Standard Project

Experimental Report

Proposal title: ***In situ*** **X-ray investigation of the UHV-CVD growth of Ge nanowires (NWs) on Si(111) using Digermane**		**Proposal number:** 20120526
Beamline: BM32	**Date(s) of experiment:** from: 26/09/2012 to: 02/10/2012	**Date of report:** 12/02/2013
Shifts: 18	**Local contact(s):** Gilles RENAUD	*Date of submission:*

Objective & expected results (less than 10 lines):

We expected to carry out the first quantitative study on both the structural (evolution of stress relaxation during growth) and morphological (NW sidewall faceting) aspects of the VSS (solid catalyst) growth of Ge NWs on Si(111) substrate, and by comparing the result with our previous data collected during VLS (liquid catalyst) processes, to be able to provide complementary information to the present understanding of the fundamental differences between the VLS and VSS processes. We also expected to conclude on the size dependencies of the growth rate, growth direction as well as NW morphology.

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

1. We didn't follow exactly the original objective of the experiment because the cylinder of digermane, the precursor gas essential to the growth of Ge NW under UHV condition, has arrived three months (end of July) later than we expected (end of April as indicated in the proposal). As a matter of fact, we managed to append the gas to our injection system just two days prior to the experiement. Consequently, we decided to start with the VSS growth of Ge NWs on Ge(111) substrate instead, as the knowledge of both homoepitaxy NW growth (i.e. Ge/Ge from this experiment and Si/Si from previous ones) is prerequisite to the understanding of the heteroepitaxy ones (i.e. Ge/Si, the original objective).
The experiment was, despite the aformentioned incident, a pleasant success.

2. We were able to cultivate state-of-the art Ge NWs on Ge(111) samples (fig. 1, left). Most NWs were grown along the (111) direction with very few occasions of kinking. The NWs are strongly tapered (fig. 2, right) as is expected for growth at relatively high temperatures (T=330°C).

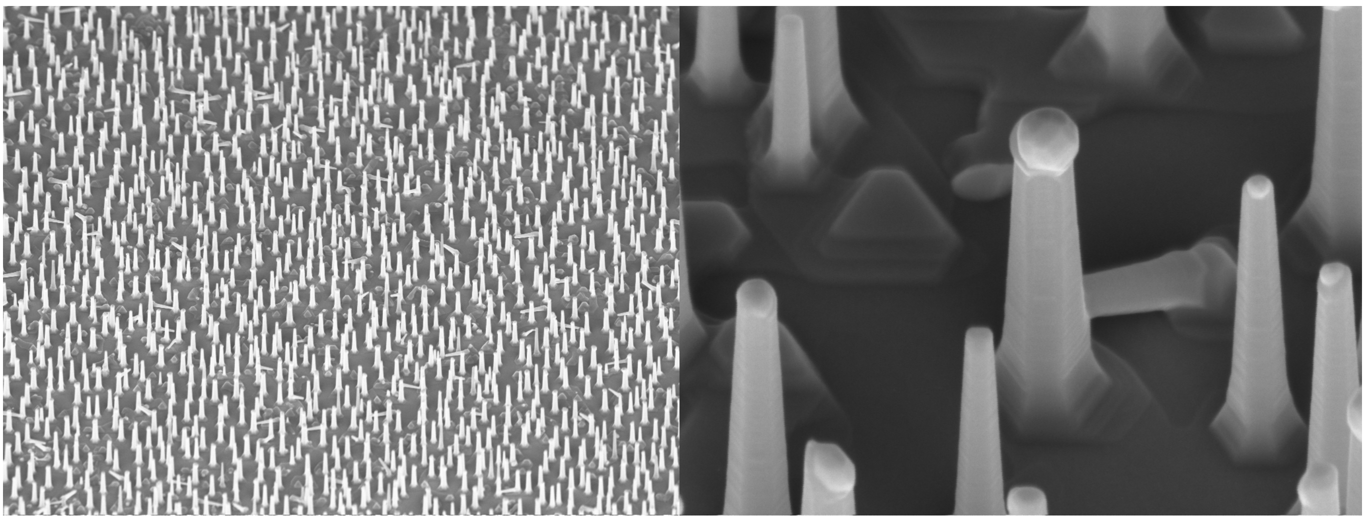

Figure 1: (left) ex situ SEM image of one Ge(111) sample with presence of large area of Ge NWs (right) the NWs grown are strongly tapered and faceted.

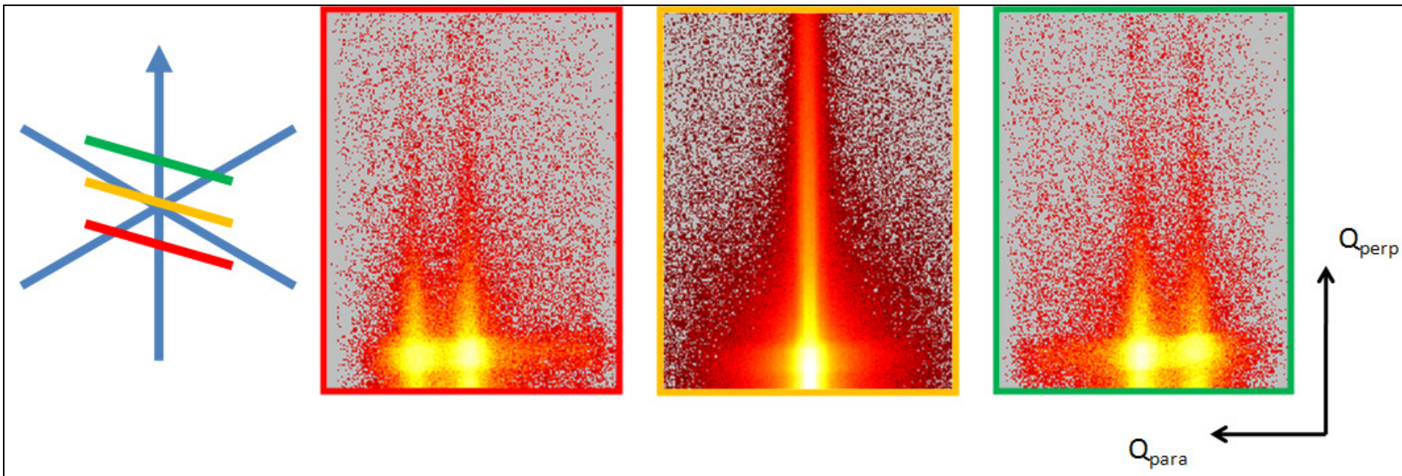

Figure 2: Results from a radial scan around an in plane Ge Bragg peak, (left) the arrow indicates the scan direction which itself is parallel to one of the facets, the in plane projection of the 2D detector is slightly tilted with regard to the scan direction, which intersects one of the two adjacent facets at closer-to-origin position, resulting in the appeareance of a double peak to the left (red) and to the right (green) of the center of the Bragg peak (yellow)

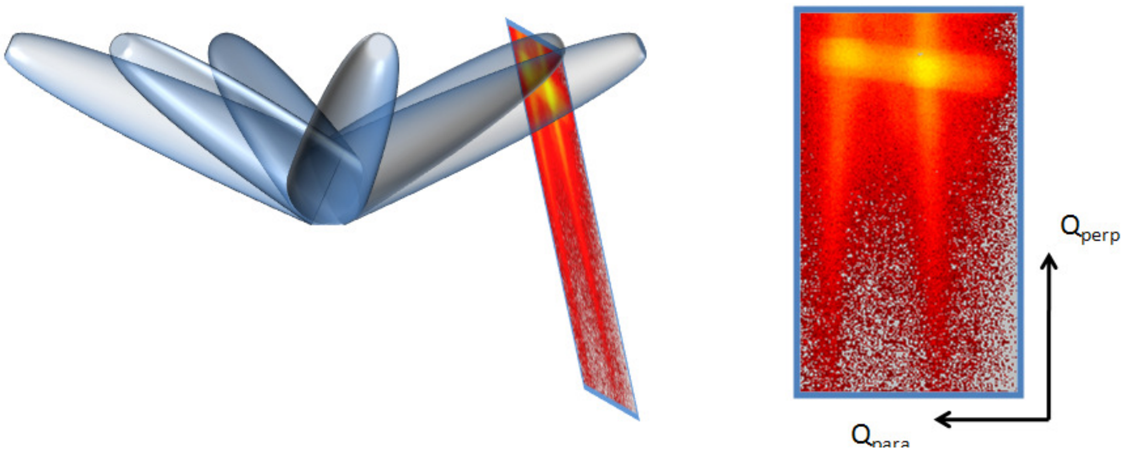

Figure 3: Results from a rocking scan around an out of plane Ge Bragg peak (left) schematic of how the detector intersects the faceting signal (right) the fact that the two peaks are found at different Qperp value is evidence to the fact that the faceting signal has an out of plane component.

The in plane (fig. 2) measurements show the presence of a six fold symmetric diffraction signal, which was further established to have a non-zero out-of-plane component by the corresponding out of plane (fig. 3) measurements. We believe that the signal arises from the large facet of the tapered NW sidewall which is only slightly tilted with regard to the surface normal. Detailed analysis is being carried out to confirm this hypothesis.

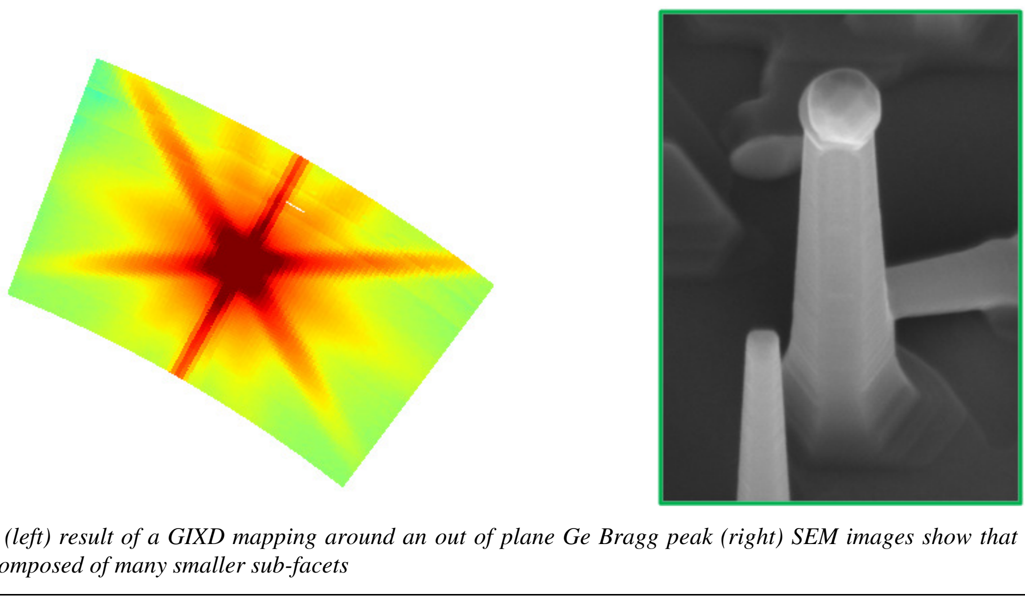

Figure 4: (left) result of a GIXD mapping around an out of plane Ge Bragg peak (right) SEM images show that the tapered NW sidewall is acutally composed of many smaller sub-facets

Out of our expectation, we found the diffraction streaks to be dodecagonal (instead of hexagonal) with a GIXD mapping around an out of plane Ge Bragg peak (fig.3 left), the origin of which might be the sub-facets observed from the ex situ SEM images (fig. 3 right) but awaits further confirmation.

We believe the aformentioned sub-facet signal could be better understood thanks to GISAXS measurements where some very broad faceting signals (small facets in real space) have been recorded during a GISAXS mapping (fig.5 left). (The analysis of the GISAXS data is still in process.) It is nevertheless worth mentioning that the NW length can be deduced from the length modulation as has previously been applied to the cases of Si NW/Si.

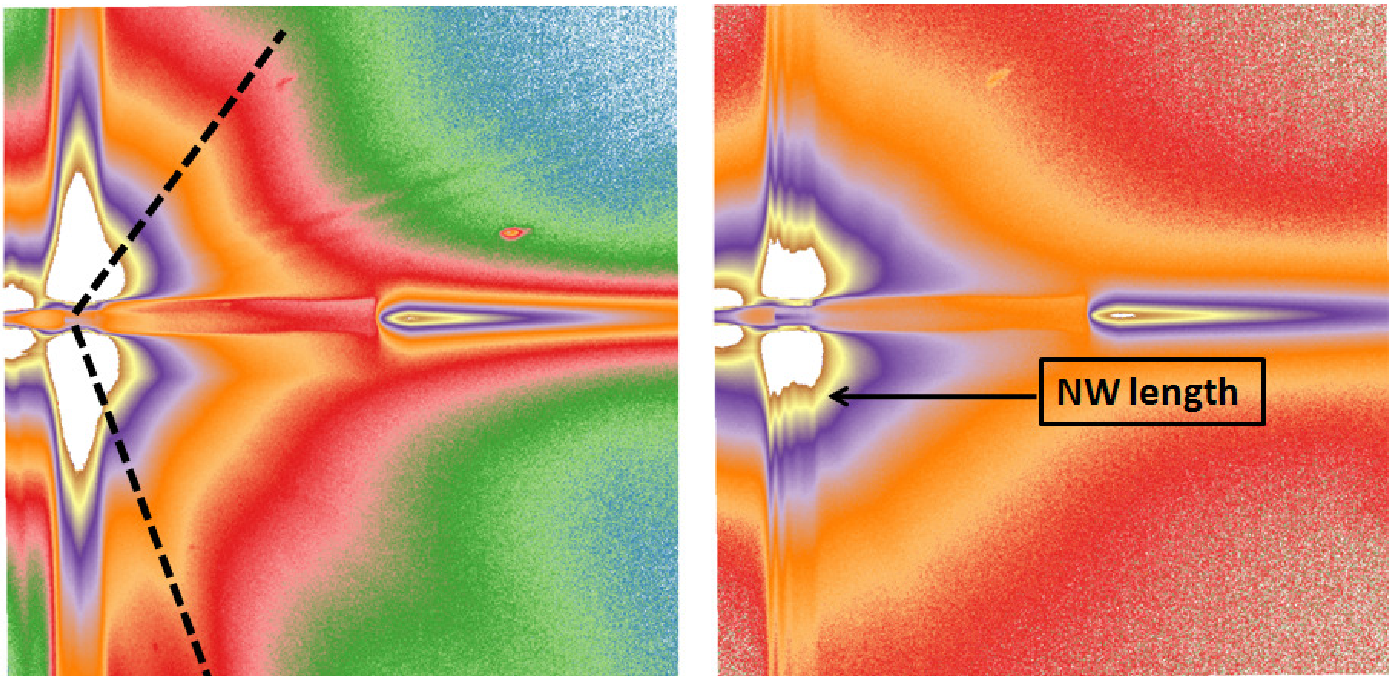

Figure 5 : (left) GISAXS measurement show presence of very broad faceting signal (right) the NW length can be deduced from the modulation period from the GISAXS measurements

Finally and most importantly, whether the growth of Ge NW below Te (eutectic temperature) undergoes the VSS (solid catalyst) or the VLS (liquid catalyst) process is to the present day, still under debate. Our preliminary measurements (fig. 6) seem to show that the catalyst becomes solid under the presence of gas, which is contrary to what has been reported in the litterature. <u>For this purpose, a more dedicated experiement is being designed and submitted to the coming run.</u>

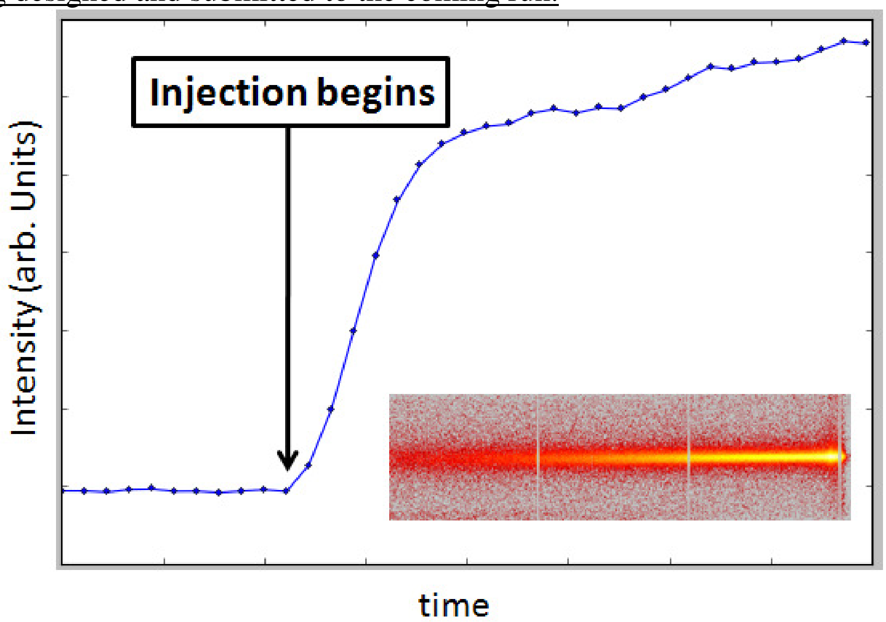

Figure 6: time scan on a "solid" Au peak during the injection (inset)the gold siganl seen by the 2D detector

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

IF-INS (BM32 ESRF) possesses, to our knowledge, the only system worldwide capable of delivering both CVD (group IV precursors) and MBE growth under UHV condition while performing *in situ* GIXD and GISAXS measurement at the same time.