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Research Article

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Synthesis and Characterization of Nano-structured ABo_3

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Abstract

Nanoparticles of $AB0_3$ (A= Ba, Sr & Ca; B=Zr) have been synthesized using modified single step combustion method for the first time. All the peaks of the XRD are indexed and no peaks are observed other than the expected peaks of the respective nano powders. The analysis results show that they are isostructural and no trace of any impurities. Only the as-prepared powder of $CaZr0_3$ needs an annealing of about 700 °C for 1 h. Their lattice parameters are agreed well with JCPDS values. In this paper r, the preparation and property analysis have been planned for single perovskites barium zirconate, strontium zirconate using a modified single-step auto-igniting combustion process.

Keywords: XRD, SEM, TEM Ethylene Diamine Tetra Acetic acid (EDTA), Perovskite Materials (barium zirconate, strontium zirconate and calcium zirconate), Debye Scherrer's Formula, EDX

Introduction

The successful synthesis of large-scale nanostructured materials with grain sizes in the range of 10-200 run represents a major achievement in the emerging field of nanotechnology. The scientific literature indicates that there are many techniques that can be used to produce nanostructured materials, including inert gas condensation or chemical vapor condensation, pulse electron deposition, plasma synthesis, crystallization of amorphous solids, severe plastic deformation, and consolidation of mechanically alloyed or cryomilled powders. However, only a few of these techniques, such as electrodeposition and consolidation of mechanically alloyed/cryomilled powders, generate nanostructures with sufficient thermal stability to permit the fabrication of bulk materials. Water, a natural solvent is known to exhibit unique properties under supercritical conditions and this has been suitably exploited to promote dissolution, diffusion, adsorption, reaction rate, nucleation and growth for the synthesis of ceramics. Over the past decade, hydrothermal synthesis Litvin and Popolitov 1984 has been widely used to produce weakly aggregated fine powders of both binary and mixed oxides under mild conditions. There is also considerable interest in the synthesis of oxide materials via microwave heating both at atmospheric pressure and under hydrothermal conditions, which notably accelerates chemical reactions. In combustion synthesis, the exothermicity of the redox (reduction-oxidation or electron transfer) chemical reaction is used to produce useful materials. By controlling the processing parameters such as microwave initiation, gravity, precursors and additives to redox mixtures, it has been possible to obtain nano size oxides using combustion synthesis. In this paper r, the preparation and property analysis have been planned for single perovskites barium zirconate, strontium zirconate and calcium zirconate using a modified single-step auto-igniting combustion process.

Synthesis of Nanocrystalline $AB0_3$

To produce powders of high phase purity, repeated grinding and re-firing is necessary to sufficiently complete the solid-state reaction of BaC03 and $Zr0_2$. $BaZr0_3$ is a highly refractory material, and extended mechanical grinding is typically required to sufficiently reduce the particle size to allow sintering to high density. For present investigations, nanoparticles of $AB0_3$ (A=Ba, Sr & Ca; B = Zr) have been synthesized using a modified single step combustion process. An aqueous solution containing ions of A (A = Ba, Sr & Ca) and Zr was prepared by dissolving stoichiometric amount of high purity $A(N0_3)_2$ (99%, CDH, India) and $ZrOC1_2 \bullet 8H_20$ (99%, CDH, India) in 200ml distilled water in a glass beaker. Citric acid (99%, CDH, India) was then added to the solution containing A (A=Ba, Sr & Ca) and Zr ions. Amount of citric acid was calculated based on total valence of the oxidizing and reducing agents for maximum release of energy during combustion [1]. In the preparation of nanoparticles of other ceramic oxides using combustion process, polyvinyl alcohol and urea were used as the complexing agent and fuel, respectively. In these cases, usual calcinations of the combustion product were essential to get a single-phase nano material. In the present combustion method, citric acid was used as the complexing agent instead of polyvinyl alcohol and urea was replaced with ammonia. Using this complexing agent and oxidant fuel system, it was possible to get a single phase $AZr0_3$

Characterization of the Structure and Phase Purity of AB03 using X-Ray Diffraction Technique

As-prepared $AZr0_3$ (A=Ba, Sr & Ca) powder obtained by the combustion method is characterized by different characterization techniques. The structure and phase purity of the powders are examined by powder X-ray diffraction (XRD) technique using X-ray Diffractometer

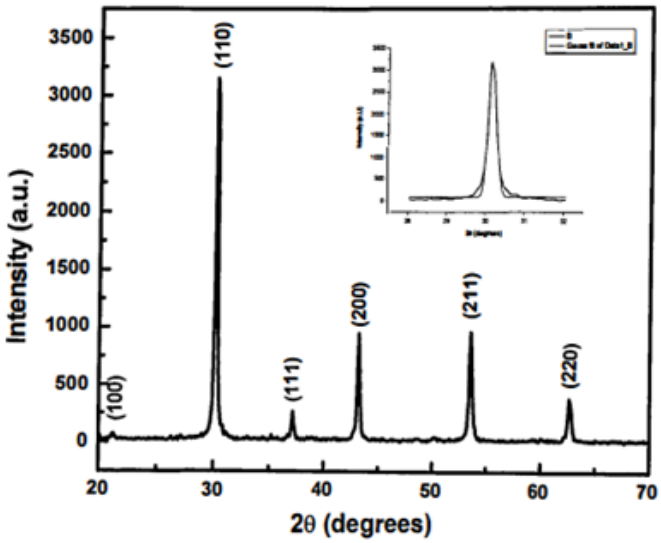


Figure 1: The XRD pattern of as-prepared $BaZr0_3$ nanocrystals. A close-up view of (110) predominant peak in the inset is used to calculate the grain size of the powder

Table 1: Interplanar $d_{hk}l$ spacings and microstrain in the $BaZr0_3$ nanopowder

Peak Position (2ɵ)	(hkl)	Interplanar Distance (d_{hkl}) (A)	Deviation in dhkl ($\Delta d_{hk}l = d_o - d_s$)	Microstrain
21.1539	(100)	4.196415	0.00195	0.000472
29.1843	(110)	5.136412	-0.00475	-0.00228
36.1722	(111)	3.16423	-0.00425	-0.0018
42.1918	(200)	2.14569	-0.00324	-0.00202
52.3877	(211)	1.24587	0.002038	-0.00423
61.6038	(220)	1.12356	0.000112	-0.00302

shows the X-ray diffraction pattern of as-prepared and annealed at 600°C powders of $SrZr0_3$ for 28 between 20° and 60°

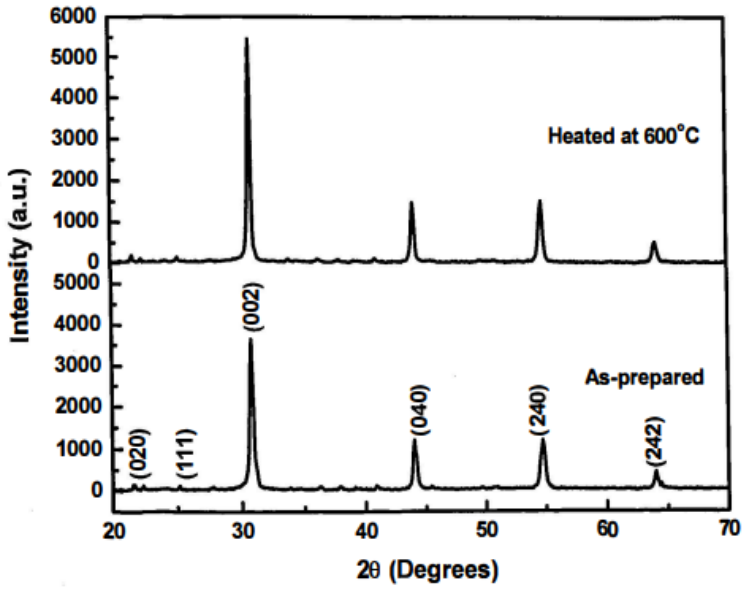


Figure 2: The XRD pattern of as-prepared and annealed $SrZr0_3$ nanocrystals at 600 °C

Table 2: Interplanar dhkl spacings and microstrain in the $BaZr0_3$ nano powde

Peak Position (2θ)	(hkl)	Interplanar Distance (d_{hkl}) (A)	Deviation in dhkl ($\Delta d_{hk}l = d_o - d_s$)	Microstrain
20.1539	(020)	3.196413	0.00175	0.000272
28.1843	(111)	4.136512	-0.00375	-0.00218
35.1721	(002)	2.15423	-0.00325	-0.00018
40.1914	(040)	2.13569	-0.00224	0.001436
42.3855	(240)	1.12587	0.001038	0.002674
64.6038	(242)	1.13156	0.000111	-0.00017

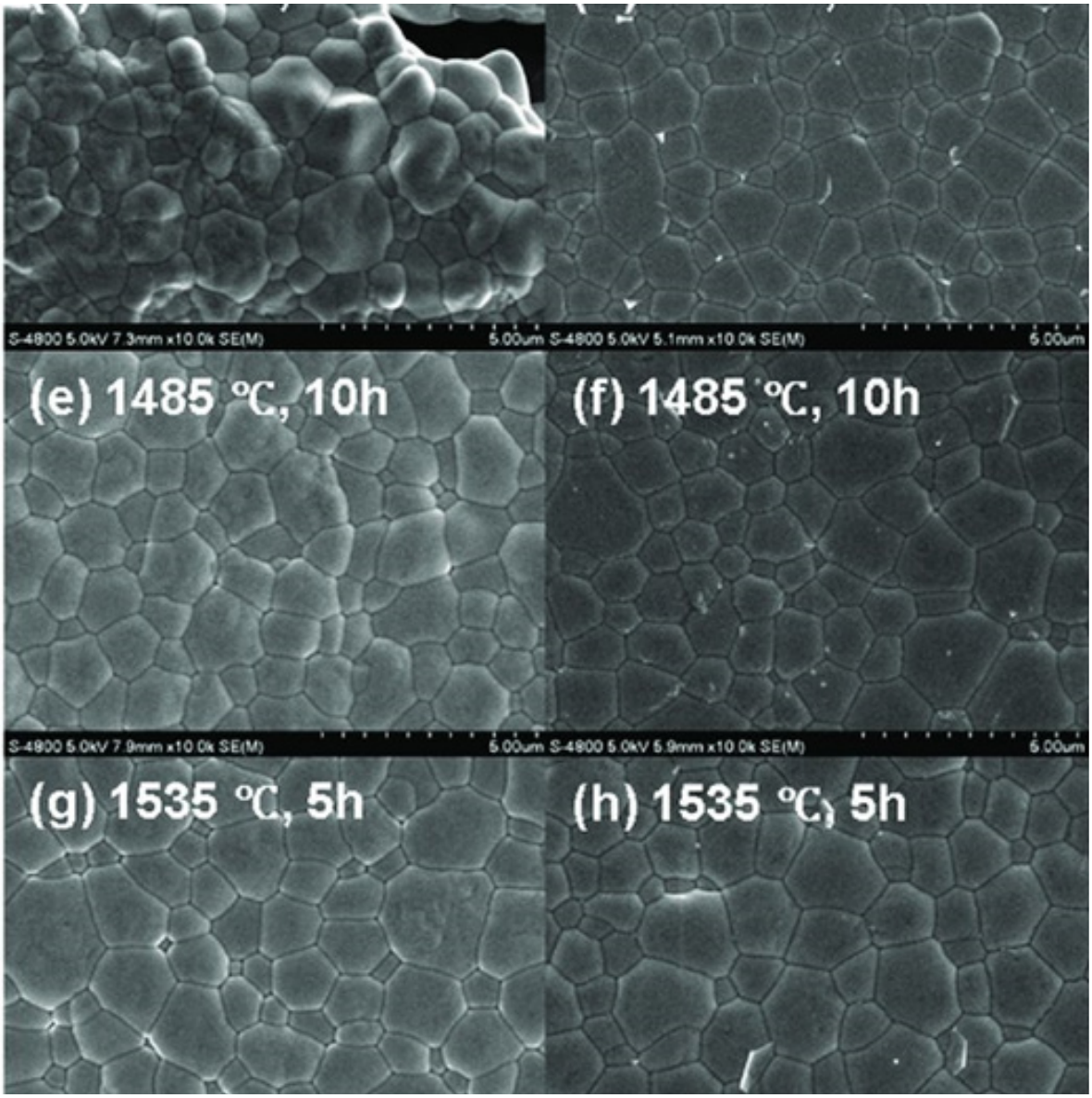
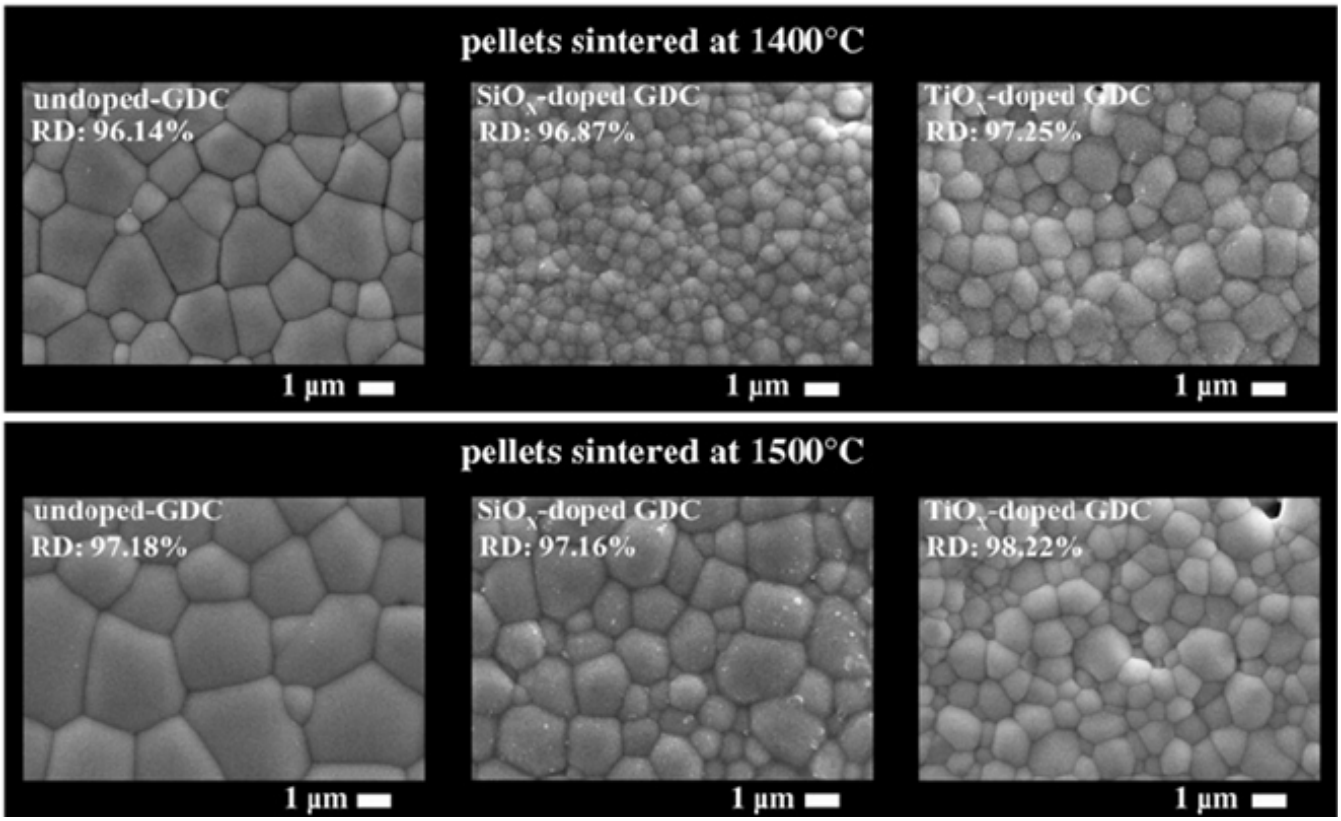


Figure 3: SEM image of sintered pellet of $BaZr0_3$ sintered at 1650°C



The scanning electron microscopic (SEM) studies on the $SrZr0_3$ sintered sample is shown in figure

Conclusion

Nanoparticles of $AB0_3$ (A= Ba, Sr & Ca; B=Zr) have been synthesized using modified single step combustion method for the first time. All the peaks of the XRD are indexed and no peaks are observed other than the expected peaks of the respective nano powders. The analysis results show that they are isostructural and no trace of any impurities. Only the as-prepared powder of $CaZr0_3$ needs an annealing of about 700 °C for 1 h. Their lattice parameters are agreed well with JCPDS values. While annealing the $SrZr0_3$ sample to 600 °C, the crystallinity increases. The scanning electron micrographs reveal a very homogeneous microstructure without agglomerates or exaggerated grain growth. There are no pores which signify the enhancement of density. Fine crystalline nature is observed in transmission electron micrographs [2-12].

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