EUROPEAN SYNCHROTRON RADIATION FACILITY

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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:** Structural Characterization of Imprinted Nanostructures in Silicon	**Experiment number**: SI-896
Beamline:	**Date of experiment**: from: 09.04.2003 to: 15.03.2003	**Date of report**: 16.03.2003
Shifts: 18	**Local contact(s)**: Bernd Struth	*Received at ESRF:*

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Report:

We have investigated several samples produced by C. Keimel at the Department of Electrical Engineering, Princeton University, Princeton, NJ 08544. A pattern of nanoscale wires with a height and width of 110 nm and 14ß nm, respectively, and lateral periods from 200 to 300 nm has been imprinted into a Si (001) substrate using the laser-assisted direct imprint (LADI) method [1]. During this process, the Si surface is melted using a short laser pulse, and the SiO_2 mold is pressed into the Si, which recrystallizes within 220 ns.

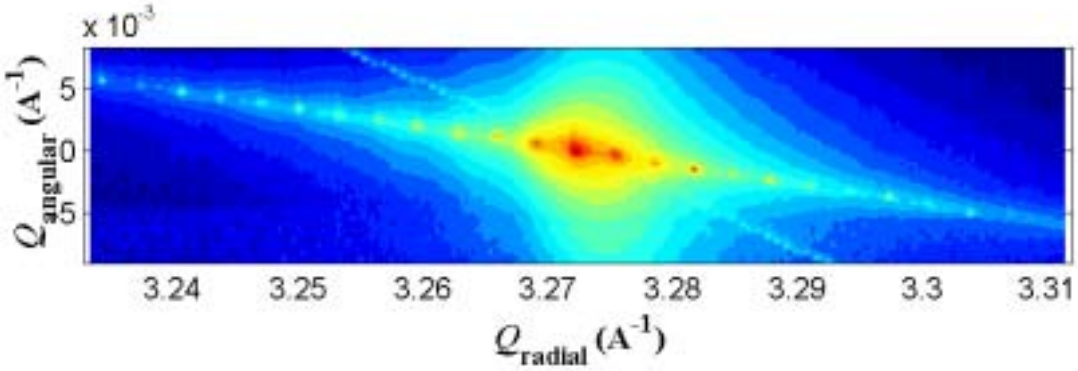
Scanning electron microscopy (SEM) reveals a quite perfect transfer of the mold pattern to the Si [1]. However, the crystal qualitiy of the rapidly melted and resolidified material cannot be directly accessed by SEM. Therefor we performed an x-ray scattering experiment, which is sensitive to the crystal structure of the sample. In order to be sensitive to the thin structure at the sample surface, we chose grazing incidence diffraction (GID) geometry. As the patterned area has a size of only about 0.5×0.5 mm, and is not entirely uniform over this area, we used a primary beam size of 0.2×0.2 mm. In order to resolve superstructure reflections according to the imprinted grating, a Si(111) analyzer crystal was used in the primary beam path, and a collimating slit of 0.2 mm width 10 cm behind the sample limited the footprint of the investigated area.

We used the (220) inplane reflection for our investigations, at an incidence angle with respect to the sample surface of 0.2°, which is below the critical angle of external reflection for the

primary beam energy of 8016 eV. Figure 1 shows an in-plane reciprocal space map of sample CK-C01 with a 200 nm period grating. Clearly, sharp satellite peaks are visible, corresponding to a crystalline grating. From the peak distance, a period of 200±3 nm follows, in very good agreement with the nominal values. From the width of the peaks we can estimate a lateral correlation length of at least 2 µm, however, the peak width is only slightly larger than the reciprocal space resolution of our experimental setup.

Therefore it is obvious that the imprinted gratings are indeed single crystalline, with the same crystal orientation than the substrate. In order to obtian information of a possible damaged surface layer of the grating, or point and extended defects within the recrystallized material, investigations of the diffuse scattering are required. However, as measurements of a reference sample without imprinted grating, but only spots melted with the laser and recrystallized, showed, no difference in the diffuse scattering as compared to a Si substrate without laser treatment could be seen. This indicated that the crystal quality of the samples is at least not significantly deteriorated by the laser melting, although atomic force microscopy showed increased roughness values in the laser-treated spots. A more elaborate investigation is required in order to resolve differences in the scattering from treated and untreated samples. In particular, cooling the samples in order to reduce thermal diffuse scattering will be required for this purpose.

Another problem turned out to be the lateral inhomogeneity of the samples. Parts of the



imprinted surface are mechanically damaged during lifting off of the mold, and the x-ray beam (precisely the footprint of the beam on the sample) is not sufficiently small in order to be sure to measure exclusively in in non-damaged areas. Therefore, diffuse scattering cannot be attributed only to crystal defects in the undamaged areas, but also the damaged areas might give a contribution, making the analysis of diffuse scattering ambiguous. A smaller x-ray spot, but at the same primary beam divergence as used in this experiment, would be required in order to eliminate this problem.

References

[1] S.Y. Chou, C. Keimel, J. Gu, Nature **417**, 835 (2002).