	Experiment title: Investigating the use of dynamical diffraction effects in resonant x-ray spectroscopy	**Experiment number**: HC-2875
Beamline: ID20	**Date of experiment**: from: 07 Dec 2016 to: 14 Dec 2016	**Date of report**: 11/3/2017
Shifts: 18	**Local contact(s)**: Christoph Sahle	*Received at ESRF:*
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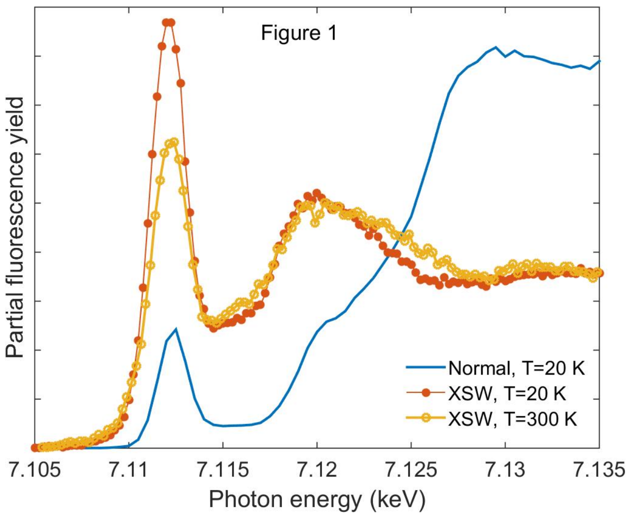
Report: This experiment was a sequel to our earlier one on x-ray standing wave (XSW) RIXS [Sci. Rep. 6, 22648 (2016)]. The experiment was performed using the ID20 RIXS spectrometer. The beam was monochromatized with a Si(111) premonochromator. We tuned the incident beam optics such that the beam divergence was minimized (~50 microrad) while keeping the beam spot size on the sample manageable (few hundred micrometers).

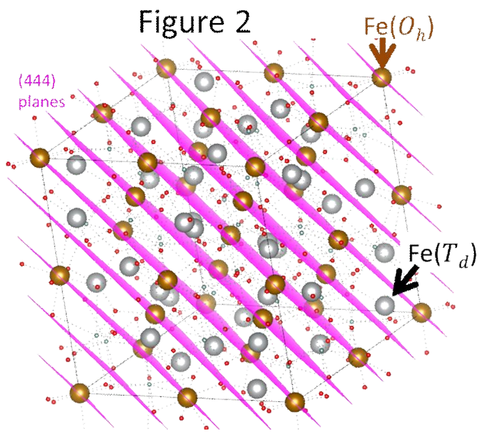
In order to optimize the setup, we first reproduced the main result of the earlier experiment, which dealt with Gd L_{III} edge resonant x-ray emission spectroscopy (RXES) on gallium gadolinium garnet (GGG). For this we used total of three bent Si(333) analyzer crystals, aligned a GGG crystal to a (008) reflection at 7.25 keV, thus creating a standing wave within the crystal and measuring RXES spectra excited by anomalously transmitted x-rays of the standing wave field. We improved the experiment since last time by this time having a wedge-shaped crystal in which the thickness is tunable for optimized transmission experiment, we cooled the sample with a cryostat in order to minimize thermal motion of the atoms, and had an better optimized beam for lower divergence. The improvement of the signal with the learning curve after the previous experiment was found to be ~10-fold.

Next we went on with a new sample of yttrium iron garnet (YIG) for 1s2p RXES (measuring Fe $K\alpha_{1,2}$ and Fe $K\beta_{1,3}$ excited at the Fe K edge) with Ge(440) analyzer crystals, utilizing a YIG(008) reflection for the XSW creation.

By creating an XSW field and tuning the location of the nodes and antinodes, one can effectively avoid the heavily absorbing atoms and the x-ray beam is anomalously transmitted (and a Laue-diffracted beam emerges from the back side of the crystal). Simultaneously, electric quadrupole absorption is enhanced.



This was seen in our earlier publication on GGG, and we could reproduce the same effect on YIG at the Fe K edge. Fig. 1 shows the partial fluorescence yield (PFY, a.k.a. high-energy-resolution-fluorescence-detected XAFS, HERFD-XAFS) spectra of YIG with and without XSW condition. The quadrupolar pre-peak is greatly enhanced with respect to the dipolar features of the spectra. The effect of temperature was also studied, and the phenomenon was stronger at low temperatures as expected (T=20 vs. 300 K).



In YIG, the 5 Fe^{3+} ions occupy two octahedral (Oh) and three tetrahedral (Td) sites. In the second part of the experiment, we turned our attention to the possible separation of the PFY spectra of the two sites. Fig. 2 shows the unit cell of YIG. Golden atoms are Fe(Oh) and gray ones Fe(Td) (oxygen red, yttrium cyan, both depicted as very small for enhanced visibility of Fe). By tuning across the YIG(444) Bragg reflection one can selectively neglect or enhance the relative signal from the Fe(Td) sites by tuning the nodes or antinodes of the XSW onto the (444) planes that contain those sites. The experiment is done by measuring PFY spectra with various rocking angles across the reflection, measuring the variations of the spectra as a function of the rocking angle. Figs. 3-4 shows the result of such a data set. The data is currently being processed. The difference of the Td vs Oh site signal is small but visible and we saw the same effect in both Kα and Kβ RXES. The spectra are expected much more sensitive to the oxidation state than the site symmetry, and thus a system with varying oxidation states is expected to yield even larger contrast, hence next experiments are being planned on a set of new systems with varying oxidation states in different crystallographic sites. The Fe 1s pre-edge PFY spectra were in our case energy-resolution-limited, but resolution can be enhanced for the next experiment.