EMCD with an electron vortex filter: Limitations and possibilities

T. Schachinger, S. Löffler, A. Steiger-Thirsfeld, M. Stöger-Pollach, S. Schneider, D. Pohl, B. Rellinghaus, P. Schattschneider

Abstract

We discuss the feasibility of detecting spin polarized electronic transitions with a vortex filter. This approach does not rely on the principal condition of the standard energy loss magnetic chiral dichroism (EMCD) technique, the precise alignment of the crystal, and thus paves the way for the application of EMCD to new classes of materials and problems. The dichroic signal strength in the L_{2,3}-edge of ferromagnetic cobalt is estimated on theoretical grounds. It is shown that magnetic dichroism can, in principle, be detected. However, as an experimental test shows, count rates are currently too low under standard conditions.

1. Introduction

The discovery in 2006 that magnetic chiral dichroism can be observed in the transmission electron microscope (TEM) [1] provided an unexpected alternative to X-ray circular dichroism (XMCD) in the synchrotron. Energy-loss magnetic chiral dichroism (EMCD) has seen tremendous progress since then [2–4], achieving nanometre resolution [5], and even sub-lattice resolution [6, 7]. The discovery of electron vortex beams (EVBs) [8, 9] has spurred efforts to use them for EMCD because of their intrinsic chirality. In spite of much progress in the production and application of vortex beams [6, 10–15], it soon became clear that atom-sized vortices incident on the specimen are needed for EMCD experiments [16–18]. Attempts to produce such beams and to use them for EMCD measurements did not show an effect so far [19]. Nevertheless, faint atomic resolution EMCD signals have been shown without the need for atom-sized EVBs using intelligent shaping of the incident wavefront with a C_s corrector instead [20, 21].

The fact that orbital angular momentum (OAM) can be transferred to the probing electron when it excites electronic transitions to spin polarized final states in the sample manifests itself in a vortical structure of the inelastically scattered probe electron. The latter could be detected by a holographic mask after the specimen. Using a fork mask as chiral filter is already established in optics. [22–24]. This ansatz opens up the possibility to measure magnetic properties of amorphous materials (or multiphase materials including both crystalline and amorphous magnetic phases [25]), since the specimen no longer needs to act as a crystal beam splitter itself. Also crystalline specimens could benefit from using the vortex filter setup and its inherent breaking of the Bragg limitation, when, for example, substrate reflections overlap with the two EMCD measurement positions which would diminish the EMCD signal strength.

2. Principle

Dealing with transition metals, dichroism measurements typically involve 2p-core to d-valence excitations at the L_{2,3} ionization edges. The L_{2,3}-edges are used, due to their strong spin-orbit interaction in the initial state. Besides their dichroic signal is an order of magnitude higher compared to using K-edges, which were originally used in X-ray magnetic circular dichroism measurements to show the dichroic effect [9, 26, 27]. The most dominant contribution to the ionisation edges are electric dipole-allowed transitions. Higher multipole transitions show low transition amplitudes contributing less than 10 % at scattering angles of < 20 mrad relevant in electron energy loss spectrometry (EELS) [28–30].

In case of an L-edge dipole-allowed transition which changes the magnetic quantum number of an atom by \( \mu \), an incident plane wave electron transforms into an outgoing wave [31]

\[
\psi_\mu(r) = e^{-\text{i} \mu \varphi} f_\mu(r)
\]

where \( \varphi \) is the azimuthal angle, and

\[
f_\mu(r) = \frac{i \mu}{2\pi} (\mu_\mu)^{1/2} \int_0^\infty dq J_{\mu}(qr) (j_1(Q)) \frac{EELS_j}{Q^3} dq.
\]

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1 Sometimes also called electron magnetic chiral dichroism. Properly speaking, this is incorrect because it is not the electron but the energy loss signal that shows dichroism.
with \((j_{\mu}(Q))_{\text{ELS}}\), the matrix element of the spherical Bessel function between the initial and final radial atomic wave functions, and \(Q = \sqrt{q^2 + q_E^2}\). Here, \(q\) is the transverse scattering vector that relates to the experimental scattering angle \(\theta\) as \(q = k_0\theta\), and \(hq_E = h\kappa_0\theta_E\) is the scalar difference of linear momenta of the probe electron before and after the inelastic interaction, also known as the characteristic momentum transfer in EELS [32]. The characteristic scattering angle \(\theta_E\) is given by \(\theta_E \sim \Delta E/2E_0\), with \(\Delta E\) being the threshold energy of the dipole-allowed L-edge being protected. The radial intensity profiles are separated by 2\(\mu\) each other.\(\sum_{\mu}|\psi_{\mu}|^2\) is the transverse wave function, and \(\psi_{\mu}\) is given by the mask aperture limit. Due to the limited extent of an electron wave [37, 38], to date no successful filtering fork mask in the DP is not straightforward, because, due to the limited space in the pole piece gap, strip apertures are used which cannot be loaded with conventional \(\varpi 3\) mm frame apertures. Even though there are proposals to use spiral-phase-plates in the DP, e.g. to determine chiral crystal symmetries and the local OAM content of an electron wave [37, 38], to date no successful

\[
\psi_{\mu}(q) = \frac{i^\mu}{2\pi} e^{-i\mu\varphi} \int_0^\infty f_\mu(qr) J_\mu(qr) r dr. \tag{3}
\]

The dichroic signal in the diffraction plane (DP) can readily be calculated via Fourier transforming Eq. 1. According to a theorem for the Fourier-Bessel transform of a function of azimuthal variation \(e^{-i\mu\varphi}\) [33], one has

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\tilde{\psi}_\mu(q) = \frac{i^\mu}{2\pi} e^{-i\mu\varphi} \int_0^\infty f_\mu(qr) J_\mu(qr) r dr. \tag{3}
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Figure 2: Principle of the vortex filter (ideal geometry): The red dot represents the scattering centre, i.e., the atom in the object plane, with its three scattering channels, $\mu = \pm 1, 0$. The resulting vortices are then incident on a fork mask in the far-field, adding topological charges $m \in \mathbb{Z}$, thereby creating a line of vortices of topological charges $\mu + m$ in the image plane. Subsequently, the EMCD signal can be derived from the difference in the vortex orders $m = \pm 1$, according to Eq. 5.

![Diagram of vortex filter](image)

| $m$  | $-1$ | 0  | $+1$ | $-1$ | 0  | $+1$ | $-1$ | 0  | $+1$ |
|------|------|----|------|------|----|------|------|----|------|
| $\mu$| -1   | 0  | +1   | -1   | 0  | +1   | -1   | 0  | +1   |
| $m + \mu$| -2 | -1 | 0   | -1   | 0  | +1   | 0    | +1 | +2   |
| $C_{m\mu}$ | 0.278 | 0.222 | 0.167 | 0.278 | 0.222 | 0.167 | 0.278 | 0.222 | 0.167 |

Table 1: Coefficients $C_{m\mu}$ for the $L_3$-edge ($j = 3/2$) taken from [6]. The weighting factors for the transitions when the final states are completely up-spin polarized show an asymmetry for $m + \mu = 0$, i.e., in the centre of the $m = \pm 1$ vortices.

implementation of a vortex mask in the DP of a TEM has been shown. On the other hand, the final image in the selected area aperture (SAA) plane (intermediate image plane) of the atom sized vortices would be too small to be resolvable at all. Both facts make this direct approach problematic. The second obstacle can be removed by defocussing the lens, observing broader vortices. The first one can be overcome by positioning the fork mask in a relatively easily accessible position, which is the SAA holder.

These ideas lead to a scattering geometry that can be easily set up in conventional TEMs, see Fig. 4a and Fig. 5c. Here, the fork mask is positioned in the SAA holder, creating a demagnified virtual image in the eucentric plane with small lattice constant. Additionally, the specimen is lifted in height by $dz$ and the C2 condenser lens is adjusted such that a focused probe is incident on the specimen.

Note that focusing the beam onto the specimen guarantees that the probability density current is mostly aligned parallel to the optical axis all over the illuminated area such that the scattering ”light cones” all point in the same direction towards the vortex filter mask. This is due to the fact that the Rayleigh range of the incident beam is of the order of 600 nm (convergence semi-angle 3.8 mrad) which is much larger than the sample thickness, in our case $\sim 70$ nm, and thus the incident wavefronts are almost flat everywhere inside the specimen, see Fig. 4b. How much they actually deviate from that assumption is estimated in Fig. 4c. It can be seen that the tilt angle at a radial position of 0.7 nm in the entrance plane amounts to $\sim 70$ µrad which can be considered negligible compared to the characteristic scattering angle $\theta_E \sim 1.95$ mrad of Co. Furthermore only a small fraction of atoms in the illuminated specimen area actually sees such ”high” tilt angles of the incident wavefront, most of the illuminated atoms do see a much less tilted wavefront.

Note that the Rayleigh range was determined using the diffraction limited spot size of the C2 aperture, the final probe diameter is much larger due to incoherent source broadening.

Neglecting the crystal field and channelling effects.
Lifting the specimen ensures that the (virtual) fork mask is now in the far field of the excited atom and creates a series of images of the ionization process as depicted in Fig. 4a. Practically, this setup is comparable to a standard STEM geometry but with the specimen lifted far off the eucentric plane. For better understanding the scattering setup, Fig. 5 compares the standard TEM setup in diffraction, Fig. 5a, and the standard STEM setup, Fig. 5b, to the setup proposed here, Fig. 5c. Note that there are slight changes in the local position of elastic- to inelastically scattered electrons in Fig. 5b. It can be seen that when the vortex filter mask is placed in the SAA holder diffracted beams (small dashing) emerge from the vortex mask in Fig. 5a,c but not in Fig. 5b because there the image of the inelastically scattered electrons in the SAA plane is much smaller than the grating periodicity such that the vortex mask is not illuminated. Lifting the specimen by $dz$ in Fig. 5c ensures that the vortex filter mask is properly illuminated. Moreover, due to electron optical reasons this lifting is essentially reducing the size of the first image of the focused probe on the (lifted) specimen, comparable to the reduction of the effective source size in the condenser system of a TEM by adjusting the C1 lens excitation.

The dichroic signal is strong in the centre of the vortices but difficult to observe because of their extension of only about 1 nm, as seen in Fig. 3. Therefore, the observation plane is set at a defocus $df$ (here 4 μm) from the specimen (preferably with the diffraction lens setting) to enhance the visibility of the dichroic signal. This can be understood from Fig. 4a and Fig. 5c. Virtual images (green) of the object intensities are observed at a defocus $df$, making the distribution broader, such that the maximum of the vortices’ radial intensity distribution, where no dichroic signal is expected to occur, moves towards higher radii. The orders $m \in \{-1,0,1\}$, with an angular separation of $2\theta_B$ are shown in Fig. 4a.

The observed vortices are calculated with Eq. 4, but now including the defocus $df$ and the spherical aberration $C_s$:

$$\psi_{m \mu}(r) = \frac{i^{m+\mu}}{2\pi} e^{-i(m+\mu)\phi_v} \int_0^{2\pi} \bar{\psi}_0(q) J_{m+\mu}(qr) e^{i(dfq^2/2ka+C_sq^4/4ka^3)} dq.$$  

When a homogeneous specimen is illuminated all atoms within the beam will contribute incoherently with their respective signals. This incoherent broadening effect according to the finite illuminated area of the specimen is taken into account by a convolution with a Gaussian as described in [39]. Thus, the final simulated radial intensity distribution is given by

$$I^\sigma_m(r) = e^{-(1/2)(c/\sigma)^2} \int_0^\infty \psi_{m \mu}(r') e^{-(1/2)(r'/\sigma)^2} I_0 \left( \frac{rr'}{\sigma^2} \right) r'dr',$$

where $I_0$ represents the modified Bessel function of first kind of order zero and $\sigma$ the amount of incoherent broadening. The resulting illuminated area (FWHM) at the specimen is $\sim 2.4\sigma$. This incoherent broadening effect
will reduce the EMCD signal as shown in Fig. 6. But still, the defocused case is preferable because the tiny differences in width for the focused case are hardly observable, see Fig. 6. In Eq. 8, there are two free parameters, defocus and broadening width, which are used to obtain best fits to the experimental data shown in the next section.

3. Experimental

The fundamental method and scattering geometry elaborated above have been realized in a proof-of-principle experiment on a FEI TECNAI F20 TEM equipped with a GATAN GIF Tridiem spectrometer (GIF) and a high-brightness XFEG. The acceleration voltage was set to 200 kV, whereas the condenser system was set up in a way to achieve a high beam current at a sufficiently small spot size, i.e. providing a beam current of $\sim 500$ pA incident on the sample in a $\sim 1.9$ nm probe (FWHM) with a convergence semi-angle of 3.8 mrad.

Fig. 7 shows the vortex filtering holographic fork mask that was placed in the SAA holder. It was produced by FIB milling into a 300 nm Pt layer deposited on a 200 nm Si$_3$N$_4$ membrane. With a diameter of 10 µm and a grating periodicity of 500 nm (back-projected: 9.4 nm), it exhibits a Bragg angle of $\theta_B = 5 \mu$rad separating the central spot from the first vortex orders in the eucentric plane by $\sim 20$ nm$^4$. As a result the vortex orders $\pm 1$ are still well separated from the central peak for defocus values of $df = 4$ µm and higher, see Fig. 8.

For the preparation of the Co sample, a 70 nm thin Co layer is deposited onto a NaCl crystal. The thin Co foil is then extracted by dissolving the NaCl in water. Afterwards, the Co foil is netted with a commercially available Cu grid, resulting in a free standing nano-crystalline Co film of 70 nm thickness, with randomly oriented 20 nm crystallites. In the following section, the experimental setup of the vortex filter experiment on the Co film is described in detail.

In imaging mode, the objective lens is set to the eucentric focus value with the sample in the eucentric height. From that position, the Co sample is then lifted by $dz = 75$ µm. The beam is focused onto the lifted specimen using C2 excitation and observing the ronchigram in the eucentric focus value with the sample in the eucentric height.

The separation distance was calculated using $2\theta_{Bragg} dz$, with a camera-length of $dz = 75$ µm and the back-projected grating periodicity $g = 9.4$ nm.

\[ \text{Figure 5: Ray diagrams (not to scale) of (a) a standard TEM diffraction setup, (b) a standard STEM setup and (c) the EMCD vortex filter setup. Full and dashed black lines represent rays of elastically scattered electrons whereas red lines depict inelastically scattered ones.} \]
Figure 6: Incoherent broadening: Radial intensity profiles of $m = \pm 1$ filtered images as in Fig. 3 with a defocus of 0 µm (dashed lines) and 4 µm (solid lines), respectively, for an illuminated specimen area of $\sim 1.9$ nm ($\sigma = 0.8$) and a $C_s$ of 1.2 mm, according to Eq. 8. Due to the incoherent broadening effect the focused vortices no longer show the characteristic dip in the center and the defocused radial profiles are hardly distinguishable. The resulting EMCD signal (dot-dashed curves) is dramatically lowered and amounts to $\sim 3\%$ in the central region of the defocused vortices and $\sim 5\%$ approximately at the beam waist for the focused ones. (Note the change of the right scale.)

Figure 7: Holographic fork mask prepared by FIB milling used as a vortex filter in the SAA holder. The mask has a diameter of 10 µm with a grating periodicity of 500 nm, giving a diffraction of the first vortex orders at $2\theta_B = 5$ µrad.

Subsequently, the frames are stacked and aligned using Image J [40]. To extract the radial intensity profiles given in Fig. 9, Digital Micrograph scripts are used to determine the exact vortex orders’ centres, cropping them and doing the rotational (azimuthal) average.

Fig. 8 shows the experimental energy filtered image of the vortex aperture from electrons which have transferred 780 eV to the Co sample. Due to the extremely low count rates, Fig. 8 is acquired taking four frames with an acquisition time of 100 s per frame with four fold binning.

Figure 8: Experimental image showing the vortex orders $m = +1$ (left), $m = -1$ (right) and $m = 0$ (middle), produced by the SAA vortex filter at the Co $L_3$-edge defocused by 4 µm.

Figure 9: Rotational averages of the outermost vortex orders in Fig. 9 (red and green full dots) and their best fits using Eq. 8 including incoherent broadening with $\sigma = 0.8$ nm and a defocus of $df = 4$ µm (full lines). The theoretically predicted EMCD signal (green, dot-dashed line) is compared to the experimental one (magenta open circles and a dashed polynomial fit curve). The error in the EMCD signal, given by the magenta shaded area, representing the RMS value including Gaussian error propagation, indicates that the faint EMCD signal cannot be discerned under present experimental conditions.

Fig. 9 shows that the simulation with the chosen parameters is in very good agreement with the experimental data. Curiously, the experimental radial profiles show a strong difference in the central region ($\sim 15\%$) which is similar to classical EMCD measurements [41] and previous vortex filter EMCD experiments [9]. However, the simulation predicts a much smaller EMCD signal ($\sim 3\%$). This discrepancy is possibly due to (i) skew optic axes which gives rise to slight differences in apparent defocus $df$ for the positive and negative vortex orders, (ii) artefacts from the mask production [42] and (iii) OAM impurities stemming from the SAA vortex mask [43].

5In fact we are working in the energy filtered selected area diffraction (EFSAD) mode [29] because the microscope is set to diffraction mode and the SAA (vortex filter mask) is used.

6For the sake of simplicity, elastic scattering inside the nanocrystalline structure of the specimen was not taken into account in the single atom approach given here.
Fig. 9 also clearly shows that the EMCD signal is much smaller than the relative root-mean-square (RMS) value of the experimental EMCD signal (∼±40%, magenta shaded region) and thus cannot be detected reliably under present experimental conditions. Since the profiles are azimuthally averaged, the absolute signal-to-noise ratio (SNR) per radial position is best for the largest radius where an average over 512 pixels was taken, and lowest for the second point (9 pixel average). This was taken into account numerically for the results shown in Fig. 9. The magenta shaded region in Fig. 9 is calculated using Gaussian error propagation, thus showing the relative RMS value in the azimuthal direction of the EMCD signal defined by Eq. 6.

Furthermore, the error in Fig. 9 (magenta shaded region) only represents the statistical error; systematic errors such as beam-drift, beam damage, artefacts due to non-isotropic vortex rings etc. have not been included. In practice, the sample’s spin polarization may be less than 100%, and the vorticity may change during the propagation of the outgoing vortex beams through the specimen [12], thereby decreasing the expected EMCD signal as well.

In view of these results and considerations, further investigations and improvement of the experimental conditions are necessary to proof the applicability and reliability of the EMCD vortex filter method.

4. Conclusions

In this work we investigated the feasibility of detecting an EMCD signal when incorporating a fork mask as a vortex filter in the SAA plane in a standard two-condenser lens field emission TEM. By lifting the sample far above the eucentric position, a vortex filter mask in the SAA plane can be properly illuminated. Thus, it produces well separated vortex orders which should, in principle, carry the EMCD information in the asymmetry of their respective central intensities. This method could become a promising method for studying magnetic properties of amorphous or nanocrystalline materials, which is impossible in the classical EMCD setup. So far the experimental tests show that the SNR is still too low and that for a successful experimental realization substantial progress in the experimental conditions is compulsory. For example, to improve the SNR future experiments should incorporate larger SAA fork masks, e.g. at least 30 to 50 µm in diameter. As the collected signal scales with the mask area, the acquisition times could then be lowered by an order of magnitude. Also, incorporating a HM in the contrast aperture holder located in the DP would simplify the experimental setup as well as increase the collection efficiency. Finally, using state-of-the-art aberration corrected microscopes it is possible to increase the lateral coherence of the probe beam while at the same time keeping the beam current high. This would enhance the EMCD signal strength by an order of magnitude. Thus, in the light of above considerations and the proof-of-principle experiment, detection of EMCD signals using HM as a vorticity filter seems to be feasible but needs thorough control of experimental parameters like spot size, vortex masks fidelity, sample- and system stability.

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