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The structure transformations in nanocrystalline zirconia

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Abstract

The special features of the structure and phase composition of nanocrystalline zirconia-based powders are studied. The investigations show that an increase in the monoclinic phase abundance is associated with a reduction in the critical size of tetragonal crystallites due to an accumulation of lattice microdistortions. This is attributed to the fact that the resultant lattice microdistortion level is inadequate to destabilize the nanocrystalline tetragonal phase. Relaxation of microdistortions on annealing causes the critical size of tetragonal crystallites to increase and the monoclinic phase is converted into the tetragonal one.

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Keywords: Nanocrystalline zirconia; Phase transformations; Lattice microdistortions

1. Introduction

Nanocrystalline powders obtained by plasma spray pyrolysis methods are a promising material for production of zirconia-based ceramics. Powders of this sort exhibit a more uniform constituent element distribution over particles as compared to those prepared by conventional processes [1], and a larger amount of excess energy is stored in these powders than in their coarse-grained cousins. In recent years, an increasing interest has been shown in dynamic processes of fine-powder treatment that will enable the powder medium to be modified in a more efficient way. The number of defects in the crystal lattice therewith increases, and increased powder "activation" may be observed. Integrated exposure of the structure and phase composition of the materials to multiple processes at high pressures offers promise for decreasing the sintering temperature of nanocrystalline zirconia-based powders [2]. In this connection, an investigation of the high-pressure effect on the structure and phase composition of the powders under consideration assumes particular importance, because it extends the possibilities to generate a preassigned structure of the materials. While the static pressure effect on the phase composition of single crystals and coarse-grained powders has been investigated in great detail, the phase transformations in materials with a nanocrystalline structure exposed to high pressures are poorly understood. The aim of this paper is to study changes in the structure and phase composition of nanocrystalline zirconia-based powders after annealing and being subjected to shock-wave treatment.

2. Experimental procedure and sample preparation

The powders were produced by plasma spray synthesis of nitrate salts of Zr, Y, and Al in a high-frequency discharge. A shock wave in the sample was generated by explosive [3] or planar impact of a high-velocity cylindrical copper or steel projectile [4].

The X-ray investigations have been carried out by means of a diffractometer and at the SR-source of VEPP-3 of the Siberian SR Centre at the Budker Institute of Nuclear Physics; Novosibirsk; the operating temperature interval was 290–1075 K. X-ray wave length was 0.15215 nm, the monochromaticity of radiation was $\Delta\lambda/\lambda = 10^{-4}$, SR beam was about 1–2 mm in linear size. The experimental X-ray

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profiles have been approximated by the analytical Caushy functions down to a minimal mean-square deviation from the experimental points.

3. Results and discussion

Data obtained by transmission electron microscopy show that the morphology of the ZrO_2 (3Y) powder particles is typical for plasma spray synthesis. The major fraction of the particles is hollow spheres, semispheres or films. Dark-field microscopy of the nanostructure of zirconia powder particles suggests that the major fraction of crystallites of size $D<50$ nm is single-domain particles. The crystallite particle size distribution are shown that the average crystallite size for ZrO_2 (3Y) powder is 20 nm. X-ray spectral microanalysis of individual powder particles shows no difference in Y_2O_3 concentration between particles made up of coarse and fine crystallites. The average amount of Y_2O_3 is approximately 3 mol%.

Fig. 1 shows fragments of X-ray diffraction patterns of ZrO_2 (3Y) samples in the initial state and after heating at different temperatures. As is seen from the figure, there are changes in the intensity of m-ZrO_2 (Y) and the lines of the t modification are broadened substantially. As seen from the figure, the line intensity of m-ZrO_2 (Y) is enhanced dramatically after loading, and the X-ray diffraction peaks of the t modification are broadened substantially.

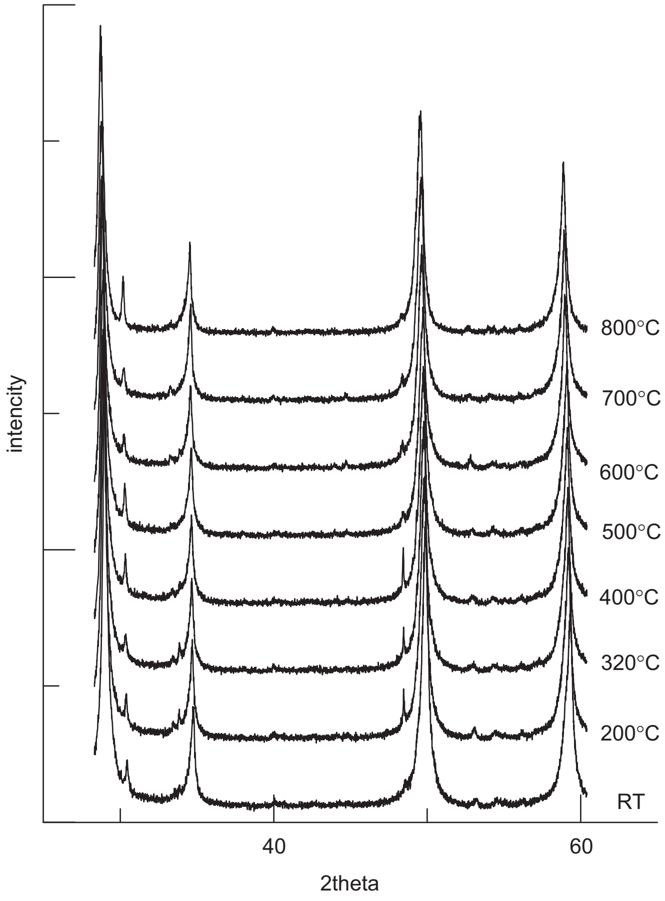

Fig. 1. Fragments of powder X-ray diffraction patterns from ZrO_2 (3Y) during heating.

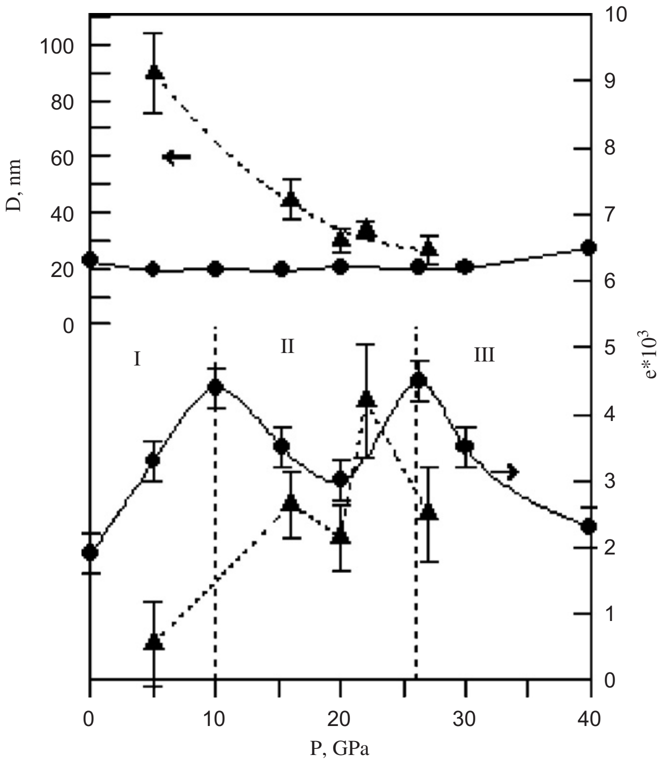

Fig. 2. Variations of the CDD average size D and lattice microdistortions in the initial phase of ZrO_2 (3Y) powder (●, solid lines) and coarse-grained ZrO_2 powder (▲, dotted lines, [3]) subjected to shock compression.

Fig. 2 demonstrates the pressure dependence of the coherently diffracting domain (CDD) average size and the t-phase lattice microdistortion in powder. Also shown for comparison are the literature data on the variations of these characteristics inherent in coarse-grained zirconia powder subjected to shock compression. After treatment of this kind, nanocrystalline powder exhibits more severe lattice microdistortions. Three characteristic ranges of change in the microdistortion level in the binary system can be recognized: region I corresponding to an increase in the microdistortion level, region III corresponding to a decrease in this quantity down to the initial level at high pressures, and region II corresponding to the microdistortion curve dip at moderate pressures. Similar behavior of the microdistortion level is observed in the coarse-grained powder under review. These data show that variations of the amount of the m modification in ZrO_2 (3Y) depicted in curve 1 are in good agreement with those found in the t lattice. Notably, this behavior of the lattice microdistortions (presence of two peaks on the plot of $<\varepsilon>$ against P) in zirconia is slightly different from that observed earlier in other ceramic powders subjected to shock-wave loading [5]. In Fig. 2, we see no increase in the CDD size in the t phase in nanocrystalline ZrO_2 (3Y) powder, which testifies that formation of the m phase in the powder is not related to the grain growth (i.e., to the change in the surface energy). On