# CHAPTER 3

## Experimental procedures

This chapter aims to describe the preparation and techniques of the various experiments. Because of the various combinations, the contents are separated into several topics as follows:

### 3.1 MWNTs preparation

#### 3.1.1 Synthesis and preparation of MWNTs

MWNTs were synthesized at the Nanomaterials Science Research Unit, Faculty of Science, Chiangmai Univesity, Thailand, using the CVD method [1]. The length of the MWNTs produced there was more than 10 μm, and the average diameter was approximately 40 nm.

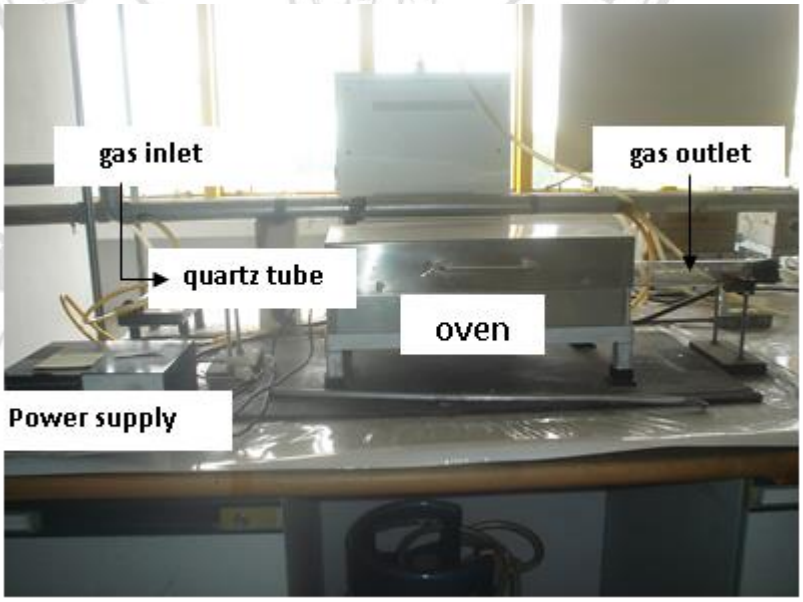



**Figure 3.1** Photograph of CVD apparatus

### 3.2 Preparation of copper and aluminium surfaces for adhesive bonding

#### 3.2.1 Preparation of copper surfaces

**1. Nitric acid/Ferric chloride**

- degrease the surfaces
- immerse for 1 to 2 minutes at room temperature in the following solution by weight: 197 parts water, 30 parts nitric acid (sp gr 1.42), to 15 parts ferric chloride solution (42%).
- rinse thoroughly
- dry as quickly as possible
- apply adhesive immediately

**2. Nitric acid**

- Bright dip in concentrated nitric acid at 16 to 21°C for 15 s or until all corrosion has disappeared.
- Rinse thoroughly
- Dry as fast as possible
- Apply adhesive immediately

**3. Acid etch (Sulfuric acid-Dichromate-Ferric sulfate)**

- Remove surface contamination by sanding, wire brushing, or sandblasting, if necessary.
- Degrease
- Immerse for 10 min at 66 °C in the following solution by weight: 8 parts water, 1 part ferric sulfate (commercial), to 75 parts sulfuric acid (sp gr 1.84).
- Rinse (with water at or below room temperature)

- Dry
- Immerse the parts until a bright clean surface has been obtained in the following solution by weight at room temperature: 17 parts water, 2 parts sulfuric acid (sp gr 1.84), to 1 part sodium dichromate.
- Rinse using cold tap water.
- Dip in concentrated ammonium hydroxide.
- Rinse in cold tap water.
- Dry quickly and apply adhesive immediately

### 3.2.2 Preparation of aluminium surfaces

Procedure

- Degrease
- Dip in chromic acid at 65-70 °C for 5-10 minutes
- Rinse using cold tap water and dry
- Apply adhesive immediately

Preparation of chromic acid

- Sodium dichromate 10 wt.%
- Sulfuric acid 96% 30 wt.%
- DI water 100 wt.%

### 3.3 Preparation MWNTs/LLDPE sheet and MWNTs/PVB sheet before coated on metal sheet

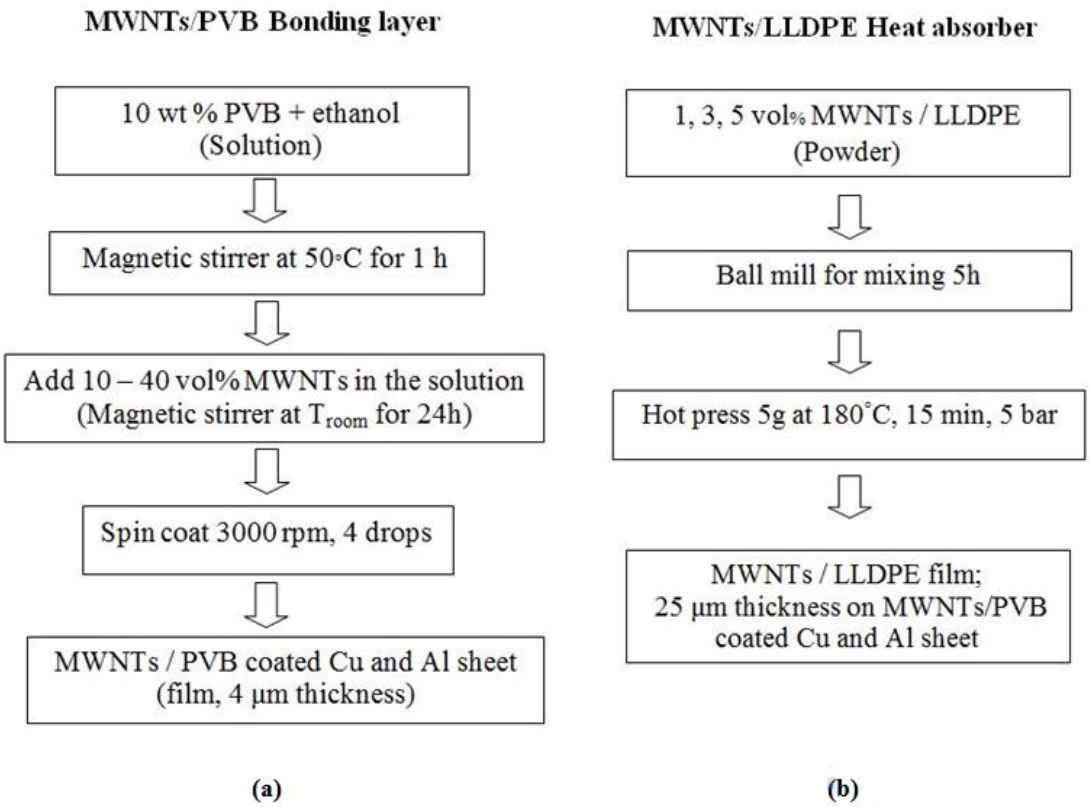
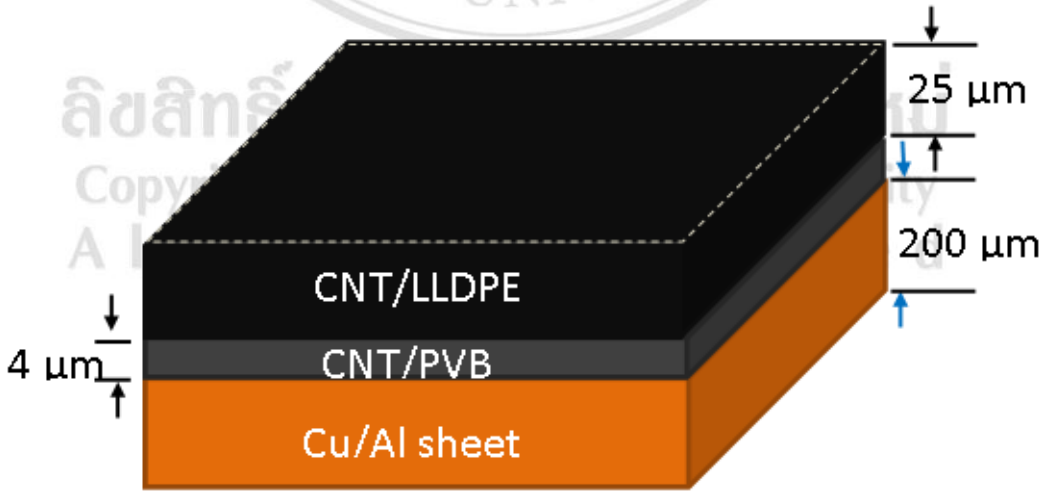



**Figure 3.2** Flow charts showing the process of (a) MWNTs/PVB coated on a Cu and Al sheet and (b) MWNTs/LLDPE film on MWNTs/PVB coated on a Cu and Al sheet.




**Scheme 3.1** Diagram for preparation MWNTs/polymer composites coated on copper and aluminium sheet

## 3.4 Sample characterization

The sample was characterized by using these microscopes as follows:

### 3.4.1 Scanning electron microscopy (SEM)

FE-SEM (FE-Scanning Electron Microscope: JSM 6335 F) was used to study MWNT agglomerates after milling and the distribution of MWNTs in the LLDPE matrix. In preparation for the cross-sectional images of the LLDPE/MWNT samples, the single- and four-step heating samples were frozen in liquid nitrogen before they were broken with pliers. Gold sputtering was performed on the cross-sectional surface before the FE-SEM images were taken.

### 3.4.2 Density test

The density of the LLDPE/MWNT composites both those prepared using step heating, and those prepared without step heating was measured by the Archimedes test compared with theory [2,3].

### 3.4.3 X-ray diffraction (XRD)

XRD (Rigaku- MiniFlex II, Desktop X-ray Diffractometer) was used to measure the X-ray diffraction patterns. The degree of crystallinity ($X_C$) was determined using the method of Hermans and Weidinger [4-6].

$$X_C = A_C/(A_C+A_a) \quad (3.1)$$

where $A_C$ and $A_a$ are the area of the crystalline and amorphous regions, respectively.


**Figure 3.3** X-ray Diffractometer (XRD)

## 3.5 Mechanical properties test

### 3.5.1 Tensile tests

An ultimate tensile testing machine (Hounsfield, Model H10KS) was used to study the mechanical properties of the LLDPE/MWNT composites, following the American standard test materials ASTM D638-10 and strain rate of 50 mm/min.

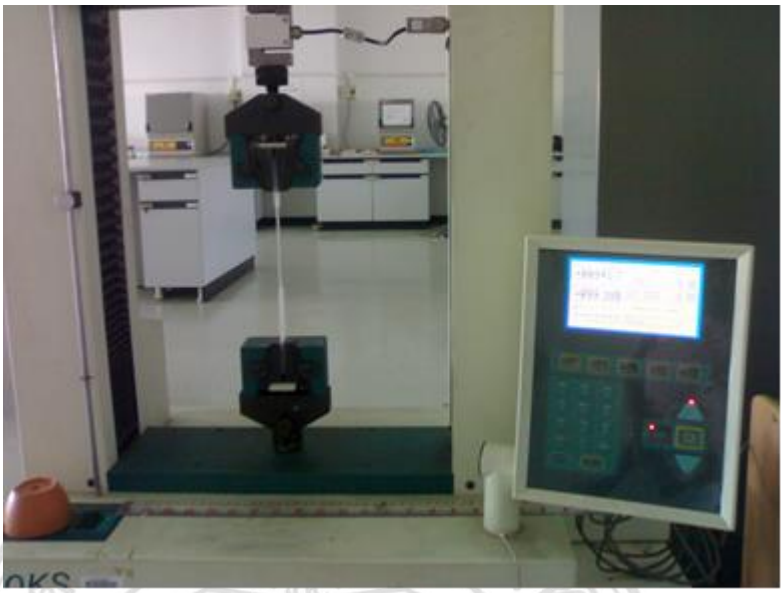


**Figure 3.4** tensile testing machine

### 3.5.2 Impact strength tests

An impact tester (charpy) machine (Toolquip, Leicester LE67 5FT: England) was used to determine the impact strength of the LLDPE/MWNT composites prepared using single- and four-step heating, following ASTM D6110-10 standard.

**Figure 3.5** An impact tester (charpy) machine

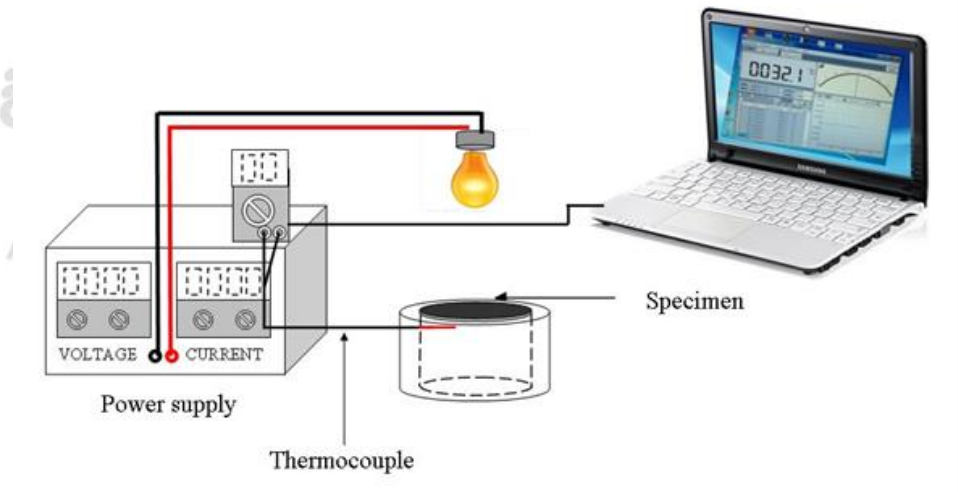
## 3.6 Thermal properties test

### 3.6.1 The reflectance of the coating

The reflectance of the coatings was measured by ultraviolet visible spectroscopy (VARIAN Cary 50 Conc) within a wavelength range of 190–1100 nm.

**Figure 3.6** An ultraviolet visible spectroscopy (VARIAN Cary 50 Conc)

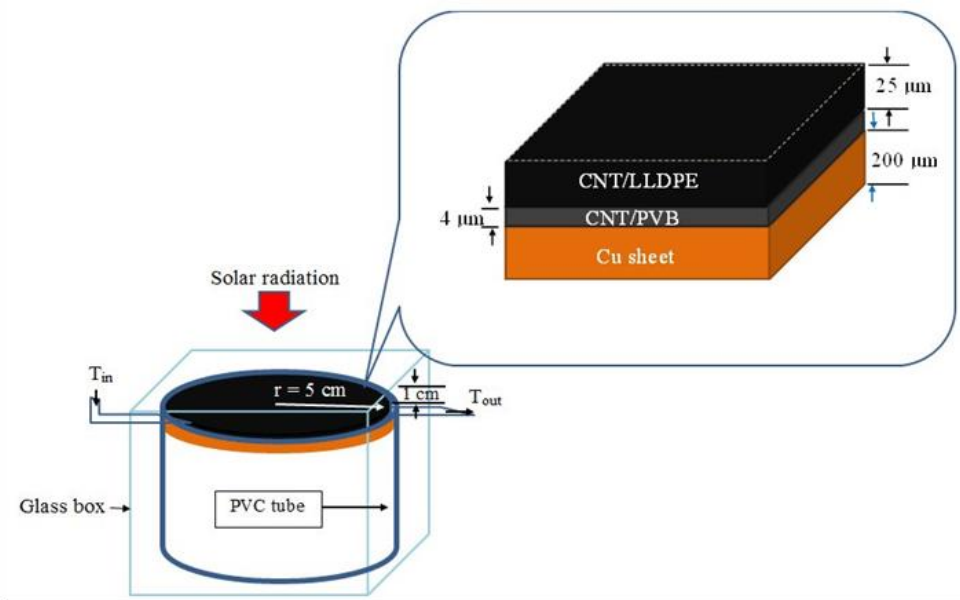
### 3.6.2 Heat absorbing test




**Scheme 3.2** Diagram of heat absorbing test

MWNTs/LLDPE composite coated on copper sheet by using MWNTs/PVB composite for the bonding layer was tested using the heat absorbing test shown in Scheme 3.2. The specimen for testing heat absorption used the MWNTs/PVB sheet at the same thickness (~4 μm) for 40 vol.% of MWNTs/PVB composite and compared the different vol.% of MWNTs (10 and 40 vol.% MWNTs/PVB) and used the distance between the specimen and light source at 10 cm.

### 3.6.3 The energy conversion efficiency was measured using a home-made method




**Scheme 3.3** Diagram of the home-made test system

In Sheme 3.3, heat absorbed by water from solar radiation was determined by using a home-made method. This consisted of the solar radiation from the sun (in a clear-sky day from 11:00 to 13:00 o'clock in April at Chiang Mai, Thailand) and a glass box 12×12×6 $cm^3$ in size. When converting the value of water heat absorbed for the energy per area of the sample, the following equation was used, $Q = mc(T_{out}–T_{in})$ where Q is the solar energy collected, c is the specific heat of the water, m is the mass of water flow rate in (kg/s).

## 3.7 References

[1] Singjai, P., Changsarn, S., Thongtem., "Electrical resistivity of bulk multi-walled carbon nanotubes synthesized by an infusion chemical vapor deposition method," Materials Science and Engineering Part A, Vol. 443, No. 1, January 2007 p.p. 42-46.

[2] Xie, X.L., Mai, Y.W., and Zhou, X.P., "Dispersion and alignment of carbon nanotubes in polymer matrix: A review," Materials Science and Engineering, Vol. 49, No. 4, May 2005, 89–112.

[3] Chu, K., Guo, H., Jia, C., et al.,. "Thermal Properties of Carbon Nanotube–Copper Composites for Thermal Management Applications," Nanoscale Research Letters, Vol. 5, No. 5, March 2010, pp. 868–874.

[4] Kundu, P.P., Biswas, J., Kim, H., et al., "Influence of film preparation procedures on the crystallinity, morphology and mechanical properties of LLDPE films," European Polymer Journal, Vol. 39, No. 8, August 2003, pp.1585–1593.

[5] Ning, Y., "Determination of the crystallinity in different type poly (oxyethylene-styrene) block copolymers by X-ray diffraction method," Chinese Journal Polymer Science, Vol. 7, No. 4, April 1989, pp. 315–321.

[6] Kuila, T., Bose, S., Mishra, A.K., et al., "Effect of functionalized graphene on the physical properties of linear low density polyethylene nanocomposites," Polymer Testing, Vol. 31, No. 1, Febuary 2012, pp. 31-38.