Applications of Controlled Atmosphere Transmission Electron Microscopy in Catalysis and Photocatalysis

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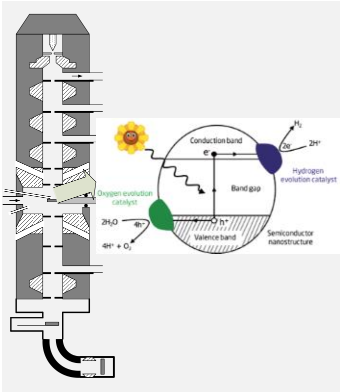

Transmission Electron Microscopy (TEM) has been an invaluable tool for catalysts scientists for decades. The addition of controlled atmosphere capabilities are of particular interest in this field as samples can be characterized under conditions approaching those under which they operate. Recently, other forms of stimuli, such as mechanical, electrical and optical, has been adopted to the technique making it even more versatile. Catalysts and photocatalysts can now be investigated at the sub-Ångstøm level under a gaseous atmosphere while exposed to light. In this paper, recent advances within in situ TEM in general and (photo)catalysts in particular.

Electron microscopy has been used extensively to characterize catalysts for decades [1]. The combination of local and spectroscopic information makes it a powerful experimental tool. The combination with *in situ* capabilities provides researchers with a one-stop solution for sample morphology and composition under simulated reaction conditions.

Adding *in situ* capabilities to a high-resolution tool like the transmission electron microscope without compromising its performance is a challenging task [2,3]. Here, the development of sample holders capable of exposing samples to optical irradiation as well as the performance of the microscope performance under *in situ* conditions the will be shown. Other examples from heterogeneous catalysis will be used to highlight the capabilities of the tool.

Cuprous oxide has been identified as an active catalyst for the water splitting and hydrogen evolution from an ethanol solution. However, Cu_2O suffers from photocorrosion. This phenomenon was investigated using controlled atmosphere transmission electron microscopy. Fig. 1 shows how the photoinduced degradation of cuprous oxide to metallic copper under an aqueous atmosphere using bright-field imaging, electron diffraction and electron energy-loss spectroscopy [4]. All three techniques show the transformation from oxide to metal.

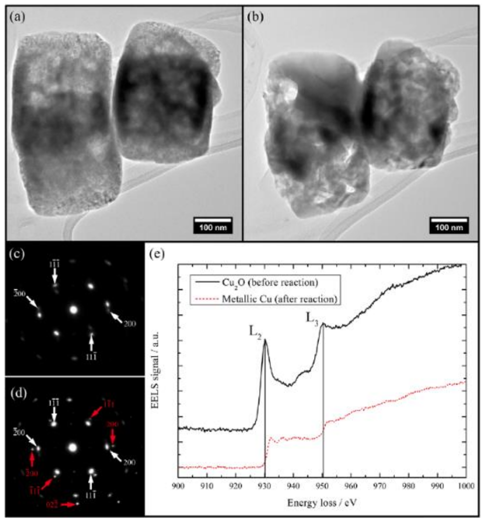

Fig. 1 Bright-field images, electron diffraction and Electron energy-loss spectra showing the photodegradation of cuprous oxide under optical illumination in an aqueous environment.

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References

[1] T. W. Hansen and J. B. Wagner, *ACS Catal*. 4 (2014) 1673.
[2] J. B. Wagner, F. C. Cavalca, C. D. Damsgaard, L. D. L. Duchstein and T. W. Hansen, *Micron* 43 (2012), 1169.
[3] *Controlled Atmosphere Transmission Electron Microscopy*. T. W. Hansen, J. B. Wagner (Eds.), Springer, 2016.
[4] F. C. Cavalca, A. B. Laursen, B. E: Kardynal, R. E. Dunin-Borkowski, S. Dahl, J. B. Wagner, and T. W. Hansen, *Nanotechnology* 23 (20012), 075705.