THE EFFECTS OF ANNEALING ON MICROSTRUCTURAL CHANGES FOR ALUMINIUM NITRIDE EPITAXIAL GROWN ON SAPPHIRE BY TRANSMISSION ELECTRON MICROSCOPY

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DEDICATION

To my lovely mother, who gave me endless love, trust, constant encouragement over the years, and for her prayers

To my spouse, for being very understanding and supportive in keeping me going, enduring the ups and downs during the completion of this thesis.

To my family, for their patience, support, love and prayers

This thesis is dedicated to them.

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ABSTRACT

The performance of semiconductor devices depends strongly upon the microstructure of the materials. Therefore the microstructural control is intrinsically important for fabrication of high performance devices. In this research, the microstructures have been analysed in detail and the mechanisms of microstructural changes in aluminium nitride (AlN) epitaxial have been clarified for the establishment of the growth method. AlN heteroepitaxial layers were made by growing an AlN buffer layer on a (0001) sapphire substrate by the Metal Organic Vapor Phase Epitaxy (MOVPE) growth process. Annealing treatments were added before and after the deposition of an AlN buffer layer. The surface roughness of AlN was observed with an Atomic Force Microscope (AFM) and X-ray Rocking Curve (XRC). The cross section of AlN heteroepitaxial was observed by using Transmission Electron Microscope (TEM) at 200 Kv and High-Angle Annular Dark-Field (HAADF) images were observed with a Scanning Transmission Electron Microscope (STEM) at 300 kV. Thin foil specimens or lamella for the TEM observation were made using a Focused Ion Beam (FIB) mill with accelerating voltage of 15 kV~3 kV for a smooth finishing of lamella. Prior to the deposition of a medium temperature MT-AlN layer, the sapphire substrate was cleaned or pre-annealed at a high temperature, T_{An} under the atmosphere of H_2. For annealing temperature, T_{An} less than 1250°C , the crystallinity improved but twisting domains appeared above the temperature. Threading dislocations (TDs) of type c and type-a+c with 10^8 cm^{-2} dislocation density was observed. However when the temperature was increased to 1350°C, threading dislocation were reduced. On the other hand, post deposition annealing at a high temperature between 1500ºC and 1700°C for 2 hours under the atmosphere of N_2+CO was carried out. Cross sectional TEM revealed that after annealing at 1500°C, cone-shaped domains and threading dislocations remained. The morphology of domains and the changes in TEM image contrast strongly suggest that the domains are inversion domains. TDs of type-a and type- a+c were visible for g =01-10 under the two beam condition. However, after annealing at 1550°C, the cone shaped domains coalesced with each other to leave a single domain boundary running in a zigzag laterally at the center of AlN buffer layer that the upper layer has the Al- polarity while the lower layer has the N-polarity determined by the HAADF analysis. The inversion domain boundary become smooth and flatter with the rising annealing temperature. The surface of MT-AlN buffer was finely rugged before the annealing, but became coarser and smoother with annealing. The changes in the surface morphology indicates the occurrence of grain coalescence. The density of TDs was reduced to roughly 5×10^8 cm^{-2} after annealing at 1650°C. Conclusively, this research confirms that pre-deposition and post deposition annealing are an effective treatment to control the microstructure and to reduce the dislocation density for advancement of semiconductor devices.

ABSTRAK

Prestasi peranti semikonduktor bergantung kuat kepada mikrostruktur bahan. Oleh itu kawalan mikrostruktur untuk mengurangkan ketumpatan kecacatan penting untuk fabrikasi peranti berprestasi tinggi. Dalam kajian ini, perubahan dan mekanisme mikrostruktur telah dianalisis secara terperinci. Lapisan nipis AlN telah di mendap atas substrat (0001) nilam dengan kaedah *metal organic vapor phase epitaxy (MOVPE)*, di mana proses pertumbuhan dalam cara yang agak konvensional, tetapi rawatan penyepuhlindapan ditambah sebelum dan selepas pemendapan. Kekasaran permukaan lapisan AlN telah diperhatikan dengan *atomic force microscope (AFM).*Lapisan AlN diperhatikan dengan menggunakan mikroskop elektron penghantaran konvensional (CTEM) pada 200kV. Tinggi sudut anulus gelap-bidang (HAADF) imej imbasan-TEM juga diperhatikan dengan *scanning transmission electron microscope (STEM)* pada 300 kV. Penipisan spesimen untuk pemerhatian TEM telah dibuat menggunakan *focus ion beam (FIB)* dengan voltan dari 15kV ~ 3 kV. Sebelum pemendapan lapisan MT-AlN, substrat nilam itu disepuh lindap pada suhu yang tinggi dalam gas hydrogen, H_2. Pada suhu penyepuhlindapan, T_{an} <1250° C, penghabluran bertambah baik tetapi domain berpusing muncul.Kecacatan bebenang jenis c, jenis (a + c) dan berpusing domain dengan 200-500 nm diameter sepanjang [2-1-10], dengan kepadatan 10^8 cm^{-2} telah diperhatikan. Walau bagaimanapun apabila suhu telah meningkat kepada 1350°C kecacatan bebenang semakin berkurang justeru telah meningkatkan lekatan antara nilam dan AlN. Di samping itu, pemendapan penyepuhlindapan telah di jalankan pada suhu yang tinggi antara 1500°C untuk 1700°C untuk 2 jam di bawah suasana N_2 + CO. Keratan rentas TEM telah mendedahkan bahawa selepas penyepuhlindapan pada suhu 1500°C, domain berbentuk kon dan kecacatan bebenang kekal. Morfologi dan keadaan pembelauan untuk kontras imej sangat menyarankan bahawa domain adalah domain penyongsangan. Kecacatan bebenag jenis-a dan jenis-a + c dapat di lihat untuk g = 01-10 di bawah keadaan dua rasuk. Walau bagaimanapun, selepas penyepuhlindapan pada 1550°C, domain penyongsangan bersatu antara satu sama lain untuk meninggalkan sempadan domain berjalan secara zigzag di tengah-tengah lapisan AlN. Lapisan atas mempunyai kutub-Al dan lapisan yang lebih rendah mempunyai kutub-N. Sempadan domain penyongsangan menjadi semakin licin dengan suhu penyepuhlindapan yang semakin meningkat. Permukaan MT-AlN penampan telah halus lasak sebelum penyepuhlindapan, tetapi menjadi lebih licin dengan penyepuhlindapan. Perubahan dalam morfologi permukaan menunjukkan berlakunya geseran sempadan domain. Ketumpatan TDS telah dikurangkan kepada kira -kira 5 × 10^8 cm^{-2} selepas penyepuhlindapan pada suhu 1650°C. Kesimpulan daripada penyelidikan ini telah sahkan bahawa pra pemendapan dan selepas pemendapan penyepuhlindapan telah menjadi rawatan yang amat berkesan bagi mengawal struktur mikro dan mengurangkan ketumpatan kecacatan bebenang untuk kemajuan dalam fabrikasi peranti semikonduktor berkualiti tinggi

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LIST OF ABBREVIATIONS

ALD	-	Atomic Layer Deposition
BSE	-	Backscatter Electron
BF	-	Bright Field
CBED	-	Convergent Beam Electron Diffraction
DF	-	Dark Field
EBSD	-	Electron Back Scatter Diffraction
ELO	-	Epitaxial Lateral Overgrowth
FIB	-	Focused Ion Beam
FWHM	-	Full Width Height Maximum
HAADF	-	High Angle Annular Dark Field Image
HCP	-	Hexagonal Close Packet
HT	-	High temperature
HVPE	-	Hydride Vapor Phase Epitaxy
ID	-	Inversion Domain
LED	-	Light Emitting Diode
LD	-	Laser Diode
MBE	-	Molecular Beam Epitaxy
MOCVD	-	Metal Organic Chemical Vapor Deposition
MOVPE	-	Metal Organic Vapor Phase Epitaxy
PL	-	Photoluminescence
RMS	-	Root Mean Square
SAW	-	Surface Acoustic Wave
SAD	-	Selected Area Diffraction
SE	-	Secondary Electron
TD	-	Threading Dislocation
TDD	-	Threading Dislocation Density
TEM	-	Transmission Electron Microscope

UV LED	-	Ultra Violet Light Emitting Diode
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

λ	-	Wavelength of incident wave
d	-	Interplanar distance
h	-	Plank's constant
mv	-	Nonrelativistic electron momentum
sin ɵ	-	Scattering angle
<uvw>	-	Components of Burgers vector
$\|b\|$	-	Magnitude of Burger's vector

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Nowadays, everyone is already familiar with semiconductors based bright blue, green and red light emitting diodes (LEDs) that light up our electronic appliances, decorate our streets and illuminate airport runways. LEDs are built mostly based on nitride semic006Fnductors such as aluminium nitride (AlN), gallium nitride (GaN), indium nitride (InN) and their alloying materials with wide wavelength ranging from the infrared to ultraviolet. In 1902, British scientist Henry J. Round discovered the physical effect of electroluminescence. Electroluminescence is an optical and electrical phenomenon whereby a material emits light in response to an electric current or an external electric field.

In 1962, the first visible spectrum LED light was produced by Nick Holonyak Jr. and was red in colour (Mukai *et al.,* 1998). As technology progressed in the 1970's, LEDs were used in applications such as calculators, digital watches and test devices. Since emitting blue light was a difficult task thus it took almost three decades to produce the first blue LED in 1971 using GaN by Jacques Pankove (Nakamura & Mukai, 1992). This is because in order to produce blue LED, it required the development of techniques for the growth of high-quality crystals as well as the ability to control p-doping of semiconductors with high bandgap to produce heterojunctions, which was achieved with gallium-nitride (GaN) (Nakamura & Krames, 2013).

LEDs consist of several layers which are p-type layers, n-type layers and active region between them. P-type layer hasa large hole concentrations, where else N-type contains majority electron carriers. Between them is an active layer or also known as the p-n junction to which the negative electrons and positive holes are driven when electric voltage is applied to the semiconductor. When electrons and holes meet, they recombine and light is emitted. This phenomenon is known as electroluminescence as shown in Figure 1.1 (Raguse & Sites, 2015).

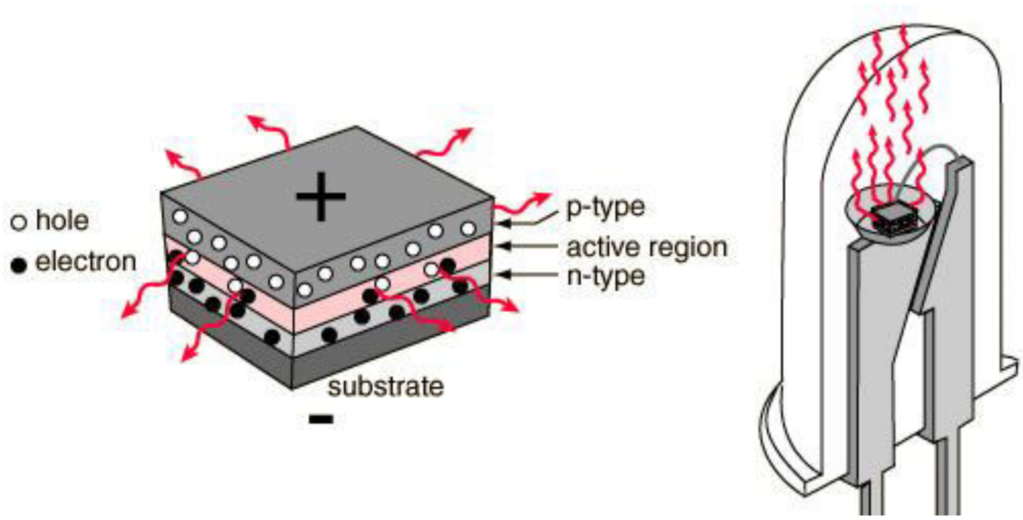

Figure 1.1 Phenomena of electroluminescence to emit light in LED (Raguse & Sites, 2015)

In order to have a good emission, it is essential to choose a suitable nitride semiconductor material which will emit light when electric current passed through it. Group III-V nitride semiconductors have been known for having outstanding optical, electronic and thermal properties. These materials have wurtzite orzinc blende crystal structures. AlN, GaN and AlGaN have gained considerable attention as promising materials for optoelectronic devices in the blue and UV regions (Kuech, 2016; Manasreh, 2000). This is because these nitrides are semiconductor with wide band gap of direct transition for example, 3.4eV for GaN, 6eV for AlGaN and 6.2eV for AlNat room temperature (Cardona & Kremer, 2014; Tripathy & Pattanaik, 2016).

Figure 1.2 shows the relationship between bandgap energy and wavelength of semiconductors. Light with wavelengths shorter than 400 nm is called ultraviolet

(UV) light. Since the emission wavelength is inversely proportional to the bandgap energy, a semiconductor with a wider bandgap emits light with a shorter wavelength. For instance, AlN has a wider direct- band gap energy of 6.3 eV and emits light with shorter wavelength, 210nm for deep ultraviolet UV LED.

On the other hand, the emission wavelength of GaN is 365nm with 3.2eV band gap energy and is used for high brightness blue LEDs and violet LDs for blue-ray equipment. Thus the wavelength of emitted light is determined by the bandgap energy of the semiconductor. Recently the winners of Noble prize physics, Akasaki's group of research invented the efficient blue LEDs using GaN, has led to white light sources for illumination. This light sources has long lifetime and requires ten times less energy than ordinary bulbs yet environmentally friendly (Akasaki &Amano, 2014).

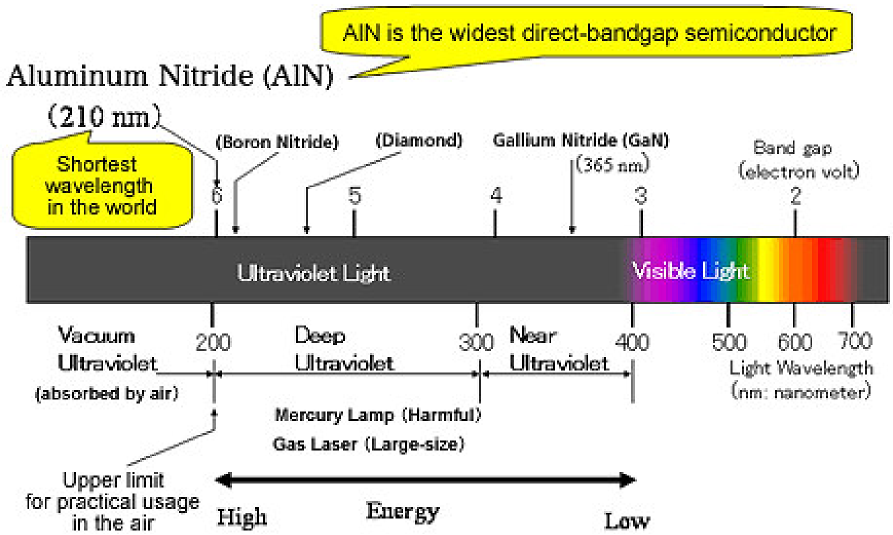

Figure 1.2 Relationship of bandgap and wavelength (Taniyasu & Kasu, 2010)

The definition of band gap is the energy needed to promote an electron from the lower energy valence band into the higher energy conduction band (Edwards, 2000). Hence, the wide band gap nitride semiconductor materials allows the devices to operate at much higher temperature, voltage and frequencies. The direct wide bandgap allows efficient absorption and emission of light. Due to this potential, semiconductor nitrides have been investigated to application for various optoelectronic devices, such as LED, Laser diodes (LD) and photo detectors for the ultraviolet region. In complimentary with this, there is a significant relationship between the band gap energy and wavelength. In the process of recombination by emitting photons to produce light, the energy state of the electron hole- pair drops. However the emitted photon has a specific energy determined by the bandgap of the material making up the LED as shown in Figure 1.3 (Narendran *et al.*, 2005).

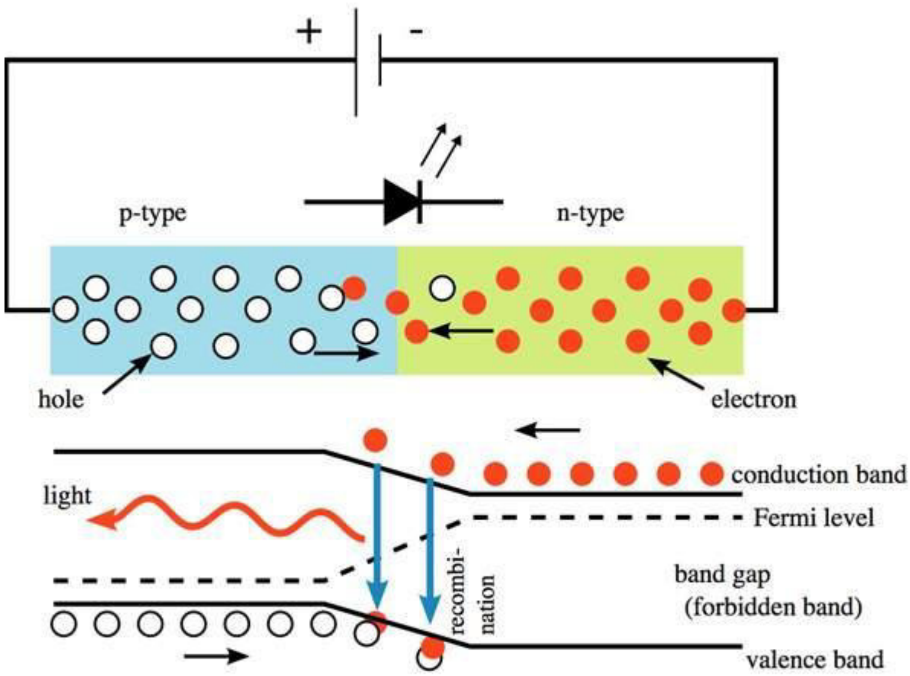

Figure 1.3 Influence of semiconductor bandgap energy with photon emission (Narendran *et al.*, 2005)

1.2 Motivation of Study

In this research, AlN was used as it stands out with the widest direct band gap among the III-V nitrides and that makes it a key component of deep-ultraviolet light source and detection. Due to it's high acoustic velocity and strong piezoelectricity, AlN is also used in the application of surface acoustic wave devices (SAW). AlN is thermally stable at high temperature with high thermal conductivity as well as high dielectric strength which promotes it to be used in high power electronic devices. In conjunction with the characteristics of conducting electricity in extreme environments, AlN demonstrates significantly higher performance while demanding less power.

AlN crystallizes in the wurtzite structure with lattice constants of a=3.111 Å and c=4.978 Å under the space group P63mc hexagonal as shown in Figure 1.4. AlN is stable at high temperature in inert atmosphere and it melts at 2800°C. It starts to decompose at 1400°C under the ambient atmosphere which makes it suitable for the usage at high temperature for heat treatment or annealing (Berger, 1997). However, the ultimate performance of AlN is limited by lattice defects that reduceit's efficiency. In addition, it is very difficult to grow high quality epitaxial layer of AlN, and several issues such as a high defect density and piezoelectricity still remain to be resolved in thin-film growth process. Globally, researchers have made a number of attempts in growing crystals including single crystals of high quality on substrates such as silicon carbide (SiC) and sapphire (Al_2O_3).

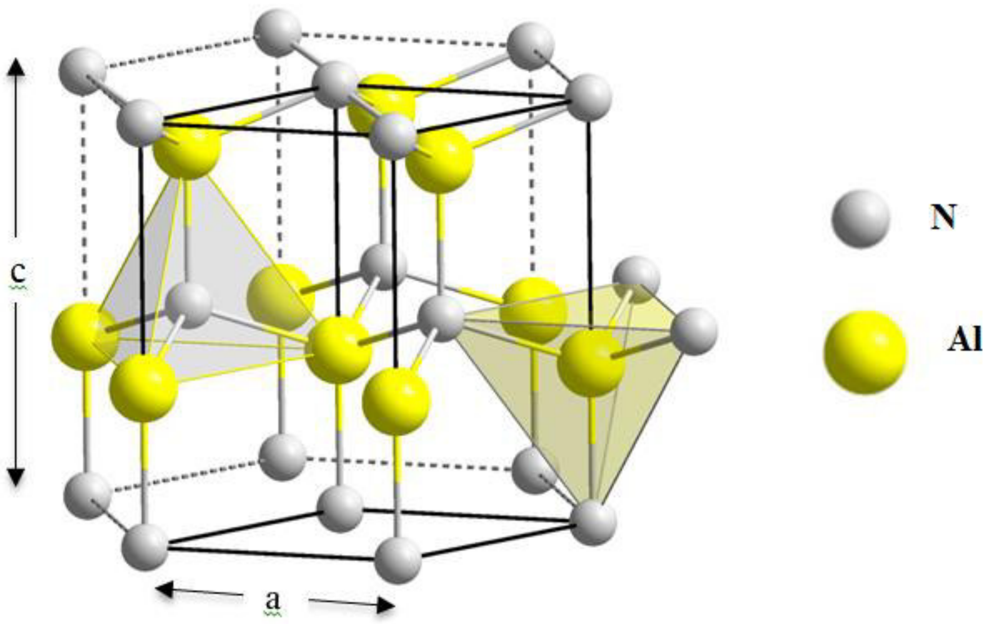

Figure 1.4 Crystal structure and lattice constant of aluminium nitride (Berger, 1997)

Various methods have been reported recently on the growth of nitride semiconductor thin films including hydride vapor phase epitaxy (HVPE), molecular beam epitaxy (MBE) and metal organic vapor phase epitaxy (MOVPE) using sapphire as a substrate (Ishida *et al.,* 2000; S. Wang *et al.*, 2015; W. Wang *et al.,* 2016; Yang *et al.*, 2015). However it has been difficult to grow high quality heteroepitaxial layer especially with a smooth top surface without cracks and other defects. This is because of the large lattice mismatch and large difference in thermal expansion coefficient between the nitride films and sapphire substrate. Table 1.1 shows the lattice constants and thermal expansion coefficient differences of AlN and GaN with sapphire substrate.

Table 1.1 : Lattice and thermal mismatches between nitride and sapphire

Elements	Lattice constant (A)	Thermal expansion coefficient x $10^{-6}(K^{-1})$
GaN	a = 3.189	5.59
	c = 5.182	7.75
AlN	a = 3.111	5.3
	c = 4.980	4.2
Sapphire	a = 4.758	7.5
	c = 12.991	8.5

The large lattice mismatch between AlN and sapphire resulted to 13% leads to dislocation defects in the AlN epitaxial. (Dovidenko *et al.,* 1996). A certain lattice mismatch is currently unavoidable but a reduction of threading dislocation (TDs) is required. Thus in order to reduce TDs, initially the growth parameters and steps should be modified. Complimentary to this, thin buffer layers also results in reduction of these defects. Recent development has succeeded in improving remarkably the surface morphology as well as the electrical and optical properties of AlN, GaN and AlGaN alloy films by preceding deposition of a thin AlN layer as a buffer layer (Ito *et al.,* 1999; Xiong *et al.,* 2013). Mainly AlN is used as a buffer layer because Al is hard and it bends the dislocation to prevent it to reach the surface of the nitride semiconductor therefore reduces the density of dislocation (Kuwano *et al.*, 2010). This phenomena will be further explained in the next chapter. As for AlGaN/GaN systems, GaN layer thickness is increased to reduce the dislocation density (Akasaki *et al.,* 1989).

In conjunction with this, a model for the growth process of high quality crystals whereby the crystallographic quality is examined using scanning electron microscope (SEM), reflection high energy electron diffraction (RHEED) and X-ray Diffraction (XRD). However usage of Transmission Electron Microscope (TEM) to clarify the microstructure inside the nitride semiconductors epitaxial films are not many. Nevertheless, cross sectional TEM observation of AlN grown on sapphire substrate is essential to provide detailed information. This is to control the microstructure and growth process of thin films including formation and annihilation process of defects for further analysis and characterization of nitride semiconductors. There are many other methods to reduce dislocations such as deposition of buffer layers, doping and epitaxial lateral overgrowth which will all be described in the next chapter. Annealing at high temperature is the method chosen by author and shall be discussed in detail. Annealing is a heat treatment process on the material which alters the materials microstructure by causing changes in physical, electrochemical and piezoelectric properties.

1.3 Problem Statement

When a thin film of AlN is grown or deposited on a sapphire substrate which has a large lattice mismatch and the large difference in the thermal expansion, it usually contains many structural defects. The density of dislocation is high normally in the range of $10^{10}cm^{-2}$. Besides threading dislocations, there are many other kinds of structural defects, such as inversion domain, stacking mismatch boundaries, voids, and stacking faults. These defects disrupts the periodicity of the crystal over the length of several atomic diameters thus degrades the optoelectronic properties. For instance, a threading dislocation acts as a nonradiative and scattering center in electron transport which effects the performance of LEDs and field effect transistor.

In addition, the inefficiency of optoelectronic devices are also caused by dislocations defects as rapid nonradiative recombination of holes without conversion of their available energy into photon causes heating up of the crystal. This leads to the deterioration of emission efficiency. Other than that optoelectronic devices have a shorter lifetime compared to devices with fewer defects. Thus the core problem is the inefficiency of devices due to defects in the material.

To my best knowledge, there is a lack of research conducted to characterize and analyze the lattice defects in details by transmission electron microscopy (TEM) although information obtained would be very useful in improving existing devices. This may be because very high skills and diffraction knowledge is required for one to characterize using TEM. The specimen preparation method is also very crucial and tedious. Moving on from here onwards, it is important to identify the right parameters which can assist to reduce lattice defects so that a high quality epitaxial film can be obtained. Many researchers have reported various ways to reduce the lattice defects, however a realistic condition in growing thin films without any defects have not be established yet. Annealing is one of the method which can reduce lattice defects in the epitaxial layer and has been employed by author before and after deposition of epitaxial layer.

Author studied the changes in microstructure due to high temperature annealing of AlN epitaxial on sapphire substrate. The core problem need to be identified at an early stage of the growth process to reduce or avoid defects in the crystal. Hence, this research aims to identify, analyse and characterize the defects in order to reduce the dislocation thus producing high quality thin films. The growth process of thin films with different parameters and conditions is also discussed in detail.

1.4 Research Objectives

The objective of this research is to determine the annealing effects in AlN epitaxial layer by:

i. Characterizing and analysing the defects in AlN thin film grown on sapphire substrate using TEM

ii. Identifying the changes of microstructure in AlN thin film after pre and post annealing treatment
iii. Determining the polarity of AlN thin film to categorize the defect type
iv. Establishing the growth parameters for higher quality AlN thin film.

1.5 Research Scope

This research was conducted in collaboration with the research groups of Mie University, Japan whereby the growth of AlN epitaxial on sapphire substrate using metalorganic vapour phase epitaxy (MOVPE) was carried out there. Annealing treatment was induced before and after the deposition of AlN buffer layer. The specimen provided by collaborators were further examined by author using electron microscopy technique at the facility of Kyushu University, Japan. After the formation process of lattice defects in the epitaxial were clarified, remedies for the condition to grow high quality thin films were proposed for establishment of the crystal growth. Especially the effects of heat treatments or annealing on the microstructure were investigated in details by using sophisticated TEM techniques.

The results of the research are important as to establish and improvise the parameters in growth conditions. In short, this research focuses on how does high temperature annealing can reduce the lattice defects in aluminium nitride thin films, thus is used for fabrication of high quality semiconductor devices.

1.6 Significance to Knowledge

The contribution of this study is to provide information to fellow researchers on the effects of annealing treatment in order to reduce the dislocation density in the AlN epitaxial layer by advanced TEM techniques. The crystallographic polarity determination also contributes to the identification of inversion domains in optoelectronic applications. Not only that, the annihilation mechanism of aluminium nitride heteroepitaxial layers lattice defects are also very useful. Furthermore, the growth conditions plays an important role thus the establishment is crucial too. This research also focuses on the development of high quality nitride semiconductor epitaxial using combinations of alloys and AlN buffer layers. By reducing the defects in the AlN epitaxial, the semiconductor based devices performance can be improved, which leads to saving cost and reduce energy consumption. In addition the reduction of defects also improves the optical, chemical and piezoelectric properties of the optoelectronic devices.

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