ESRF	**Experiment title:** GISAXS kinetic evolution of Ge quantum dots self organization	**Experiment number:** Si-689
Beamline: ID32	**Date of experiment:** from: 12/06/01 to: 02/07/01	**Date of report:** 10/07/01
Shifts: 48	**Local contact(s):** Bruce Cowie	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
D. Buttard*, J. Eymery*, G. Guenot*, R. Lazzari*, F. Leroy*, M. Noblet*, G. Renaud*, O. Ulrich*
CEA Grenoble, *Département de Recherche Fondamentale sur la Matière Condensée, Service de Physique des Matériaux et Microstructures*
17 rue des Martyrs, 38054 Grenoble Cedex 9

Report:

Aim of the experiment : Sub-surface dislocation networks realized by wafer bonding, have been shown to be efficient to control both size and density of quantum dots (QDs). Our samples are Si bicrystals, obtained by ¨ Direct Wafer Bonding ¨ on full 4-inch wafers. The upper crystal is thinned to about 10 nm to increase the interface bonding strain field to the surface. Accommodation of the disorientations of molecular bonding is done by two kinds of dislocation networks : a twist of two crystals around the [001] axis induces a square network of pure screw dislocations, whereas a tilt angle (miscut of the surface) induces 60° dislocations. STM previous experiments have shown the first results of lateral self organization of Ge QDs grown onto these surfaces. It has been proposed in this experiment to study with GISAXS the initial substrate and the *in situ* growth of Ge QDs.

Experimental setup : The study was performed *in situ*, under UHV (base pressure: 10^{-9} Torr), with different sources for molecular beam epitaxy : silicon, and germanium. We have used a $1242 * 1142$ pixels CCD camera (CRG/IF) to acquire GISAXS pictures. It took us roughly one of the three weeks to complete the installation in the experimental hutch (MBE chamber, CCD camera and under vacuum beamline elements).

Measurements : To prepare the ID32 experiment, we had performed first GISAXS measurements on the SUV setup of the BM32 beamline. This preliminary experiment has shown that GISAXS was well suited to observe the growth of Ge QDs onto our sample, including the 2D-3D Stranski-Krastanov transition. Different sample, with different tilt and twist angles, have been studied. Three families can be distinguished (A, B and C) Samples A: pure twist, Si upper layer thickness 100 Å. Samples B : twist and tilt, upper layer thickness 90 Å. Samples C: Si upper layer thickness 100 Å, twist about 5° and tilt about 1.4°, with 12 Å thick oxide and Si nanocrystals deposited by LPCVD. We have started experiments by performing GISAXS measurements at room temperature. Samples A and B have been annealed and Ge was deposited at about 350 °C, up to 15 Å, by 1 Å steps. GISAXS measurements were performed between each growth step. After last step, we have performed annealing to observe the evolution of Ge QDs (Ostwald ripening). Samples C were annealed up to 850°C, to observe the behavior of Si nanocrystals deposited on SiO_2.

Preliminary results : For sample A (1(a)), we can see truncature rods in the [-110] direction, as well as in the [110] direction. This group of rods is assigned to the square dislocation network of twist, and we deduce from the period a twist angle of 0.998°, very close to the target of 1°. Figure 1(a) also shows diffusion from facetted objects, which is attributed to the roughness of the upper surface. The angle between specular beam and diffusion from the facets is roughly 56°, which corresponds to $< 111 >$ planes. It is

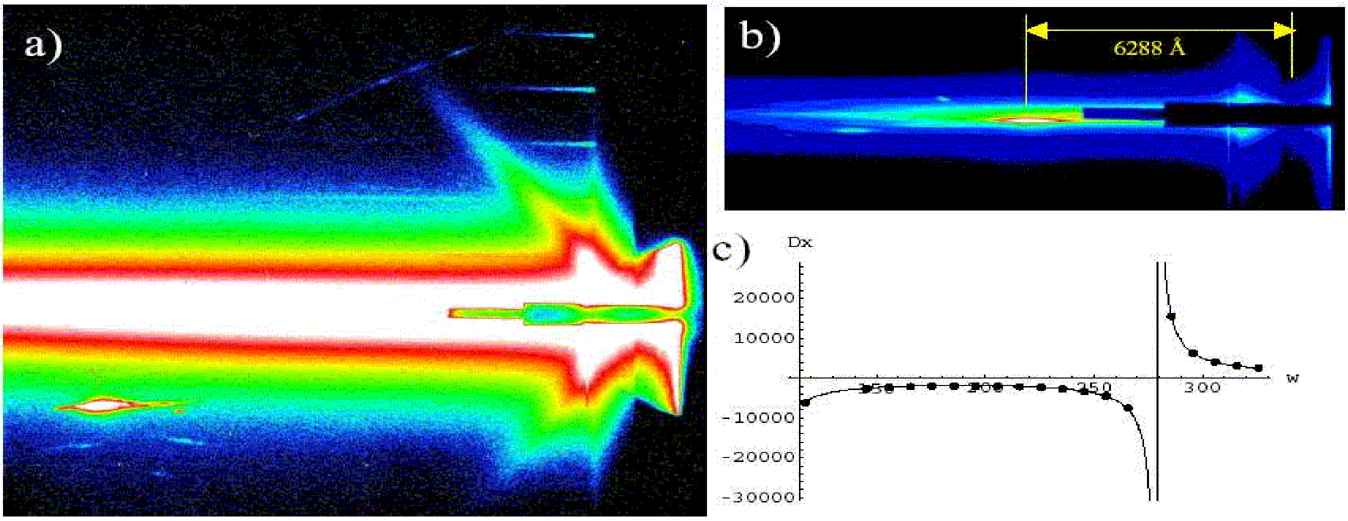

Figure 1: a) Sample A. Truncation rods from the twist square dislocation network can be seen, as well as diffusion from [111] facets. b) Sample B. Measurement of q_x momentum transfer. c) Sample B. Experimental data for $D_x(nm)$ versus ω and modeling.

not yet possible to determine unambiguously whether the truncation rods signal can be attributed to the electronic density variation of dislocations core, or to the surface correlated roughness (STM measurements have shown correlated roughness with a typically 8 Å amplitude). For samples B, we can observe, for every azimuth, a strong intensity enhancement close to the specular beam. This peak (Fig. 1(b)) is attributed to the q_x momentum transfer due to the tilt. Calculations of the distances related to this feature are shown in the figure 1(c) as a function of the azimuth angle ω (dark points), and simple modeling is also shown on this figure (dark line). The period measured by q_x variations is very close to the tilt induced roughness period observed with STM.

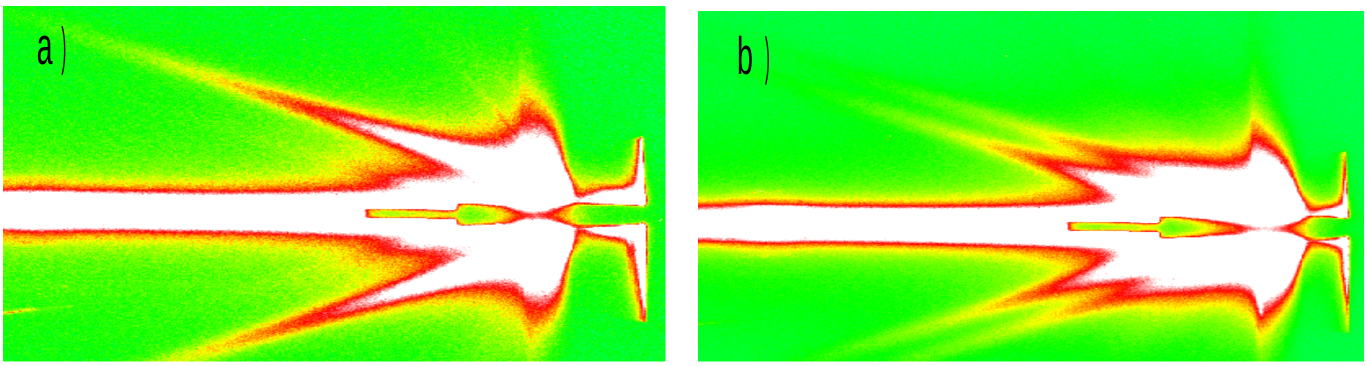

Figure 2: Post growth annealing. Illustration of DWBA with incident angle a) $\alpha = 0.3°$ and b) $\alpha = 0.2°$

We have grown Ge on samples A and B, at substrate temperature 350°C, by equivalent thickness steps of 1 Å. We have observed classical Stranski-Krastanov growth mode, with a 2D wetting layer up to 6Å, followed by nucleation of 3D Ge QDs. Analysis of GISAXS pictures gives for these dots an height of 3 nm, a diameter of 5 nm, and a mean dot to dot distance of 10 nm. When we anneal these dots, we can see the apparition of facets. With an incident angle $\alpha = 0.3°$, facetted dots induce a single diffusion streak, whereas with an incident angle $\alpha = 0.2°$, close to the critical angle (0.18° for Si at $h\nu = 10keV$), there are two streaks (see Fig. 2 a) and b)). We attribute this doubling to a second order term of the DWBA theory.
For samples C, during annealing up to 850°C, we did not observe Si nanocrystals self organization, as opposite to previous STM experiments.

In conclusion : We have observe very interesting features of the scattering of bonded samples (dislocation scattering) and of dynamical effect induced by the growth of QDs. But it has been difficult to reproduce any self organization feature during this experiment. We attribute this fact to the difficulty to control the vacuum and the temperature in the setup, and therefore to control how clean were the surfaces.