The Environmental TEM in the in situ Toolbox for Materials Science

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The Environmental TEM in the *in situ* Toolbox for Materials Science

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Visualizing gas-solid interactions at the sub-nanometer scale has significant impact on materials science fields such as catalysis and bottom-up nanofabrication.

Environmental Transmission Electron Microscopy (ETEM) is one way to approach high-resolution imaging of the processes taking place under reaction conditions. Here, a few examples on how ETEM has been used to understand materials science will ben presented.

Combining imaging and spectroscopy capabilities with a spatial resolution approaching 1Å at elevated temperatures and controlled gaseous environments makes ETEM a unique tool. However, as with any other characterization technique, ETEM has drawbacks. Limitations in sample geometry and field of view call for use of complementary *in situ* characterization techniques such as *in situ* XRD, XAS and EXAFS. The combination of techniques will paint a more complete picture of the sample under a reactive environment.

In order to take full advantage of the complementary *in situ* techniques, transfer under reaction conditions is essential. A dedicated TEM transfer holder allow for *in situ* transfer of sample in a gaseous environment at temperatures up to approx. 900°C between different *in situ* technique set-ups [1]. Figure 1 shows the concept of the transfer holder. Cuprous oxide has been reduced in an *in situ* XRD set-up in hydrogen and transferred to the ETEM in 10^5 Pa of H_2 at 220°C keeping the copper metallic.

In general, the catalytic behaviour of nanoparticle systems can be greatly influenced by the synthesis procedure used. The different stages of the formation process such as drying, calcination, and reduction can all be optimized by tweaking parameters such as temperature, time of treatment and gaseous environment. Following the process of identified volume subsets of the catalyst material by means of electron beam based characterization at the different stages of synthesis gives insight in the formation mechanisms of individual nanoparticles from precursor to active catalyst.

Growth of nanostructures by cracking of gaseous precursors is a complex process, which in some cases suffers from lack of full control. Systematic ETEM studies of such processes give fundamental insight in the growth independent of the chemical processes of the gaseous precursors leading to solid material growth are controlled by seed particles and catalysis [2] or by the electron beam [3].

An example of the *in situ* growth of layered carbon structures from C_2H_2 on Ni particles is shown in Figure 2. The time-dependent growth rate of the carbon layers is easily extracted from the movie frames.

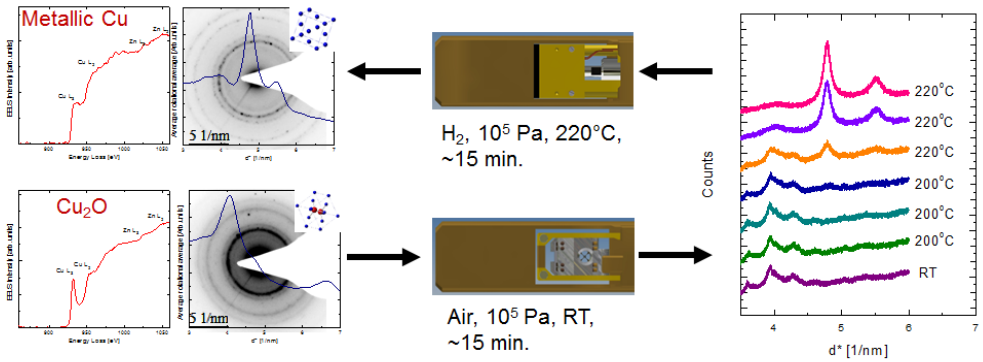

Figure 1: In situ transfer of copper containing nanoparticles between ETEM and in situ XRD setup. The cuprous oxide particles are reduced in the XRD and transferred under elevated temperature in hydrogen to the ETEM keeping the copper metallic.

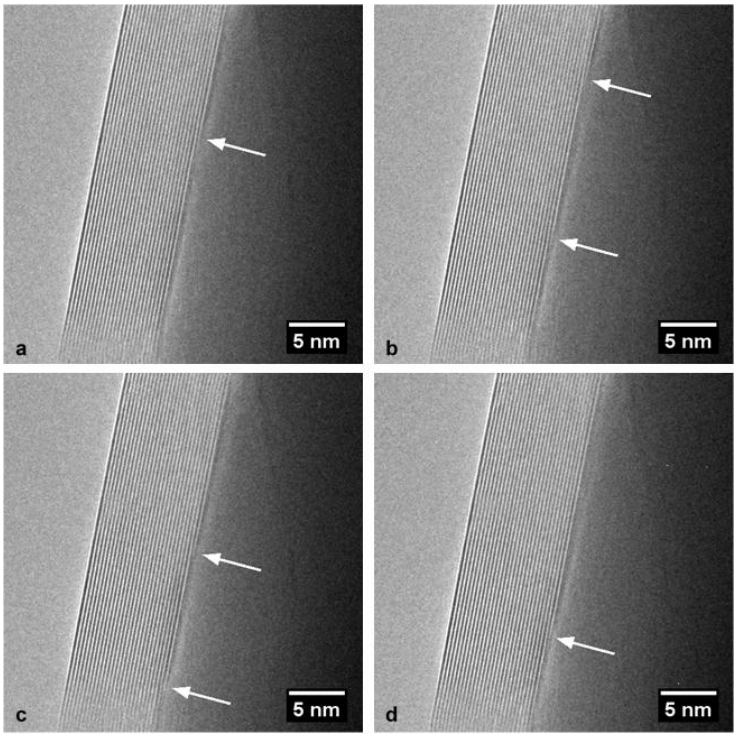

Figure 2: Image series with 0.61s between the images. The in-plane growth of the carbon layers is easily observed (see arrows). The images are acquired approximately 100s after introduction of C_2H_2. T= 650 °C, P(C_2H_2)= 3×10^{-2}Pa.

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[1] C. D. Damsgaard et al., Microsc. and Microanal. 20 (2014) 1038.
[2] M. He et al., *Scientific Reports* 3 (2013) 1460.
[3] W. F. van Dorp et al., *ACS Nano* 6 (2012) 10076.