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 via the User Portal:
https://wwws.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 form 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: Probing the hierarchical organization of Bagworm silk by scanning X-ray nanodiffraction	Experiment number: SC-5011
Beamline: ID13	**Date of experiment**: from: 1/10/2020 to:5/10/2020	**Date of report**: 9/9/2021
Shifts: 9	**Local contact(s)**: Manfred Burghammer	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

T. Yoshioka

T. Kameda

C. Riekel*

Report:

We studied single bagworm (BW) fibers as well as longitudinal- and cross-sections from paraffin-embedded fibers. All samples were supported by Si_3N_4 membranes. Scanning nano X-ray diffraction (nanoXRD) was performed in transmission geometry using a monochromatic beam of λ=0.08158 nm, focused to ~250 nm (hxv) fwhm (full-width-half-maximum) with a flux of $\Phi\sim2*10^{11}$ photons/s. NanoXRD patterns were recorded by an Eiger 4M detector.

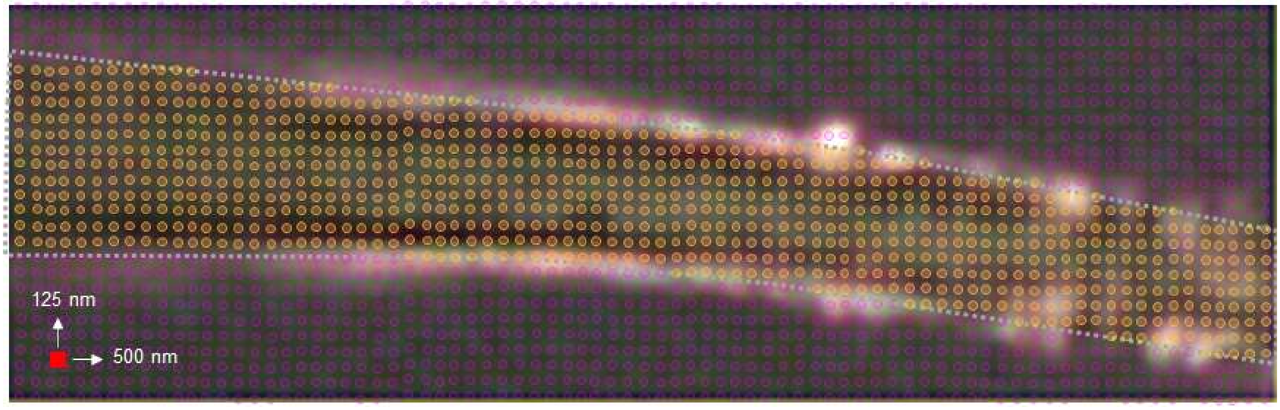

Fig.1 Optical microscopy of BW fiber with overlay of nanoXRD probing positions.

The optical image of a BW fiber with an overlay of probing positions is shown in Fig. 1. The averaged SAXS/WAXS pattern from 100 probing positions agrees to the SAXS/WAXS patterns obtained from fibers bundles[1] (Figs. 2A,B). For typically 50 ms exposures we used rectangular probing meshes of 50-250 nm horizontal and 500-1000 nm vertical step-increments to limit radiation damage[2]. Hierarchical structural information is obtained from composite SAXS/WAXS images (called also “density maps”) as shown in Figs. 2C,D,E. We also collected SAXS/WAXS pattern from 0.5 µm longitudinal and 1.0 µm cross-sections of single BW fibers embedded in paraffin matrix. The quality for the results is shown in this report for the longitudinal section. Indeed, the optical microscopy image shows two fibroin sections embedded in the paraffin matrix (Fig. 3A). The composite SAXS image reveals contours of fibroin section and surrounding paraffin matrix (Fig. 3B). A single scan-line across the section reveals intensity modulated equatorial streaks which will be used for simulating the mesoscale structure.

1. T. Yoshioka, T. Tsubota, K. Tashiro, A. Jouraku, T. Kameda., *Nat Comm* **10**, 1469 (2019).
2. C. Riekel, M. Burghammer, C. Ferrero, T. Dane, M. Rosenthal., *Biomacromolecules* **18**, 231 (2017).

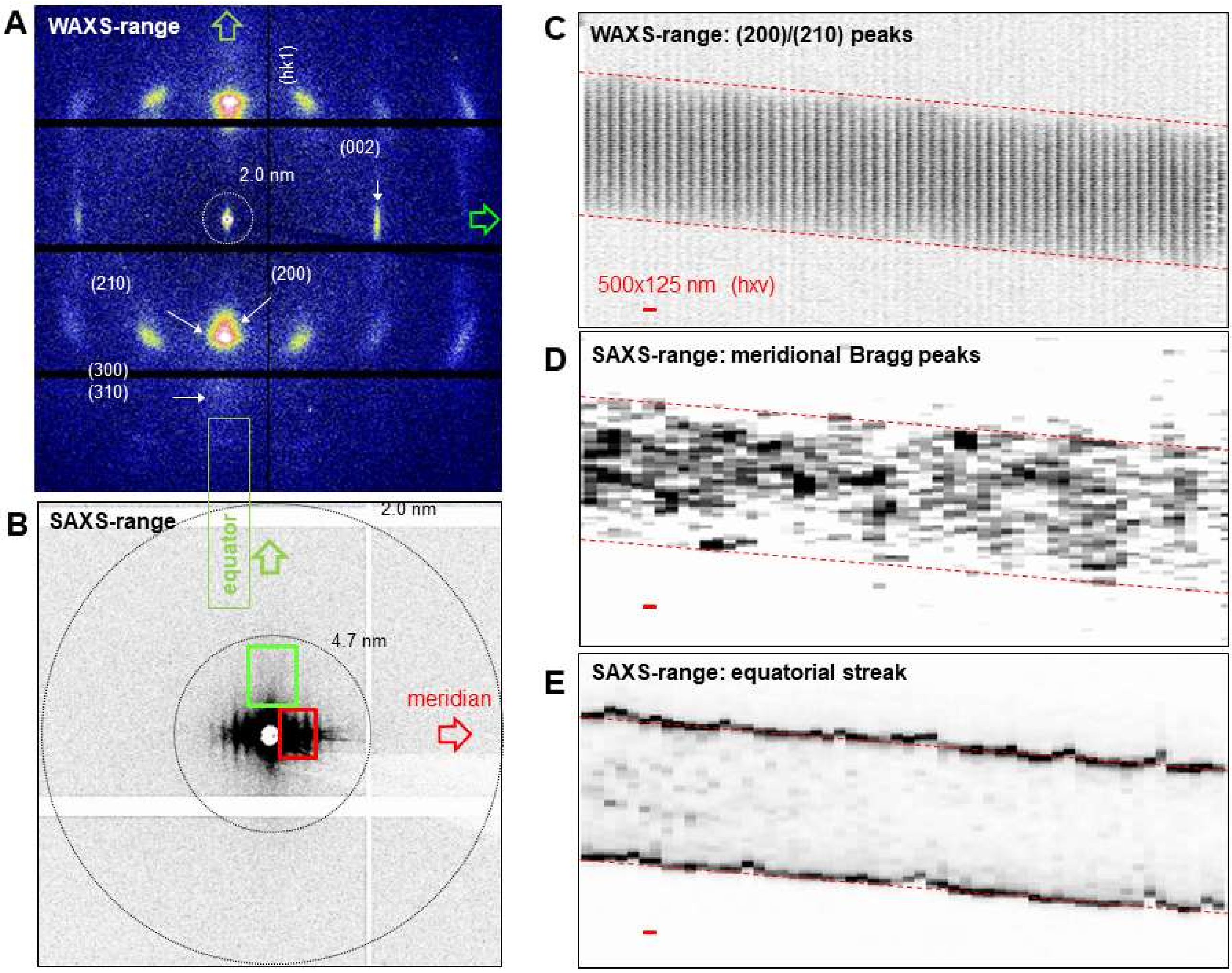

Fig.2 A: Averaged WAXS pattern (5 s overall exposure) from probing positions on BW fiber (Fig. 1). Selected reflections indexed for silkworms' lattice. B: SAXS-range of (A). C: Composite WAXS image based on pixels covering (200)/(210) reflections. D: Composite SAXS image based on meridional Bragg peaks (red rectangle in B). E: Composite SAXS image based on equatorial streak (green rectangle in B). The strong skin-layer scattering in (E) is due to a sericin layer.

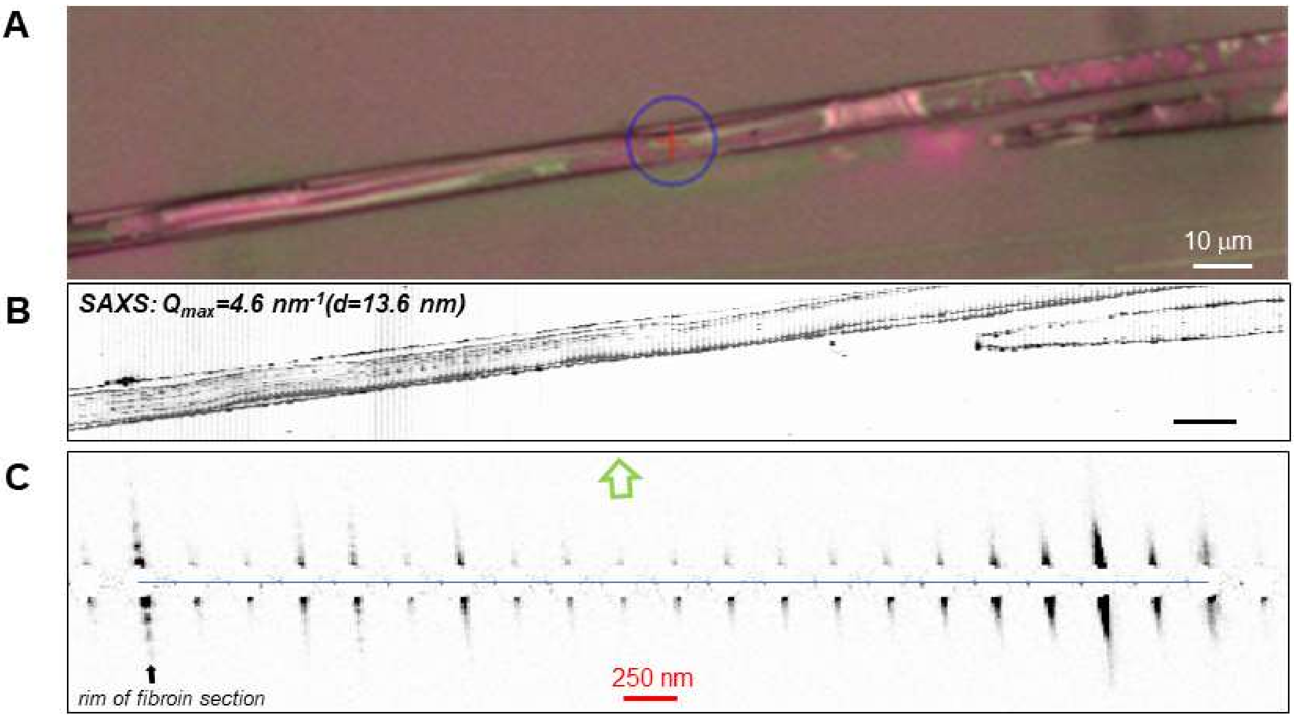

Fig.3 A: Optical image of two BW sections with 500 nm thickness. B: Composite SAXS image of (A) based on equatorial streak (green rectangle in Fig. 1B, Q_{max}=4.6 nm^{-1} pixel limit; 250_{vert}x1000_{hor} nm^2 step-increments). C: One scan-line across BW section in (B) revealing modulated SAXS streaks.