STUDIES REGARDING ELABORATION AND CHARACTERIZATION OF CeO_2 POWDERS

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***Abstract**: This paper describes the synthesis and the characterization of CeO_2. The preparation of high surface area metal oxide is currently the subject of extensive research due to their multiple applications in different areas (electronic devices, sensing, biomedicine, catalysis, etc.). SPVD – Solar Physical Vapor Deposition offer the possibility of an innovative oxide elaboration. XRD spectra performed on initial targets and on the nanopowders obtained have been determined. Scanning Electron Microscopy (SEM) observations support the results.*

***Keywords**:* SPVD, oxide, nanopowder.

INTRODUCTION

CeO_2 has attracted much attention for its potential applications in electronic ceramic, ultra-precise polishing, gas sensor, catalysis, solid oxide fuel cells (SOFC) and so on [1–3]. For example, CeO_2 is one of the abrasive materials for chemical–mechanical polishing in advanced integrated circuits [3]. Because of its excellent ionic conduction, doped-cerium oxide nanoparticles are promising electrolyte materials for SOFC [2]. The capacity of the modern automotive exhaust treatment catalysts containing CeO_2 is much more effective than that of the predecessors due to its high "oxygen storage capacity" [1]. Therefore, the extensive synthesis of CeO_2 becomes an urgent task for further research and applications. Many synthesis approaches, such as chemical solution precipitation, sol–gel method, microwave-assisted hydrothermal processes, mechano-chemical processing, polyvinyl pyrrolidone (PVP) solution route, electrochemical synthesis, combustion method, direct sono-chemical route and gas–liquid co-precipitation have been used for preparation of CeO_2. Several works have investigated the methods for producing fine cerium oxide powder. In the specific literature some researchers synthesized the cerium oxide with the particle size range between 30 and 50 nm via hydrothermal process. Hanawa et al. synthesized a nano-grade of cerium oxide by simultaneously introducing cerium nitrate and ammonia into a container, then injecting steam at 100^0C and stirring [4]. The powder formed by that process had a grain size in the range 10–80 nm. Bondioli et al. prepared the nano-grade cerium oxide by the flux method, adding cerium ammonium nitrate to eutectic mixture of molten salts [5]. Those products had the particle size range in 10–20 nm. Wang and Lu used cerium nitrite and urea as reactants via hydrothermal process to form cerium hydroxide carbonate [5]. The cubic structure cerium oxide was synthesized by further calcinations of that intermediate at 500^0 C. Some special reacting conditions, such as high temperature, high pressure, capping agents, expensive or toxic solvent have been involved in the preparation of CeO_2. In this contribution, we present a successful example of synthesis of CeO_2 nanoparticles using a new technique such as Solar Physical Vapor Deposition. Observations by scanning electron microscopy (SEM) are useful methods to achieve the studies made by XRD.

EXPERIMENTAL

1.Elaboration in a solar reactor or solar physical vapor deposition (SPVD)

The solar physical vapor deposition or solar PVD (SPVD) is an original process to prepare nanopowders. Pure ZnO, $Zn_{1-x}Al_xO$, $Zn_{1-x}Co_xO$, $(Zn_{1-x}Bi_xO)$, $Bi_{1-y}Zn_y)_2O_3$ and metallic Zn nanophases have been prepared by this method. The parameters such as melting point 2400 °C and boiling point

3500 °C permit o synthesize the nanopowders by this method. Each of the 12 solar furnaces built at Odeillo/FontRomeu around 1970 are constituted by mobile plane mirrors (‘‘heliostats’’) tracking the sun and reflecting the radiation on a parabolic concentrator. At the focus, is placed a cooled holder or a more or less sophisticated reactor (see Fig. 2). The reactors are constituted by glass balloons. Inside, a target, made with the material to be melted and vaporized or sublimated (depending on its nature), is placed on a cooled support at the focus of the concentrator. The vapor pressure around the target depends on the material and on the atmosphere inside the balloon; it is more or less large. In the last case, a smoke is generally visible and is depositing by condensation on a cold finger (water cooled copper tube). In a similar reactor, the smoke is collected on a metallic or a nanoporous ceramic filter through which the gas atmosphere is flowing.

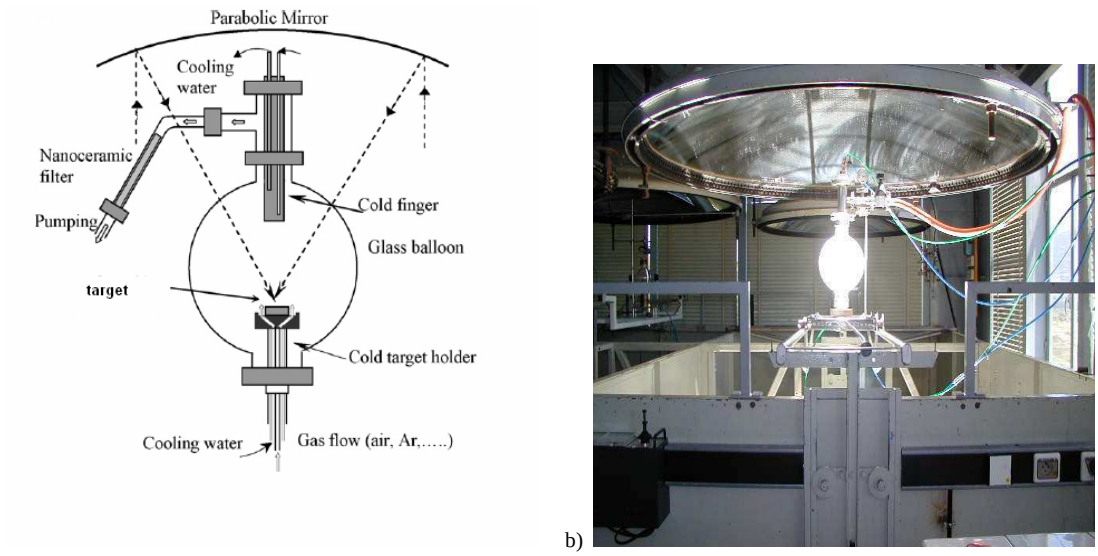


Fig. 1. a) Solar physical vapor-deposition process in the “helitron” solar reactor; b) view of the “helitron” solar reactor

The most recent reactor, the ‘‘heliotron’’, associates the two processes (see Fig.1): collection by a cold finger and pumping through a filter, which decreases the phenomena of condensation on the walls of the balloon and increases the effectiveness of the collection. The precursors, called target, are the powder of commercial CeO_2. The nanopowders are collected by scraping the deposit formed on a cold finger (water cooled copper tube), or by trapping them on a nanoceramic filter through which the smokes produced are pumped. Fig. 1a and b shows the more recent solar reactor of this type, called “heliotron”.

The CeO_2 nanopowders are collected by aspiration on a nanoporous ceramic filter and by condensation on a cold finger. The production rate of the nanopowders depends on the vapor pressure of the material. The synthesis parameters(pressure, solar flux, reactor position) are presented in table 1.

Table 1. Synthesis parameters

SAMPLE /CODE	P(pressure)	F(solar flux	H_0(reactor position)
GP_1	5hPa(air)	964W/m^2	63mm
GP_2	5hPa(air)	950W/m^2	63mm
GP_3	20hPa(air)	982W/m^2	63,2mm
GP_4	60hPa(air)	1010W/m^2	63mm
GP_5	100hPa(air)	1010W/m^2	63,5mm

2. Characterization

The synthesized CeO2 was characterized by XRD with X'Pert MPD Pro diffractometer. The phase and structure of CeO_2 were identified by powder X-ray diffraction analysis with Cu Kα radiation (30 kV, 30 mA). The XRD specters are represented in figure 2.

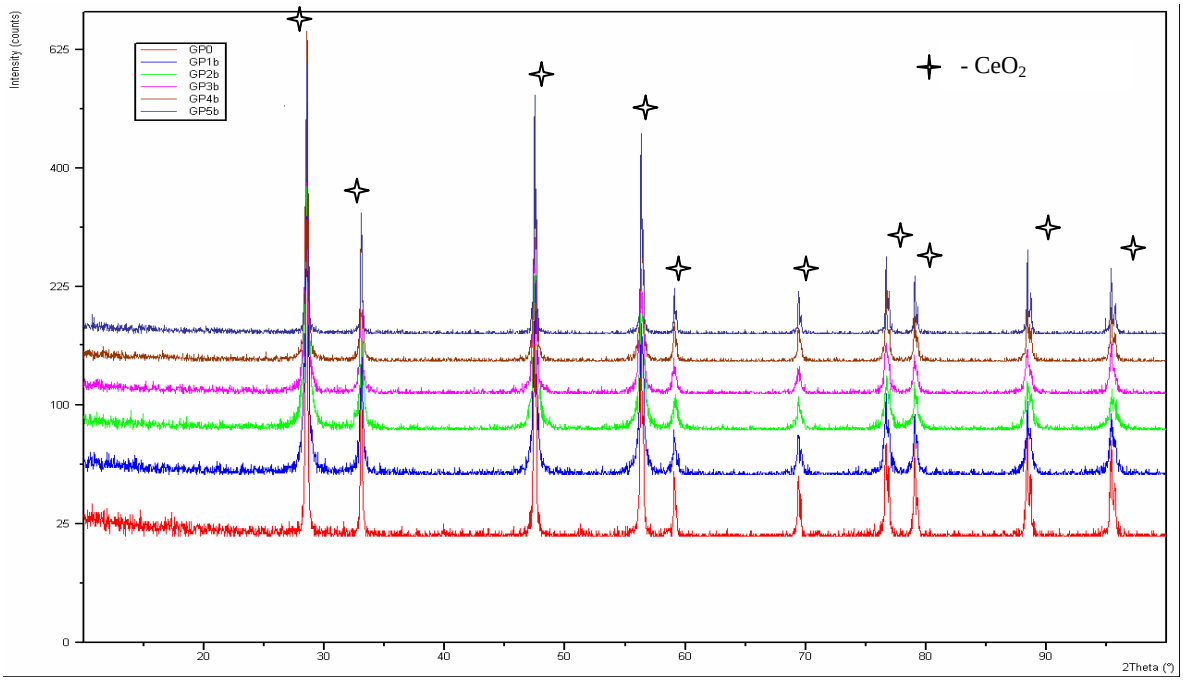
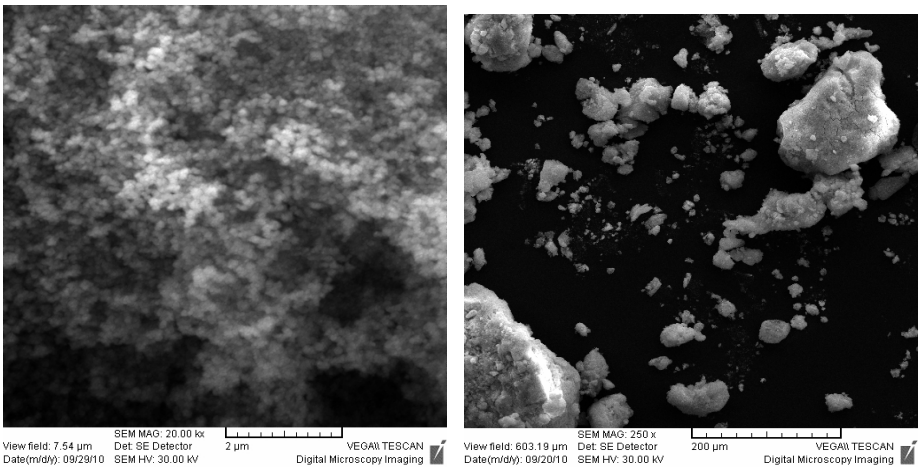


Fig 2. XRD specters of CeO_2 prepared by SPVD

The diffraction peaks show that the particles were the cubic structure of cerium oxide according to fiche ASTM nr. 01-075-9470. As a first approximation, they are in agreement with literature data for pure CeO_2: cubic structure, a=b=c=5,4113, space group Fm-3m). The Scanning Electron Microscopy with TESTAN VEGA II, LMU is more adapted for the observation of this samples because of the depth of the reaction field and increases the possibility of observing the most important morphologies. The morphology of cerium oxide shows that the spherical and polycrystalline agglomeration like in figure 3.



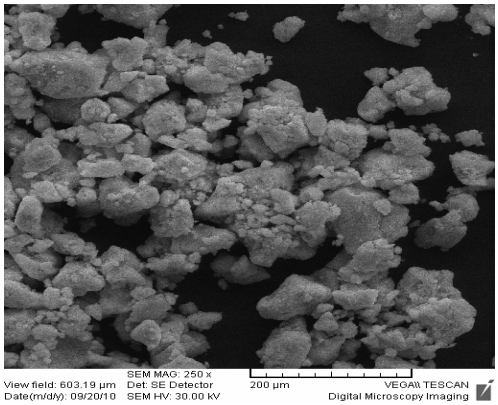


Fig. 3. Morphologies of CeO_2 powders obtained by SEM

Electron Microscopy observations are in agreement with the XRD analysis. This morphologies show many agglomerations appear in purenanopowders prepared by SPVD due to the nanometric rage of particle.

Conclusions

Powders of pure CeO_2 have been prepared by an original method (SPVD) and their nanostructure was characterized by XRD and SEM. The sintering of the nanopowders, producing a massive nanomaterial, will lead the possibility of studding the electrical and anti-corrosive properties of this oxide. The nanocomposites obtained from CeO_2 and SiO_2 mixtures appear to be promising materials for applications such as preparing a new solid electrolyte for SOFC. Nevertheless, new investigations are needed.

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