CHAPTER III
EXPERIMENTAL

3.1 Materials and Equipments

Iron nitrate nanohydrate and alumina received from Aldrich are used for catalyst preparation. High purity hydrogen, argon and carbon monoxide are obtained from Thai Industrial Gas.

Reaction system shown in Figure 3.1 and 3.2 consists of quartz tube reactor, temperature controller, furnace and mass flow controllers.

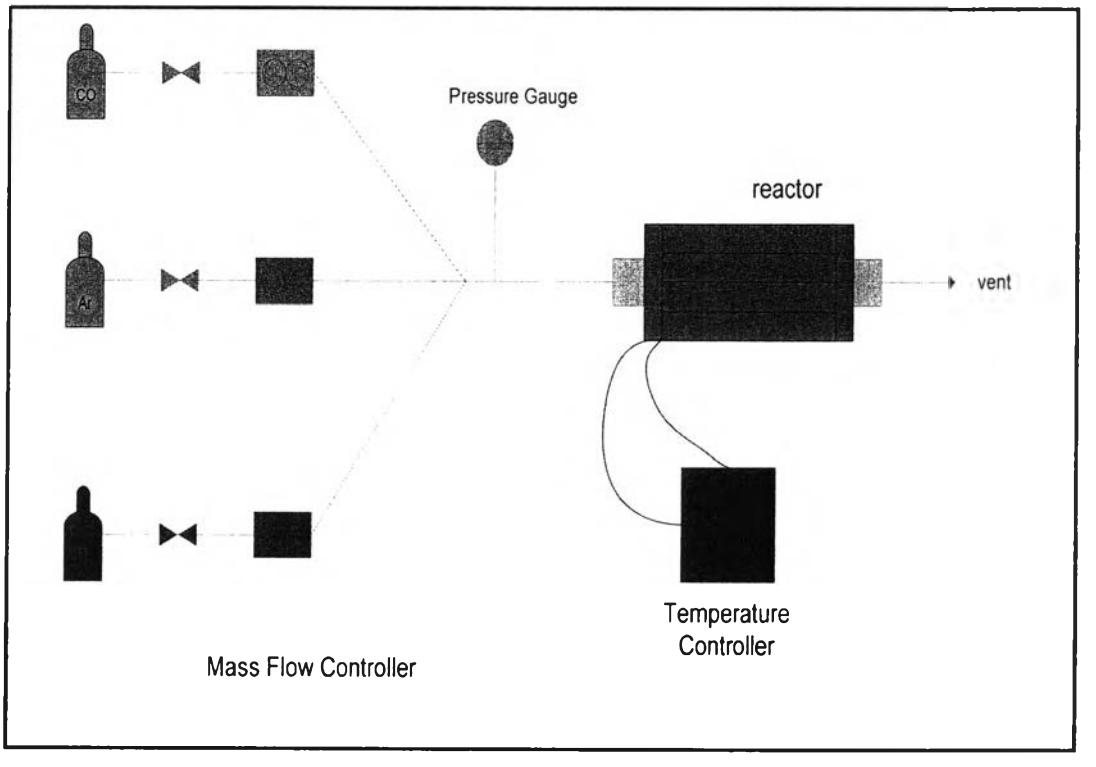

Figure 3.1 Schematic diagram of reaction system.

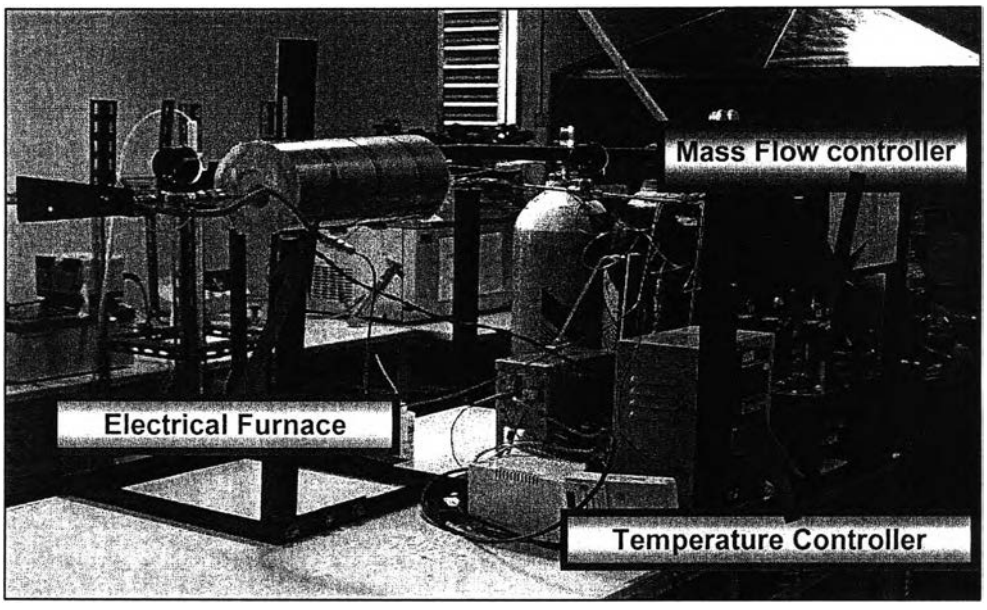

Figure 3.2 The photograph of the reaction system.

3.2 Methodology

The catalysts had been prepared first, and then carbon nanotubes were grown on them.

3.2.1 Catalysts preparation

Iron nitrate nanohydrate and alumina received from Aldrich are used as catalyst precursor. Incipient wetness impregnation (IWI) was applied to prepare Fe/Al_2O_3 at 1, 10, 20 and 30% loading. After impregnation, the catalysts were left at room temperature for 2 hours, dried over night and calcined at 600°C for 3 hours.

3.2.2 CNTs and CNFs growth

0.1 g. of prepared catalyst was put on a small ceramic boat and then placed at the center of the quartz tube reactor. Prior to synthesis, the catalyst was reduced at 600°C with 1:3 H_2/Ar for 3 hours before reaction with CO/H_2 at 400-900°C for 1 hour.

3.2.3 Characterization

The temperature-programmed reduction (TPR) experiments on the calcined catalysts were conducted in a Thermo Finnigan TPDRO 1100 equipped with a thermal conductivity detector (TCD). The system was operated by passing a continuous flow H_2 over approximately 50 mg of the calcined catalysts, while the temperature was linearly increased at a rate of 10°C/min. The intensities of thermal conductivity, which corresponds to the amount of hydrogen consumption by the samples, were plotted with the temperature.

The temperature-programmed oxidation (TPO) experiments on an CNTs and CNFs containing sample were conducted in a TPO apparatus equipped with a flame ionization detector (FID). The system was operated by passing a continuous flow of 2% O_2 in He balance over approximately 5 mg of the sample, while the temperature was linearly increased at a rate of 10°C/min. The intensities of the FID, which corresponds to the amount of oxygen consumption by the samples, were plotted with the temperature.

The SEM, JOEL model JSM-5200-2AE, was used at acceleration voltage of 20 kV.

Transmission electron microscopy was performed on a JEOL JEM-2000FX at an acceleration voltage of 160 kV. The sample was prepared by sonicating the spent catalyst in an isopropanol solution, putting a few drops of the suspension onto a lacey carbon grid, and then allowing it to dry.