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Understanding Bimetallic Catalysts Via *Ex-Situ* TEM Studies

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Bimetallic catalysts remain an interesting but poorly understood area of science. The effects of sequential reduction - oxidation - reduction (ROR) treatments on the nanostructures of these materials are often unknown. However, with the use of *exsitu* TEM techniques[1, 2] recent studies have begun to explore these effects. Previous *ex-situ* TEM studies revealed that a simple RO treatment of small, Cu metal particles created torus structures of Cu oxide[3,4]. The evolution of this Cu oxide structure was related to differences in Cu and O ion diffusion rates. Similar but more extensive *exsitu* TEM investigations are now being conducted on bimetallic particles.

In this work, CuAu particles were deposited onto a standard, 200 mesh, holeycarbon-coated, Mo TEM grid using an Edwards high vacuum (10^{-5} Torr) deposition system. The TEM grid was transferred into ExxonMobil's *ex-situ* treatment facility (Figure 1) where it was thermally annealed for 1 h at 500 ^{o}C under flowing (20 sccm) hydrogen. This treatment gave rise to bimetallic particles that appeared well distributed on the carbon support film (Figure 2a). The TEM grid was then transferred back into the *ex-situ* TEM facility, and the CuAu particles were given a 6 h air oxidation treatment at 200 ^{o}C. The grid was placed back into the TEM, and the same CuAu particles that were imaged subsequent to the thermal anneal in hydrogen were re-examined (Figure 2b).

The air oxidation treatment resulted in a significant change in the bimetallic particles' morphology. In the case of this low temperature air oxidation treatment, the bimetallic CuAu particles transformed from uniformly dark, alloy structures into particles having two distinct regions: a light grey coating around a dark core (compare Figure 2a with Figure 2b). EDS data collected from the light grey regions of the particles indicated that these areas were preferentially enriched in Cu. Thus, significant phase separation occurred within each particle during the low temperature air oxidation treatment. Interestingly, the final 1 h treatment at 300 ^{o}C under flowing (20 sccm) hydrogen altered both the original materials' original particle size distribution and its chemistry by creating a mix of small, Cu-rich particles and Cu-depleted bimetallic structures (compare Figure 2a and Figure 2c).

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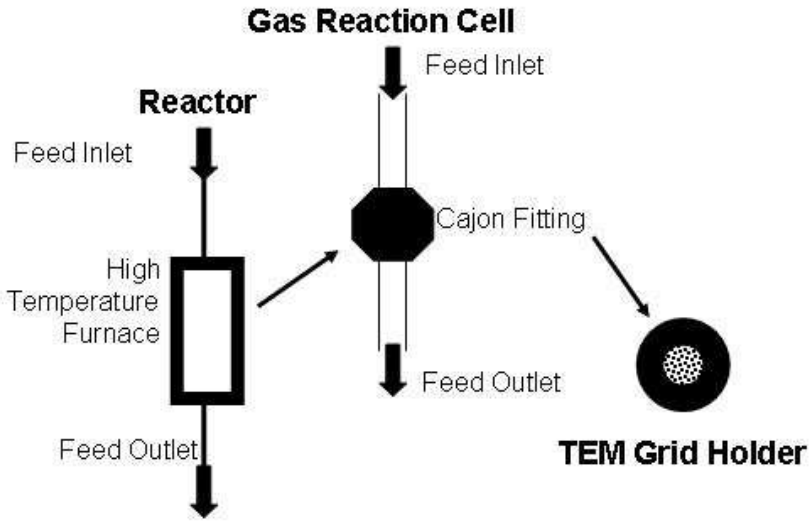
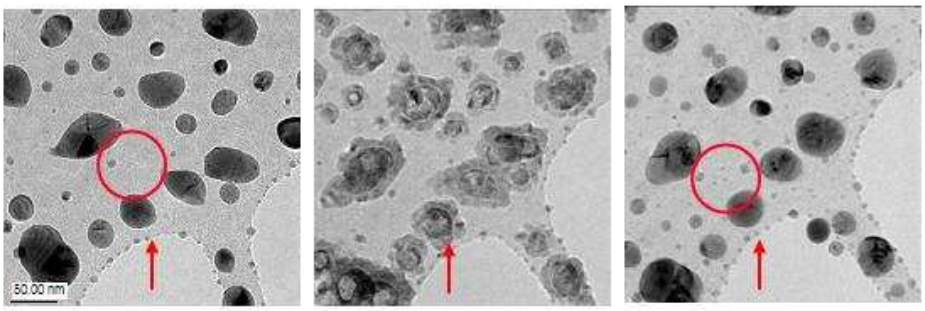


Figure 1: Schematic of "ex-situ" gas reactor system.



a b c

Figure 2: Bright field TEM image showing the general morphology of reduced CuAu particles subsequent to (a) initial reduction, (b) air oxidation, and (c) re-reduction.

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