Novel monoclinic CuO nanobundles, 0.8–1 μm in size, were synthesized at 130 °C in the presence of sodium dodecyl benzenesulfonate (SDBS) by a simple hydrothermal method. Each nanobundle was comprised of many nanorods with one end growing together to form a center and another end radiating laterally from this center. The length and the diameter of these assembled nanorods are in the range of 200–300 nm and about 20–30 nm, respectively. HRTEM and SAED results indicated that the CuO nanorods grow along the [010] direction. An investigation of the hydrothermal process revealed that the reaction time, temperature and surfactant play important roles in the formation of the resultant CuO nanostructures. Isolated CuO nanorods were obtained when the temperature was increased to 190 °C, and CuO microflowers composed of many nanosheets were produced at 130 °C when cetyltrimethylammonium bromide (CTAB) was employed instead of SDBS. The possible mechanism for the formation of these CuO nanostructures was discussed simply on the basis of the experimental results.

Keywords: CuO, Nanostructures, Hydrothermal Synthesis.

1. INTRODUCTION

Over the past few decades, the synthesis and investigation of low-dimensional (0D, 1D and 2D) nanomaterials have attracted particular attention in view of their wide range of potential applications.1,2 The fabrication of three-dimensional (3D) complex architectures consisting of low-dimensional nanostructures is of great interest to material scientists and chemists because this type of material not only shows unique properties but also gives insight into the construction of micro- and nano-scale devices.3 A variety of 3D nanomaterials have been investigated extensively via a soft template-assisted approach. For example, Guo et al. synthesized dendritic silver crystals in a cetyltrimethylammonium bromide (CTAB) and sodium dodecyl benzenesulfonate (SDBS) mixed surfactant solution at room temperature, and found that the concentration of CTAB/SBDS and molar ratio have a significant influence on the final silver shape.4 Uniform cobalt microspheres composed of ordered nanoplatelets with a thickness of approximately 20 nm were obtained in the presence of SDBS.5 Flowerlike bismuth tungstate structures, which were assembled by nanosheets consisting of numerous square nanoplatelets, were fabricated by varying the amount of poly(vinylpyrrolidone) (PVP) surfactant.6 Different driving mechanisms, including surface tension, capillary effects and magnetic forces, were proposed for the formation of 3D complex nanostructures.7–9 Among all complex 3D architectures, there are few reports on bundle-like nanostructures10,11,41,42 because of the extremely novel shape. Hong et al. proposed that a planar network structure of gadolinium oxalate served as a molecular template to fabricate Gd(OH)3 nanobundles.10 A decrease in surface energy was proposed to explain the formation of sodium tungstate nanobundles with the driving force for the assembly of nanorods originating from lateral capillary forces.11 All these bundle-like nanostructures were constituted directly by parallel nanorods connecting together.

As an important p-type semiconductor, cupric oxide (CuO) has drawn much attention in recent years on account of its promising applications. It forms the basis of several high-\( T_c \) superconductors and materials with...
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is in the range of 400–500 nm and the diameter is about 20–30 nm.

Figures 2(a and b) shows typical TEM images that are in accordance with the FESEM observations. As can be seen, a single bundle possessed many nanorods with one ends radiating from a central point, which was formed by another ends growing together. The structures and shapes were still preserved even after the CuO nanobundles were dispersed in ethanol by ultrasonic vibration for 30 min before being deposited on a carbon-coated copper grid for the TEM observations. This suggests that the center of the nanobundle was produced as an integral part when one end of the nanorods grew together, rather than loosely aggregated. The novelty of this nanostructure is that the entire CuO nanobundle exhibits lateral radiation from this center. The full size of a single bundle was approximately 1 μm, and the typical length as well as the diameter of the nanorods is in the range of 350–500 nm and close to 20–30 nm, respectively. Figure 2(c) is a corresponding SAED pattern taken from an individual nanorod of CuO nanobundles. The SAED pattern can be indexed to be the [001] zone axis of a monoclinic phase CuO nanorod, indicating that it is single crystalline. In contrast, SAED of the entire CuO nanobundle produced a complex polycrystalline pattern (Fig. 2(d)). The ED rings were not continuous but were composed of discrete spots, suggesting a preferential orientation of the collective assembled nanorods constituting the CuO nanobundles. HRTEM image taken from the tip of a single CuO nanorod is shown in Figure 2(e), which reveals that the nanorod is a single crystal in nature. The distance between the adjacent fringes was examined to be 0.270 nm, corresponding to that of the (110) planes of CuO. Based on SAED and HRTEM analyses, it can be demonstrated that the CuO nanorods grow along the [010] direction. The direction is the same with the previous reports of CuO nanoleaves and nanoplatelets.²⁵,²⁶

The structure and crystal phase of the resultant CuO nanobundles were investigated by XRD pattern as shown in Figure 2(f). All the observed peaks were indexed to the monoclinic phase CuO (JCPDS 05-0661, \(a = 4.684\) Å, \(b = 3.425\) Å, \(c = 5.129\) Å and \(\beta = 99.47°\), space group \(C2/c\)). No other characteristic peaks for impurities, such as Cu(OH)₂ or Cu₂O were detected, indicating the high purity of CuO nanobundles obtained via our current synthetic route. The XRD results are well consistent with the above HRTEM and SAED analyses. The samples of CuO nanobundles were further examined by Raman spectra. CuO belongs to the \(c_{2h}^6\) space group with two molecules per primitive cell. There are 12 zone-center optical phonon modes including six infrared active modes \((3A_u + 3B_u)\), three acoustic modes \((A_u + 2B_u)\), and three Raman active modes \((A_g + 2B_g)\). Figure 2(g) shows the Raman spectra of the as-synthesized CuO nanobundles. It can be seen that

**Fig. 2.** TEM images of (a) CuO nanobundles with a panoramic view and (b) high-magnification view of half of a CuO nanobundle; (c) SAED pattern of a single assembled nanorod circled in (b); (d) SAED pattern of the whole CuO nanobundle circled in (a); (e) HRTEM image of a single assembled nanorod circled in (b); (f) XRD pattern and (g) Raman spectra of the CuO nanobundles.
there are three Raman peaks at 273, 320 and 608 cm\(^{-1}\) corresponding to the \(A_2\), \(B_3\) and \(B_1\) modes of CuO nanostructures, respectively. The results are in good agreement with the reported values\(^{19}\) and demonstrate that CuO nanobundles are single crystal with a monoclinic structure.

In order to investigate the growth mechanism of the as-prepared CuO nanobundles, systematic time-dependent experiments were carried out at 130 °C. Figures 3(a–d) shows the SEM images of CuO samples synthesized at 130 °C with a reaction time of 100 min, 2, 10 and 18 h, illustrating the morphological evolution of the CuO nanobundles. A large number of flake-like CuO were obtained when the hydrothermal reaction proceeded for 100 min and the typical SEM image was shown in Figure 3(a). The surface of some CuO nanoflakes was rough and some flakes connected to each other. At the initial stage, the orthorhombic Cu(OH)\(_2\) precursor precipitated as small nanorods or nanoflakes due to the connection of (010) planes through H-bonds.\(^{18}\) In the following hydrothermal period, Cu(OH)\(_2\) lost H\(_2\)O molecules by breaking the interplanar H-bonds, resulting in the formation of monoclinic flake-like CuO nanostructures. It should be noted that 100 min was the appropriate time to complete the reaction, as investigated by the XRD pattern shown in Figure 3(f). Some blue precursors coexisted with the black CuO products when the reaction time was less than 100 min, which suggested the transformation from the Cu(OH)\(_2\) precursor to the final resultant CuO was not fully complete. When the reaction was prolonged for 2 h, the edges of most CuO nanoflakes partially split into

![SEM images of CuO products prepared with SDBS at 130 °C with a hydrothermal reaction time of (a) 100 min; (b) 2 h; (c) 10 h and (d) 18 h. (e) SEM image of the sample prepared without SDBS for 24 h and (f) XRD pattern of products prepared with SDBS at 130 °C for various time.](image-url)
many irregular short nanostrips due to the splitting process. By careful observation, some tips of the short strips were curved while the center part of the flakes was still integrated (Fig. 3(b)). This process is similar to the formation of CuO nanoribbons and nanorings. With the assistance of SDBS in the low temperature region, the CuO nanoflakes split easily into small nanoribbons possibly because the intense Brownian movement of the surfactant molecules might destroy the integrity of CuO crystal facets. As the reaction time was increased further, the partially split parts of these CuO nanoflakes might grow thicker and larger to produce rod-like structures. As the SEM image shown in Figure 3(c), many irregular rod-like structures with a variety of lengths were produced when the hydrothermal reaction time was 10 h. One ends of these nanorods were connected together in the central part of flakes, which was preserved well as those in the previous stage. However, the nanorods were not straight and the diameter varied according to the position. It was obvious that the CuO rod-like structures were formed in situ from the split parts of nanostrips and the various lengths might result from the different extent of partially splitting. When the hydrothermal treatment was progressed for 18 h, the CuO nanobundles were formed to some extent (Fig. 3(d)). Compared to the CuO products obtained from 24 h hydrothermal treatment process, the nanobundles produced at this stage were not perfect. However, many straight nanorods existed with one end connecting to form a whole as center of the bundle. The whole center was the one preserved from the nanoflake at the initial reaction stage. With reaction time prolonging, the CuO nanobundles will continue to grow into perfect ones, as what are displayed in Figures 1(a and b). In contrast, the sample prepared at 130 °C for 24 h without surfactant of SDBS only included some small nanoplatelets and some layered CuO nanostructures due to the direct decomposition of Cu(OH)$_2$ precursor (Fig. 3(e)).

A possible growth mechanism for the formation of bundle-like CuO nanostructures is proposed based on the above controlled experimental results. The growth of CuO nanobundles in our case consisted of a five-step process as schemed in Figure 4:

1. The Cu(OH)$_2$ precursor precipitates as very small nanoflakes due to the H-bonds existing and connecting its (010) planes (step a).
2. Flake-like CuO nanostructures with some rough surfaces form through both the decomposition of Cu(OH)$_2$ molecules and the breakage of H-bonds (step b).
3. The edges of the CuO nanoflakes split partially with the assistance of the surfactant SDBS, while the central part is still integrated (step c).
4. Irregular CuO nanorods with different lengths are formed in situ at the previous partially split positions. The rods are connected together with one end at the central part of CuO nanoflakes (step d).
5. The irregular CuO nanorods transform gradually into straight ones and perfect bundle-like CuO nanostructures are produced (step e) finally.

It is obvious that SDBS is very crucial during the entire CuO growth process. In the beginning, SDBS interacts directly with the CuO facets to produce partially split CuO nanoflakes, and subsequently it acts as a soft template for the growth of CuO nanorods and the formation of the final bundles.

![Chemical reaction diagram](image)

**Fig. 4.** Schematic diagram of a possible growth mechanism for CuO nanobundles. The entire procedure includes five main steps. (a) blue Cu(OH)$_2$ precursor precipitates as very small nanoflakes due to the H-bonds in its (010) planes; (b) Irregular CuO nanoflakes with a rough surface form by the dehydration of Cu(OH)$_2$ molecules; (C) The edges of CuO nanoflakes split partially as a result of an interaction between SDBS and the CuO crystal surface, while the central area is still integral; (d) Irregular nanorods are formed in situ at the previous partially split positions; and (e) Straight nanorods and perfect CuO nanobundles are formed finally.
Temperature is also served as one of the important factors, which influences the final morphology of resultant CuO nanostructures. On the basis of our experimental results, temperature determines the extent of splitting process for the nanoflakes. Figure 5 shows SEM images of CuO samples prepared at different temperatures over a 24 h period. When the synthesis was carried out at 100 °C, only thick strip-like CuO was generated, and many tips of the partially split strips were curved to form a hook-like shape. At 100 °C, the low temperature could not provide sufficient energy for the intense movement of surfactant molecules and the crystal facets showed less destruction. As a result, only very slight splitting process occurred originally and resulted in the preservation of large integrated central areas of nanoflakes, as displayed in Figure 5(a). It appears that 130 °C is the optimal temperature for the formation of perfect CuO nanobundles, possibly because the appropriate extent of splitting of the initial CuO nanoflakes can be obtained with the assistance of surfactant molecules under this synthetic temperature, and the related SEM image is also shown in Figure 5(b). If we kept increasing the reaction temperature to 160 °C or above, it was clearly observed from Figures 5(c–d) that CuO nanobundles became much less prevalent while isolated nanorods were dominant. In particular, for the samples prepared at 190 °C, almost uniform separate CuO nanorods were achieved. In the higher temperature region in our present work, CuO nanoflakes were split completely into nanostrips at the initial stage of the reaction and the central part of the flakes could not be preserved any more. Therefore, isolated nanorods developed from the split nanostrips could be finally obtained. It is interesting that the length of the isolated CuO nanorods is almost comparable to that of nanorods constituted the CuO nanobundles.

The final morphology of the CuO nanostructures is determined by many factors instead of only the reaction time or temperature, as investigated above. It is widely accepted that the final shape of the resultant CuO is also relevant to the concentration of the starting materials, pH of the system, solvent, surfactant type and so on. For example, only flower-like CuO consisting of nanosheets could be obtained when the same approach and synthetic parameters were employed except that the surfactant of SDBS was substituted with cetyltrimethylammonium bromide (CTAB). From the FESEM and TEM images in Figures 6(a–c), we found that most nanosheets were in the range of 0.5–2 μm, while the size of each CuO microflower was approximately 2–4 μm. A close observation (Fig. 6(c)) revealed that the surface and edges of these nanosheets were not smooth. The SAED patterns (inset in Fig. 6(c)) and typical XRD pattern shown in Figure 6(d) demonstrate that the CuO microflowers are single crystalline in the monoclinic phase with no impurities.

Generally, the surfactant is considered to kinetically control the growth rates of different crystallographic facets of CuO nanostructures through preferentially adsorbing and desorbing on these facets. However, the mechanism of morphology-control using surfactants is quite complicated since different surfactants may play different roles for a definite nanomaterial, which might lead to the formation

![Fig. 5. SEM images of CuO products prepared at (a) 100 °C; (b) 130 °C; (c) 160 °C and (d) 190 °C with a hydrothermal reaction time of 24 h.](image-url)
of different final shapes. The molecule of SDBS has a hydrophilic group of DBS\(^{-}\) that can coordinate with Cu\(^{2+}\) ions to produce Cu(DBS)\(_2\) (Eq. (1)). With increasing temperature and the presence of NaOH, the compound Cu(DBS)\(_2\) can be transformed gradually into Cu(OH)\(_2\) precursor and then this blue precursor can precipitate (Eq. (2)). When the temperature is high enough, CuO can be produced by the dehydration of Cu(OH)\(_2\) and the simultaneous release of DBS\(^{-}\) anions (Eq. (3)), which can determine the extent of splitting of the initially generated CuO nanoflakes by interacting directly with its surfaces as well as act as a template for the following crystal growth. The detailed chemical reactions occurring during the hydrothermal process can be expressed as follows:

\[
\begin{align*}
\text{Cu}^{2+} + 2\text{DBS}^{-} & \rightarrow \text{Cu}($$\text{DBS}$$)_2 \quad (1) \\
\text{Cu}($$\text{DBS}$$)_2 + 2\text{OH}^{-} & \rightarrow \text{Cu}($$\text{OH}$$)_2 + 2\text{DBS}^{-} \quad (2) \\
\text{Cu}($$\text{OH}$$)_2 & \rightarrow \text{CuO} + \text{H}_2\text{O} \quad (3)
\end{align*}
\]

Although SDBS was found from the above analysis to play important roles as both ligand and soft template, the practical amounts of starting materials for SDBS and copper salts (0.021 M/0.037 M) in the experiments was less than the stoichiometric ratio based on the reaction equations which probably make the SDBS act more as template to large extent. SDBS was closely associated with alkali during the entire reaction process, which made its role more complex than that of CTAB in the synthesis of CuO microflowers. The detailed reasons for SDBS- and CTAB-assisted formation of different final CuO nanostructures needs to be studied further and the related work is currently under way.

4. CONCLUSIONS

In summary, we have employed a simple hydrothermal method to prepare CuO nanobundles with size of 0.8–1 μm in the presence of SDBS. These nanobundles were assembled by many nanorods with one ends growing together to form a center and another ends radiating laterally from it. The length and the diameter of these CuO nanorods are in the range of 200–300 nm and about 20–30 nm, respectively. It was found that reaction time, temperature and surfactants played important roles in the formation of such novel CuO nanostructures. Isolated CuO nanorods were obtained when the temperature increased to 190 °C, and CuO microflowers composed of nearly rhombic nanosheets were achieved at 130 °C when CTAB was employed instead of SDBS.
Acknowledgments: The authors are grateful for the financial support from the BK21 Project through School of Advanced Materials Science and Engineering.

References and Notes


Received: 19 May 2009. Accepted: 4 August 2009.