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Nanoparticle Structure and Dynamics Studied Using Controlled Atmosphere Transmission Electron Microscopy

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Nanoparticle and catalysis research makes extensive use of transmission electron microscopy (TEM). In particular, environmental transmission electron microscopy (ETEM) has attracted considerable attention in recent years. This technique allows us to expose samples to gaseous environments at elevated temperatures in order to investigate local structural changes at the atomic level as the environment changes. Recently, the technique has also been used in nanowire, graphene and electron optical lithography research among others.
Recent developments in TEM instrumentation include monochromation of the electron source and aberration correction of both condenser and objective lenses. These developments have now been introduced onto the ETEM column. The improved spatial resolution and interpretability provided by these additions are beneficial for imaging the surface structure and dynamics of catalyst nanoparticles and provides exciting new possibilities for investigating chemical reactions. In order to take full advantage of this, an understanding of both the interaction of fast electrons with gas molecules and the effect of the presence of gas on high-resolution imaging is necessary.
Using an FEI Titan ETEM equipped with a monochromator and an aberration corrector on the objective lens, we have investigated sintering of supported metal nanoparticles often used in catalysis. A model system consisting of supported gold nanoparticles were prepared by sputter-depositing the metal onto graphene and boron nitride substrates. These samples were imaged under hydrogen at increasing temperatures. Gas was introduced into the environmental cell using digitally controlled mass flow controllers providing accurate and stable control of the pressure in the cell. As the temperature was increased, migrating particles were observed on the support. As they came into contact, a neck was formed between the particles and subsequently, the particles coalesced entirely (Fig. 1). Growth patterns have also been investigated for platinum and palladium nanoparticles supported on silicon oxide substrates. Here, anomalously large particles were observed as the particles were sintered in oxygen atmospheres at temperatures at elevated temparatures. Such large particles have also been observed for industrial catalysts. In this study, we will try to elucidate the mechanisms of metal nanoparticle sintering.
The analytical capabilities of the microscope can be further augmented by adding stimuli such as optical or electrical though the sample holder. Using a holder capable of exposing the sample to light, the redox properties of cuprous oxide have been investigated. Cuprous oxide has been identified as an active catalyst for the water splitting and hydrogen evolution from an ethanol solution. However, Cu2O suffers from photocorrosion. This phenomenon was investigated using controlled atmosphere transmission electron microscopy. Fig. 2 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. All three techniques show the transformation from oxide to metal.
Effects of imaging in various elemental as well as di-molecular gases and their effect on imaging and spectroscopy in the environmental transmission electron microscope will also be discussed.

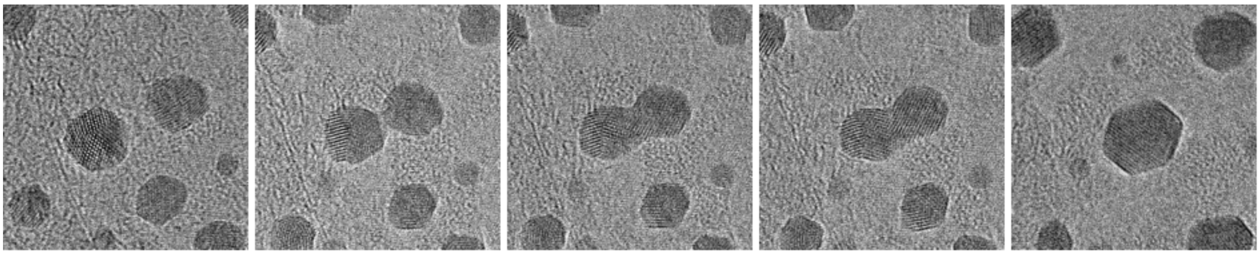

Figure 1: ETEM image sequence of Au/graphene in 200 Pa H2 at 104°C. The image series shows different stages of two Au particles coalescing resulting in a single Au particle. Image dimensions: 18x18 nm.

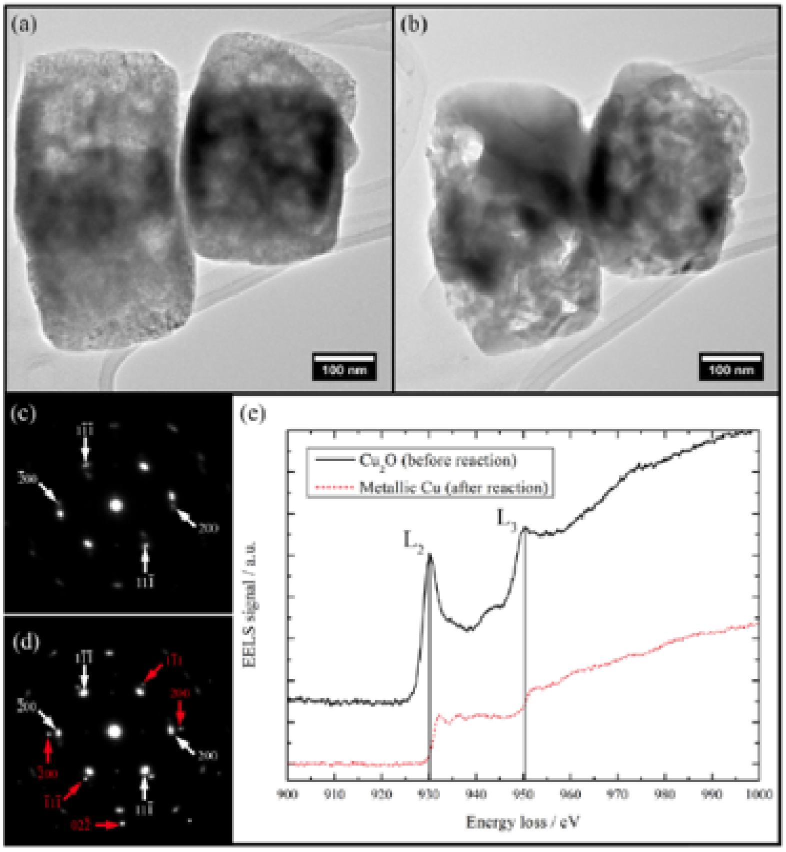

Figure 2: Bright-field images, electron diffraction and Electron energy-loss spectra showing the photodegradation of cuprous oxide under optical illumination in an aqueous environment.