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# Preparation of inverse opal cerium dioxide for optical properties research

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#### 1. Introduction

Since Yablonovich theoretically showed the ability of three dimension periodic dielectric materials to possess a photonic band gap in 1987 [1], photonic band gap materials have become an important and popular area of research. The strong interest in photonic band gap materials stems from their potential to confine and control the propagation of light with minimal losses. Inverse opal structure, as an important three dimensional periodic dielectric material, has great promising applications in optical communication, photonic computing, switching, lasing, solar cells, etc. [2–4].

Many methods have been invented to prepare inverse opal [5–8]. Among them, the sol–gel method is very prevalent, economic and convenient to prepare inverse opal. A series of materials, including TiO<sub>2</sub> [8], SiO<sub>2</sub> [9], ZnO [10], ZnS/ZnO doped SiO<sub>2</sub> [11], etc. have been prepared following this method of opal template and sol–gel method.

 $CeO_2$  is an important rare earth oxide and has been applied in integral optical instruments, laser glass, fluorescent lamps, phosphors, and so on [12,13]. Inverse opal  $CeO_2$  [14,15] and doped  $CeO_2$ materials [16–19] have been prepared in the past and optical properties of them are studied in detail [14]. However, the optical properties were tested by changing the angle of incident light on

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#### ABSTRACT

Inverse opal cerium dioxide  $(CeO_2)$  is prepared with polystyrene (PS) microsphere opal template and solgel method, and characterized in detail. The opal template and inverse opal CeO<sub>2</sub> show different colors in the light of halogen tungsten lamp and have a long-range ordered three dimensional periodic structure. Comparing with the opal template, the size of the inverse opal CeO<sub>2</sub> shows shrinkage. The randomly selected positions of opal and inverse opal almost have the same peak position. The true filling factor of CeO<sub>2</sub> is different from the theoretical value and calculated by testing the reflecting peak. The inverse opal CeO<sub>2</sub> can modulate light by changing the diameter of air sphere and have directional stop band gap in the visible spectrum, which is expected to be some advanced and promising applications.

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an inverse opal  $CeO_2$  generated using on one opal template. In another case [15], one opal template and corresponding inverse opal  $CeO_2$  were prepared, the optical properties were not investigated at all.

In this work, different diameter porous inverse opal  $CeO_2$  are prepared and the optical properties are investigated in detail, the filling factors of inverse opal  $CeO_2$  are investigateded.

#### 2. Materials and methods

Different opal templates were prepared by the self-assembling method [20] with spheres of mean diameters of 340 nm, 390 nm and 435 nm polystyrene microsphere over a silica substrate [21]. CeO<sub>2</sub> inverse opals were prepared by immersing opal templates into 0.08 mol/L Ce(NO<sub>3</sub>)<sub>3</sub> sol–gel solution for 15 min, then taking the opal templates into 55 °C oven for 30 min. These operations above were repeated three times. Then, the infiltrated opal templates were annealed in an oven with a rising rate of 50 °C/h from room temperature to 500 °C and maintained for 2 h to completely remove PS microsphere, then naturally cooled down to room temperature. For reference, CeO<sub>2</sub> film was prepared on the clean silica substrate with the same operation.

#### 3. Results and discussion

Opal template and inverse opal  $CeO_2$  were observed with RZ-F300C and microscope (MA2001, COIC) in the light of halogen









Fig. 1. Microscope images of different diameter PS microsphere opal and corresponding inverse opal CeO2. (A and B) 340 nm; (C and D) 390 nm; (E and F) 435 nm.

tungsten lamp. The microscope images show as Fig. 1. The left column is opal, the right is inverse opal. The arrows represent the growth direction of opal and inverse opal.

The exhibiting colors are induced by the reflection of the opal and inverse opal structure which have directional stop band gap in special wavelength, which is the basic property of photonic crystal [22]. In the opals, there are some black lines which are cracks. In the inverse opal, there are many great cracks, and the domain sizes for uniform structures are close to one square millimeter. These results may be attributed to (1) the presence of defect and cracking in the opal template and (2) shrinkage of the sample after removing the template by calcination [23].

Fig. 2(a) shows the typical SEM image of the opal made from PS microsphere with mean diameter of 340 nm. The opal has long-ranged order in large-area. A face center cubic close packing structure with (111) planes along the substrate is shown. The opal

made from PS microsphere with 390 nm and 435 nm have the same structure. The thickness of the opal films are controlled about 20  $\mu$ m with 20–50 layers.

A long-range ordered macropore and mesopore network structure remains after the dissolution of the precursor solvent of  $CeO_2$  and removing the PS microsphere by calcination. The typical SEM image of the inverse opal  $CeO_2$  based on 340 nm PS microsphere opal shows as Fig. 2(b). It is obvious that the inverse opal  $CeO_2$  belongs to the shell structure. The sol–gel covers the inner surface of the opal only, leading to shell systems [24]. The center-to-center distance of macropores diameter in Fig. 2(b) is estimated 230 nm by direct measurement. This is attributed to the shrinkage of infiltrated  $CeO_2$  precursor due to the pore elimination during the annealing process. The measured holes' diameters of inverse opals  $CeO_2$  based on 390 nm and 435 nm PS microsphere opal and shrinkage rate show in Table 1.

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