EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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 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.

ESRF	**Experiment title:** Structural dynamics of excimer formation in thin films of Perylene	**Experiment number**: HS-3104
Beamline: ID09B	**Date of experiment**: from: 26/01/2007 to: 30/01/2007	**Date of report**: Monday, February 26, 2007
Shifts: 12	**Local contact(s)**: Marco Cammarata	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The original purpose of the beamtime was to study the excimer formation in thin films of Perylene in the α-form. In a previous beamtime[1] we saw some promising results, which we were going to investigate further during this beamtime. Unfortunately the chopper used at ID09b to create the very short X-ray pulses was being repaired for the full duration of our scheduled beamtime, hence only a time resolution of 50μs was obtainable, which is much to slow for us to observe the excimer formation or decay, as we have seen in previous experiments that the system is back to its original state within 30μs after laser excitation.

We instead used the beamtime to test and implement several improvements to our experimental setup, which will allow us to better control our experiment. We also examined the long term heating effects arising from continuous laser irradiation, which we have observed in earlier experiments, hoping to gain a better understanding of this and possibly find a way of avoiding the heat build-up in the sample during future time-resolved experiments without having to decrease the laser intensity.

The first improvement we implemented was a second motor allowing us to rotate not only the phi angle, but also the chi angle – the idea is that running the motor continuously during X-ray exposure will allow us to average over several of the nano-crystallites in the sample, which will give a smoother peak structure and thus allow us to better determine the time evolution of the peak positions. Also rotating the sample during exposure will give each sample site a longer break between laser and X-ray exposures, which we hope will not only lengthen the lifetime of the sample but also help eliminate the long term heating effects.

[1] ME-1186

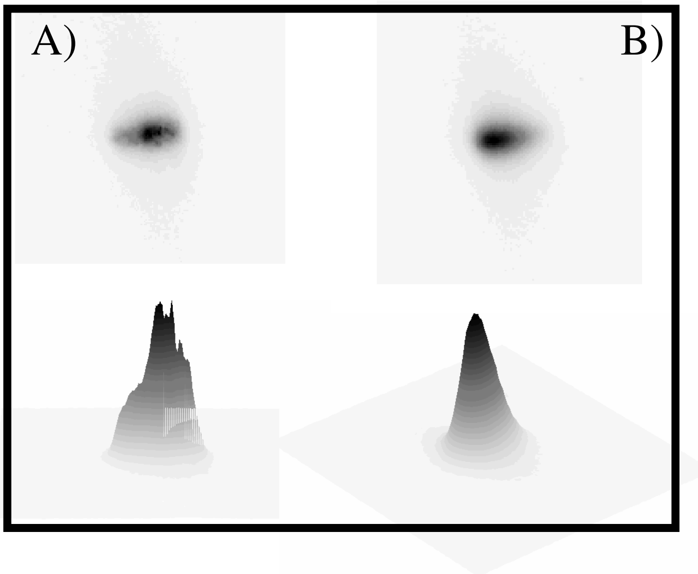

Fig. 1) Images of a peak with and without rotating the sample during X-ray exposure. Top a 2-D projection and bottom a 3-D image. A) No rotation of the sample during exposure. B) Rotation of the sample during exposure.

As seen in Fig.1 rotating the sample during the X-ray exposure really smoothes the peak giving us a much better peak profile for determining the peak center, which will be a great help for determining the peak shifts observed in earlier experiments.

The second improvement is the addition of a spectrometer, which will allow us to monitor the fluorescence spectra of the sample during the experiments. This will not only make it possible for us to ensure that excimers are being formed after laser excitation, but also allow us to optimize the number of excimers by looking at the fluorescence signal, while changing different parameters like laser polarization and intensity. Finally it will allow us monitor any changes in the sample quality during the experiments.

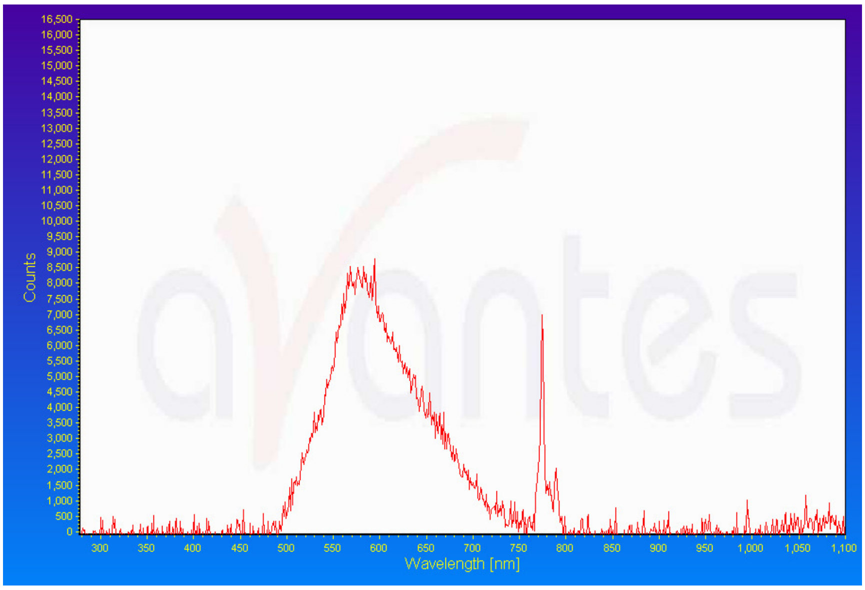

Fig. 2) Fluorescence spectrum recorded with the spectrometer showing the number of photon counts as a function of wavelength. The broad peak is the excimer fluorescence and the small thin peak is leftover 800nm from the laser.

We were able to see the fluorescence from the excimers as seen in spectrum in Fig. 2, but we discovered that the spectrometer was probing the fluorescence from a too large area of the sample, hence we were not able to see when the sample were destroyed in the X-ray probed area just by looking at the fluorescence spectra. To solve this problem we are at the moment making and imaging setup, which will allow us to image just a small area of the sample onto the fiber of the spectrometer; this will hopefully give us the needed sensitivity for these experiments.

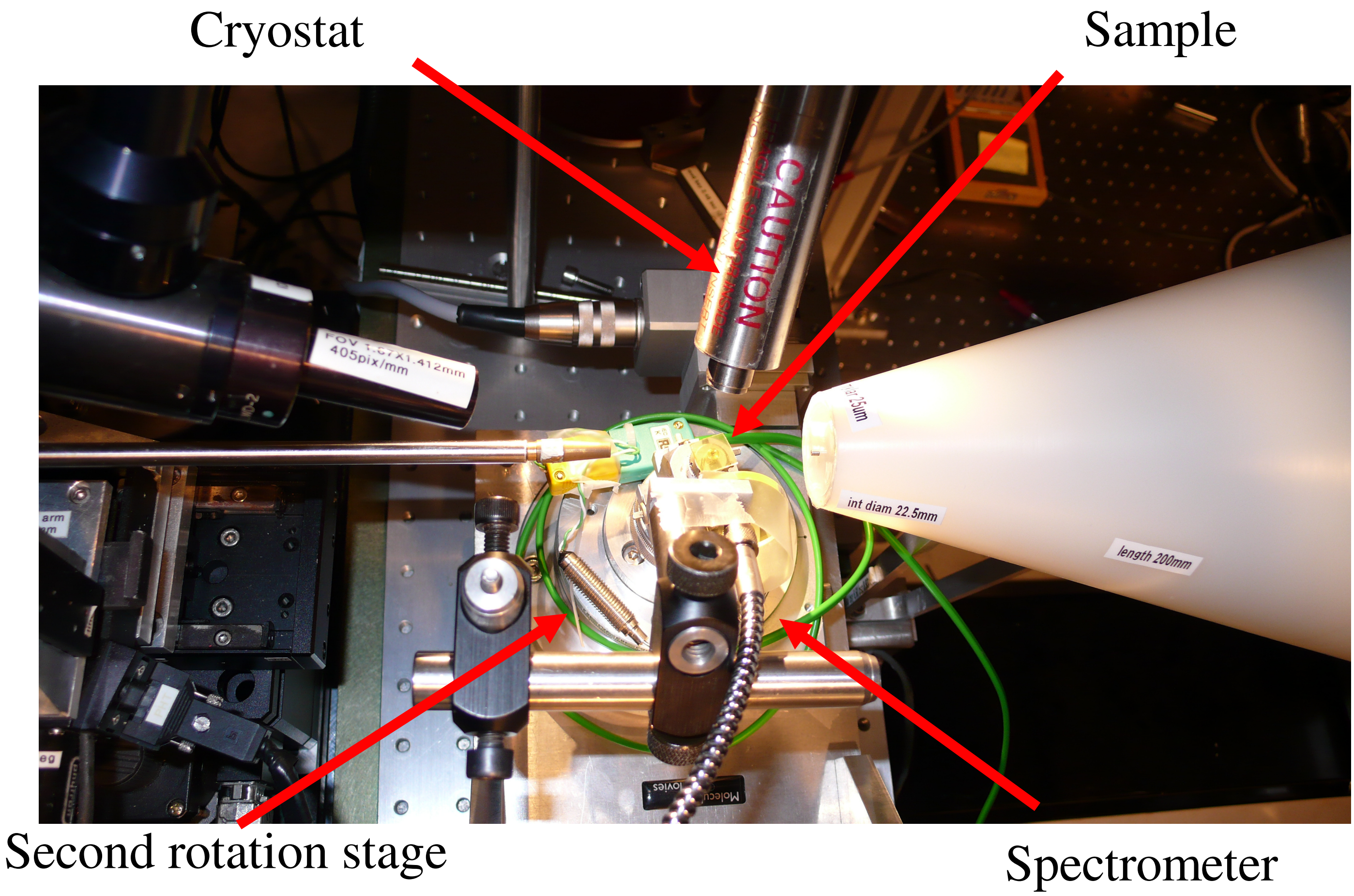

Fig.3 The final setup after all improvements were added.

To study the long term heating effects we kept the sample stationary and recorded a series with the time delays 0, 100, 300 and 900µs and compared the images to laser off images at different laser intensities and different cooling conditions. This allowed us to investigate if the sample was back to its original temperature before being hit by the next laser pulse, if the sample is not back to its original temperature before the next excitation pulse hits the sample, it will slowly heat up during the experiment, which might influence our results or at least complicate the data analysis.
We discovered that even just having the cryostat letting out a very small air stream keeping the sample at room temperature had a positive effect and helped the sample release the excess heat that the laser excitation produces in the sample.

To sum up the results, despite the problems with the beamline chopper, the excellent support from the beamline staff made it possible for us to have a very productive beamtime, where we managed to implement two improvements that will help us a great deal in future experiments. Also we managed to study the long term heating effects, which have been giving us trouble in the data analysis and found ways of reducing them greatly.