



# An investigation on the influence of milling time and calcination temperature on the characterization of nano cerium oxide powder synthesized by mechanochemical route

M. Aminzare<sup>a,\*</sup>, Z. Amoozegar<sup>b</sup>, S.K. Sadrnezhad<sup>c</sup>

<sup>a</sup> Materials Engineering Department, Science and Research Branch, Islamic Azad University, Tehran, Iran

<sup>b</sup> Department of Materials and Metallurgical Engineering, Iran University of Science and Technology (IUST), Tehran, Iran

<sup>c</sup> Department of Materials Science and Engineering, Sharif University of Technology, Tehran, Iran

## ARTICLE INFO

### Article history:

Received 22 July 2011

Received in revised form 11 June 2012

Accepted 14 June 2012

Available online 13 July 2012

### Keywords:

A. Ceramics

B. Chemical synthesis

C. Electron microscopy

## ABSTRACT

The synthesis of nano-sized CeO<sub>2</sub> powder was investigated via mechanochemical reactions between hydrate cerium chloride and sodium hydroxide as the starting materials. The process was followed by a subsequent calcination procedure. Characterization of as-synthesized powder was performed using X-ray diffraction, FTIR spectroscopy, Brunner–Emmett–Teller (BET) nitrogen gas absorption, scanning electron microscopy (SEM) and particle size analyzer (PSA). The precursors were milled for different milling times and then were subjected to different heat treatment procedure at variable temperatures from 100 to 700 °C. According to the results, milling time and calcination temperatures induce paramount effects on crystallite size and surface area of as-synthesized powders. In addition, the average activation energy for the growth of nanocrystals during calcination was determined to be about 12.53 kJ/mol, suggesting the influence of interfacial reactions on the crystallite growth during the calcination procedure.

© 2012 Elsevier Ltd. All rights reserved.

## 1. Introduction

Nanostructures and nanomaterials have significantly different physical or chemical properties compared to their ultrafine or micron sized counterparts due to their greater surface area which provides them with a higher reactivity. These fantastic properties have attracted interests in nano materials for researchers and investigators [1].

Cerium dioxide is an attractive oxide ceramic due to its interesting properties such as good chemical stability as well as high relative permittivity. This ceramic material represents a fluorite like cubic structure with the space group *fm3m* [2]. The very noteworthy issue, in this regard, is the absence of any reports in the literature relative to the crystallographic transformations through the heating procedure of this material from room temperature up to 2700 °C (ceria melting point) [3]. The aforementioned valuable properties have resulted in the widespread exploitation of ceria for many applications in various fields such as capacitors, hydrogen storage materials, ultraviolet absorbers, buffer layers of superconducting materials as well as

catalysts [4–6]. Since ceria is a convenient solvent for the high solid solubility of different cations, it has been utilized accompanied by numerical foreign cations such as Gd, La, Ca, Mg, Sm, Y and Zr as the dopant material in many different fields including gas sensors, fuel cells, oxygen pumps and automobile exhaust catalysts [6–13].

Production of ceria nanoparticles has been extensively investigated, since these materials display unique properties as a typical result of the size reduction to nanometer range. For instance, one of the most important variations, resulted from shifting the size to the nanometer range is the transformation of atomic structure and broadening of band gap in nano ceria [14]. Several synthesis techniques including solid state reaction [15], polyol method [16], electro-synthesis [17], gel-combustion [18], hydrothermal [19,20], precipitation [21,22], composite-hydroxide-mediated (CHM) approach [23] and other methods [24,25], have been proposed to prepare nanosized cerium dioxide powders. The main factors governing the size of the obtained powders are synthetic route, pH gradient, environmental conditions, temperature, pressure, and time [15–25]. In other words, several parameters may be influential on the variations in size and morphology of synthesized ceria nano powder resulting in distinct properties in the final product.

Mechanochemical synthesis is one of the promising methods to prepare nanosized materials. This method which combines the benefits of both chemical and physical routes, has the potential to

\* Corresponding author. Tel.: +98 9109330176; fax: +98 21 77240291.

E-mail addresses: [masoudaminzare@yahoo.com](mailto:masoudaminzare@yahoo.com), [masoudaminzare@gmail.com](mailto:masoudaminzare@gmail.com) (M. Aminzare).