

Sintering of zirconia ceramics by intense high-energy electron beam



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ABSTRACT

A comparative analysis of the efficiency of zirconia ceramics sintering by thermal method and high-energy electron beam sintering was performed for compacts prepared from commercial TZ-3Y-E grade powder. The electron energy was 1.4 MeV. The samples were sintered in the temperature range of 1200–1400 °C. Sintering of zirconia ceramics by high-energy accelerated electron beam is shown to reduce the firing temperature by about 200 °C compared to that in conventional heating technique. Ceramics sintered by accelerated electron beam at 1200 °C is of high density, microhardness and smaller grain size compared to that produced by thermal firing at 1400 °C. Electron beam sintering at higher temperature causes deterioration of ceramics properties due to radiation-induced acceleration of high-temperature recrystallization at higher temperatures.

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1. Introduction

One of the current problems of modern science and technology is to manufacture and study the properties of tool and construction purpose ceramic materials of high mechanical strength. There are two basic approaches to solve this problem. The first approach implies production of ceramic materials with nano-sized structural components. At present, this problem can be solved due to advances in the technology of ultradisperse powders (UDP). UDP can be sintered at lower temperatures since the surface energy is high as compared to that of coarse powders. This allows reduction of the rate of grain growth caused by extensive recrystallization, and production of ceramics with submicron-sized grains. The second approach based on the effect of polymorphic transformations under external mechanical loads is of primary interest as it can increase the fracture toughness of ceramics. Both of these approaches can be realized for stabilized zirconia based ceramic structures.

Zirconia is characterized by polymorphic phenomena. It can exist mainly in three crystalline modifications, namely low temperature monoclinic (m) modification and high temperature tetragonal (t) and cubic (c) modifications. Invalent cations, which increase the anionic sublattice defectiveness, are introduced in the ZrO₂ crystal lattice to ensure the tetragonal phase at room temperature. This state is referred to as stabilized zirconia. When a crack appears and propagates under critical loads, metastable tetragonal grains transform into monoclinic phase and their volume increases. As a result of structural transition, the crack loses

its energy and stops propagating, thereby increasing the material strength.

As shown by numerous studies, stabilized zirconia based ceramic materials possess a unique combination of mechanical, thermal, electrical and other properties that makes them widely used in various fields of science and technology [1].

The basic method to manufacture zirconia ceramics is high-temperature sintering of compacted powders. The diffusion mobility of the substance components grows at increased temperature. Therefore, sintering temperature and duration should be reduced to limit recrystallization and growth of the grain and to retain its small size. Simultaneously, the ceramics fabricated should be of high density and minimum porosity. However, these conditions are difficult to fulfill as grain coarsening and densification are thermally activated processes occurring simultaneously. Various powder sintering techniques are proposed to overcome these problems. Among these are sufficiently laborious hot-pressing technique [2], which requires time-consuming two-step sintering [3,4], spark plasma sintering (SPS) [2,5–8,11] and microwave sintering [8–11], which employ alternative heating sources. The studies have shown that the above techniques make possible to decrease the sintering temperature and to obtain ceramic structures with most essential technical characteristics superior to those of the ceramics manufactured using conventional firing technique.

Russian scientists suggested a new method to activate ceramic sintering through heating by high-intensity beam of high-energy electrons [12–16]. This new direction appeared due to the emergence of a new class of industrial technological purpose electron accelerators that have been developed at the Institute of Nuclear Physics SB RAS (Novosibirsk, Russia) for many years. These

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accelerators generate electron beams with maximum beam power of up to 160 kW. The electron energy ranges from 1 to 4 MeV. The electron beams with specified parameters provide bulk heating of the target up to (1500–2000) °C. The processes occurring in materials under electron beam heating and methods and techniques based on these referred to as radiation-thermal (RT) processes.

Diffusion-controlled solid-phase processes in inorganic materials under RT effect are significantly different from those observed for thermal heating techniques. This difference is due to the electron-beam induced change in the defective state of crystal lattice.

As is well known, high-energy electrons, when passing through condensed media, transfer energy to both electronic and nuclear subsystems of the crystal lattice and turn these into excited state. This results in the formation of various defects of vacancy nature, displaced atoms, electron-hole pairs and excitons. Electron-phonon interactions change the vibrational spectrum of the particles in the crystal lattice.

In these conditions, the rate of the diffusion processes, which are highly sensitive to the changed defect state of the solid and the potential relief of the crystal lattice, can increase. Therefore, high-temperature diffusion-controlled solid-state reactions, which make the basis for ceramic production, can be significantly accelerated as well. Reliable experimental data accumulated by now proves increase in heterodiffusion coefficients in ionic dielectrics [17–20] and bulk and grain boundary diffusion coefficients in polycrystalline lithium ferrosinels [21] under RT effects. Experimental evidence was obtained for RT activation of diffusion-controlled synthesis of complex oxide compounds [22–24] and sintering of some types of ceramics [12–16].

As is obvious from the foregoing, the electron beam heats the material and, in addition, it significantly accelerates high-temperature mass transfer in a broad class of ionic structures and, hence, the rate of practically important diffusion-controlled solid-phase reactions.

This paper aimed to explore the potential of the RT technique for sintering zirconia ceramics from UDP. The problem was solved through the comparative analysis of ceramic sintering using the accelerated electron beam and that performed by the conventional technique.

2. Experimental

Stabilized zirconia powder, TZ-3Y-E grade (Tosoh Corporation, Japan), of the composition (in mol%): $97\text{ZrO}_2\text{-}3\text{Y}_2\text{O}_3$, prepared through hydrolysis was sintered to manufacture ceramics. Stabilized ZrO_2 state was obtained with 3 mol% Y_2O_3 introduced into the crystal lattice. The specific surface area (S_{sp}) of the powders was measured using the Brunauer-Emmett-Teller (BET) method. The values obtained for S_{sp} were used to calculate the average primary particle size (D) according to the formula $D=6/\rho \cdot S_{\text{sp}}$, where ρ is the material density. $D=75$ nm.

The sample pellets were prepared through dry uniaxial compression at pressure 150 MPa. The relative density of the compacts was $\approx 48\%$ of the theoretical density. The compacts made under the same conditions were divided into two parts. One part was sintered using thermal technique, and the other part was sintered by accelerated electron beam.

The kinetics of sample densification during thermal firing in the temperature range (1200–1500) °C was investigated using a high-sensitivity DIL 402 C dilatometer (NETZSCH, Germany). The heating rate was 10 °C/min. The isothermal holding time was 1 h.

RT sintering of ceramics was carried out with the electron accelerator ELV-6 of continuous action (Institute of Nuclear Physics SB RAS, Novosibirsk). The energy of the accelerated electrons was

1.4 MeV. The absorbed dose was ($10^4\text{-}10^5$) Gy/s. The electron beam current density was varied in the range of (12–16) $\mu\text{A}/\text{cm}^2$ that provided heating of the target (compact) up to the sintering temperature range (1200–1400) °C.

During RT firing, the samples were placed on the bottom of the cell made of lightweight chamotte. The side of the cell exposed to radiation was covered with radiation-transparent protector. The temperature was controlled using a thermocouple (type S), which was in contact with the test sample located in the immediate vicinity of the sintered samples.

The density of the ceramic samples was measured by hydrostatic weighing in distilled water using Shimadzu analytical balances equipped with special attachment. The X-ray diffraction (XRD) was performed with the ARL X'tra diffractometer using $\text{CuK}\alpha$ X-ray radiation. The obtained X-ray patterns were subjected to the Rietveld analysis using Powder Cell 2.4 software code. The microstructure of the ceramic samples was examined by the scanning electron microscopy (SEM) using the Hitachi TM-3000 electron microscope. The microhardness of the samples was measured with the ZHV1M microhardness tester (Zwick, Germany) with indentation load 300 g. The exposure time under loading was 10 s. Ten indents were performed on the sample surface.

3. Experimental results and discussion

3.1. Thermal sintering

Fig. 1 shows the kinetic features of compact shrinkage for different firing modes. The samples were heated and cooled at a rate of 10 °C/min. The isothermal holding time was 1 h.

The analysis of the dilatometric (shrinkage) curves showed that a significant change in the linear dimensions of the samples starts at ≈ 950 °C. At 1200 °C, shrinkage is observed to be low. At 1300 °C, shrinkage sharply increases. The degree of the shrinkage during heating grows, and during isothermal holding, it decreases. A further increase in the temperature has little effect on the shrinkage.

Fig. 2(a) and (b) shows the dependences of the ceramics density, its microhardness and the average grain size on the firing temperature.

It can be seen that ceramics density sharply increases in the firing temperature range (1200–1400) °C, which is consistent with the data of the dilatometric studies. In this temperature interval, the average grain size does not significantly change. The observed

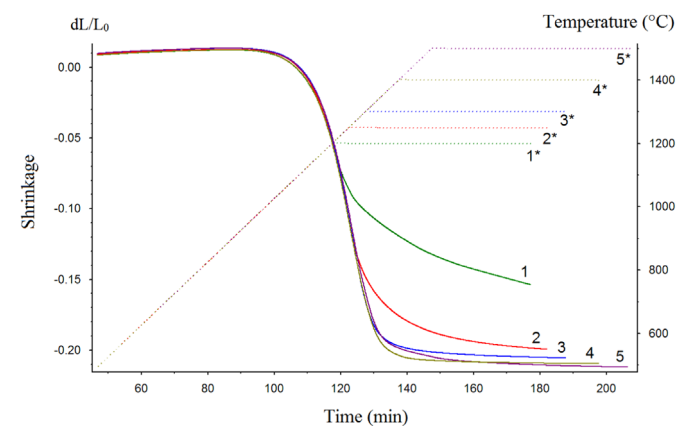


Fig. 1. Kinetic behavior of liner shrinkage (curves (1–5)) of compacts heated to (1200–1500) °C at a heating rate of 10 °C/min and during 1 h isothermal holding: 1, 2, 3, 4 and 5 are for 1200, 1250, 1300, 1400 and 1500 °C, respectively. (1*–5*) indicate thermal sintering regimes.

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