

# Breaking barriers: Innovative methods for 4DSTEM diffraction data acquisition and processing in SEM

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Background incl. aims

Electron diffraction is a reliable tool to obtain information about the crystalline structure of nanoscale materials. With the recent emergence of more sensitive detector technology (Direct electron detectors e.g., Timepix and Medipix) the requirement for the dose applied to the sample in experiment drastically decreases.

Diffraction requires a far simpler setup as compared to conventional transmission electron microscopy as the diffraction pattern can be obtained without any lenses. This lends itself ideally to an implementation in a conventional SEM tool having several advantages such as providing more space for sample stimuli and manipulation, lower cost, and much smaller footprint. Typically, the acceleration voltage in SEM is lower compared to TEM. However, in the past years the tendency for particles' size decrease can be observed and SEM could become an attractive alternative to TEM for such materials.

Rather than taking a single diffraction pattern, the combination of a SEM tool and a fast direct electron detector allow for the efficient acquisition of 4DSTEM diffraction data, having a diffraction pattern for each probe position in a STEM scan.

There are however a few challenges:

- A lower acceleration voltage compared to TEM leads to a decrease of maximum sample thickness that can be used.
- The field of view in a SEM is much larger than in TEM. On the one hand, this is advantageous to gather statistical information on a much higher number of particles. On the other hand, a large part of this field of view is often empty resulting in long recording times with large portions of the data being empty.
- Distinguishing diffraction reflexes from the background can become challenging at the reduced acceleration voltage. This reduces the intensity of the reflections and results in poor averaged diffraction patterns which are dominated by the background signal.

To negotiate these issues new methods for data acquisition and processing should be developed. In the current work we aim to push the limit of transmission electron diffraction in SEM, mitigate beam damage and sample contamination, to make SEM a versatile tool for a high-resolution electron diffraction structure investigation.

## Methods

Experiments made on a Tescan Mira FEG SEM. Diffraction data collected via an Advacam AdvaPix. Sample manipulated by custom stage of 3 Xeryon linear piezo motors in XYZ configuration mounted on rotation stage for tomography. Data processing is done in Hyperspy, controls are made with Python, image denoising is achieved with a convolutional neural network, Edge detection is realized with OpenCV.

## Results

To reduce the acquisition time and avoid empty areas in the 4DSTEM dataset we use a fast overview scan from which we detect all particles positions and shapes with computer vision. In a second step we determine a scan pattern to visit only those areas which likely to give diffraction patterns. We call this approach ROI 4DSTEM: each detected particle is marked with a bounding box. We then use the assumption that particles or agglomerated particle sets tend to be much thinner on their outer boundaries as compared to their centers. These boundaries are therefore the ideal positions to get good quality diffraction data from if the assumption holds that the core of the particles is the same crystal structure as its perimeter area. We call this method Edge Detected 4DSTEM. By utilizing image denoising via a modern convolutional neural network and edge detection algorithms we were able to create with high precision a mask of scan positions that is later used to position the incident beam only at those areas that most likely will lead to good quality diffraction patterns.

This approach allows us to perform 4DSTEM in SEM in a much more effective manner. Indeed, for a standard 4DSTEM raster scan of 1024x1024 scan positions we would get 1,048,576 diffraction patterns, or approximately 182 GB of data to store and 18 minutes to collect all this data at 1 ms exposure time. In our ED4DSTEM method, we only collect 23,000 positions, resulting in 4 GB of data and 23 seconds of total recording time for the same 1ms exposure time. This is almost 50 times faster/more efficient as compared to conventional raster scanning resulting in a significant reduction of beam damage and contamination on top of the benefit of pre-filtered diffraction data of much higher average quality.

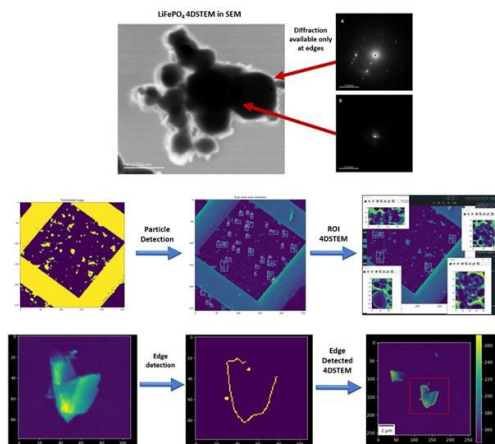
By processing each individual diffraction pattern and storing only the peak positions and their intensities we can obtain a massive data reduction that considerably simplifies the following data analysis steps.

This reduced dataset can then be used for any kind of diffraction analysis. In our case we are collecting “virtual” ring patterns as a radially integrated histogram of the scattering angle of all observed peaks, weighted by their intensity. This method has the advantage of suppressing the background in the diffraction pattern that originated from substrate/noise/amorphous content in the sample that otherwise would dramatically deteriorate the quality of a positionaveraged diffraction pattern. An added benefit is the sub-pixel position accuracy that can compensate for beam convergence and pixel size effect.

### Conclusion

In this work we present methods that allow for more effective 4DSTEM diffraction data acquisition on dispersed crystalline nanoparticles at low acceleration voltages as well as methods for more reliable data processing. Our method, compared to conventional 4DSTEM raster scan, drastically decreases the total 4DSTEM acquisition time, beam damage and contamination while providing a dramatic data reduction in the process that could significantly simplify follow the data analysis. These approaches are also amenable to TEM and could be especially useful in case of beam-sensitive objects.

### Graphic:



**Keywords:**

4DSTEM, SEM, TEM, EdgeDetection, ImageRecognition

**Reference:**

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