EELS signal enhancement by means of beam precession in the TEM

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ABSTRACT

EELS is nowadays a most relevant characterization tool as it provides chemical and electronic information with an extraordinary spatial resolution. When a crystal is viewed in zone axis in the TEM, there is channelling of the electrons along the atom columns, which strongly reduce the EELS signal, so that it is generally advised to work slightly off the zone axis to collect EELS data, which may not always be possible or advantageous. In the present work, we demonstrate the use of precession to compensate for the reduction of EELS signal when in the zone axis.

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1. Introduction

Electron energy-loss spectroscopy (EELS) performed in the transmission electron microscope (TEM) provides chemical and electronic information about the considered solid state sample, through the determination of the energy lost by the incident electrons, with an extraordinary spatial resolution, that can be as high as one atomic column in aberration-corrected instruments [1–4].

If one solves the Schrödinger equation for an electron in a crystalline potential, the resulting wavefunctions are Bloch waves. When a number of Bragg beams are excited, the same number of Bloch waves propagate in the crystal. In the most simple case, in the two beam condition, two Bloch waves are present: one that is peaked at the specimen atoms (type 1) and another that is peaked at half way between the atoms (type 2), both with the same intensity. When tilting the specimen out of the exact Bragg condition, if the beam-crystal plane angle is reduced, type 1 wave is enhanced, and, if the aforementioned angle is increased, then, the type 2 wave is enhanced.

The orientation dependence of Bloch waves amplitude is a generalised phenomenon and may affect the relative intensity of the EELS edges, depending on the position of the different atoms in the unit cell respective to the exact Bragg plane, which is sometimes used to determine the polarity of a thin film or nanostructure [5].

When in exact zone axis, the Bloch wave description may not be the most suitable one. It is well established that, when a crystal is viewed parallel to the atom columns, the obtained high resolution images show a one-to-one correspondence with the actual atom positions if the balance between the resolution of the instrument and the atomic distance is favourable enough; thus, in this case, the exiting wave function mainly depends on the projected structure, and the physical explanation for this to occur has been proposed [6–8] to be the channelling of the electrons along the atom columns parallel to the beam direction: through the positive electrostatic potential of the atoms, a column acts as a channel for the electron, within which it can scatter dynamically without leaving the column. This elastic and highly directed incident electron–atom interaction is bound to strongly reduce the EELS signal [9,10].

The obvious answer to this is to remain off-axis to perform EELS experiments. Now, this is not always possible. Consider, for instance, the EELS analysis of a region delimited by one or several interfaces, where getting away from the zone axis means collecting signal from outside this region.

More interestingly, when working in aberration-corrected microscopes, one does want to keep the zone axis condition in order to allow for the chemical analysis of one given atomic column. In this case, electron channelling may give rise to further experimental problems. Artefacts may be encountered, especially if the sample is thick enough. If an inelastic scattering event is suffered by an electron channelling down an atomic column, there is a non-null probability of this electron swapping to the next atomic column and, thus, giving some chemical information about the wrong atomic column.

Precession can be considered to overcome the problems associated with channelling: if one combines EELS with...
precession, it is possible to recover an effective two-beam condition while remaining in the zone axis [11–13].

2. Material and methods

To prove this precession-assisted signal enhancement, several EEL spectra were obtained in a Jeol J2010F coupled with a GIF spectrometer and a SpinningStar precession system.

The considered EELS edges for testing the hypothesis were the Si L\textsubscript{2,3} edge in Si and the O K and Ti L\textsubscript{2,3} edges in SrTiO\textsubscript{3} (STO).

The used probe size was 0.5 nm, convergence and collection semiangles were 9 mrad and 10 mrad, respectively, and the acquisition time was of 3 s for the Si L\textsubscript{2,3} edge in Si and 6 s for the O K and Ti L\textsubscript{2,3} edges in STO.

Sample thicknesses were determined from low-loss EELS to be about 30 nm for the Si sample, and about 35 nm for the STO sample.

3. Results and discussion

Let us consider the Si L\textsubscript{2,3} edge in a Si crystal in the [110] zone axis first. Precession angles between 0° and 1.92° are investigated.

For each precession angle, an EEL spectrum is obtained—an example is given in Fig. 1. Then, for every spectrum, background is extracted and signal is integrated over a 100 eV energy window to obtain an intensity I. The signal enhancement (SE) for a given precession angle \( \alpha \) will then be given by \( SE = \frac{I(\alpha)}{I(0)} \), where \( I(0) \) designates the intensity obtained without precession and SE is expressed in percentage.

The SE(\( \alpha \)) plot in Fig. 2 clearly shows that precession compensates for the reduction of EELS signal when in the zone axis. It is also apparent from Fig. 2 that signal enhancement saturates at angles as low as \( \alpha \sim 0.5° \). Furthermore, for precession angles over \( \alpha \sim 0.5° \) the off-zone-axis EELS signal is recovered.

Let us now consider the O K and Ti L\textsubscript{2,3} edges in a SrTiO\textsubscript{3} (STO) crystal in the [001] zone axis. Again, precession angles between 0° and 1.92° are investigated. In this case, for every spectrum, background is extracted and signal is integrated over a 50 eV energy window.

SE(\( \alpha \)) plots for these edges are given in Fig. 3. Although the data are considerably noisier when compared to the Si example, the same tendencies are observed, namely: signal is effectively enhanced by precession, and this enhancement saturates for angles as low as \( \alpha \sim 0.5° \).

The given experimental data clearly prove that precession compensates for the reduction of EELS signal when in the zone axis.

4. Conclusions

In the present work it has been proposed that precession of the beam is to compensate the reduction of EELS signal when in zone axis conditions. This compensation has been experimentally observed in the case of the Si L\textsubscript{2,3} edge in a Si crystal in the [110] zone axis and in the case of the O K and Ti L\textsubscript{2,3} edges in a SrTiO\textsubscript{3} (STO) crystal in the [001] zone axis.

These findings can be of great interest in interferometric EELS techniques such as circular dichroism [14,15] where the combination of both techniques can allow obtaining more significant signal to noise ratios. They may also help in the interpretation of EELS data, as channelling effects make it very difficult to accurately determine EELS signal from theoretical calculations.

As electron beam precession has been recently reported to present several applications as an imaging technique [16], it might be also interesting to explore the use of electron beam precession to enhance EELS signal when performing the chemical analysis of one given atomic column in aberration-corrected microscopes.
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