Multi-microscopy Characterisation of III-nitride Devices and Materials

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This dissertation is submitted for the degree of
Doctor of Philosophy

Wolfson College

April 2016

I would like to dedicate this thesis to my loving parents …

Declaration

I hereby declare that except where specific reference is made to the work of others, the contents of this dissertation are original and have not been submitted in whole or in part for consideration for any other degree or qualification in this, or any other university. This dissertation is my own work and contains nothing which is the outcome of work done in collaboration with others, except as specified in the text and Acknowledgements. This dissertation contains fewer than 65,000 words including appendices, bibliography, footnotes, tables and equations and has fewer than 150 figures.

Christopher Xiang Ren
April 2016

Acknowledgements

And I would like to acknowledge ...

Abstract

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Nomenclature

Acronyms / Abbreviations

1-D	One-Dimensional
AlN	Aluminium Nitride
EL	Electroluminescence
ET	Electron Tomography
FIBT	Focussed Ion Beam Tomography
GaAs	Gallium Arsenide
GaN	Gallium Nitride
InN	Indium Nitride
LED	Light-Emitting Diode
SPS	Single Photon Source

Nomenclature

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Chapter 1

Introduction

Gallium nitride (GaN) has been termed the 'most important semiconductor material since silicon' [1], and indeed the influence of this incredible material and it's associated alloys (termed III-nitrides) is pervasive in modern society. The impact of III-nitride materials is perhaps best evidenced by the global transition from traditional lighting sources to semiconductor lighting solutions based on III-nitride materials. Since the first demonstration of a high-brightness blue light emitting diode (LED) in 1991 by Shuji Nakamura [2], the widespread use of LEDs for general lighting purposes has blossomed into a multi-billion pound industry. The extraordinary optical properties of III-nitride materials have enabled their application outside of the lighting industry: the development of III-nitride based lasers has found applications in telecommunications [3], medicine [4] and data storage . Furthermore, III-nitride optical emitters have been used as single photon sources (SPSs) which have applications in cryptography for secure communications [5].

The optoelectronic properties of III-nitride materials are somewhat astonishing: GaN suffers from a defect density several orders of magnitude higher than other optically active semiconductor materials such as gallium arsenide (GaAs) [6] yet is still optically active. Despite this, the effects of defects originating from the heteropitaxial growth of GaN are clearly deleterious when considering III-nitride device operation. This work aims to explore the manner in which the microstructural properties of photonic III-nitride devices affect their performance by combining multiple microscopy techniques, an approach we term 'multi-microscopy', thus allowing us to link specific structural features with emissive properties at the device level. The experimental research in this thesis is separated into four main sections.

The first section details the investigation of inhomogeneous electroluminescence (EL) of indium gallium nitride (InGaN) quantum well (QW) LEDs. By employing the use of scanning probe techniques, electron microscopy and spectroscopy the underpinning cause of LED behaviour was elucidated and reported.

The second section involves microscopy-based investigation into the mechanisms behind incomplete etching in the fabrication of III-nitride based microdisk cavities and the effect of this issue on the overall optical performance of these cavities
The third section describes the microscopy of one dimensional (1-D) photonic crystal cavity (PCC) 'nanobeam' cavities. The intrinsic resistance of III-nitride based materials can often result in improperly etched features, which can results in high optical losses in cavities. This section concerns the use of tomographic techniques such as electron tomography (ET) and focussed ion beam tomography (FIB-T) to investigate the effect of these issues on the emission of III-nitride nanobeam cavities.

1.1 III-Nitride Material Properties

1.1.1 Crystal Structure

GaN can crystallise into two distinct crystal structures: hexagonal (wurtzite) and cubic (zinc blende and rock salt). Under ambient conditions, wurtzite GaN is the most commonly studied form as it is the most structurally stable. Thus, the work discussed in this thesis concerns wurtzite III-nitrides. A schematic of a wurtzite III-nitride crystal structure is shown in Fig.**??** and consists of stacked hexagonal close-packed planes following an ABABAB stacking sequence. Atoms of the respective elements are tetrahedrally bonded to one another. However, in the case of III-nitrides this structure deviates from ideal tetrahedral bonding and results in a non-zero dipole moment for each unit cell which will be discussed in the following sections.
A 4-index Miller-Bravais notation (hkil) is used to denote the crystal planes where the index i is defined by the relation:

$$i = -(h+k) \tag{1.1}$$

The crystallographic planes (0001), (1-100) and (11-20) shown in Fig.**??** are often termed the c, m and a-planes in the literature. The fundamental unit cell of the wurtzite GaN crystal structure and its associated lattice parameters **a** and **c** is shown in Fig.1.1

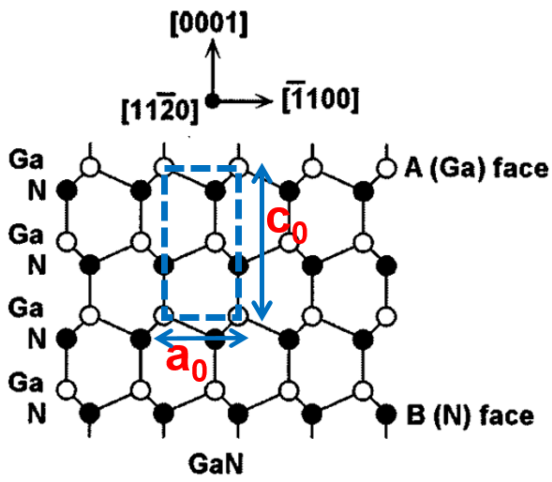

Fig. 1.1 Unit cell (dashed line) for GaN crystal structure and lattice parameters $\mathbf{a_0}, \mathbf{c_0}$. Adapted from [7]

Other members of the III-nitride materials such as indium nitride (InN) or aluminium nitride (AlN) have different lattice parameters due to the differing atomic radii of aluminium and indium relative to gallium.

Alloy	**a** (Å) at T = 300K	**c** (Å) at T = 300K
GaN	3.189	5.185
InN	3.545	5.703
AlN	3.112	4.982

Table 1.1 Room temperature lattice parameters for GaN, InN and AlN [8].

III-nitride photonic devices are often heterostructures consisting of ternary alloys the materials shown in Table **??**. Lattice parameters of a relaxed ternary alloy $A_xB_{1-x}N$ can be estimated using Vegard's law [9]:

$$\mathbf{a} = x\mathbf{a}_{AN} + (1-x)\mathbf{a}_{BN} \tag{1.2}$$

$$\mathbf{c} = x\mathbf{c}_{AN} + (1-x)\mathbf{c}_{BN} \tag{1.3}$$

Typical indium compositions for blue LEDs range between 15-20 %, which leads to a considerable lattice mismatch of approximately 2 %, resulting in considerable amounts of strain in these GaN/InGaN heterostructures.

1.1.2 Band Structure

One of the principal driving factors behind the interest in III-nitrides for photonic devices is their direct bandgap which collectively spans the visible spectrum and beyond.

1.1.3 Built-in Fields

III-nitride materials in wurtzite structure are termed 'polar' materials, due to the fact they exhibit a spontaneous polarisation field [10]. This occurs due to III-nitride bonding structure deviating from an ideal tetrahedral structure along the (0001) axis along the crystal, combined with the ionicity of the bond [11]. This deviation causes each unit cell to possess a non-zero dipole moment along the principal axis of the tetrahedral bonding structure, resulting in an overall spontaneous polarization in the crystal. As the III-nitride wurtzite structure is non-centrosymmetric, the direction of the polarization depends on whether the crystal exhibits (+ *c*) or (-*c*) polarity, as shown in Fig.1.2

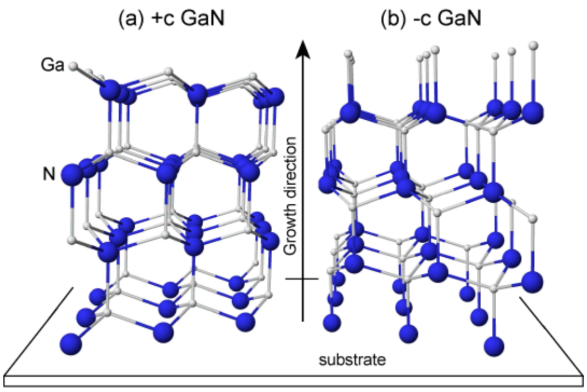

Fig. 1.2 Illustration of Ga-face (+ *c*) and N-face (-*c*) GaN wrutzite crystal exhibiting polarity along the *c*-axis [12].

This non-zero dipole moment is particularly strong for III-nitrides relative to other III-V semiconductors due to the strong electronegativity and small size of nitrogen compared to other group V elements, resulting in a metal-nitrogen bond with greater ionicity than other III-V bonds [13]. Fig.1.3 shows a GaN unit cell with lattice parameters *c* and *a* denoted.

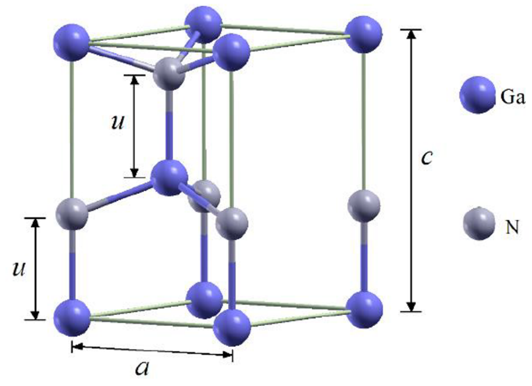

Figure 1. Unit cell 1x1-MN/GaN (M = V, Cr and Mn) multilayers.

Fig. 1.3 GaN unit cell with lattice parameters c and a [14]

If all nearest neighbour bond lengths are equal, an ideal hexagonal closed packed crystal exhibiting zero spontaneous polarisation would have a ratio of lattice parameters denoted by:

$$\frac{c}{a} = (\frac{8}{3})^{0.5} = 1.63299 \tag{1.4}$$

The degree of spontaneous polarisation observed in III-nitride materials is thus determined by the amount their lattice parameter ratio deviates from this ideal value. The values for bulk III-nitride materials are given in Table.1.2.

Alloy	$\mathbf{\frac{c}{a}}$
GaN	1.6259
InN	1.6116
AlN	1.6010

Table 1.2 Bulk $\frac{c}{a}$ ratios for GaN, InN and AlN [11].

A lower $\mathbf{\frac{c}{a}}$ ratio indicates a higher angle between the three bonds at the base of the tetrahedral bonding structure, resulting in a lower compensation polarisation along the (0001) axis and a higher spontaneous polarisation. Thus according to Table.1.2 the strongest spontaneous polarisation is observed in AlN and the weakest in GaN.
It is important to note that materials which exhibit spontaneous polarisation also exhibit piezoelectric polarisation [10]. Strain experienced by the material results in the distortion in of the crystal lattice, which can either alleviate or exacerbate the deviation from the ideal tetrahedral structure resulting in an additional polarisation. This piezoelectric polarization is a crucial consideration in III-nitride devices which often consist of QW heterostructures:

lattice mismatches with underlying layers result in the expansion or contraction of III-nitride films. Interestingly two different polarisation configurations are obtained for AlGaN and InGaN coherently strained to GaN. In the case of InGaN the piezoelectric field acts against the spontaneous field, whilst the opposite is true for AlGaN strained to GaN. Within the context of visible light LEDs, InGaN containing QWs are dominated by the piezoelectric contribution to the polarization fields [15] due to the sizeable lattice mismatch between GaN and InN (11%) [16].

The Quantum Confined Stark Effect

As previously discussed, III-nitride photonic devices often make use of heterostructures known as quantum wells, which enhance radiative efficiency by confining carrier wavefunctions over a range of several nanometres. Given the presence of built-in fields in III-nitride materials, it is important to consider the effect polarisation fields will have on the band structure and thus optical properties of quantum wells as shown in Fig.1.4

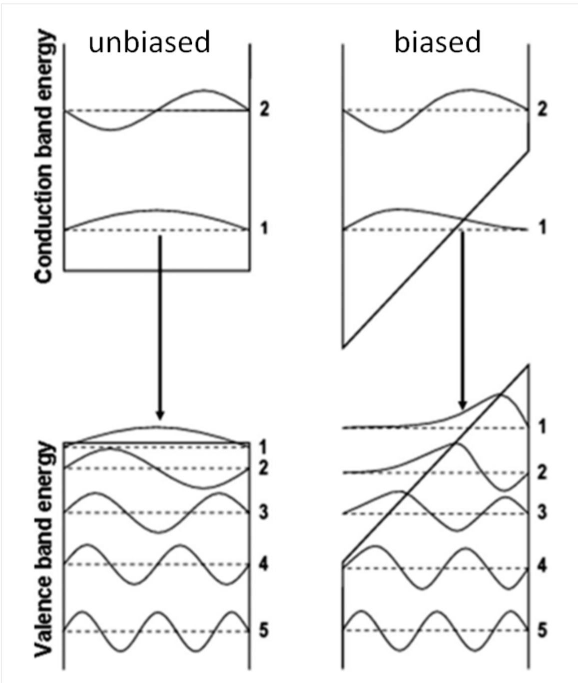

Fig. 1.4 Unbiased and biased quantum well energy levels with associated carrier wavefunctions. Under an applied field the overlap between the electron and hole carrier wavefunctions is reduced [17].

The transition from a rectangular to a 'sawtooth'-shaped potential well results in the reduction in energy of the optical transition, meaning the photons emitted from the QW are red-shifted. However, as the carrier density within the QW is increased, by either optical

or electrical injection, the polarization fields are effectively screened resulting in a carrier density-dependent optical transition energy.
A further effect of the polarization fields is to spatially separate the carrier wave functions, thus reducing their overlap as shown in Fig.1.4. This results in a reduced probability for the radiative recombination carriers thus reducing the efficiency of III-nitride QW emitters.

1.1.4 Defects in III-nitrides

1.2 III-nitride Cavities

1.2.1 Cavity characteristics

1.2.2 Cavity Designs

Chapter 2

Experimental Methods

2.1 Atomic Force Microscopy

2.2 Hyperspectral Electroluminescence Mapping

2.3 Scanning Electron Microscopy and Cathodoluminescence

2.4 Photoluminescence

2.5 Dual Beam Scanning Electron microscopy with a Focussed Ion Beam

2.5.1 Fabrication

2.5.2 Tomography

2.6 Transmisson Electron Microscopy

2.6.1 Tomography

2.7 Hidden section

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Subplots

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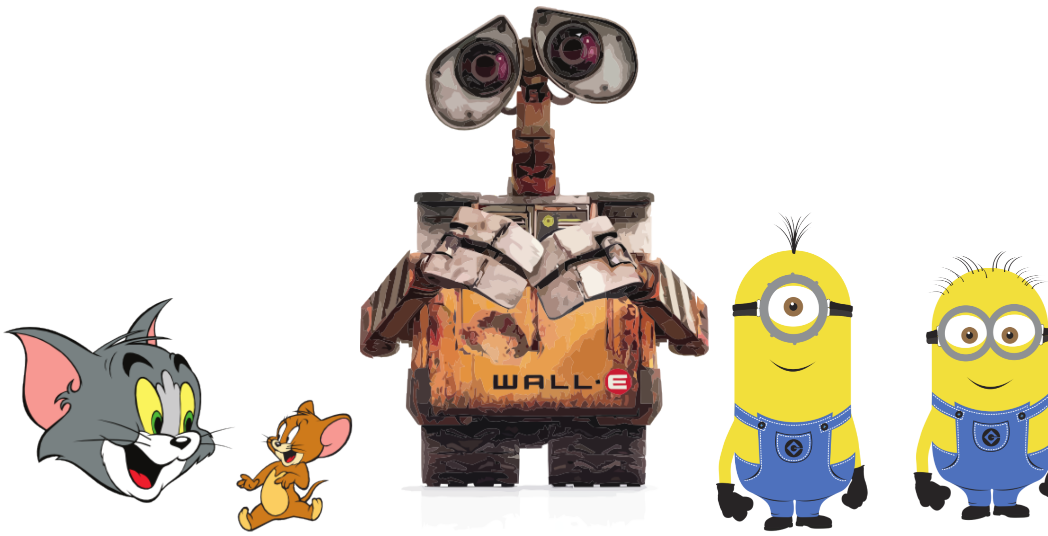

(a) Tom and Jerry (b) Wall-E (c) Minions

Fig. 2.1 Best Animations

Chapter 3

Inhomogeneous Electroluminescence in InGaN QW LEDs

3.1 Background

$In_xGa_{1-x}N$/GaN quantum well (QW) structures are key structures in present day light emitting diodes in the visible wavelengths. Despite the growth of III-nitride LEDs into a gigantic market with a projected overall worth of 64 billion EUR by 2020, III-nitride alloys suffer from a plethora of material issues arising from heteroepitaxial growth on foreign substrates with large lattice mismatches [6]. A notorious issue in III-nitride growth is the high density of threading dislocations which are the source of highly undesirable effects in diode structures such as non-radiative recombination [18] and leakage current [6].
Threading dislocations have been shown to result in inverted pyramidal defects at the surface of nitride epilayers, known as 'V defects'. The effect of these defects on LED performance is hotly debated in literature as they are expected by many to hinder LED performance due to their association with TDs. However, it has been shown that narrower QWs along the sidewalls of V-defects serve to screen carriers from the non-radiative centres at TDs

Enumeration

1. The first topic is dull
2. The second topic is duller
   (a) The first subtopic is silly
   (b) The second subtopic is stupid

3. The third topic is the dullest

itemize

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  - The second subtopic is stupid
- The third topic is the dullest

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3.2 Hidden section

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Subplots

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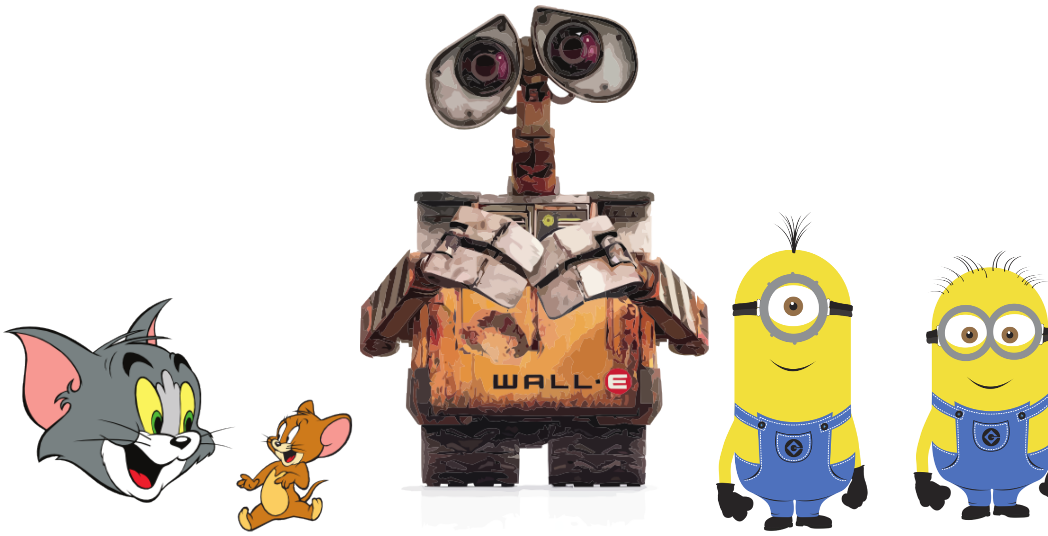

(a) Tom and Jerry (b) Wall-E (c) Minions

Fig. 3.1 Best Animations

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Appendix A

How to install LaTeX

Windows OS

TeXLive package - full version

1. Download the TeXLive ISO (2.2GB) from https://www.tug.org/texlive/
2. Download WinCDEmu (if you don't have a virtual drive) from http://wincdemu.sysprogs.org/download/
3. To install Windows CD Emulator follow the instructions at http://wincdemu.sysprogs.org/tutorials/install/
4. Right click the iso and mount it using the WinCDEmu as shown in http://wincdemu.sysprogs.org/tutorials/mount/
5. Open your virtual drive and run setup.pl

or

Basic MikTeX - TeX distribution

1. Download Basic-MiKTeX(32bit or 64bit) from http://miktex.org/download
2. Run the installer
3. To add a new package go to Start » All Programs » MikTex » Maintenance (Admin) and choose Package Manager

4. Select or search for packages to install

TexStudio - TeX editor

1. Download TexStudio from
http://texstudio.sourceforge.net/#downloads

2. Run the installer

Mac OS X

MacTeX - TeX distribution

1. Download the file from
https://www.tug.org/mactex/

2. Extract and double click to run the installer. It does the entire configuration, sit back and relax.

TexStudio - TeX editor

1. Download TexStudio from
http://texstudio.sourceforge.net/#downloads

2. Extract and Start

Unix/Linux

TeXLive - TeX distribution

Getting the distribution:

1. TexLive can be downloaded from
http://www.tug.org/texlive/acquire-netinstall.html.

2. TexLive is provided by most operating system you can use (rpm,apt-get or yum) to get TexLive distributions

Installation

1. Mount the ISO file in the mnt directory

   ```
   mount -t iso9660 -o ro,loop,noauto /your/texlive####.iso /mnt
   ```

2. Install wget on your OS (use rpm, apt-get or yum install)

3. Run the installer script install-tl.

   ```
   cd /your/download/directory
   ./install-tl
   ```

4. Enter command ‘i’ for installation

5. Post-Installation configuration:
   http://www.tug.org/texlive/doc/texlive-en/texlive-en.html#x1-320003.4.1

6. Set the path for the directory of TexLive binaries in your .bashrc file

For 32bit OS

For Bourne-compatible shells such as bash, and using Intel x86 GNU/Linux and a default directory setup as an example, the file to edit might be

```
edit $~/.bashrc file and add following lines
PATH=/usr/local/texlive/2011/bin/i386-linux:$PATH;
export PATH
MANPATH=/usr/local/texlive/2011/texmf/doc/man:$MANPATH;
export MANPATH
INFOPATH=/usr/local/texlive/2011/texmf/doc/info:$INFOPATH;
export INFOPATH
```

For 64bit OS

```
edit $~/.bashrc file and add following lines
PATH=/usr/local/texlive/2011/bin/x86_64-linux:$PATH;
export PATH
MANPATH=/usr/local/texlive/2011/texmf/doc/man:$MANPATH;
export MANPATH
```

```
INFOPATH=/usr/local/texlive/2011/texmf/doc/info:$INFOPATH;
export INFOPATH
```

Fedora/RedHat/CentOS:

```
sudo yum install texlive
sudo yum install psutils
```

SUSE:

```
sudo zypper install texlive
```

Debian/Ubuntu:

```
sudo apt-get install texlive texlive-latex-extra
sudo apt-get install psutils
```

Appendix B

Installing the CUED class file

LaTeX.cls files can be accessed system-wide when they are placed in the <texmf>/tex/latex directory, where <texmf> is the root directory of the user's TeXinstallation. On systems that have a local texmf tree (<texmflocal>), which may be named "texmf-local" or "localtexmf", it may be advisable to install packages in <texmflocal>, rather than <texmf> as the contents of the former, unlike that of the latter, are preserved after the LaTeXsystem is reinstalled and/or upgraded.

It is recommended that the user create a subdirectory <texmf>/tex/latex/CUED for all CUED related LaTeXclass and package files. On some LaTeXsystems, the directory look-up tables will need to be refreshed after making additions or deletions to the system files. For TeXLive systems this is accomplished via executing "texhash" as root. MIKTeXusers can run "initexmf -u" to accomplish the same thing.

Users not willing or able to install the files system-wide can install them in their personal directories, but will then have to provide the path (full or relative) in addition to the filename when referring to them in LaTeX.