European Synchrotron Radiation Facility

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON



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: Coherent Scattering and Reconstruction of Nanoporous Structures and Nanoparticles	Experiment number: SC 1863
Beamline:	Date of experiment:	Date of report:
ID 10c	from: 10.5.2006 to: 15.05.2006	5.7.2006
Shifts:	Local contact(s):	Received at ESRF:
18	A. Robert	
Names and affiliations of applicants (* indicates experimentalists):		
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Report:

We performed a coherent x-ray imaging experiment for the determination of structures on nanometer length scales. The investigated samples ranged from Ni structures to assemblies of colloidal particles deposited on thin silicon-nitride membranes. In order to position the small structures in the beam via the Ni fluorescence signal we temporarily changed the energy to 8.39 keV, i.e., slightly above the Ni K edge. For the imaging experiment an X-ray energy of 8.31 keV slightly below the Ni K edge was employed. A partially coherent x-ray beam was produced by a pair of polished roller blade slits with a 10 x 10 μ m² opening. The parasitic Fraunhofer scattering from these slits was partially blocked by a guard slit (also roller blade slits) further downstream. Approximately 2.6 10⁸ photons/s were illuminating the samples. We measured a value of the Fraunhofer fringe visibility of ca. 30 % - 40 % which ensured us that the x-ray beam is sufficiently coherent to perform imaging experiments. A princeton direct illumination ccd camera was positioned 3.75 m downstream of the sample. From the q-range covered we estimate a spatial resolution of approximately 50 nm. A beamstop of 2 mm diameter was placed in front of the ccd.

Fig.1 shows a typical ccd image (exposure time 200 seconds) recorded from the scattering of the Nickel structures. In this case the structure mirrors the letter H (the inset shows the corresponding SEM picture) with an overall size of 4 x 4 μ m². The height of the Nickel structures is 300 nm and the size of the individual bars is 400 nm. We estimate that the oversampling ratio is around 6. Due to the high symmetry of the object the scattering intensity is mainly distributed along two perpendicular directions in reciprocal space. Around the beam stop parasitic scattering from the guard slits is visible preventing the use of smaller beam stops. The missing forward data caused by the beam stop is one of the central problems in coherent imaging in the SAXS regime. If the area of missing data is sufficiently small than modified phase retrieval algorithms are able to reconstruct an image successfully[1]. However, the missing data area in our experiment is too large for this approach and so we decided to paste a calculated diffraction pattern in the missing data area, like e.g. done in [2]. With this we can employ the HIO phase retrieval algorithm and Fig. 2 shows an example of a reconstructed image. Depending on the exact shape of the support the quality of the reconstructed. Further Nickel samples have been measured (lattice structures of different spacings, different letters) and the analysis is ongoing.

Figure 3 shows as a further example a ccd image recorded from the scattering of a 800 nm large assembly of colloidal particles. Unfortunately, the parasitic scattering is even stronger here as can be seen from the large areas of missing data. Nevertheless, a reconstruction similar to the one outlined above will be done on this data.

In summary, we successfully performed coherent imaging experiments on samples providing nanometer structures. A first successful reconstruction has been presented. The need for effective suppression of parasitic scattering is apparent and will be a major point for further investigations.

[1] Y. Nishino et al. Phys.Rev.B. 68, 220101(R) 2003

[2] J. Miao et al. Nature 400, 342 (1999)



Figure 1: Scattering image of a Nickel structure. The SEM image is shown in the inset.



Figure 2: Reconstructed image of the Nickel structure.



Figure 3: Scattering image of an assembly of colloidal particles. The SEM picture is shown in the inset.