CHAPTER 3

Experimental

Experimental study details are given in this chapter. In the first section, Iron, Cobalt Carbon Nanotubes fabrication method and experimental aims were explained, and in the second part CNT growth by low pressure chemical vapor deposition method (LPCVD), model (CV-6SLX) was explained. The last section is focusing on characterization technique which is scanning electron microscopy (SEM, model: JSM–6460 LV), results was explained in chapter 4.

3.1 Experimental tools

Carbon nanotubes synthesis by low pressure Chemical vapor deposition (LPCVD), model (CV-6SLX) is used to produce MWNTs by metal particles (iron and cobalt) under different reaction conditions. List of recipes and description of the growth process and reaction conditions should be optimized by (Argon gas flow rate, Acytelen C_2H_2 and Hydrogen H2 gases flow rates for dehydrogenation processes, applicable time t and pressure P for CNT growth). Scanning electron microscopic SEM (model: JSM–6460 LV), used to characterize carbon nanotubes CNTs.

3.2 Experimental Aims

1. To produce Carbon Nanotubes (CNTs) using nanocatalysis materials synthesis as an Iron or Cobalt (Fe or Co) metallic materials.
2. To study the effects of acetylene gas rates and temperature variations in carbon nanotubes morphology.

3.3 Experimental Parameters

In this experiment the cobalt and iron nanoparticles powder (Fe, Co NPs) were bought with 99.99% purity The Fe and Co samples and sample holder were supported by ceramic and putted in LPCVD device, model (CV-6SLX). Each samples were annealed at 450, 650, 850, 950ºC respectively for 20 minutes in a flow of 100 sccm of Argon and 50 sccm of Hydrogen and then LPCVD growth was carried out by the addition of 10,20,30 and 40 sccm of acetylene C_2H_2 for constant timing at 20 minutes. All experiments processed under pressure of 30 torr. The total samples numbers were 16 for both iron and cobalt. Hereby, the surface morphology for each samples of the carbon nanotubes for iron and cobalt were studied by scanning electron microscope (SEM, model: JSM–6460 LV).

3.4 Experimental Process at LPCVD

Low pressure chemical vapor deposition system LPCVD method was used to synthesize CNTs. List of recipes and description of the growth process and reaction conditions should be optimized. The system consists of two parts, first part is a Lindberg/Blue M 1100 ºC Split Mini Furnace and the other part is its controller. All experiments were done at a high temperature so samples in a quartz boat were placed in a 1 inch diameter quartz tube. The upper temperature limit of the furnace is 1100 ºC. In this study, CNT growth experiments were performed at atmospheric pressure. Firstly, a catalyst pretreatment took place with Ar, H_2 or Ar-H_2 mixture for 20 Minutes at 450, 650, 850, and 950 ºC respectively. Ar gas sent into the system to remove the contamination and to prevent the oxidation of the samples, and H_2 gas sent into the system to prevent amorphous carbon formation and to provide the reduction from metal oxide catalyst to metal catalyst which are more suitable for CNT growth. After reaching the desired temperature C_2H_2 gas flow was started to initiate CNT growth. Ar and H_2 continued to flow during CNT growth. Different gas flow rates for Ar, H_2 and C_2H_2 were investigated. In this study growth temperature in the range of 450 to 950 ºC was studied.

Finally; Growth time was also another parameter for the CNT growth, one growth times were investigated, 20 min. When the growth was finished, the hydrocarbon gas was turned off first but H_2 gas and Ar gas was still flowing through the system and the temperature was set to 0 ºC so the system was left for cooling under again Ar, H_2 or Ar-H_2 mixture ambient (see appendix A).

3.5 Characterization Methods

Several instrumental techniques can be used to characterize CNTs catalytic result of Fe or Co catalysts. For example, transmission electron microscopy (TEM) is a helpful tool to show the investigation of the tubes' internal structure. The X-ray diffraction (XRD), small angle X-ray scattering (SAXS) Fourier-transformed infrared spectroscopy (FTIR), ultrasonic techniques, four-point probe, BET, SEM as well as high-resolution scanning electron microscopy might be focused to characterize the CNTs catalytic results.

Quality, structure and physical properties of carbon nanotbes depend strongly on the growth conditions e.g. temperature, catalyst and environment condition. All this makes a proper characterization of the Carbon Nanotubes (CNTs). Characterization Techniques Methods is a parametric study whose aim to find optimal growth and pretreatment conditions for high quality and high yield, therefore, through characterization of obtained CNTs is essential. Therefore, the catalyst was characterized by scanning electron microscopy (SEM), and purified and unpurified CNT samples were analyzed with SEM,

3.5.1 Scanning Electron Microscope Theory

SEM (Scanning Electron Microscopy) images the sample morphology by scanning the surface with a high energy beam of electrons. SEM is the first step to characterize the CNTs. Using SEM, morphology of CNTs, their dimensions and their orientations can be seen *(Thess, et al. 1996, Liu, et al. 2004, Li, et al. 2002)*. Diameters of CNTs also can be measured roughly with SEM. In light microscopy, a specimen is viewed through a series of lenses that magnify the visible-light image. However, the scanning electron microscope (SEM) does not actually view a true image of the specimen, but rather produces an electronic map of the specimen that is displayed on a cathode ray tube (CRT).

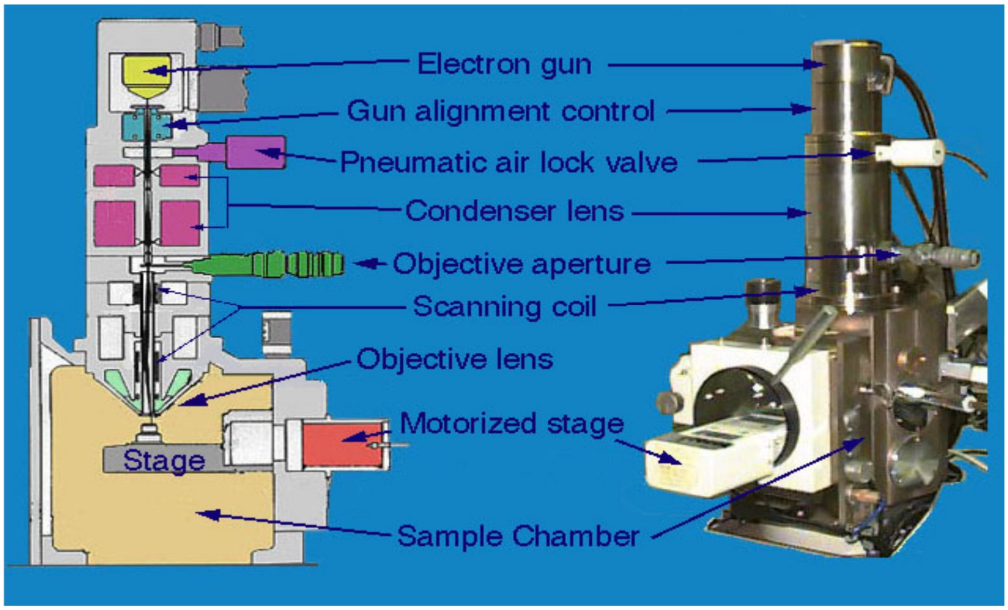

Figure 3.5.1 Scanning Electron Microscope(SEM*) (Source: Daenen, et al. 2003)*

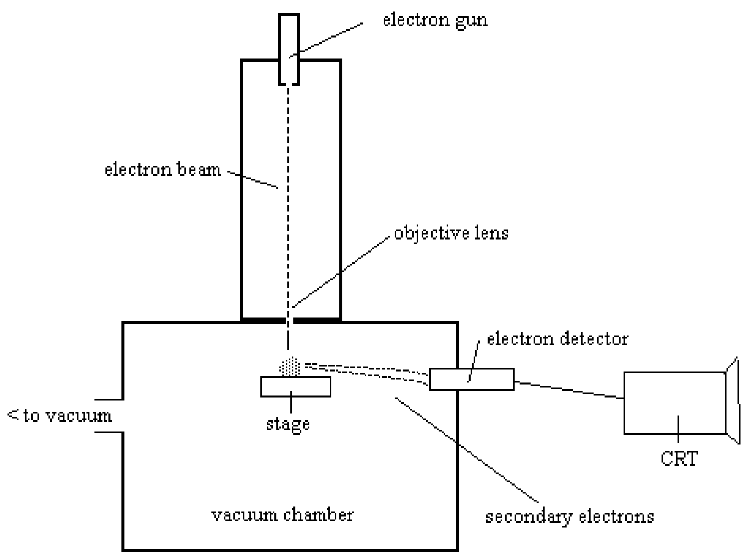

Figure 3.5.2 Schematic Diagram for SEM Open System
(Source: Daenen, et al. 2003)

Figure 3.5.1 and figure 3.5.2 shows a schematic for a generic SEM. Electrons from a filament in an electron gun are beamed at the specimen in a vacuum chamber. The beam forms a line that continuously sweeps across the specimen at high speed. This beam irradiates the specimen, which in turn produces a signal in the form of either x-ray fluorescence, secondary or backscattered electrons. The signal produced by the secondary electrons is detected and sent to a CRT image. The scan rate for the electron beam can be increased so that a virtual 3-D image of the specimen can be viewed.