OPEN ACCESS
Global Journal of Research in Engineering & Computer Sciences
Volume 01| Issue 02 | Nov-Dec | 2021
Journal homepage: https://gjrpublication.com/journals/
Original Research Article

Synthesis and Characterisation of Zirconium oxide

*Dr Sumit Kumar Gupta[1], Rachna Sharma[2] & Pankaj Kala[3]

[1]Assistant Professor Parishkar College of Global Excellence,India
[2]Research scholar SunRise University, India
[3]PGT Physics Rukmani Birla Modern School Jaipur

Submission Date: 20th Dec 2021 | Published Date: 31st Dec 2021

*Corresponding author: Dr Sumit Kumar Gupta

Abstract

Zirconia is a widely used multifunctional material. Its interesting properties, such as high chemical resistance, thermal stability and high mechanical toughness, have turned this material into object of study within fields such as optics, electronics, and magnetism, among others In this article, we report the structural Zirconium oxide (ZrO2) nanoparticles were characterised and synthesised by the wet-chemical processes. Products were characterised using X-ray diffraction (XRD), scanning electron microscopy (SEM) &, transmission electron microscopy (TEM). The crystal structure was determined by XRD. In addition, the formation mechanism of zirconia nanostructures was discussed.

Keywords: X-ray diffraction (XRD), scanning electron microscopy (SEM) &, transmission electron microscopy (TEM).

INTRODUCTION

Presently, Zirconium oxide or zirconia ($Zr0_2$) attracts much interest since their dielectric constants are theoretically predicted to be much higher with values in the range 30-45 (Zhao and Vanderbilt 2002). It is an interesting material in fundamental studies as well as applications-oriented research on account of its remarkable properties like sensible chemical and dimensional stability, high melting point; low thermal conduction and high wear resistance.

$Zr0_2$ exists in three different phases namely room temperature stable monoclinic phase and high temperature stable tetragonal and cubic phases (metastable phases at room temperature). In nature, zirconium dioxide is found in small quantities as the mineral baddeleyite (monoclinic zirconia). The materials are wide employed in the business as thermal and chemical barrier coatings and buffer layers for high temperature superconducting films (Taylor 1992; Atik and Aegerter 1992; Shappir et al. 1986)

Experimental procedure

Synthesis of nanocrystalline $Zr0_2$

Various methods have been formulated to synthesize nanomaterials. The metastable phases of zirconia (tetragonal and cubic), which may be prepared by various wet-chemical processes (Dodd and McCormick 2001; Hu et al. 1999; Cabanas et al. 2001; Clearfield 1964; Mazdiyasni et al. 1966; Roy and Ghose 2000), have attracted much attention due to their scientific and technological importance. The wet-chemical processes area unit advantageous over typical solid-state synthesis in terms of homogeneity and powder characteristics. Success of these wet-chemical processes is based on the nanocrystalline nature of the produced powder and the particle size induced phase transition in $Zr0_2$.

In a typical synthesis, an aqueous solution containing ions of Zr was prepared by dissolving typical amount of high purity ZrOCb.8H20 (99%, CDH, India) in distilled water (- 200 ml) in a glass beaker. Citric acid (99%, CDH, India) was then added to the solution containing Zr ions. Amount of acid was calculated supported total valence of the oxidizing and reducing agents for max unleash of energy throughout combustion. Oxidant/fuel magnitude relation of the system was adjusted by adding acid and ammonium ion hydroxide; and therefore the magnitude relation was unbroken at unity. The ensuing clear answer was heated on a hot plate (at concerning 200±250°C) till it become a viscous answer. The solution boils upon heating and undergoes dehydration accompanied by foam show in Fig-1

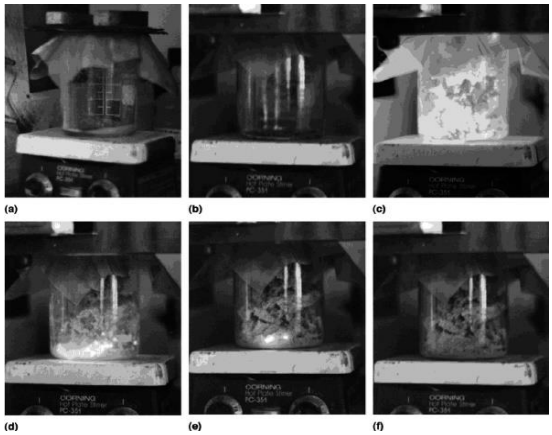

Fig-1: Photographs of a typical nitrate+citrate combustion process for nanocrystalline zirconia powders. (a) Clear starting precursor, (b) Turned into gel, (c) Combustion process, (d) Combustion about to complete, (e) View of the set-up inside the ventilated fume hood, (f) fluffy product from combustion (g) Grey ashes of single phase nanocrystalline ZrO_2.

RESULTS AND DISCUSSION

Structural Characterization of Zr02

X-ray diffraction (XRD) data collection on the single-step combustion-synthesized powder was carried out for phase identification and crystallite size determination using a Bruker D-8 X-ray diffractometer (CuKa radiation, Ni-filter).

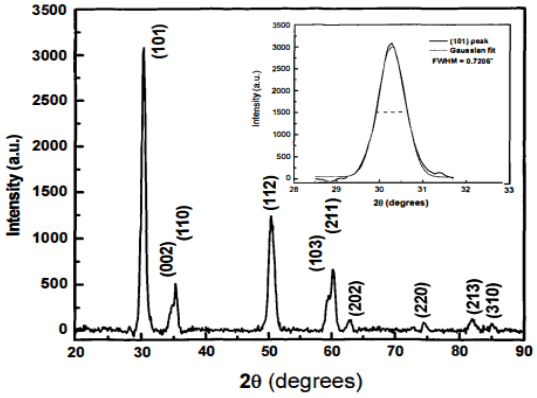

Fig-: 2 XRD patterns of as-prepared Zr02 nano powder sample. A close-up view of (1 0 1) predominant peak in the inset is used to calculate the grain size of the powder

Table-1: Interplanar $d_{hk}l$ spacings and microstrain in the tetragonal structure (t-Zr02). Nanopowder

Peak Position (2θ)	(hkl)	Interplanar Distance (d_{hkl}) (A)	Deviation in d_{hkl} ($\Delta dhkl = d_o - d_s$)	Microstrain
30.2738	(100)	2.9499	-0.0092	-0.0031
34.5578	(110)	5.136412	-0.00475	-0.00228
39.1720	(111)	3.16423	-0.00425	-0.0018
45.3918	(200)	2.14569	-0.00324	-0.00202
53.3876	(211)	1.24587	0.002038	-0.00423
61.6038	(220)	1.12356	0.000112	-0.00302
65.5025	213	1.11324	-0.00011	-0.00201
69.4532	310	1.10231	-0.0006	-0.00101

SEM microstructures of Zirconium oxide nanopowder sample for two different magnifications

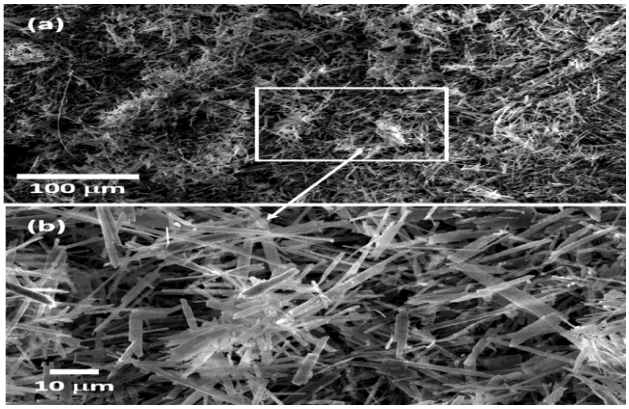

Fig-3: SEM microstructures of Zirconium oxide nanopowder sample for two different magnifications

Energy dispersive spectrum of the as prepared Zirconia powder

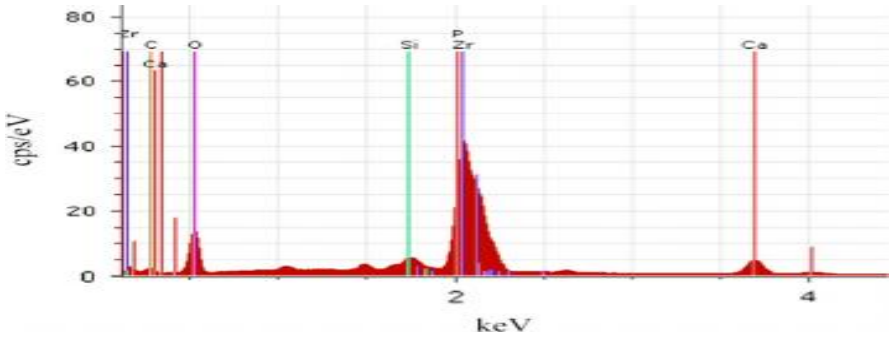

Fig-4: Energy dispersive spectrum of the as prepared Zirconia powder

TEM micro image of the as-prepared Zr02 nanopowder

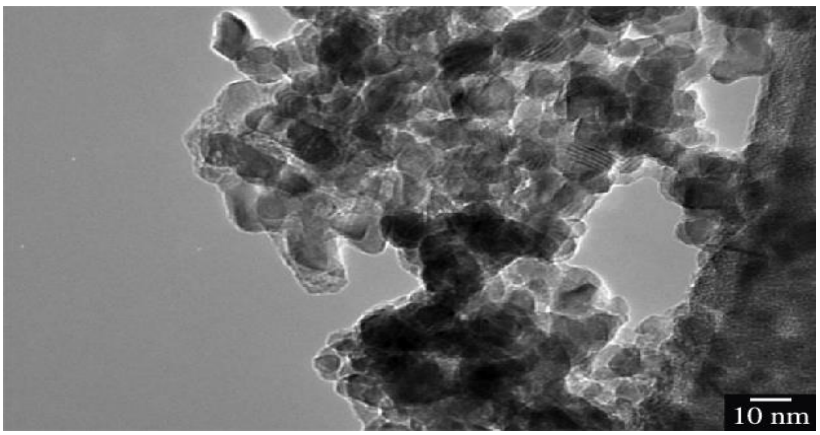

Fig-: 5 TEM micro image of the as-prepared Zr02 nanopowder

CONCLUSION

From the SEM observation, morphology of these agglomerated particles is dispersed and the fracture mechanism appears to be intergranular. The individual grain size range is about l-42µm. The crystallite size observed from TEM was in agreement with the results obtained through X-ray line broadening. The high frequency permittivity of the prepared sample was 28 and the loss factor was nearly O. l 7x 10-4 which supports the usability of the material in dielectric resonators. The EDAX result shows that the as-prepared Zr02 powder sample is stoichiometric, containing considerably equal proportion of zirconium and oxygen. It also indicates that the homogeneous nanosized composite powder could be obtained by this combustion process.

REFERENCE

1. Phillips, J. M. (1996). Substrate selection for high-temperature superconducting thin films. Journal of Applied Physics, 79(4), 1829-1848.
2. Pollet, M., & Marinel, S. (2003). Lowering of CaZrO3 sintering temperature using lithium–calcium fluoride flux addition. Materials Science and Engineering: A, 362(1-2), 167-173.
3. Potdar, H. S., Deshpande, S. B., Patil, A. J., Deshpande, A. S., Khollam, Y. B., & Date, S. K. (2000). Preparation and characterization of strontium zirconate (SrZrO3) fine powders. Materials Chemistry and Physics, 65(2), 178-185.
4. Potdar, H. S., Deshpande, S. B., Godbole, P. D., & Date, S. K. (1993). Synthesis of microcrystalline BaZrO3 via molecular precursor route. Journal of materials research, 8(5), 948-950.
5. Pramanik, P. (1999). A novel chemical route for the preparation of nanosized oxides, phosphates, vanadates, molybdates and tungstates using polymer precursors. Bulletin of Materials Science, 22(3), 335-339.
6. Perng, L. H., Chin, T. S., Chen, K. C., & Lin, C. H. (1990). Y-Ba-Cu-O superconducting films grown on (100) magnesia and sapphire substrates by a melt growth method without crucible. Superconductor Science and Technology, 3(5), 233.
7. Shende, R. V., Krueger, D. S., Rossetti, G. A., & Lombardo, S. J. (2001). Strontium zirconate and strontium titanate ceramics for high-voltage applications: synthesis, processing, and dielectric properties. Journal of the American Ceramic Society, 84(7), 1648-1650.
8. Rammakrishnan, T. V., & Rao, C. N. R. (1999). Superconductivity today. Universities Press.
9. Katahira, K., Kohchi, Y., Shimura, T., & Iwahara, H. (2000). Protonic conduction in Zr-substituted BaCeO3. Solid State Ionics, 138(1-2), 91-98.
10. Kear, B. H., Colaizzi, J., Mayo, W. E., & Liao, S. C. (2001). On the processing of nanocrystalline and nanocomposite ceramics. Scripta Materialia, 44(8-9), 2065-2068.
11. Levin, I., Amos, T. G., Bell, S. M., Farber, L., Vanderah, T. A., Roth, R. S., & Toby, B. H. (2003). Phase equilibria, crystal structures, and dielectric anomaly in the BaZrO3–CaZrO3 system. Journal of Solid State Chemistry, 175(2), 170-181.
12. Iwahara, H., Yajima, T., Hibino, T., Ozaki, K., & Suzuki, H. (1993). Protonic conduction in calcium, strontium and barium zirconates. Solid State Ionics, 61(1-3), 65-69.
13. Koo, J., Kim, Y., & Jeon, H. (2002). ZrO2 gate dielectric deposited by plasma-enhanced atomic layer deposition method. Japanese journal of applied physics, 41(5R), 3043.
14. Hahn, H., Logas, J., & Averback, R. S. (1990). Sintering characteristics of nanocrystalline TiO2. Journal of Materials Research, 5(3), 609-614.
15. Haile, S. M. (2003). Materials for fuel cells. Materials today, 6(3), 24-29.
16. Gömöry, F., Šouc, J., Vojenčiak, M., & Klinčok, B. (2007). Phenomenological description of flux pinning in non-uniform high-temperature superconductors in magnetic fields lower than the self-field. Superconductor Science and Technology, 20(9), S271.
17. Buchanan, R. C., & Park, T. (1997). Materials crystal chemistry. CRC Press.
18. Büchel, K. H., Moretto, H. H., & Werner, D. (2008). Industrial inorganic chemistry. John Wiley & Sons.