



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://www.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.



	Experiment title: Structural dynamics of a 1D conducting thin film	Experiment number: MA-1864
Beamline: ID09B	Date of experiment: from: 22 april 2013 to: 27 april 2013	Date of report: March 2014
Shifts: 12	Local contact(s): Dmitry KHAKHULIN, Michael WULFF	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Morten Christensen*, Christian Rein*, Kristoffer Haldrup*, Tim Brandt van Driel*, Martin Meedom Nielsen*, Kasper Skov Kjaer*, Asmus Ougaard Dohn§, Tobias Harlang# Elisa Biasin, Peter Vester ^ Affiliation, unless otherwise indicated: Dept. of Physics, Technical University of Denmark # Chemical Physics, Lund University, Sweden ^ University of Copenhagen, Denmark § Dept. of Chemistry, Technical University of Denmark		

Report:

Purpose, Expected Results, Preparation:

The purpose of the beamtime was to study the structure-function relationship in a 100 nm thin film of unidirectionally aligned chains of potassium tetracyanoplatinate.

The semiconducting compound potassium tetracyanoplatinate(II) ("TCP") forms needle-like crystals with uniaxial growth along the Pt-Pt axis. Zone-casting type thin film fabrication of the compound produces thin films ideal for studying laser-induced structural changes at the molecular scale: Upon exposure to light in the UV-region, electrons populate the conduction band of the Pt-chain thereby inducing 1D-conduction properties for the excited state system. Photo excitation in the UV causes population of the $\sigma(6p_z)$ -orbital between the Pt-nuclei, which in addition to the conduction character has bonding-like properties.

By laser-exciting a thin film of aligned TCP-films, we thus expected to monitor a contraction of the Pt-atoms along the Pt-Pt axis, in the affected domains of the thin film.

The TCP-films was produced on glass slides using a novel, zone-casting type fabrication technique. Prior to the beamtime, the samples where characterized using atomic force microscopy (AFM) polarized UV-VIS absorption and emission spectroscopy and grazing incidence x-ray scattering using a standard lab-source.

Method:

The laser pump – X-ray probe setup at beamline ID09B was used. An array of rotating chopper wheels isolates a single X-ray pulse from the synchrotron and the sample is excited by a femtosecond laser pulse with selected arrival time prior to arrival of the X-ray pulse. A rotation stage for Grazing Incidence X-ray Diffraction of thin films was mounted on the goniometer, and the sample was placed in GIXD-geometry for observation of selected hkl-reflections.

Setup:

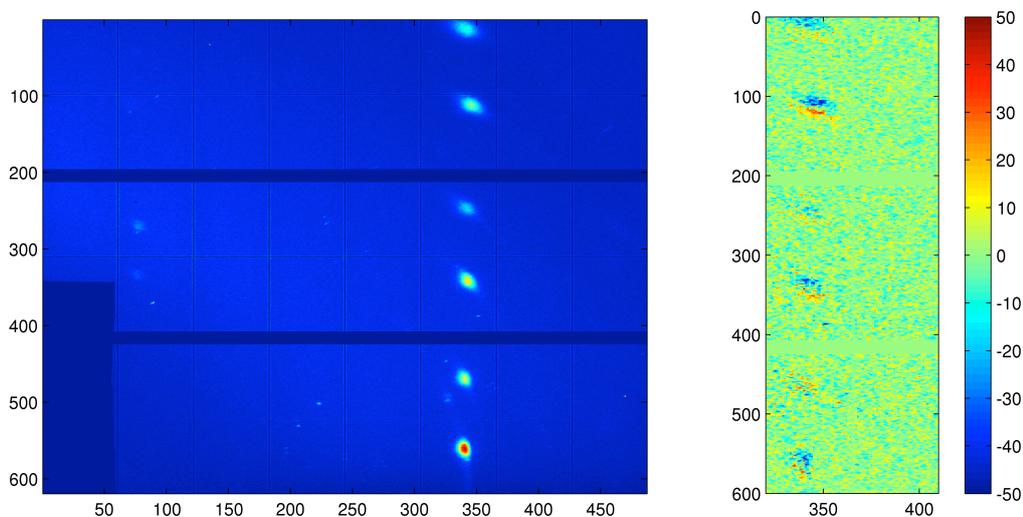
The experiment was performed using a pink 15 keV X-ray beam reflected off the Ru-multilayer to get rid of un-desired X-ray arising from e.g. higher harmonics. The sample was excited using a 266 nm laser beam and the diffraction images collected on a Pilatus 300 K detector.

Results:

We managed to get nice steady-state diffraction data for a variety of samples (cf. image below) but was not able to monitor any structural change other than what could be attributed to trivial heating induced by the laser beam. Furthermore, we tried measuring films with different thickness in order to be able to characterize the acoustic phonon contribution to the signal, but again without being able to accurately catch any phonons within experimental uncertainty.

We noted loss of scattering intensity, that was accelerated by application of nitrogen flow (in order to cool the sample). We attributed this loss in intensity to degradation of the crystal due to heating and air-flow induced loss of crystal water from the films.

Single image & difference between an off image(-3ns) and an on image (900ps)



Conclusions and Outlook:

Despite not being able to acquire the data we expected, we have learned some vital points about the thin film systems and their behavior in a Laser Pump/X-ray Probe experiment. These learnings will be incorporated in the forward strategy for thin film systems in our group and will hopefully result in future X-ray studies of the thin films.