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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

Reports supporting requests for additional beam time

Reports can 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.

	Experiment title: Structural effects in self-assembled peptide nanotubes: phase transitions and lattice dynamics	Experiment number: SC-4587
Beamline: ID27	Date of experiment: from: 10/11/2017 to: 14/11/2017	Date of report: 01/02/2018
Shifts: 12	Local contact(s): Volodymyr SVITLYK (email: svitlyk@esrf.fr)	*Received at ESRF:*

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Report:

The project was aimed at: 1) revealing the structure modifications of FF nanotubes at various temperatures and pressures, and 2) understanding the dynamics of water–FF interaction and related quantities. The first aim required to use ID27 beamline, whereas the second – ID28. Since only ID27 beamline has been assigned, we restricted ourselves with the first aim.

During the project we have performed single crystal X-ray diffraction experiments of three types: 1) determination of FF nanotubes' structure at high pressure, 2) determination of FF nanotubes' structure at low temperatures, and 3) determination of the structure of crystals of derivatives of FF dipeptides at ambient pressure and temperature. We have studied single crystalline microtubes representing bundles of arranged FF nanotubes and needle-like solvate crystals of cycled FF monomers formed in the solution. Both microtubes and crystals have typical lengths up to 5 mm and diameters 50-100 μm, which were suitable for the beamline station. It was found that the intensity of diffraction peaks is quite low. Therefore, careful integration of the obtained diffraction patterns was required, and this took more time than we expected. For the moment we have processed only data for temperature measurements and for solvate crystals and have solved their crystal structures.

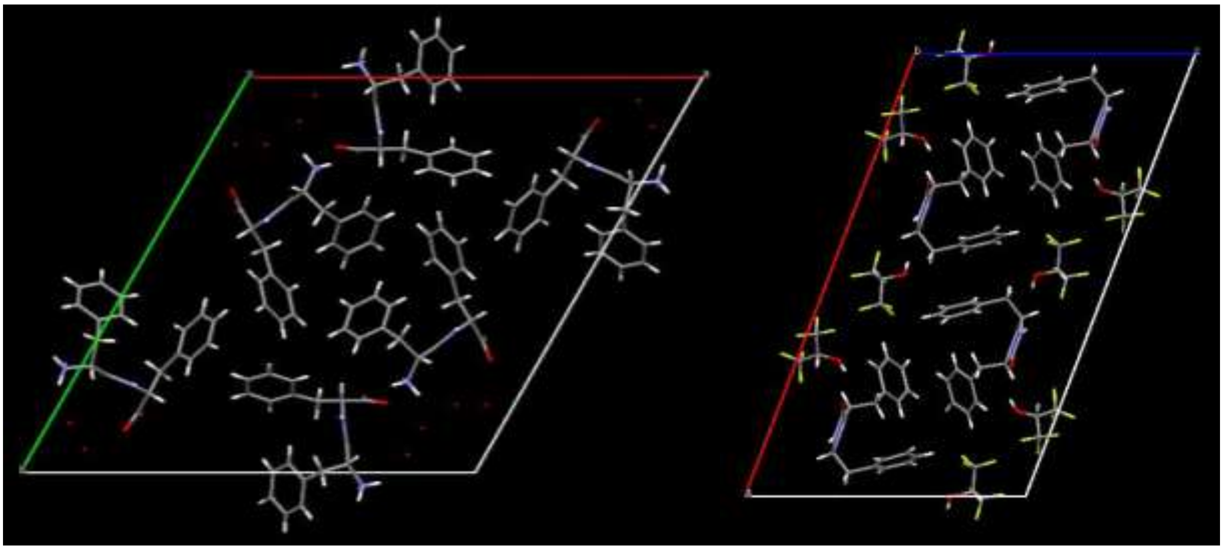
Pressure measurements were performed in helium filled DAC in pressure range 0.11–14.5 GPa. In total 18 measurements have been performed. At each pressure point a survey diffraction pattern has been measured and then a set of detailed patterns with a 0.5 degree step were collected. The integrating of the obtained diffraction patterns is in progress and the results will be reported in the forthcoming paper.

Temperature measurements were performed in the temperature range 95- 290 on cooling and heating. In total 14 measurements have been performed. To control the temperature a CryoStream system was used. At each temperature point a survey diffraction pattern has been measured and then a set of detailed patterns with a 0.5 degree step were collected.

The obtained results reliably showed that at ambient conditions FF nanotubes belong to $P6_5$ space group, whereas $P6_1$ is usually reported in the literature. It is known, that 230 crystallographic space groups were obtained for the right-handed coordinate system, whereas for a number of chemical compounds a left-handed system is necessary. Therefore this difference can be attributed to the chirality of the used dipeptide. In our study we used D-Phe-D-Phe form, whereas L-Phe-L-Phe is usually reported. The results will be reported in the paper (in preparation).

The solved crystal structure at ambient conditions is presented at Fig. 1a. Parameters of this structure coincide with those reported in the literature. Analysis of low-temperature crystal structures demonstrated non-monotonic behavior of cell volume and cell parameters of the nanotubes with local minima at about 200 K. At the same temperature an anomaly in the temperature dependence of dielectric permittivity has been observed by us. Thus the results obtained in this project allowed us to shed light on the origin of this anomaly.

We also have solved the structure of solvate crystals of cycled FF monomers (Fig. 1b). Similar cycled monomers occur at elevated temperatures (above 140°C). The solvate form of monomers' FF crystal has not been studied yet. The results are being prepared for the publication.



(a) (b)

Figure 1. Solved crystal cell of (a) FF microtube consisting of individual nanotubes, and (b) solvate crystal of cycled FF monomers.

To conclude, we suppose that the project SC-4587 has been succsesfully realized. The results revealed important details of the crystal structure of FF nanotubes and allowed to shed light on data, obtained earlier by other methods. For sure, further analysis will bring new valuable information important for the understanding the role of water in the process of self-assembly of peptide nanotubes, phase transitions under varying external conditions, and FF–water interactions. The obtained results are going to be published in high-impact scientific journals and presented at international conferences.