Synthesis of Carbon Nanotube by Chemical Vapor Deposition (CVD) Method

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***Abstract*-We synthesize the carbon nanotube (CNT) by chemical vapor deposition (CVD). The morphological images have been observed before and after the CNT growth on the Si/SiO_2/Co substrate. Scanning electron microscope (SEM) images confirmed the growth of CNT onto the Si/SiO_2/Co substrate. The SEM image of Si/SiO_2/Co substrate having no CNT was found little dark due to having Co catalyst on the top, whereas lots of amorphous carbon was existed on the Si/SiO_2/Co/CNT surface according to SEM image. The growing CNT has no regularity and directionality.**

***Index Terms*-Carbon Nanotube, Chemical Vapor Deposition, Morphology, Amorphous Carbon**

I. INTRODUCTION

Carbon nanotubes (CNTs) have been recognized originally by Sumiolijima in 1991 [1]. CNTs are made of carbon atom by rolling up a graphene sheet with tubular nanostructure. CNTs have large variety of physical properties due to having different individual graphene layer which is rolled up into a tube. The wide variety of electronic structures in combination with a mechanically strong nanoscale lattice and outstanding optical properties are among the main reasons for the large interest in using CNTs in future electric and optical applications [2]. Due to having many interesting features and applications in the field of electronics, optics, and medicines, it needs precise fabrication and quality assurance.

There are several methods to fabricate the CNT such as laser ablation, arc discharge, chemical vapor deposition (CVD) and so on. Among them chemical vapor deposition (CVD) method is easy and cost effective. This method is capable of high-quality, defect free and large-scale production of CNT. Furthermore, it could be possible to gain control over the various parameters which are involved in CVD experiments. In this process, P^+-Si with 200 nm thickness of SiO_2 is used as a substrate. Cobalt nanoparticles and ethanol are used as the catalyst and carbon source respectively for CNT growth.

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CVD synthesis can be divided into two main categories: Supported catalyst growth and floating or gas-phase growth. In the supported growth process, the catalyst is prepared in advance and deposited in some way on a support medium. This is inserted into a flow apparatus, typically a tube at atmospheric pressure in a temperature-controlled furnace, where it can be exposed to flowing carbon-rich gas at elevated temperatures (typically 500–1100^{o}C) for a certain period. For floating-catalyst growth, a high-temperature flow-furnace is still used, however the catalyst and carbon source are injected into the system simultaneously, either in gas phase or as an aerosol, where the subsequent decomposition and reaction can either occur completely suspended in the gas flow or following self-deposition on a surface in the reactor.

Supported growth is the most widely used CVD-based method of nanotube synthesis. The type of catalyst used, the way in which it is prepared and the choice of support all have significant impact on the nature of the material produced. The most common support media are metallic Si and Si wafers [3–11] or various SiO_2 based materials, [12–15] although graphite and various metallic thin films have also been used [16–19]. Fe, Co, and Ni have proven to be the most successful catalysts, with Fe being the most frequently used. The catalysts are often deposited as small particles or islands on the substrate and result in localized mats of nanotubes sprouting from the catalyst covered areas. Electrochemical, lithographic, sputtering, spin coating, and other techniques are commonly used for catalyst deposition.

An interesting observation is that the diameter of the carbon nanotube becomes proportional to the particle size when it is reduced to the scale of single tubes (a few or few 10s of nanometers) [13] making it possible to tune the nanotube diameter by controlling the catalyst deposition. Acetylene is the most widely used hydrocarbon, with methane, ethylene, propylene, and a few aromatic compounds also finding common use. Preparing the catalyst in advance provides the opportunity to define specific patterns on the substrate. The ability to control the position and direction of growth could have significant benefits, including simple routes to achieving nanotube-based electronic devices. Techniques such as photolithography, [7,20] electron beam lithography, [5,21,22] laser etching, [14,15] micromoulding, [23] and even ink-jet printing [24] have been successfully used to achieve patterned nanotube growth.

Both Cheng et al. [25] and Yang et al. [26] describe the production of macroscopic fibers of SWNT using a combination of ferrocene, benzene, and thiophene in a dual-furnace system. The fibers, observed to be up to 100 mm in diameter and a few centimeters in length, were composed of numerous roughly aligned bundles of individual SWNT with diameters of 1.5–3.0 nm. Ago et al. observed the production of SWNT by injecting a colloidal solution of CoMo nanoparticles (11nm diameter) in toluene into a 1200^{o}C oven [27]. With the addition of 1% thiophene to the solution SWNT with diameters of 1.1–1.9 nm was favored, while using 10% thiophene resulted in only MWNT. A group at Rice University has developed a very successful method of SWNT synthesis using CO [28,29]. Bronikowski et al. useed high pressure jets (up to 10atm) of preheated CO to rapidly heat mixtures of iron pentacorbonyl in CO as they enter a high-temperature furnace (900–1100^{o}C) [29].

In this work, we used chemical vapor deposition (CVD) technique to fabricate the carbon nanotubes due to having some unique advantages compared to the other techniques. Arc discharge and laser vaporization are both batch-scale processes which ultimately limit their production capacities. However, CVD is most promising method which potentially offers controlled synthesis and continuous operation. The energy required is lower. The carbon feedstocks are abundant and inexpensive. The setup of process is simple and it is easy to control and manipulate. In this process, CNT are synthesized with high quality and large scale production. With these aforementioned advantages, CVD method was chose to synthesize CNT in this work. Characterization including SEM analysis has been carried out to ensure quality fabrication and to explore the correct mechanism for CNT production.

II. MATERIALS AND METHODS

Chemical Vapor Deposition (CVD) technique was used to synthesize CNT. In this system, ethanol was used as the carbon source to synthesize CNT. Temperature was controlled by a temperature controller. Argon gas, which was managed by a mass-flow controller, was used to keep the pressure inside the chamber stable before and after supplying ethanol. A rotary pump was used to make vacuum inside the chamber. Chamber's pressure value was displayed by a manometer. In order to fabricate CNT on $Si/SiO_2/Co$ substrate, a commercial SiO_2 (100nm)/p^{+}-Si substrate was cleaned with piranha solution. Then, we deposited 5-nm-thick cobalt film as the catalyst onto the substrate by using e-beam evaporator MUE-ECO-EB (ALVAC) with deposition rate of 0.01 nm/s. The substrate with Co catalyst thin film was introduced into the CVD system for CNT growth. Firstly, the quartz tube chamber was evacuated to around 10 Pa to pull out residual oxygen gas in the upper part of ethanol bottle at 800°C. Then the furnace was cooled down in order to put the sample into chamber. The chamber was then evacuated while increasing temperature. After reaching a desired temperature (850^{o}C), Ar gas (100 sccm) was introduced by mass flow controller for 10 minutes in order to get thermal equilibrium state of the whole system. Then ethanol was introduced into the chamber so that the pressure was kept fixed at 400 Pa for 30 min. After growing CNTs, the chamber was cooled down to room temperature naturally in order to take out the sample. The as-grown CNT was investigated by scanning electron microscopy (SEM) image.

III. RESULTS AND DISCUSSION

SEM analysis has been conducted to confirm the fabrication of CNTs. Fig. 1(a) and 1(b) exhibits the morphological images of $Si/SiO_2/Co$ substrate and CNT grown on $Si/SiO_2/Co$ substrate respectively. It seemed that, lots of amorphous carbon was existed on the $Si/SiO_2/Co/CNT$ surface according to SEM image. The magnification for both the SEM images was 2μm.

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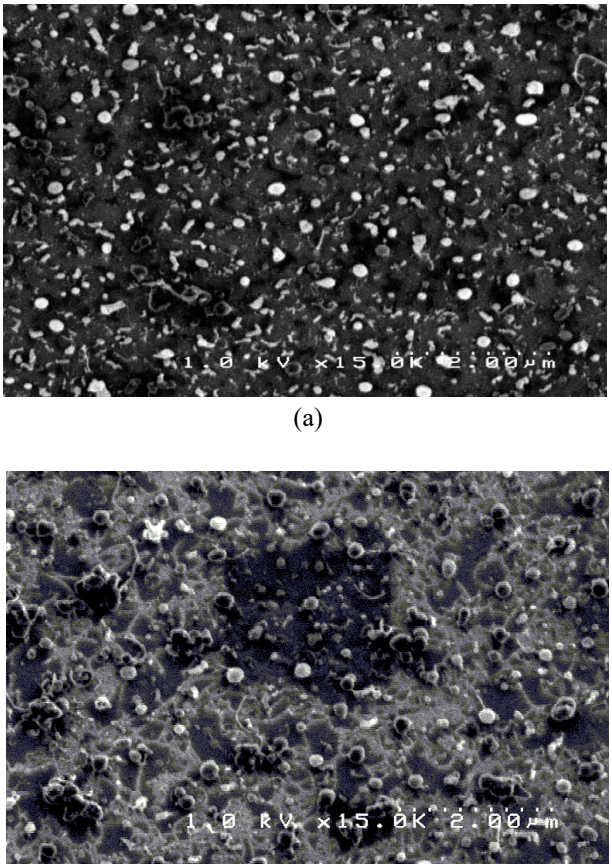

Fig. 1. SEM image of (a) $Si/SiO_2/Co$ substrate
(b) $Si/SiO_2/Co$ substrate with randomly grown CNT.

Fig. 1(a) exhibit the SEM image of $Si/SiO_2/Co$ substrate which looked little black and this is due to the cobalt catalyst on the top. However, CNT randomly grown on $Si/SiO_2/Co$ substrate is shown clearly in Fig. 1(b). The growing CNT has no regularity and directionality. But the morphological image confirmed the growth of CNT onto the $Si/SiO_2/Co$ substrate.

IV. CONCLUSION

Chemical vapor deposition (CVD) shows the greatest promise as a true industrial scale process, particularly floating catalyst techniques which, in principle, can be operated in a continuous fashion. The prospects of controlled patterned growth are also very favorable for applications like molecular scale sensors, mechanical and electronic devices. Due to having such unique character and advantages we chose to synthesize CNT by CVD method. We observed the morphological images before and after the CNT growth on the $Si/SiO_2/Co$ substrate. Scanning electron microscope (SEM) images confirmed the growth of CNT onto the $Si/SiO_2/Co$ substrate. The SEM image of $Si/SiO_2/Co$ substrate having no CNT was found little dark due to having Co catalyst on the top, whereas lots of amorphous carbon was existed on the $Si/SiO_2/Co/CNT$ surface according to SEM image. The growing CNT has no regularity and directionality.

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