ESRF	Experiment title: EXAFS analysis of the Ge-environment in $Ge_xC_ySi_{1-x-y}$/Si(001) epitaxial single layer with compressive null and tensile average lattice strain.	**Experiment number:** CH 305
Beamline IF D32	**Date of experiment:** from: January 18th to 21th and 23th,1996	**Date of report:** Feb 25th 1997
Shifts: 12	**Local contact(s):** J. L. Hazemann and C. Revenant Brizard	*Received at ESRF:*

Longer Report

Here, we report on the EXAFS experiment performed for the second time at beam-line IF with Total Electron Yield (TEY) detector on a series of $Si_{1-x-y}Ge_xC_y$ samples. In the following we are going to analyse what is necessary for the exploitation of the experiment, why the detector used should satisfy all the requirements of the experiment and what worked, and what didn't, during the experiment. A paper describing TEY detector is enclosed. First of all must be remember that necessary conditions to exploit the experiment are the following:

1) Collection of EXAFS spectra with polarization of the electrical field parallel (horizontal configuation=H) and perpendicular (vertical configuration=V) to plane interface. In fact, differences among the two configurations concerning different distances of NNN in or out of the plane parallel to the interface allow to get relationtionships with the tetragonal distortion of the unit cell.

2) Collection of the EXAFS spectra at liquid nitrogen temperature in order to reduce termal vibrations

3) Rotation of the sample during spectra collection in order to avoid the presence of spurious Bragg peaks.

Point 1 is perfectly satisfied because the sample-holder can be easly rotated without changing other the experimental parameters.

Setting: At begining we found the same problem of beam alignment found during the first experiment inducing a non correct measurement of the incident photocurrent, I_0. On the basis of the previuous experience this problem was solved rather quickly.
Afterward, all our effort was devoted to a correct measurement of the TEY detection, i.e. Il. First of all, we checked the dark noise of I_1/I_0 with and without the rotation of the sample holder at room-temperature. We found the noise with rotation was a factor 10

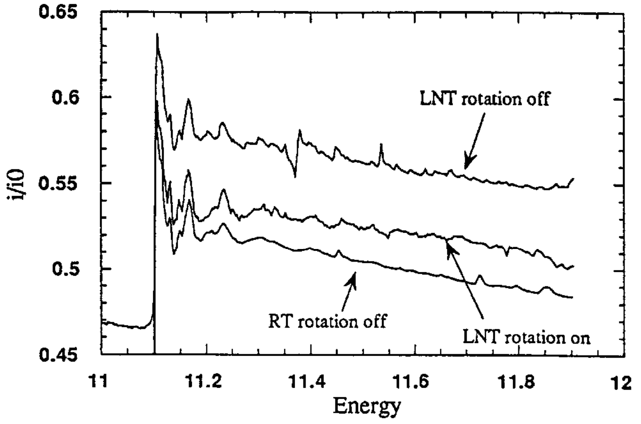
grater than in no-rotating case. Therefore, a clear problem of noise must be addressd to the rotation. Secondly, the noise to signal ratio was checked with photon-beam using integration time longer than rotation period (2 seconds) to avoid only partial suppression of spurious Bragg peaks. Nevertheless, again the I_1/I_0 value, 1.3%, was an order of magnitude grater than reference value without rotation 0.12%.

In the following we cooled down the system to liquid nitrogen.

The detector works at atmospheric pressure in atmosphere of cooled helium injected directly in the scattering chamber that is not vacuum isolated by air. The helium flux is devoted to perform thermal conduction between sample and the wall of the scattering chamber, avoid the presence of of water steam and therefore of its frezeeing and, finally, reduce the electrons neutralization. As a matter of fact, the helium flux and the correct procedure of cooling (followed according to the experience of the first experiment of June 96) avoided the formation of ice crusts on the walls of the scattering chamber's . On the contrary, the freezing of the humidy on the sample-holder cannot avoided during its re-introduction in the cool scattering chamber after the change of the sample at room temperature. As a consequence, the formation of small ice crystals produced an ageing of the rotation gears that, as a matter of fact, stopped many times. On line reparations of the rotations mechanisms allowed only a partial functionality recovery of sample holder that rotating with many vibrations and releases increased the overall noise to signal ratio.

In fig. 1 are compared the EXFAS signal of the same spectrum collected at RT without rotation and at LNT with and without rotation. The spectrum at RT looks good nevertheless the low statistic even if small Bragg peaks already appear. On the contrary, cooling the sample, a large Bragg peaks appear due to the reduction of the thermal atomic vibration. Finally, fig. 1 show that with rotation all the Bragg peaks disappers but the spectrum appears quite noisy with artificial superimposed periodicity due to rotation. For these reason we collected some EXAFS spectra at RT but no one of at LNT was reasonable.

Developments of the TEY sample holder are work in progress. In order to reduce the noise, we replaced the metallic contacts under the rotating stage by graphite contacts. Moreover, we are going to replace all the mechanics by a steel one in order to prevent an exagerate aging of the mechanical parts. Specialized technician of the in-house research beamtime division on the CRG/IF beamline will test these changes.



Scientific report. One the scientific aims of this experiment was the investigation of the coupling between Ge and Carbon. Some of us have shown that the strain release efficency of carbon is higher than that predicted following Vegard's [1]. This result could be explained with an enhanced probability of Ge-C coupling. If carbon and germanium occupy random position then the probability to have carbon as NN of Ge bonds should be equal to the carbon atomic concentration, 4y. On the contrary, if each C atom were coupled with at list one Ge atom then the number of Ge-C bonds should be equal to 4y/x (y/x=0.148 with y=2 and x=13.5%. x is the same for all the samples). The only information that we can get from the few RT spectra concern only first shell and a complete data reduction is working in progress. However preliminar results show that the number of carbon NN is quite close to 4y/x, confirming the supposed enhanced probability of Ge-C coupling.

Summary. The problems concerning the beam-line alignment and the correct cooling procedure of the detector characterizing the first experiment have been overcome. Vibration and electrical noise worsening the signal detection have been pointed out during the second experiment. We are goning on a deep revision of the sample-holder considering structural developments that allow TEY detection in vacuum atmosphere. Preliminar results show a very intersting preferential coupling between Ge-C. Because the aims of experiment have not been obtained because of technical problems, we apply for other beam time as soon as possible. We confirm that TEY is the right detection system for this experiment, however other shifts can be allocated only after the work in progress developments will be tested or, insted, considering as alternative effective detection the flourescence mode. In the last case, both IF and GILDA beam-lines are equipped for multi detectors fluorescence detection that could be a valid alternative to sample rotation to avoid Bragg spuriuos peaks.