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Growth and Characterization of Carbon Nanostructures

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Abstract. Carbon nanostructures were grown by arc-discharge method and characterized by SEM, AFM, STM, and XRD techniques. We observe broadening in the radial distribution of nanofibers grown with Co and Ni application. The nanofibers grown with Co/Ni application were straighter and shorter in length than nanofibers without Co/Ni application. This might be due to catalyst particle poisoning resulting in termination of the growth process earlier than expected.

Keywords: Carbon nanostructures, nanotubes, nanofibers, arc-discharge in solution.

PACS: 73.63.Fg, 74.78.Na, 78.67.Bf, 78.67.Ch, 79.60.Jv.

INTRODUCTION

Carbon nanostructures in the form of carbon nanofibers, nanotubes (single wall carbon nanotubes, SWNT and multiwall carbon nanotubes, MWNT), and carbon nanoballs attract both scientific and industrial attention due to their extraordinary properties [1]. Among these, carbon nanotubes (CNTs) are most widely studied one. Nanostructure growth can be classified under three main techniques: arc-discharge, laser vaporization and chemical vapor deposition. These methods can satisfy the needs for scientific research, however, fail to provide sufficient, inexpensive yield of nanostructures needed for industrial applications.

In arc-discharge method, two electrodes brought to close separation under low pressure background gas. CNTs were actually discovered while trying to grow fullerenes only known form of carbon nanostructures then [2]. In this method anode electrode can be doped with catalyst particles to grow single wall carbon nanotubes (SWNT). Pumping to very low base pressures, followed by several purging steps, necessary cooled vacuum motion feedthroughs to keep electrode separation constant, and breaking of the vacuum to collect soot after growth, make this method very time consuming and expensive, thereby increasing end product cost with low yield.

It was first demonstrated by Ishigami *et al.* that nanostructure growth by arc-discharge can be achieved in a more simplified manner [3] by having protective environment around the electrodes. This environment

can simply be a solution (liquid nitrogen, deionized water, etc.) not necessitating complex growth process. Sano *et al.* [4] proposed a growth mechanism for this method leading to nanostructure growth. With a simple automation, this method holds promise to meet industry requirements of mass production of carbon nanostructures.

EXPERIMENTAL

Two %99.99 pure graphite electrodes were used to create plasma discharge in de-ionized water ($R \sim 18.2 \text{ M}\Omega$). A DC power supply was used to run about 30A current through the electrodes. At electrode separation of about 1 mm the arcing can be started and can be sustained by continuously lowering the anode. The high temperature ($\sim 4000\text{K}$) [5] during arcing at the tips of the electrodes creates necessary large temperature gradient between arcing site and water. Large temperature at this site creates protective carbon vapor environment for nanostructure growth, at the same time boils some amount of water. Only anode electrode gets consumed by the process which provided carbon source. The experiment can continue as long as there is enough supply of anode electrode and by chilling of water.

Growth runs were separated into two groups. In the first group the "normal" growth proceeded as described above. In the second group, the cathode electrode was covered with Co/Ni at several Co to Ni ratio to serve as catalyst. At the end of growth

products were collected from the surface of the cathode and from the top and bottom of the deionized water supply

The grown material was studied by Scanning electron microscope (SEM), Atomic force microscopy (AFM), scanning tunneling microscope (STM) and x-ray diffraction (XRD).

Some growth runs were also annealed at 350-500 °C in stagnant air for various annealing times to remove amorphous carbon.

RESULT AND DISCUSSIONS

In normal growth mode nanofibers had an average length of about 1 μm with diameters ranging from 10-30 nm with majority 20 nm in diameters. Nanofibers were entangled with some amorphous carbon and nanospheres resembling spaghetti with meatballs. SEM picture of typical nanofibers grown by the normal mode are shown in Figure 1.

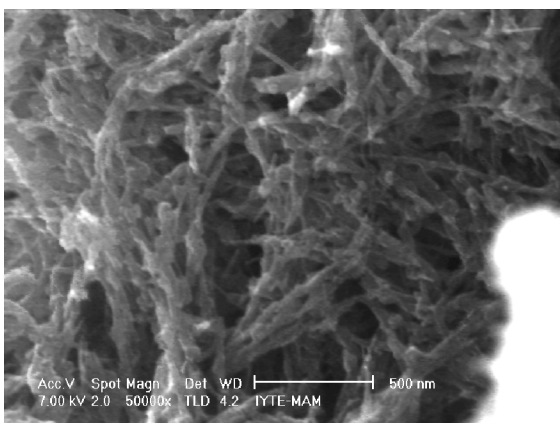


FIGURE 1. SEM picture of the nanofibers grown without Co/Ni.

We saw a widening in the radial distribution of nanofibers grown with Co and Ni ranging from 5nm-50nm. This broadening might be due to Co and Ni particle sizes, i.e. larger catalyst particles might promote growth of larger diameter nanofibers. The nanofibers grown with Co/Ni application were straighter and shorter in length than nanofibers without Co/Ni application, resembling balls and sticks (Figure 2). This structural change can also be explained by suggested bottom growth mechanism [6] and catalyst particle poisoning resulting in termination of the growth process earlier than the normal mode.

AFM and STM measurements also supported the structures observed by the SEM measurements. XRD data exhibit sharp (002) peak symmetric about $2\theta=26^\circ$. A weak (100) peak was also observed which is present if sample contains MWNTs.

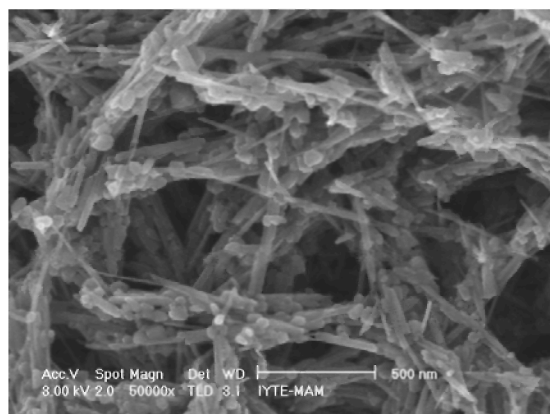


FIGURE 2. SEM picture of the nanofibers grown with Co/Ni.

CONCLUSIONS

Carbon nanostructures were grown by arc-discharge method and characterized by several techniques. This method remains viable method to grow large amount of nanostructures for industrial applications.

The observed broadening in the radial distribution of nanofibers grown with Co and Ni can be explained by assuming catalyst particle sizes playing deciding role in growth of nanofibers. The nanofibers grown with Co/Ni application were straighter and shorter in length than nanofibers without Co/Ni application. This can also be explained by catalyst particle poisoning resulting in termination of the growth process earlier than the normal mode.

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