Chapter 3. Experimental Procedure

3.1 Fabrication of GaN-nanowire based M-S-M device

The entire fabrication process of GaN-nanowire based MSM device was shown in Fig. 3.1. First of all, a commercially available single-crystalline sapphire wafer [Al_2O_3(001)] was used as a substrate for MSM device fabrication. Single-crystalline doped GaN epilayer (2.6μm) was then grown on sapphire substrate by well-developed molecular beam epitaxy (MBE) technique. The GaN-deposited sapphire substrate was subsequently spin-coated with photoresist (PR) and followed by photolithography and lift-off technique to create the patterns on the GaN epilayer. The Nickel layer was deposited by ion beam sputtering (IBSD) method on the patterned GaN epilayer, serving as either the hard mask or electrode for electrical measurement. Inductively coupled plasma reactive ion etching (ICP RIE) was utilized to etch the GaN epilayer while the Ni layer was taken as hard mask during the etching process. Prior to the growth of GaN nanowires, a Au thin film was then deposited on the patterned GaN epilayer, serving as the catalyst. Finally, the GaN nanowires were grown across the after-etched GaN film by catalytic chemical vapor deposition (CVD) technique.

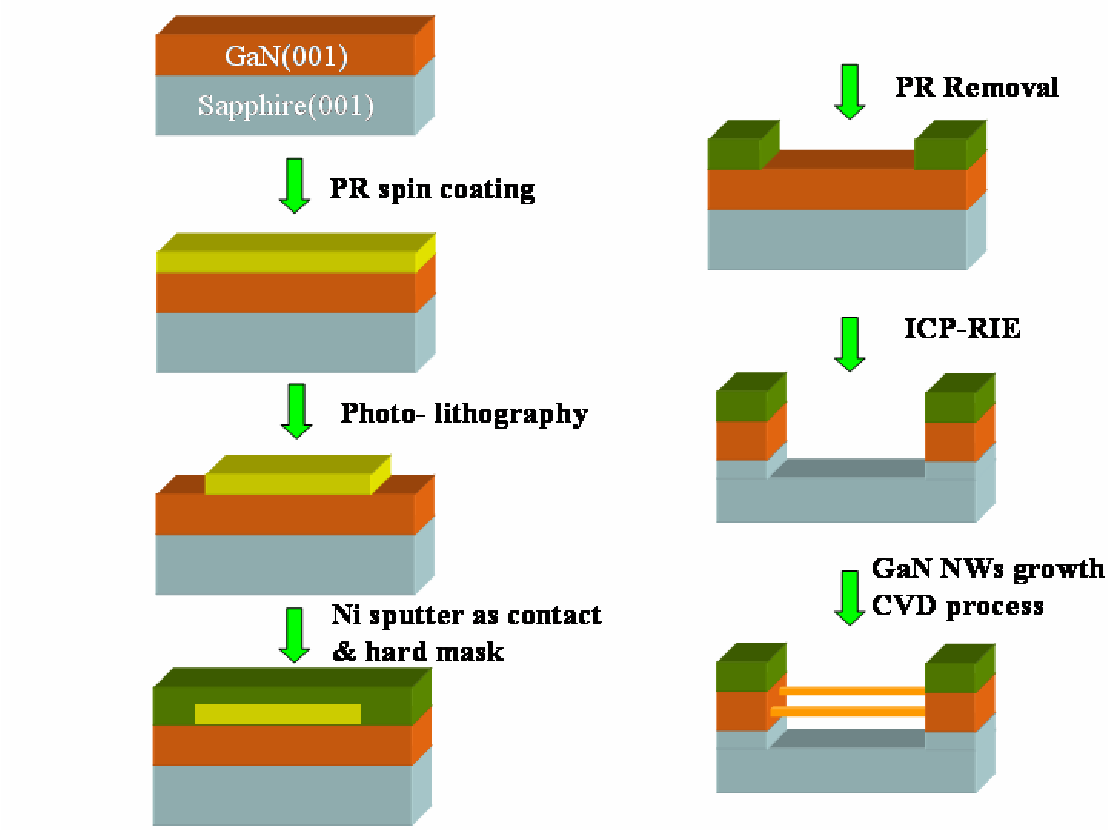

Fig. 3.1 Schematics of epi-GaN nanowire-based M-S-M structure fabrication.

3.2 Catalytic CVD growth of 1D GaN Nanowire

Figure 3.2 depicts the schematic of thermal-CVD reactor for the fabrication of GaN nanowires. The synthesis of GaN nanowires was carried out in a quartz tube (25 mm in outer diameter) furnace. The gold thin films (10 nm) serving as the catalyst weas then deposited on the patterned GaN epilayer by dc sputtering (EMITECH, K550X). Pure gallium powders (ACROS, 99.999%) were placed in an alumina boat to provide the gallium vapor source. Prior to growth, the quartz tube was degassed and then purged with ammonia NH_3 (30 sccm) and followed by increasing the temperature up to 900 °C at a rate of 50℃/min. During the growth period, the furnace was maintained at 900 °C for 30 min and followed by annealing in NH_3 ambient at 800 °C for 1 hr, as shown in the temperature program (Fig 3.3).

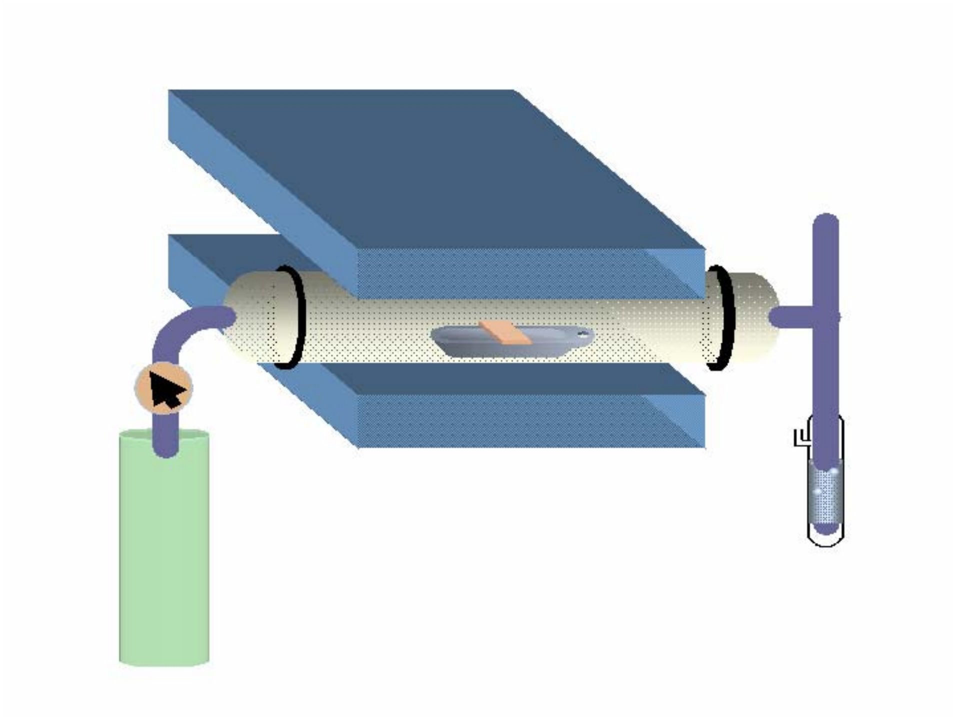

Fig.3.2 Schematic of thermal-CVD reactor.

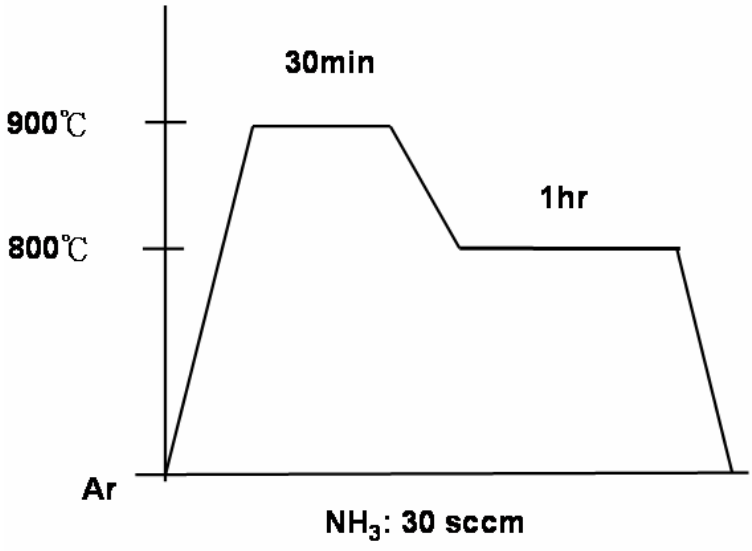

Fig.3.3 Temperature program of grown GaN NWs

3.3 Structural analysis and optical properties of GaN nanowires

3.3-1 Field Emission Scanning Electron Microscopy (FESEM)

The surface morphology of the nanowires was observed by field emission scanning electron microscopy (FESEM) using a JEOL 6700 system.

SEM is ton the most widely used technique used in characterization of nanomaterials and nanostructures. The resolution of the SEM approaches a few nanometer, and the instruments can operate at magnifications that are easily adjusted from ~10 to over 650,000. Not only does the SEM produce topographical information as optical microscopes do, it also provides the chemical composition information near the surface.

In a typical SEM, a source of electron is focused into a beam, with a very fine spot size of ~5 nm and having energy ranging from a few hundred eV to 50 KeV, that is rastered over the surface of the specimen by deflection coils. As the electrons strike and penetrate the surface, a number of interactions occur that result in the emission of the electrons and photons from the sample, and SEM images are produced by collecting the emitted electrons on a cathode ray tube (CRT). Various SEM techniques are differentiated on the basis of what is subsequently detected and imaged, and the principle images produced in the SEM are of three types: secondary electron images, backscattered electron images and elemental X-ray maps.

Fig.3.4　Field emission SEM（JEOL JSM-6700F）

3.3-2 High-Resolution Transmission Electron Microscopy (HR-TEM)

The high-resolution transmission electron microscopy (HR-TEM) was employed to analyze the microstructure and determine the growth direction of nanowires. Other than that, we also use HR-TEM to investigate the epitaxial relationship between the nanowrie and the substrate. The HRTEM (JEOL-4000EX) used in this study can be accessed at the department of material science and engineering at Tsing Hua University.

Fig.3.5 Schematic of CM 200 HRTEM

3.3-3 Cathodoluminescence Spectroscopy (CL)

The FESEM was also equipped with a cathodoluminescence system, which can be used for the luminescence studies of single nano-object. Besides, the measuring temperature of CL experiments can be tailored from room temperature to 4K using a liquid helium system. The liquid helium system is currently commercially available, based on a continuous-flow liquid helium cryostat. The advantage of cooling the observed samples are (1) there are substantial increase in CL intensity; (2) the decrease in measuring temperature can also facilitate the improvement of the signal-to-noise ratio; and (3) the rate of electron bombardment damage in electron beam-sensitive material is reduced. The powerful tool allows us to study the size effect of single nanowire on the bandgap.

3.4 Electrical measurement of GaN-nanowire M-S-M device

Current-voltage (I-V) and photoconductivity (PC) measurements of the GaN NWs detector was carried out using a micro-probe system (Everbeing Int'l Corp.) which is equipped with a microscope for controlling two tungsten needles (tip size ~ 2 μm) to precisely touch onto the micrometer-sized patterned electrodes. The Keithley model 230 voltage-source unit and Keithley model 617 multimeter were utilized to applied the external bias and to measure the electrical current in the I-V and PC experiments. The light source generated from a 150 W xenon lamp was introduced from the monochromator (PTI model 101) into the black box. The variable monochromic light beam was aligned and focused onto the sample in dark surroundings at room temperature. The light power was carefully calibrated and the power at 3.5 and 4.0 eV are 122 and 70 μW, respectively. Usually, the scan range of the photon energy is from 2.0 to 4.0 eV (λ = 620 – 310 nm) for each PC experimental run.