

## Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.**

Once completed, the report should be submitted electronically to the User Office via the User Portal:

<https://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

### ***Reports supporting requests for additional beam time***

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

### ***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

### ***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

### **Deadlines for submission of Experimental Reports**

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

### **Instructions for preparing your Report**

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.



<b>Experiment title:</b> Growth of SiGe and GeSi core-shell quantum dot lattices in alumina matrix	<b>Experiment number:</b>
<b>Beamline:</b>	<b>Date of experiment:</b> from: 30.1.2013 to: 5.2.2013
<b>Shifts:</b> 18	<b>Local contact(s):</b> Dr. Carsten Baetz
<b>Date of report:</b> 27.2.2013  <i>Received at ESRF:</i>	

**Names and affiliations of applicants (\* indicates experimentalists):**

Dr. Maja Buljan, Institute Rudjer Boskovic, Zagreb, Croatia\*

Dr. Joerg Grenzer, Forschungszentrum Dresden-Rossendorf, Germany\*

Dr. Vaclav Holy, Charles University in Prague, Czech Republic\*

**Report:**

Core/shell semiconductor nanocrystals are widely studied nowadays because of their prospective application in photovoltaics and photoelectronics. The aim of the experiment was to study the growth of Ge/Si and Si/Ge nanoparticles Ge/Si/Al<sub>2</sub>O<sub>3</sub> and Si/Ge/Al<sub>2</sub>O<sub>3</sub> multilayers by magnetron sputtering by x-ray reflection and grazing-incidence small-angle scattering (GISAXS method) in situ during deposition.

For the deposition we used the magnetron growth chamber attached to the large sample goniometer at the BM20 beamline. For the growth we used two magnetron systems, namely a DC system for Si and Ge deposition with the power of 100W as well as rf magnetron for the deposition of alumina (20 W), Ar was used as a working gas. During the growth, the sample was irradiated by a focused primary beam with the wavelength of 1.083 Å. The scattered beam was detected by a linear detector MYTHEN1K mounted on the delta/nu arm of the goniometer in the direction perpendicular to the sample surface.

We deposited a series of multilayers on Si substrates with the periods Al<sub>2</sub>O<sub>3</sub>/Ge/Si and Al<sub>2</sub>O<sub>3</sub>/Si/Ge, in each deposition run we deposited 10 periods. During the growth we measured specular and diffuse x-ray reflection keeping constant the incidence angle  $\alpha_i = 1$ deg; one detector frame corresponds to the distribution of scattered intensity for various exit angles  $\alpha_f$  and the same azimuthal direction  $\nu = 0$ . Figure 1 shows the example of a time/ $\alpha_f$  intensity map measured during the growth of the sequence Al<sub>2</sub>O<sub>3</sub>/Ge/Si.

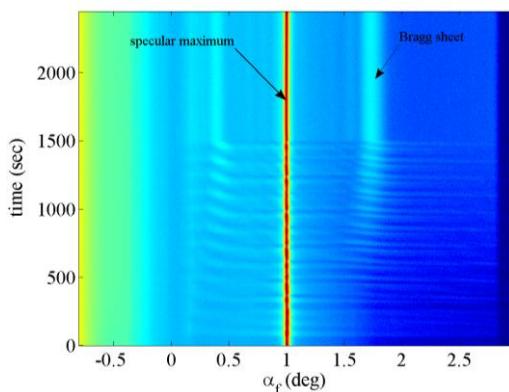


Fig. 1 time/ $\alpha_f$  scan measured during the growth of an Al<sub>2</sub>O<sub>3</sub>/Ge/Si sequence

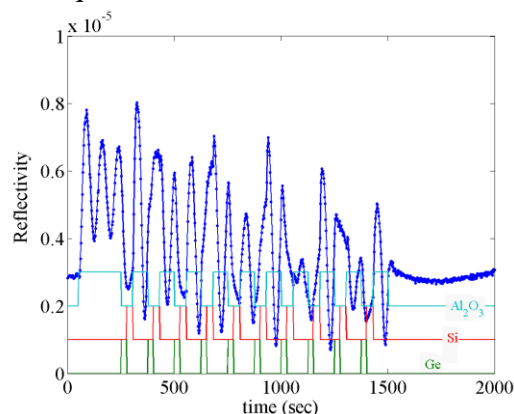


Fig. 2 The time dependence of the specular reflectivity extracted from the data plotted in Fig. 1

The sharp maximum in the intensity map at  $\alpha_i = \alpha_f = 1\text{deg}$  corresponds to the specularly reflected beam; broad stripes at  $\alpha_f \approx 0.4\text{ deg}$  and  $1.6\text{ deg}$  are the Bragg sheets caused by the correlation of the random roughness profiles at various interfaces in the multilayer [1]. From the intensity map we extracted the time-dependence of the specular reflectivity (Fig. 2).

After completion of the growth we measured a standard specular reflection curve (Fig. 3) and a GISAXS intensity map. For the GISAXS maps we used a nu-scan with constant incidence angle  $\alpha_i = 0.2\text{ deg}$  close to the critical angle of total external reflection. Each detector frame in the GISAXS measurement corresponds to a distribution of scattered intensity with given azimuthal angle  $\nu$  and various exit angles  $\alpha_f$ . Finally we converted the intensity distribution to reciprocal space (Fig. 4).

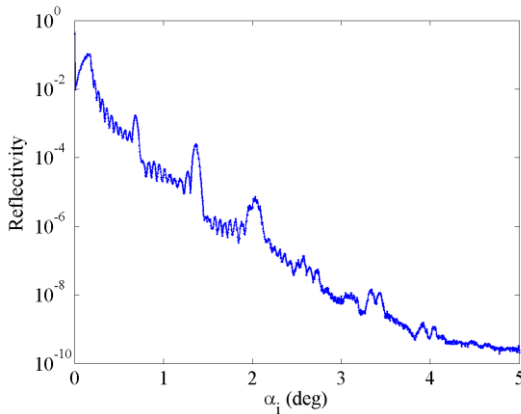


Fig. 3. Post-growth specular reflectivity curve

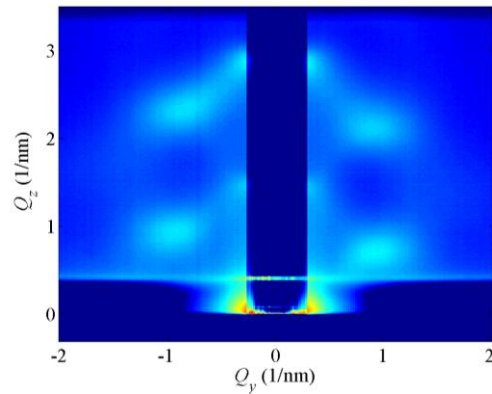


Fig. 4 Post-growth GISAXS map

Satellite maxima in Fig. 4 indicate the presence of a self-order three-dimensional array of nanoparticles; the apparent zigzag pattern of the maxima corresponds to an ABAB or ABCABC-type of particle stacking at subsequent multilayer interfaces.

After the growth we also measured a fast WAXS scan to detect the presence of crystalline particles. For this measurement we used the same linear detector and performed a fast delta-scan. Figure 5 presents an example of the WAXS scan along with the theoretical positions of the Ge and Si diffraction maxima

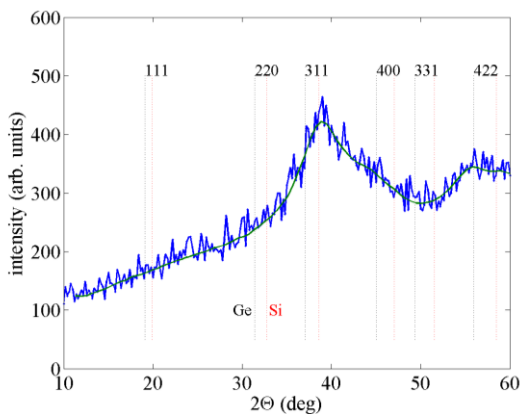


Fig. 5 The diffraction curve (WAXS scan) measured after the growth. The dotted line represents the intensity extracted from a single pixel of the linear detector; the line depicts the intensity integrated over all pixels.

The analysis of the experimental data is still in progress. From the data we determine the time evolution of the morphology of the growing surface and the evolution of the ordering of the nanoparticles during deposition. From the comparison of the results obtained during the growth of  $\text{Al}_2\text{O}_3/\text{Ge}/\text{Si}$  and  $\text{Al}_2\text{O}_3/\text{Si}/\text{Ge}$  sequences we will analyse the wetting/dewetting properties of Si/Ge and Ge/Si interfaces.

[1] Holý V., Baumbach T., Phys. Rev. B **49**, 10668 (1994).