CHAPTER III

EXPERIMENTAL

In this chapter, the materials and chemicals, microwave plasma system, substrate preparation and synthesis of DLC thin films procedure will be explained. The most widely characterization techniques of DLC thin films, Scanning Electron Microscope (SEM), Raman Spectroscope, Atomic Force Microscope (AFM), and nanoindentation tester which have been used in this work will be presented.

3.1 Materials and Chemicals

The chemicals used in these experiments, carried out at the Plasma Laboratory were specified as follows:

1. Methanol (Commercial grade) was purchased from CTL.
2. Acetone (Commercial grade) was purchased from ZEN POINT.
3. De-ionized water was donated from Faculty of Science, Chulalongkorn University.
4. Sodium Hydroxide was purchased from SIGMA-ALDRICH.
5. Diamond powder (virgin diamond powder 0.1 μm type MB-1-um form EID).
6. Ultra high purity hydrogen gas was purchased from Thai Industrial Gas Co., Ltd. (TIG).
7. Methane gas was purchased from Thai Industrial Gas Co., Ltd. (TIG).

3.2 Microwave plasma system

The microwave plasma reactor was designed to be assembled as an economical home-made prototype by Rujisamphan [50], the photograph and schematic of the system are shown in Fig. 3.1 and 3.2, respectively. The system consists of vacuum chamber, microwave guiding components, and gas flowing system.

3.2.1 Vacuum chamber

The vacuum chamber was made from stainless steel. The chamber was equipped with many plates; the front plate was used for sample loading while the bottom plate was connected with three ports, leading to an Edwards Speedivalve, an air leak valve, and a substrate holder. The substrate temperature was measured by a thermocouple embedded within the substrate holder. The left plate was connected with the Edwards Penning and the Edwards Pirani vacuum gauge. The chamber was evacuated using a rotary vane pump Edwards model RV3 and the turbo molecular pump Edwards model ACX75. The top plate was connected with a doughnut-plate in which was designed for water cooling and gas inlet line.

3.2.2 Microwave guiding components

Microwave guiding components composed of cylindrical cavity, waveguide, and magnetron power supply. Traditional microwave plasma research, mostly utilized at 2.45 GHz, in which its frequency is the same as that used in conventional microwave oven. The microwave plasma system used the magnetron source that generates a microwave for forming plasma from the conventional microwave oven powered by a phase controlling power supply. A waveguide unit that guided the microwave was generated by the magnetron source toward the cylindrical cavity via an axial antenna. An antenna made of a conductive material provided a lot of microwave radiation for radiating the microwave was guided by the microwave guide unit toward the chamber. A discharge called a plasma ball was generated above the substrate.

3.2.3 Gas flowing system

The gas flowing system consists of methane (CH_4) and hydrogen (H_2) tanks, regulators, and mass flow controllers (Aalborg model GFC-17 and Dwyer model GFC-2102). These gaseous were regulated by mass flow controllers with an accurate the gas flow rate. Each of gaseous flew through a manual on-off valve and mixed in the gas inlet line. The unit of gas flow rate is the standard centimeters cubed per minute (sccm).

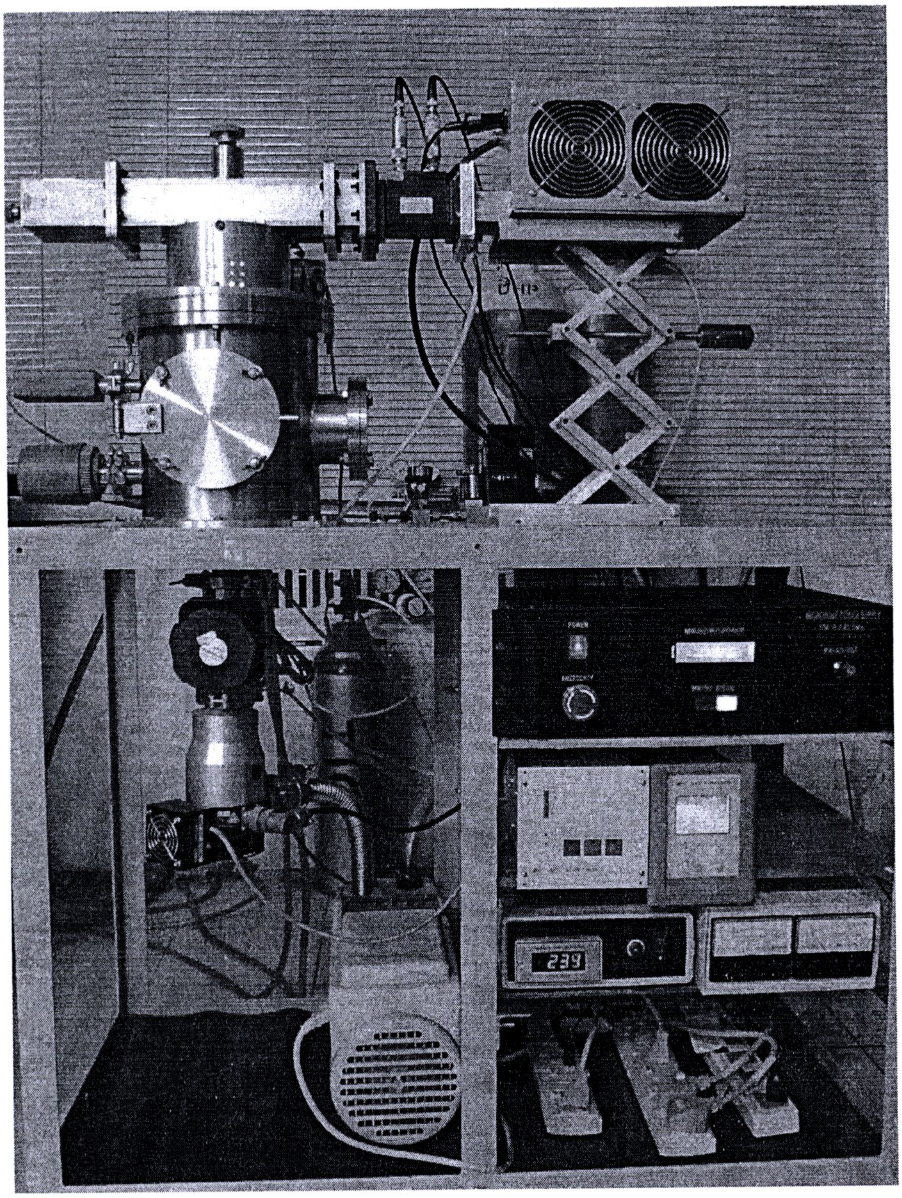
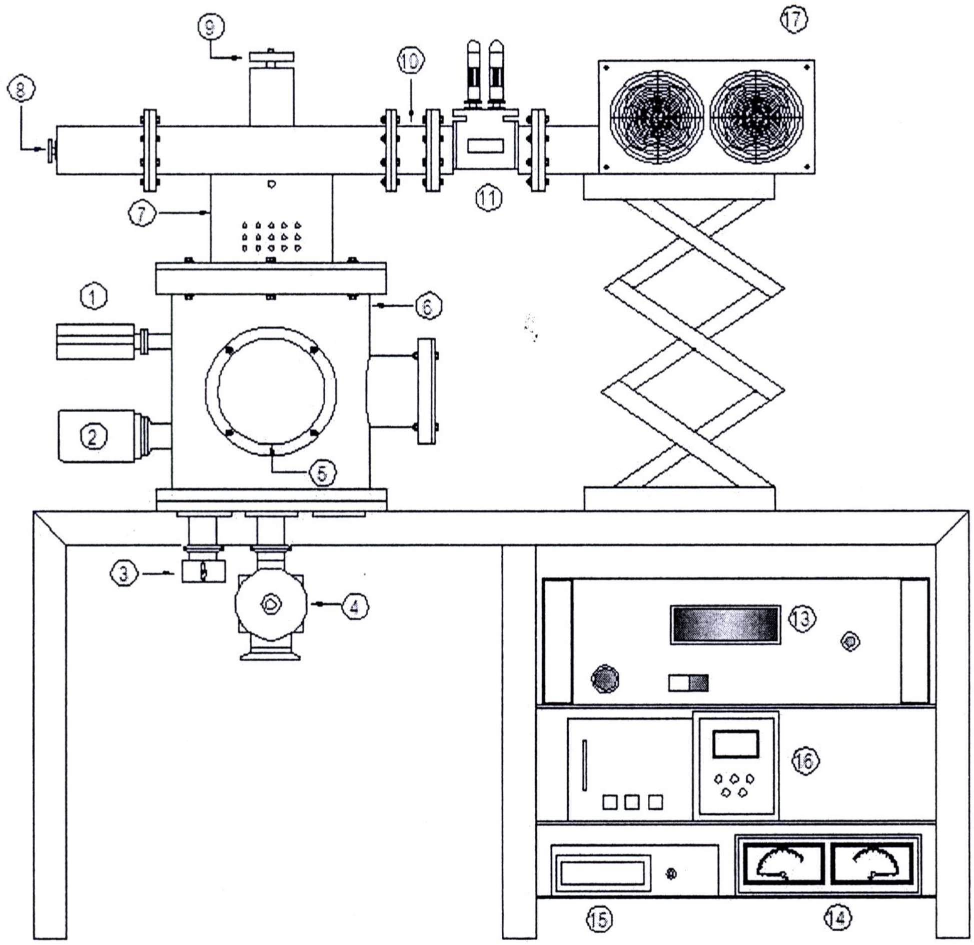


Figure 3.1 The photograph of MW-PECVD reactor.



[1] Penning Gauge
[2] Pirani Gauge
[3] Air-leak valve
[4] Speedivalve
[5] Loading door
[6] Chamber
[7] Cylindrical cavity
[8] Short-circuit plunger
[9] Brass antenna
[10] Waveguide
[11] Dual Power Monitor
[12] Turbo controller
[13] Power supply
[14] Microwave power meter
[15] Temperature indicator
[16] Gauge monitor
[17] Magnetron source

Figure 3.2 The schematic diagram of MW-PECVD reactor.

3.3 Substrate preparation

In this research, prior to the deposition of diamond films, the alumina substrates were pretreated by cleaning with detergent, scratching with diamond powder (0.1 μm) by hand for 30 min, cleaning by ultrasonic for 30 min in acetone, methanol, and de-ionized water respectively. Finally, they were blown dried with nitrogen gas.

3.4 Synthesis of DLC thin films procedure

The substrates were loaded in the reactor chamber pumped by a rotary pump. This pump evacuated air in the chamber until the total gas pressure reached at about 2-2.6 x 10^{-2} torr. After the based pressure, a turbo pump evacuated air until the pressure between 1.2-1.5 x 10^{-5} torr. Then, the gaseous mixture of methane and hydrogen were fed into the chamber and deposition pressure was adjusted by the controlling valve of pump. Finally, the MW-PECVD technique was applied to deposit DLC thin films on alumina substrates. In this research, the methane (CH_4) concentration, deposition pressure and deposition time were investigated. After the growth process completed, all substrates were cooled down to room temperature within the reactor chamber under evacuating conditions. The range of growth conditions using in this study are shown below in Table 3.1.

Table 3.1 Deposition conditions for the DLC thin films.

Parameters	Range
Gas composition (%)	
$CH_4 : H_2$	0.5-5
Gas flow rate (sccm)	
H_2	100
CH_4	0.5-5
Deposition pressure (Torr)	10-50
Microwave power (W)	700
Substrate temperature (°C)	300-350
Deposition time (hr)	5-30

3.5 Characterization of DLC thin films

The obtained films were analyzed by the following methods:

3.5.1 Scanning electron microscopy (SEM)

SEM observation with a JEOL mode JSM-6480LV, at Faculty of Science, Chulalongkorn University was employed to investigate the morphology of DLC films as shown in Fig. 3.3. The samples for SEM analysis were coated with gold particles by ion sputtering device to provide electrical contact to the specimens.

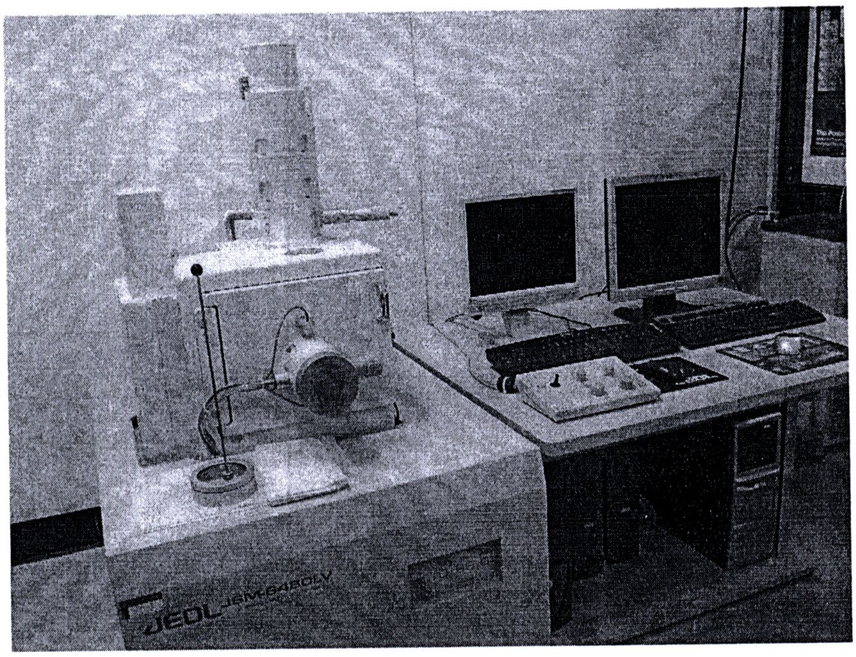


Figure 3.3 Photograph of Scanning electron microscope (SEM).

3.5.2 Raman spectroscopy

Raman spectrums were measured by Renishaw invia raman microscope (Fig. 3.4) using 514.5 nm line of an argon ion laser, at The Gem and Jewelry Institute of Thailand (Public Organization) (GIT), Bangkok.

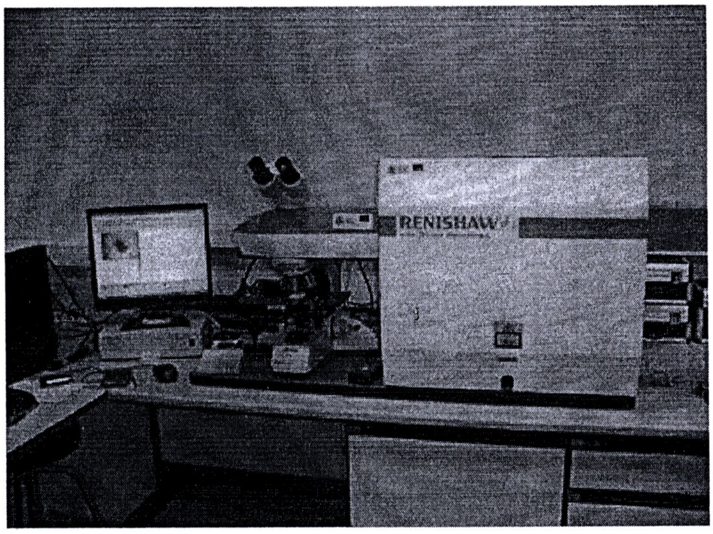


Figure 3.4 Photograph of Renishaw invia raman microscope.

3.5.3 Nanoindentation test

The hardness of CVD diamond films were determined by nanoindentaion using a CSM nano hardness tester (NHT), at Nano Shield Company Limited, Samutprakarn as shown in Fig. 3.5. Hardness of the films was calculated from the load-displacement curves. These curves were obtained with a Berkovich diamond indenter and a loading and unloading rate of 10 mN min^{-1} up to maximum load of 5 mN. For each samples, a number of indentations have been performed at different position.

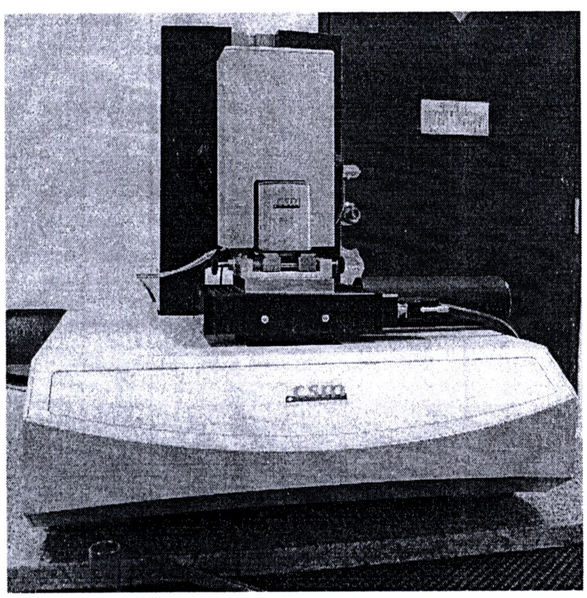


Figure 3.5 Photograph of CSM nano hardness testers (NHT).

3.5.4 Atomic force microscopy (AFM)

Atomic Force Microscope (AFM) shown in Fig. 3.6 was used to examine the surface roughness of the diamond film using Nano Scope IV with tapping mode, at Scientific Technological Research Equipment Center, Chulalongkorn University.

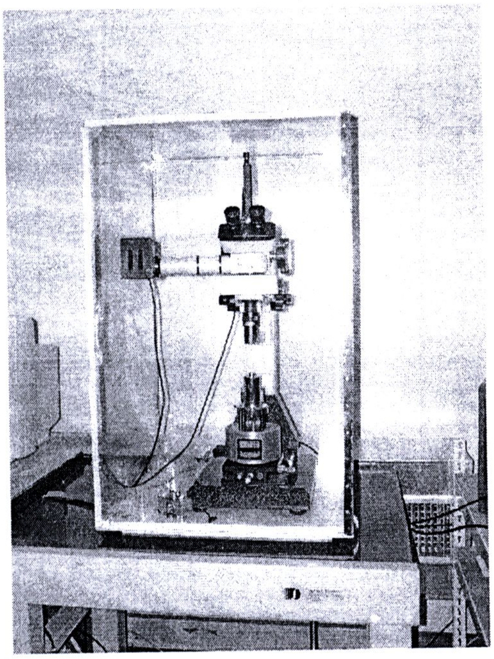


Figure 3.6 Photograph of Atomic Force Microscope (AFM).