USING PROTOCHIPS AXON SOFTWARE FOR TRACKING ELECTRON FLUX AND CUMULATIVE DOSE

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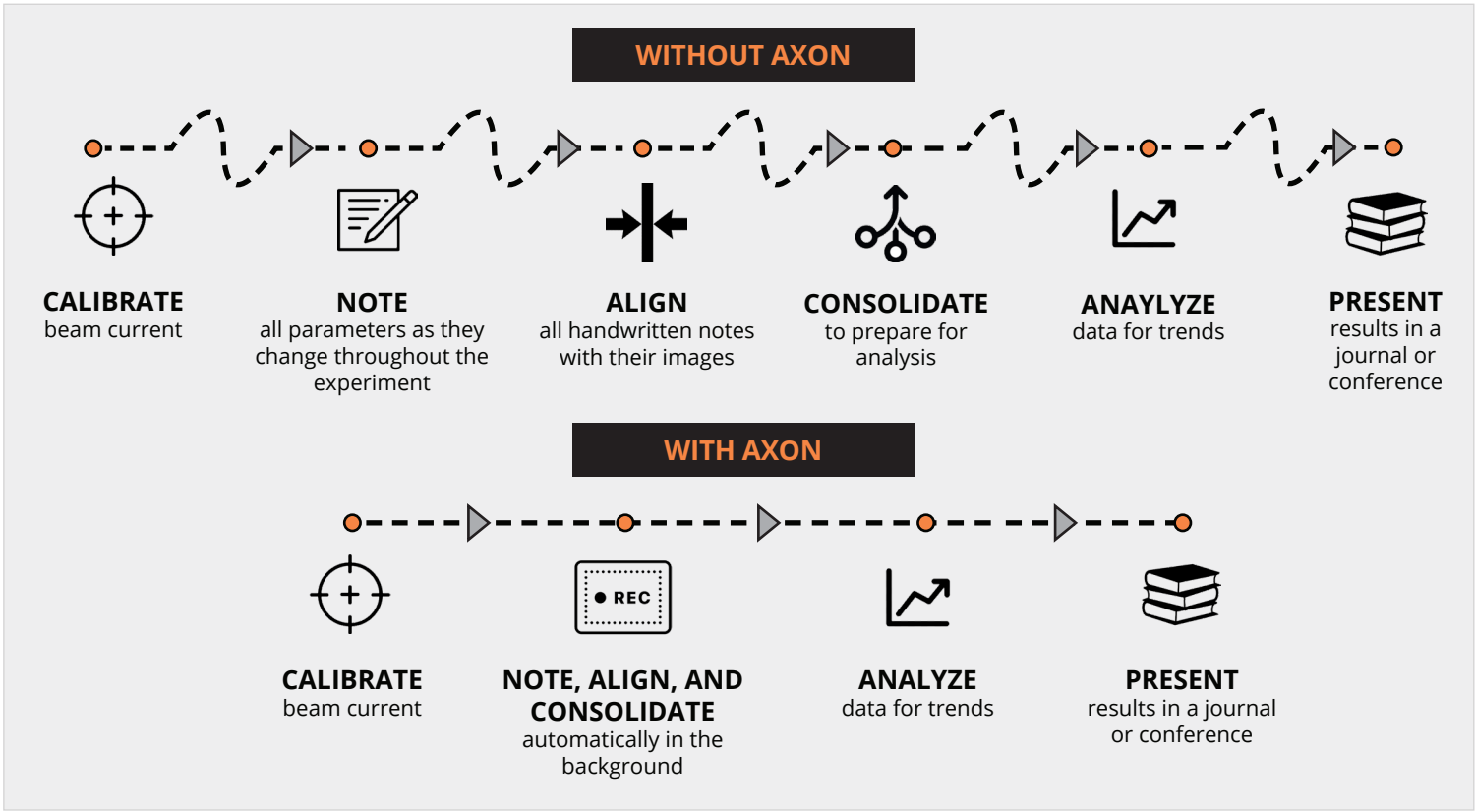
WHY TRACK ELECTRON FLUX AND CUMULATIVE DOSE?

The liquid cell TEM community has identified electron flux (electrons/Å^{2}s) and cumulative electron dose (electrons/ Å^2) used to acquire an image as critical parameters that should be reported with every *in situ* liquid TEM experiment in order to improve experiment reproducibility and enhance result validity. The interaction of the electron beam with the liquid during an *in situ* experiment can cause radiolysis and ionization processes to occur which leads to the formation reactive radiolytic products. The type and concentration of radiolytic products that form are directly related to both the incident electron flux (d) and the cumulative dose (D) to which a sample or region is exposed. These products influence the local chemical environment and must ultimately be taken into account when interpreting the results of the experiment.

HOW DOES AXON HELP YOU CALCULATE AND TRACK ELECTRON FLUX AND CUMULATIVE DOSE?

AXON takes the frustration out of this critical process by automating the recording, alignment, and consolidation of all the dynamic parameters needed for flux and cumulative dose calculations for the entirety of the experiment, straightening and shortening the path to your final results. There is no need to manually track any of them anymore. Once the experiment is over, you can then export a .CSV file with the entire set of parameters for every image captured and use the full capabilities of your favorite spreadsheet software to calculate flux and cumulative dose for every image, as well as analyze trends against other AXON-integrated experimental parameters. With AXON, you end up with higher quality results and conclusions in significantly less time with a lot less effort than trying to track manually.



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CALCULATING ELECTRON FLUX AND CUMULATIVE ELECTRON DOSE FOR IN-SITU EXPERIMENTS

$$d = \frac{i_e}{eA}$$

d = electron flux (electrons/ Å²s)*
i_e = beam current (C/s)
e = the elementary charge (C/electron)
A = the area of the STEM scan (Å²)
D = cumulative electrons (electrons/Å²)
[Dwell time] = scan speed

$$D_{STEM} = d * [dwell\ time]$$

$$D_{TEM} = d * [exposure]$$

Step #	Task	Required Parameters for Calculations			Dynamic During Experiment	Tracked by AXON
			STEM	TEM		
1	CALIBRATE BEAM CURRENT	Spot Size	✓	✓	✓	✓
		Condenser Aperture	✓	✓	✗	✗
		Accelerating Voltage	✓	✓	✗	✓
2	RUN YOUR EXPERIMENT	Spot Size	✓	✓	✓	✓
		Scan Area	✓	✗	✓	✓
		Frame Size	✓	✓	✓	✓*
		Dwell Time	✓	✗	✓	✓
		Exposure Time	✗	✓	✓	✓
3	ANALYZE DATA	Export Parameters to Excel	✓	✓		
		Enter Beam Calibration	✓	✓		
		Calculate Dose	✓	✓		

* Because the electron exposed area of the TEM beam is NOT equivalent to the image frame (unlike STEM), the beam spread must be adjusted at the start of the experiment to just fill the frame and cannot be changed during the experiment. If the beam spread is fixed to the image frame area and held constant during the experiment, the frame size recorded by AXON may be utilized for the illuminated area during dose calculations.

CALIBRATING BEAM CURRENT

In order to accurately calculate the electron dose, the microscope's beam current must be calibrated. This can be done by either using a Faraday cup to directly measure the beam current or acquiring an image of the beam/ probe on the CCD and using the camera conversion efficiency value to convert the counts to electrons.[2] This calibration is performed by measuring the beam current for each spot size and condenser aperture combination for a given acceleration voltage. Because AXON records the spot size for each image, which is a dynamic parameter in an experiment, it becomes easy to use standard Excel functions to apply the correct calibration for each image's conditions and calculate flux and cumulative dose.

CONSIDERATIONS FOR STEM MODE

Scanning transmission electron microscopy (STEM) forms an image by rastoring a condensed electron probe over a pre-determined scan area. When imaging in STEM mode, increasing or decreasing the magnification of an image is performed by increasing or decreasing the scanned area. Thus, if the scanned area is changed by adjusting magnification, the electron flux will also change, since the flux is inversely proportional to the area over which the beam is scanned [1-3].

CONSIDERATIONS FOR TEM MODE

Unlike STEM, the electron flux (d) when using TEM mode is not affected by changes in magnification [R], but rather is affected by changing the brightness. Since the illuminated region is not defined by the area of the recorded image (as it is in STEM) this must be taken into account when setting up the experiment. There are two strategies for controlling and calculating dose when working in TEM mode:

1) Maintain a constant brightness based on the calibrated TEM beam, or
2) Maintain a constant beam size throughout the experiment [3].

REFERENCES AND RESOURCES

1. Abellan et al. “Factors Influencing Quantitative Liquid (Scanning) Transmission Electron Microscopy,” ChemComm, (2014) 50, 4877-4880.

2. Ahmad, N. (November 23, 2017). Nanoscale *in situ* studies of Au and Au-Cu Nanoparticle synthesis by liquid cell transmission electron microscopy, (Doctoral Thesis) Sorbonne Paris Cité, Paris France.

3. Woehl et al. “Direct *In Situ* Determination of the Mechanisms Controlling Nanoparticle Nucleation and Growth,” ACS Nano (2012) 6 (10): 8599-8610

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