



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.



Experiment title:
Creation of silicon nanocrystals in insulating multilayer matrices

Experiment number:
38114

Beamline:
BM25B

Date of experiment:
from: 19.11.2015 to: 23.11.2015

Date of report:
01.03.2016

Shifts: 12

Local contact(s):
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Received at ESRF:

Names and affiliations of applicants (* indicates experimentalists):

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Report:

This was our first experience using excellent facilities of ESRF.

Our main goal was to confirm the formation of crystalline silicon nanocrystals in annealed multilayered structures of initial composition (1) $\text{SiO}_x/\text{Si}_3\text{N}_4$; (2) $\text{SiN}_x/\text{SiO}_2$ and (3) $\text{SiN}_x/\text{Si}_3\text{N}_4$ obtained using PECVD technique. The samples contained from 10 to 40 pairs of layers with different thickness of active and barrier layers varying from 1.5 to 6 nm. The multilayered structures were grown on silicon substrates. As a reference sample we used $\text{SiO}_x/\text{SiO}_2$ structure with 40 pairs of layers, 5 nm thick active layers and 3 nm barrier layers. Annealing of the samples at temperatures up to 1150 °C should result in the self-organisation of silicon nanoclusters in non-stoichiometric SiO_x or SiN_x layers. The crystalline phase of silicon nanocrystals in the reference sample was demonstrated previously using different techniques.

The results of the GIXRD scans collected at a grazing incidence angle of 0.3° for multilayered $\text{SiO}_x/\text{SiO}_2$, $\text{SiO}_x/\text{Si}_3\text{N}_4$ and $\text{SiN}_x/\text{Si}_3\text{N}_4$ structures, annealed at 1150 °C are shown in Fig.1. Small overall thickness of the structures, small dimensions of the nanoparticles as well as the presence of silicon everywhere made the task non-trivial. Preliminary measurements of X-ray reflection confirmed that the contrast in the samples is sufficient to performed the measurements. The results presented in Fig.1 confirm the formation of silicon nanocrystals in all the matrices studied. As to our knowledge, this is the first time that the formation of silicon nanocrystals in silicon nitride matrix was demonstrated.

The measurements were performed on a number of samples of the series and for further investigations such parameters as the layer thickness and number were defined to ensure reasonable signal to noise ratio.

Our further plan was to use GISAXS technique to investigate possible ordering of the nanoclusters, however for our beamtime the CCD camera of the beamline was not available and with the camera borrowed from the pool we didn't see much. This is what we plan to apply for for the next beamtime.

We planned also to see annealing temperature dependence on the formation of nanocrystals, but didn't have time for that since annealing has to be done *ex-situ*.

The results of this beamtime are included in a paper, which is under preparation at the moment.

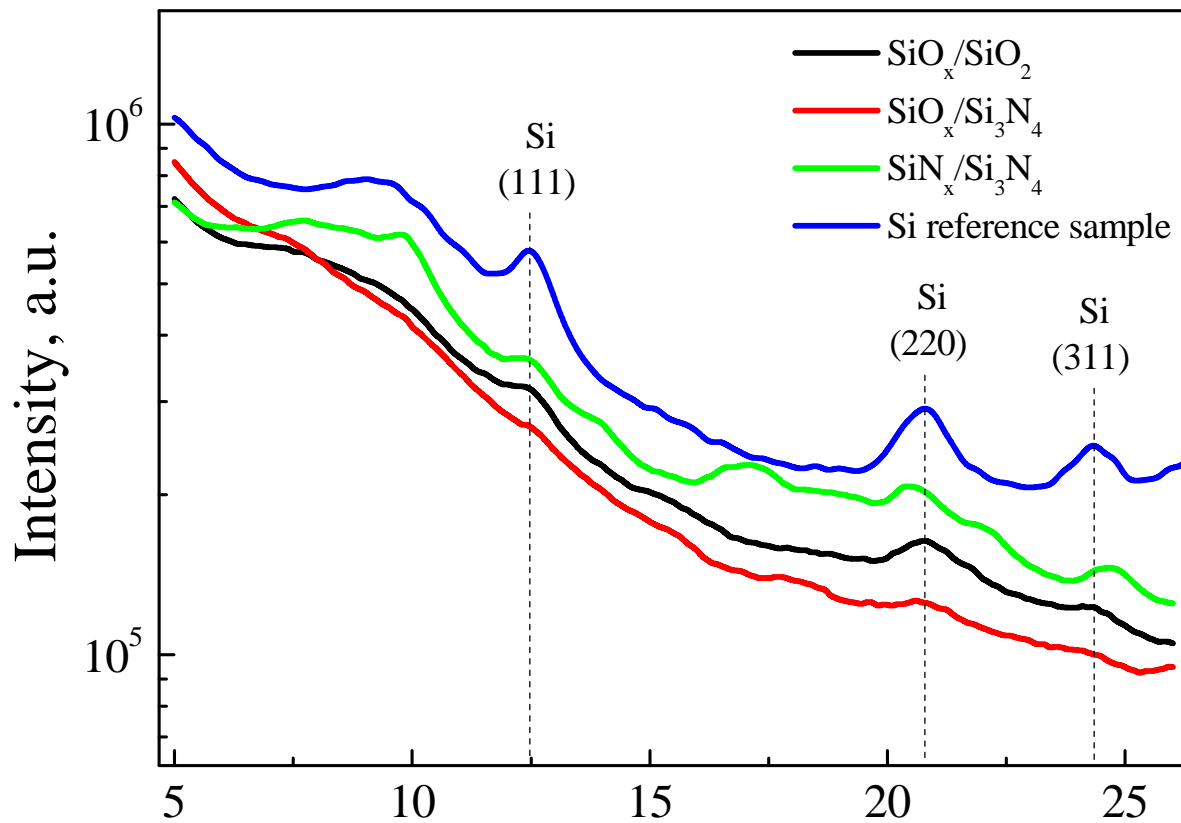


Figure 1. GIXRD results

