EUROPEAN SYNCHROTRON RADIATION FACILITY

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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 via the User Portal:

https://wwws.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.

ESRF	**Experiment title: Nanosecond surface X-ray diffraction study of Pt(110)/(1x1) -> (2x1) reconstruction formation kinetics**	**Experiment number**: HC-2294
Beamline: ID03	**Date of experiment**: from: 29.09.2016 to: 05.10.2016	**Date of report**: 15.03.2017
Shifts: 18	**Local contact(s)**: Maciej Jankowski <maciej.jankowski@esrf.fr>	*Received at ESRF:* *15.03.2017*

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Report:

During the scheduled beamtime the nanosecond grazing incidence pump-probe surface X-ray diffraction experiment has been carried-out at ID03 beamline for the first time.

During the experiment we have sucessfully prepared the Pt(011) single crystal surface under UHV conditions. Bright (0,3/2,0.01) surface X-ray diffraction peak of 1e7 cts originating form the Pt(110)-(1x2) “missing row” reconstruction has been observed.

In the initial phase of the experiment we have captured the “single bunch” signal by delaying the Pilatus 300K X-ray detector gate with respect to the ring clock. We have observed a non-uniform gating of the Pilatus photosensitive area that has not been reported before. The results of this part of the experiment are now described in a short communication letter submitted to the Journal of Synchrotron Radiation.

In the next step of the experiment the “time zero” has been determined by detecting the laser pulses arrival time at the sample position with respect to the ring clock and the detector gate. A fast oscilloscope, a photodiode and a delay generator was used to monitor and control the laser emission timing.

To calibrate the laser excitation power and to identify the time-zero more precisely we have first colleced the transient signal in the vicinity of Pt(110)-(022) Bragg peak in the grazing incidence geometry. It was necessary to use the relatively large “grazing” angle of 2 degrees due to the finite size of the laser beam spot. The time zero has been determined with the accuracy of around 1 nanosecond limited by the laser emission jitter. Using the area X-ray detector we have collected transient X-ray diffraction signal in certan volume of the reciprocal space along the 02L crystal truncation rod in the time range of +-50 ns after the laser excitation. Figure 1a shows the KL-slice of the reciprocal space through 022 Bragg peak at time zero and Figure 1b shows the same region recorded at 25ns after the laser excitation. Figure 1c shows the signal difference map demonstrating strong transient shift of the CTR signal along L towards negative direction. This is known as the transient thermal expansion effect of the near-surface crystal lattice due to the impulsive laser pulse heating. The estimated probing depth at this conditions is on the order of 200nm and the peak transient temperature did not exceed 150K. The important new observation is the fact that the CTR signal shifts only along L, without any statisticvally significant effect in the in-plane HK-direction. This effect may be due to the ultrafast nature of the thermal expansion in this experiment and is currently under analysis.

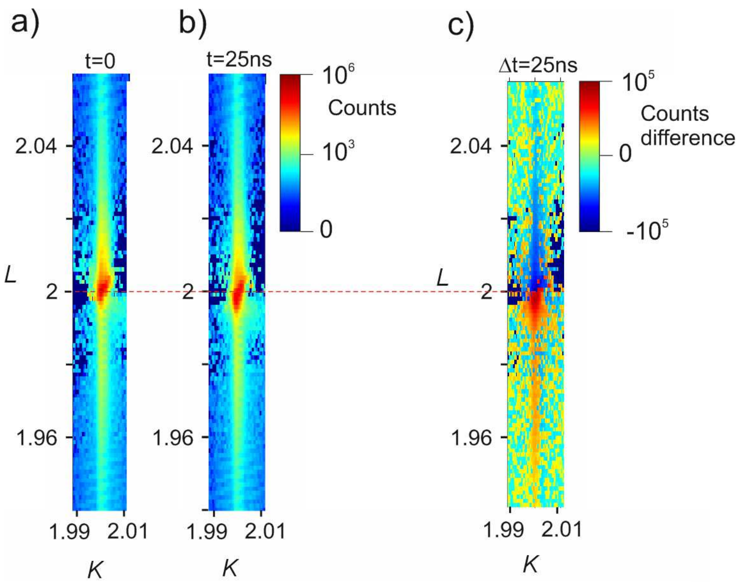

Figure 1. a) KL-slice through reciprocal space recorded near 022 Bragg peak at “time zero” and b) recorded at 25ns after the laser excitation, c) their corresponding difference map

In the next step of the experiment we have recorded a set of transient CTR signals from the (0 3/2 0) surface Bragg peak. At room temperature and even at elevated up to 300C temperatures the surface peak did not exhibit any statistically significant dynamics on ns timescales. Employing the two facts that the thermal expansion in our experiment results in the peak shifts exclusively along the L-coordinate and that the (0 3/2 0) surface reflection has rod-like structure also along the L-coordinate we concluded that the thermal expansion effect cannot be easily seen from the shifts of the surface diffraction peaks. As the next step we attempted to heat the crystal to the near (1x2)<->(1x1) phase transition point to promote the surface atom dynamics. Unfortunately, due to the technical failure in the sample heating system we were not able to proceed with sample heating to the HT. The post beamtime investigation showed that we have got a short circuit between the filament and the ground eliminating the possibility of thermoelectron emission heating requried for reaching the order-disorder surface temperatures of 600C.

The heating stage is now replaced by a new one. Evaluating the first results as “very promising” we are very interested in repeating the experiment. Therefore, we resubmit the original proposal.