Standard Project

Experimental Report template

Proposal title: In situ study on Ge condensation by low temperature SiGe oxidation		Proposal number: 20160924
Beamline: BM02 (D2AM)	**Date(s) of experiment:** from: 12/04/2017 to: 18/04/2017	**Date of report:** 15/09/2017
Shifts: 18	**Local contact(s):** Nathalie Boudet	*Date of submission:*

Objective & expected results (less than 10 lines):

The objective was to study in situ the kinectics of the low temperature Ge condensation process with combied GIXD, GIMAD and XRR. We wanted (1) to follow in real time the changes in composition, strain, thickness, position of the Ge rich layer during oxidation, (2) to study the effect of temperature, (3) the possible compliance of the UTSOI substrate and (4) the critical thickness of the Ge rich layer.

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

The experiment was a great success. We were able to measure a total of 10 samples (compared to the 7 samples that we initially planned), each starting at a different stage of condensation and/or under a different temperature (see fig.1).
So far the GIXD data and the XRR data have been analyzed. With XRR (see fig. 2), we were able to extract the thickness evolution of each layer (The top Si oxide layer, Ge rich layer, initial SiGe 20% layer, ultra thin Si layer and buried oxide layer). This allows us to estimate the amount of Si that was oxidized and hence the Ge composition in the Ge rich layer. Below we will demonstrate some typical GIXD results obtained on two of the samples (170417 and 180417).
For sample 170417, we started with an already condensed SiGe 50% layer (the ultrathin Si layer of the SOI has been totally consumed, see fig. 1). With in situ condensation (fig. 3), we were able to identify four phases. Phase 1 (blue) is the reference measurement at 675°C under N_2, there is no condensation at this stage. Phase 2 (red) is the condensation at 675°C under 200sccm of O_2. In phase 3 (yellow) we have increased the temperature to 700°C. And finally Phase 4 (green) is the relaxation of the 100% Ge layer. With GIXD, we can follow the in plane and out of plane component of the (224) Bragg reflection. It shows how the out of plane lattice parameter of the Ge rich layer gradually increases (due to higher Ge composition) while its in plane lattice parameter stays relatively constant (because the layer is still pseudomorphic) in phase 3 (yellow). We can also see a change of oxidation speed between phase 2 and phase 3 due to the temperature change. With the calculated lattice parameter, we were able to then estimate the Ge composition in the Ge rich layer and it showed how it increases from the initial 50% to around 100% at the end of phase 2. With further annealing (oxidation), the Ge rich layer starts to relax, this is evident from the increase of in plane lattice parameter in phase 4. However, the continuous increase of the out of plane lattice parameter is unexpected, as one would expect it to decrease according to the Poisson relation. A possible explanation is that when dewetting occurs one is left with embedded islands which may be under complex stress states (somewhat triaxial) and the simple Poisson relation between in-plane and out-of-plane may not hold anymore.
For sample 180417, we made sure that with the large area detector we always scan through both the (224) Bragg peak of the Ge rich layer and that of the Si substrate. The peak of the Si substrate is then used as a reference to calibrate the thermal expansion and any sort of sample misalignment. For this sample, we started again with an already condensed SiGe 50% layer (the ultrathin Si layer of the SOI has been totally consumed). However, instead of reaching the end of the condensation process (like sample 170417), we decided to stop the oxidation half way (after about 2000 seconds) and proceed with further annealing under N_2. The results (see fig. 4) is also quite intriguing. The in plane and out of plane lattie parameters behaved as expected in the oxidation phase (yellow). However, once N_2 is introduced in the furnace and O_2 pumped. We saw an decrease in the out of plane lattice parameter while the in plane lattice parameter stayed constant. This is once again against our expectations. One would expect the in plane lattice parameter to increase if the Ge rich layer dewets into relaxed islands. Else, one would expect the out of plane lattice parameter to remain constant if the Ge rich layer dewets into pseudomorphic islands. Both of which were not observed here. A possible explanation here is that the Ge rich layer has indeed dewetted into pseudomorphic islands, but the complex stress states makes it that the simple Poisson relation does not hold anymore.
With in situ GIXD it is possible to measure precisely the in plane and out of plane lattice parameter with a fine temporal resolution. While the final interpretation is still under debate among our group, we are confident that in situ X-ray measurement is the ideal tool for us to finally understand this complex process.
To summarize :
(1) We were indeed able to follow in real time the changes in composition, strain, thickness, position of the Ge rich layer

during oxidation with a combination of synchrotron X-ray scattering techinques.
(2) We were able to study the effect of temperature by performing condensation on four different temperatures.
(3) We did not observe any compliance of the UTSOI substrate in our measurement.
(4) With in situ measurement we were able to determine the critical thickness of the Ge rich layer.

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

With our team members taking shifts, we were able to meaure a total of 10 samples (as opposed to the 7 that we originally expected). For each sample, we were able to perform successful in situ GIXD, XRR and GIMAD (GIMAD was not performed on all samples in order to save time) measurements at different temperatures. We were able to reproduce the same condensation process as we had in our home lab with a different sample environment (furnace). The results are intriguing, and by understanding them we hope to finally understand the fascinating Ge condensation process, and to apply them to prototype microelectronics fabrications.

Publication(s):
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