## 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 using the **Electronic Report Submission Application:** 

http://193.49.43.2:8080/smis/servlet/UserUtils?start

#### Reports supporting requests for additional beam time

Reports can now 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.

| <b>ESRF</b>   | <b>Experiment title:</b><br>Rapid surface kinetics of semiconductor polymers probed with an area detector | Experiment<br>number:<br>28-01-658 |
|---|---|------------------------------------|
| Beamline:   | Date of experiment:   | Date of report:                    |
| BM28  | from: 20 July 2004 to: 26 July 2004   | 21 Sept 2005                       |
| Shifts:   | Local contact(s):   | Received at ESRF:                  |
| 18  | Dr. Danny MANNIX  |                                    |
| Names and affiliations of applicants (* indicates experimentalists):  |   |                                    |
| J E Macdonald*, A Das*, H Thomas*   |   |                                    |
| School of Physics and Astronomy, Cardiff University, 5 The Parade, Cardiff CF24 3YB, UK                               |   |                                    |
| S Droege*   |   |                                    |
| Department of Physics, The University of Hull, Cottingham Road, Kingston upon Hull HU6 7RX, UK                        |   |                                    |
| <b>R A L Jones</b><br>Department of Physics and Astronomy, University of Sheffield, Hounsfield Road, Sheffield S3 7RH |   |                                    |

### **Report:**

The main aim of the experiment was to install and use the MAR area detector for surface-sensitive diffraction for the first time, to investigate the scattering from poly(9,9-dioctylfluorene) (F8), a blueemitting liquid-crystalline polymer. We have characterised the structure of thin films of F8 using grazing incidence x-ray diffraction (GIXRD): the chains layer normal to the interface with the inplane ordering depending on the temperature [1], hence it is a suitable material for developing the area detector facility.

The experimental requirements are quite challenging. When measuring the surface structure with angles of incidence  $\alpha < \alpha_c$ , the beam penetration is 5-7 nm and the scattering from a polymer surface is very weak ( $\leq 10^{-8}$  of the incident beam flux). This signal is easily swamped by scattering from any windows in the direct beam that are visible to the detector. With point detectors, this scattering is trivially discriminated using collimating slits on the detector arm. When using an area detector, it is not possible to shield this scattering and hence windows need to be excluded from the space visible by the detector.

In the proposal we described a vacuum chamber designed to reduce the scattered background from windows, which was sent to an external company for detailed design and drawing. We also built a 25 cm diameter scattering chamber in Cardiff with kapton windows for a helium atmosphere as a reserve chamber. Due to delays in the design stage, this chamber was employed for the experiment and performed extremely well.

Fig. 1 shows the area detector image of the scattering from the F8 film together with the corresponding point detector scans performed previously [1]. The peaks are consistent with an orthorhombic unit cell with a = 2.56 nm, b = 2.34 nm, c = 3.32 nm (with the *b*-axis normal to the substrate surface). Fig. 2 shows sections for different  $\alpha$  values, indicating limited difference in structure between the structure within 5nm of the top surface of the film and that in the bulk of the film.



Fig. 2 Scattering in the plane of the surface for F8 for varying angles of incidence. The critical angle for the F8 is  $0.18^{\circ}$  and  $0.20^{\circ}$  for the thermal oxide on the silicon surface.



#### References

 X-ray diffraction study of the structure of thin polyfluorene films, S. Kawana, M. Durrell, J. Lu, J. E. Macdonald, M. Grell, D.D.C. Bradley, P. Jukes, R.A.L. Jones and S.L. Bennett, Polymer 43 (2002) 1907-1913