ESRF	**Experiment title:** Time-resolved reflection XAS to probe the growth of semiconductor heterostrucutres: a feasibility study	**Experiment number:** MI - 474
Beamline: ID24	**Date of experiment:** from: 27-9-00 to: 2-10-00	**Date of report:** 9-2-01 *Received at ESRF:*
Shifts: 15	**Local contact(s):** S. Pascarelli	

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Report:

We have performed a feasibility study for applying time-resolved XAS to the investigation of the growth process of Ge dots on Si substrates. Our goal was to assess the potential of beamline ID24 (the dispersive XAS beamline at the ESRF) to tackle growth kinetics studies on surfaces, using a newly developed experimental setup based on dispersive XAS in the reflection mode. This has become possible thanks to recent technical developments, aimed at increasing the beam stability on one side (installation of the local horizontal feedback on the ID24 straight section) and at decreasing the vertical spot size on the sample on the other (implementation of a third re-focusing mirror in the vertical plane).

In the allocated beam time we worked on the optimization of the sample alignment and data acquisition procedure. We tuned the polychromator at the Ge K-edge (E ~ 11.1 KeV), and focused the beam to 30 μm x 30 μm (FWHM). The sample (typically a few mm^2) was fixed on a double goniometer at grazing incidence angles $1 \leq \theta$ (mrad) ≤ 4 with respect to the beam, at a distance of 0.6 m from the center of the re-focusing mirror. The CCD detector was positioned D=1.7 m behind the sample. Particular care was taken to adjust the angle of rotation of the plane of the sample around the axis of the beam, in order to intercept all the energies of the polychromatic fan at the same grazing incidence angle.

Three samples were investigated:

sample 1: an oxidized Ge wafer (with an oxide layer d_{GeOx} ~ 110 A, as measured by ellipsometry)
sample 2: a thick (~ 480 A) Ge layer on Si(001)
sample 3: a thin Ge layer (~ 70 A) with Ge dots on Si(001).

The first tests were aimed at establishing our surface sensitivity. Figure 1 shows calculated reflectivity curves at $E=11.1$ KeV as a function of grazing incidence angle, for different Ge layers on Si. The figure shows that different depths of sample can be probed by adjusting θ between 1 and 4 mrad.

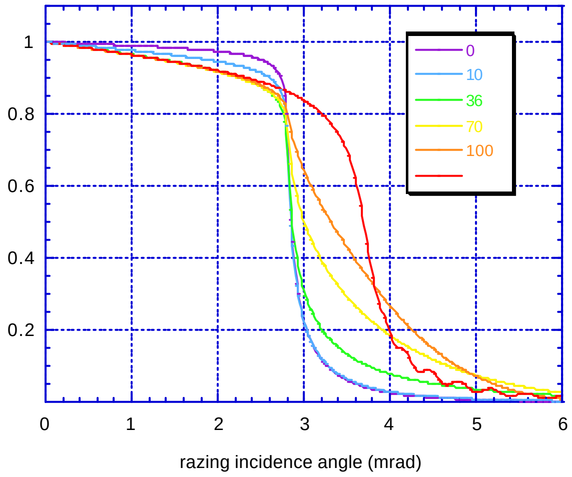

Figure 1: Calculated reflectivity curves for different Ge layers (thicknesses in A) on Si

Figure 2 shows a Ge K-edge spectrum (360 ms aquisition time) on sample 1. The small shoulder on the high energy side of the edge corresponds to the absorption onset of the GeO_x surface layer. The inset on the right shows that by adjusting θ between 1 and 3 mrad the intensity of this shoulder varies according to the portion of oxide layer probed.

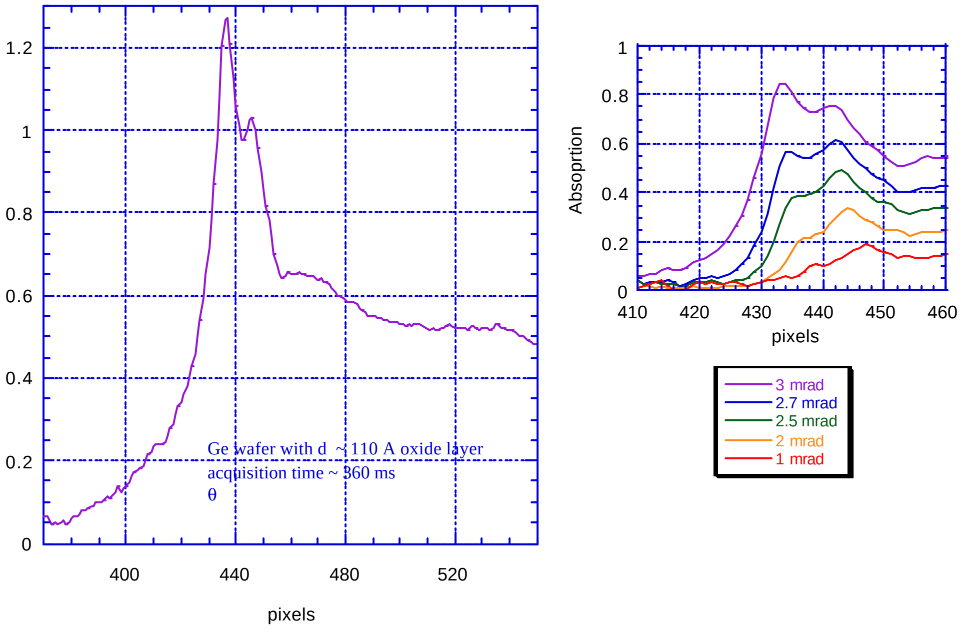

Figure 2: Ge K-edge spectrum on sample 1. Inset on right: variation of edge features with q.

In Figure 3 we plot spectra relative to sample 3 for different values of θ. The absorption of the 70 A Ge layer is maximized for $\theta < 3$ mrad, as expected. In both cases (Figures 2 and 3) the spectra have a very limited energy range (ΔE max above the edge ~ 100 eV), due to non-statistical noise arising from a lack of proper normalization between the I_0 and I_1 intensities (this will become clear below where we describe the data acquisition method).

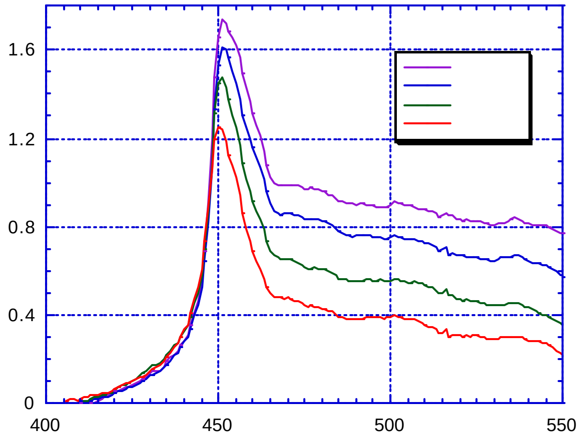

Figure 3: Ge K-edge spectra on sample 3 for different grazing incidence angles q.

In the second part of our tests, we tried to evaluate our sensitivity to detect early structural modifications around Ge as it is deposited on the Si substrate. For this, we used as reference the Ge K-edge spectra relative to 2 model situations: Ge bulk and Ge impurities embedded in Si, Ge:Si (Figure 4). This figure shows strong modifications of the near-edge structures in the two limit situations, and at the absorption onset the relative difference between the spectra [Ge:Si -Ge bulk] / [Ge bulk] reaches values of the order of 50%, due mainly to a shift of the edge towards lower energies.

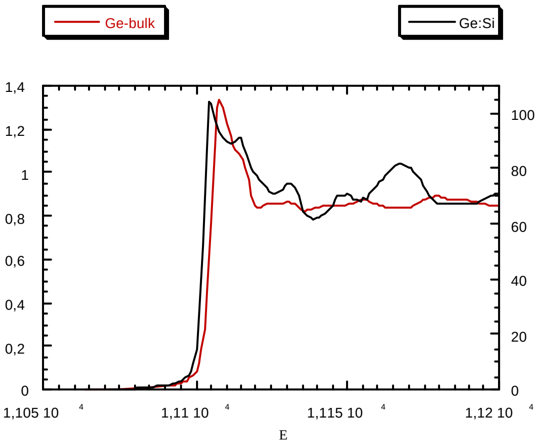

Figure 4: Ge K-edge spectra relative to 2 model situations: Ge bulk and Ge impurities embedded in Si, Ge:Si

Note that the energy range of Figure 4 is comparable to those of Figures 2 and 3: this indicates that even with such a restricted energy range it should be possible to obtain detailed information on the growth kinetics, if the noise level is low enough and if the time resolution for data acquisition can be matched to the relevant timescale for growth.

Our time-resolved data acquisition procedure consisted in:

i) record an I_{01} spectrum through air
ii) vertically translate the sample upward to intercept the beam and lift the CCD detector an amount 2θD to intercept the reflected beam.
iii) record $(I_1)_i$ spectra reflected by the sample with i=1,N.
iv) vertically translate the sample downward to remove it from the beam and lower the CCD detector an amount 2θD to intercept the direct beam.
v) record an I_{02} spectrum through air
vi) perform ln $[I_{0i}/(I_1)_i]$, where I_{0i} is obtained by a linear interpolation of I_{01} and I_{02}.

During these feasibility tests, no treatment was applied to the sample, so the time-resolved tests were uniquely devoted to assess the best time-resolution we could obtain with a reasonable S/N. Acquisition times of the order of 200 – 500 ms were used for the $(I_1)_i$ spectra. Figure 5 shows the difference signal between two successive I_1 spectra on sample 2, with an acquisition time of 270 ms each. The total time elapsed between the two acquisitions is 490 ms since it includes the readout time of the CCD camera (220ms). Figure 5 shows a peak-peak noise level in the difference signal lower than 1%. This noise level is sufficiently low to enable the detection of the first few A of Ge during growth.

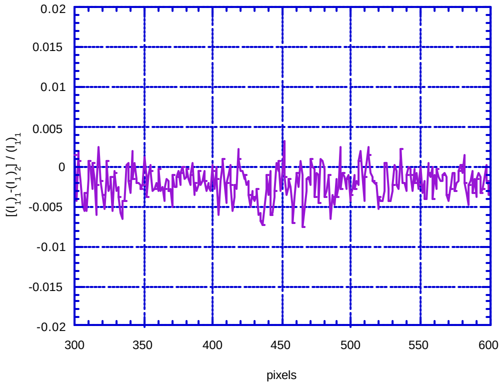

Figure 5: difference signal between two successive I_1 spectra on sample 2, with an acquisition time of 270 ms each