Resonant diffraction with polarisation analysis of YFe_2O_4

T. Mueller, J. de Groot, S. Adiga and M. Angst

Peter Grünberg Institut PGI and Jülich Centre for Neutron Science JCNS, JARA-FIT, Forschungszentrum Jülich GmbH, 52425 Jülich, Germany

At P09 at Petra III we measured resonant x-ray diffraction, which was not done on stoichiometric YFe_2O_4 before, and searched for anisotropy, possible caused by orbital ordering, in the x-ray polarization on superstructure reflections. Stoichiometric YFe_2O_4 showed multiple charge ordered phases below 240 K in recent electron diffraction experiments [1]. We have grown single-crystals of $YFe_2O_{4-\delta}$ in a CO/CO_2-atmosphere to tune δ. Optimized crystals, unlike previously available crystals, exhibit a magnetic behaviour identical to highly stoichiometric powder samples, i.e. two hysteretic phase transitions at 228 K and 180 K upon cooling [2]. Four different charge ordered phases were observed in a preliminary XRD experiment down to LN_2-temperature. At P09 we observed an additional phase at 10 K with ($\frac{1}{3}$, $\frac{1}{3}$, half-integer)-reflections (Figure 1 (left)), which might be equal to the low temperature phase of $LuFe_2O_4$. The right part of figure 1 shows a scan along reciprocal l-line trough these peaks. The peak at $l = 15.5$ in the left part of figure 1 is much

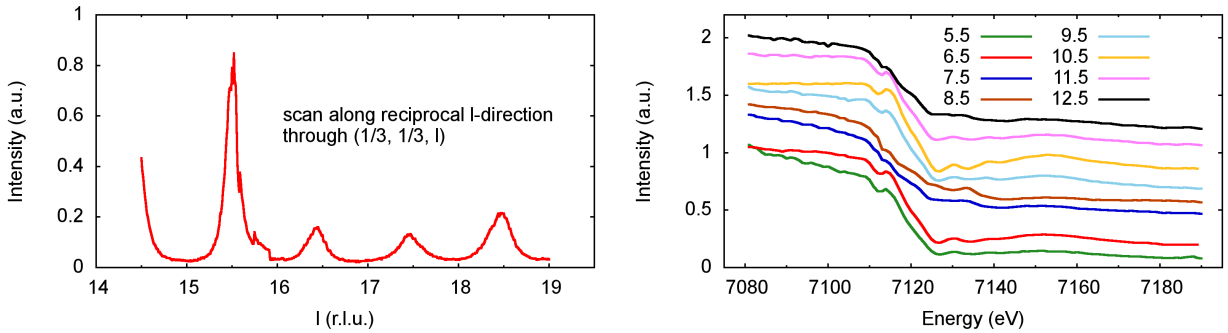

Figure 1: **(left)** reciprocal l-scan along ($\frac{1}{3}$, $\frac{1}{3}$, l) at 10 K — **(right)** Energy spectra around the Fe K-edge on ($\frac{1}{3}$, $\frac{1}{3}$, $X.5$) for varying X at 10 K

stronger than the other peaks, through a possible misalignment of the UB-matrix, and aligning on this peak before the measurement. The right part of figure 1 shows the energy spectra over the iron K-edge for different half integer l-values. Although the shown curves are the mean of four independent energy scans each, the statistical fluctuations are most likely the reason for the variations, most present in the difference between $l = 5.5$ and $l = 8.5$. The characteristic up-down variation of the post edge features observed by Mulders et al. [4] in $LuFe_2O_4$ is, also after absorption correction, not observable in YFe_2O_4. This indicates that the low-temperature phase differs from that of $LuFe_2O_4$, although it has the same ($\frac{1}{3}$, $\frac{1}{3}$, half-integer)-reflections. The left part of figure 2 compares the resonant spectra in $\sigma \rightarrow \sigma$ to that in $\sigma \rightarrow \pi$. The feature at 7116.5 eV, being relatively larger $\sigma \rightarrow \pi$, indicates some anisotropy. But the full polarization analysis (Fig. 2 left) is similar to that calculated for an ideal Thompson scatterer. Therefore the scattering contribution is dominated by isotropic charge scattering. This could be explained by the weakness of the feature present in $\sigma \rightarrow \pi$ in comparison to the intensity in $\sigma \rightarrow \sigma$. Therefore even at the resonance energy the polarization analysis mainly probes the structure. Additional to the peaks at ($\frac{1}{3}$, $\frac{1}{3}$, half-integer), shown in figure 1 (left), we observed peaks at half integer h and k positions. Further studies revealed that they were induced due to beam heating. To examine these peaks we did energy scans at 120 K, where the ($\frac{1}{3}$, $\frac{1}{3}$, half-integer)-peaks are absent and one at ($\frac{1}{2}$, $\frac{1}{2}$, 10.75) is present without beam heating. The energy spectra over the iron K-edge, shown in the left part of figure 3, are normalized

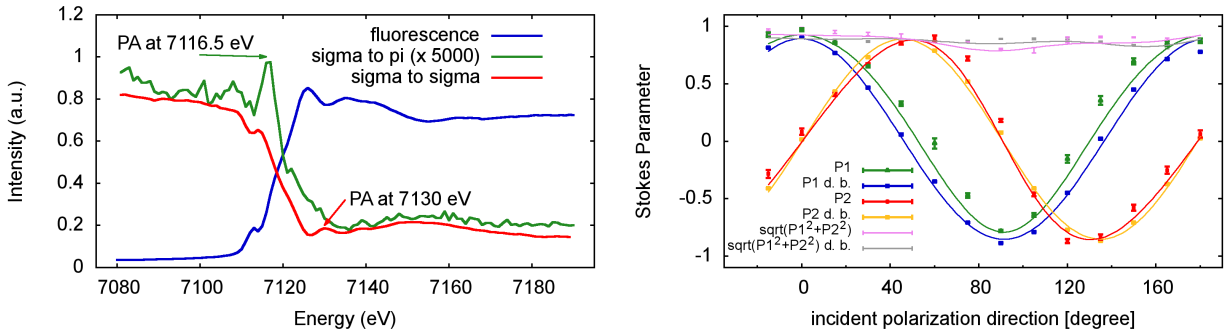

Figure 2: **(left)** Energy spectra around the Fe K-edge on $(\frac{1}{3}, \frac{1}{3}, 6.5)$ at 10 K — **(right)** Stokes Parameter P1 and P2 of the full linear polarization analysis on the $(\frac{1}{3}, \frac{1}{3}, 6.5)$-reflection, compared to the direct beam, both measured at 10 K and 7116.5 eV. The solid lines are sinusoidal fits for the direct beam and calculations for P1 and P2 for ideal Thomson scattering [4].

but not corrected for absorption, which is proportional to the shown fluorescence. At both pre edge features in the $\sigma \rightarrow \pi$ curve full polarization analysis was performed, for the feature at 7125.5 eV the resulting Stokes parameter are given in the right part of figure 3. The aberration in the polar-

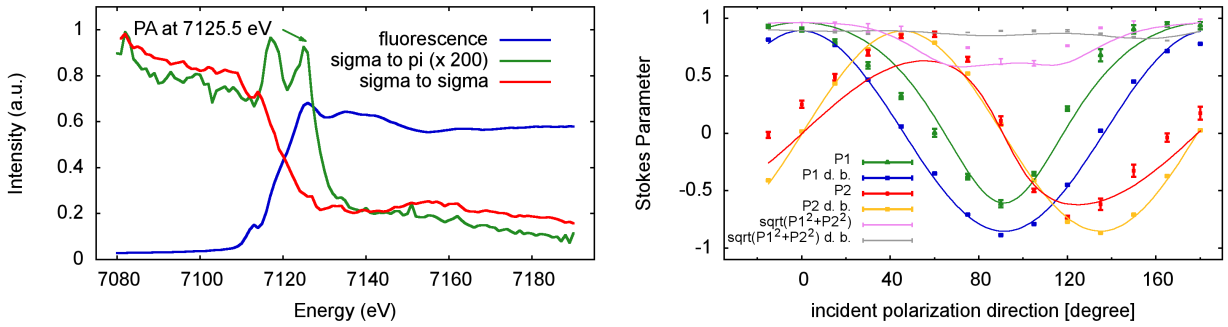

Figure 3: **(left)** Energy spectra around the Fe K-edge on $(\frac{1}{2}, \frac{1}{2}, 10.75)$ at 120 K — **(right)** Stokes Parameter P1 and P2 of the full linear polarization analysis on the $(\frac{1}{2}, \frac{1}{2}, 10.75)$-reflection, measured at 120 K and 7125.5 eV, compared to the direct beam (same data as in figure 2). The solid lines are sinusoidal fits for the direct beam and calculations for P1 and P2 for ideal Thomson scattering [4].

ization analysis from the ideal Thomson scatterer (solid red and green curve) is much stronger for the $(\frac{1}{2}, \frac{1}{2}\ 10.75)$-reflection than for the $(\frac{1}{3}, \frac{1}{3}, 6.5)$-reflection. Although the variation to the direct beam is mostly caused by the $2\theta \approx 55°$ which is closer to $90°$ than the $2\theta \approx 34°$ of the $(\frac{1}{3}, \frac{1}{3}, 6.5)$-reflection, the polarization analysis gives indication for an anisotropic scattering contribution. As the resonant features are weak, the polarization analysis mainly tests non-resonant diffraction, thus structural anisotropy. In contrast to $LuFe_2O_4$ with no indications for anisotropy connected to orbital order, there are indications for weak anisotropy in the resonant x-ray diffraction of YFe_2O_4.

References

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[4] using equation 35 and 36 from C. Detlefs et al., arXiv:1106.4446v1 [cond-mat.str-el]