Synthesis of Cu and CuO_x Nanowires via Reduction of CuO Nanowires

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One-dimensional metal nanostructures attracted great interest because of their novel physical and chemical properties and their potential applications in the new generation of nanoscale devices and systems. Copper nanowires are of particular interest due to their potential applications in fabricating nanoscale electronic devices and CuO_x is of particular interest to heterogeneous catalysis. Copper nanowires have been synthesized by many methods such as template synthesis [1], electrochemical synthesis [2] and reduction synthesis [3]. For example, aligned Cu nanowires have been synthesized by the reduction of aligned CuO nanowires in electron cyclotron resonance hydrogen plasma [3]. The direct thermal reduction of CuO nanowires in H₂ would not produce the expected Cu nanowires because of the coalescence and deformation of newly formed Cu nanowires [3]. We report here the successful fabrication of aligned Cu nanowires and CuO_x nanowires via the use of less reducing gasses to avoid the coalescence of Cu nanowires during the thermal reduction process.

The CuO nanowires were synthesized by direct thermal oxidation of Cu substrates in a tube furnace filled with air, similar to the synthesis processes reported earlier [4]. The synthesized CuO nanowires were then loaded into a tube furnace with either flowing nitrogen or argon. Various reduction temperatures were used to explore their effect on the final Cu nanowires. The degree of reduction period, the types of reducing gases used, etc. A field emission SEM equipped with an energy dispersive X-ray spectrometer and a Robinson backscattered electron detector was used to characterize the morphology, composition and size distribution of both the CuO nanowires and the reduction product Cu nanowires.

Figure 1 shows the SEM images of the CuO nanowires prior to the thermal reduction, revealing the general morphology of the fabricated CuO nanowires as well as the presence of very long CuO nanowires (figure 1a). The unreduced CuO nanowires have diameters from 100 nm to 200nm with an average length of 8-10 μ m; some of the very thin CuO nanowires grew as long as 100 μ m or longer. Most of the CuO nanowires are aligned with their growth directions perpendicular to the local surface of the Cu substrate. Figure 2a and 2b show SEM images of nanowires obtained after reducing the CuO nanowires in nitrogen at about 360°C for 35 minutes. After reduction, the nanowires modified their morphology and the end of the nanowires lost their shape and collapsed onto each other. Removal of oxygen from the CuO lattice during the reduction process may have caused the nanowires deform. Figure 2c shows the curling of the tip of the Cu nanowires; high resolution SEM images and TEM images showed the presence of potholes in the Cu nanowires. EDS analysis showed that the nanowires contain mostly Cu with a small amount of oxygen probably due to air exposure of the nanowires. It is interesting to note that the most of the Cu nanowires still maintains their integrity and are firmly connected to the substrate and are aligned with respect to each other. At higher reduction temperatures, all the CuO nanowires were reduced and collapsed to form Cu nanoparticles. At lower reduction temperatures, nanowires of CuO_x were formed and the oxygen content depends on the reduction temperature, the reduction period and the type of reduction gasses. Detailed reduction processes of CuO nanowires will be discussed [5].

References

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Figure 1. SEM images of CuO nanowires prior to reduction in nitrogen.

Figure 2. SEM images of nanowires after reduction at 360°C for 35 minutes in nitrogen. EDS analysis showed that the nanowires are most probably pure Cu. Very thin Cu nanowires were curled up and potholes were formed on their surfaces.

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