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Splitting and Flattening of Helical Carbon Nanofibers by Acid Treatment

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Helical carbon nanofibers (HCNFs), such as the carbon nanocoil (CNC) and the carbon nanotwist (CNTw), were synthesized by catalytic chemical vapor deposition using a substrate. HCNFs are classified into round and angular types according to the fiber cross-sections. When four types of HCNFs (round-type CNC and CNTw, angular-type CNC and CNTw) were acidified in a 30% hydrogen peroxide solution, only the angular type CNC was found to show a drastic shape change. The shape change observed was a splitting followed by a flattening of the angular type CNCs. The CNC was split into two or three thinner flat fibers. As a function of the treatment temperature, the weight of the CNCs decreased above 80 °C and the CNCs were etched effectively at 140 °C. The longer the reaction time the lower the weight of the CNCs, and the weight loss reached a saturation point when the reaction time was greater than 45 min at 140 °C. The splitting and flattening of the CNC occurred during the weight loss process. To analyze the CNC structure, electron tomography of the as-grown and acid-treated CNCs was obtained using a computerized tomograph system with transmission electron microscopy (TEM). The 3D-images were constructed using the TEM images collected at different tilt angles. The 3D image reconstructions showed that the CNCs had a tubular structure and were composed of several helical fibers which act as frames.

Keywords: Carbon Nanocoil, Acid Treatment, Splitting and Flattening, Transmission Electron Microscopy, Electron Tomography.

1. INTRODUCTION

A helical carbon nanofiber (HCNF), which is a coiled or twisted carbon nanofiber, is synthesized by the catalytic chemical vapor deposition (CVD) method.[1–10] HCNFs are considered to be potential materials for electron field emitters,[5, 11–13] fillers in materials[14] and nano-springs.[15] HCNFs can be categorized into two types: a carbon nanotwist (CNTw) which has a twisted shape without an inner diameter[4, 5] and a carbon nanocoil (CNC) which has a spiral spring shape with an inner diameter. In most cases, the CNTw and the CNC tend to be synthesized with a Ni-based and a Fe-based catalyst, respectively. We have used the Ni–Cu- or Ni–Sn-based catalysts for the synthesis of the CNTw and the Fe–Sn-based catalyst for the synthesis of the CNC.[6, 7] The CNTw of nearly 100% purity can be prepared with a uniform shape and fiber diameter, whereas the CNC has been prepared with only a 30–40% purity which lacks uniformity.

As mentioned above, the purity of the CNCs prepared by CVD was rather low. Short carbon fibers and carbonaceous particles with no specific form are co-produced with CNCs. Carbonaceous particles are also generated as impurities when CNFs and carbon nanotubes (CNTs) are synthesized and can be removed by acid treatment in the same manner as disposing of catalyst particles.[16] Some as-prepared CNCs were unexpectedly found to split into several CNCs with a flattened fiber shape when processed using an acid treatment. Consequently, it is important to understand the basic structure and growth mechanism of the CNCs by analyzing the flattened fiber species.

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