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Low-temperature Growth of Single-crystal $SrCO_3$ Nanoneedles

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Abstract: Single-crystal $SrCO_3$ nanoneedles were synthesized in reverse micelles at low temperature. The products were characterized by X-ray diffraction, X-ray energy dispersive spectrometer, transmission electron microscopy and selected area electronic diffraction. The influences of experimental conditions on the morphologies of the products were discussed. The growth mechanism of $SrCO_3$ nanoneedles in reverse micelles were proposed.

Keywords: Nanoneedles, $SrCO_3$, reverse micelle.

Owing to the important role of dimensionality in the properties of nanomaterials, various types of 1D nanostructured materials, including metals and alloys, inorganic oxides, inorganic salts and polymers have been synthesized and their novel properties were also extensively studied in the recent years[1-5]. Various preparation methods towards diverse 1D-nanomaterials, including templating methods, catalytic growth, electrochemistry, chemical vapor deposition, solution-based solvothermal treatment and reverse micelle, have been extensively developed. However, some of these methods require special conditions, such as high temperature and pressure and the catalysts. Among these methods, reverse micelle or microemulsion is demonstrated to be promising for the fabrication of 1D nanomaterials. The shape, size, and size distribution of nanowires, prepared in the reverse micelles, can be controlled by reaction temperatures, surfactants, additives, surfactant concentrations and molar ratios of water to surfactants[6].

Carbonate materials are widely applied to the inorganic-organic hybrid composite materials as fillers. $CaCO_3$ and $BaCO_3$ nanorods or nanowires have been synthesized in reverse micelles[7-8]; however, to the best of our knowledge, strontium carbonate nanoneedles or nanowires have not been prepared in reverse micelles. It has been proved that $SrCO_3$ materials has unique performance in low-temperature catalytic oxidation of VOC(volatile organic compound) and chemiluminescence sensors[9]. However, the morphology and size are much more significant to determine the properties of $SrCO_3$ materials[10]. Dendritic strontium carbonate has been synthesized through the hydrothermal approach[11]. Herein, we introduce a facile reverse micelle method to synthesize strontium carbonate nanoneedles at low temperature.

The $SrCO_3$ nanoneedles were obtained by rapidly pouring the reverse micelle A (3

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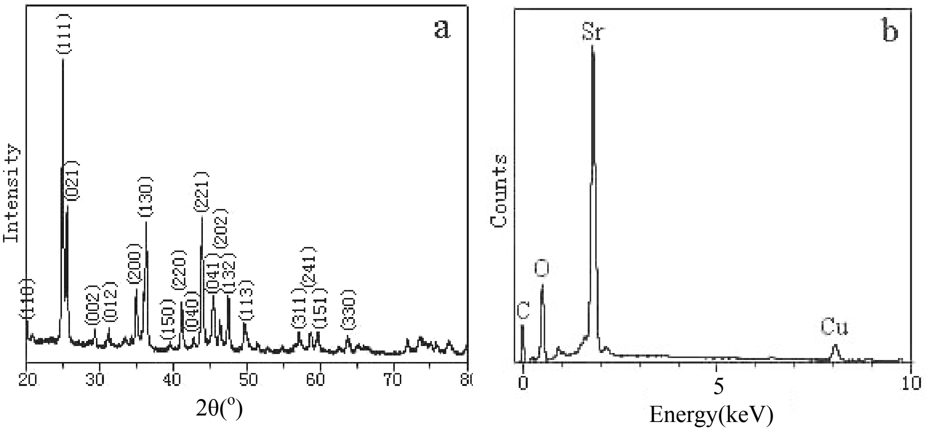
mL Tx-100, 3 mL *n*-butylalcohol, 30 mL cyclohexane, 0.5 mL 0.1 mol/L $Sr(NO_3)_2$ aqueous solution) into the reverse micelle B (3 mL Tx-100, 3 mL *n*-butylalcohol, 30 mL cyclohexane, 0.5 mL 0.1 mol/L Na_2CO_3 aqueous solution) while stirring for 5 minutes; the mixtures (reverse micelles) were then aged at 303 K for 48 hrs without stirring.

The X-ray diffraction (XRD) spectra of strontium carbonate (**Figure 1a**) can be indexed on an orthorhombic phase according to JCPDS 05-0418. It also indicates that these peaks are attributed to the pure strontianite-type crystal according to the XRD standard spectrum of $SrCO_3$ powder. The X-ray energy dispersive spectrometer (EDS) data indicates that these nanoneedles are composed of Sr, O, C (**Figure 1b**). It is worth noting that in the XRD spectrum of strontium carbonate the (200) peak is strengthened, which maybe imply the anisotropic growth along the [100] direction.

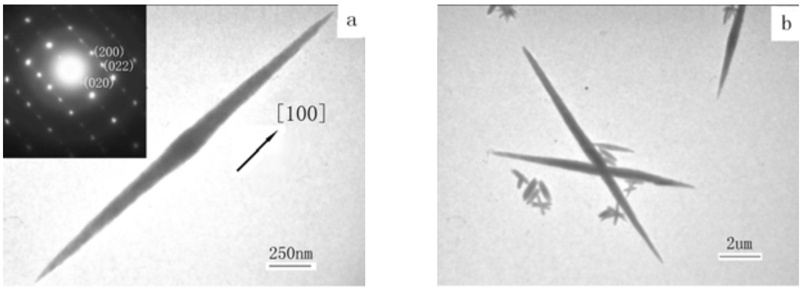
Fig 1 XRD spectra (a) and EDS analysis (b) of SrCO3 nanoneedles



The typical transmission electron microscopy (TEM) and selected area electronic diffraction (SAED) images of the samples are illustrated in **Figure 2**. **Figure 2a** is the SAED patterns and its corresponding image. The needlelike $SrCO_3$ was prepared at 303 K and the aging time of the samples was 24 hrs. The SAED patterns of the sample are consistent with the good crystallinity and mainly the nanowires grow along the [100] direction. The diffraction spots can be indexed on the orthorhombic phase. Those results are in good agreement with the results of XRD. However, because the sources of Sr^{2+} and CO_3^{2-} were limited, the length of needle was not long enough and the aspect radio of the product is only about 20. In order to obtain long $SrCO_3$ nanoneedles, 1 mL 0.1 mol/L $Sr(NO_3)_2$ aqueous solution was used in the reverse micelles, not 0.5 mL. The gotten sample is just like **Figure 2b**, which exhibits some longer nanoneedles and the length of the longest nanoneedles is about 20 μm.

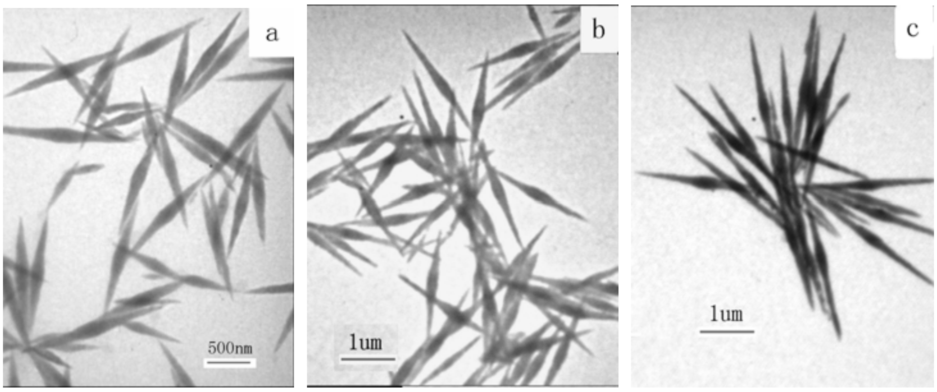
$CaCO_3$ and $BaCO_3$ nanorods or nanowires have been prepared in reverse micelles. The nanoparticles→ network →aggregated to short wires →developed to long wires mechanism was proposed by Daibin Kuang *et al.* and the nanorod-derect oriented attachment growth mechanism was proposed by Biao Liu *et al.*[8,12]. The growth processes of $SrCO_3$ nanostructures in reverse micelles were also followed by examining

Figure 2 TEM and SAED patterns of $SrCO_3$ nanoneedles.



(a) The SAED pattern of needlelike $SrCO_3$ and its corresponding image; (b) the TEM pattern of long nanoneedles.

Figure 3 Illustration of the growth of the nanoneedles at different aging time.



(a) 6hrs; (b) 24hrs; (c) 48hrs.

the early stages of their formation and the "Ostwald ripening process" growth mechanism was proposed[13]. In the Ostwald ripening process, the larger particles will grow at the cost of the small ones due to the difference of energy between large and small particles of a higher solubility based on the Gibbs-Thompson law. As shown in **Figure 3a**, after 6 hrs of aging, lots of nanoneedles formed and the length of them was about 1 μm. At the same time, a few of shorter nanoneedles were appeared. The larger nanoneedles would then grow at the cost of the small ones. Just like **Figure 3b**, after 24 hrs of aging the length of the nanoneedles became 2 μm and few of shorter nanoneedles could be found. At last, with increasing of aging time the longer nanoneedles appeared (**Figure 3c**). It exhibites that the morphologies of the nanoneedles are uniform.

In summary, single-crystal $SrCO_3$ nanoneedles were synthesized at low temperature by a facile reverse micelle method. The experimental conditions, which influenced on the morphologies of the products, were discussed. The aspect radio of the product is about 20 and the length of the longest one is about 20 μm. The growth processes of $SrCO_3$ nanostructures were followed and the "Ostwald ripening process" growth mechanism was proposed.

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