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SYNTHESIS OF NANO-PARTICLES OF CeO_2 BY AUTO-COMBUSTION METHOD

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ABSTRACT

Nano-particles of CeO_2 with the smallest average particle size (from 10 to 20 nm) and uniform morphology, well- dispersion, especially with single crystal of CeO_2 were obtained from a mixed solution of $Ce(NO_3)_3$, citric acid, polyvinyl ancohol by auto-combustion method. The synthesized CeO_2 powders were characterized as the crystalline phase identification by X-ray diffraction (XRD), the morphology and particle size by trasmission electron microscopy (TEM), the single crystal of CeO_2 was determinated by selected area electron diffraction (SAED).

I - INTRODUCTION

Application of materials depends on their size and morphology, therefore, much emphasis has been laid on the size and shape control recently [1, 2]. Research has proved that organic molecules containing functional groups such as N, O, S atoms are able to adjusting the size and morphology of inorganic oxides by coordinating with inorganic oxides or absorbing on the surface of inorganic oxides. Especially, organic molecules containing functional groups O atoms can be bonded on the surface of the inorganic oxides by hydrate bonds the absorption or attraction of electrons. And there are no impurities after the precusor is calcined at a high temperature. So, there are potential values exploiting the organic molecules with functional group O atoms to control the size and morphology of particles [3].

There is a demand for advanced ceramic materials made with ultrafine powers for increasing performance. The use of monodispersed nano crystalline powders as starting materials has considerable potential for improving the properties of subsisting ceramic composition. Ceria is used in many fields (for example, solid electrolytes in solid oxide fuel cell, catalysis, optical additive, comsmetic materials and polisher for chemical mechanical planarization [4].

Several techniques have been developed for preparing nanosized pure CeO_2 or cation- doped ceria particles that yield homogenous and high-density sintered bodies as hydrothermal, sol-gel, co-precipitation, homogenous deposition… However, the technique of these methods is difficult and the equipment is expensive, so it is impossible to produce ultrafine CeO_2 particles with the methods in the industry [5].

In this work, we focused on the shape and size control of CeO_2 particles by auto-combustion method. The process exploits the advantages of cheaper precusors, a simple preparation method and a resulting ultrafine, homogenous powder [6].

II - EXPERIMENTAL

$Ce(NO_3)_3.6H_2O$ (Merck) was a source of Ce^{3+}, polyvinyl alcohol (Peking) was chosen as an adjusting agent of particle-size and morphology, citric acid ($C_6H_8O_7.H_2O$) (Merck)

was chosen as a ligand and a determinant factor in the formation of the sol-gel.

Several experimental were carried out to find out the optimal condition for the preparation of CeO_2 powders, we found that the weight of Ce^{3+}, citric acid and polyvinyl alcohol affected not only the particle size but also the morphology of CeO_2 powders. And base on the received data, optimal condition was as : the aqueous solution of $Ce(NO_3)_3$, citric acid, polyvinyl alcohol were dissolved with the molar ratio of Ce^{3+} to citric acid was 1:3, the weigh ratio of $Ce^{3+}(NO_3)_3$ to polyvinyl alcohol was 10:3.

The mixed solution was evaporated by continous stirring at temperature of 80 - 90°C on a sterrer. After about two hours of evaporation, the clear solution turned milky. And gel was formed within 30 minutes. The gel was translucent with a honey-like color and viscosity.

The gel was dried at 150°C and ignited immediately when it was dry. Some different types of thermal decomposition started at the hottest zone and a ring-shape reaction zone move to the center of the crucible. During the combustion of the gel, much brown gases (probably NO_x and CO_2) were released. After 10 to 15 minutes later, yellow CeO_2 powders were obtained.

The phase of CeO_2 powders were identified by X-ray diffraction (XRD, D8 ADVANCE, Buker- German) using CuKα radiation. The size and morphology of the synthesized particles were determinated by transmission electron microscopy (TEM, JEM-1010, JEOL, Japan). Single crystal of CeO_2 was identified by selected area electron diffraction (SAED, JEM-1010, JEOL, Japan).

III - RESULTS AND DISCUSSION

Fig. 1 shows that the XRD pattern of synthesized products, there are typical diffraction peaks of crystalline CeO_2 in the pattern. This is clearly show that synthesized products were CeO_2 crystals and synthesized CeO_2 have cubic structure and exists as single phase.

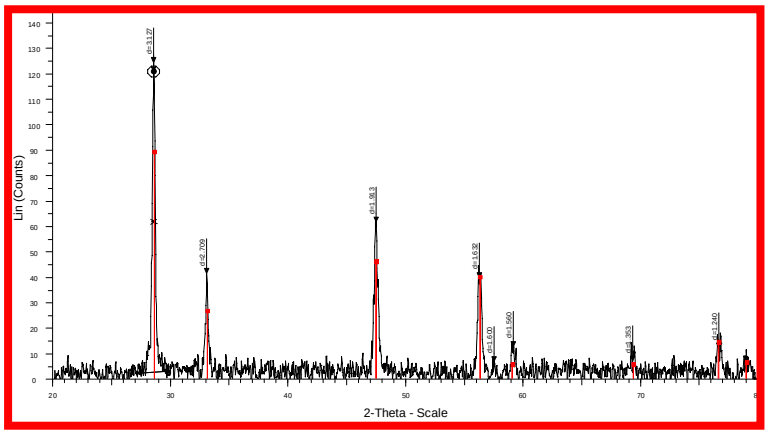

Fig. 1: XRD pattern of synthesized products

TEM image of CeO_2 particles were shown in Fig. 2. The results show that synthesized CeO_2 powders have uniform morphology, well-dispersion and particles have nanosize (about 10- 20 nm).

The results show that an appropriate quantity of polyvinyl alcohol can be used to control the particle- size and morphology of CeO_2powders assisting citric acid. Because polyvinyl alcohol has a large molecular weight and a long chain structure, it can be adsorbed on the surface of CeO_2 to perform a dispersive and stable action and spatial obstruction could defend the particles from aggregation.

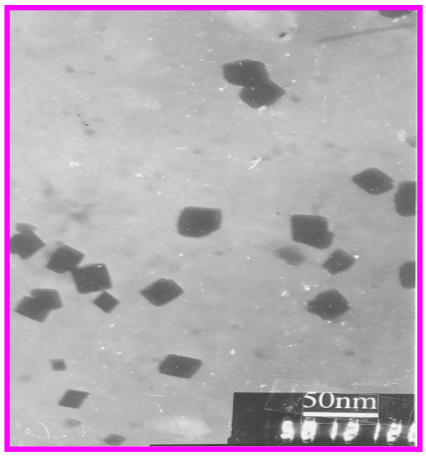

Fig. 2: TEM image of CeO_2 particles especially, synthesized powders have single crystal of CeO_2(as shown in Fig. 3)

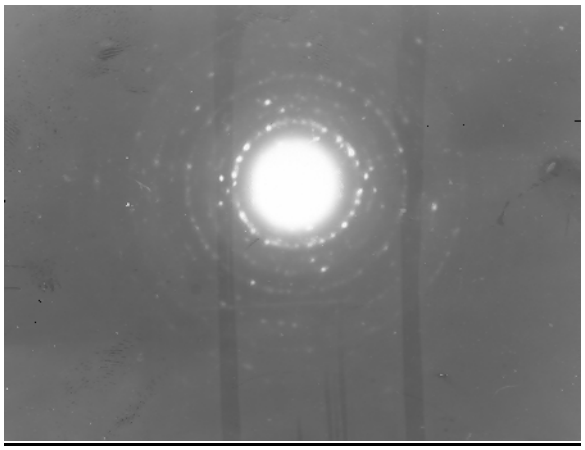

Fig. 3: Image of selected area electron diffraction of CeO_2

IV - CONCLUSIONS

Well-crystallized and monodispersed nano-ceria powders were obtained by auto-combustion method using citric acid as a ligand and determinant agent for the formation of the sol- gel and polyvinyl ancohol as adjusting agent of particle- size and morphology.

It was concluded that the cubic structure was only existed in sample from result by XRD without other phases. Especially, the synthesized CeO_2 powders were nanosized particles (about 10-20 nm) and the existing as single crystal of CeO_2 was identified by selected electron diffraction.

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