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Fabrication of Materials for Next-Generation Clean Energy Devices from Salts

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Summary

Cobalt oxide (CoO) whiskers were significant owing to their potential applications based on electric, magnetic, catalytic, and gas-sensing properties. In addition, it used to be applied to the raw materials of lithium cobalt oxide for lithium ion secondary batteries. There are various techniques to make CoO whiskers, including anodization, sol-gel method, hydrothermal growth and flux growth. Among these techniques, flux growth is an environmentally friendly process and can produce high-quality crystals at temperatures below the melting points of the solutes. In this study, we report the growth of superlong CoO whiskers via chloride flux method and the observation of their growth manner.

Reagent-grade cobalt powders were used as a solute, and a mixture of KCl and LiCl powders was chosen as the flux. Furthermore, vapor-grown carbon fibers (VGCF) were also used as an additive. Each mixture (solute, flux and additive) was put into a platinum crucible. The Pt crucibles were heated to 600-900 °C and held at each temperature for several hours. Subsequently, they were cooled to room temperature at various cooling rate. The crystal products were separated from the remaining flux in warm water. The obtained crystals were studied using X-ray diffraction, scanning electron microscopy, and transmission electron microscopy.

High quality, superlong CoO whiskers were successfully grown by the cooling of chloride fluxes. The obtained whiskers had average size of up to 1200 μm × 1.2 μm and aspect ratios of up to 1000. The shape and crystal phase of cobalt oxides strongly depended on the temperature, kinds of carbon species, and cooling rate. CoO whiskers were obtained even without addition of carbon species at relatively higher temperature. In case of activated carbon, CoO whiskers could not be obtained. These results indicated that mild oxidative atmosphere in flux is key factor to grow one-dimensional shape. For growth of whiskers, cooling rate over $100°C \cdot h^{-1}$ were required.