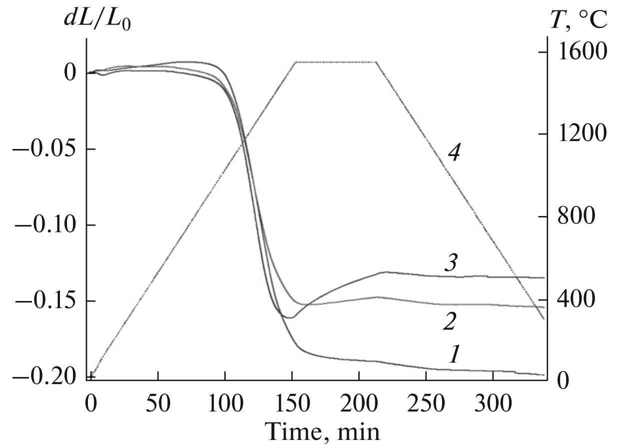

Fig. 1. Effect of processing time in the planetary mill of zirconia powders on the kinetics of sintering of zirconium ceramic: (*1–3*) processing time of 7.5, 15, and 30 min, respectively; (*4*) temperature regime of sintering.

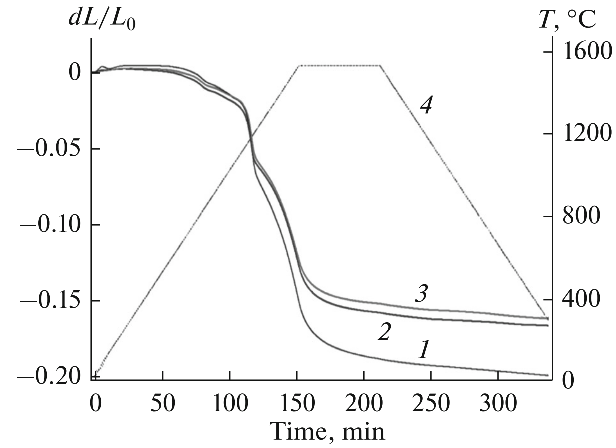

Fig. 2. Influence of treatment time in the planetary mill of ZrO_2–Al_2O_3 powders on the kinetics of sintering of composite ceramic: (*1–3*) processing time of 7.5, 15, and 30 min, respectively; (*4*) temperature regime of sintering.

sity of ceramic. Processing of the powder in planetary mill for 15 and 30 min leads to a slight increase in the density of the compact, which is ≈17 and 5% in the case of zirconia powder and the powder composition ZrO_2–Al_2O_3, respectively. However, in this case, the density of the zirconium ceramic sintered from them decreases, while the density of ZrO_2–Al_2O_3 composite hardly changes.

In Fig. 1, typical kinetic dependences of the linear shrinkage ($\Delta L/L_0$) of zirconia compacts prepared from powders P1, which were pretreated in PM for a different period, are given.

As follows from the data in Fig. 1, an increase in the processing time of the zirconia powder in PM causes disturbance of the shrinkage of the compact. This manifests itself in that the expansion of the sintered compacts occurs at the stage of isothermal holding. In addition, the larger the period of grinding of the zirconia powder in PM and the higher the density of the compacts produced from it, the larger is the degree of expansion of the specimen during isothermal holding. The expansion of specimens can be explained by the decrease in the density of zirconium ceramic with an increase in the period of processing of the powder in the PM (Table 1).

The results of the study of the effect of processing of powders of composition P2 in the planetary mill on the kinetics of their shrinkage are given in Fig. 2.

It is clear that, during sintering of the powder nanocomposites of the ZrO_2–Al_2O_3 system, an increase in the isothermal holding period causes a decrease in linear dimensions of the specimens rather than their increase, as was characteristic of zirconium ceramic.

The set of the studies showed that grinding ultradisperse plasma-chemical powders in a planetary mill as compared to the first method of mechanical processing of powders, at which a lower density of the compact is achieved, leads to sintering of both types of ceramic with reduced density. Thus, dry grinding of the powders in planetary mill cannot provide a composition ceramic with increased density. The best and almost identical results were obtained when using the first and third methods of powder preparation. In this case, the relative density of the compact prepared by static pressing using the powder exposed to thermal sintering achieves the value corresponding to ρ_{rel} = 0.59 of the theoretical value.

CONCLUSIONS

In order to improve the technological properties of ultradisperse powders $97ZrO_2$–$3Y_2O_3$ and $80Al_2O_3$–$20(ZrO_2$–$Y)$ prepared by the sol-gel and plasma-chemical methods, the method of mechanical processing is the most convenient, which involves the prepressing of powders at elevated pressure and their further grinding in a ball mill.

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