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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 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.

ESRF	**Experiment title:** Lattice dynamics of diphenylalanine nanotubes	**Experiment number**: SC-4714
Beamline: ID28	**Date of experiment**: from: 01/03/2018 to: 05/03/2018	**Date of report**: 20/06/2018
Shifts: 9	**Local contact(s)**: Alexei Bosak (email: alexei.bossak@esrf.fr)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

KHOLKINE Andrei, Ural Federal University, 19 Mira str., 620000 Ekaterinburg, Russia

*VASILEV Semen, Ural Federal University, 19 Mira str., 620000 Ekaterinburg, Russia

*VASILEVA Daria, Ural Federal University, 19 Mira str., 620000 Ekaterinburg, Russia

*ZELENOVSKII Pavel, Ural Federal University, 19 Mira str., 620000 Ekaterinburg, Russia

Report:

The project objectives were: (i) to reveal the real phonon dispersion in FF nano- and microtubes, and (ii) to determine several related quantities (sound velocities, elastic constants, phonon density of states and specific heat) essential to their applications in nanotechnology. To do this we requested to use ID28 beamline, and 9 beamshifts were allocated.

During the project we have performed single crystal X-ray diffraction experiments on several individual FF microtubes grown from the solution and have chosen the best one with pure single crystal diffraction pattern without any evidence of twins or poly-crystallinity. At the same time we have chosen the working temperature (240 K) and conducted several experiments at this temperature. It is essential that we also revealed the gradual damage of the samples under the synchrotron beam and determined the parameters for more or less non-destructive regime of measurements. The estimated time for one measurement was about 6 minutes.

The chosen sample was then used for performing inelastic X-ray scattering measurements with optimized parameters. After the long settings of the setup we could measure the inelastic X-ray scattering in the vicinity of M-points of Brillouin zone. The obtained spectra are presented in Fig. 1. No peaks corresponding to inelastic scattering was observed. Probably the reason is insufficient intensity of the X-ray beam, which was reduced because of the tube damage.

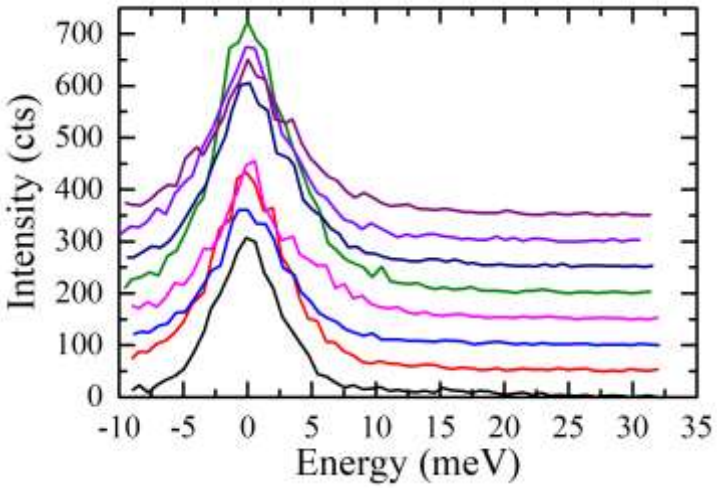

Figure 1. Inelastic X-ray scattering spectra measured in the vicinity of M-point of Brilloin zone of FF nanotube.

During the sample selection we noticed the formation of a set of additional reflexes in X-ray patterns of microtubes dried for several hours at elevated temperature. An example of such additional reflexes is presented in Fig. 2. The observed reflexes copy the main ones, but lies between them. Their period is 7 times smaller, than the period of main reflexes that corresponds to formation of superstructure with the 7x period of lattice parameter along c-axis of the tube. The nature of this effect is not clear yet and requires additional investigation using the facilities of ESRF. It is related to the behavior of nanoconfined water in the hydrophilic environment and should be studied in the future.

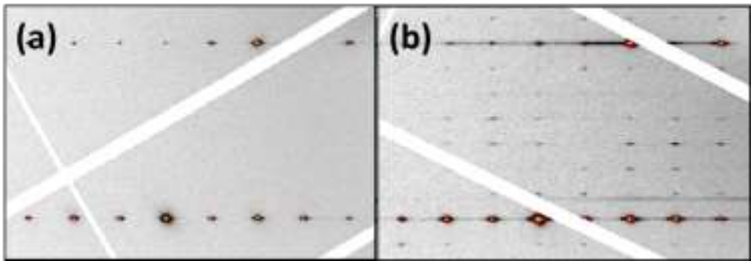

Figure 2. The superstructure formation in dry FF nanotubes. Diffraction patterns in H0L plane for (a) as-grown and (b) dry FF nanotubes.

To conclude, we suppose that regardless the problems with observation of inelastic X-ray scattering the project SC-4714 brought about new important results. Formation of the superstructures in FF nanotubes during their drying was not reported earlier. For sure, revealing the nature of these superstructures is of importance for understanding the role of water in phase transitions and physical properties of FF nanotubes. The obtained results will be published in high-impact scientific journals and presented at international conferences.