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.

ESRF	**Experiment title:** Van der Waals heterostructures based on 2D polymers	**Experiment number**: MA-5750
Beamline: ID10	**Date of experiment**: from: 12.05.2023 to: 16.05.2023	**Date of report**: 28.06.2023
Shifts: 6	**Local contact(s)**: Dr. Oleg Konovalev	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Proposal Objective

In this proposal, we studied the precise structure of novel van der Waals heterostructures (vdWHs) using different 2D materials like 2D polymers (2DPs), metal organic frameworks (MOFs), graphene or transition metal dichalcogenide. Although we have recently shown that the synthesis of 2D polyimide-graphene (2DPI-G) vdWHs at the liquid-air interface is possible there is still a lot to learn about the formation and precise structure of this novel class of material system and the combination of 2D materials in heterostructures.

Results

This was the first time that we measured with this setup at ID10, so the first task in analysing the data was to convert it into a format that can be imported into our visualisation and analysis software WxDiff. We were able to convert the data in binary image files including a calibration file that could now be analyzed.

In the course of our experiment we studied a variety of individual films and heterostructures (including 2DPs, 2D MOFs, MoS_2 and graphene) that have been prepared either at the water surface or via chemical vapor deposition (CVD). In addition to the fabrication method the deposition conditions have also been varied.

Here, we will focus on vdWHs made from MoS_2 and the 2D MOF Cu-BHT [copper(II) benzenehexathiolate] prepared under different conditions. Specifically, different types of MoS_2 were first fabricated by CVD at different temperatures and then thin films of Cu-BHT (around 20 nm) were grown on top of these MoS_2 layers by a second CVD step. As an example, we show the 2D GIWAXS images of a heterostructure of individual MoS_2 crystals with the Cu-BHT film grown on it (Fig. 1a,b) and one with a large are MoS_2 film and Cu-BHT deposited with the same growth conditions (Fig. 1c,d). Both samples show diffraction rings as well as spots with the sample with the MoS2 crystals showing stronger intensities. The diffraction spots are clear signatures of MoS_2 in the form of a Bragg rod at around $Q_{xy} = 2.3$ $Å^{-1}$. The actual position of the peaks varies slightly between the two samples resulting in different unit cells. We calculated the following cell for the sample with smaller MoS_2 crystals (Fig. 1a,b): a = b = 2.75 Å, c = 6.28 Å, $\alpha = 75.37°$, $\beta = 83.66°$ and $\gamma = 97.38°$. For the sample with the large area MoS_2 film (Fig. 1c,d) we determined the following lattice: a = b = 3.20 Å, c = 6.43 Å, $\alpha = 75.52°$, $\beta = 89.73°$ and $\gamma = 120.50°$. We also extracted possible unit cells of the Cu-BHT grown on top but here it needs to be mentioned that due to the limited number of diffraction rings for the sample with the MoS_2 film there is certain uncertainty in the unit cell meaning that there are other similar cells that could explain the diffraction pattern. We assume the unit cell to be monoclinic based on previous literature reports (DOI: 10.1002/anie.201707568) and the lattice for the film on the small MoS_2 crystals was calculated to be: a = 10.53 Å, b = 7.25 Å, c = 3.63 Å, α = 90°, β = 93.55° and γ = 90° and for the film on the MoS_2 film: a = 7.68 Å, b = 5.68 Å, c = 4.45 Å, α = 90°, β = 105.26° and γ = 90°.

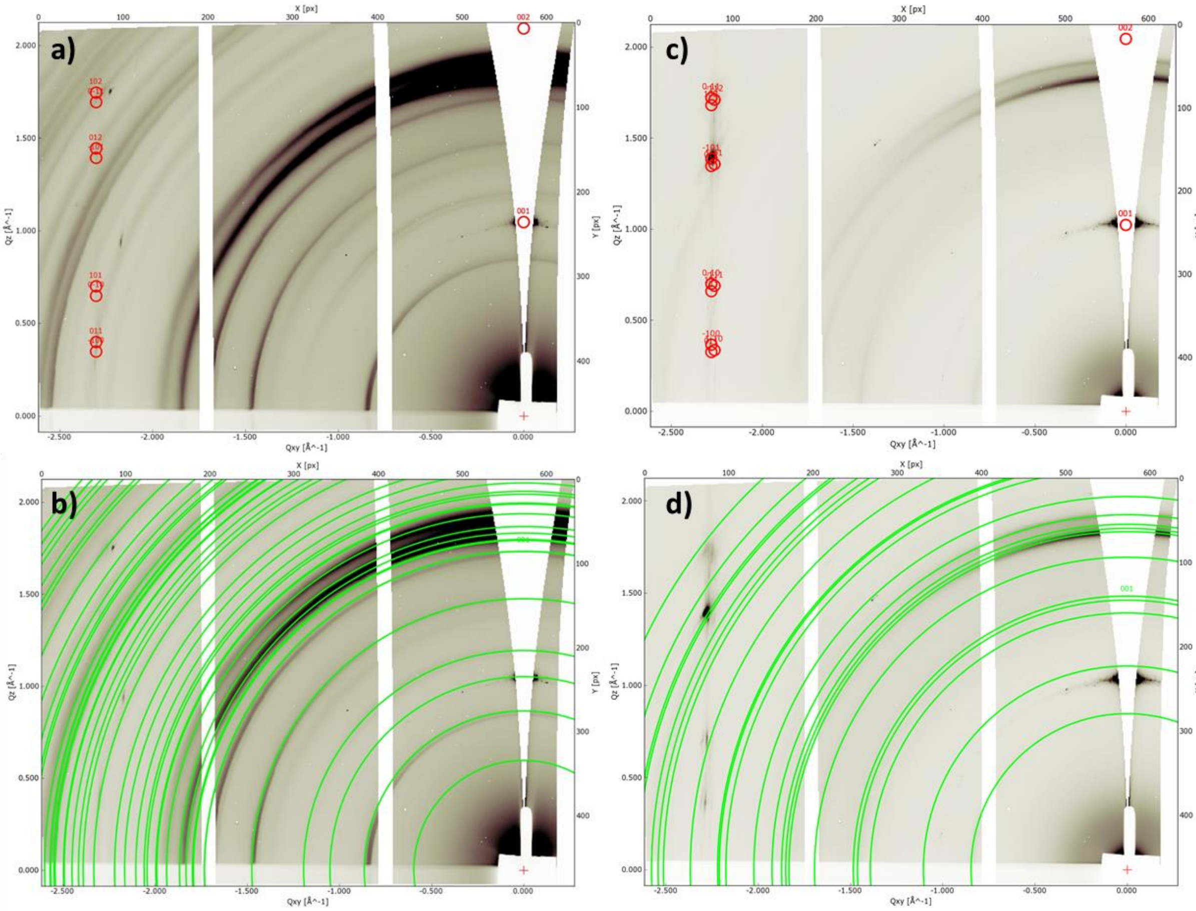

Figure 1: 2D GIWAXS pattern of a,b) a Cu-BHT film grown on MoS_2 single crystals and c,d) the same Cu-BHT on a MoS_2 thin film. The red circles in the images represent the calculated peak positions of the MoS_2 and the green rings the ones of the MOF.

Outlook

Further analysis of the collected data is currently ongoing for a variety of individual layers as well as heterostructures of various of these. The analysis will include indexing of the observed peaks and calculating the unit cells of the different crystalline films. We expect to be able to publish some of the data as part of scientific manuscript in high-impact journals. Furthermore, we are planning to submit new proposals for ID10 due to the very promising results of the experiment.