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Imaging and Understanding Metal Organic Frameworks Using Advanced Scanning Electron Microscopy Techniques

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Metal organic frameworks (MOFs) are a structurally tuneable class of hierarchical porous materials with a wide range of host-guest chemistry. Their design acts as a platform for advanced functional materials resulting in properties ranging from charge conductivity, catalytic metal centres, high surface area and organic capacitance. As a result, MOFs have a wide range of applications and can be employed in catalysis, health care, batteries, supercapacitors, and carbon capture[1].

Due to their organic and porous nature, MOFs are incredibly difficult to structurally characterise using typical scanning electron microscopy methods. Beam stability, along with non-conductive nature and porous framework result in a combination of problematic issues for nanoimaging and structural milling.

An example of the degradation MOFs suffer from is electron beam interaction causing surface damage as well as difficulty in imaging of nanoscale structures due to charging artifacts. Alongside this the amorphous nature of adsorbing MOFs results in severe sensitivity to electron beam current resulting in material loss and degradation. The preparation of MOF composite TEM lamellae by FIB milling can prove time consuming and laborious.

Herein we demonstrate a novel technique to imaging, 3D volumetric chemical analysis and TEM lamellae preparation using MOF-74 type analogue for carbon capture and mixed membrane composite CPO-27-Ni in collaboration with the University of St Andrews and the University of Cambridge [2,3]. Using imaging strategies that include high resolution variable pressure microscopy with optimised beam path lengths using NanoVP charge reduction mode, we demonstrate superior imaging at a low vacuum, improving imaging quality and eliminating sample charging and low accelerating voltage to reduce material degradation. Alongside this we employ a cryogenically cooled *in situ* stage to undertake 3D volumetric analysis of a MOF composite membrane in conjunction with energy dispersive x-ray spectroscopy. In doing so we show a new methodology for TEM lamellae preparation, 3D volumetric analysis of MOF composites and best practice imaging using low pressure, low kV scanning electron microscopy.

Figure 1: cryogenically cooled *in situ* stage to undertake 3D volumetric analysis of a MOF

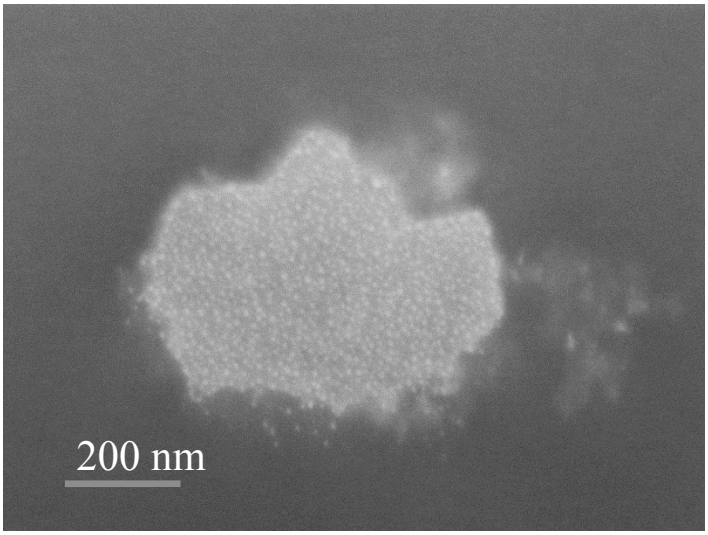
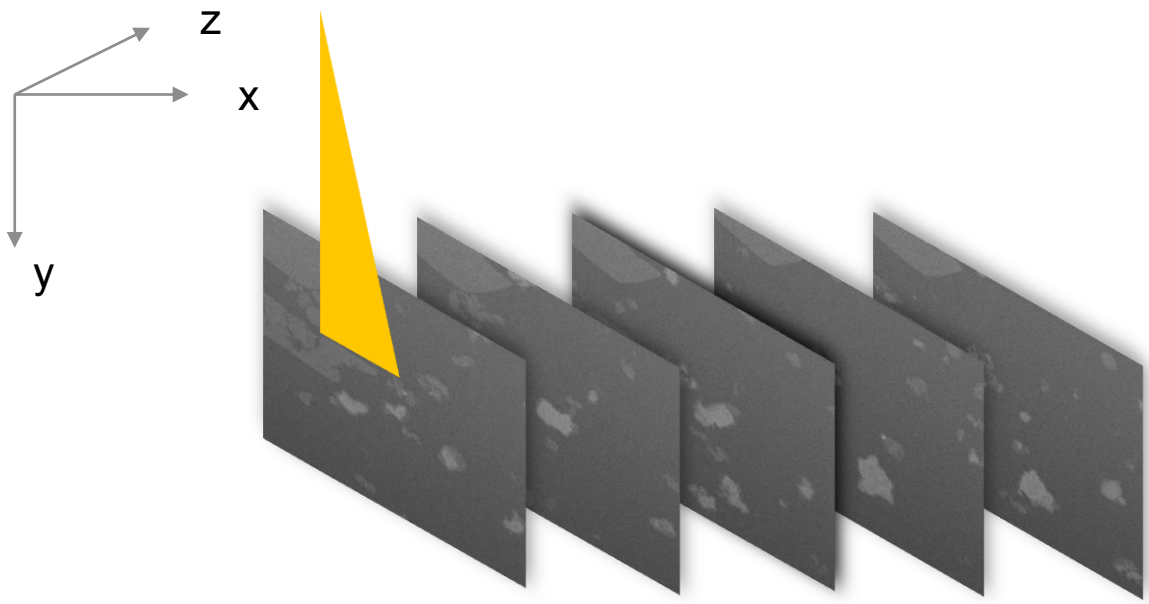


Figure 2: FIB Milling small 1-5µm step size guarantees a consistent 3D model



References:

[1] AE Baumann, Communications Chemistry **2**(1) (2019), p. 86.
[2] JH Choe, H Kim and CS Hong, Mater. Chem. Front. (2021).
[3] SM Vornholt et al., ACS Applied Materials & Interfaces **12**(52) (2020).

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