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"No-dose" imaging

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When trying to collect high resolution information of material formation processes and resulting structures by electron microscopy often the beam sensitivity of the sample poses a limit to imaging and not the electron optics. As sketched in Figure 1A the beam sensitivity of materials, i.e., the critical damage threshold expressed in accumulated dose, is commonly associated with the loss of structural features or the onset of visible changes to the specimen and can vary significantly [1,2,3]. However, the critical damage threshold may not be a fixed value as often assumed, considering that sample preparation, applied electron flux and temperature during imaging can be used to extend it [3]. Moreover, imaging modalities (Figure 1B) and dose distribution schemes can also be optimized to maximize the information content of the acquired data [4,5,6].

In this contribution I will be discussing how appropriate averaging schemes can be utilized to access the structure of organic photovoltaics [3,4] and the dynamics of polymer vesicle formation [7] from noisy cryo-TEM and liquid-phase TEM data. These approaches can also be employed to obtain compositional information of beam-sensitive materials at high resolution from cryo-STEM-EELS [4,8]. Furthermore, for liquid-phase STEM experiments it will be shown how quantifying the reaction/assembly dynamics of amorphous cobalt carbonate (Figure 1C) in dependence of the applied electron flux permits extrapolation to "no-dose" imaging conditions, i.e., zero-electron flux [9]. This method is general and can be applied to many other systems, not only tracking their evolution in time, but more importantly, providing insights that are as close as possible to laboratory synthesis conditions. Along the same lines, it will be shown how dynamic control over the liquid layer thickness in a liquid cell can be achieved to mediate diffusion limitations for most part of the synthesis while at the same time enabling a rapid reduction in thickness whenever needed to overcome dose or resolution limitations [10]. Finally, the benefits of precise control over electron exposure in time via ultrafast beam blanking (Figure 1D) are introduced as a means to extend

the critical damage threshold for beam sensitive materials [8].

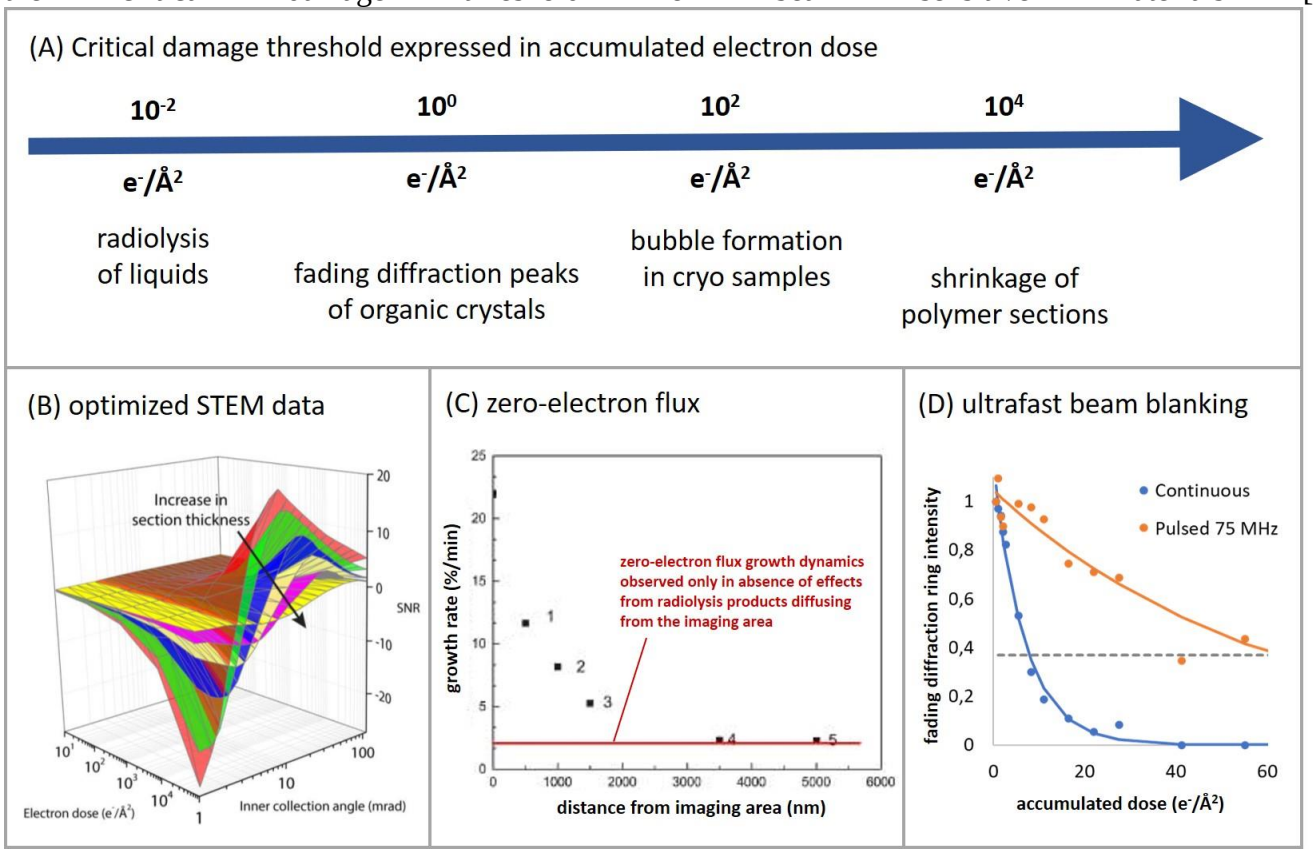

Figure 1. Figure 1 (A) Scheme illustrating differences in beam sensitivity of materials; (B) signal-to-noise ratio (SNR) of carbon nanotube (CNT) plotted as a function of electron dose, inner collection angle of ADF detector for various polymer section thicknesses 100 nm (red), 200 nm (green), 500 nm (blue), 1 µm (gold), 2 µm (magenta), and 5 µm (yellow). Note: Negative SNR represents the absolute SNR of ABF images (i.e. dark CNT in bright background) and positive SNR refers to the ADF images (i.e. bright CNT in dark background) [5]; (C) growth rate of cobalt carbonate nanoparticles measured from liquid-phase STEM data in dependence of distance from imaging area where electrons generate CO32- ions by a reaction cascade [9]; (D) fading of P3HT diffraction ring intensity in dependence of accumulated electron dose for continuous (blue) and a pulsed electron beam at 75 MHz (orange). The critical dose threshold at which the intensity has decreased to 1/e is indicated by the gray doted horizontal line [8].

References

[1] H. Wu, Heiner Friedrich, J. P. Patterson, N.A.J.M. Sommerdijk, N. de Jonge "Liquid phase electron microscopy for soft matter science and biology" ***Adv. Mater.*** 32 (2020) 2001582.

[2] H. Friedrich, P.M. Frederik, G. de With, N.A.J.M. Sommerdijk "Imaging self-assembled structures: the interpretation of TEM and cryo-TEM images" ***Angew. Chem. Int. Ed.*** 49 (2010) 7850-7858.

[3] Z. Leijten, G. de With, H. Friedrich "Quantitative Analysis of Electron Beam Damage in Organic Thin Films" ***J. Phys. Chem. C*** 121 (2017) 10552-10561.

[4] Z.J.W.A. Leijten, M.J.M. Wirix, M. Strauss, J.M. Plitzko, G. de With, H. Friedrich "Low-dose (S)TEM elemental analysis of water and oxygen uptake in beam sensitive materials" ***Ultramicroscopy*** 208 (2020) 112855.

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[5] K. Gnansekaran, G. de With, H. Friedrich, "Quantification and optimization of STEM image contrast for beam sensitive materials." ***R. Soc. open sci.*** 5 (2018) 171838.
[6] D. Chen, H. Friedrich, G. de With "On Resolution in Electron Tomography of Beam Sensitive Materials" ***J. Phys. Chem. C*** 118(2014) 1248-1257.
[7] A. Ianiro, H. Wu, M. van Rijt, M. P. Vena, A. Keizer, A. C. C. Esteves, R. Tuinier, H. Friedrich, N.A.J.M. Sommerdijk, J.P. Patterson "Liquid–liquid phase separation during amphiphilic self-assembly" ***Nat. Chem.*** 11 (2019) 320-328.
[8] Z.J.W.A. Leijten, M.J.M. Wirix, W. Verhoeven, O.J. Luiten, G. de With, H. Friedrich "Nanoscale chemical imaging of beam sensitive polymeric materials by cryogenic electron microscopy" ***J Polym Sci.*** (2021) accepted DOI: 10.1002/pol.20210012
[9] H. Su, L. B. Mehdi, J. P. Patterson, N. A. J. M. Sommerdijk, N. D. Browning, H. Friedrich "Growth kinetics of cobalt carbonate nanoparticles revealed by liquid phase STEM" ***J. Phys. Chem. C*** 123 (2019) 25448−25455.
[10] H. Wu, H. Su, A. D. A. Keizer, L. S. van Hazendonk, M. Wirix, J. P. Patterson, J. Laven, G. de With, H. Friedrich "Mapping and controlling liquid layer thickness in liquid phase (S)TEM" (2021) submitted.

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