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JOURNAL OF RARE EARTHS, Vol. 32, No. 11, Nov. 2014, P. 1027
JOURNAL OF RARE EARTHS
www.re-journal.com/en/

Preparation of Nd_2O_3 nanorods in SDBS micelle system

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Received 28 November 2013; revised 18 July 2014

Abstract: Well-crystallized Nd_2O_3 nanorods were prepared in the aqueous solution containing neodymium nitrate, sodium hydroxide (dissolved in ethanol) and sodium dodecyl benzene sulfonate (SDBS). One dimensional nanorods of neodymium hydroxide were synthesized first, which was then placed at different temperatures (600 and 800 °C) in a calcar for 10 h to form Nd_2O_3 nanorods. The morphology and crystal structure of the products were investigated by X-ray diffraction, transmission electron microscopy, field emission transmission electron microscopy, Fourier transform infrared spectroscopy and fluorescence spectrometry. By using SDBS micelles as a template, this method manufactured uniform morphology of hexagonal one-dimensional neodymium oxide nanorods with a diameter ranging from 20 to 70 nm. The length of the nanorods increased with prolonged reaction time.

Keywords: Nd_2O_3 nanorods; preparation; rare earth elements; SDBS; micelle

One dimensional nanomaterial offers great potential as new type of material for applications in catalysis, photonics, hydrogen storage material, and so on. For example, Li and Shen reviewed the morphology of rod-shaped metal oxides[1]. Du et al.[2], Zhang et al.[3] and Maitarad et al.[4,5] made many studies in shape-controlled synthesis and catalytic application of nanomaterials. The neodymium oxides have been widely used in magnetic materials[6–8], superconductor[9], photonics[10], superfine ceramics, modified glass[11], luminescent/laser materials[12,13], protective coatings[14,15], catalysts and catalyst promoters[16,17]. Different from the traditional neodymium oxides, the nanometer Nd_2O_3 is of interest for many potential applications due to their small-size effect, tunneling effect and interfacial surface effect. In recent years, the preparation and characterization of Nd_2O_3 nanomaterials with superfine structure have attracted much attention because of their wide range of applications.

Nd_2O_3 nonmaterial is usually synthesized by sol-gel process[18,19], direct precipitation[20], and hydrogen plasma-metal reaction[21]. In this study, Nd_2O_3 nanorods have been synthesized by using surfactant as template. In order to obtain Nd_2O_3 nanorods with uniform morphology, the effects such as structures and concentrations of surfactants and reaction time on the morphologies have been studied. In addition, the Nd_2O_3 nanorods obtained were light blue colored, which should be very interesting since the blue color is one of the trichroism (red, green and blue are the three primary colors), and the blue color crystal is hence highly significant in microelectronics application and luminous material.

1 Experimental

1.1 Materials

Neodymium nitrite (Shanghai Chemical Co., A.R.), sodium dodecylbenzene sulfonate sodium (SDBS, Fluka, A.R.), ethanol (Shanghai Chemical Co., A.R.), sodium hydroxide (Shanghai Chemical Co., A.R.) and distilled water were used.

1.2 Synthesis of Nd_2O_3 nanorods

The preparation of Nd_2O_3 nanoparticles in SDBS micelle solution is shown below.

$Nd(NO_3)_3+3HOC_2H_5=3Nd(OC_2H_5)_3+3HNO_3$

$Nd(OC_2H_5)_3+3H_2O=Nd(OH)_3\downarrow+3HOC_2H_5$

$Nd(OH)_3\rightarrow dried\rightarrow Nd_2O_3$

Generally, neodymium nitrite (0.4 g) was added into ethanol containing sodium hydroxide. Under continuous stirring SDBS micelle solution (16 mL) was added and neodymium hydroxide was obtained as a light purple precipitate after 12 h.

The purple precipitate was filtered and washed with distilled water and alcohol, respectively. The product was dried in vacuum at 80 °C for 24 h first and then put in a calcar at different temperatures (500, 600 and 800 °C) for 10 h, leading to light blue colored Nd_2O_3 nanorods with hexagonal phase.

1.3 Characterization

The morphology and size of the sample were exam-

Foundation item: Project supported by Laboratory of Environment Materials and Project in Jiangsu Province (017375003k11034) and National Natural Science Foundation of China (20773106)

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DOI: 10.1016/S1002-0721(14)60178-2

ined by a transmission electron microscope (TEM, TECHAI-12 Philip Apparatus Co., USA) and Tecnai G2 F30 S-TWIN (FEI Co., USA). The X-ray diffraction patterns of samples were recorded with an X-ray diffractometer using Cu Kα radiation of 0.15406 nm (XRD, AXS D8 Brucker Co., Germany). The Fourier transform infrared (FTIR) spectrum of the sample was recorded at room temperature in the range of 4000–400 cm^{-1} with an infrared spectrometer (Nicolet-740, USA) using KBr pellet technique. The fluorescence measurements were taken on a PerkinElmer LS-55 spectrofluorometer with a 700 W Xenon lamp as excitation source.

2 Results and discussion

The morphology of the synthesized Nd_2O_3 nanoparticles was obtained with high-resolution TEM (HRTEM). Fig. 1(a) shows the TEM images of Nd_2O_3 nanorods, from which it can be seen that Nd_2O_3 nanorods are 50 nm to 1 μm in length and have a uniform diameter in the range of about 20 nm. The magnified images inserted in Fig. 1(a) show the clear (001, left) and (100, right) lattice fringes with the interplanar spacing of 0.6 and 0.38 nm, respectively.

Fig. 1(b) is the EDS spectrum of the Nd_2O_3 nanorods. It should be mentioned here that C and Cu peaks in this figure come from the Cu grids. Therefore, Fig. 1(b) illustrates that there are only Nd and O existing in the nanorods.

The phase purity and crystal structure of $Nd(OH)_3$ and Nd_2O_3 nanorods obtained were examined by XRD pattern (Fig. 2). Fig. 2 shows X-ray diffraction spectra of $Nd(OH)_3$ and Nd_2O_3. All the peaks of the XRD pattern of $Nd(OH)_3$ (Fig. 2(a)) are identical with those in literature[22]. The X-ray diffraction peaks of Nd_2O_3 at 600 °C calcination (Fig. 2(b), curves (1–3)) correspond to the (001), (100), (002), (011), (012), (003) and (103) planes, which can be indexed to the hexagonal structure for one-dimensional neodymium oxide nanorods with a lattice constant of $a=b$=0.384000 nm, c=0.601000 nm, according to JCPDS card No. 00-074-1147. The X-ray diffraction peaks of Nd_2O_3 at 800 °C calcination (Fig. 2(b) curves (4–6)) correspond to the (100), (002), (101), (102), (110), (103), (112), (201), (202), (104), (203), (210), and (211) planes, which can be indexed to the hexagonal structure for one-dimensional neodymium oxide nanorods with a lattice constant of $a=b$=0.38297 nm, c= 0.59987 nm, and $2\theta_{Max}$=30.827 according to JCPDS card No. 00-041-1089. Besides, no obvious peaks corresponding to neodymium hydroxide or neodymium nitrate were observed in Fig. 2(b) curves (4–6). Therefore, the Nd_2O_3 nanorods synthesized in the present paper are pure hexagonal phase. The X-ray diffraction peaks of Nd_2O_3 at 500 °C calcination according to JCPDS card No. 00-021-0579, can be indexed to the cubic structure for one-dimensional neodymium oxide nanorods with a lattice constant of $a=b=c$=1.108 nm, and $2\theta_{Max}$=27.939, which are the same as in the literature[22] (dates not shown). Obviously, different crystalline Nd_2O_3 can be obtained at different temperatures at calcar.

Based on Debye-Scherrer expression $D=K\lambda/(\beta\cos\theta)$[23], when the size of crystal is smaller than 100 nm, remarkable diffraction peak becomes wide, with the size of crystal becoming small. Therefore, comparing the curves (1–3) (or (4–6)) in Fig. 2(b), it is evident that Nd_2O_3 nanorods have better crystallinity when SDBS concentrations is 50 mmol/L.

The concentration of SDBS was found to affect the morphology of $Nd(OH)_3$. Fig. 3 shows the TEM image of Nd_2O_3 nanorods prepared in the micelles having different SDBS concentration. At SDBS concentration lower than 1 mmol/L (<cmc_1), the particles formed are sphere, with the diameter ranging from 20 to 100 nm (Fig. 3(a)). When SDBS concentration was higher than cmc_1, the rod-like Nd_2O_3 was obtained and with increasing SDBS concentration (30–50 mmol/L), the Nd_2O_3 nanorods became longer and wider (Fig. 3(b–d)); the size ranged from 20 to 70 nm in width and >50 nm in length. Therefore Nd_2O_3 nanorods were formed using SDBS micelles as the template. Moreover, Nd_2O_3 nanorods be-

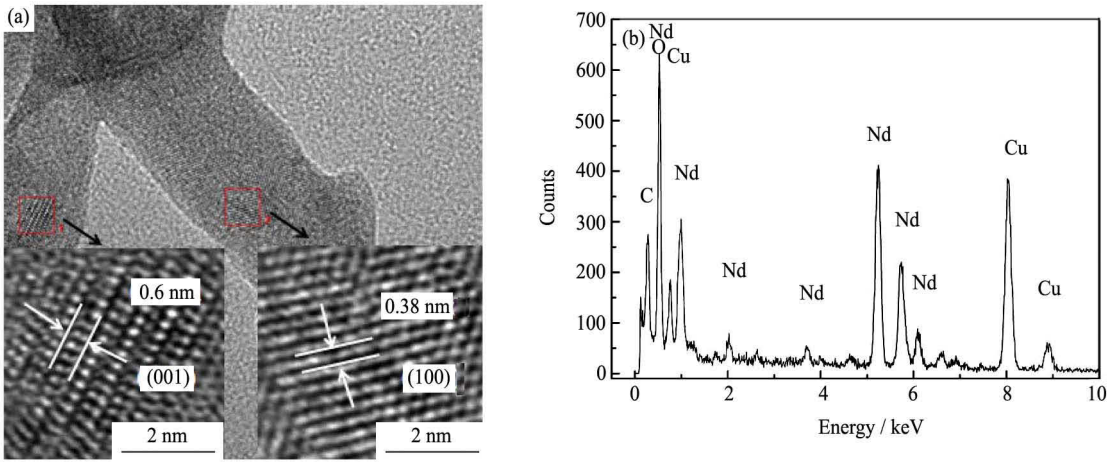

Fig. 1 HRTEM image (a) and EDS picture (b) of hexagonal phase of Nd_2O_3 nanorods