Low dose, high resolution analytical transmission electron microscopy

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In order to expand the application of transmission electron microscopy to emerging engineering systems, particularly in the area of soft and hybrid materials, it is vital to address both sample preparation and electron beam damage issues. Here we report on the application of high resolution TEM and bright field STEM (together with EELS and EDX) to the analysis of organic crystalline materials (both monolithic and also dispersed in polymers. i.e. a formulation) as well as to beam sensitive inorganic crystalline materials (carbonates and zeolites).

This work has involved developing a more fundamental understanding of beam damage mechanisms in different materials, the identification of accurate measures of sample damage during electron exposure, investigation of the reliance of sample damage and damage rate on different microscope operating parameters (e.g. kV, TEM versus STEM, presence of a surface contamination layer etc.) as well as the accumulated electron fluence and fluence rate, developing predictive models to assess electron beam stability in new materials, as well as the investigation of cryo-preservation techniques using plunge freezing or cryo-FIB preparation in order to limit damage.

We demonstrate the development of a semi-predictive model to assess electron beam stability in organic crystals based on descriptors of molecular structure [1], using this we have shown the ability to achieve lattice imaging in highly beam sensitive materials allowing defects and surface structure to be elucidated [2]. We also demonstrate the determination of crystallinity in ultrafine inorganic surfactant-mediated nanoparticles via low dose TEM and STEM [3]. We discuss the potential for analytical measurements at high spatial resolution and also the use of compressed sensing techniques as a means to limit dose whilst retaining information content.

References

[1] M S'ari et al 2017 Microscopy and Microanalysis, 23(S1), 1194-1195

[2] J Cattle et al 2015 J. Phys.: Conf. Ser. 644 012030

[3] R W M Hooley et al 2017 J. Phys.: Conf. Ser. 902 012005