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ESRF

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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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.

ESRF	**Experiment title: Formation Mechanism and Instability of Porous Silicon Layer**	**Experiment number**: SI–475
Beamline: ID01	**Date of experiment**: from: 26/11/99 to: 30/11/99	Date of report**:** **25/11/99**
Shifts: 8	**Local contact(s)**: D. Le Bolloc'h	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The aim of this experiment was to study porous silicon structure with grazing incidence small angle x–ray scattering techniques. Due to its nanoporous structure, porous silicon has important physical properties, such as luminescence at room temperature in the visible range and well–defined optical index variations due to the modulated electron density [1]. The material is easily obtained by the anodisation of a single–crystal silicon wafer in a HF electrolyte and the properties of the elaborated layer can be well controlled by the adjustment of the fabrication parameters. But the formation mechanisms of porous silicon are not completely understood. For this reason an accurate knowledge of its nanoporous structure is of great interest. For thin layers, results concerning the interface structures of the PS layer can be obtained in a non destructive way, with specular and off–specular measurements, performed in the incident plane. But for thicker layers, the nanoporous structure leads to a strong bulk scattering contribution at larger values of momentum transfer $\mathbf{q}$ [2], only accessible with out of plane geometry.

Nine samples were studied mostly with "thick" PS layers (in the micrometer range). A specular reflectivity curve is first obtained to measure the critical angle ω_c of the porous silicon layer. Then, the GISAXS experiment is performed for several incidence angles (smaller and larger than the critical one) in the $(\mathbf{q}_z, \mathbf{q}_{//})$ plane with a 2D detector (the indices z and // describe respectively the direction perpendicular and parallel to the sample surface). The first studied sample is a thick p^- type PS layer ($\omega_c = 0.16$ deg) (this kind of sample has already been characterised by other techniques, such as transmission electron microscopy [3] and classical small angle x–ray scattering [4]). Figure 1 presents various scans obtained from the 2D data, for two incidence angles (0.3 and 0.12 deg); a diffuse hump related to the typical size of the porous structure is always observed (see for instance fig. (c)). Along $\mathbf{q}_{//}$, for $\mathbf{q}_z$ chosen at the maximum of the scattered intensity (fig. (a) and (b)), a strong in plane correlation appears (the maximum of the correlation is marked with a vertical dotted line on the graphs). For smaller angles, a Yoneda peak is also observed (fig (d)), which is the sign of surface scattering.

From these scans, various quantitative results can be obtained, such as the in–plane correlation length (around 14 nm) or the typical size of the scattering particles (5 nm). In the latter case, one must pay attention to the contribution of the correlation signal which perturbs the shape of the scattered intensity resulting from a single particle. This size agrees with previous observations [3] but the correlation is a new effect.

The same technique has been applied to all typical kinds of porous silicon structures. The corresponding typical size a of scattering particles and the in–plane correlation length $\xi_{//}$ are reported in the following table [5]:

PS type	a (nm)	$\xi_{//}$ (nm)
p^-	5	14
p^+	11.5	22
n^+	6	15
n	5	11
n^-	5.5	14.5

In addition to the particle size which is a new result for most of n–type materials, a new feature is the strong in plane correlation. In some case, a correlation ring indicates that the particles are also correlated in the $\mathbf{q}_z$ direction. It is some time possible to observe on the transmitted beam the scattered signal from the cylindrical pores, which can not be observed on the reflected one.

For thin PS layers, the same procedure has been applied. For incidence angles larger than the critical one, the scattered intensity results mostly from a scattering phenomena at the interface between porous silicon and bulk silicon : this is due to the weak absorption of the beam and to a large interface roughness; moreover, the porous volume is limited (thin layer). On the contrary, for incidence angles smaller than the critical one, the bulk scattering is stronger as the surface scattering is weak (small roughness) and as the evanescent wave plays the major role by probing the porous medium. For these samples, the cross–over between bulk and surface scattering has been qualitatively observed [5], but other measurements with optimised samples are needed to obtain more quantitative conclusions.

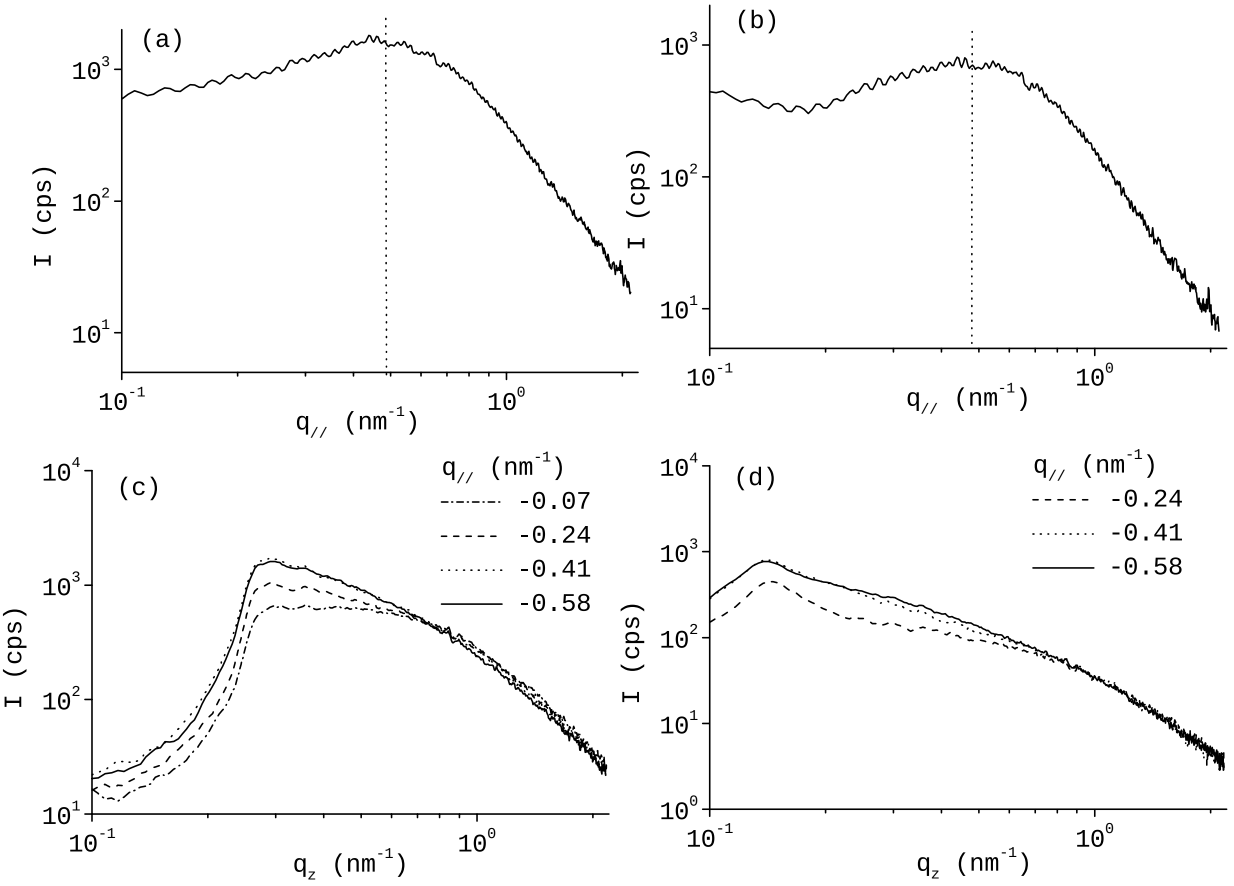

Fig. 1 : Log–log scale representation of $\mathbf{q}_{//}$ scans measured at $\mathbf{q}_\mathbf{z} \cong 0$ for incidence angles of (a) 0.3 and (b) 0.12 deg and $\mathbf{q}_\mathbf{z}$ scans measured at various $\mathbf{q}_{//}$ positions (indicated on the graph) for incidence angles of (c) 0.3 and (d) 0.12 deg. All the results were obtained with a thick p^- type PS layer (2 µm, $\omega_c = 0.16$ deg). The indices z and // are related to the directions perpendicular and parallel to the surface sample.

References :

[1] L.T. Canham, editor. *Properties of porous silicon*. EMIS Datareviews Series No. 18, INSPEC, (The Institution of Electrical Engineers, London), 1997.

[2] V. Chamard, G. Dolino, and J. Stettner. *Physica B.*, **283**, 135 (2000)

[3] M. I. J. Beale, N. G. Chew, M. J. Uren, A.G. Cullis and J. D. Benjamin. *Appl. Phys. Lett.*, **46**, 86 (1985).

[4] V. Vezin, P. Goudeau, A. Naudon, A. Halimaoui and G. Bomchil. *Appl. Phys. Lett.*, **60**, 2625 (1992).

[5] V. Chamard. PhD thesis, Joseph Fourier–Grenoble 1 University (October 2000).