Experimental Progress to obtain atomic resolution EMCD measurements in the TEM

Hasan Ali[1, 2], Jan Rusz[3], Tobias Warnatz[3], Devendra Negi[4], Ling Xie[2], Björgvin Hjörvarrson[3] and Klaus Leifer[2]

[1]Department of Materials and Environmental Chemistry, Stockholm University, 10691, Stockholm, Sweden
[2]Department of Materials Science and Engineering, Uppsala University, Box 534, 75121, Uppsala, Sweden
[3]Department of Physics and Astronomy, Uppsala University, Box 516, 751 20 Uppsala, Sweden
[4]Stuttgart Center for Electron Microscopy, Max Planck Institute for Solid State Research, Heisenbergstrasse 1, 70569 Stuttgart, Germany.
Corresponding author: hasan.ali@mmk.su.se

The advancements in nanotechnology call for characterization techniques capable to determine the atomic scale magnetic properties of the materials. Typically used techniques like STEM-DPC, magnetic exchange force microscopy, electron holography and x-ray magnetic circular dichroism (XMCD) are either restricted to surface analysis or they are limited by spatial resolution. Electron magnetic circular dichroism (EMCD), an electron energy loss spectroscopy (EELS) based technique, has the potential to measure the magnetic properties of the materials with atomic resolution while preserving the depth information of a few tens of nm. Being a transmission electron microscope (TEM) based technique, another advantage is that the structure of the materials can be characterized and can be locally correlated to the magnetic properties.

EMCD was proposed in 2003 and first experimental demonstration was shown in 2006 [1]. From the time of its discovery, the EMCD technique has been continuously developed to improve the S/N ratio of the EMCD signals and the spatial resolution of the analysis. Although the technique has been recently used to detect the magnetic signals from single atomic planes [2], it is still not straight forward to obtain quantitative EMCD signals with sub-nm spatial resolution due to several challenges.

One of the challenge is the requirement to acquire at least two EELS spectra from two different angular positions in reciprocal space, the difference of which gives the EMCD signal. This requirement leads to scan the region of interest multiple times in the STEM mode, making it difficult to keep the experimental conditions same for all the scans due to sample drift, beam damage, contamination and other instrumental instabilities. We addressed this challenge by building a double hole aperture in the reciprocal space which can be used to acquire the two EELS spectra simultaneously in a single STEM scan [3]. This not only improves the accuracy of the EMCD signals but also enhances the S/N ratio of the signals compared to the previously adopted techniques.

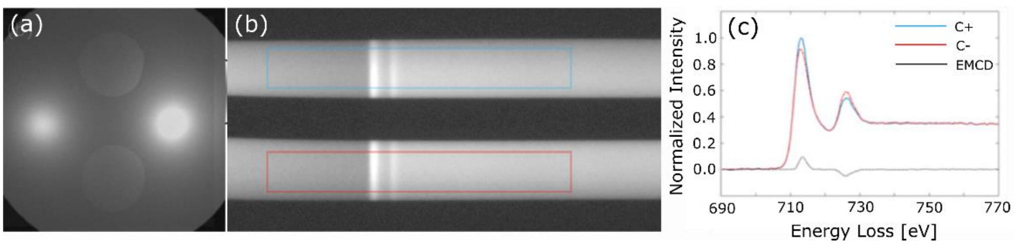

Figure 1. (a) Electron diffraction pattern with the sample tilted to 2-beam orientation, overlaid by the CCD

image of double hole aperture (b) Momentum-resolved 2D EELS spectra (c) Background subtracted and normalized EELS spectra and their difference (EMCD) signal

The EMCD signals are highly sensitive to the orientation of the sample and it is crucial to know how a change in orientation will affect the measured EMCD signal. This kind of study is challenging as it needs the two EELS spectra and the diffraction patterns acquired from the same region of the sample with a perfect spatial registration which is non-trivial with the conventional acquisition techniques. We developed a method to map the EMCD signal as well as the orientation of the sample simultaneously by the use of a quadruple aperture in the diffraction plane. With the experimental results and simulations, we built an accurate relationship between the crystal orientation and the EMCD signal [4].

The conventional EMCD measurements are carried out with the sample tilted into two-beam or three-beam orientation. In this orientation, the atomic planes are parallel to the electron beam while hiding the atomic (column) resolution. To obtain the atomic column resolution, the EMCD measurements must be carried out in a zone axis orientation where the distribution of the EMCD signal is much more complex than the simple case of 2-beam orientation. Negi et al. proposed the use of ventilator apertures to acquire the EMCD signals in zone axis with atomic resolution electron probes. We experimentally utilized such apertures to detect the EMCD signals using electron probes with semi-convergence angles up to 10 mrad. In a probe-corrected microscope, a 10 mrad semi-convergence angle would correspond to a diffraction limited probe size 1.2 Å which is sufficient to resolve the atomic columns of many magnetic materials [5].

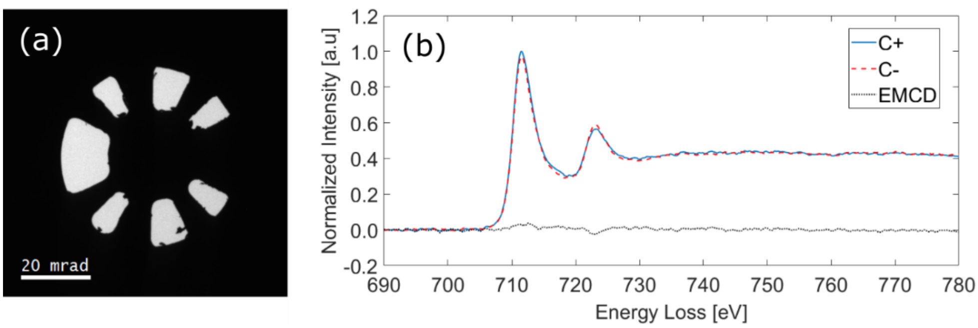

Figure 2. (a) The mirrored ventilator aperture (VA) which can simultaneously acquire the positive and negative EMCD components in zone axis (b) the + and – EELS spectra and their difference (EMCD) signal acquired using the VA from an Fe sample in [001] zone axis with 5 mrad convergent electron probe.

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