Solid State Dynamics Visualized by Environmental EM

Jakob B. Wagner

Center for Electron Nanoscopy, Danchip/CEN, Technical University of Denmark, Kgs. Lyngby, Denmark

A good understanding of the dynamics and formation mechanisms of low-dimensional nanostructures from bottom-up processes is of great importance in order to exploit the controllability of the nanostructures and their applications in photovoltaics, electronics, sensors, *etc.* on an industrial scale.

In situ environmental electron microscopy allows for monitoring gas and heat induced dynamics at the nanoscale in real time. Examples of such reactions include growth of 2D materials (graphene)[1], thin metal films [2], pseudo 1D materials (carbon nanotubes)[3] and semiconductor oxides such as CuO[4].

The high spatial resolution enabled in electron microscopy combined with spectroscopic capabilities enables fundamental insights during the dynamical processes of the nanostructured materials directly linked to growth parameters such as atmosphere composition and temperature.

Thin metal films are widely used in micro and nanofabrication for a vast variety of devices. Downscaling of such devices and thereby, thin films makes them more prone to dewetting due to process temperatures. In situ studies of the dewetting behavior shines light on the mechanism of adhesion at the nanoscale (Fig. 1).

Growth and growth termination of single wall carbon nanotubes are very dependent on the local environment of the catalytic seed particle forming the tubes [4]. Topology of the seed particle support, the state of the catalytic particle, and carbon source play a crucial role in the growth dynamics. Observing the initial solid carbon formation on seed particles at the atomic scale reveals the conditions leading to encapsulation of the catalyst particles (no carbon nanotube growth) and a carbon layer lift-off resulting in nanotube growth, respectively.

Nanostructures can also be formed by simple oxidation of metals such as copper. CuO nanowires are formed by oxidation of metallic copper in situ in the electron microscope. The exact geometry of the resulting structures depends strongly on the partial pressure of oxygen during the heat treatment. Low pressure results in 2-dimensional copper oxidation in contrast to the 1-dimensional CuO nanowires forming at higher pressures. Post-growth treatment of the oxide nanowires at elevated temperature at reduced partial pressure of oxygen results in dissolution of the formed nanowires [5]. The direct observation of oxidation and disintegration reveals the nature of the facet dependent reactions.

Solid state dynamics at the nanoscale directly visualized by means of controlled atmosphere electron microscopy is a key factor in the understanding of the formation mechanisms and thereby a stepping stone towards the controlled synthesis of tailored materials.

[1] J. Kling *et al.* Carbon 99, 261 (2016)
[2] A. Bastos Fanta *et al.* Materials Characterization 139, 452 (2018)
[3] M. He *et al.*, Scientific Reports 3, 1460 (2013)
[4] S. Rackauskas *et al.*, Nano Letters 14, 5810 (2014)
[5] L.. Zhang *et al*. ACS Nano 11, 4483 (2017)
[6] S. Rackauskas *et al.,* Scientific Reports 7, 12310 (2017)

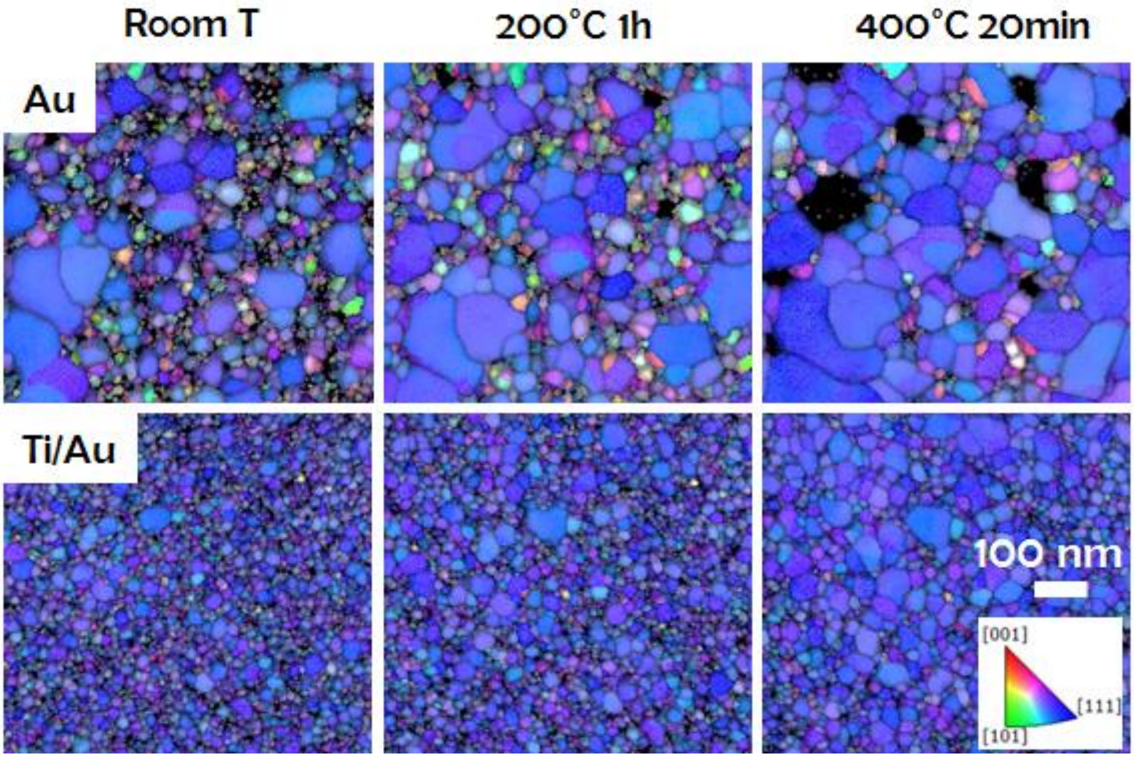

Figure 1. In situ Transmission Kikuchi Diffraction of thin Au film on SiN. The adhesion layer (Ti) clearly influence the stability and dewetting of the film.

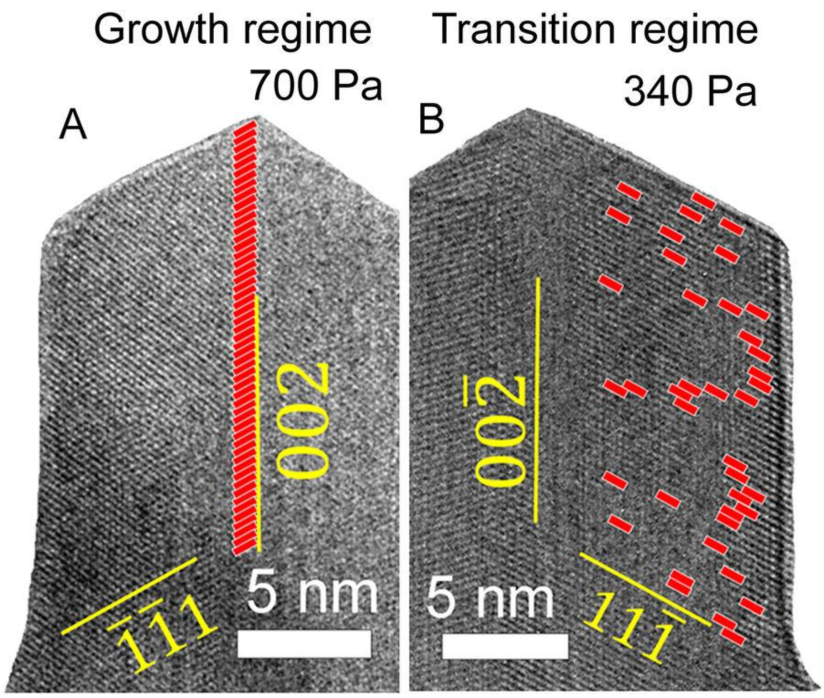

Figure 2. Oxidation of copper into a high aspect ratio CuO nanowire monitored by means of in situ environmental transmission electron microscopy. The layer-by-layer growth of CuO is heavily influenced by the vapor pressure of oxygen in the microscope. Figure adapted from [6].