Chapter 2

Experimental Techniques

In this chapter, we will describe the experimental techniques to grow and characterize the GaN epilayer on sapphire (0 0 0 1) and silicon (1 1 1) substrates. They include growth method: molecular beam epitaxy (MBE); material characteristic analysis method: High Resolution X-ray Diffraction (HRXRD), Field Emission Scanning Electron Microscopy (FESEM), Photoluminescence (PL) and Atomic Force Microscopy (AFM).

2.1 Molecular beam epitaxy (MBE) system

The ULVAC molecular beam epitaxy (MBE) is newly installed and only for growing group III nitride and their alloys. This MBE is equipped with in-situ monitoring reflective high energy electron diffraction (RHEED) and residual gas analyzer (RGA). Fig 2-1 shows the system description of this RF-MBE. The ULVAC MBE system can be divided into three functional groups:

1. Vacuum equipment: Chambers, Pumps, Valves, Vacuum gauges
2. Epitaxy equipment: Sample Manipulators, Kundsen cells, Cracking cell, RF plasma
3. Analytical instruments: RHEED, beam flux monitor, RGA

There are two chambers in ULVAC MBE system: loading chamber and growth chamber. The oil-free rotary pump and turbo pump are used for loading chamber and pre-pumping to 2×10^{-6} pa. The growth chamber is pumped by oil-free rotary pump, turbo pump and ion pump. In the baking procedure, the titanium sublimation pump is used to absorb the inert gas, steam and hydrogen. After 48hrs baking procedure, the growth chamber is pumped to 1.5×10^{-8} pa for group III nitride growth.

Six effusion cells are installed. Solid metal of gallium, indium, aluminum are used as group III sources. Nitrogen source is generated by two RF plasma generators or catalytic. Purity of 6N nitrogen and ammonia are connected through mass flow control (MFC) to RF plasma and catalytic, respectively. In this thesis, nitrogen source is supplied by RF plasma. With fine tuning of the thermal controller parameters, the temperature reading could be controlled in ±0.1℃.

The RF plasma consists of four major parts: ULVAC RF Plasma source, RF matching unit, RF generator and water cooling flow control assembly. The maximum RF power can be operated at 500 watts. The normal operation RF power used in this thesis is 300 and 500 watts.

The ULVAC RHEED system is equipped for in-situ monitor. This RHEED has advantages such as small focus spot (<100μm) could be obtained, beam size remains small at large working distances and small beam divergence could be reached. The normal operated filament current is set as 1.7A and the beam energy is set as 20kV.

2.2 Material Characteristic Analysis

2.2.2 High Resolution X-ray Diffraction (HRXRD)

Crystal structure was examined by the HRXRD analysis using the Bede D1 system. The x-ray source is copper (kα) line wavelength λ= 0.154 nm.

X-ray diffraction is a non-destructive method to investigate the crystal structure information, such as lattice constant, layer composition, strain, film thickness, etc. X-ray has wavelength comparable to the lattice constant in the crystal. Thus, X-ray scattered by the atoms in the crystal will be diffracted. As the diffraction condition is under the Bragg condition, the diffraction pattern will be very intensive because the Bragg condition is

$$n\lambda = 2d_{hkl}\sin\theta_B \qquad \text{(Eq. 2-1)}$$

where λ is the wavelength of X-ray, n is an arbitrary integer, θ_B is the diffraction Bragg angle, and d_{hkl} is the spacing of the adjacent parallel planes of atoms with Miller indices h, k, l. The relation between d_{hkl} and the lattice constant, a, is

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad \text{(Eq. 2-2)}$$

The rocking curve is to scan the diffraction beam intensity as a function of the diffraction angle of the sample with a fixed incident X-ray beam. In order to obtain high angular resolution for determining the lattice constant, two silicon crystals, channel-cut crystals (CCC), are positioned in front of the X-ray source to obtain a monochromated X-ray beam with only 12” divergence. We usually make the symmetric (0 0 0 2) and asymmetry $(1\ 0\ \bar{1}\ 2)$ rocking curve measurements to examine the crystal quality of GaN epilayer. The result of symmetric (0 0 0 2) reflections showed the density of screw and/or mixed threading dislocation. Broadening of the asymmetric $(1\ 0\ \bar{1}\ 2)$ reflections compared to symmetric reflections is indicative of a defect structure with a large pure edge threading dislocation due to the symmetric reflections wouldn’t show the information of edge dislocation.

2.2.3 Field emission scanning electron microscopy (FESEM)

Scanning electron microscopy (SEM) is one of the well-known and most widely used surface analytical techniques. Field emission scanning electron microscopy (FESEM) equipped with a field-emission cathode in the electron gun of a scanning electron microscope provides narrower probing beams at low as well as high electron energy, resulting in both improved spatial resolution and minimized sample charging and damage. High-resolution images of surface topography, with excellent depth of field are produced using a highly focused scanning electron beam. The primary electrons enter a surface with the energy of 0.5 ~ 30 keV, and generate many low energy secondary electrons. The intensity of these secondary electrons is largely governed by the surface topography of the sample. An image of the sample surface can thus be constructed by measuring secondary electron intensity as a function of the position of the scanning primary electron beam. We used the FESEM, i.e. Hitachi S-4700I, to obtain the surface and sidewall morphology. From the plane view image, we know that the surface is smooth or rough and compared with the RHEED pattern. From the cross section image, we can see the general structure and the thickness of film.

2.2.1 Photoluminescence (PL)

Photoluminescence (PL) is the optical radiation emitted by semiconductor crystal after excitation with incident light source. Most of the light results from the difference in energy of the excited electron returning to its ground state. PL has the advantage of the ability to discriminate between species involved in radiative recombination and can provide simultaneous information on many type centers (defect levels). Low temperatures are necessary for two principal reasons. First, specific information about the centers, the donors and acceptors, which promote electrical conductivity, can be obtained only when the electrical carriers are frozen out in these centers. Once the electrical carriers are thermally liberated, the impurities or defects that released them reveal their presence only through some inhibition of carrier mobility. The second advantage of low temperature is the dramatic reduction of spectral broadening due to vibration processes.

The experimental was set-up for the photoluminescence measurement. The 325 nm line of a He-Cd laser was used as an excitation source for the PL spectroscopy, and the emission from the sample was analyzed by the SPEX 1403 double grating spectrometer equipped with a thermal electric-cooled photomultiplier tube. Samples were cooled in a closed-cycle refrigerator at 10 K.

2.2.5 Atomic Force Microscopy (AFM)

Surface morphology was examined by the AFM measurement and analyzed by the Digital Instrumen, Dimension 3100. The measurement was carried out using the tapping mode. The scan steps in x, y directions were both 9.7 nm. The resolution is 0.01 nm in the z direction. The shape of silicon tip is conic.

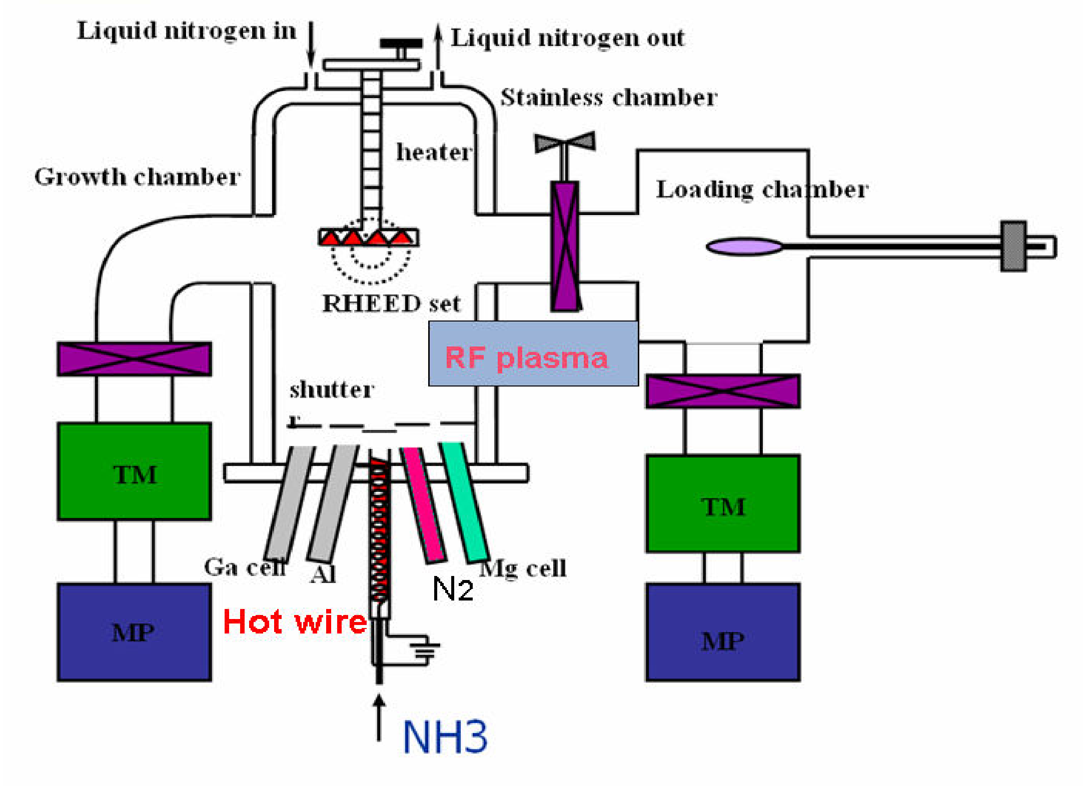

Fig 2-1 The system description of this ULVAC MBE.