Fabrication and Characterization of Fe:Ni Nanoparticles for Carbon Nanotube Growth

Tippawan Saipin, Udom Tipparach* and Supakorn Pukid

Department of Physics, Faculty of Science, Ubon Rajathanee University, Warinchamrab, Ubon Ratchathani 34190, Thailand

**Corresponding author. E-mail: udomt@hotmail.com*

ABSTRACT

We have studied the growth of Fe:Ni nanoparticles on silicon substrates by a thermal evaporation method and surface morphology by using an Atomic Force Microscope (AFM) and Scanning Electron Microscope (SEM). The nanometer-sized particles of Fe:Ni, served as a catalyst for the growth of carbon nanotubes, were prepared under different argon atmospheres of pressures ranging from 200 mtorr to 500 mtorr. The catalyst particle sizes favorable for the chemical vapor deposition growth of carbon nanotubes are between 20 nm and 50 nm in diameters.

Key words: Metal nanoparticles, Catalyst, Carbon nanotubes

INTRODUCTION

Since being discovered by Iijima in 1991, carbon nanotubes (CNTs) have been considered as one of the most attractive nanomaterials due to their unique physical properties. The feasible potentials for technological applications of carbon nanotubes are gas sensors, battery electrodes and field-emitting devices. Several growth methods have been developed for the synthesis of carbon nanotubes such as arc discharge and thermal chemical vapor deposition (CVD). The choice of the growth process depends on the purpose of the use of carbon nanotubes. Carbon nanotubes growth by thermal CVD is both efficient and versatile (Li et al., 1996; Kong et al., 1998; Li et al., 2001; Wongwiriyapan et al., 2005) and it is promising to study an individual carbon nanotube as electron field emission devices (Chai, 2004; Saito et al., 2000), therefore, the technologically most relevant process for the synthesis of these interesting nanomaterials. Thermal CVD growth of nanotubes from hydrocarbon precursor molecules such as methane and ethanol is facilitated by nanoparticle sizes of transition metals (Campbell et al., 2002; Nerushev et al., 2003;) such as iron, cobalt and nickel at elevated temperatures. Identification and control of critical growth parameters are crucial for process optimization and development of growth models. The dimensions of carbon nanotubes have an effect on performances of carbon nanotube-based devices such as field emitters. The purpose of this work is to study the effect of nanoparticle size of an Fe:Ni catalyst on the growth of the carbon nanotubes by the thermal CVD technique.

EXPERIMENTAL PROCEDURES

Catalytic nanoparticles of iron and nickel were prepared on silicon (001) substrates by thermal evaporation. The set up apparatus are shown in Figure 1. The powder of 10 milligrams of iron and nickel metals with the ratio of 50:50 by weight was put in a tungsten boat and then placed the boat in a vacuum chamber. After the chamber vacuum was pumped down

to 10^{-6} Torr, the inert gas Ar was filled into the chamber and the Ar pressure was maintained at about 200–500 mtorr. The Fe:Ni powder was evaporated by applying electric current about 80 Amperes to the boat. The nano-sized particles of an Fe:Ni catalyst were deposited on silicon substrates with different Ar atmospheres, mainly 200, 300 and 400 mtorr. By changing the pressure of the Ar gas and distance between the boat and the substrates, we can roughly adjust the catalytic particle sizes.

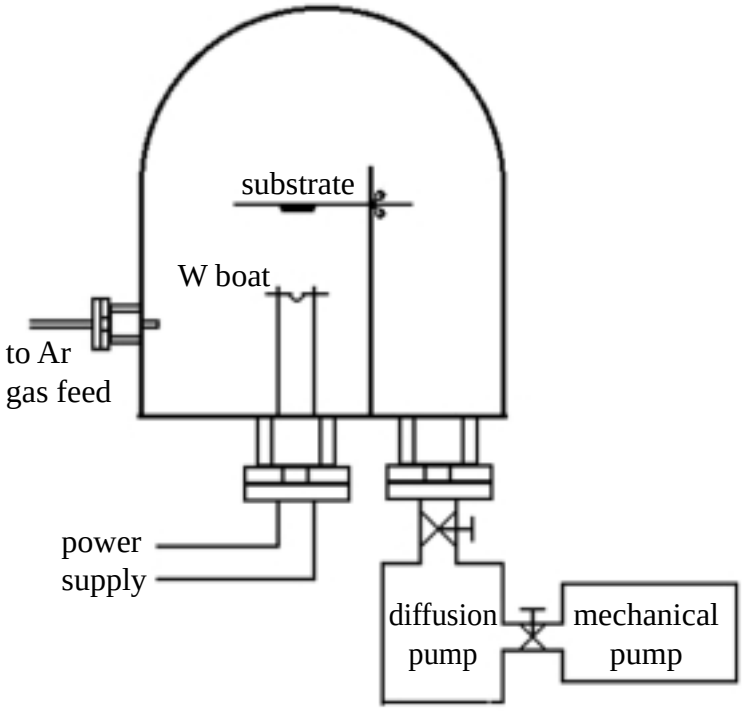


Figure 1. Schematic diagram of the thermal evaporation system.

The sizes of the particles were determined by AFM and SEM. Next, the silicon substrates coated with Fe:Ni nanopartices were loaded with face up direction on an alumina boat and then the boat was put into a quartz tube furnace as shown in Figure 2. The furnace was turned on and set the temperature at 900°C and at the same time Ar and H_2 were introduced to the reactor (tube furnace) with the flow rate of 70 sccm and 65 sccm respectively. When the temperature reached the setting point, 900°C, and it was stable, then H_2 gas was turned off. The Ar gas was used to purge the reactor and to dilute the H_2 and H_2 gas was used to prevent the oxidation of Fe:Ni catalyst while raising the temperature. After that methane (CH4) gas was introduced with the flow rate of 10 sccm for 30 minutes while Ar gas was allowed to flow continuously. CH_4 gas was used as a source of carbon. Finally, CH_4 gas and temperature controller were turned off, but Ar gas was allowed to flow for 15 minutes and let the samples cool down to room temperature. The samples were characterized by SEM and Energy Dispersive Spectrometer, EDS.

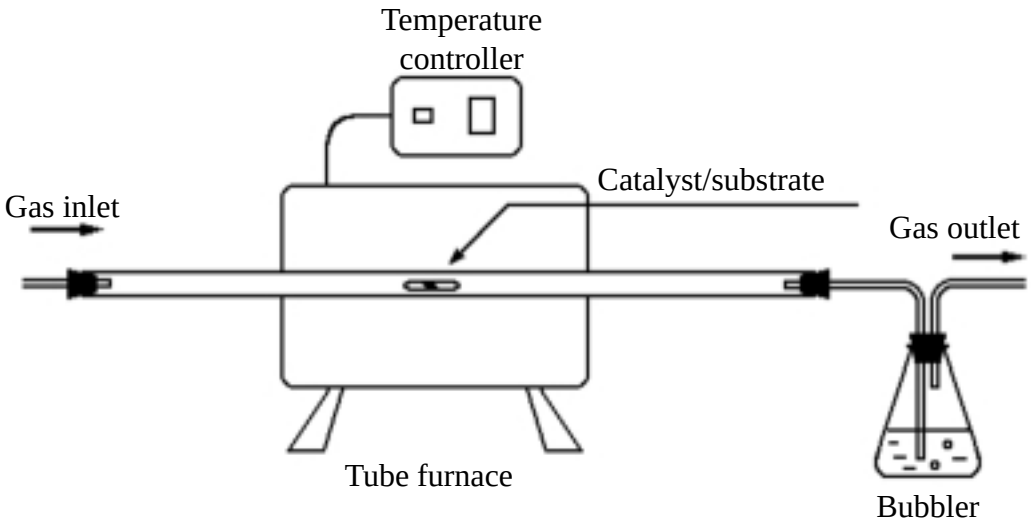
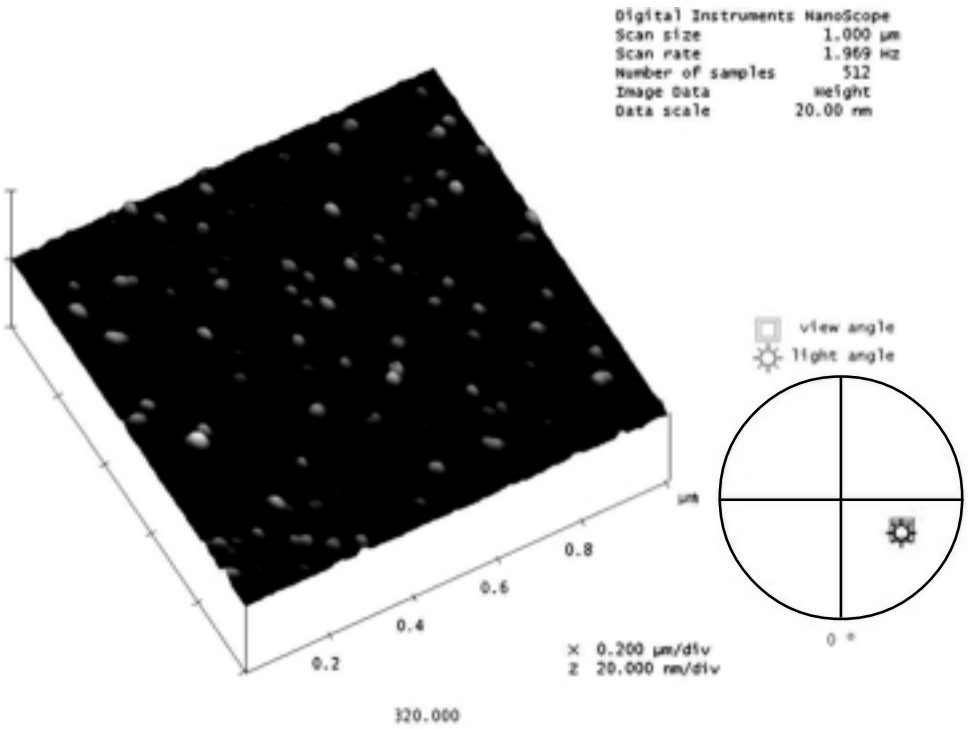


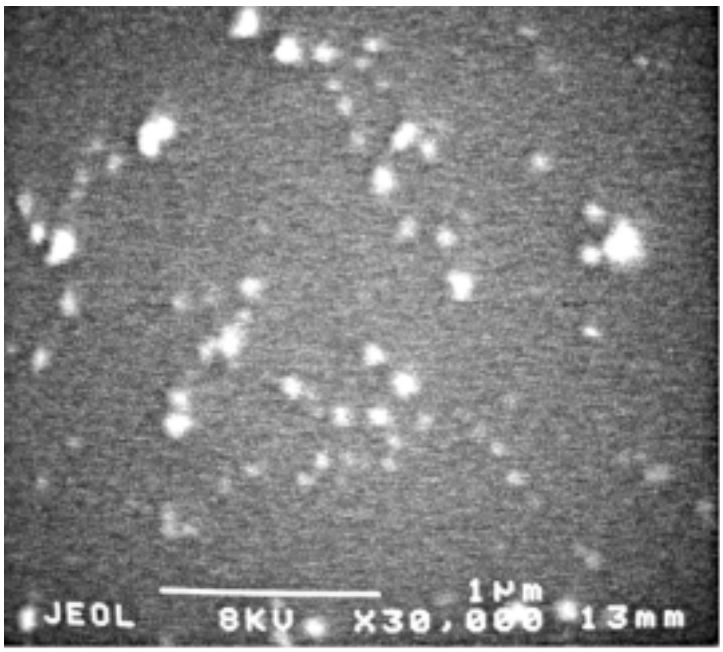
Figure 2. Experimental setup of the carbon nanotube growth.

RESULTS AND DISCUSSION

The catalysts deposited with 200 mtorr, 300 mtorr and 400 mtorr Ar atmospheres have a film shape, particle shape (as shown in Figure 3) and amorphous respectively. The particle sizes of the catalyst in Figure 3 are approximately between 10 nm and 100 nm. Carbon nanotubes were observed on the silicon substrates with only the catalytic nanoparticle shapes as shown in Figure 4. Undesired carbon such as carbon fibers and amorphous carbons may occur besides carbon nanotubes. The straight lines with nanometers in diameter shown in Figure 4 are carbon nanotubes and the rest of then are carbon fibers and amorphous carbon. Our carbon nanotubes have diameters between 10 nm and 20 nm. The lengths of the carbon nanotubes range from 10 µm to 40 µm. They are likely expected to be multiwalled carbon nanotubes. The growth mechanism seems to follow the theory of Baker (1998) which states that carbon atoms, produced at the surface of the catalytic particle by decomposition of the hydrocarbon gas, are dissolved as interstitials in the metal lattice of the particle. After reaching a critical concentration, a graphene layer forms at the particle surface, initiating filament (carbon nanotubes) growth. In this view, the catalytic nanoparticle is required to seed carbon nanotubes. This result is in agreement with the experiments observed by Li et al., (2001) and molecular dynamic simulation done by Ding et al., (2004).

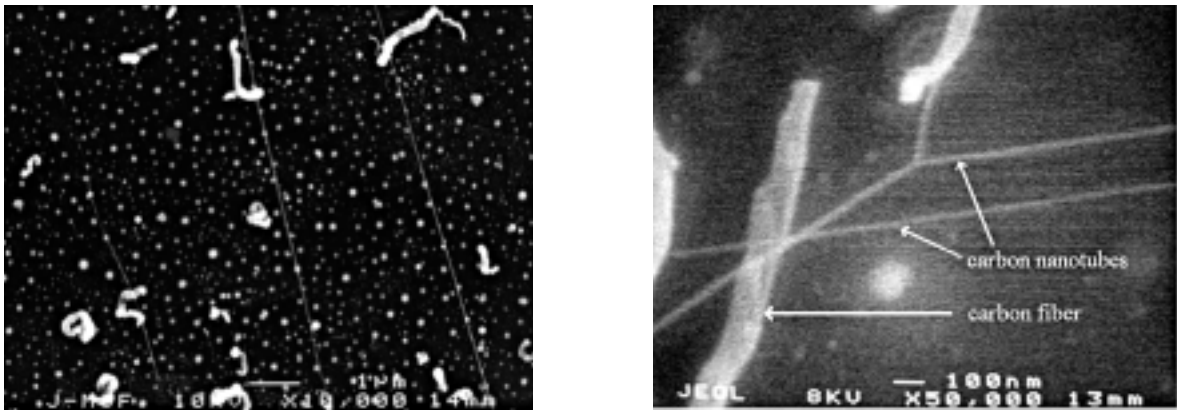


1(a)



1(b)

Figure 3. Images of Fe:Ni catalyst on the silicon substrate prepared with 300 mtorr Ar atmosphere, (a) AFM image, (b) STM image.



4(a) 4(b)

Figure 4. SEM images of carbon nanotubes grown on the silicon substrate coated with Fe:Ni nanoparticles at 300 mtorr of Ar atmosphere.

CONCLUSION

We conclude that Fe:Ni nanoparticle-sized metals prepared by thermal evaporation technique under Ar atmosphere of 300 mtorr can be used as a catalyst for the growth of carbon nanotubes. Film shape or amorphous shape of a catalyst may not be favorable for growing of carbon nanotubes. Carbon nanotubes are likely to grow on the nanoparticles and the nanopartices (nanoislands) may serve as a seed for the growth of carbon nanotubes. The carbon nanotube growth is sentitive to the catalyst size. Under our conditions such as the flow rates, growth temperature and carbon source used, the best sizes of the catalytic nanoparticles for carbon nanotube growth by thermal CVD are in the range of 20 to 50 nanometers in diameters.

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