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Thermal Stability and Degradation Analysis

of GaN/AlGa_N Heterostructure

A thesis submitted in partial satisfaction

of the requirement for the degree Master of Science

in Material Science and Engineering

by

Teng Zhang

2018

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ABSTRACT OF THE THESIS

Thermal Stability and Degradation Analysis

of GaN/AlGaN Heterostructure

by

Teng Zhang

Master of Science in Materials Science and Engineering

University of California, Los Angeles, 2018

Professor Dwight Christopher Streit, Chair

AlGaN/GaN heterostructure is one of the most important materials in applications like High Electron Mobility Transistors (HEMT) and other high speed high power devices. Harsh environment is often encountered for these devices so the thermal stability of the AlGaN/GaN heterostructure is essential. Despite large efforts spent in the last decades, the reliability of

GaN HEMT and GaN related devices still represent an issue. The role of high temperature in the degradation of AlGaIn/GaN heterostructure is controversial and multiple process occur simultaneously upon thermal activation.

This work presents a study of thermal stability of AlGaIn/GaN heterostructure in various temperature and different atmosphere. X-ray diffraction and Raman spectroscopy were utilized to characterize the structure degradation. For N₂ atmosphere annealing, crystal structure can be maintained up to 1000°C, with better crystallinity due to recrystallization.

The FWHM drop for N₂ annealed sample is up to 38.9%. Significant degradation is observed when annealed in air: (i) irreversible lattice relaxation; (ii) oxidation and defect propagation; (iii) phase separation of AlGaIn. Starting from 750°C, no crystal structure of AlGaIn can be detected. Possible failure mechanism is discussed, and these results may be instructive for future device fabrication and optimization.

The thesis of Teng Zhang is approved.

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Jamie Marian

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University of California, Los Angeles

2018

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1. Introduction

1.1 Motivation

In the age of information, the increasing demand of wireless communication prompts the development of semiconductor technologies. In the past decades III-nitride semiconductors have been widely explored, for its great potential in applications such as cellular, personal communication, and radar, some of which are already available on market.

Table 1.1 Properties of several semiconductors¹

Properties	Si	GaAs	4H-SiC	GaN
Band-Gap (eV)	1.12	1.43	3.26	3.4
Dielectric Constant	11.4	13.1	9.7	9.5
Electron Mobility (cm ² /Vs)	1350	8500	700	900
Saturated Electron Velocity (10 ⁷ cm/s)	1.0	2.0	2.0	2.5
Breakdown Field (MV/cm)	0.4	0.5	2.0	3.3
Maximum Temperature(°C)	300	300	600	700

Gallium Nitride is an ideal material for high power microwave devices and circuits, the properties of GaN material compared with other semiconductors are listed in Table 1.1. The wide

band gap enables higher power per unit width for device active layer. The high breakdown voltage allows higher operation voltage and thus higher efficiency. The bulk mobility for GaN may not be the highest but it can retain its high mobility in devices, if not higher, whereas other type of semiconductors requires high-level doping which introduces massive scattering center and decreases the mobility. For GaN/AlGa_N heterostructure, 2D electron gas(2DEG) can be formed without intentionally doping, due to the polarization effect, which will be elaborate later. With its high saturation electron velocity, GaN devices can amplify high-frequency signals up to hundreds of gigahertz. Well-developed epitaxy technique allows us to grow GaN structure on Silicon Carbide and Sapphire with limited lattice mismatch, rendering good thermal conductivity, and making it easier to dissipate heat from a small package.

With such superior properties, GaN High-Electron-Mobility-Transistors (HEMT) can easily operate in conditions that cannot be achieved in other materials. Starting from 2004, the GaN HEMT device is commercially available on market, and lots of efforts have been devoted to the optimization of GaN HEMT device as well as fabrication technique. Up to now, the drain voltage of GaN HEMT can reach some hundreds of volts².

However, the very high voltage applied can induce high electric field within the active layer and cause various kinds of degradation. To improve the reliability of GaN-based HEMT, a better understanding of the degradation mechanism is required.

1.2 Objective

Basically, the failure of GaN HEMT can be classified into three categories: thermally induced failure, bias induced failure and those caused by the piezoelectric nature of GaN³. More often than not, it is the combination of these failure mechanism that cause a device to fail. In the past decade, lots of efforts have been devoted to the exploration of electrical degradation, such as current collapse, gate leakage and several mechanisms have been proposed^{1,3-6}. These reports give practical guidance on the improvement of GaN HEMT devices and its stability.

High temperature is often encountered in both processing and operation of GaN HEMT devices and is a major factor of degradation. Certain temperature range (200°C-600°C) is reported to have the effect of aggravating electrical degradation³, by generating trapping site or activating dislocation propagation. Thermally-induced delamination of passivation layer and contact failure may also result from high temperature. At higher temperature, the heterostructure undergoes a

rather complex change including structure deformation and composition variation. The mechanism behind these changes remain unknown to date, this dissertation is dedicated to exploring the structural change in the AlGaIn/GaN heterostructure upon high-temperature annealing. Such mechanism may be good instruction for material processing and device packaging of GaN HEMTs.

2. Background

2.1 GaN, AlGaN and AlGaN/GaN heterostructure

GaN is first synthesized in 1932 by Johnson *et al* and is considered a stable compound with great hardness. These properties make GaN good material for protective coatings. Later, people start to realize that its wide energy gap is suitable for device operation under high power and caustic environment. By the end of the twentieth century, main obstacles for GaN material, including etching, growth of single crystal GaN, and p-type doping, have been successfully solved, and soon after, commercial GaN device started to appear on the market.

AlN, similar to GaN, also has good thermal and mechanical properties, with a much higher energy gap. Combined with its good thermal match with Si and GaAs, it is an ideal material for electronic packaging and insulating. It is its ability to form alloys with GaN, however, producing $\text{Al}_x\text{Ga}_{(1-x)}\text{N}$ that attracts so many interests. The band gap of the alloy ranges from 3.4eV to 6.2eV, which is from the green wavelength well into the ultraviolet, the E_g of AlGaN alloy can be calculated by Eq.2.1

$$E_g = xE_g(\text{AlN}) + (1-x)E_g(\text{GaN}) - bx(1-x) \quad (2.1)$$

where b is the bowing parameter having controversial values ranging from -1eV to 1eV, depending on the composition, and $E_g(\text{AlN})=6.2\text{eV}$, $E_g(\text{GaN})=3.4\text{eV}$.

Three crystal structure can be found in group-III nitrides: wurtzite, zinc blende and rocksalt structure. The wurtzite structure is thermodynamically favored in ambient condition and will transform into rocksalt under very high pressure. Zincblende can be stabilized by epitaxy on $\{011\}$ crystal plans of cubic substrate. A great deal of interests is in the wurtzite structure, which has no inversion symmetry that will lead to spontaneous polarization^{7,8}. As shown in figure 2.1, the wurtzite structure has a hexagonal unit cell with tow lattice constant a and c . For AlGaN, the Ga atoms are replaced by Al atoms.

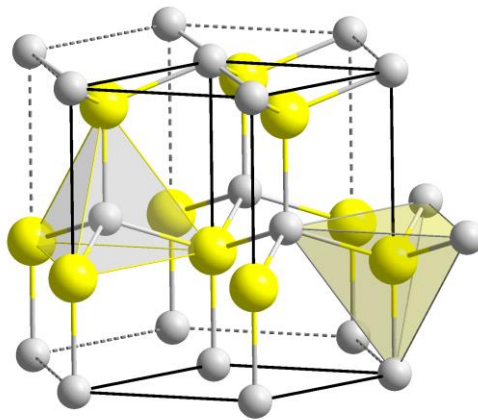


Figure 2.1 Wurtzite structure of GaN. Yellow sphere represents Ga atom and gray ones represent N atom.

In AlGaIn/GaN heterostructure, AlGaIn act as barrier and a quantum well (QW) can be formed at the interface. The electronic and optical properties of GaN and AlGaIn structure have been extensively studied. It is calculated that the Ga 3d electron is strongly hybridized with both the upper and lower valence band s and p levels⁹. This hybridization has profound influence on properties of GaN such as band-gap, lattice constant, acceptor level, etc. In Al, however, there is no 3d core states thus no hybridization effect between N 2s states. As a result, AlN possess much different properties as GaN. Furthermore, it has been predicted that in wurtzite structure such as ZnS or ZnSe, acceptors like Cu, whose d electrons are resonance with the lower valence band, are repelled by d-hybridized upper valence band resulting in a deep level energy state. Impurities without d electrons, such as Mg, can form shallow level, which makes them good candidate for room temperature p type dopant. It is worth mentioning that due to hydrogen compensation, Mg doped GaN need to be activated by annealing or other approach to become conductive p type semiconductor.

The AlGaIn/GaN heterostructure can be used in heterostructure field-effect transistor (HFET). In traditional silicon, good transport property is achieved by inducing an inversion layer.

The channel can be formed on the interface of silicon with its native oxide SiO_2 which is easy to obtain. However, in III-V semiconductor, no such oxide is available. A heterostructure like AlGaIn/GaN with large band gap difference is needed to form the channel. As shown in figure 2.2, a wide band gap material (AlGaIn) depleted by build-in voltage of the metal semiconductor contact is used instead of an oxide insulating layer.

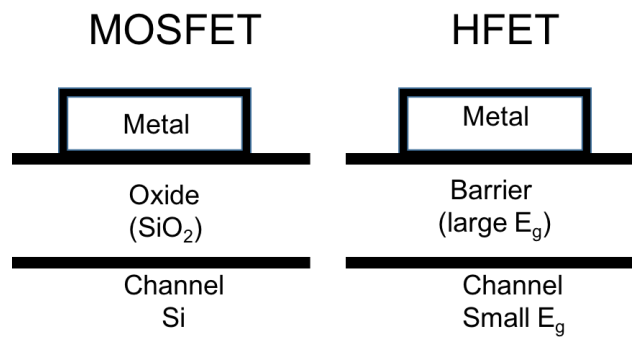


Figure 2.2 The structure of different types of FETs

At this kind of interface an inversion layer of electron (or holes) can be formed, which also known as 2D electron gas(2DEG). The polarization effect of AlGaIn/GaN structure allows the GaIn HFET to achieve very high carrier density without intentionally doping. Also, without dopants acting as scattering center, the carrier mobility is mainly determined by the interface quality and, as recently reported, can reach as high as $1200\text{cm}^2/\text{V s}$ at room temperature.

2.2 Polarization effect in AlGaN/GaN heterostructure

As previously mentioned, wurtzite structure lacks inversion symmetry, combined with the relatively large electronegativity difference between III atom and N, III-nitride exhibit high spontaneous polarization. Without any strain, AlN possesses a spontaneous polarization only 3 to 5 times smaller than that of a particular ferroelectric material^{10,11}. Also, the wurtzite structure has three non-vanishing independent components in its piezoelectric tensor, which leads to piezoelectric effect. Compared with traditional III-V or II-VI semiconductors, the spontaneous and piezoelectric polarization in Wz GaN and AlN is several times larger. Furthermore, due to the lattice constant difference, strain will be induced at the interface of AlGaN/GaN heterostructure, causing additional electrical field. It is found that a total electrical field up to 5MV/cm can be formed at the interface, result in high interface charge density.

As can be seen in figure 2.1, tow layer sequence can be distinguished in non-center-symmetric wurtzite structure: AB and BA (or A face and B face, Ga face and N face, corresponding to [0001] and [000 $\bar{1}$] direction). In the epitaxy procedure of AlGaN/GaN heterostructure, the polarity is of great importance and in GaN. Different etching rate of Ga face and N face can be

used to determine the polarity of GaN.

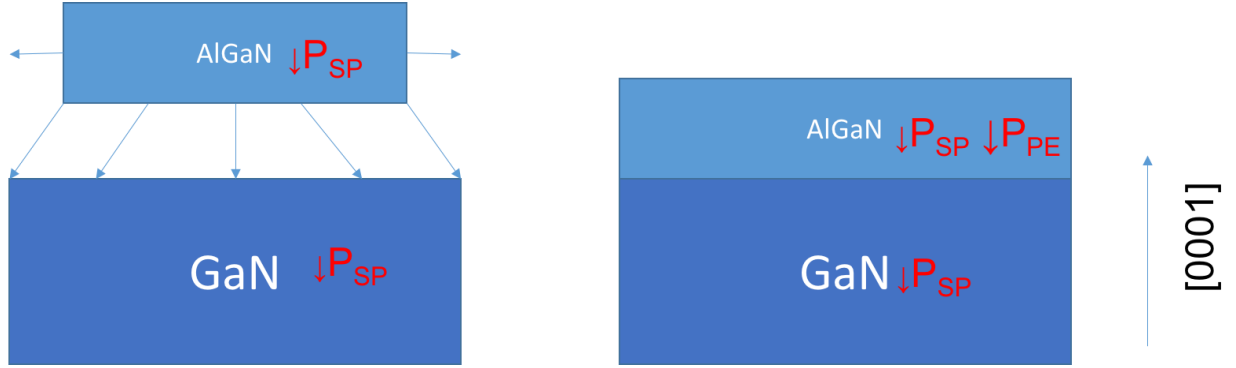


Figure 2.3 Spontaneous and Piezoelectric polarization in AlGaIn/GaN heterostructure

The total polarization $\mathbf{P}$ is the sum of spontaneous polarization $\mathbf{P}_{SP}$ and strain induced piezoelectric polarization $\mathbf{P}_{PE}$. The spontaneous polarization of GaN and AlN is negative to the basal direction, i.e. to the $[000\bar{1}]$ direction as shown in figure 2.3. The magnitude of $\mathbf{P}_{SP}$ is increasing as x goes from 0 to 1.

$$P_{SP}(x) = (-0.053x - 0.029) \text{ C/m}^2 \quad (2.2)$$

where x is the Al content in $\text{Al}_x\text{Ga}_{(1-x)}\text{N}$. Due to 2.4% lattice constant difference between GaN and AlN (at 300K), piezoelectric polarization is induced at the interface. The polarity of piezoelectric field is pointing from A face to B face, i.e. from the cation terminated face to anion terminated face, and the magnitude is

$$P_{PE}(x) = 2 \frac{a(x)-a(0)}{a(0)} [e_{31}(x) - e_{33}(x) \frac{c_{13}(x)}{c_{33}(x)}] \quad (2.3)$$

where $a(x)$ and $a(0)$ is lattice constant of $\text{Al}_x\text{Ga}_{(1-x)}\text{N}$ and GaN , respectively, e is piezoelectric constant, C is elastic constant (both e and C are in tensor form).

Thus, the overall charge density at the interface is

$$\begin{aligned}\sigma &= P(\text{AlGaN}) - P(\text{GaN}) \\ &= P_{SP}(\text{AlGaN}) + P_{PE}(\text{AlGaN}) - P_{SP}(\text{GaN})\end{aligned}\quad (2.4)$$

In the case of A face orientation, the positive charge induced by polarization will be located at the AlGaN/GaN heterointerface. These positive charges will be compensated by electrons that fell into a quantum well structure at the interface as shown in figure 2.4. Since the bandgap of AlGaN is larger, electrons in the “valley” is hard to climb over the energy barrier, thus 2DEG formed.

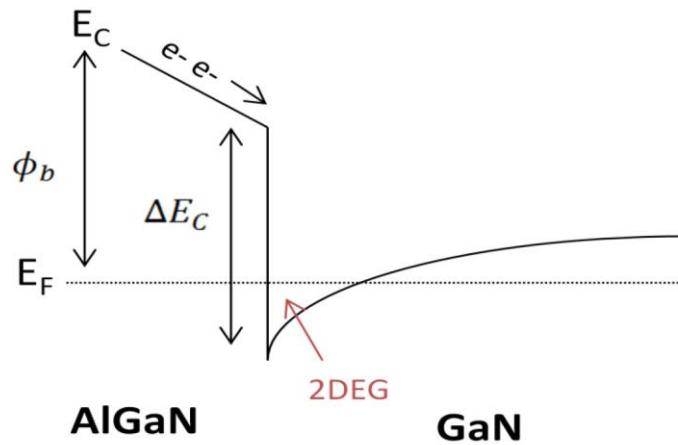


Figure 2.4 Band structure at the AlGaN/GaN interface

Since the carriers in the channel layer is induced by charges in the barrier layer, these electrons have an increased mobility compared with those in bulk materials because they are spatially separated from the dopants^{12,13}. In unintentionally doped AlGa_N/Ga_N structure, even better transport property can be expected because of better quality of crystal structure¹⁰.

2.3 Effect of barrier width and Al content on 2DEG

As discussed above, the positive charges in the barrier layer induce 2DEG in the channel, yet the origin of the 2DEG is more complex to explain.

Surface donor-like states is believed to be responsible for the 2DEG formation, and simulations based on this hypothesis fits well with experimental results^{10,14,15}. Substitutional donor such as oxygen might be incorporated in the AlGa_N surface, and dangling bonds or vacancies may also act as donor state. These states sit in deep level within AlGa_N energy gap and is occupied when barrier width d is small, as can be seen in figure 2.5 (a). As barrier width increases, donor states approach fermi level and finally the fermi level at surface slides down below donor energy, as shown in figure 2.5(c), and they start emptying.

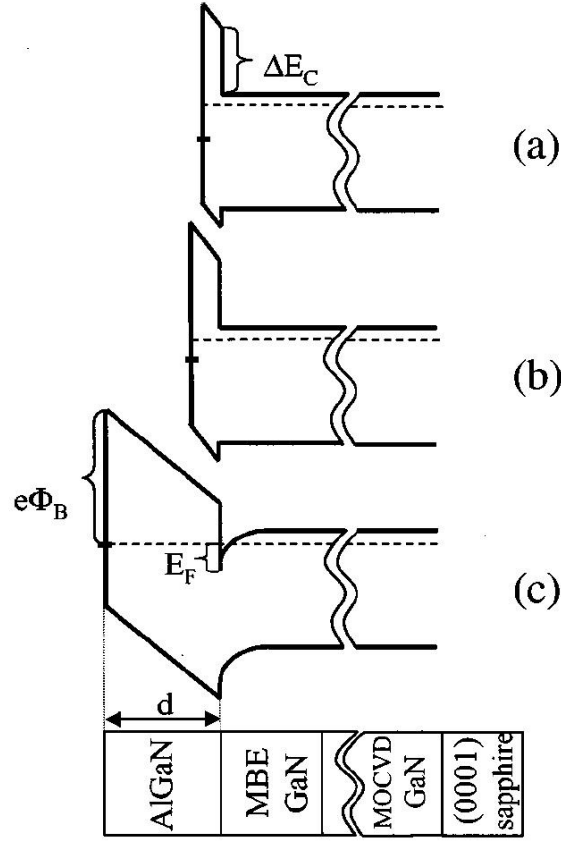


Figure 2.5 Band structure with increasing barrier width d

The Al content x is another crucial factor influencing the properties of the structure in many ways. First, x influences the energy gap, which directly determines the energy offset ΔE_C between GaN and AlGaN. When ΔE_C is small, the quantum well height is insufficient to confine the electrons and result in low electron density. Meanwhile, increasing the Al content in barrier layer can also increase the piezoelectric and spontaneous polarization effect. In a moderate range of Al mole fraction, 2D electron density N_s increases in a linear-like fashion with increasing Al fraction

x^{14-17} . The 2D electron density can be expressed as a function of x :

$$N_S(x) = \frac{\sigma(x)}{e} - \frac{\epsilon_0 \epsilon(x)}{de^2} [e\Phi_B(x) + E_F(x) - \Delta E_C(x)] \quad (2.5)$$

where $\epsilon(x)$ is the dielectric constant, d is the barrier width, $e\Phi_B$ is the surface state height, E_F

is fermi level and ΔE_C is conduction band offset.

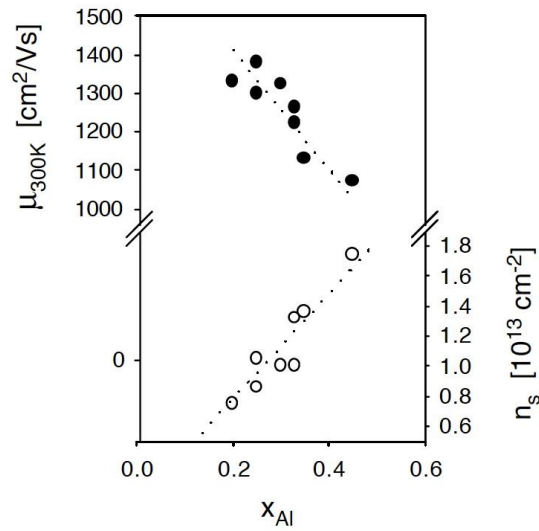


Figure 2.6 Dependence of charge carrier and sheet mobility of AlGaIn/GaN 2DEG¹⁸

As shown in figure 2.6, higher Al content x result in lower sheet mobility. As x_{Al} increases, the interface becomes rougher and it's harder to grow smooth and abrupt heterostructure. Also, the higher strain and higher extent of local strain variation would result in much more structural defects in the near surface region. In very high Al content (beyond 65%), the poor crystal structure and high strain make the sample show no 2DEG behavior.

2.4 2DEG mobility and the influence of high temperature

The scattering mechanism affecting the mobility of GaN/AlGaN heterostructure and the influence of high-temperature annealing is well studied, here we give a summary of it.

The major difference between the carriers in bulk semiconductor and 2DEG is their low-temperature behavior. In doped bulk semiconductors at very low temperature (below 100K), the carriers will “freeze-out” and impurity scattering dominate, causing poor conductivity. For 2DEG, the carrier concentration does not vary significantly with temperature and since carriers are spatially separated with ionized donors, mobility does not show strong temperature dependence.

At higher temperature range 100K to 500K, phonon scattering is the dominant scattering mechanism, with Coulomb scattering and alloy disorder scattering play a non-negligible role to the overall mobility^{10,19–21}. In this temperature range, electron scattering relaxation time is dominated by phonon scattering, which will decrease with increasing T ²². At even higher temperature, strain relaxation and structural defects may cause further degradation of mobility. The overall 2DEG mobility is summarized by Hsu *et al* in figure 2.7.

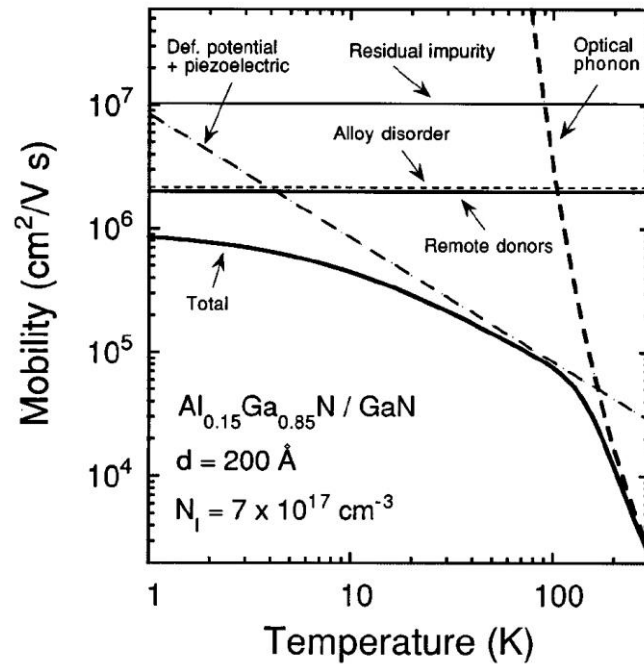


Figure 2.7 Temperature dependence of 2DEG mobility²⁰

3. Experiment Method

3.1 MBE Fundamentals and growth of GaN/AlGaN Heterostructure

Molecular Beam Epitaxy (MBE) is an important growth technique with refined control of growth parameter and in-situ monitoring capabilities. It is the well-developed MBE technology that allows the booming growth of semiconductor devices and electronic industry, such as semiconductor lasers, light emitting diodes (LEDs), photodetectors and high electron mobility transistors (HEMTs). Figure 3.1 shows a typical schematic cross section of MBE system. Solid source materials such as Ga, Al, or Mg, Si for dopant are contained in the effusion cells and will be evaporated after electrical heating. The chamber is pumped into Ultra High Vacuum (UHV) condition and a molecular beam builds from the effusion cell. The molecular beams impinge on the substrate and the epi-layer grows. The beams from different cells can be independently controlled by means of shutters. The single-crystal substrate is usually heated to a certain temperature to allow optimum growth quality. The typical growth rate is in the order of several layers per second. Although higher growth rate can be obtained, lower rate allows surface migration of impinging atoms and smoother surface. Furthermore, since MBE is a UHV based

technique, it is compatible with a wide range of surface analysis techniques, such as reflection high-energy electron diffraction (RHEED), Auger electron spectroscopy and quadrupole mass spectroscopy for gas analysis.

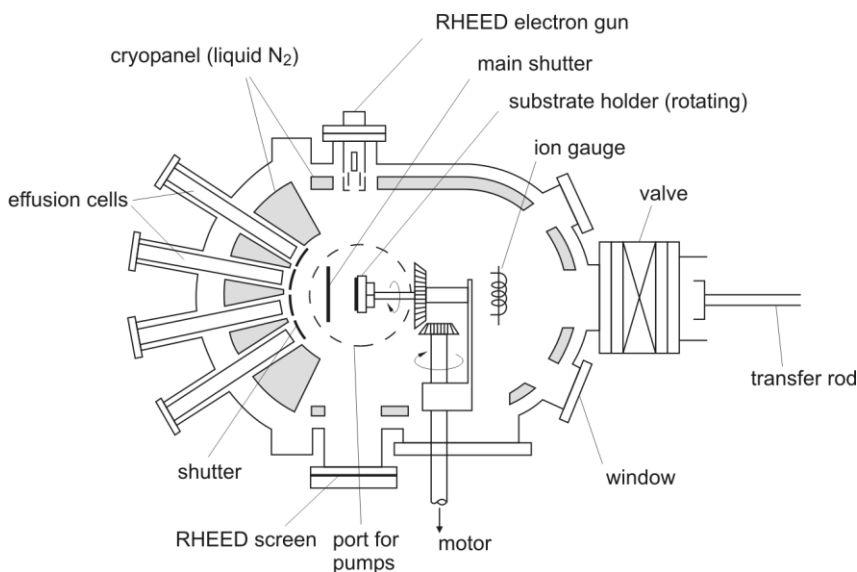


Figure 3.1 Schematic cross section diagram of an MBE system

For nitrides growth, which is somewhat difficult compared with compound III-V semiconductors, the temperature encountered is much higher and the system thus would be more complex. The nitrogen in molecular is inert and is hard to chemically-absorbed on surfaces such as GaN below 950°C due to the very high N-N bonding energy. Atomic nitrogen or molecules with weaker bonds should be provided to get absorbable nitrogen source. To get high quality III-nitride, RF or ECR plasma sources are usually employed to activate the nitrogen species.

The heterostructure used in this experiment were grown on sapphire substrate (0001) using the MBE system at Northrop Grumman Aerospace System (NGAS). 600 angstroms of AlN seed layer is deposited on sapphire substrate, and AlGa_xN/GaN heterostructure is grown on top. The wafer is diced into 8mm x 8mm samples by Disco DAD321 dicing saw at The Center of High Frequency Electronics (CHFE), UCLA.

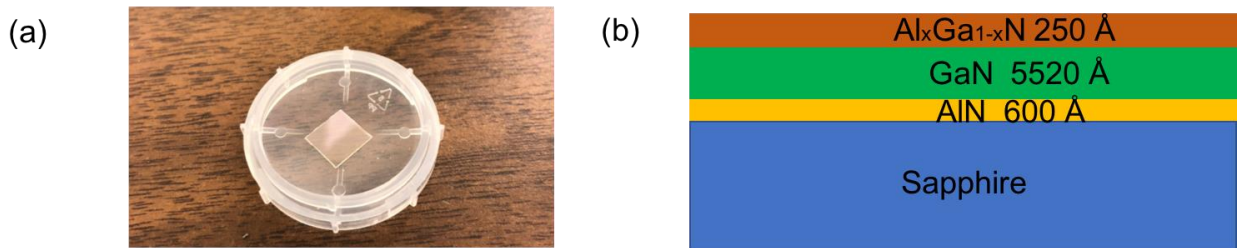


Figure 3.2 (a) 8mmx8mm AlGa_xN/GaN sample (b) AlGa_xN/GaN structure

As grown characterization was done at NGAS, selected results are listed in table 3.1. The sample shows good electrical and structural characteristics.

Table 3.1 Specs Sheet of as-grown AlGa_N/Ga_N sample

Sample ID	Sheet Resistivity	AFM-RMS	Thickness Uniformity
D16016	508.85 (Ω/sq)	0.581	0.5
Xray_GaN-FWHM	Carrier Concentration	Carrier Mobility	Al content in AlGa _N
718(arcsec)	$1.79\text{E}+13(\text{cm}^{-2})$	$737.9(\text{cm}^2/\text{V s})$	0.38

3.2 High-Resolution X-Ray Diffraction (HRXRD)

X-Ray Diffraction is one of the most commonly used methods for obtaining crystal information. X-rays are produced upon decelerate of electrically charged particles, usually electrons, with sufficient kinetic energy. A continuous spectrum is resulted from the electron deceleration, with sharp peaks in some specific wavelength. The sharp peaks, unlike the continuous spectrum, can only exist when the voltage applied to an x-ray tube is above a certain value. This is because when the electrons have enough kinetic energy it can knock out the inner shell electrons in the target metal, the vacancy is then filled with outer shell electrons and emit a characteristic x-ray. Since K shell vacancies is more likely to be filled by L shell electrons, intensity of K_{α} line is

usually higher. Also, the K_{α} lines have shorter wavelength than L or M lines do, it's less likely to be absorbed and is most suitable for x-ray diffraction. To get monochromatic convergent K_{α} x-ray beam, crystal filters and monochromator is needed. These are systems with well-designed crystals that can absorb x-rays with undesired wavelength and redirect divergent x-rays to be convergent. The x-ray going out from the tube can be considered highly monochromatic and convergent.

$$n\lambda = 2d \sin \theta \quad (3.1)$$

At certain angle, when x-rays satisfy the Bragg condition (equation 3.1, where λ is the wavelength of x-ray, d is the interplanar spacing and θ is Bragg angle), the diffracted x-rays will constructively interfere each other and result in peaks.

For hexagonal system, the interplanar distance d can be calculated by a/c ratio as:

$$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad (3.2)$$

where h, k, l are Millar indices of a crystallographic plane.

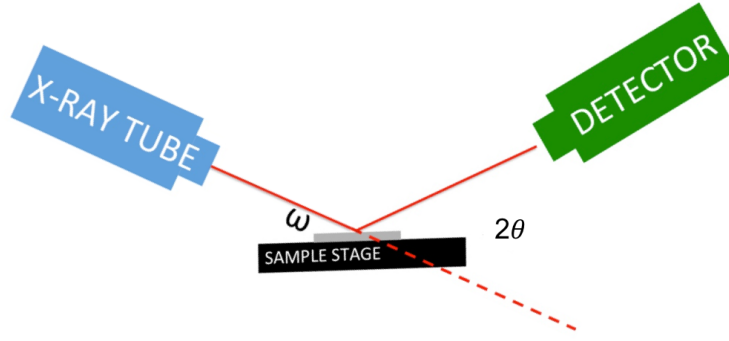


Figure 3.3 Schematic diagram of XRD system setup

The XRD system setup is shown in figure 3.3, ω is the angle between sample and incident x-ray and 2θ is the angle between incident beam and detector. In a symmetric scan, 2θ is twice of ω . In powder or polycrystalline samples, we can get all the diffraction peaks in symmetric ω - 2θ scan. In the case of single crystal layers, like the AlGaIn/GaN epi-layers, we can only get diffraction peaks from planes normal to the zone axis. Other crystallographic planes, however, can be observed by asymmetric scans, glancing incidence (G.I.) and glancing exit (G.E.). In asymmetric scans the ω is not equal to θ but deviate by an angle φ

$$\omega = \begin{cases} \theta - \varphi, & \text{for G.I.} \\ \theta + \varphi, & \text{for G.E.} \end{cases} \quad (3.3)$$

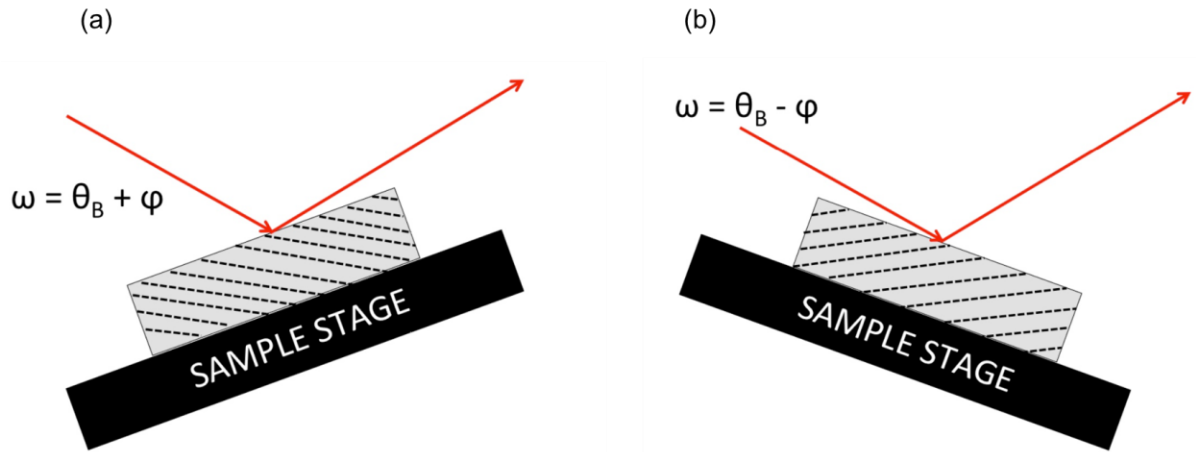


Figure 3.4 Schematic diagram of (a) glancing exit scan and (b) glancing incident scan. The dashed line represents the crystallographic plans that not perpendicular to the surface normal.

One thing worth noted is that in glancing incident condition, due to the wide exiting angle the resolution is worse than that of glancing exit. For this reason, G.E. is more often used for asymmetric reciprocal space map.

Basically, coupled ω - 2θ scan can give us crystallographic information in vertical direction to the reciprocal point, as shown in figure 3.5, while ω scans give rise to reciprocal point in tangent direction to the zone axis. In order to mapping out the reciprocal space, we can use looped scans, in which ω - 2θ scan is measured while looping with different ω angel.

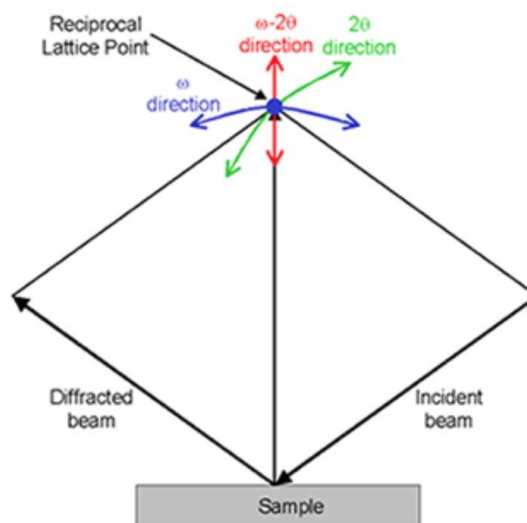


Figure 3.5 Schematic diagram of different scan axis in reciprocal space

In this work, **Bede D1 High-Resolution Diffractometer** with a copper x-ray ($\text{Cu K}\alpha$, $\lambda=1.54\text{\AA}$) source tube is used. This system is capable of both double axis diffraction (DAD) and triple axis diffraction (TAD). Schematic diagram is shown in figure 3.6. The axis 1 consists of a 3-bounce channel cut (111) silicon monochromator and serves as the incident beam conditioner. Axis 2 is simply the sample stage. If we turn on the third axis, the system switched from DAD to TAD. Axis 3 is the analyzer crystal which consists of a 4 bouncing (220) channel cut silicon. In the triple axis condition, only x-rays whose wavelengths are very close to the $\text{Cu K}\alpha$ line ($\lambda=1.54\text{\AA}$) can be selected to enter the detector. Thus, the resolution can be improved significantly, while losing some intensity. In fact, all the x-rays we use are to some extent non-monochromatic, so sometimes it's

hard to distinguish whether the crystal defect or the multi-wavelength x-ray source is the cause of a peak widening. So triple axis HR-XRD is necessary in some circumstances.

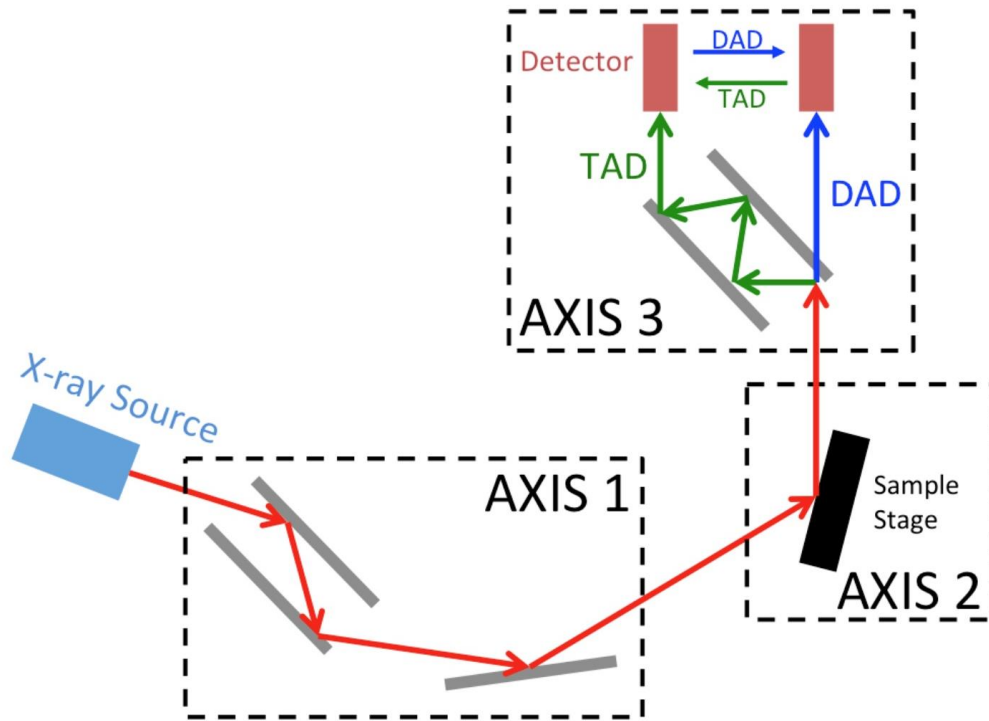


Figure 3.6 Schematic diagram of the BEDE B1 high-resolution x-ray diffractometer

In this work, TAD is used for symmetric coupled ω - 2θ scan and symmetric sapphire (0006) reciprocal space map, DAD is used for asymmetric sapphire (11 $\bar{2}$ 9) reciprocal space map.

3.3 Raman Spectroscopy

Raman spectroscopy is used to probe phonon vibration modes of a material. A

monochromatic laser source hits the sample surface and the incoming photons will interact with the phonon vibration modes within the lattice. Those interactions will cause either energy loss or gain for the photons, which we called Stokes and anti-Stokes shift respectively as shown in figure 3.7. After the interaction the photons will return to the aperture where they came and deflected to a CCD detector. Such detectors will detect photons with energy close to the Rayleigh line and give us Raman signals. Since the vibrational modes solely depend on the bonded atomic species and bonding strength, it can be the fingerprint of a specific material. Furthermore, the vibration modes within an alloy would gradually change with different alloy content, it is a desired way to study alloy composition and related properties.

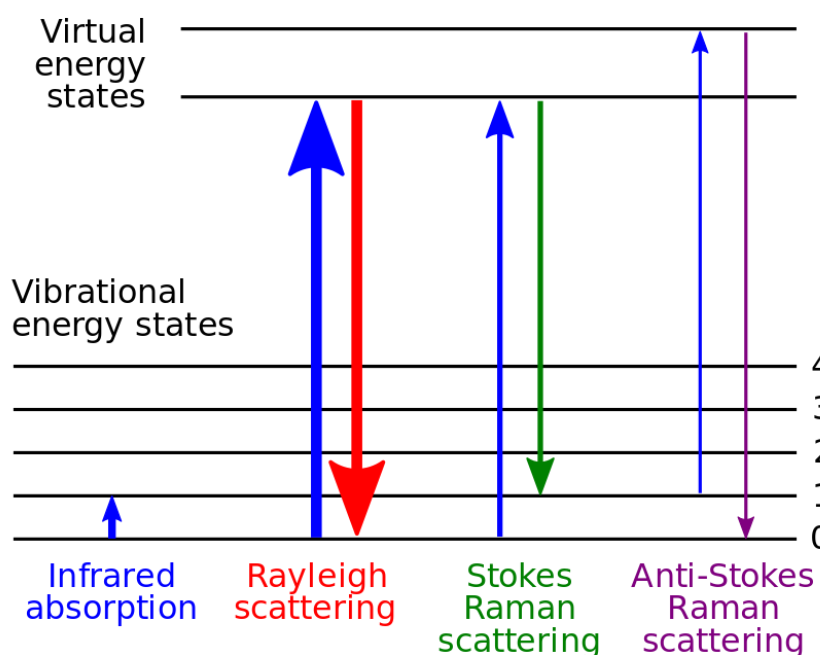


Figure 3.7 Illustration of Stokes and Anti-Stokes Raman scattering.

In this work, **Renishaw inVia Raman Microscope** is used for Raman spectroscopy. The laser wavelength is 633nm. 5X, 20X and 50X objective lens with low chromatic distortion was employed to focus and collect the laser light. A silicon sample was used to calibrate the relative peak position.

3.4 Heat Treatment

In this work, **Lindberg/Blue M™ 1700°C** Tube Furnaces were used for N₂ heat treatment and **Barnstead Thermolyne FB1415M** Muffle Furnace used for air. For the N₂ annealing,

stagnant atmosphere was first adopted, and it turns out to show leakage problem, in later experiments it was changed to flowing N₂ System. The ramp rate is set to 3 degrees/min and 3 hours hold at target temperature. For air annealing, there is no ramp rate control, so the ramp up rate is at its maximum (which is not specified) and the hold time is also 3 hours.

4. Results and Discussion

4.1 As grown wafer characterization

The AlGa_N/Ga_N wafer grown at NGAS is characterized by x-ray diffraction without any treatment. Figure 4.1 shows asymmetric reciprocal space map of (10 $\bar{1}$ 3) Ga_N and AlGa_N peaks. Symmetric (0002) coupled ω -2 θ scan of the sample is shown in figure 4.2 and compared with simulated result from Rocking Curve Analysis by Dynamical Simulation (RADS).

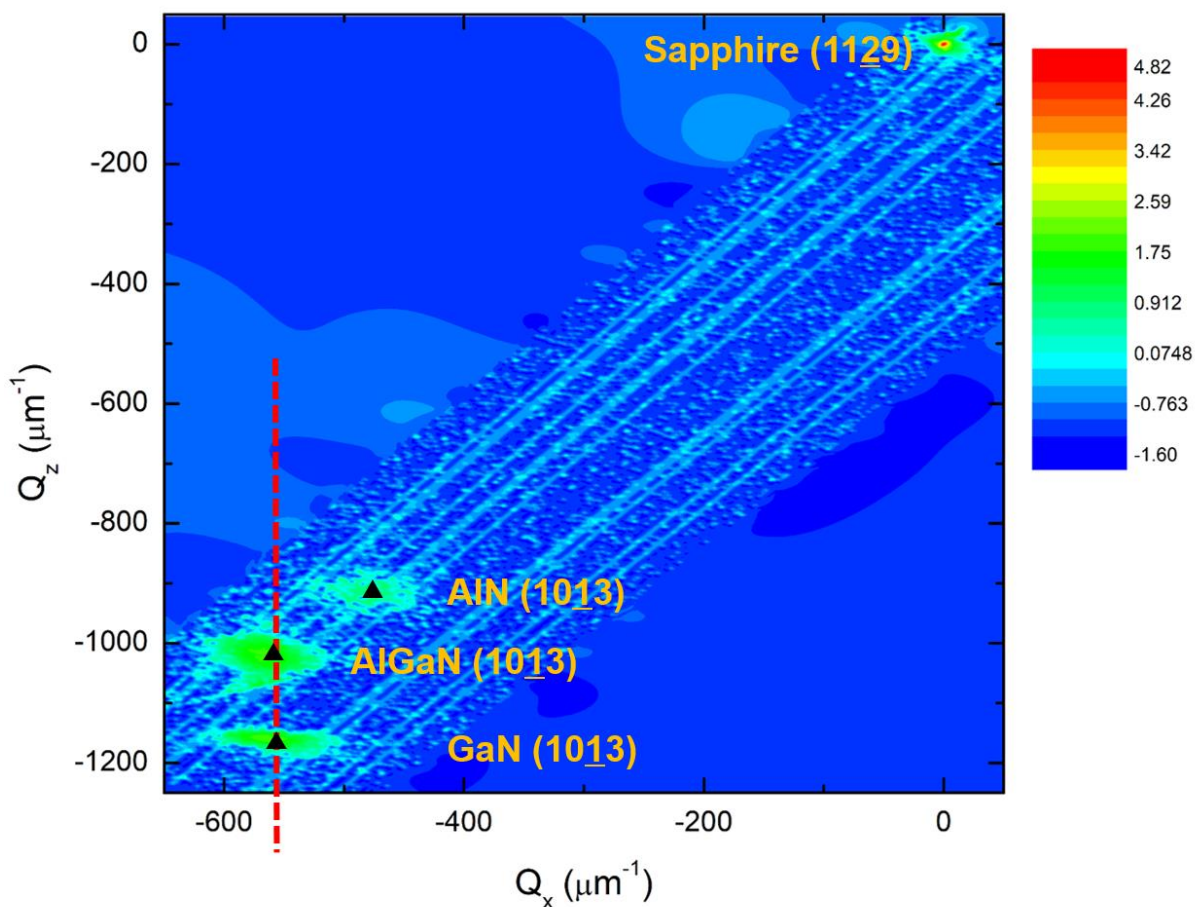


Figure 4.1 Reciprocal space map of (10 $\bar{1}$ 3) Ga_N, (10 $\bar{1}$ 3) AlGa_N and (11 $\bar{2}$ 9) sapphire peaks for unannealed sample.

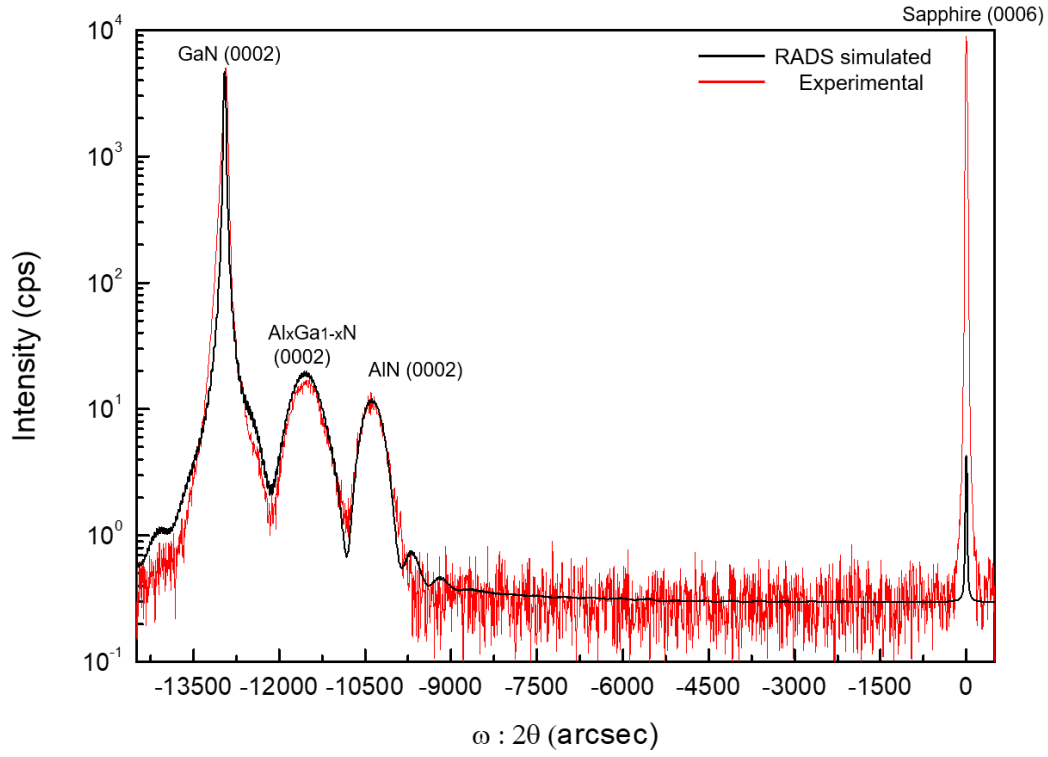


Figure 4.2 Symmetric (0002) coupled ω -2 θ scan versus RADS simulation result of untreated sample.

The stress and strain in the AlGaIn/GaN heterostructure is crucial for the HEMT performance since it's directly connected with the polarization. The stress and strain in the epilayer usually calculated as relaxation, which is defined as:

$$R = \frac{a_{\parallel} - a_s}{a_{x,R} - a_s} \quad (4.1)$$

Where R is relaxation, $a_{\parallel}$ is the in-plane lattice parameter of strained lattice, a_s is the relaxed substrate lattice parameter and $a_{x,R}$ is the in-plane intrinsic lattice parameter of the epilayer. In reciprocal space map, the amount of relaxation of AlGaIn epilayer can be determined as follow.

First, the reciprocal points of GaN (10 $\bar{1}$ 3) and AlGa $\bar{N}$ (10 $\bar{1}$ 3) is chosen as the highest intensity point in figure 4.1. The angel between the vectors that joint the origin and GaN, AlGa $\bar{N}$ point is calculated and compared with totally relaxed angel. The quotient between these two angles gives us the relaxation of the AlGa $\bar{N}$ to the GaN layer. Note that the true origin of the reciprocal point is not what shown in the picture but is calculated using the following expression²³

$$Q_x = \lambda^{-1}[\cos(\omega - \Theta) - \cos \omega]$$

$$Q_z = \lambda^{-1}[\sin \omega - \sin(\omega - \Theta)] \quad (4.2)$$

Where ω and θ is determined by a specific diffraction peak, in this case: Sapphire (11 $\bar{2}$ 9). As shown in figure 4.1, the AlGa $\bar{N}$ peak and GaN peak is almost lined up vertically, meaning a pseudomorphic layer, relaxation for all the layers are listed in table 4.1. The relaxation amount is then used for the RADs fitting and the results show good match. The pseudomorphic structure is beneficial for piezoelectric polarization which can be confirmed by the good electrical properties shown in table 3.1.

Table 4.1 Calculated relaxation of each layer

	Relaxation
$\text{Al}_{0.38}\text{Ga}_{0.62}\text{N}$	0%
GaN	81%
AlN	100%

4.2 HRXRD Results

To get high-temperature stability of AlGaN/GaN heterostructure, heat treatments under different temperature and different atmosphere were done.

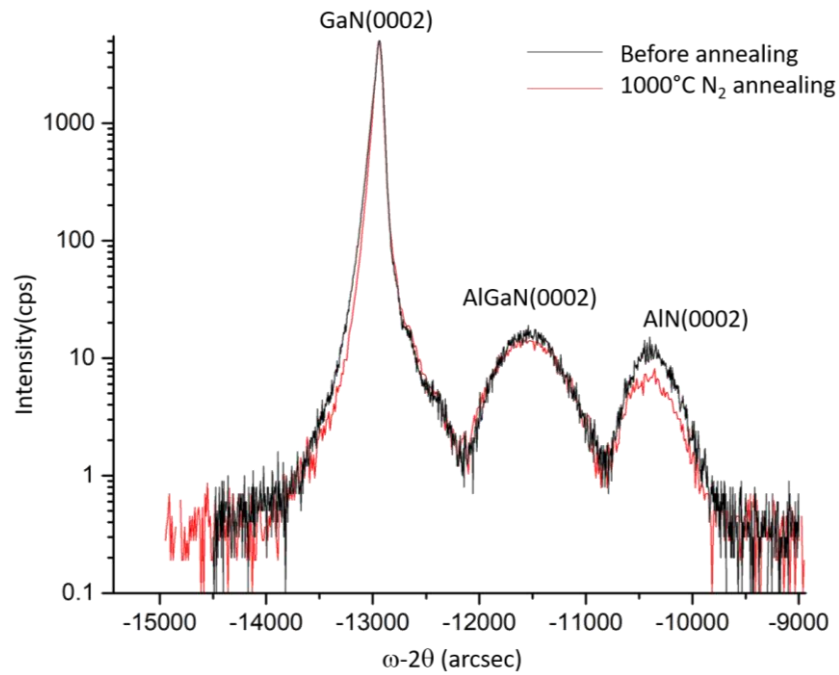


Figure 4.3 Coupled ω - 2θ scan profiles of 1000°C nitrogen annealing and before annealing sample.

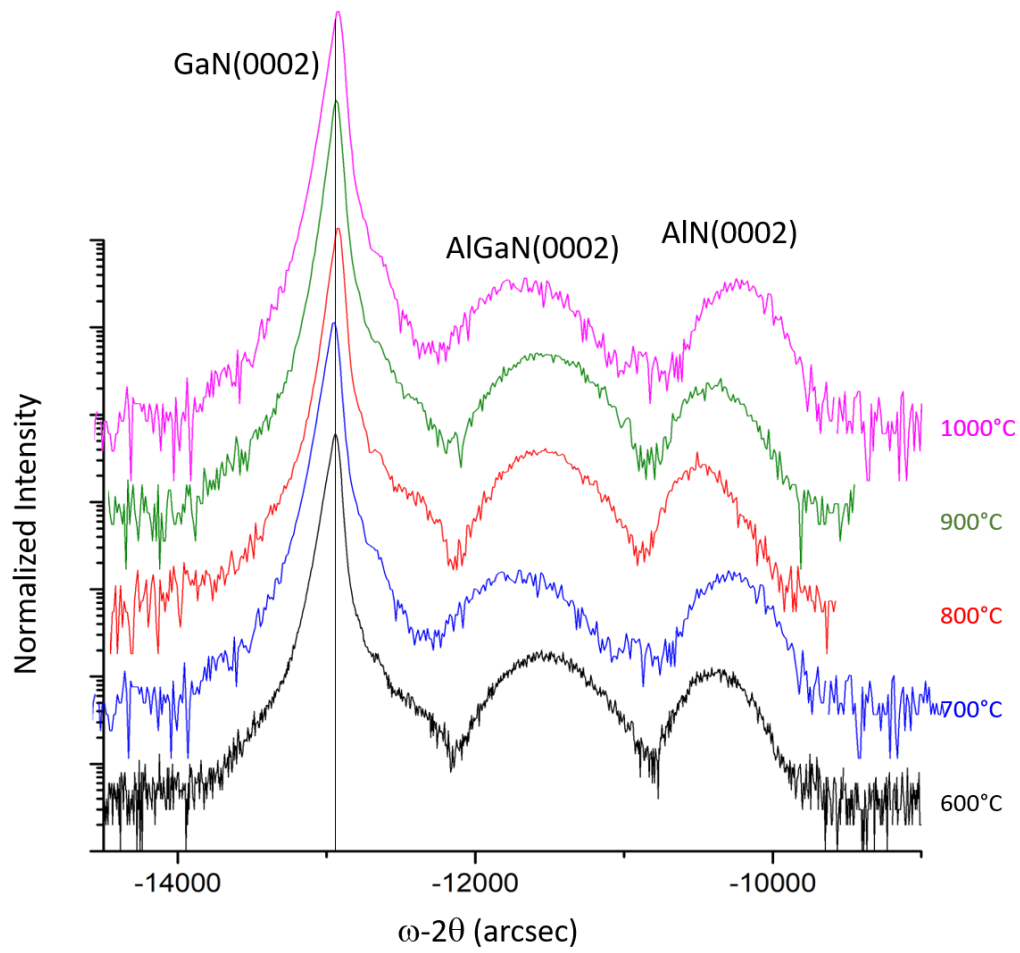


Figure 4.4 Stacked coupled ω - 2θ scan profiles for nitrogen annealing samples under different temperature.

For nitrogen annealed samples, the AlGaN/GaN samples remain good crystallinity up to 1000°C, as can be seen in figure 4.3. The Bragg angle change of GaN and AlGaN (0002) peaks is less than 30 arcsec and the result lattice parameter change is in the order of $10^{-3}\text{\AA}$.

For those annealed in air, degradation of AlGaN can be observed as shown in figure 4.6

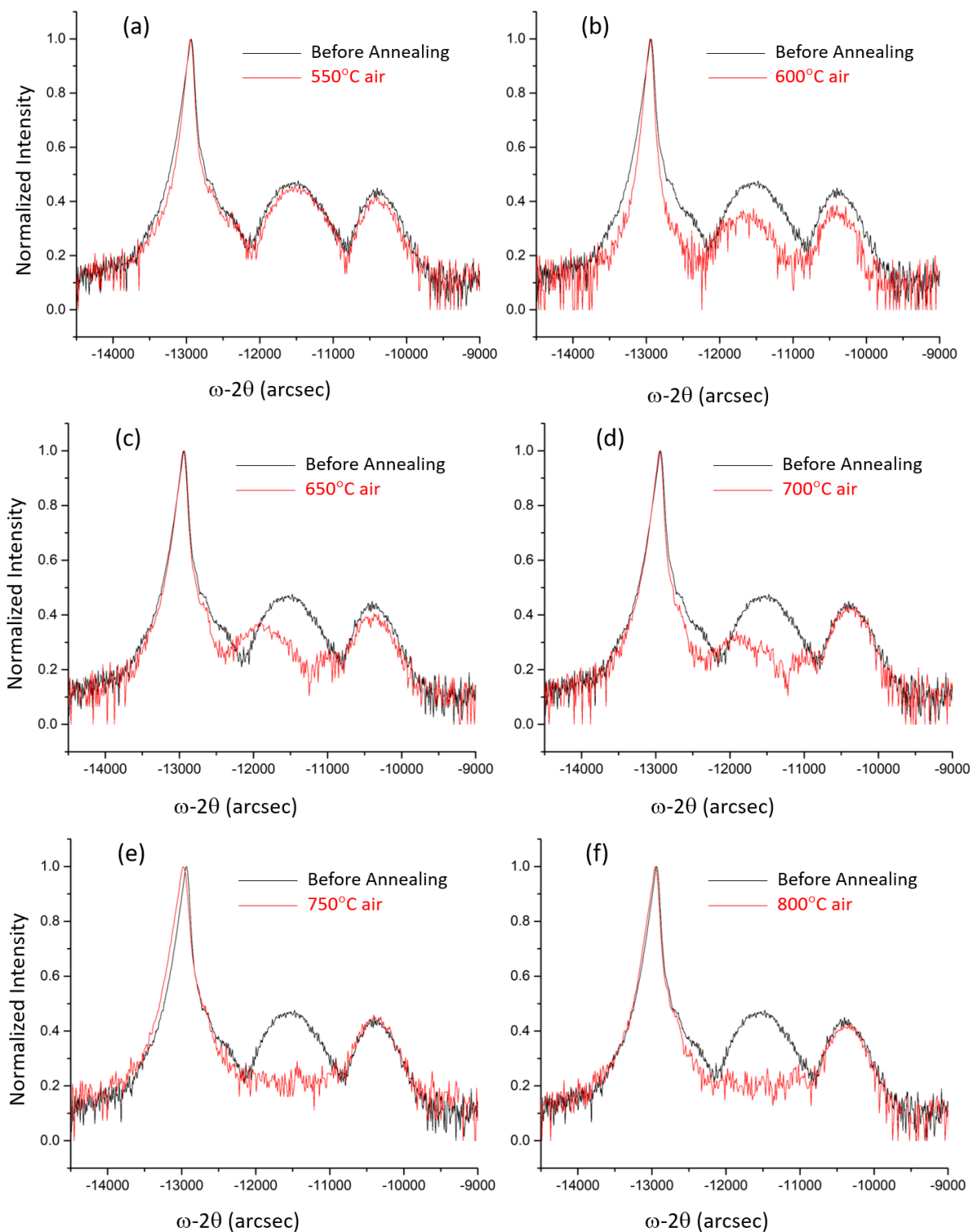


Figure 4.6 ω - 2θ profile of unannealed sample compared with air annealed sample in different temperature:(a)550°C
(b) 600°C (c) 650°C (d) 700°C (e) 800°C

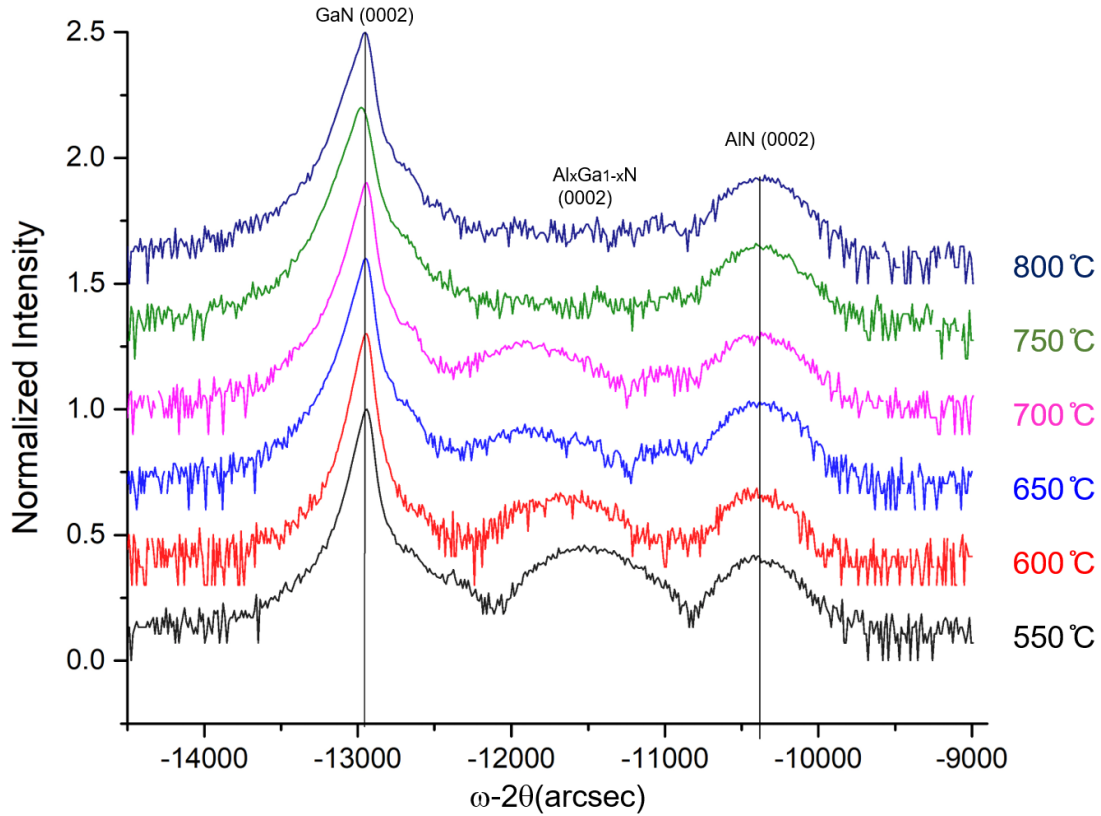


Figure 4.7 Stacked ω -2 θ profile of air annealing samples.

and 4.7. Starting at 600°C, the Bragg angle of AlGa_N (0002) peak shifts to smaller value while that of the GaN (0002) peak remain unchanged, the AlGa_N structure was totally degraded at 800°C. In partially degraded condition, such as 600°C, irreversible lattice relaxation occurred as indicated by the AlGa_N peak shift and the amount of relaxation increases as the temperature goes up before fully degraded. Follow the same procedure in section 4.1, RSM and RADs fitting is done to get the relaxation of 600°C air annealed sample and results are shown in figure 4.8 and 4.9, calculated relaxation is listed in table 4.2.

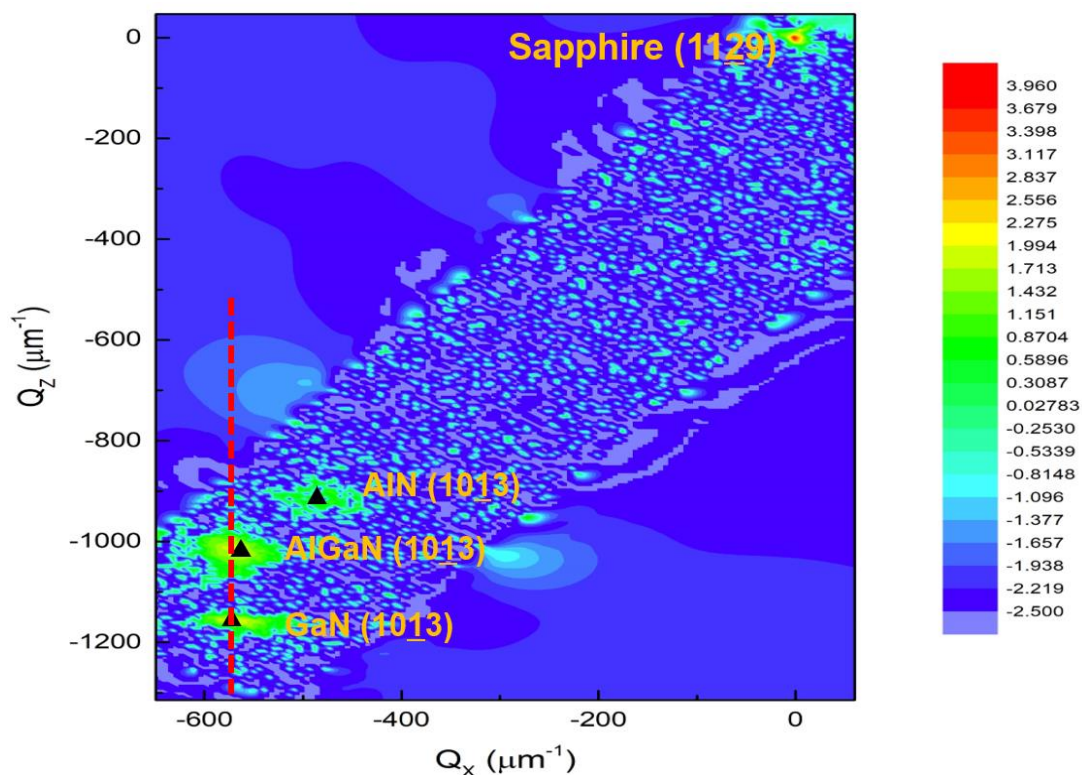


Figure 4.8 Reciprocal space map of (1013) GaN, (1013) AlGaIn and (1129) sapphire peaks for 600°C air annealed sample.

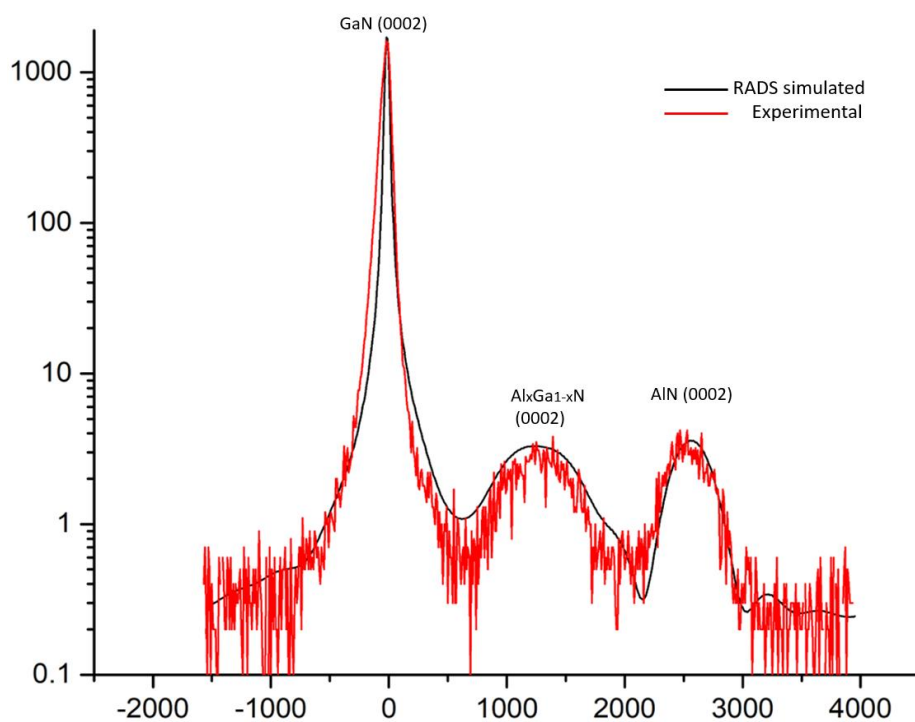


Figure 4.9 Symmetric (0002) coupled ω - 2θ scan versus RADS simulation result for 600°C air annealed sample.

Table 4.2 Calculated relaxation for 600°C air annealed sample

	Relaxation
$\text{Al}_{0.38}\text{Ga}_{0.62}\text{N}$	62.75%
GaN	83%
AlN	100%

The comparison of N_2 and air annealed sample indicates that it was the introduction of oxygen that creates massive defects and dislocations. And the high-temperature irreversible lattice relaxation can be attributed to the propagation of cracks and thermally activated dislocation motion. Such amount of lattice relaxation will decrease polarization and lead to lower 2DEG concentration^{24–26}. Rocking curves (omega scan) were measured to get information about crystal quality and dislocation density. Figure 4.10 shows the FWHM and FW0.01M of the samples. For nitrogen annealed samples, both GaN and AlGaN peak width decrease, indicating better crystal quality. The drop of FWHM and FW0.01M for GaN annealed in N_2 from room temperature to 1000°C is 38.9% and 38.1%, for AlGaN it is 32% and 21.2%. This means that

annealing in N_2 will lead to recrystallization of GaN/AlGaN heterostructure.

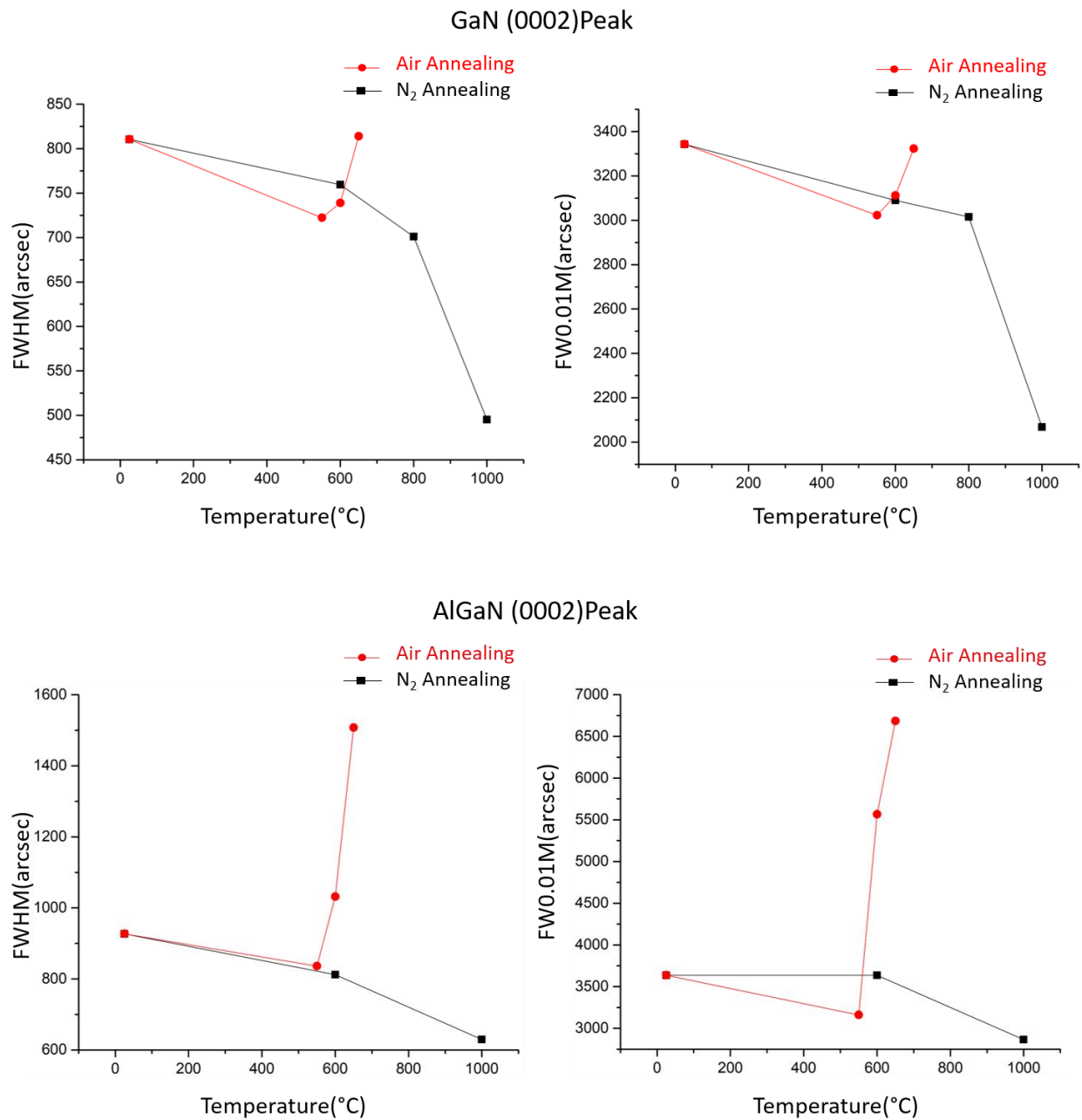


Figure 4.10 FWHM and FW0.01M of ω scan versus temperature

For those annealed in air, the widths first decrease then increase at higher temperature.

Oxygen is no doubt one of the major cause of such degradation. Under high temperature the oxygen molecules incorporated into the AlGa_{0.38}N layer and form compound with Al and N. This would in turn lead to interface degradation and affect the quality of GaN layer. The inflection points in figure 4.10 can be related to the Debye Temperature (831K for Al_{0.38}Ga_{0.62}N) as proposed by many authors^{27,28}. Above the Debye Temperature, lattice vibrations are so strong that either recrystallization or propagation of cracks would be greatly enhanced.

If we assume that the diffusivity D is inverse proportional to the Rocking curve intensity or the area covered by the AlGa_{0.38}N peak and plot it versus inverse temperature, we can get straight line as shown in figure 4.10 and 4.11, which can be fit into the following equation:

$$\ln(D) = -\frac{E_A}{RT} + \ln(D_0) \quad (4.3)$$

where R is ideal gas constant, D_0 is pre-exponential factor (constant) and E_A is activation energy.

The calculated activation energy is 0.848eV for areal assumption and 0.828eV for intensity assumption. This means that interstitial diffusion could be the main mechanism for oxygen molecules to diffuse into the AlGa_{0.38}N layer under high temperature.

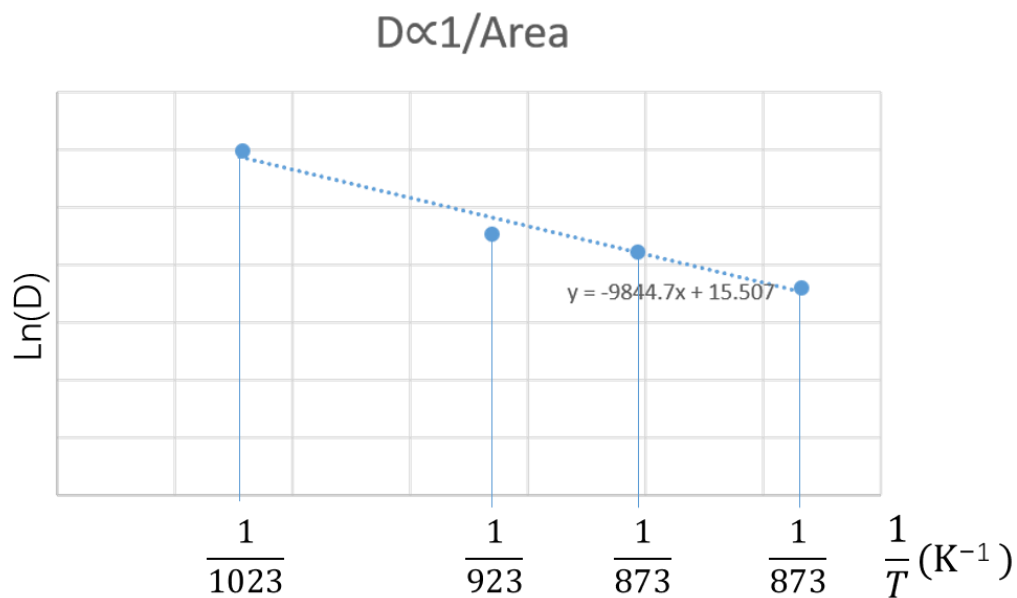


Figure 4.10 Plot of $\text{Ln}(D)$ versus inverse temperature, assuming D is inverse proportional to XRD peak area.

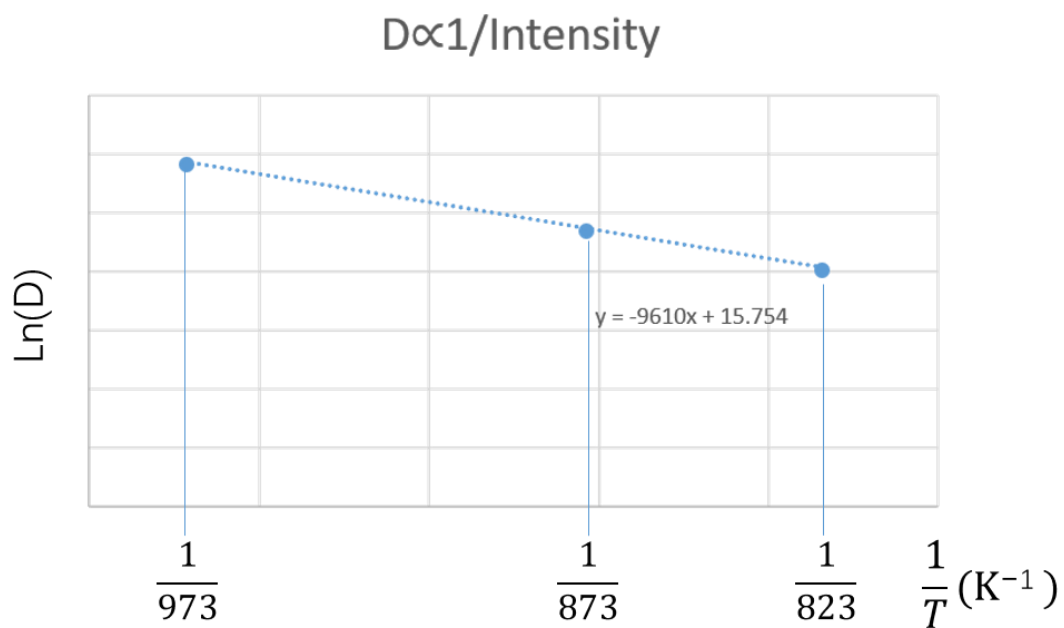


Figure 4.11 Plot of $\text{Ln}(D)$ versus inverse temperature, assuming D is inverse proportional to XRD intensity.

4.3 Raman Spectroscopy

In this work, Raman spectra was collected using $Z(X, \cdot)\bar{Z}$ backscattering geometry. The

allowed phonon modes in this configuration is A₁ (LO) and E₂ for wurtzite GaN and AlGaN.

Similar to the XRD results, the Raman spectra of the Al_{0.38}Ga_{0.62}N sample is unaffected up to 1000°C annealing in N₂, as shown in figure 4.12.

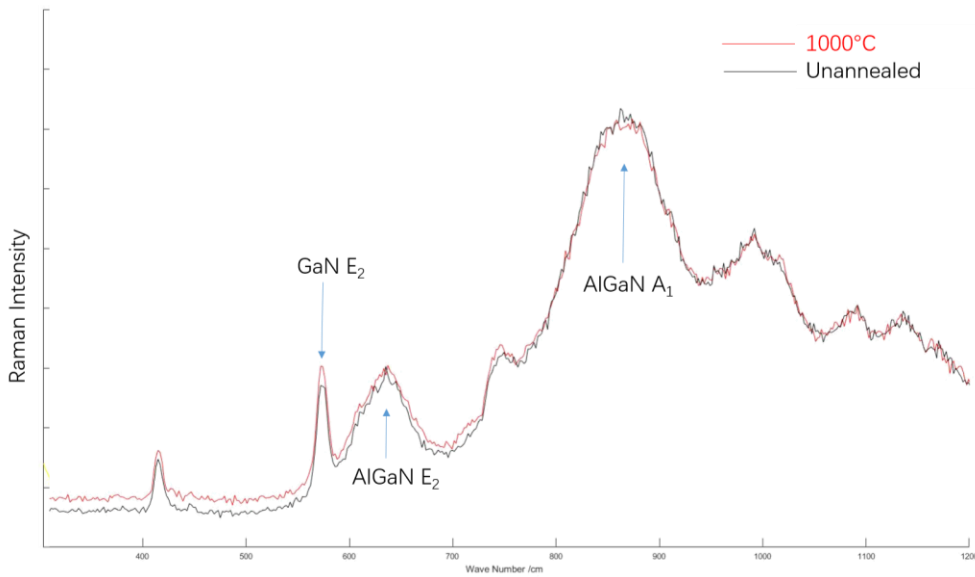


Figure 4.12 Raman spectra of N₂ annealed and unannealed sample.

For the air annealed samples, broadening happened to the AlGa_xN A₁ peak starting from 550°C. Up to 800°C, two separated peaks can be distinguished from simulated peak split as shown in figure 4.14, the newly emerging peaks are 843 cm⁻¹ and 881 cm⁻¹. This phenomenon is observed in many other studies²⁵ and can be used to determine the pathway of AlGa_xN degradation. Referring to the Raman spectra data as function of Al content²⁹, the two peaks correspond to Al_{0.32}Ga_{0.68}N

and $\text{Al}_{0.9}\text{Ga}_{0.1}\text{N}$ for 843cm^{-1} and 881cm^{-1} respectively. Thus, besides oxidization, AlGa_{0.9}N will

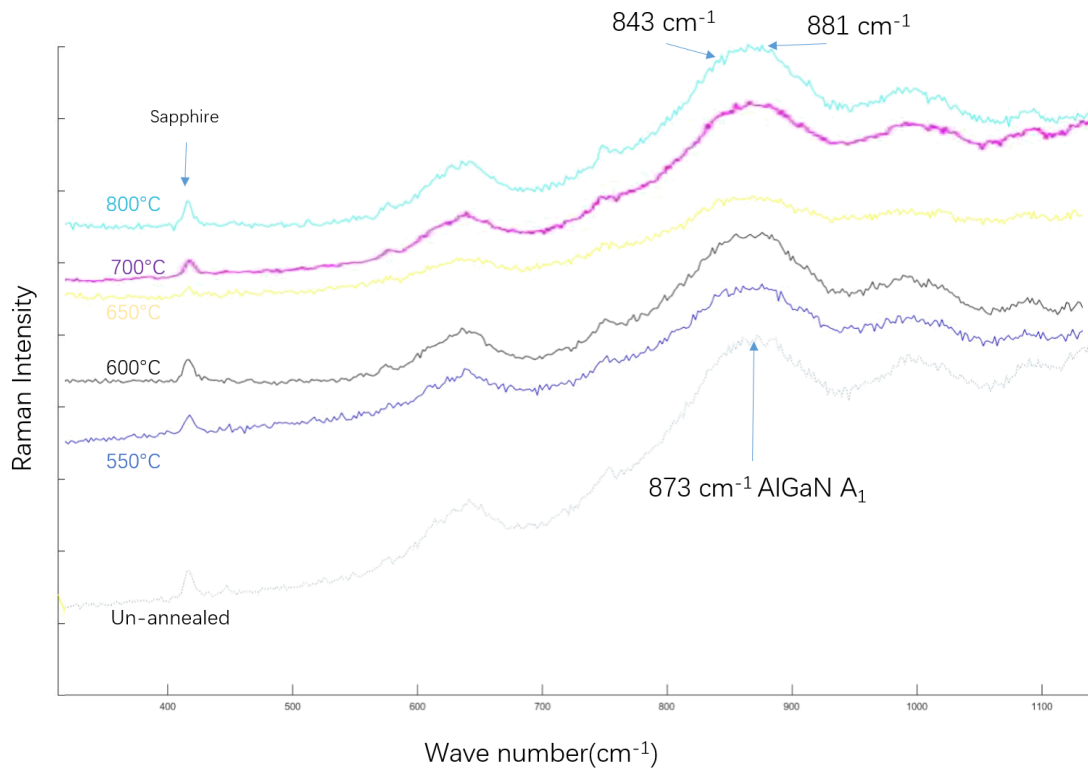


Figure 4.13 Stacked Raman spectra of air annealed sample at various temperature.

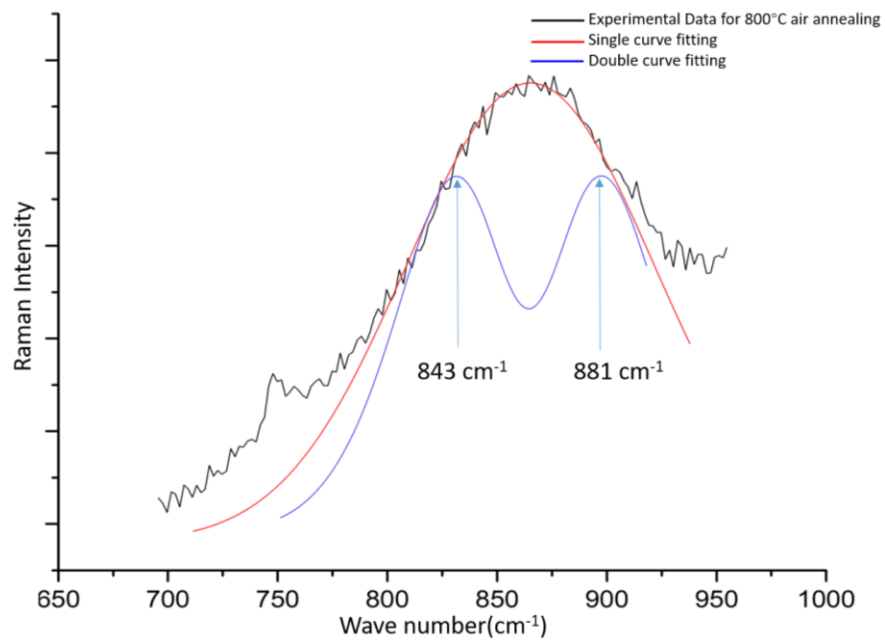


Figure 4.14 Raman spectra curve fitting of 800°C air annealing sample.

undergo a phase separation and emerging into a high Al content phase and a low Al content phase under very high-temperature condition. Since the crystallinity of the emerging high Al phase is so poor, very weak signals can be observed in figure 4.6 and 4.7. The high Al phase can also act as a cap layer protecting the GaN layer underneath.

5. Conclusions and future work

AlGaIn/GaN HEMT heterostructures are characterized after various temperature annealing in nitrogen and air atmosphere. In N₂ atmosphere study, the crystal structure of both GaN and AlGaIn layers are unaffected up to 1000°C, proving good thermal stability. The FWHM and $\Delta 2\theta$ of N₂ annealed samples decreased at higher temperature, indicating better crystallinity and recrystallization happened. When annealed in air, major degradation occurred to the AlGaIn layer due to the oxidation and defects created during oxidation process. Irreversible lattice relaxation occurred. Above Debye temperature the propagation of dislocations severely destroys the crystal structure. The diffusion energy of oxygen is presumed to be 0.838eV with interstitial diffusion mechanism. Raman spectra indicate that there exists two emerging AlGaIn phase under high-temperature annealing, one being very high Al content (>90%) and the other with lower Al content. The exact phase separation mechanism remains unclear.

Further study can be done using different characterization tools to explore the diffusion of oxygen and AlGaIn phase separation. Depth sputtering XPS can be utilized to study O content at different depth in the AlGaIn layer. Cross-section TEM is capable of monitoring crystal

morphology and interface quality of the heterostructure. The effect of oxygen and AlGaN phase separation on GaN HEMT devices needs to be specified.

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