

#### Dopant distribution in zinc oxide photocatalysts

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The successful use of nanoparticulate ZnO in various applications, such as UV-screening agents or as a photocatalyst for the destruction of chemical waste requires the development of techniques for controlling its photocatalytic activity. A widely examined method for achieving this goal has been the incorporation of transition metal oxide dopants. However, experimental studies regarding the effects of particular dopants have often reported conflicting results. For example, some studies have found that cobalt doping of zinc oxide reduces the photocatalytic activity whereas other studies have reported enhanced activity. Such conflicting results are largely due to insufficient characterisation of how the dopants are distributed within the photocatalyst. In this study, transmission electron microscopy with energy dispersive x-ray spectroscopy has been used to determine the distribution of transition metal oxide dopants in nanoparticulate zinc oxide powders that were synthesised by various methods, including mechanochemical processing, hydrothermal processing, and wet chemical precipitation. The results obtained in this study demonstrate the importance of using multiple analytical techniques to characterise the distribution of dopant elements in photocatalytic materials.

#### Direct imaging of endogenous biometal distributions within millimetre-scale organisms at micrometre resolution – X-ray Fluorescence Tomography

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First-row transition metals are required for all forms of life on earth. The high reactivity of these elements means that an array of mechanisms has evolved to regulate key processes governing their transport and binding action. Tracking metals within biological tissue is non-trivial; tagging approaches suffer from lack of specificity, and can fail to find strongly-bound species; in addition, tags can interfere with normal biochemistry. Electron microscopy provides stupendous resolution, but probes miniscule volumes due to the short penetration of electrons.

With  $\mu\text{m}$  sensitivity, X-ray Fluorescence Microscopy (XFM) can probe endogenous metal concentrations at resolutions at the  $\mu\text{m}$  length scale. Elemental maps are quantitative. With penetration depth and depth of field well matched at around 0.5 mm, the method can be up-scaled to 3-D visualisations via tomography. Here we report on our application of X-ray fluorescence tomography of Zn, Cu, Fe, and Mn in *C. elegans* and discuss recent progress in developing self-absorption corrections that will enable accurate mapping of light elements.

#### Interleaving patterning method to reduce focused ion-beam induced damage in beam sensitive materials

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Most ion-beam sensitive materials, such as biological samples and polymers, experience significant heat damage during Focused Ion Beam (FIB) milling processes<sup>1</sup> apart from other undesirable effects such as ion implantation and amorphization. The heat generated by the energy loss of the ion beam can cause dramatic local temperature increases in a material with low thermal conductivity<sup>2</sup>. Cryogenic conditions do not necessarily prevent or delay the degradation of organic samples caused by the local heating<sup>3</sup>.

An Interleaving Patterning (IP, called interlacing patterning in literature) strategy<sup>4,5</sup> was first used for electron beam deposition to eliminate proximity effects caused by consecutive deposition<sup>4</sup>. Later it was used to eliminate thermal stress during FIB processing<sup>5</sup>. Recent development work was focused on improving pattern acuity in nanofabrication<sup>6</sup>.

The IP technique is extremely effective in preventing local temperature spikes during FIB processing. In IP subsequent dwell points do not overlap, and thus temperature increases are not additive. Heat dissipates before the ion beam return to modify an area twice. A trial on a polymer film showed that rapid milling and large area cross-section can be achieved without visible heat damage. Here we employ the IP technique on biological materials. IP FIB milling was used to cross-section freeze-dried *Collembolea* (springtails) to study the effect of climate change on their cuticular morphology. The specimen morphology was retained during FIB milling and the cuticle ultrastructure<sup>7</sup> was revealed. Further experiments on cross sectioning and TEM lamella lift-out of a fixed mouse hypothalamus specimen confirmed that specimen morphology is well retained and that ultrastructure can be imaged.

The IP technique is able to retain specimen ultrastructure, increase contrast and improve resolution, even for biological specimens without chemical fixation and staining. Further opportunities for high-quality specimen preparation will be possible when IP is integrated into the control software in a Cryo-FIB.

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