

# Chapter 3 Experiments

## 3-1 Sample Preparation

The Si-doped GaN (GaN:Si) film samples were grown by the Low Pressure Metalorganic Chemical Vapor Deposition (MOCVD) system from “Chung Shan Institute of Science and Technology”. The GaN films were deposited on the (0001) sapphire, by using Trimethylgallium (TMGa) and Ammonia (NH<sub>3</sub>) as the Ga · N sources, and the high purity H<sub>2</sub> as the carrier gas. Prior to growth, a standard cleaning procedure was performed on the sapphire substrate. A 25nm GaN buffer layer was prepared on sapphire at 520°C before 2μm undoped GaN growth at 1120°C. 2μm Si-doped GaN films were then deposited on it by flowing SiH<sub>4</sub> at rates of 2, 8, and 16sccm, respectively, for samples A, B and C with V-defects on them. Carrier concentrations in samples A, B, and C are 2.5 · 6.7 · 11·10<sup>17</sup>cm<sup>-3</sup>, respectively, as deduced from 300K Hall measurements.

For comparison, 2μm undoped GaN (*u*-GaN) and Si-doped GaN (*n*-GaN) with SiH<sub>4</sub> flow rate at 2sccm were prepared after 25nm GaN buffer deposited on sapphire at 520°C, with concentrations 5·10<sup>16</sup>cm<sup>-3</sup> and 2.7·10<sup>17</sup>cm<sup>-3</sup>, respectively.

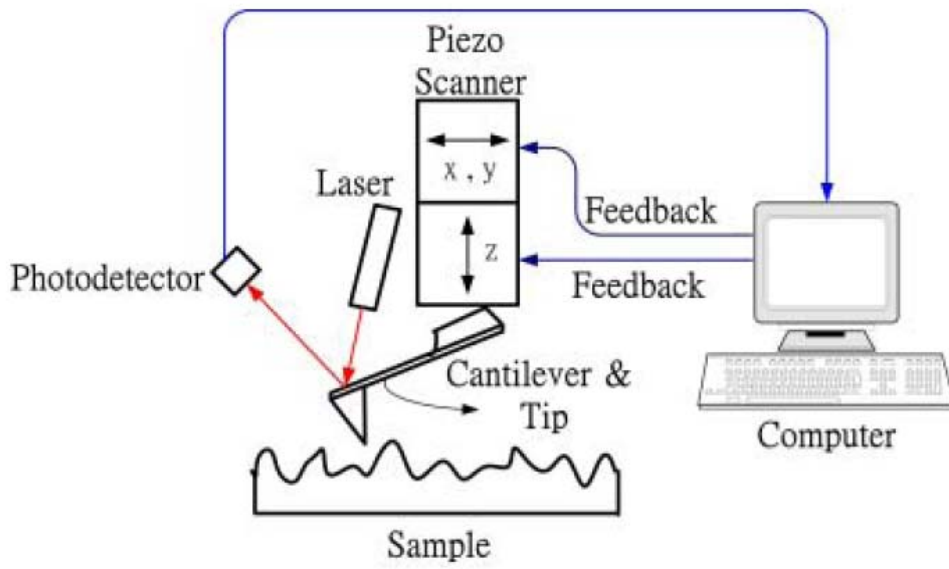
### 3-2 Atomic Force Microscopy (AFM)

Shown in Fig.3-2-1 is the basic operation principle of atomic force microscopy. Cantilever is biased with AC and DC voltages, that will vibrate at large amplitudes as AC signal was tuned to the resonance frequency. One may move the probe with sharp tip on the cantilever to the sample surface. The attractive and repulsive forces change the amplitude or displacement of the cantilever as the distance between the tip and the sample is reduced. Thus, one may aim the laser spot at the cantilever and detect the reflection from it. From the reflective signals, we can analyze the sample morphology. Three traditional operation modes are used to obtain the sample morphology. In contact mode, the tip touches the surface and scans over the sample. Although high resolution can be obtained in this way, the deformation of tips or samples often happens. Non-contact mode is preferred to avoid the probe deformation since it utilizes the long range Van der Waal's force between the tip and sample. However, the sensibility and resolution is limited because of the interference from ambient environment. Tapping mode is a combination of contact mode and non-contact mode. Detection of tapping mode is more sensitive than that of non-contact mode and less destructive on probes than that in contact mode.

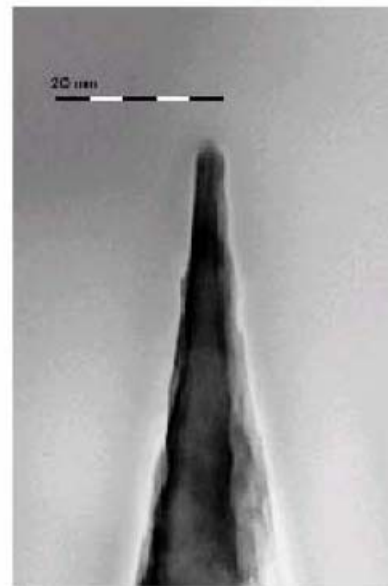
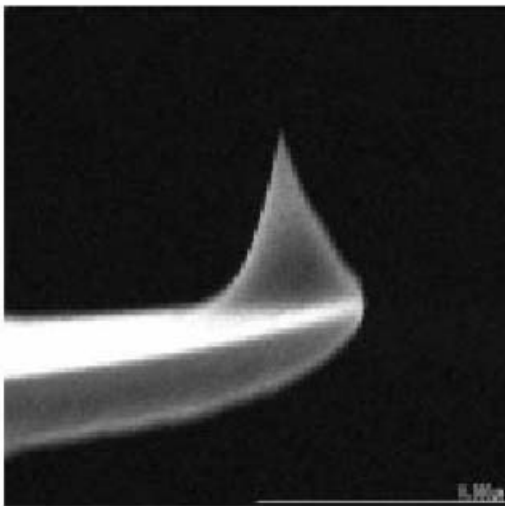
Scanning probe microscopy (SPM) system used in our study is Solver P47H, manufactured by the “Molecular Devices and Tools for Nano Technology” (NT-MDT) in Russia. It can be operated in multi-modes such as AFM for morphology measurements, EFM, MFM, and SKM, etc. In our study, the

V-defect's profile is obtained by tapping mode in order to optimize the resolution and avoid probe destruction. The AFM probes, which have a cantilever about 50 or 80  $\mu\text{m}$  and a sharp tip with a radius of curvature about 10 nm, were also from NT-MDT.





(a) The operation of the AFM system



(b) Cantilever and the tip

Fig.3-2-1 AFM system and Probe

### 3-3 Micro-Raman System

Fig.3-3-1 shows the block diagram of our micro-Raman system. It consists of an Olympus BX-40 confocal microscope, a double monochromator, and a thermoelectric cooled Princeton Instruments IRY-1024G diode array detector. Coherent Innova 70 Ar<sup>+</sup>/Kr<sup>+</sup> ion laser at 488nm was used as the pumping source. Laser plasma line was cut off by an inserted interference filter (at 488nm). The laser beam was guided into the microscope by four mirrors and a long-pass edge filter (at 488nm). The Olympus 100X objective lens (N.A.=0.9) focused the beam on the sample with a spot size about 2 $\mu$ m. Reflection and scattering signals from the sample were collected by the same objective lens. However, Rayleigh scattering and reflection were blocked by long-pass edge filter and only the Stoke-Raman signals passed and guided into the double monochromator by optical fiber. The double monochromator (Jobin-Yvon U-1000) served as the dispersion element, which consists of a cosecant-driver and two 10cm $\times$ 10cm holographic gratings of 1800 grooves/mm, and provided the spectral resolution about 0.5cm<sup>-1</sup> for 200 $\mu$ m slit. The dispersed signals were then directed to the multichannel detector that contains 1024 discrete pixels. The Raman spectrum is obtained by subtracting the “dark” from the “signal” spectra. The “dark” spectrum is measured by reducing the laser light and keeping the other experimental conditions the same as in taking the “signal” spectrum. Finally, the received Raman signals were processed by a computer.

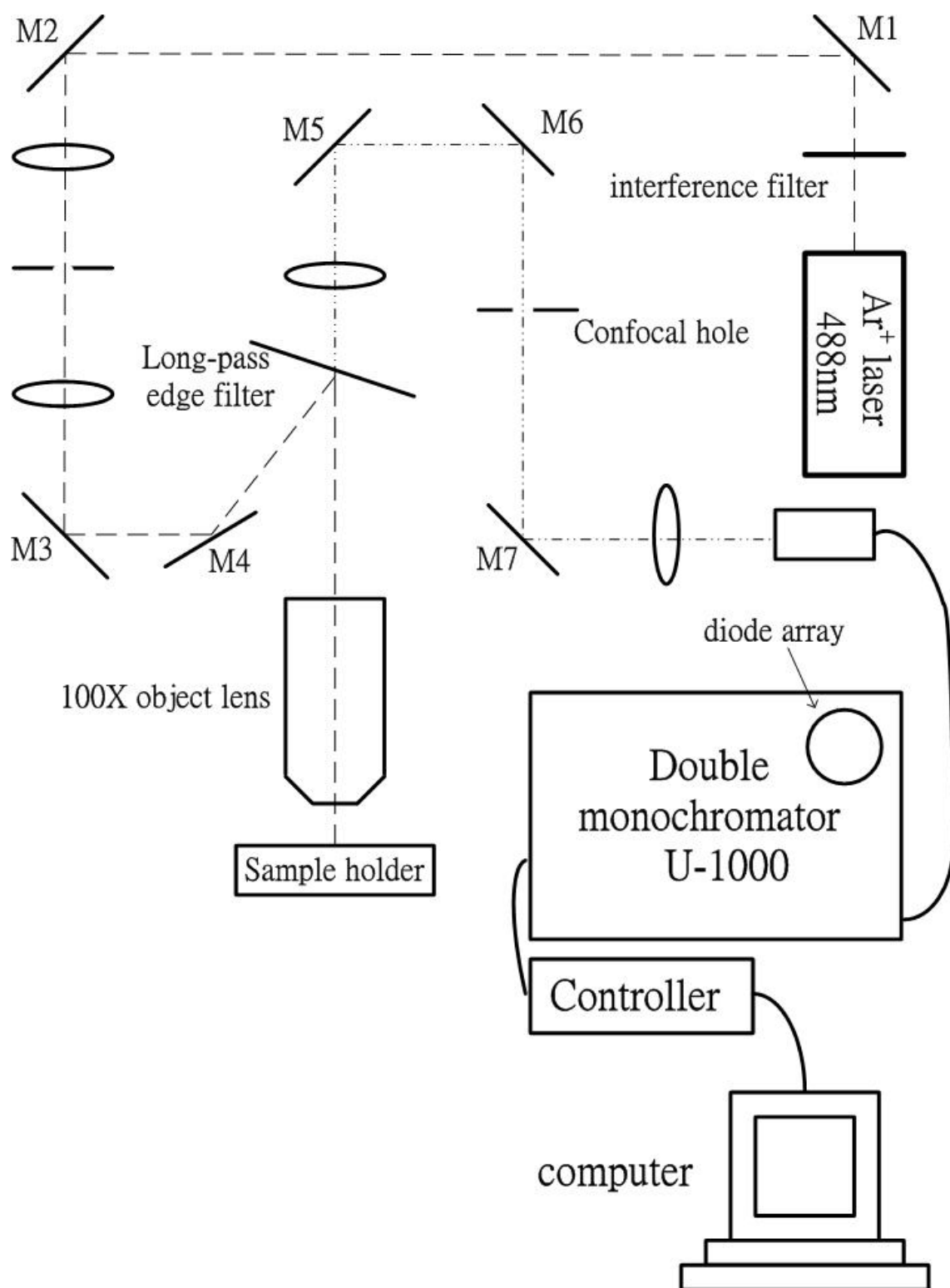


Fig.3-3-1 Micro-Raman system block diagram