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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:** Sliding mode in an organic aperiodic composite	**Experiment number**: HS 857
Beamline: ID28	**Date of experiment**: from: 10/04/2016 to: 16/04/2016	**Date of report**: 12/09/2016
Shifts: 18	**Local contact(s)**: Alexei Bosak	*Received at ESRF:*

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Report:

This experiment was a continuation of the experiment HS4656 performed on ID28 in 2012. Incommensurate composite structures can be considered as a new state of matter with specific structure, properties and dynamics. We proposed to measure this dynamics in the prototype organic host-guest alkane-urea inclusion compound. The previous experiment showed the richness of this material in term of dynamics. Several results were obtained concerning the phonons in the nonadecane/urea, measuring far away in the reciprocal space. These phonons were measured around main Bragg peaks of the aperiodic composites. During this experiment, we foccussed on the dynamics emerging from the pure satellite Bragg peaks, where phason modes are expected to be visible.

Experimental conditions:

We kept the same experimental conditions and crystals than we used for experiment HS4656, which was very successful (see report):

- (Si 12,12,12) analysers configuration.
- A large nonadecane-urea single crystal of ~10 mm long and ~8 mm large with an exceptionally good mosaicity (less than 0.1 degree).

As compared to the first experiment, two major improvements had been obtained:

- The experimental resolution had been rigorously measured before our experiment using a Ge crystal. We performed a carefull analyse of these data which provided us a better knowledge of the instrument limitations.

- The displex system was improved to work properly with our very large crystal. Temperature regulation was very reliable, even at low temperature (100K)

Measurements and results:

In periodic crystal, the number of Bragg peaks in a selected region of the reciprocal space in defined by the periodicity of the lattice. On the contrary, in aperiodic crystal, the nodes of the reciprocal lattice at three dimension are nothing else but a projection of a periodic lattice of higher dimension. Thus, the number of Bragg peaks (or more precisely the so called "satellite" Bragg peaks) is in principle infinite, these satellite Bragg peaks being infinitely close to each other. In the case of a single aperiodic direction (here **c**), a four dimensional superspace group is required (here hexagonal $P6_122(00\gamma)$ at room temperature) and four indices are needed to characterize all these Bragg peaks $Q_{hklm}=h\mathbf{a}^*+k\mathbf{b}^*+l\mathbf{c_h}^*+m\mathbf{c_g}^*$. The misfit parameter $\gamma=c_h/c_g=c_g{}^*/c_h{}^*$ is equal to 0.418 in the case of *n*-nonadecane/urea. The common (periodic) ($\mathbf{a}^*$,$\mathbf{b}^*$) plane contains the common (hk00) Bragg peaks, (hkl0) reflects the host (urea) periodicity, (hk0m) the guest (alkane) periodicity, and (hklm) with l and m non-zero are the pure satellite which testify the intermodulation between both sublattices. Several studies have been performed as function of the temperature and for different polarisations around common Bragg peaks (0 0 6 0), (0 0 12 0). Their analysis give information on the coupling between phonon and phason mode. The phason mode is the excitation along the fourth dimension of the crystallographic superspace: it corresponds to an anti-phase sliding of the two host and guest sublattices. This mode is theoretically gapless showing an acoustic-like dispersion, but at the difference of an acoustic Goldstone mode, it is expected to be overdamped. Experimentally, we foccussed very much at the reciprocal position (0 0 6 1), that is 6.418 $\mathbf{c_h}^*$, and around it, at different temperatures. A quasi-elastic componant is reported with a maximum intensity around (0 0 6 1), see figure [1]. This result is obtained at 145K in the so-called phase II of nonadecane/urea. In practise, the number of observable satellite peaks is expected to be very limited for intensity reasons. This appeared to be no longer true when working with the exeptionally sensitive detector of ID28 beamline. So the major difficulty was the number of elastic peak increasing while decreasing temperature and limiting the available space for inelastic measurement. In phase III (100K), this elastic contribution prevented us from measuring acccuratly any inelastic contribution around (0 0 6 1).

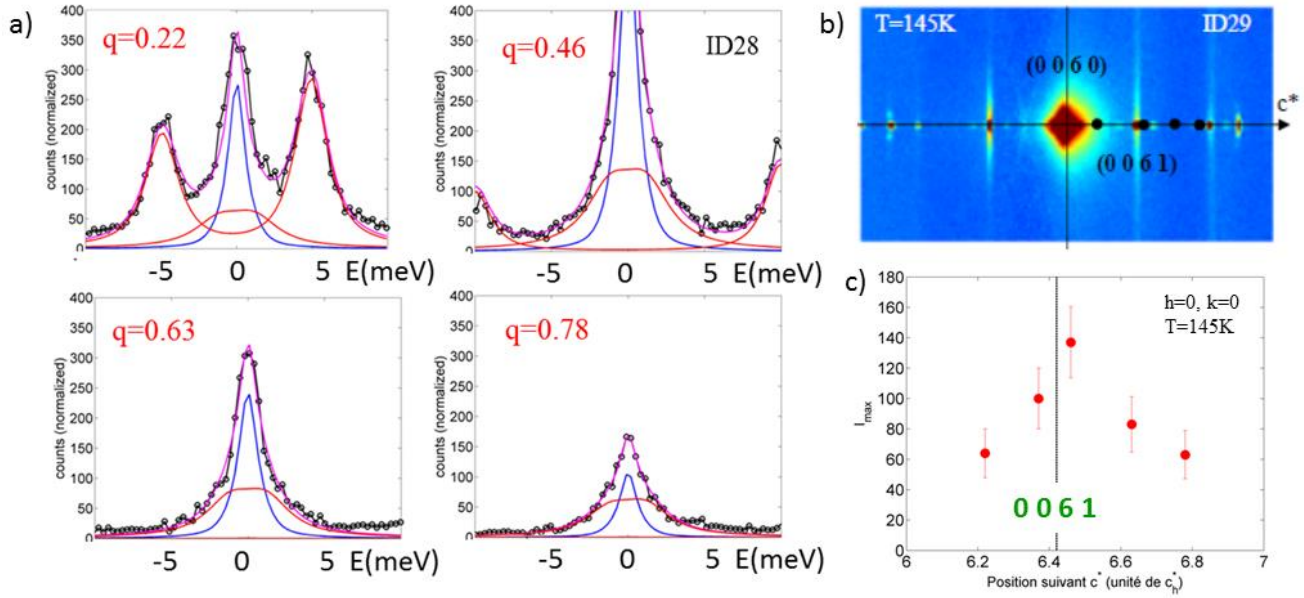

FIG : a) Energy spectra of *n*-nonadecane/urea as measured on ID28 spectrometer at T=145K at position (0 0 6+q 0) with q=0.22, 0.46, 0.63 and 0.78 showing in red the supplementary quasi-elastic component; b) Reconstructed diffraction image from measurements on ID29 beamline at T= 145K around that position, showing as black point the location of the inelastic spectra; c) Evolution along **c*** of the supplementary quasi-elastic component (in red on fig. a)).

[1] C. Mariette, Thèse de doctorat de l'Université de Rennes 1, 2013.