EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** Simultaneous observation with combined AFM and scanning near-field X-ray microscope	**Experiment number**: MI-403
Beamline:	**Date of experiment**: from: 21/06/00 to: 27/06/00	**Date of report**: 16/8/00 *Received at ESRF:*
Shifts:	**Local contact(s)**: Dr Raymond Barrett	

Names and affiliations of applicants (* indicates experimentalists):

Professor R E Burge*, Cavendish Laboratory, University of Cambridge, UK

Dr M T Browne*, Physics Department, King's College London, UK

Dr P Charalambous*, Physics Department, King's College London, UK

Report:

1. Intention.

The intention, based on the expectation to have available X-rays of "water-window" energies, was to continue the development of the near-field X-ray microscope by introducing a fine collimating tube at the X-ray focus of the collimator zone plate under near-field imaging conditions to achieve spatial resolutions approaching 10nm, and to make a detailed investigation of the simultaneous measurements of X-ray transmission and surface topography.

The AFM tip was pierced by a central aperture, which, in principle, allowed both the measurements of X-ray transmission and the surface profile to be made simultaneosly at the same specimen location.

2. Experimental.

In practice , for the 3keV photons actually available, it was not possible to gain significant X-ray transmission through the aperture and, instead, the focussed X-ray beam at the specimen was provided by the "condenser" zone plate, which actually became the objective zone plate of a scanning transmission X-ray microscope. Thus X-

ray and topographic measurements were made simultaneously but at the adjacent, rather than coincident, locations of the AFM tip and the focus of the objective zone plate.

X-rays of 3keV energy were anticipated, and as a consequence considerable effort was expended in seeking to make apertures in the AFM tips that would define a transmitted probe of this energy with reduced diameter as compared with the probe formed by the condenser zone plate itself. A new method was tried whereby a fine tip some 2um long and about 50nm diameter was grown on the standard AFM tip in carbon contamination. This was done in a scanning electron microscope. The contamination spike was then coated with a layer of sputtered gold, following which the top of the spike was cleared away by ion milling to allow the carbon core to be visible. The X-ray absorption of carbon is much less than that of an equivalent thickness of gold and the carbon spike was left in situ with small effect on X-ray transmission through the core.

Two problems appeared in the application of these (long) collimator tubes to give unacceptably small transmission. The first was the uncontrolled bending of the cantilever making the axial alignment of the tube extremely difficult; the second was the effect of surface roughness giving incoherent X-ray scattering at the tube walls. Both problems will be significantly reduced for the much shorter collimators (~ 0.25um) appropriate to the initial concept for photon energies of 300-500 eV.

The experimental work was, therefore, concentrated on the simultaneous, but physically separated, collection of data on X-ray transmission and specimen height fluctuations. Although the data were actually collected from AFM tip and zone plate focus a few um apart on the specimen surface, for the energy of 3 keV, it was shown to be possible, because of the low X-ray absorption of the AFM tip, actually by scanning the zone plate laterally, to make X-ray transmission measurements through the tip location and have the two simultaneous images precisely aligned.

The procedure adopted to standardise imaging under near-field conditions was to bring the specimen by means of an axial piezo movement into contact, as judged by a voltage setting, with the AFM tip at each pixel at which position the X-ray signal, for an image focused on the tip, was also recorded with a dwell time of (for example) 10msec. Before moving to the next pixel the AFM tip was released from the specimen to return to a standard position before being brought up at the adjacent pixel; the total duty cycle of tip movement, X-ray imaging and tip release is about 30msec.

Excellent topographic images of gold coated polystyrene latex spheres about 1um and 500nm in diameter, some ion-milled to produce circular gold discs with a wider range of topographic features, were produced using fabricated carbon fibre needles, about 100nm in diameter and 3um long, mounted on the original AFM tips. X-ray transmission images were gained by using a germanium zone plate 230um in diameter with 85nm outer zone width, focal length for 3keV photons ~48mm. The transverse resolutions of the two images were about, 5nm (AFM) and 100nm (X-ray transmission). The (arbitrary) actual displacement of the centres of simutaneously-taken images was about 8um and the images were brought to coincidence by off-line image processing. It is of interest that the X-ray transmission is finite only through the silicon nitride supporting film on the silicon specimen mount, whereas the AFM image continues to include specimen areas where the X-ray transmission is zero.

The question of the axial sensitivity of the tip measurements of surface topography is an important one. Calibration experiments were carried out to a displacement of 60um with good linearity from 10um. For smaller axial displacements due to expected hysteresis effects the linearity needs to be further investigated, as do the parameters controlling the tip contact and tip release. Assuming linearity for small displacements, axial distances of order 5nm can be measured using the AFM tip in the present set up, or, in 3D, discrimination is possible between cubes of material about 5nm on a side.

3. Conclusions.

The scanning transmission X-ray microscope with AFM tip incorporated needs further development to characterise the tip displacement, but in general is now a working instrument. The X-ray image in the first order zone plate focus will be limited in resolution only by the physical action of the zone plate, assuming adequate optical design of scanning stages etc and vibration isolation. This resolution will no doubt be improved by further zone plate development and imaging at higher orders of the zone plate focus. The use of a cylindrical collimator to improve spatial resolution of the transmitted X-ray image for high photon energies will always be difficult. With further development both the transverse and the axial resolutions of the AFM tip in the present application can be improved, but the current performance is adequate to add a third dimension to the two dimensions of the transmission X-ray image which is the dominant partner for relevant applications.

The new 3D facility is seen particularly in the context of quantitative X-ray measurements from areas of non-uniform specimens where the specimen thickness is required, and with dynamic experiments conducted in the microscope involving e.g. radiation damage, heating, specimen straining etc. The question of improved X-ray microscope resolution using a small collimator in analogy to the scanning near-field optical microscope must be put on hold until lower photon energies are available at the ESRF.

It is a pleasure to acknowledge the helpful advice and expertise on the beam line-and much else-provided throughout the work by Dr Jean Susini and Dr Raymond Barrett.