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Preparation of CeO_2 Nanoparticles by Using Mesoporous Active Carbon as Template

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CeO_2 nanoparticles were prepared by using mesoporous active carbon as template. The prepared CeO_2 nanoparticles were cubic phase with an average size of 8 nm and narrow distributing. The results indicate that the mesoporous active carbon template plays an important role in preparing CeO_2 nanoparticles.

Key Words: CeO_2 nanoparticles, Mesoporous active carbon.

INTRODUCTION

In recent decades, nano-sized cerium dioxide (CeO_2) has been widely studied and applied in optics[1], electrochemistry[2], oxygen sensors[3], solid oxide fuel cells (SOFCs)[4] and three-way catalysts (TWC)[5] due to its oxygen vacancies with easy reducibility and high mobility, high oxygen storage capacity (OSC) and electronic conductivity. For these applications, it is of critical importance to regulate the size of the particles. To date, several methods of preparing CeO_2 nanoparticles, including hydrothermal[6], reversed micelles[7], co-precipitation[8], electrochemical[9], solvothermal[10], solid-state reactions at ambient temperature[11] and sol-gel[12], have been developed. To the best of our knowledge, the synthesis of CeO_2 nanoparticles by using mesoporous active carbon as template has not been reported. Herein, we report a novel route for the preparation of CeO_2 nanoparticles by using mesoporous active carbon as template and urea as precipitator.

EXPERIMENTAL

All the reagents are of analytical purity, obtained from Shanghai Chemical Reagent Ltd. Co. of China and used without further purification. In a typical procedure, CeO_2 nanoparticles were prepared as follows: 5.0 g $Ce(NO_3)_3 \cdot 6H_2O$ was dissolved in 100 mL distilled water in a beaker and then 8.0 g mesoporous active carbon was added into the solution slowly under vigorous stirring. Then 2 g urea was added into the solution and

heated at 90°C for 1 h. The precipitate was filtrated, washed several times with hot distilled water, dried at 100°C for 12 h and calcined at 550°C for 5 h.

RESULTS AND DISCUSSION

The X-ray diffraction (XRD) pattern of the samples is shown in Fig. 1. All of the reflection peaks can be readily indexed to a pure cubic phase of CeO_2 with lattice constant a = 0.5411 nm, which agrees well with the reported data (JCPDS card no. 81-0792). No other impurity peaks are detected. The XRD pattern suggests that the reflections are very board, which indicates that the crystallites are relatively small. The average size of CeO_2 nanoparticles is about 8 nm calculated by the Scherrer equation.

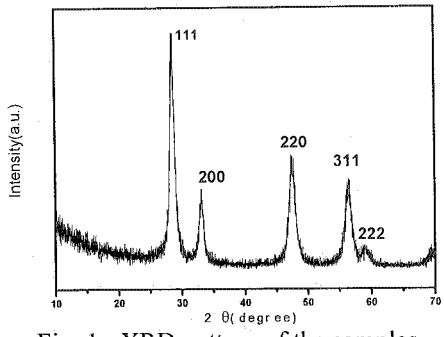

Fig. 1. XRD pattern of the samples

The general morphology of the samples is investigated by transmission electron microscopy (TEM) and shown in Fig. 2a. It reveals that the most of the samples are nanoparticles with an average size of 8 nm and narrow distributing. Electron diffraction (ED) pattern (Fig. 2b) is consistent with cubic phase CeO_2. The perfect diffraction pattern and lattice fringes indicate that the individual CeO_2 nanoparticles are well-crystallized. The HRTEM image (Fig. 2c) reveals that the lattice space is 0.312 nm, which agrees with the (111) lattice plane of cubic phase CeO_2. The ED pattern and the HRTEM image indicated that the samples are cubic phase CeO_2, which is constant with the result of XRD.

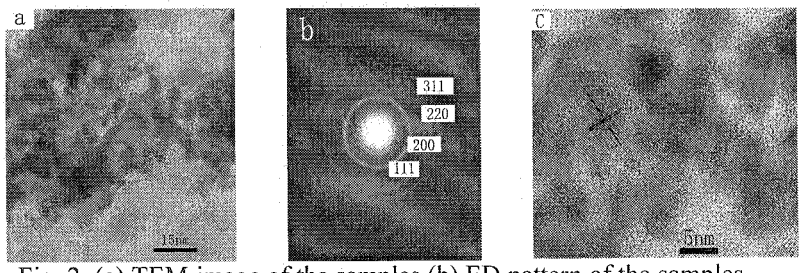

Fig .2. (a) TEM image of the samples (b) ED pattern of the samples (c) HRTEM image of the samples

Further evidence for the quality and composition on the surface of the samples are obtained by X-ray photoelectron spectroscopy (XPS) and the XPS spectra of CeO_2 nanoparticles are identified. The XPS spectra (Fig. 3) indicated that there are no other elements or impurities except for Ce and O on the surface of the samples. The binding energy of Ce $3d_{5/2}$, Ce $3d_{3/2}$ (Fig. 3b) and O 1s (Fig. 3c) are about 882.8 eV, 900.8 eV and 531.5 eV, which indicates that the oxygen element exists in the form of O^{2-} and cerium element exists in the form of Ce(IV)[13].

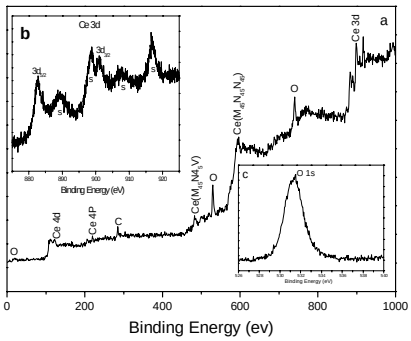

Fig. 3. XPS analysis of the samples (a) survey spectrum (b) Ce 3d spectrum (c) O 1s spectrum.

As shown in Fig. 4, the specific surface area of the mesoporous active carbon is 1677 $m^2 \cdot g^{-1}$ caculated by the Braunauer-Emmett-Teller (BET) method. The medium pore size is about 3.608 nm. When the above solution was heated at 90ºC, the precipitate was formed. The part of the precipitate was in the pore of the mesoporous active carbon, while most of which were on the surface of the mesoporous active carbon. The thickness of the precipitate was quite thin and the precipitate dispersed well due to the large surface area of the mesoporous active carbon. When the samples were calcined at 550ºC, the mesoporous active carbon was changed to CO_2 air current, which prevented from the aggregation of CeO_2 particles during calcinations. The large surface area and CO_2 air current play an important role in the formation of small homogeneous nanoparticles.

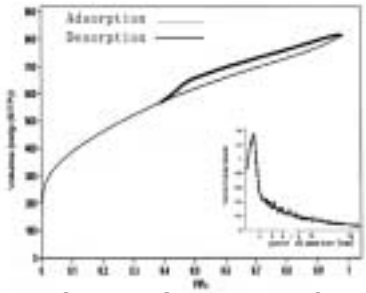

Fig. 4. Nitrogen adsoption-desorption isotherm and the pore size distribution curve of mesoporous active carbon

In order to further investigation, the effect of the mesoporous active carbon on the preparation of CeO_2 nanoparticles, a similar experiment was carried out. All the procedures were the same except for the using of the mesoporous active carbon. The products are cuboid with the average size 500 nm × 500 nm × 1.5 μm (Fig. 5).

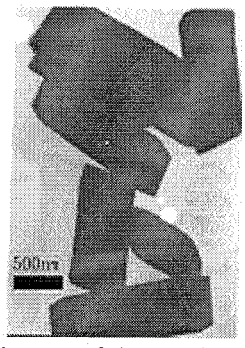

Fig. 5. TEM image of the products prepared without mesoporous active carbon

Conclusion

CeO_2 nanoparticles with an average size of 8 nm and narrow distribution have been prepared successfully by using mesoporous active carbon as template. The results indicate that the mesoporous active carbon template plays an important role in preparing CeO_2 nanoparticles. This route is simple and low cost, which offers a great opportunity for scale-up preparation for industry application and a novel approach preparing other nano-oxides.

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