# CORRELATION OF SELECTED PROBLEMS DURING GaN MOVPE EPITAXY ON Si SUBSTRATES WITH IN-SITU INTERFEROMETER OBSERVATION

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In this paper, selected problems occurring during GaN epitaxy on Si substrates are correlated with in-situ interferometer observations based on structures fabricated using  $3\times2$ " Thomas Swan Close Coupled Showerhead MOVPE system. Samples were epitaxially grown on highly resistive (>  $500\,\Omega{\rm cm}$ ) p-type 2" silicon substrates. The growth was observed using an interferometer acquiring reflectance traces in-situ. To avoid issues that are shown in this paper, different approaches of the so-called buffer layer were used by depositing them on Si before the growth of GaN. Different material qualities of grown GaN layers were obtained by variation in films constituting the buffer layer grown before the desirable GaN film. The study shows the evolution of the grown samples, improvement of various parameters and also gradual reduction of material issues discussed in this paper. Improvement in GaN layer was mainly observed by SEM characterization and studying the growth mechanisms through observation of reflectance traces obtained in-situ.

Keywords: Gallium nitride, GaN on Si, MOVPE, transition AlGaN, in-situ observation

#### 1 INTRODUCTION

GaN and nitride materials are very attractive for high-frequency, high-power electronics and are commonly grown on sapphire or SiC substrates. However, nowadays much attention is paid particularly to GaN grown on silicon due to low cost and availability of large dimensions substrates. This also provides opportunity towards integration with Si electronics. Along with benefits of using silicon as a substrate many issues arise that have already been eliminated or minimized [1]. Problems occurring during GaN growth on Si can hinder to obtain high quality GaN layers with desirable electrical and optical parameters. It is shown that gradual improvement can be achieved by varying the composition and technological parameters used during the deposition process of transition layers.

## 2 COMPARISON OF MATERIAL PROPERTIES

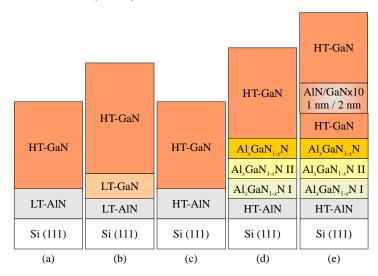
In this section the properties of various materials used for MetalOrganic Vapour Phase Epitaxy (MOVPE) of gallium nitride are compared and the choice of Si substrate is justified. All substrates materials for GaN growth were chosen due to their cell structure, both lattice constant and thermal expansion coefficient close to gallium nitride, chemical stability and ability to withstand high temperatures >  $1000\,^{\circ}$ C that is necessary in order to grow high quality GaN by MOVPE. We can distinguish a few applicable substrates for GaN growth such as silicon carbide, sapphire, and silicon, which are heteroepitaxial substrates, and homoepitaxial GaN substrate, which has re-

cently entered the marked. GaN substrates bring many opportunities and possibilities into GaN-based devices. However, enormous price and relatively low dimensions of GaN substrates make them virtually unusable, mainly from the economic point of view. Each of heteroepitaxial substrates exhibits some advantages and disadvantages depending on the desired application of the grown structures. Nevertheless, the important factor is the price and availability of large dimension substrates if the fabricated structures are expected to be put in practical use. Choosing silicon substrates for GaN growth involves a number of advantages, like the ability to withstand high temperatures, good heat conductivity 1.5 W/cm K and the possibility of integration with other Si integrated devices. Moreover, Si substrates still ensure the lowest price and a variety of physical properties — wide range of resistivity, p- or n-type conductivity, as well as the possibility of obtaining largest diameters of single wafers which can reach up to 12". From the point of view of optoelectronic application, all types of conductivity that can be achieved in Si substrate, is considered as an major advantage. The properties of substrates are reviewed in Tab. 1. Silicon oriented in (111) direction was chosen due to pseudohexagonal arrangement of atoms that are considered to be the best option for GaN growth. Using silicon as a substrate for GaN epitaxy involves some issues that will be discussed further.

### 3 EXPERIMENTAL

Silicon oriented in (111) direction was chosen due to the pseudo-hexagonal arrangement of atoms that is

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 $\bf Fig.~1.$  Scheme of grown GaN layers

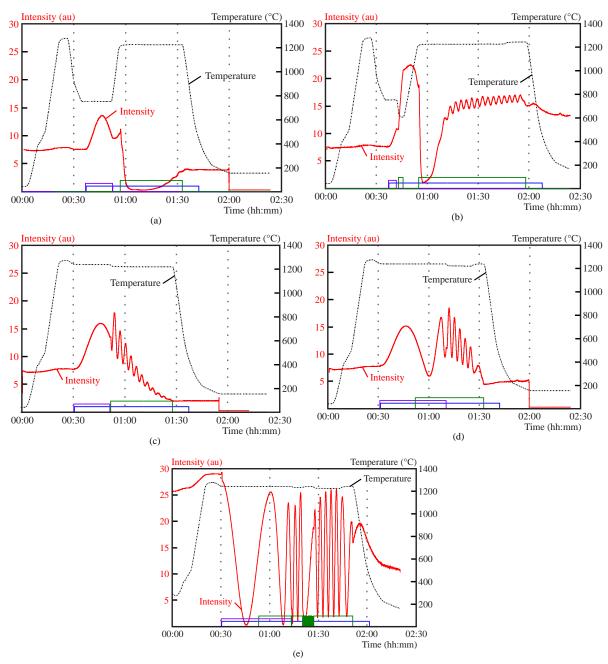


Fig. 2. Scheme of grown GaN layers

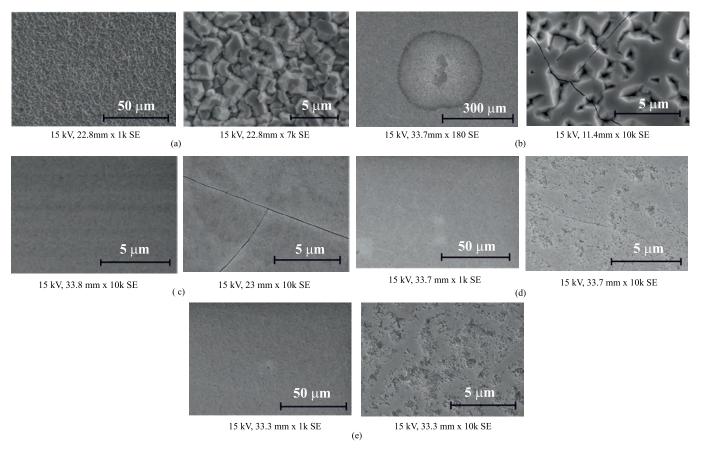


Fig. 3. SEM surface images of grown samples: left (a) to (e)—view of the GaN surface, left (b)—example of melt back etching, right (a)—not coalesced GaN islands, right (a) to (e)—gradual improvement in coalescence of grown layers, visible cracked surfaces

Table 1. Comparison of the most significant properties of substrate materials for GaN epitaxy (averaged values, may differ at low and high temperatures, listed in respect to GaN, prices averaged based on offers the author has been given from producers) [1,2]

Material	$Al_2O_3(0001)$	SiC (6H, 4H)	Si (111)	GaN
Diameter up to	8"	4"	12"	2"
Average price per 2" substrate	15-50 \$	800-1400 \$	7–30 \$	> 2000 \$
$a( ext{Å})$	4.76	3.08	5.43	3.19
$c( ext{Å})$	12.99	$15.1,\ 10.1$	_	5.19
$TEC (10^{-6}/K)$	7.5	4.2, 4	2.59	5.59
Thermal conductivity	0.5	4.9, 3.7	1.56	1.3
$\Delta a/a(\%)$	16	3.4	17	_
$\Delta  \mathrm{TEC}/\mathrm{TEC}  (\%)$	-34	25, 28	54	_

considered as the best option for GaN growth. Using  $3\times 2$ " Thomas Swan Close Coupled Showerhead MOVPE system, GaN samples were grown on Si(111) high resistive (>  $500\,\Omega\,\mathrm{cm}$ ) p-type substrates. Trimethylgallium (TMGa), trimethylaluminium (TMAl) and ammonia (NH<sub>3</sub>) were used as gallium, aluminum and nitride precursors, respectively. H<sub>2</sub> was used as a carrier gas.

The growth process consisted of annealing the silicon substrate in hydrogen atmosphere, growth of different transition layers before thick ( $\sim 1500 \text{ nm}$ ) GaN for each structure. With respect to Fig. 1: (a) — low temperature GaN (LT-GaN) deposited on low temperature AlN nucleation layer (LT-AlN), (b) — LT-AlN, (c) — high temperature AlN (HT-AlN), (d) —  $\text{Al}_x \text{Ga}_{1-x} \text{N}$  transi-

tion layer that ensures smooth transition from AlN to GaN on HT-AlN, (e) —  $\mathrm{Al}_x\mathrm{Ga}_{1-x}\mathrm{N}$  transition layer and strained layer superlatice (SLS) AlN/GaN which serves the purpose of relieving stresses present in the grown layers. Photoluminescence spectra were measured of each sample at room temperature. PL peaks are vivid and around 365 nm wavelength. Slight variation of the peaks can be correlated with various types and amounts of strains present in the GaN/Si multilayer systems In each case the 20 seconds pre run of TMAl before introducing to the reactor other precursors was performed in order to avoid issues connected to reactivity of Ga, N and Si. Reflectance traces were acquired in-situ by a 635 nm interferometer mounted on the reactor of the epitaxial sys-

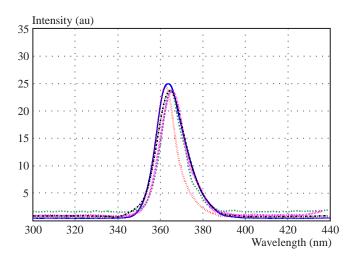


Fig. 4. Photoluminescence spectra of each sample measured in room temperature. Straight line – sample A, dash line – sample B, dot line – sample C, dash dot line – sample D, short dash line – sample E

tem. SEM images were taken using HITACHI SEM microscope. Smooth monocrystal layers with two-dimensional (2D) growth mode are grown when the oscillations observed on traces are regular with a high value of least-to-maximum intensity. The ideal case of discussed in situ reflectance traces can be found in another author's work [3].

#### 4 DISCUSSION

Issues occurring during heteroepitaxial GaN growth by MOVPE are described, discussed and examples of work in our laboratory are shown. The high in-plane lattice mismatch is considered for Si (111) plane and it results in a very high initial dislocation density in GaN layer. Calculating dislocation densities one can obtain values of the order of  $10 \times 10^{13} \text{cm}^{-3}$  present at GaN/Si interface. The initial dislocation density quickly decreases within a few hundred nanometers, this feature is presumed to be caused by the high binding and dislocation activation energies of nitrides [1]. Another consequence of large lattice mismatch is the difficulty to grow the layer formed in continuous matter, to completely transit to two-dimensional growth (Fig. 2). Reactivity of Ga, N and Si prevents direct growth of GaN on Si. At high elevated temperatures that are consistent with GaN epitaxy, gallium interacts with silicon leading to the so-called meltback etching (Fig. 3). Once initiated, it is not possible to stop this phenomenon, which leads to a rough surface with deep hollows in the substrate [1]. Moreover, nitrogen present in one of the precursors, ammonia, at high temperatures reacts with silicon forming a  $Si_xN_x$  amorphous layer [2]. On such areas the growth of GaN will be prevented, which can either be of disadvantage or can be used as intentional masking for selective growth. However, the most relevant problem is large TECs difference. During GaN layer epitaxy on Si, MOVPE process is performed at elevated temperatures, usually above 1000 °C. After the process the reactor has to be cooled down to room temperature. Large thermal mismatch between the grown GaN and the Si

substrate leads to introducing tensile stress in GaN layer during the cooling procedure. The amount of adhibited stress depends on many factors, such as the thickness of the deposited layer, initial growth mechanism, composition and type of buffer layer, but also on technological parameters (i.e., temperature, pressure in the reactor, and V/III ratio of the precursors). Present stress directly leads to crack formation impeding future device processing (Fig. 3). Additionally, such cracks can partially expose the Si substrate and initiate meltback etching reaction that is described above. Reflectance traces acquired in situ can be correlated with the features observed on SEM images (Figs. 2,3). In sample A, absence of oscillation is observed that is a direct result of the lack of coalescence. It is visible that GaN grows in the form of separate blocks. Sample B is partially coalesced, but additionally melt back etching occurred due to the application of a too thin seed LT-AlN layer. In samples C and D, a decrease in reflectance signal is observed due to both cracks that are formed during the growth and increasing surface roughness. Sample E was grown with almost a crack free surface, stable oscillations are observed indicating complete coalescence of the grown GaN layers and transition to 2D growth mode.

Photoluminescence spectra were measured of each sample at room temperature. PL peaks are vivid and around 365 nm wavelength. Slight variation of the peaks can be correlated with various types and amounts of strains present in the GaN/Si multilayer systems.

#### 5 SUMMARY

Gradual improvement of the deposited GaN-based multilayer systems was shown and discussed. The described issues were either overcome or minimized. Improvements in the growth mechanisms are observed on the reflectometer traces but simultaneously indicating incomplete transition to the two-dimensional growth mode, which can be correlated with both rough surfaces and

cracks present on the grown samples. Application of various approaches indicates that further improvement can be achieved and observed by correlation with reflectometer traces observed in-situ.

# Acknowledgement

This work was co-financed by the European Union within European Regional Development Fund, through grant Innovative Economy (POIG.01.01.02-00-008/08-05), National Centre for Science under the grant no. N N515 495740 and DEC-2012/07/D/ST7/02583, by National Centre for Research and Development through Applied Research Program grant no. 178782, by Wrocław University of Technology statutory grants and Slovak-Polish International Cooperation Program no. SK-PL-0005-12.

This paper summarizes in brief the essential issues of a work presented at ADEPT-2014, Advances in Electronics and Photonic Technologies, an international conference held in Tatranska Lomnica, High Tatras, Slovakia, on 1-4 June 2014.

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Received 15 June 2014

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