ESRF

ESRF	Experiment title: **UHV-CVD growth of monodisperse Ge NWs on Si(111): sub-eutectic metastability, incubation time, catalyst out-diffusion, NW-substrate interaction, overall growth kinetics**	**Experiment number**: SI-2473
Beamline: BM32	**Date of experiment**: from: 18/07/0212 to: 23/07/2012	**Date of report**:
Shifts: 18	**Local contact(s)**: Gilles RENAUD	*Received at ESRF:*

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Objective

The objective of this experiment was to determine the parameter dependencies (for example, catalyst size, gas pressure, growth temperature) of the various phenomena observed during Ge NWs VLS/VSS growth on Si substrate, such as the temperature / gas pressure dependency of the AuGe catalyst droplet, the incubation time, catalyst out-diffusion, NW substrate interactions as well as the overall growth kinetics.

Background :

The IF-INS (BM32 ESRF) possesses, to our knowledge, the only system worldwide capable of delivering both CVD (Chemical Vapor Deposition) and MBE (Molecular Beam Epitaxy) growth under UHV (Ultra High Vacuum) condition while performing *in situ* GIXD (Grazing Incidence X-ray Diffraction) and GISAXS (Grazing Incidence Small Angle X-ray Scattering) measurement at the same time.

Until June 2012, the gas injection system is equipped with three different precursor gases: disilane (Si_2H_6), silane (SiH_4) and germane (GeH_4). Unfortunately, only the first precursor gas has proven itself to be sufficiently reactive under our restricted growth condition ($P_{chamber}$<5e-5 mbar, mainly to comply with ESRF's safety standards).

In situ x-ray scattering measurements on the VLS (Vapor-Liquid-Solid) growth of Si NWs using disilane have been carried out at BM32 ever since the optimal growth condition was found in Jul. 2011 (please refer to experiment report 32-03-704).

With the success with disilane, we can't help thinking of moving on to Ge NWs growth, but this time, by ordering a bottle of digermane, which is reported to be 10-100 times more reactive than germane, and is used by different groups across the world for NW growth under UHV condition.

Experiment

The objective of the experiment has not been attained.

As a matter of fact, the delivery of the digermane (from the U.S. to France via U.K.) took much longer than what we previously estimated, mainly due to the extremely high toxity of the precursor gas as a cargo to be shipped in normal containers.

Even though we finally managed to install the gas just before the experiment, we were left with no time for test growth, not to mention the time for finding optimal growth conditions.
With X-ray diffraction, we were able to observe the presence of Ge atoms once the gas injection started, i.e. the decomposition of the precursor gas at the sample surface was confirmed. However, the SEM image taken after growth was disappointing.
Fig. 1 shows one Si(111) sample after several hours of exposure to digermane.It is easy to arrive at the conclusion that the yield of the growth is rather poor and even though several NWs are present, they did not grow along the [111] direction.
Fig. 2 shows what we are capable of growing with disilane. (We expect to see very much the same with digermane). It is worth mentioning that it took us several months to find the optimal growth condition for disilane proper to our chamber.

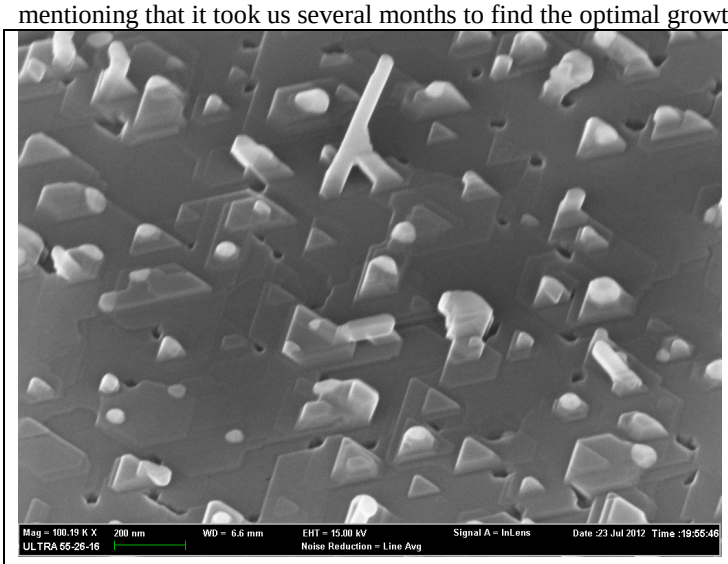

Figure 1 : SEM image of growth with digermane (Jul. 2012)

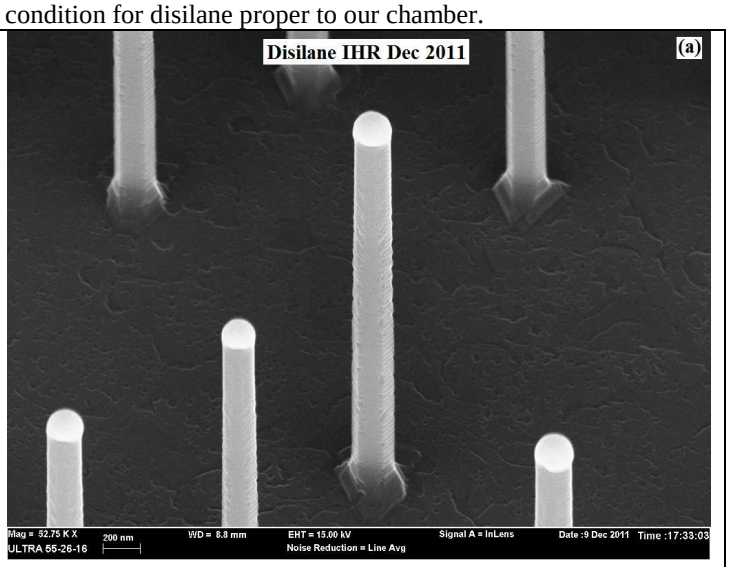

Figure 2 : SEM image of growth with disilane (Dec. 2011)

Outlook

One advantage of growing Ge NWs resides in the fact that they can be grown at much lower temperatures (~ 300°C) compared to that of the growth of Si NWs (~500°C). The growth mechanism is different too, as the catalyst alloy is in its solid state ($T_{growth} < T_{eutectic}$, Vapor-Solid-Solid mechanism) and not in its liquid state ($T_{growth} > T_{eutectic}$, Vapor-Liquid-Solid mechanism).
As a result, the optimal growth condition found for disilane no longer applies in the case of digermane. Nevertheless, keeping in mind the reported successful growth with digermane of other groups around the globe, and with our previous experience with disilane (please refer to experiment report 32-03-712), we are confident that, given more time for testing, the objective of this experiemnt will eventually be achieved.