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CeF_3 nanoparticles: synthesis and characterization

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Abstract

CeF_3 nanoparticles 5–10 nm in size were prepared using the polyol method. $CeCl_3$ and HF were heated up in ethylene glycol. At a temperature of 180 °C crystalline CeF_3 nanoparticles were formed. The material was washed with ethanol, centrifugated and dried. The particles were characterized by EDX, XRD and TEM.

Keywords: A. Fluoride; A. Inorganic compounds; A. Nanostructures; B. Chemical synthesis; D. Optical properties

1. Introduction

A number of exciting size- and surface-dependent properties of nanometer-sized semiconductor particles, which lie between molecular and bulk forms of matter, have stimulated an exponential development of nanochemistry and nanophysics.

Cerfluoride nanoparticles attracted interest for academic reasons, because of their Faraday effect, which is applicable to optoelectronics such as an optical isolator, optical switches or optical memory [1]. As Cerflouride nanoparticles exhibit a high Faraday effect combined with very low absorption in the visible they are well qualified for magneto-optical applications.

Extensive research work was carried out in the field of synthesis and characterization of semiconductor nanoparticles [2–7]. In the case of particles consisting of rare earth materials only few investigations are known. The sol–gel technique was used to produce nanoparticles of Tb_2O_3 [8] and rare earth doped yttria [12]. In spite of their interesting properties and potential applications no synthesis of well defined CeF_3 is known. Feldmann et al. [9–11] developed the so-called polyol process to synthesize nanoscale oxide, sulfide and phosphate materials. The surface of the nuclei is complexed by polyol medium right after formation which limits the growth of particles and stabilizes them against agglomeration.

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We report here about the synthesis and charcterization of monodisperse cerfluoride nanoparticles by polyol technique.

2. Experimental

2.1. Synthesis

CeF_3 particles were prepared by first suspending 200 mg $CeCl_3$ in 100 ml diethylene glycol in a teflon flask. Afterwards a mixture of 0.8 ml of an aqueous solution of 40% HF in 10 ml ethanol was added and the mixture then heated in a silicon oil bath to 180 °C under reflux for 2 h. After 2 days the mixture became turbid. After two more days the CeF_3 was separated via centrifugation and washed three times with ethanol to remove the chloride ions and the diethylene glycol. The yield of the reaction is around 25%. We assume that we lost some materials during the centrifugation process because the particles are very small and the rotational speed of our centrifuge was limited to 11,000 rpm.

2.2. Methods

The crystallinity and phase-purity of the products was monitored by powder X-ray diffraction (XRD) using a Guinier-Huber camera 600 with Cu $K\alpha_1$ radiation. The energy disperse X-ray analysis (EDX) was carried out on a Joel raster electron microscope (JSM-840A).

Morphological investigations were performed using a transmission electron microscope (Zeiss EM 900) (TEM). The samples were prepared by dropping diluted aqueous solution of CeF_3 nanoparticles onto 400-mesh carbon-coated copper grids with excess solvent evaporating after several hours.

High resolution transmission microscopy (HRTEM) were performed on a Phillips CM 30 with 300 kV.

3. Results and discussion

Figs. 1 and 2 show a typical TEM overview of CeF_3 nanoparticles. The average size estimated from TEM micrographs was 5 nm. The photo shows that most of the nanoparticles are single particles and that only a few particles are agglomerated.

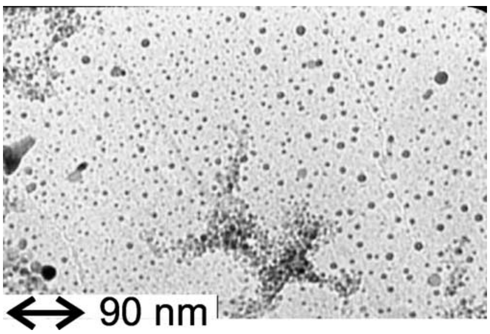

Fig. 1. TEM pictures of the CeF_3 nanoparticles.

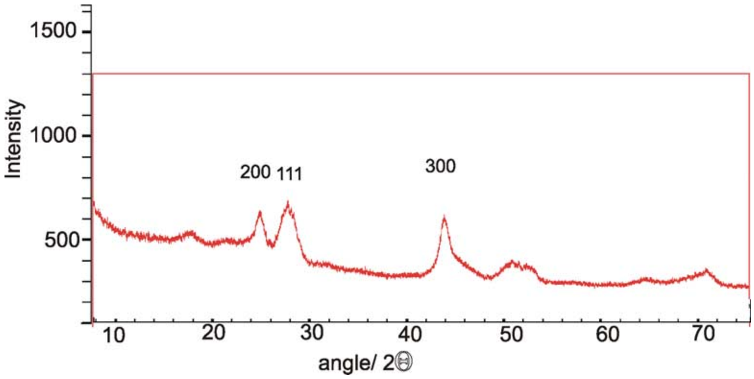

Fig. 2. X-ray diffraction pattern of the CeF_3 nanoparticles.

EDX measurements on CeF_3 nanoparticles indicated the presence of Ce and F in a ratio of 1:3, and in addition, 2% C due to the ethylene glycol which stabilises the individual particles in the solution as well when the particles are dried.

Powder X-ray diffraction has been performed on the powder of CeF_3. The diffractogram confirmed the crystallinity of the particles. The position of the reflection peaks matched that expected a the CeF_3 structure with the space group $P6_3$/mcm. The width of the peaks corresponds to the small particle size. The mean nanocrystal sizes obtained from the full width at half-maximum intensity are 2 nm. Fig. 3 shows a

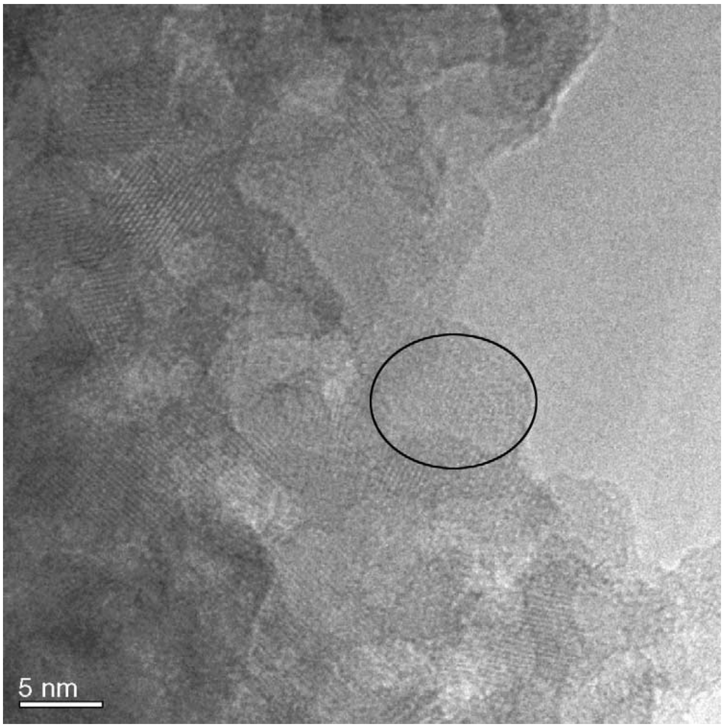

Fig. 3. HRTEM of the crystalline CeF_3 nanoparticles.

HRTEM overview image of CeF_3 nanoparticles. The existence of lattice planes on the HRTEM image further confirmed the crystallinity of the particles. The average sizes estimated from HRTEM micrographs were generally larger than those obtained from XRD patterns, a trend already observed for CdS and CdSe particles [2].

These CeF_3 nanoparticles are of interest due to their high Faraday rotation combined with low absorption which is deed to fabricate nano-composite-material with novel tunable magneto-optical properties. For example, they can be embedded in a thin transparent film [13].

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