ID: 5012

Use of Sol-Gel Process to Obtain Isotropic Properties of a Titanate Pyrochlore

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ABSTRACT

A $(Ce_{0.5}Y_{0.5})_2Ti_2O_7$ pyrochlore titanate was synthesized by the dry process, and its physicochemical properties were studied. In order to improve the isotropy characteristics , the material was synthesized by the sol-gel process using urea and hexa-methylene-tetramine (HMTA), as a dispersant in the liquid phase, the ratio nHMTA-urea/nM to the number of moles of cations (referred to as M) is set at 1.2,meaning that (nHMTA/nM=1.2). Archimedes density of the ceramic gave a value of 3.146 g/cm^3. Phase Identification by X-ray diffraction (XRD) showed a predominance of the pyrochlore phase, identified by comparison with JCPDS standard compound. Fourier Transform Infrared (FTIR) Spectroscopy revealed an absorption corresponding to the Ti-O elongation bond appearing at 417 cm^{-1} and 514 cm^{-1}, and a broad absorption band in the 450-600 cm^{-1} range, due to the elongation mode of the Y-O, Ce-O and Ti-O bonds. Analysis by Scanning Electron Microscopy (SEM) has enabled us to assess the microstructure, the particles are well agglomerated and give a compact appearance with good densification, EDS analysis confirms the presence of the $(Ce_{0.5}Y_{0.5})_2Ti_2O_7$ phase.

Keywords: Sol-Gel, Pyrochlore, XRD, SEM, FTIR.

1 Introduction

Pyrochlores (Fd3m, Z=8),presented by $A_2B_2O_7$ chemical formula, are a deformation of the fluorite structure (Fm3m, Z=8) [1]. They have attracted significant attention due their interesting properties such as, high melting point, low thermal conductivity and high resistance to irradiation, which select them as matrices for immobilization of (HLW) [2]. Pyrochlores, have been synthesized by many techniques, including solid state reaction, hydrothermal synthesis, which have their own advantages but also some drawbacks [3]. The sol–gel method offers considerable advantages of good mixing of the starting materials, excellent chemical homogeneity and phase purity of the synthesized powders [4]. The aim of this study is to improve the isotropic properties of a pyrochlore with $(Ce_{0.5}Y_{0.5})2Ti_2O_7$ chemical formula, previously synthesized by solid state reaction at 1350°C for 12 hours, by sol-gel process.

2 Experimental

A $(Ce_{0.5}Y_{0.5})_2Ti_2O_7$ pyrochlore: was synthesized by sol- gel method. The following reagents were employed: $Ce.N_3O_96H_2O$ (ACROS ORGANICS, 99.5%), $Y.N_3O_9.6H_2O$ (ACROS ORGANICS, 99.9%), urea CH_4N_2O(ALDRICH,>99%), (HMTA) hexa-methylene-tetramine, ($C_6H_{12}N_4$ SIGMA), $Ti(NO_3)$ (MERCK, 99.9%). All reagents are dissolved separately in ultra-pure water, and stirred for two hours. Emulsifiers, such as (HMTA) and urea, are also added to aqueous solutions, setting the molar ratio n HMTA-urea/n M = 1.2. Ultrapure water is heated to 90°C. The precursors $Ce.N_3O_9.6H_2O$,$Y.N_3O_9$. $6H_2O$, $Ti(HNO_3)$, HMTA and urea, are cooled down between 4 and 6°C, overnight. They are placed in separating funnels at the moment of the chemical reaction. The solutions are slowly mixed drop by drop, under strong agitation to ensure good dispersion, it's the solution forming. After 24 hours of reaction in solution, 48 hours of washing and PH adjustment, then 24 hours drying of the resulting gel, the dry substance was then ground.

DOI: 10.21467/abstracts.170 ISBN: 978-81-970666-1-0 (eBook)

Calcination was carried out in a Carbolite BLF 1800 furnace. The gel was first preheated at 350°C for 4 hours, and then Calcined at 600°C, The resulting powder is compacted in 13 mm diameter pellets of various heights. The pellets are sintered at 1100°C for 10 hours. The powders densities (ϱA) are measured by the Archimedes method. The phase identification is carried out by X-ray diffraction (XRD) analysis, with a Philips X'Pert PRO apparatus. Phases identification is performed using a Philips X'Pert plus 2004 software [5]. The scanning electron microscopy (SEM) observations, was conducted on a Philips XL30 microscope. The energy-dispersive spectrometer (EDS) was used for elemental analyses. The FTIR analysis is conducted using a 380 NICOLET spectrometer. The spectral range is from 4000 to 300 cm^{-1}. Spectral processing is performed using OMNIC software [6].

3 Results and Discussion

Archimedes density is ranging between (2.01-4.510) g/cm^3, it's due to the growth of bonds, between the grains and the reduction of pores. XRD analysis shows that the TiY_2O_5 structure is beginning to form, along with a $TiYO_3$ perovskite phase and mixed Ce-Y oxide phases. Pyrochlore is micrographed in (SE) and (BSE) modes. The (SE) micrographs are made up of irregularly shaped agglomerates formed by nanomitric grains (figure 1.).Micrographs in (BSE), show the presence of three phases (figure 2.) already found by XRD. EDS analysis confirms the presence of the $(Ce_{0.5}Y_{0.5})_2Ti_2O_7$ phase.

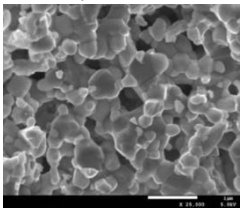

Figure1 : Micrographie en (SE)

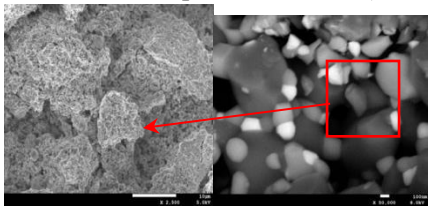

Figure2 : Micrographie en (BSE)

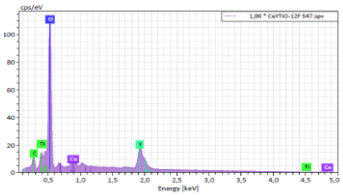

Figure3 : Global (EDS) analysis

FTIR analysis revealed the vibrations of the Ti-O elongation bonds at 417 cm^{-1} and 514 cm^{-1}. A broad absorption band in the interval (450-600 cm^{-1}), due to the elongation mode of the Y-O, Ce-O and Ti-O metal bond.

4 Conclusions

A $(Ce_{0.5}Y_{0.5})_2Ti_2O_7$ pyrochlore was synthesized by sol-gel process, urea and (HMTA) are used as dispersants. The HMTA-urea ratio is fixed at 1.2. The obtained Archimedes' density is 4.510 g/cm^3. Both XRD and SEM analysis identified three main crystalline phases, rutile (TiO_2), ($Ce_{0.9}O_{1.95}Y_{0.1}$), finally pyrochlore ($Y_2Ti_2O_7$). FTIR analysis revealed vibrations of the Ti-O , Y-O and Ce-O elongation bonds.

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ISBN: 978-81-970666-1-0

Series: AIJR Abstracts

DOI: 10.21467/abstracts.170