| ESRF | **Experiment title:** Temperature dependent Fe 1s2p RIXS-LD on $Fe_3O_4$ single crystal | **Experiment number:** HC-2889 |
|---|---|---|
| **Beamline:** ID20 | **Date of experiment:** from: 29 Nov 2016 to: 06 Dec 2016 | **Date of report:** 27/02/17 |
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## Report

### Introduction:

We investigated the angular dependence of Fe 1s2p RIXS linear dichroism (LD) on a (110) $Fe_3O_4$ single crystal with the aim to understand the electronic and magnetic ground state of Fe. The experiment makes use of the constraints imposed by the crystal symmetry and the scattering geometry and can hence reveal information smaller than the instrumental energy bandwidth. A new setup was developed at ID20 which enables us to rotate the sample and the magnetic field independently in a cryostat (Fig. 1).

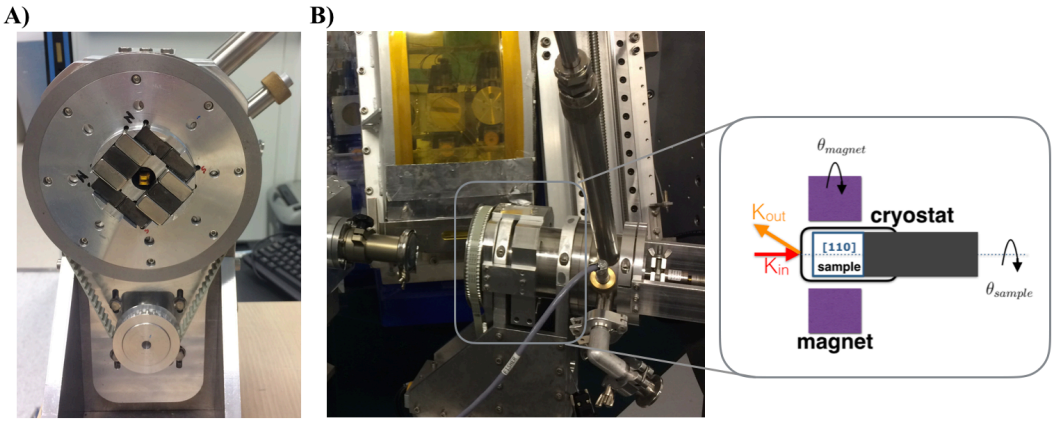


**Figure 1:** Setup developed at ID20. **A)** Front view showing the magnets arrangement surrounding the cryostat. The magnetic field is ~0.5T in the centre. **B)** Side view showing the scattering geometry used. A cartoon on the right illustrates possible rotations in the setup ($\theta_{sample}$ and $\theta_{magnet}$ are the rotations of the sample and the magnet).

### Measurements:

We studied two $Fe_3O_4$ single crystals that were skull melter grown at Purdue University and characterised using XRD and magnetic measurements to ensure their correct orientation and stoichiometry.

| Sample | T=273K | T=140K | T=20K | HERFD | RIXS | Magnet rotations | Sample rotations |
|---|---|---|---|---|---|---|---|
| (110) | ✓ | ✓ | | ✓ | ✓ | ✓ | ✓ |
| (100) | ✓ | | | ✓ | | ✓ | ✓ |

**Table 1:** List of the measurements performed during the beam time

## Results:

We observed strong dichroic signal at the Fe pre-edge of $Fe_3O_4$. Fig. 2A shows Fe HERFD-XANES measured with the incident polarisation ($E_{in}$) parallel to [0 0 1] direction and parallel to [1 -1 0] (the orientations are labeled as (0,0) and (90,90) orientation respectively). We observe four main dichroic features of around 3% modulation with respect to the edge as shown in Fig. 2B. We studied the angular dependence at the four observed features by rotating the sample together with the magnet (B) about the surface normal (Fig. 2C). Based on multiplet calculations we assigned features 1,2 and 4 to $Fe^{2+}$ $O_h$ site.

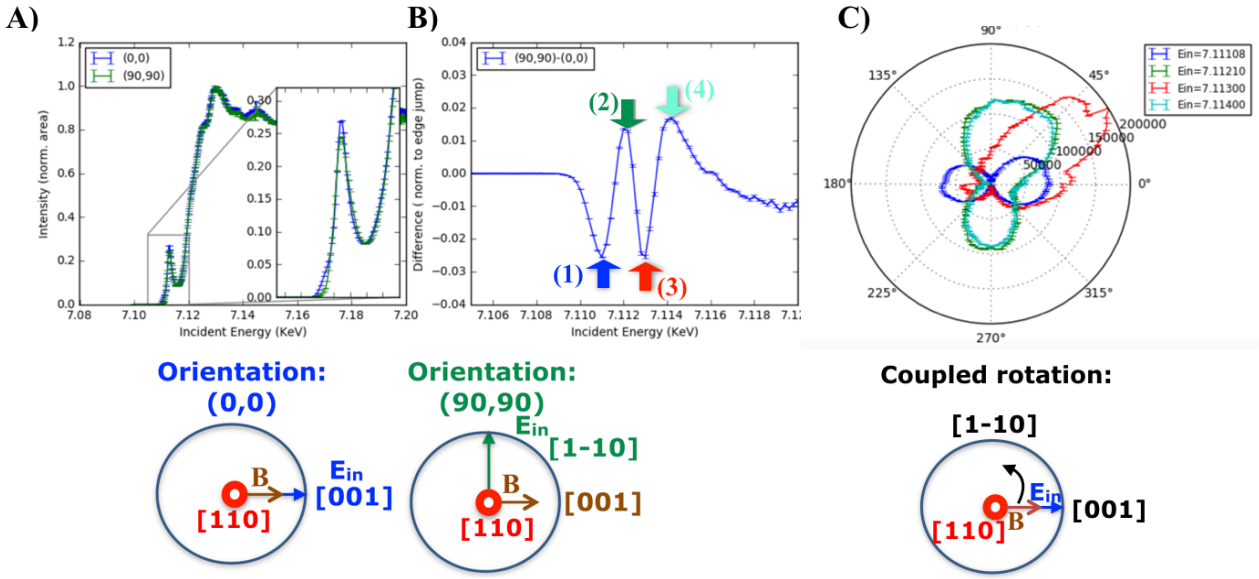


**Figure 2:** Fe K-edge measurements of $Fe_3O_4$. **A)** HERFD-XANES measured at two orientation: in blue with the incident polarisation ($E_{in}$) and the external magnet (B) // [0 0 1] direction, in green with $E_{in}$//[1 -1 0] and B//[001]. The cartoon below shows a sketch the scattering geometry. **B)** The dichroism signal. **C)** The angular dependence of the four dichroic features measured rotating the sample and the external magnet.

In order to probe the magnetic moments of Fe in $Fe_3O_4$, we rotated B relative to the sample. As the direction of B changes, the net spin moment aligns with B. If the orbital moments are not fully quenched, the orbital moments consequently also re-align. The final orientation of the orbital moments depend on the strength of the competing interactions such as spin-orbit coupling, distortion and temperature. Fig. 3 shows the angular dependence observed as we rotate the magnet at four different crystal orientations.

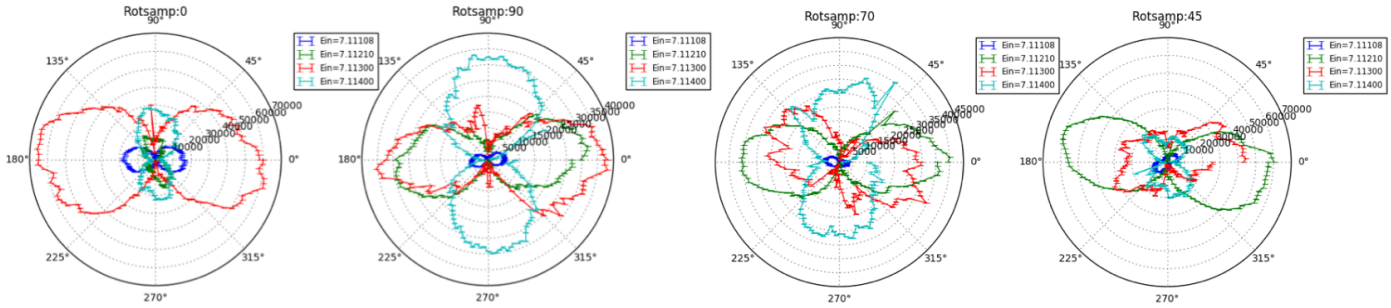


**Figure 3:** Angular dependence as we rotate the external magnet relative to the sample. The measurements are at four incident polarisations ($E_{in}$) orientations namely $E_{in}$// [0 0 1],[1 -1 0], [0.7 -0.7 0.3] and [0.5 -0.5 0.7].

## Conclusion:

RIXS-MLD is a sensitive probe of the magnetic and electronic structure that reveals interactions smaller than the instrumental energy bandwidth because the experiment makes use of the constraints imposed by the crystal symmetry and the scattering geometry. Our results show that the orbital moments of formal $Fe^{2+}$ sites are not fully quenched as evident by the strong magnetic angular dependence. We expect further detailed 1s2p RIXS-XMLD measurements together with detailed calculations will elucidate the ground state and quantify the orbital moments of Fe in $Fe_3O_4$.