

# Carbon nano-materials and micro-materials prepared in graphite boat

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## Abstract

Carbon nanomaterials and micromaterials were prepared using Ni/activated carbon nanotubes as catalyst that were placed in a graphite boat. The products were characterized by TEM. Most of them were carbon nanotubes (CNTs) that interlaced with each other. The CNTs have curled morphology with even diameter about 10 ~ 20 nm. Several kinds of interesting carbon materials were found in the products: very long carbon nanocoils with flake structure, carbon nanotube coils, carbon microcoils and carbon microfibers with fishbone structure. The coil pitch of carbon nanotubes coils and carbon microcoils are about 250 nm and 530 nm, respectively.

## Introduction

Activated carbon nanotubes are one kind of outstanding catalyst-support materials, since they have large surface areas, modified surface properties and pore structure [1-3]. Activated CNTs supported metals (Ni, Co and Fe) have been used for catalytic decomposition of hydrocarbons to obtain carbon nanomaterials and micromaterials, such as carbon nanotubes, carbon nanofibers and carbon nanocoils/microcoils, etc [4-6]. Those results show that carbon nanotubes have very high selectivity and efficiency to synthesize fibrous carbon materials. In this paper, we prepared some special kinds of carbon materials in a graphite boat used the activated carbon nanotubes (ACNTs) supported Ni as catalyst. The structures and morphologies were characterized by TEM.

## Experimental

CNTs were produced by catalytic pyrolysis method [7] and activated by KOH [3]. Figure 1(a) shows the representative CNTs that have straight morphologies and smooth walls. The diameters of most CNTs are in the range of 40 ~ 100 nm. The TEM image of ACNTs is shown in Figure 1(b). As can be seen, the outer walls but also the inner walls of ACNTs were etched irregularly and the inner diameter of ACNTs became larger than that of the non-activated CNTs.

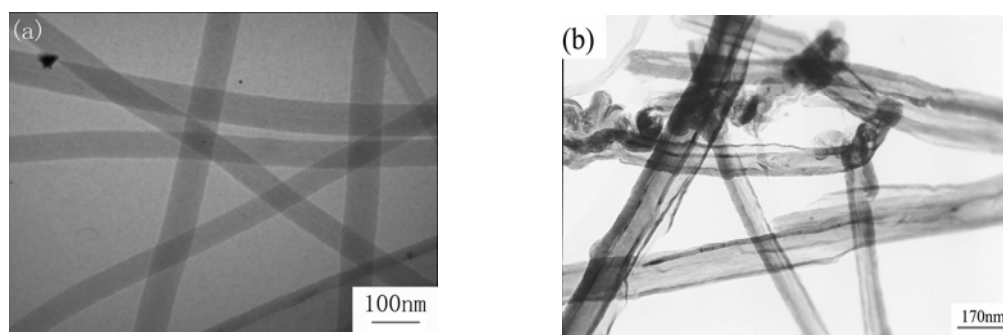


Figure 1 Typical TEM images of non-activated CNTs and activated CNTs

For loading of Ni catalyst, ACNTs were impregnated in  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  ethanol solution followed by sonication for 30 min. The mass ratio of Ni to ACNTs = 1 : 10. Then the Ni-containing ACNTs were obtained. The preparation of carbon materials was carried out in a horizontal furnace with a quartz tube (100 mm inner diameter and 1200 mm length) on about 200 mg Ni-containing ACNTs in a graphite boat in nitrogen atmosphere (120 sccm). When the reaction temperature reached, hydrogen was firstly introduced into the reaction chamber to reduce metal catalyst with cutting off nitrogen. After 30 min, a gas mixture of acetylene, thiophene and hydrogen was introduced. The reaction conditions were: reaction temperature 800 °C, reaction time 30 min, the gas flow rate of acetylene 100 sccm, and the total hydrogen 450 sccm with 150 sccm hydrogen bubbling through thiophene.

### Results and Discussion

Figure 2 shows the TEM images of the products. Most of the products are the curled carbon nanotubes, as showed in Figure 2(a). The diameters of those carbon nanotubes are about 10 ~ 20 nm. The very interesting thing is that some special kinds of carbon nanomaterials and micromaterials were also observed, which is showed in Figure 2(b) ~ (d). Figure 2(b) shows one carbon nanocoil whose diameter is about 120 nm and length is about 100  $\mu\text{m}$ . The aspect ratio is about 800. The inserted figure (1) is the amplified image of the tip part that contains a nickel catalyst particle. The inserted figure (2) shows the amplified image of the middle part of the carbon nanocoil. As we can see, the coil is composed of two helix carbon sheets, which is different from the ordinary carbon coils that are composed of round carbon fibers. The morphology of the carbon nanocoil is very similar to that of DNA. Some double-coiled carbon nanocoils were also found in the products as showed in Figure 2(c). The diameter of the tube is about 50 nm with 200 ~ 300 nm of the coil diameter. Figure 2(d) shows one fish-bone carbon micro-fiber. This carbon micro-fiber has double-layer structure. The inner layer is main bone whose diameter is about 100 nm, while is outer layer is side bone whose diameter is about 350 nm. This is a very special kind of carbon fiber. Those special kinds of carbon materials are not the main products, so the research is still being carried out in order to prepare one kind of those carbon materials selectively.

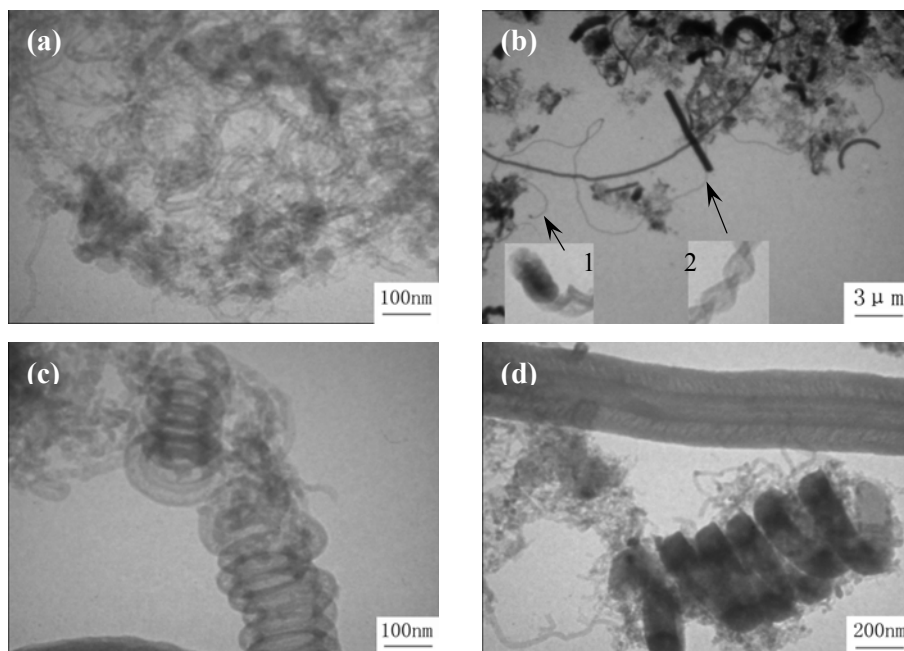


Figure 2 TEM images of carbon nanomaterials and micromaterials

### **Conclusions**

Some special kinds of carbon nanocoils and carbon microfibers were prepared using Ni/ACNTs as catalyst in a graphite boat, such as the long-aspect-ratio carbon nanocoils, double-coiled carbon nanotube coils and fish-bone carbon microfiber.

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