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

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

- 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 each project 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:** RIXS signal multiplication through the Kossel effect from X-ray cavities	**Experiment number**: MI 1335
Beamline: ID20	**Date of experiment**: from: 18/07/2018 to: 24/07/2018	**Date of report**: 02.03.2021
Shifts: 17	**Local contact(s)**: Blanka Detlefs	*Received at ESRF:*

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Report:

The experiment aimed at using the Kossel effect in artificially grown planar X-ray cavities to excite fluorescence / resonant inelastic X-ray scattering (RIXS) at selected depths inside the cavity as well as enhance the obtainable signal through focussing the emitted signal into Kossel cones. In order to explore all dependecies of the effect most efficiently, a Von Hamos analyzer with a 2D detector was used to record the emission spectral and angular distributions at the same time, while scanning incident energy and incident angle.

Setup:

The planar X-ray cavities were grown on super-polished silicon wafers with platinum layers to act as mirrors below the critical angle. Carbon was used as mostly transparent spacer medium and either metallic iron or iron oxide (Fe_2O_3) was grown in the centre of the cavity as sample layer. The cavities were irradiated by the x-ray beam at an adjustable grazing incident angle. A photodiode monitored the specular reflection of the beam and allowed to observe the "incident" cavity modes and their dependence on incidence angle and energy. The fluorescence was emitted from the cavity in cones of constant emission angle to the cavity surface (for each energy and cavity mode) and dispersed onto a 2D detector by a spherically bent Von Hamos analyser crystal. The crystal had a radius

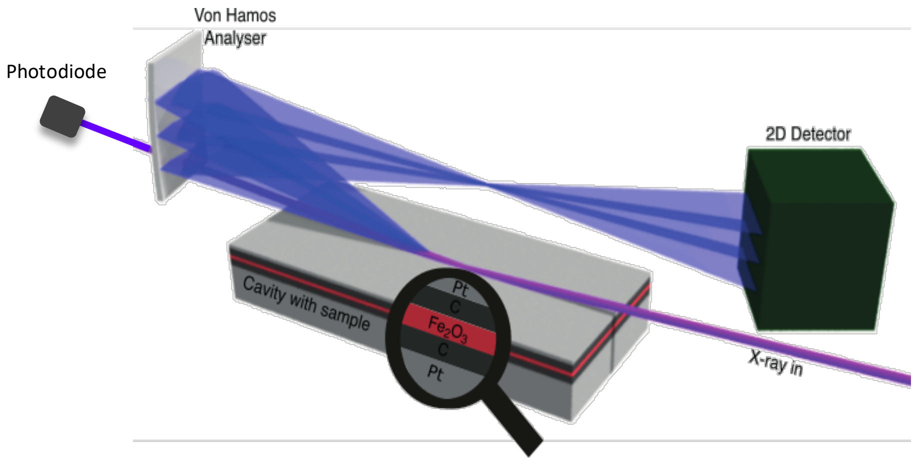

Figure 1: Schematic layout of the X-ray cavity and spectrometer setup at the ID20 beamline.

of 250 mm and used the Si (333) planes to disperse horizionally. The detector was placed behind the focus to conserve the information in the vertical direction. A Pilatus 300K-W 2D detector was used to record the Fe $K\alpha$ emission lines while resolving the emission angle from the sample.

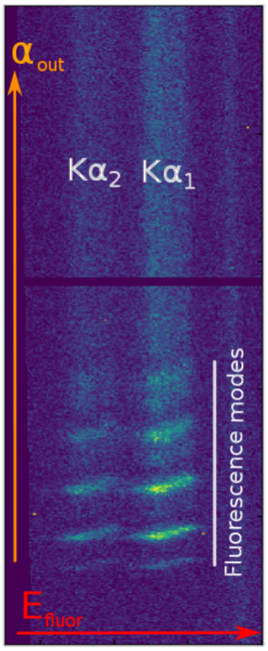

Figure 2: Exemplary detector image from the Pilatus detector, showing the Fe $K\alpha$ emission as a function of emission angle.

Experimental Results:

The iron fluorescence shows a strong modulation with emission angle, as is visible on each detector image (see Figure 2). Thus, each acquisition maps the fluorescence intensity as a function of emission angle and emission energy. However, the fluorescence also depends strongly on both the incident energy and incident angle of the synchrotron beam onto the cavity. This dependence was carefully mapped by scanning incident angle and incident energy, while recording the 2D detector images as well as total fluorescence and the specular reflection intensity with two separate photodiodes.

Apart from smaller datasets on a cavity with metallic iron, the main result of this beamtime comprises of a four-dimensional dataset on a cavity with Fe_2O_3, mapping the fluorescence intensity over the incident energy (7.1 keV to 7.15 keV), incident angle (0.32 ° to 0.46 °), emitted energy (6.38 keV to 6.41 keV) and emission angle (0.0 ° to 4.0 °).

Encountered Problems:

Initially, we observed only weak signals and a substantial amount of stray light. This was mitigated by covering most of the beam path with a helum-filled pipe and by shielding potential stray light paths to the detector with lead. Furthermore, we observed degradation of the cavity performance over time, which we connected to a growing contamination layer on the cavity surface under X-ray irradiation in air. A rough vacuum hood with Kapton windows was built around the cavity and reduced further contamination.

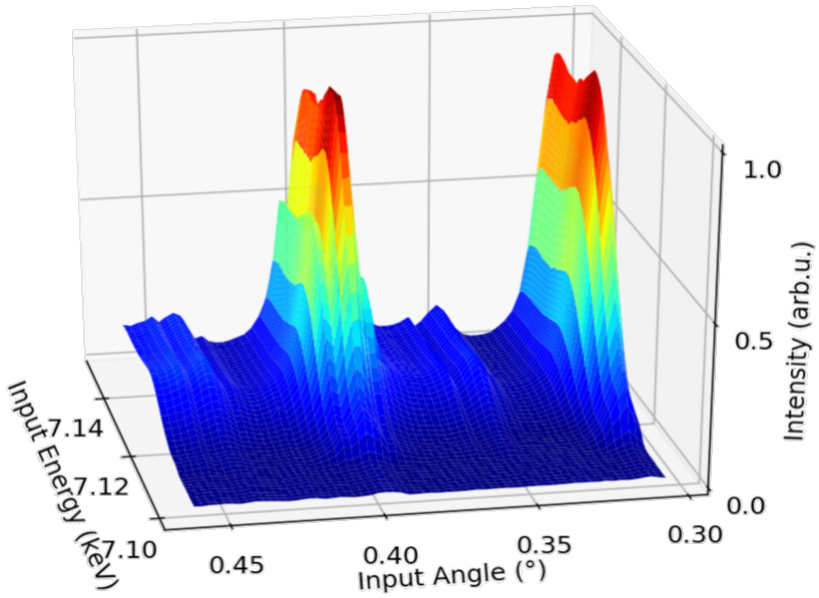

Figure 3: Fluorescence yield from Fe_2O_3 ,integrated over emission angle (0 ° to 4 °) and energy (6.38 keV to 3.41 keV), covering four modes of standing waves of the incident beam within the cavity. For modes with the standing wave maxima within the sample layer (at incident angles 0.41 ° and 0.32 °), the inelastic signal is enhanced, while it is suppressed for modes with the node located at the sample layer (at incident angles 0.45 ° and 0.36 °).

Data Quality:

While the dataset on the metallic iron cavity only spans a small parameter space in input energy and angle with consistent quality (due to the problems mentioned above), the four-dimensional dataset of the Fe_2O_3-containing cavity shows a variety of interesting effects (see Figure 3) with good and publishable data quality. The observed effects have more structure than expected from a simple model and quantitative simulations are ongoing for a detailed explanation.

We currently plan to publish the data in two papers.