Standard Project

Experimental Report

Proposal title: In situ monitoring of strain relaxation during the deposition of the shell in GaN/AlxGa1-xN core-shell nanowires grown by PAMBE.		**Proposal number:** 20100807
Beamline: IF-INS	**Date(s) of experiment:** from: June 29th, 2011 to: July 5th, 2011	**Date of report:** *November 28th, 2011*
Shifts: 18	**Local contact(s):** Dr. Valentina Cantelli	*Date of submission:* *September 15th, 2010*

Objective & expected results (less than 10 lines):

Using the SUV cabin of the beamline IF-INS, our first objective was to understand the process of nucleation of GaN nanowires grown by Plasma Assisted Molecular Beam Epitaxy. The questions we aimed at answering were related to the very first stages of the GaN nanowires growth. What happens on the Si surface when exposed to Ga and N simultaneously? when exposed to N only ? when exposed to Ga only ? what conditions make the start of nucleation possible? how is it related to the Ga flux involved ?

Our second aim was to monitor *in situ* the evolution of strains involved when depositing an AlN shell around these GaN nanowires.

Results and the conclusions of the study (main part):

1) Nucleation

Although it wasn't included in the proposal we submitted, this part of the work we performed was the most sucessful one. As a matter of fact, we discovered that when exposed to the slightness beam of active nitrogen, the Silicon surface is nitridated. This nitridation consists in the formation of a crystalline structure detectable by its corresponding peak in the reciprocal space (Fig. 1). Interestingly, the intensity of such peak increases and very quickly (less than 1 minute after the start of the nitridation process) saturates. Then, when further exposed to N (up to 10 minutes), the intensity of the peak remains constant. Such a surface was afterwards simultaneously exposed to Ga and N. We noticed that a *sine qua non* condition for the GaN to start growing (i. e. for the peak related to crystalline GaN to appear) is the decrease of the intensity of the peak related to nitridated Silicon (Fig. 2)

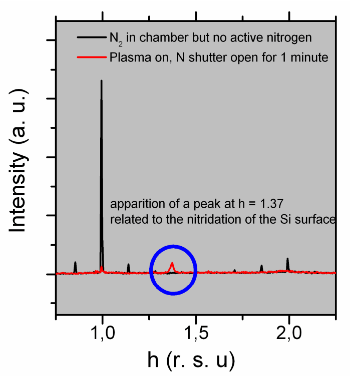

Fig. 1. h-scan around $[100]_{Si}^{hex}$ before (black line) and after (red line) turning on the plasma. Without the presence of active nitrogen, one can see the typical Si 7x7 reconstruction. With the introduction of active nitrogen, the 7x7 has disappeared and a peak at h = 1.37 has shown up. This peak is related to the formation of crystalline Si_xN_y.

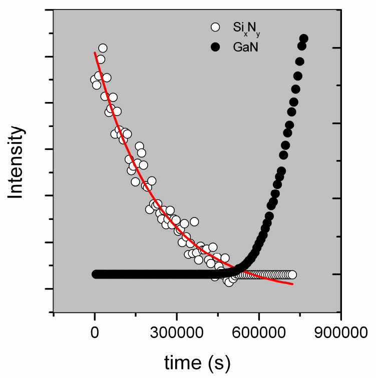

Fig. 2. Evolution in time of the peak related to crystalline Si_xN_y $[1.37\ 00]_{Si}^{hex}$ and of the peak related to GaN $[3.62\ 00]_{Si}^{hex}$ while depositing Ga and active N.

This decrease was first understood as the destruction of the crystalline layer but later on, rather interpreted as an amorphisation of the layer. Interestingly, no matter how important the Ga flux is, the SiN peak always takes more or less the same time to dissapear. An other interesting result is the fact that even when attempting to protect the silicon surface by leaving it under a constant Ga flux prior to the turning on the plasma cell, the nitridation happens anyway.

On the other hand, when playing with AlN, the behaviour is totally different. AlN is often used as a buffer layer to improve the nanowires orientation. It is usually grown by alternatively supplying Al and N. In this case, it was interesting to observe that although the surface is covered with aliminium, the SiN related peak appears and almost instantly its intensity fades. Once the thin AlN buffer was grown (corresponding to about 40s of deposited Aluminium), it was also observed that the GaN nanowires start growing much faster than on bare Silicon (about 5 minutes on AlN VS 2h00 on bare Silicon in this case)

2) Strains in core-shell nanowires

The requirements for a successful measurement of non –biased strains in core-shell nanowires are the following: straitness of the GaN cores, homogeneity of the shell thickness from nanowire to nanowires and symmetry of the shell on a given nanaowire. Such features were supposed to be obtained by performing Selective Area Growth, that is beeing able to properly control the density of nanowires and therefore better managing their final morphology. However, prior to the experiment, it was not possible to perform such growth and therefore to determine the appropriate growth conditions. As a consequence, we decided to attempt growing a core-shell sample on bare Silicon. It can be seen from the pictures below (Fig. 3) that the nanowires are too dense and the shell totally inhomogenious along the nanowire length. The measured strains are therefore difficult to interprete.

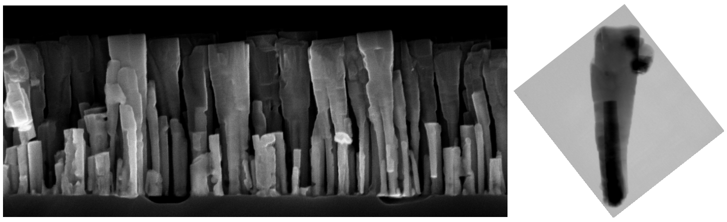

Fig. 3. Scaning Electron Micrographs of the GaN/AlN core shell nanowires grown during the experiment

Justification and comments about the use of beam time (5 lines max.):

It was absoluteley necessary to get enough intensity in grazing incidence to detct signal from growing GaN and to discover the Si_xN_y peak.

Publication(s):

Nothing has been yet published about this synchrotron run but an article about the nucleation is in process of writing.