



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>

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal (“relevant report”)

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a “*preliminary report*”),
- even for experiments whose scientific area is different from the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as “relevant report(s)” in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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: X-ray phase-contrast imaging technique using an X-ray interferometer based on Si refractive lenses	Experiment number: MI-1333
Beamline:	Date of experiment: from: 20 Jun 2018 to: 23 Jun 2018	Date of report: 12 Sep 2021
Shifts:	Local contact(s): Martin Rosenthal (email: martin.rosenthal@esrf.fr) ID13	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Dr Dmitrii Zverev (Immanuel Kant Baltic Federal University) Dr Irina Sniguirev (ESRF) Dr Anatoly Sniguirev (Immanuel Kant Baltic Federal University)		

Report:

We demonstrated the phase-sensitive X-ray imaging technique based on the bilens interferometer. The essence of the method consists of scanning a sample, which was set upstream of the bilens across the beam of one lens of the bilens, and recording changes in the interference pattern. This optical scheme involves fine-tuning the position of the sample on the optical axis, while a small deviation can lead to some distortion of its reconstructed phase profile. Also, the advanced optical layout was considered. Knowing that the bilens generate two diffraction-limited focal spots, the sample can be placed in the focal plane of the bilens CRLs. In this case, the small size of the focused beams provides excellent phase sensitivity and high spatial resolution allowing to avoid possible distortions of the phase profile completely. The capabilities of both optical schemes were studied theoretically and experimentally.

An experimental demonstration of the phase-sensitive imaging technique based on a bilens interferometer was performed at the ESRF ID13 undulator beamline (Grenoble, France). The bilens interferometer consisting of 58 double concave parabolic lens in each lens array was placed at a distance of 96 m from the X-ray source. At X-ray energy of 12.7 keV the theoretical focal distance f of lenses in the bilens interferometer is 18 mm, the effective aperture is 13.3 μm , and the expected diffraction-limited focal spot size is to 87 nm.

The interference fringe patterns were recorded using X-ray high-resolution CCD detector FReLoN with a spatial resolution of 0.9 μm (pixel size of 0.37 μm). The detector was installed at a distance of 1.7 m from the foci formed by the interferometer. At this distance, the period of the interference pattern is 2.77 μm (more than 7 pixels per period), that is corresponds to the upper limit of phase resolution for the proposed imaging technique of about 840 mrad ($\sim 0.27\pi$ rad). It should be noted given the large number of fringes observed in the interference pattern, the determination of the fringe shift can be performed with much greater accuracy than the detector pixel size. The approximation of the intensity distribution in the cross-section of the interference pattern by a harmonic function allowed achieving excellent phase resolution of 190 mrad ($\sim 0.03\pi$ rad) for the experimental setup under consideration.

To demonstrate the phase sensitivity of the proposed imaging technique the boron fiber is a coaxial structure consisting of a weakly absorbing boron shell with a diameter of 100 μm and a very absorbing tungsten core inside with a diameter of 15 μm was chosen as a sample. Even though boron fiber overlapped both beams in the

bilens, it was possible to reconstruct the absolute values of the phase profile by the relative shifts of the interference fringes.

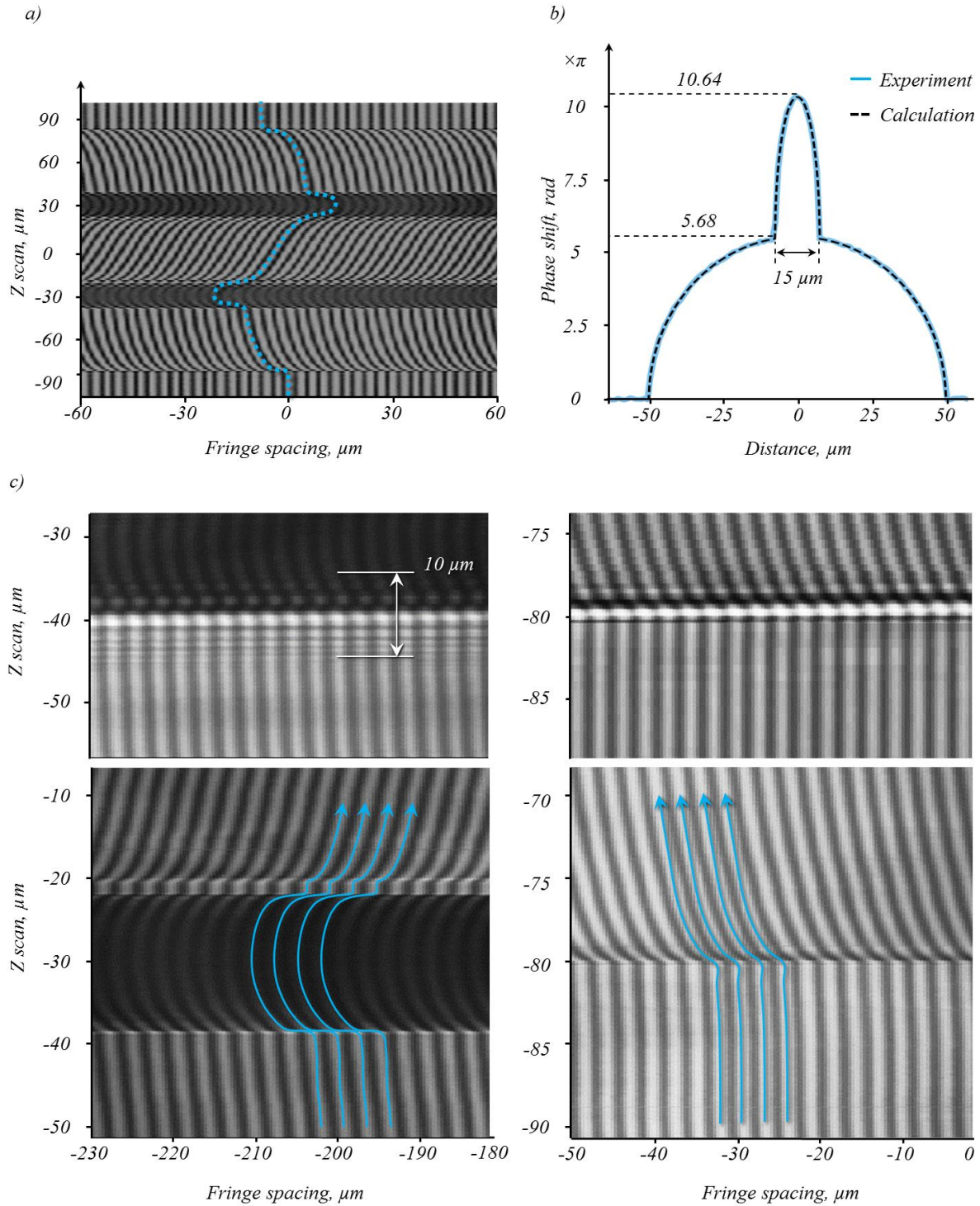


Figure 1. The result of an experimental demonstration of a phase-sensitive imaging technique based on a bilens interferometer. (a) Experimental scan image. (b) The reconstructed phase profile of boron fiber with a tungsten core. (c) The fragments of the scan images in the area of a tungsten core and edge of boron fiber obtaining experimentally with the scanning step of 100 nm in both optical layouts where the sample was placed in front of the bilens (top row of images) and in its foci (bottom row of images).

The fiber scanning process was carried out as follows. First, when moving the fiber perpendicular to the optical axes of the bilens, a set of interference patterns was recorded. The scanning step was 1 μm , and the scanning area ranged was $\pm 100 \mu\text{m}$ relative to the bilens centre. Then, cross-sections were cut out from the corresponding interference pattern for each fiber position, and by combining them, an experimental scan image was formed.

The corresponding experimental scan image is demonstrated in Figure 1(a). This image shows the shifts of interference fringes observed during the scanning process, which are the result of phase delays of the beam generated by the fiber. The absolute values of the phase delay are easily measured from the corresponding values of the shift of the fringes.

The observed shifts of the fringes are indicated by a dashed line. During the scan, the fringes are first shifted in one direction by more than five periods relative to the interference fringes without sample, and then symmetrically in the other direction. It is related to the axial symmetry of the fiber. Thus, in the zero position of the scan, when the tungsten core of the fiber is located opposite the center of bilens, the shift of the fringes is not observed.

The reconstruction of the phase profile of boron fiber, taking into account its large size, is presented in Figure 1(b). The theoretical curve calculated for the expected coaxial structure of the boron fiber, consisting of 15 μm tungsten core and 100 μm boron shell, well describes the experimental result. Thus, the measured maximum value of the phase shift, equal to 10.64π , corresponds to the joint contribution to the phase delay of boron with a thickness of $100 - 15 = 85 \mu\text{m}$ with a refractive index decrement of $2.8 \cdot 10^{-6}$ and 15 μm tungsten with a refractive index decrement of $1.9 \cdot 10^{-5}$. It is worth noting that for boron with a thickness of 100 μm , the corresponding phase shift will be 5.68π , which was marked on the phase profile for clarity. The experimental results are fully consistent with the corresponding theoretical calculations.

For comparison the spatial resolution, the fragments of the scan images in the area of a tungsten core and edge of boron fiber were obtained experimentally with the scanning step of 100 nm in both optical layouts (see Figure 1(c)). In the top row, the distortions of the scan images formed in the optical scheme where the sample placed in front of the interferometer are observed. The measured characteristic size of the distortion is about 10 μm , which corresponds to the effective aperture of the bilens CRLs. The bottom row of images corresponds to the advanced optical layout where the sample was placed at the lens foci. Due to the tiny focal spot size the spatial resolution of the technique is determined by the larger scan step of 100 nm. The smooth shifts of the fringes occur.

The ability to directly detect the phase shift is an absolute advantage of X-ray interferometric imaging using bilens among other phase-sensitive methods, such as diffraction enhanced imaging (DEI) or Talbot interferometric imaging which detect only the spatial gradient of the phase shifts. Furthermore, tracking the change of the shape of the envelope of the interference fringes and their visibility during the scanning of the sample, it is possible to obtain the corresponding absorption and scattering profiles. As a result of these advantages, the proposed approach has the highest resolution in density and provides a way to perform precise studies of biomedical and organic samples without using any additional methods for their preparation. Also, the proposed phase-sensitive X-ray imaging technique is an effective tool for phase diagnostics of the wavefront of the synchrotron beam. The transverse phase profile of the beam can be obtained by scanning it with bilens.

We would like to emphasize that the proposed approach is not limited by 1D objects but also allows studying of two-dimensional samples, for instance by adding an additional imaging lens-system with a scanning of the sample across the bilens axis. We expect to perform this in the future.