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: <u>https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do</u>

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, <u>you must submit a report on each of your previous measurement(s)</u>:

- 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 <u>each project</u> 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: In-situ formation and annealing in waterborne polymer coatings	Experiment number: 26-02-902
Beamline:	Date of experiment:	Date of report:
	from: 02/Nov/2018 to: 06/Nov/2018	
Shifts:	Local contact(s): Daniel Hermida-Merino	Received at ESRF:
Names and affiliations of applicants (* indicates experimentalists): Giuseppe Portale (Uni Groningen) Apostolos Vagias (Uni Groningen) Jingjin Dong (Uni Groningen)		

Report:

A system to allow in-situ GISAXS during slot-die coating (Fig. 1) of waterborne coatings was successfully tested during this beamtime at BM26B.

The great performances of the beamline allowed high quality data to be acquired with second time resolutions.



Automated slot die coating apparatus (DUBBLE BM26B, ESRF, Grenoble & MINA, RUG)

Figure 1. Experimental setup used for in-situ slot die coating experiments.



Figure 2. (left) GISAXS patterns at selected time for the hard (top row) and the soft (bottom row) investigated polyacrylic coatings. (right) Time evolution of the scattered intensity for the two studied coatings as revealed by in-situ GISAXS. Drastic intensity rise is related to formation of a "well defined" solid/air interface.

Three different formulations were investigated, named here H, S and HS. The beamtime went smooth and we had no problems with the performances of the beamline and the quality of the beam. The support of the beamline scientist was great. Our results are unexpected and clearly show the presence of a shear banding behaviour, especially for the hard colloidal coating. This is unexpected as generally the scientific community neglects shear behaviour during the slot-die and bar coating processing. Moreover, the structural evolutions is over within 10-15 min, while 30 minutes are necessary for macroscopic drying of the films according to weight changes with time.

We are currently finalizing the data analysis and we plan to write a manuscript with these high quality results.

Moreover, the success of these experiments and the previous 26-02-875 beamtime, have led to a successful new PhD project started on 1st December 2019 that will last for 4 years and will continue this investigation mostly at the ESRF and making use of the new capabilities of BM26 after the EBS upgrade.

Part of the acquired results have been included in a recent publication:

Vagias, Apostolos, et al. "Investigation of the Nanoscale Morphology in Industrially Relevant Clearcoats of Waterborne Polymer Colloids by Means of Variable-Angle Grazing Incidence Small-Angle X-ray Scattering." ACS Applied Polymer Materials 1.9 (2019): 2482-2494.