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Experimental Techniques
2.1 Sample Preparation

2.1.1 Cleaning of Samples

The GaN surface is very reactive with ambient oxygen, resulting in a native oxide which acts as a barrier for carrier transport and consequently increases the specific contact resistance. Also, the presence of oxides and contaminants on the GaN surface prior to metallization has a direct influence on the values of Schottky barrier height and ideality factor of Schottky contacts. Thus, proper cleaning and removal of oxides and contaminants on semiconductor surface prior to metal deposition is very important for ohmic and Schottky contacts [1-5].

Though a number of groups have investigated GaN cleaning procedures for device fabrication, currently there is no standard method of cleaning GaN surface prior to metallization. The method followed for the current work involves the following.

The first step in the cleaning process is to ultrasonically degrease the p- or n-type GaN layer in warm trichloroethylene, acetone and methanol for 5 min each. Then the surface oxides are removed by dipping the sample in boiling aqua regia [HNO₃: HCL (1:3)] or buffered oxide etch (BOE) solution for 10 min followed by deionized (DI) water rinse and dry nitrogen blow [6-13]. The details of fabrication of the samples, metal schemes under investigation in the present work will be discussed in chapters 3.1.2, 3.2.2 and chapter 4.2.

2.1.2 Electron Beam (e-beam) Evaporation System

The electron beam evaporation system used for the current work consists of a metallic chamber with a glass window. The system has two sets of pumps: the high vacuum and rotary pump. Using the four source
electron gun assembly, metals including titanium, tungsten, platinum, silver, gold and rhodium have been evaporated in the system. The purity of the metals varies from 99.9999 % to 99.99, %, depending upon the supplier and the type of metal. The source material is placed in one of the crucibles that are part of the water cooled anode. A cathode assembly consisting of a tungsten filament and a permanent magnet deflects the thermionic electrons onto the metal targets. The anode is fixed onto a linear motion feedthrough and can be positioned to have any of the four targets aligned for evaporation.

The power supply for the electron beam system is a 3 KW beam control unit. Output current is remotely controlled and can be set to any value in the range 0-500 mA. Since the anode gets heated rather quickly when the system is evaporating, water cooling is employed for anode. To ensure that the coolant water is flowing through the anode when the system is switched on, a flow switch is connected in the anode water cooling loop. The switch is set to trip “ON” when the water flow through the anode is greater than 5 liters per minute. If the flow is less than this, the high voltage will not be enabled, and the target evaporation will be stopped to save the anode from over heating.

The digital thickness meter (DTM-101) determines the thickness of the deposited metal from the change in oscillations of the quartz crystal. Deposition rates from 0.2 to 1.0 angstrom units per second have been obtained for most metals of interest.

2.1.3 Thermal Evaporation System

Thermal evaporation is used to evaporate low melting point metals such as aluminum (Al), silver (Ag) etc. It consists of a heating filament or
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a boat made of tungsten or molybdenum, through which a current is passed to heat the metal to the temperature equal to its melting point and to evaporate. The only disadvantage of the thermal evaporation is that the quantity of metal required is more and the evaporation is spread over a wide area which results in the wastage of the metal.

2.1.4 Rapid Thermal Annealing (RTA) System

Annealing conditions also significantly affect the contact properties. Heat treatment performed in various ambient results in different reaction products. Moreover, the depth of diffusion is a function of annealing temperature and time, thereby changing the extent of contact reaction depending on the annealing conditions chosen.

RTA System is used for rapid thermal annealing of samples in nitrogen ambient and air at normal atmospheric pressure. The system consists of a heating chamber with an automatic heat control system. The wafer to be processed is placed on a quartz tray, which slides into a quartz tube in the heating chamber. To measure the sample temperature, thermocouple and pyrometer are used. Annealing temperature is fixed/set by a preset adjustment on the front panel of the system. The temperature is automatically monitored and controlled so that it remains constant during annealing. The dry nitrogen gas flow can be controlled using a flow gauge built-in to a controller.

2.2 Characterization Techniques

2.2.1 Auger Electron Spectroscopy (AES)

Pierre Auger, in 1923, discovered the Auger process/effect and Auger electrons while irradiating samples with x-rays. The basic Auger process consists of back scattered electron due to bombardment with an
electron beam and removal of an inner shell electron to form a vacancy. The inner shell vacancy is filled immediately by a second atomic electron from a higher shell. In a radiation-less process, the energy liberated in this transition is simultaneously transferred to a third electron in the higher shell and emits an Auger electron. This process of an excited ion decaying into a doubly charged ion by ejection of an electron is called the Auger process. The Auger electrons are emitted with discrete energies that are characteristic of the elements present on the sample surface. By analyzing Auger electrons as a function of energy, the peak positions are used to identify the elements and the chemical states present. All elements in the periodic table, except hydrogen and helium, can be detected. The depth of analysis is in the range of 3-5 nm or top 2-20 atomic layers. As the electron beams can be focused to a very small probe size, excellent spatial resolution (up to 0.5 μm) [14] can be achieved.

The process of Auger electron emission in a semiconductor is illustrated in figure 2.1. The energy band diagram shows different energy bands including lower core levels, K and L levels, with discrete energies $E_K$, $E_{L1}$ and $E_{L2,3}$. A primary electron from an electron gun knocks an electron out from the K shell. This K shell vacancy is immediately filled by an electron in the L$_1$ shell. The difference energy, $E = E_{L1} - E_K$, is transferred to a third electron – the Auger electron – from the L$_{2,3}$ shell. A fraction of this energy is used to overcome the binding energy of this third electron and the remainder is retained by the emitted Auger electron as kinetic energy. Auger spectroscopy is based on the measurement of the kinetic energies of the emitted electrons. Each element in a sample being studied will give rise to a characteristic spectrum of peaks at various kinetic energies.
AES depth profile is a popular method of looking at the buried layers at the sample surface. During depth profile, when the sample is brought into the UHV environment from air, it is coated with carbon and oxygen. This coated material is removed by sputtering before the sample surface is investigated. Sputtering involves directing a beam of argon ions at energies between 500 eV and 5 keV at the sample [figure 2.2]. This process cleans the surface and erodes the sample to reveal the structure beneath the surface. Obviously this is a destructive technique.
2.2.2 Glancing Angle X-Ray Diffraction (GXRD)

Glancing angle x-ray diffraction is a versatile, non-destructive technique that reveals detailed information about the surface structure, chemical composition and thickness of thin films.

X-ray diffraction is governed by Bragg equation that describes the condition for constructive interference for x-rays on scattering from the periodic array of atoms in crystals. The Bragg equation is given by

\[ n\lambda = 2d \sin\theta, \]

where \( n = 1, 2, 3, \ldots \), \( \lambda \) is the wavelength of the incident x-rays, \( d \) is the inter-planar spacing of the crystal, and \( \theta \) the Bragg angle [figure 2.3]. Different \( d \)-spacings in the crystal satisfy the Bragg's condition for constructive interference at different incident angle. Plotting the angular positions \( \theta \) or \( 2\theta \) and intensities of the resultant diffracted peaks of radiation produces a pattern, which is characteristic of the sample.

![Figure 2.3: X-ray diffraction by a crystal](image)

2.2.3 Atomic Force Microscopy (AFM)

The atomic force microscope (AFM) is a very high-resolution type of scanning probe microscope, with a resolution of fractions of an Angstrom,
more than 1000 times better than the optical diffraction limit. The AFM was invented by Binnig, Quate and Gerber in 1986, and is one of the primary tools for imaging, measuring and manipulating matter at the nanoscale.

The AFM consists of a microscale cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface [figure 2.4]. The cantilever is typically silicon or silicon nitride with a tip radius of curvature of the order of nanometers. When the tip is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law. Depending on the condition, forces that are measured in AFM include mechanical contact force, Van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces, Casimir forces, solvation forces etc. Typically, the deflection is measured using a laser spot reflected from the top of the cantilever into an array of photodiodes. To avoid the risk of collision of tip of the cantilever with the sample surface, a feedback

Figure 2.4: Schematic of atomic force microscope
mechanism is employed and the tip-to-sample distance is adjusted to maintain a constant force between the tip and the sample. The sample is mounted on a piezoelectric tube (PZT, in figure), which moves the sample in the z direction for maintaining a constant force, and in the x and y directions for scanning the sample. The resulting map of the area represents the topography of the sample.

2.2.4 Scanning Transmission Electron Microscopy (TEM) and High-Resolution Transmission Electron Microscopy (HREM)

Transmission electron microscopes work much like optical microscopes; both consist of a series of lenses. Transmission electron microscopes use electrons instead of light and therefore the much lower wavelength of electrons makes it possible to get a resolution a thousand times better than with a light microscope. The resolution of transmission electron microscopes approaches 1.8 to 2 Å. A fine beam of electrons from an electron gun are accelerated to high voltages (typically 100 to 400 kV), and focused on the sample with the help of condenser lenses. The transmitted and forward scattered electrons form a magnified image in the image plane. With additional lenses the image is either projected onto a fluorescent screen for viewing and for photographic recording.

In scanning transmission electron microscopy (STEM), a fine beam of electrons (beam diameter <100 Å) is scanned across the sample in a raster fashion. The objective lens recombines the transmitted electrons from all points scanned by the probe beam to a fixed region in the back focal plane, where they are detected by an electron detector. The detector output is fed to an electronics setup for viewing and/or storing the image.

Electron optics engineering has evolved over the past few decades and has improved the precision of electron microscopes by counteracting
the image blurring effects of lens imperfections, or 'aberrations' [15-16]. These high-resolution transmission electron microscopes have so much resolution that they can be used to obtain images of individual atoms inside a material. High-resolution transmission electron microscopy (HREM) is used to investigate the microstructures of oxide-semiconductor, metal-semiconductor and semiconductor-semiconductor interfaces at atomic levels. High-resolution transmission electron microscopy is also known as lattice imaging, and is a very important tool for interface analysis.

2.2.5 Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) Analysis

Scanning electron microscopy is used for inspecting topographies of specimens at very high magnifications up to 300,000 X. In SEM, a high energy electron beam (typically 10 keV) is focused on a spot volume of the specimen, resulting in the transfer of energy to the spot. These bombarding electrons, also referred to as primary electrons, dislodge electrons from the specimen itself. The dislodged electrons, also known as secondary electrons, are attracted and collected by a positively biased grid or detector, and then translated into a signal. To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, analyzed, and translated into images of the topography being inspected. Finally, the image is shown on a CRT. Figure 2.5 illustrates secondary electron emission in scanning electron microscopy [14].

Apart from secondary electrons, the primary electron beam results in the emission of backscattered (or reflected) electrons from the specimen. Backscattered electrons possess more energy than secondary
electrons, and have a definite direction. All emissions above 50 eV are considered to be backscattered electrons.

Figure 2.5: Secondary electron emission in scanning electron microscopy

A scanning electron microscope may be equipped with an energy dispersive x-ray analysis (EDX) system to enable it to perform compositional analysis on specimens. EDX analysis is useful in identifying materials and contaminants, as well as estimating their relative concentrations on the surface of the specimen.

In EDX, an electron beam of sufficiently high energy (in the range 10-20 keV) is incident the material surface. The high energy electron bombardment causes x-rays to be emitted from the material surface. The energy of the x-rays emitted depends on the material under examination. By moving the electron beam across the material, an image of each element in the sample can be acquired. Due to the low x-ray intensity, images usually take a number of hours to acquire. Elements of low atomic number are difficult to detect by EDX.
2.3 Electrical Characterization

2.3.1 Current-Voltage (I-V) Measurements

Schottky barrier heights are usually deduced from current-voltage and capacitance-voltage methods. One tacit assumption in using these measurement techniques is that the SBH under investigation is homogeneous. In an I-V experiment, the junction current is measured as a function of the applied bias voltage (both forward and reverse bias).

Energy-band diagram of a metal-semiconductor junction under forward and reverse bias is illustrated with figure 2.6 [14]. Figure 2.6 (a) shows band diagram for a typical rectifying metal-semiconductor contact at equilibrium without any bias applied. The net current flow is zero. When a positive bias ($V_f$) is applied to the junction (figure 2.6(b)), the Fermi energy of the semiconductor is raised with respect to the Fermi energy in the metal. This decreases the built-in potential across the semiconductor from $V_{bi}$ to $(V_{bi} - V_f)$. A positive current flows through the junction when the bias voltage is comparable to the built-in potential. On the other hand, when a negative bias ($V_r$) is applied (figure 2.6(c)), the Fermi energy of the semiconductor is lowered with respect to the Fermi energy in the metal. This increases the built-in potential across the semiconductor from $V_{bi}$ to $(V_{bi} + V_r)$.

In most Schottky contacts fabricated on moderately doped semiconductors, thermionic emission over the barrier is usually assumed. The current equation for an ideal metal-semiconductor contact is given by [17]

$$I = I_s \exp \left[ \frac{q(V - IR)}{nkT} \right] - 1 ,$$

(2.2)
where $I_s$ is the saturation current, $q$ is the electron charge, $V$ is the applied voltage, $A$ is the area of the contact, $R$ is the series resistance, $n$ is the ideality factor, $A^{**}$ is the effective Richardson constant, and $\phi_b$ is the Schottky barrier height [17]. The values of Schottky barrier height and ideality factor are determined by plotting the forward bias current versus voltage. The ideality factor $n$ of the diode can be determined from the

\begin{equation}
I_s = A A^{**} T^2 \exp \left( \frac{-q \phi_b}{kT} \right),
\end{equation}

\(2.3\)

Figure 2.6: Energy-band diagram of metal n-type semiconductor
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slope of the linear curve of $\ln I$ versus $V$, using the relation

$$n = \frac{q}{kT \times \text{slope}}. \quad (2.4)$$

In general, ideal diodes have ideality factor in the range $1.02 < n < 1.1$. Deviation of $n$ from this range can be caused by many different factors such as interface layer, recombination in the depletion region etc. The value of $\phi_0$ can be deduced directly from the I-V curves if the effective Richardson constant, $A^{**}$ is known. The theoretical value of $A^{**}$ is 26.4 A cm$^{-2}$ K$^{-2}$ based on the effective mass ($m^* = 0.22 m_0$) of n-GaN [18]. A graph of $\ln I/[I-\exp(-qV/kT)]$ versus $V$ yields $\ln I_s$ as the intercept. Once $I_s$ is found, the Schottky barrier height can be determined using the equation (2.3) as

$$\phi_0 = \frac{kT}{q} \ln \left( \frac{AA^*T^2}{I_s} \right). \quad (2.5)$$

2.3.2 Capacitance-Voltage (C-V) Measurements

Capacitance-voltage (C-V) measurements employ reverse biasing of the contact to determine the barrier height. A DC reverse bias is applied to the contact with a small AC voltage superimposed onto the DC voltage. As a result of the application of AC, charges of opposite polarity are induced on the semiconductor and metal surfaces. This alignment of charge gives rise to a junction capacitance. In this technique a range of reverse bias is applied to the contact and the resulting capacitance is measured.

The depletion region width ($W$) can be expressed by the equation [17-19]

$$W = \sqrt{\frac{2e_s e_o}{qN_d} \left( V_{bh} - V - \frac{kT}{q} \right)}, \quad (2.6)$$
where $V_{bi}$ is the built in voltage, $V$ is the applied reverse bias, $N_d$ is the donor concentration, \( \varepsilon \) is the permittivity of vacuum, and \( \varepsilon_{sc} \) is the semiconductor dielectric constant. The charge of the depletion region is expressed as

$$Q_{sc} = qN_dW = \sqrt{2q\varepsilon_{sc}\varepsilon_0N_d\left(V_{bi} - V - \frac{kT}{q}\right)}.$$ (2.7)

The capacitance can be expressed as

$$C = \frac{\partial Q_{sc}}{\partial V} = \frac{q\varepsilon_{sc}\varepsilon_0N_dA^2}{\sqrt{2\left(V_{bi} - V - \frac{kT}{q}\right)}}.$$ (2.8)

Equation (2.8) can be rewritten as

$$\frac{A^2}{C^2} = \frac{2\left(V_{bi} - V - \frac{kT}{q}\right)}{q\varepsilon_{sc}\varepsilon_0N_d}.$$ (2.9)

All the variables in the above equation are constants except the applied voltage ($V$). Therefore, the plot of $1/C^2$ versus $V$ should be linear. The slope of the characteristic curve gives

$$slope = \frac{2}{q\varepsilon_{sc}\varepsilon_0N_d},$$ (2.10)

from which carrier concentration can be determined. The intercept on the voltage axis $V_0$ can be used to determine the barrier height as follows. $V_0$ is related to built potential $V_{bi}$ by the equation $V_{bi} = V_0 + kT/q$, where $T$ is the absolute temperature. The barrier height is given by the equation $\phi_b = V_0 + V_n + kT/q$, where $V_n = (kT/q) \ln (N_c/N_d)$. The density of states in the conduction band edge is given by $N_c = 2(2\pi m^* kT/\hbar^2)^{3/2}$, where $m^* = 0.22m_0$ and its value $2.53 \times 10^{18}$ cm$^{-3}$ for GaN [20].
In general, the value of barrier height ($\Phi_b$) measured using C-V method is more than I-V method, since current-voltage measurements are more sensitive to the defects and imperfections at the metal-semiconductor interface. Current-voltage (I-V) measurements are more sensitive to non-uniformity of the interface than C-V measurements. Capacitive-voltage (C-V) measurements are sensitive to defects in the gap of the semiconductor.
References


