CHAPTER III

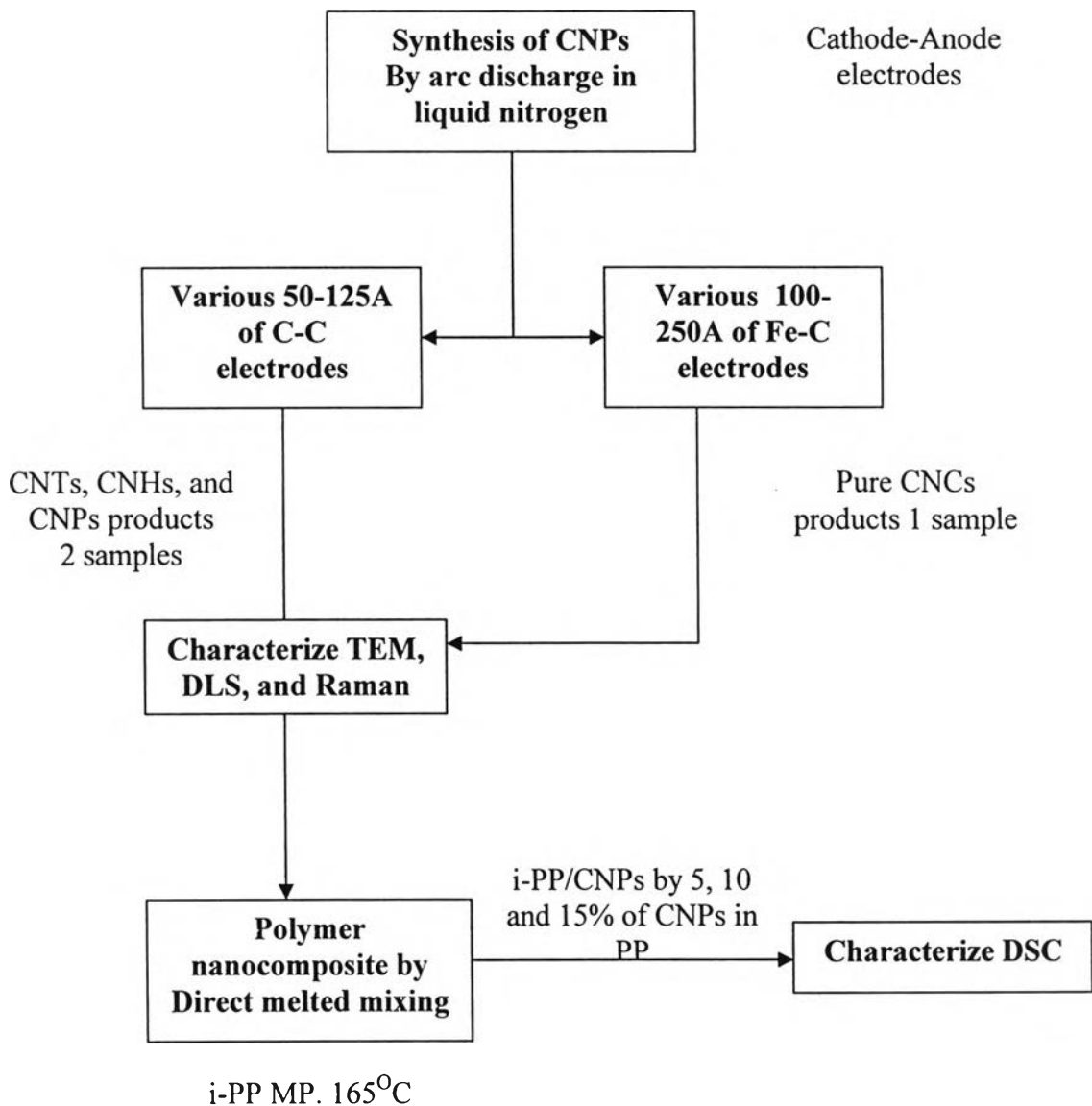# EXPERIMENTAL

3.1 Experiment procedure

Figure 3.1 Experiment procedure

3.2 Experiment method of arc discharge of synthesized CNPs

3.2.1 Materials and instruments

3.2.1.1 Pure graphite (ToyoTanso) diameter 6 mm and length 5 cm

3.2.1.2 Pure iron diameter 2 cm and length 2 cm

3.2.1.3 Welding power supply (ARC, DP300) current range 0-300A

3.2.1.4 Pyrex glass 2L. and Stainless cylinder 2L.

3.2.1.5 Liquid nitrogen (Thai industrial gases public company limited).

3.2.1.6 The experimental apparatus of arc discharge.

3.2.2 Arc discharge by C-C electrodes

A direct current (DC) welding power supply (ARC, DP300) has been used to generate the arc plasma. Pure graphite (ToyoTanso) anode and pure graphite cathode with diameters of 6 and 20 mm are used respectively as a movable anode and a cathode. The lengths of anode and cathode electrodes were 5 and 2 cm, respectively. Conduct experiments by varying electricity current of the obtained nanoparticles are range 50-125A. These electrodes are aligned in vertical in 2000 ml of liquid nitrogen felled in a Pyrex glass. The arc plasma is initiated by contacting two electrodes, and then keeping the gaps between the electrodes 1 mm to maintain stable discharge a certain period of time. After the arc discharge is terminated about 3-5 minutes per 1 batch, two types of products coal are. One is deposit at cathode end, and the other is fine particles at the vessel bottom and the vessel is leaved for 3 hrs to ensure that the dispersed particle is completely fallen down at vessel bottom. Then these products are dried in the oven $100^{\circ}C$ for 1 day, and kept in sample bottles for analysis later.

3.2.3 Arc discharge by Fe-C electrodes

This operation likes 3.2.2 arc discharge by C-C electrodes operation. For comparison, pure graphite anode and pure metallic cathode are also used.

A direct current (DC) welding power supply (ARC, DP300) has been used to generate the arc plasma. Pure graphite (ToyoTanso) anode with diameters of 3-6 mm and pure metallic cathode with diameters of 20 mm are used synthesizing carbon nanoparticles. The lengths of anode and cathode electrodes were 5 and 2 cm, respectively. Conduct experiments by varying electricity current of the obtained nanoparticles are range 100-250A These electrodes are aligned in vertical in 2000 ml of liquid nitrogen felled in a stainless cylinder. The arc plasma is initiated by contacting two electrodes, and then keeping the gaps between the electrodes 1 mm to maintain stable discharge a certain period of time. After the arc discharge is terminated, two types of products coal are. One is fine particles at the vessel bottom and the vessel is leaved for 3 hrs to ensure that the dispersed particle is completely fallen down at vessel bottom. Then these products are dried in the oven for 1 day, and kept in sample bottles for analysis later. Electrodes arrangement for arc discharge in liquid nitrogen is shown in Figure 3.2
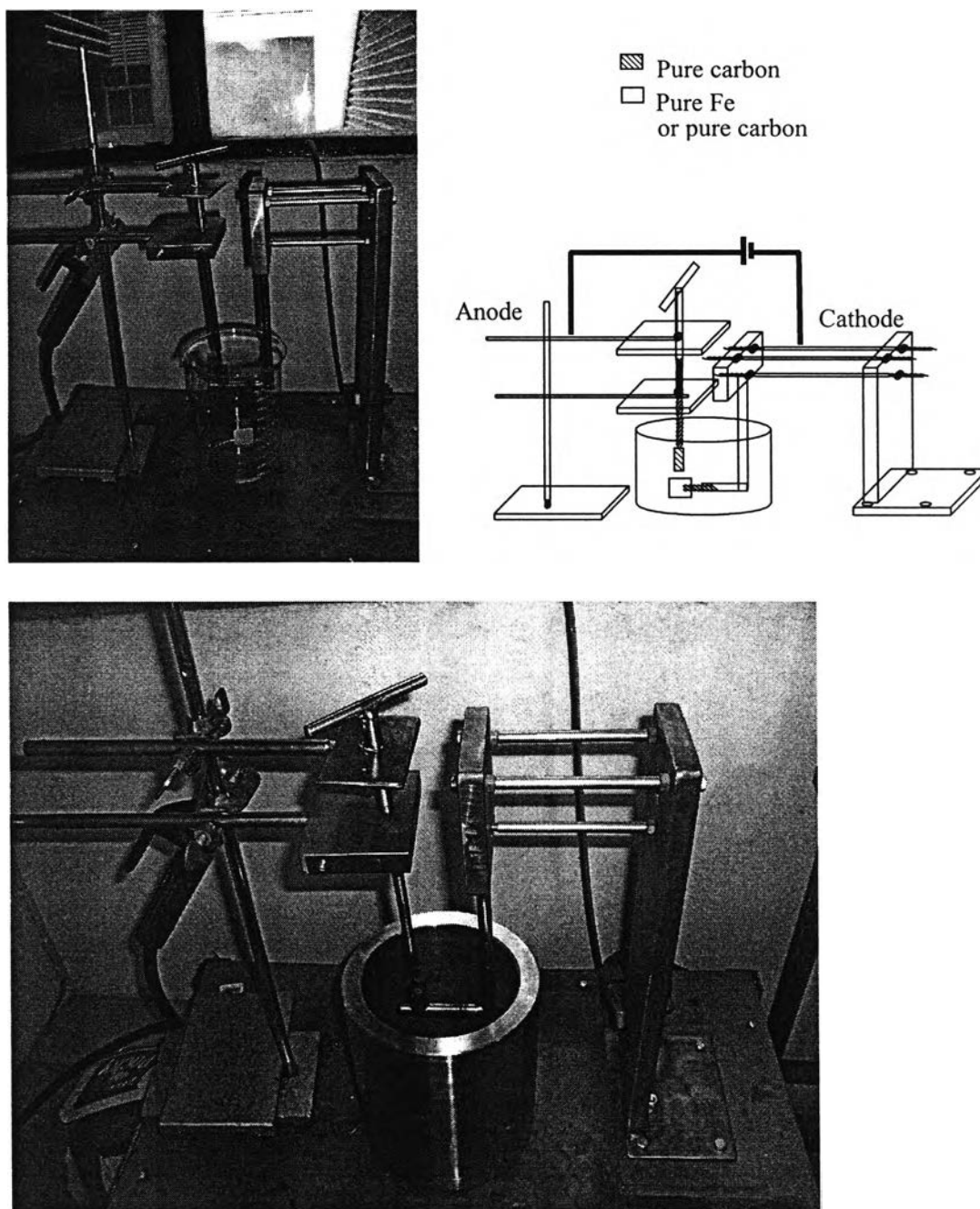
Figure 3.2 Electrodes arrangement for arc discharge in liquid nitrogen

3.3 Experiment method of mixed Polymer nanocomposite

3.3.1 Materials and instruments

3.3.1.1 Carbon nanoparticles (These experimental are 3.2).

3.3.1.2 Hot plate, spoons and Teflon

3.3.1.3 i-Polypropylene powder commercial grade (Basell, HMC Polymer company limited with a HP648N grade)

3.3.2 Melt mixing by direct melting

This operation is prepared by mixing concentration CNPs (5, 10, and 15% of iso-polypropylene (i-pp) by weight) and i-pp (1.0 g). Firstly, i-pp is melted about 30 minutes by mixing in Teflon (6in x 6in) on hot plate ($200^{\circ}C$) at room temperature. Moreover, various concentration CNPs (5, 10, and 15% of i-pp by weight) is melted mixing into melting i-pp and then mixed two samples 40 minutes. Finally, these products (i-pp/CNPs) are leaved for 15 minutes at room temperature, and kept in sample bags for analysis later.

3.4 Preparation the samples for analysis

3.4.1 Transmission Electron Microscopy (TEM)

TEM specimens were elaborately prepared by gently grinding as-grown fine particles by a mortar, subjecting to high power ultrasonic treatment (150 W, 15Hz) in toluene 99.5% with sufficient time (3-5 min.) for ensuring its uniform dispersion and then transferred to a high-grade Cu grid coated with a porous carbon film. These specimens were loaded into sample chamber, waiting for 30 min. until it became steady state inside the chamber. After using TEM, the films were developed by using

three chemical liquids in the dark room. Transmission Electron Microscopy (TEM)

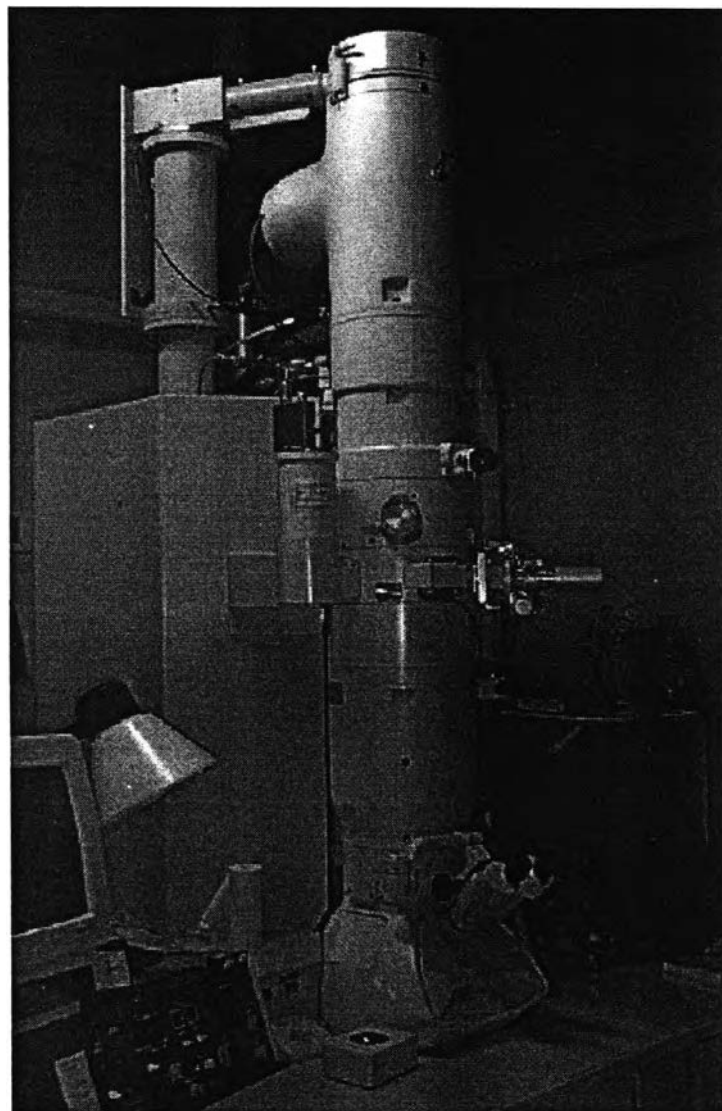machine is shown in Figure 3.3

Figure 3.3 Transmission Electron Microscopy (TEM) (JEOL 2010)

3.4.2 Field Emission Electron Microscopy (FSEM)

FESEM specimens were prepared by grinding as-grown fine particles by the

mortar similar to TEM specimens, and then directly sprinkling the appropriate

amounts onto a conductive carbon coated microscope grid. The specimens were

loaded into sample chamber, and then immediately starting the observation with using

image catcher scanner for taking the photos. Field Emission Electron Microscopy (FSEM) machine is shown in Figure 3.4
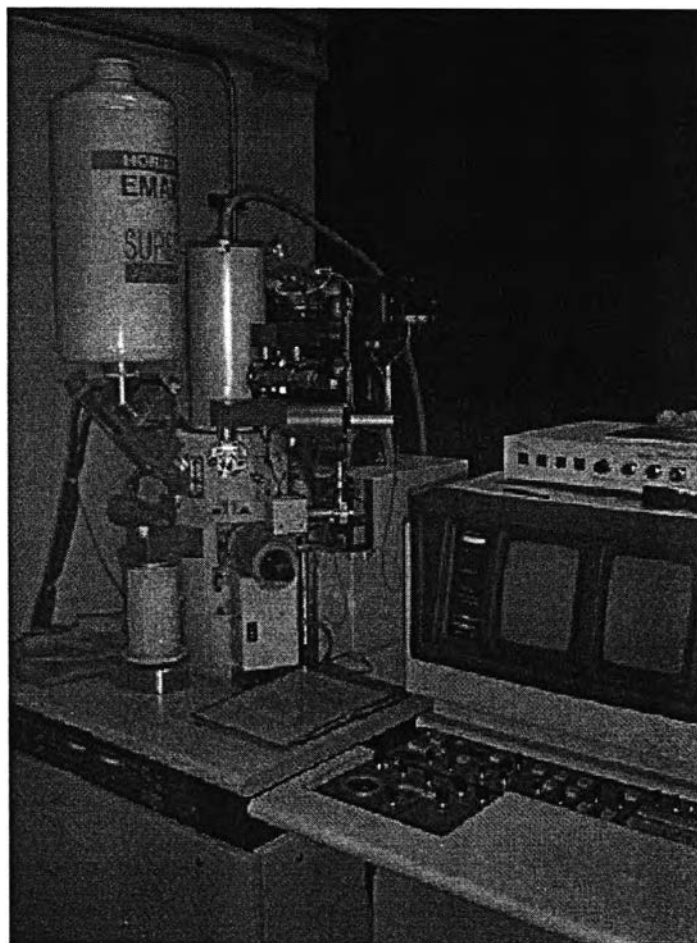

Figure 3.4 Field Emission Scanning Electron Microscopy (FESEM) (HITACHI, S-900)

3.4.3 Dynamic Light Scattering (DLS)

DLS was used to analyze the particle size distribution of the obtained products. It should be noted that DLS is based on the measurement of the dispersion of light scattering by particles motion in a static solvent such as toluene, acetone or ethanol, the measured particle size should correspond to hydrodynamic diameter but not to the real diameters of the particles with complex structures. However, DLS results were

expected to give at least the qualitative trend in particle sizes. Before the analysis,

DLS specimens were also prepared by grinding as-grown particles by the mortar, and

then subjecting to high power ultrasonic treatment (150 W) in toluene for 1 min. Next,

the specimens were diluted by toluene until them became transparent, subjecting to

ultrasonic disperser again for 1 min. and then loading to the sample cell for analysis.

The Dynamic Light Scattering (DLS) machine is shown in Figure 3.5

Figure 3.5 Dynamic Light Scattering (DLS) (MALVERN ZETASIZER300HSA)

3.4.4 Raman Spectroscopy

As-grown substrate of solid deposit was filled into the hole of specimen holder

and then was bombarded by an Ar ion laser (514 nm), number scans: 64, resolution

16.0 cm^{-1}, and laser power 1000mW at the room temperature. In the cases of carbon

nanoparticles (CNPs), two peaks can be found in the wavenumber between 1200 and

1900 cm^{-1}. One is a strong peak at 1590 cm^{-1} (Graphite band, G band) arises from an

in-plane oscillation of carbon atoms in the sp^2 graphene sheet. Another is a weak peak

at 1340 cm^{-1} (Disordered band, D band) reflects the degree of defects or dangling bonds contained in the sp^2 arrangement of graphene sheet. The Raman spectroscopy machine is shown in Figure 3.6

Figure3.6 Raman Spectroscopy (Perkin Elmer, Spectrum GX NIR FTRaman)

3.3.5 Differential Scanning Calorimetry (DSC)

DSC analyses were carried out in a thermal analyzer under flowing nitrogen atmosphere. The samples were initially heated from 90 to 200°C at 20°Cmin^{-1}, held at that temperature for 1min to eliminate thermal history effects, and then cooled to 50°C at 20 °C min^{-1}. They were kept there for 1min, heated again to 200°C at 20°C min^{-1}, and cooled to 50°C at the same rate. Onset and peak temperatures of melting and crystallization, as well as melting and crystallization enthalpies, were determined from the second scan, where ΔHm is the melting enthalpy of the samples calculated from the main melting peak. The Differential Scanning Calorimetry (DSC) machine is shown in Figure 3.7
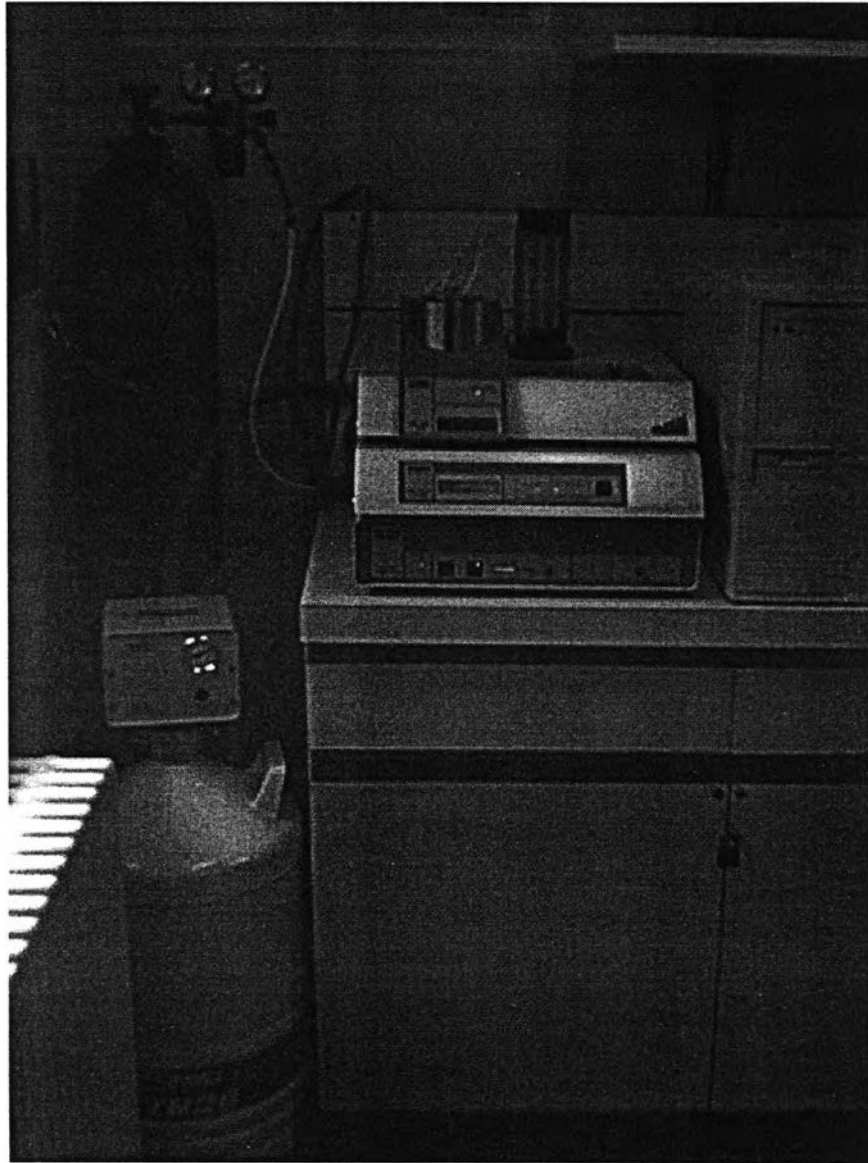
Figure 3.7 The Differential Scanning Calorimetry (Perkin Elmer, Diamond DSC

Differential Scanning Calorimeter)

3.5 Characterization

3.5.1 Carbon nanoparticles produce

The synthesized carbon nanoparticles are characterized using several analytical techniques which are TEM (Transmission Electron Microscopy,

JEOL2010), DLS (Dynamic Light Scattering, MALVERN, ZETASIZER 300HSA), and Raman Analysis (Perkin Elmer, Spectrum GX NIR FTRaman).

3.5.2 Polymer nanocomposite

The synthesized polymer nanocomposites are characterized using several analytical techniques which are Differential Scanning Calorimetry (Perkin Elmer, Diamond DSC Differential Scanning Calorimeter).