

**Experiment title:**

Time-resolved reflection XAS to probe the growth of semiconductor nanostructures: a feasibility investigation

**Experiment number:**

MI 587

<b>Beamline:</b> ID24	<b>Date of experiment:</b> from: 20/06/2002 to: 24/06/2002	<b>Date of report:</b> 28/02/2003
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**Report:**

We have performed a feasibility study for applying time-resolved XAS to the investigation of the Ge film on Si substrates during annealing at temperature of the order of Ge dot formation. 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.

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 ( $E \sim 11.1$  KeV), and focused the beam to  $30 \mu\text{m} \times 30 \mu\text{m}$  (FWHM). The sample (typically a few  $\text{mm}^2$ ) was fixed on a double goniometer at grazing incidence angles  $1 \leq \theta$  (mrad)  $\leq 4$  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.

The investigated samples were:

sample 1: an oxidized Ge wafer (with an oxide layer  $d_{\text{GeO}_x} \sim 110$  Å, as measured by ellipsometry)

sample 2: a thin planar Ge film 1.5 nm thick (sample 910)

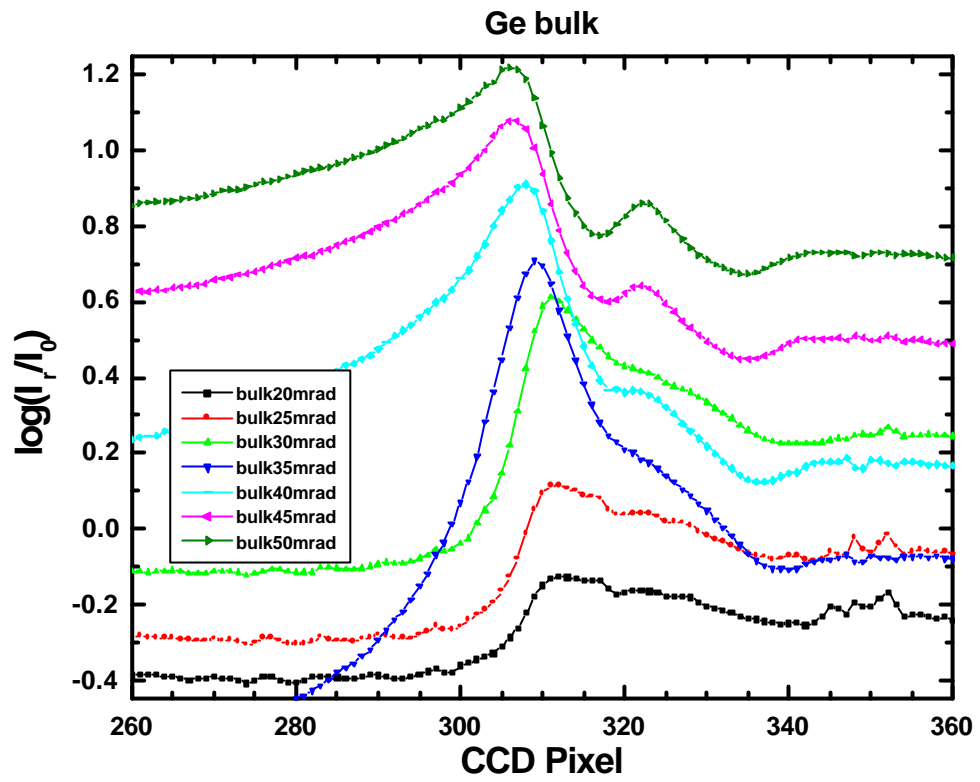
sample 3: Ge dot su Si(001) grown at  $T=500^\circ\text{C}$  (sample # 906, Deposition time= 160s).

sample 4: Ge dot su Si(001) grown at  $T=650^\circ\text{C}$  (sample # 907, Deposition time= 90s).

sample 5: Ge dot su Si(001) grown at  $T=650^\circ\text{C}$  (sample # 908, Deposition time= 180s).

The samples 3 and 4 have the same nominal equivalent Ge thickness.

As first step the samples were analyzed before annealing. The measurements were performed as a function of the incidence angle. The Ge dot sample were investigated in order to compare the Ge dot create during the post growth annealing with the structure and composition of Ge islands nucleated directly during the growth.



The obtained results are reported in the following figures

Fig. 1: Ge K edge spectra on Ge bulk (sample 1) for different incidence angles. The curves are shifted along the y axis for clarity.

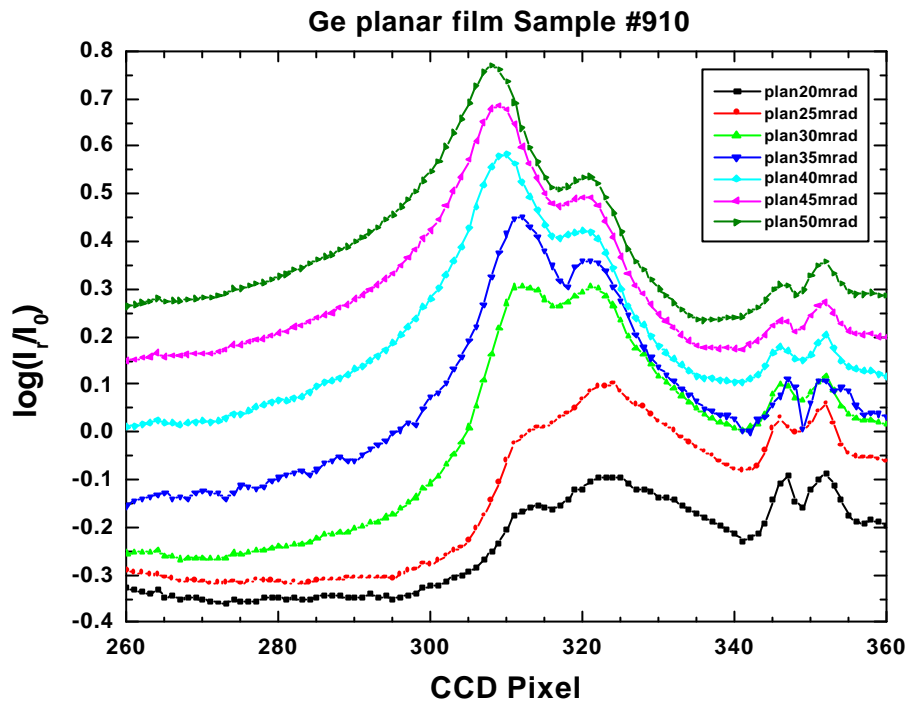


Fig. 2: Ge K edge spectra on planar Ge film grown on Si(001) (sample 2) for different incidence angles.

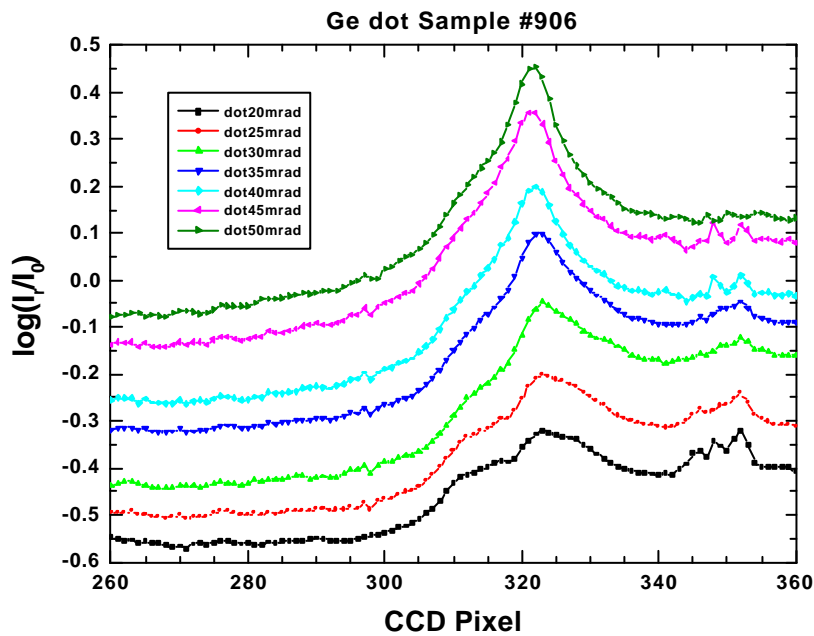


Fig. 3: Ge K edge spectra on Ge dots deposited on Si(001) (sample 3) for different incidence angles.

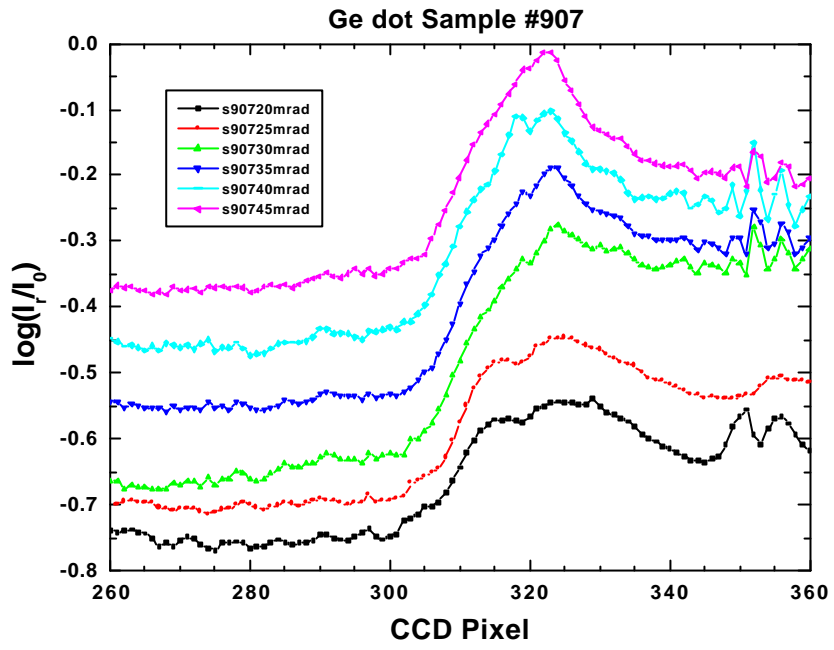


Fig. 4: Ge K edge spectra on Ge dot sample for different incidence angles.

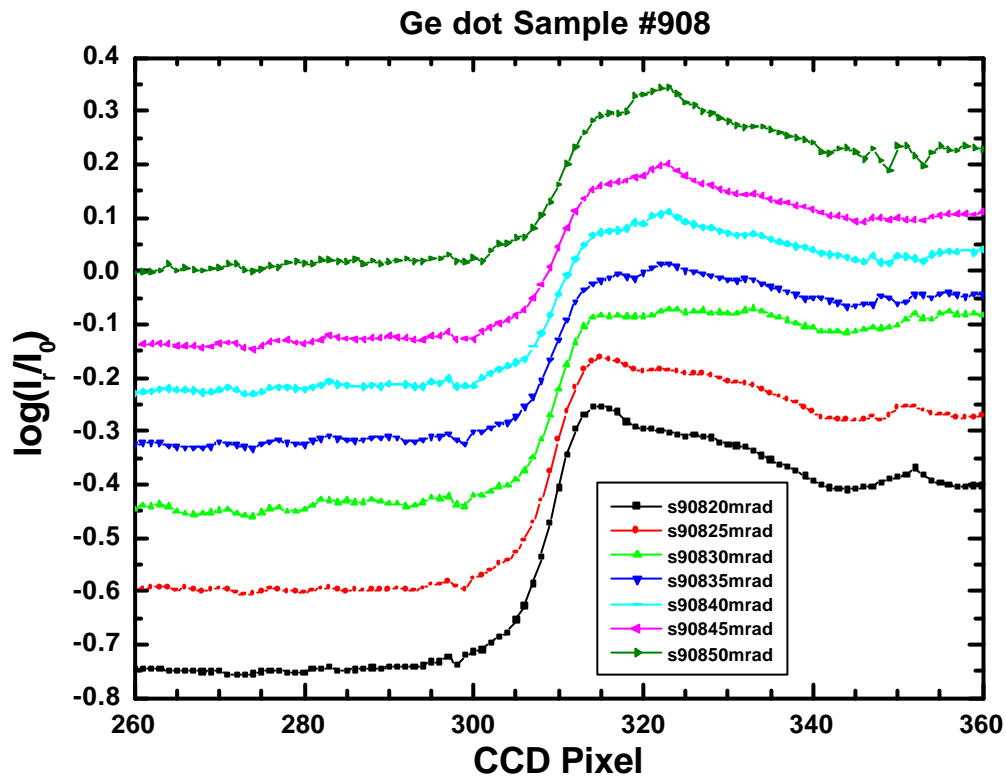


Fig5: Ge K edges of Ge dots on Si(001) (sample 908)

In the second part of our experiment we tried to perform time resolved measurements during the annealing of the Ge planar film. The annealing was performed in vacuum.

It was not possible to collect significant data during the annealing because during heating thermal volume variation together with mechanical clamping determine misalignment.

The acquired XAS data on samples before annealing are going to be analyzed