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Characterization of Iron-Containing Nanoparticle Catalyst Using STEM Techniques

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Non-noble metal catalysts, especially, single atom catalysts containing non-noble metallic elements, have attracted great attention recently. These catalysts have some economic and environmental advantages, such as, reduced cost, greater supply and lower toxicity. We have successfully synthesized Fe-Nx catalysts supported on carbon nanotubes and characterized them using different techniques. Here we report some STEM results.

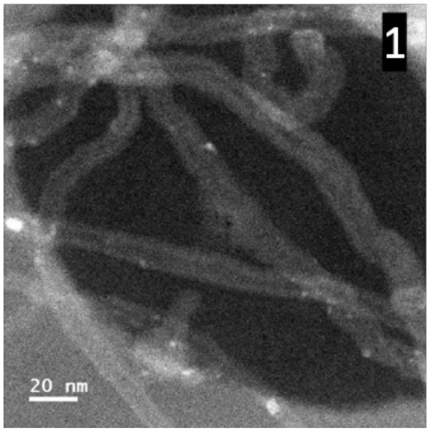
Figure 1 is a HAADF STEM image showing the morphology of the catalysts, where Fe-containing particles (bright spots) on the carbon nanotubes can be seen readily. Carbon nanotubes have distinct lattice fringes with the interplanar spacing of 0.34nm. Figure 2a is a bright-field STEM image, showing such kind of 0.34nm lattice fringes. Small Fe-containing nanoparticles do not show up in Figure 2a clearly, but corresponding HAADF STEM image (Figure 2b) highlights these particles. Some particles may be smaller than a couple of nanometers.

To confirm that these nanoparticles contain Fe, different nanoanalysis techniques such as EELS and EDS were applied. Figure 3 displays an EELS spectrum from one nanoparticle, where carbon K-edge and Fe L-edge are marked. An inset with an enlargement of EELS spectrum in the range of 600-800 eV is included to show Fe L-edge more clearly. Elemental maps were carried out using EELS and EDS. Figures 4b and 4c show the carbon and iron maps using EELS in a small area (Figure 4a), respectively. The 5nm Fe particle shows up in the Fe map nicely. Because small size of electron beam was used to achieve high resolution imaging, smaller particles have weaker EELS signal and cannot be identified easily in the map, but Fe edge signal can be seen from EELS mapping data.

High-resolution HAADF STEM images were also obtained to reveal individual Fe atoms. The STEM work was conducted on JEOL Grand ARM 300 kV S/TEM with double aberration correctors, providing high resolution STEM image at single atom level. Figure 5a shows some iron particles on the carbon tubes. Atomic resolution was achieved in the image, where Fe atom related contrast can be seen clearly in one particle marked in a circle. Single Fe atoms show up obviously at the edge of the carbon marked with a square. Figure 5b is the enlargement of the area marked in the square in Figure 5a, revealing Fe individual atoms as bright spots.

In conclusion, Fe-containing nanoparticles and single atom catalysts have been made and aberration corrected STEM is powerful to reveal single atoms. More detailed study of the Fe-Nx catalyst using other techniques will be reported later [1].

References:
[1] The catalyst samples were synthesized at Nanjing Tech University and S/TEM work was conducted at UCI. The authors would acknowledge the use of JEOL Grand ARM TEM in IMRI facilities at UCI.



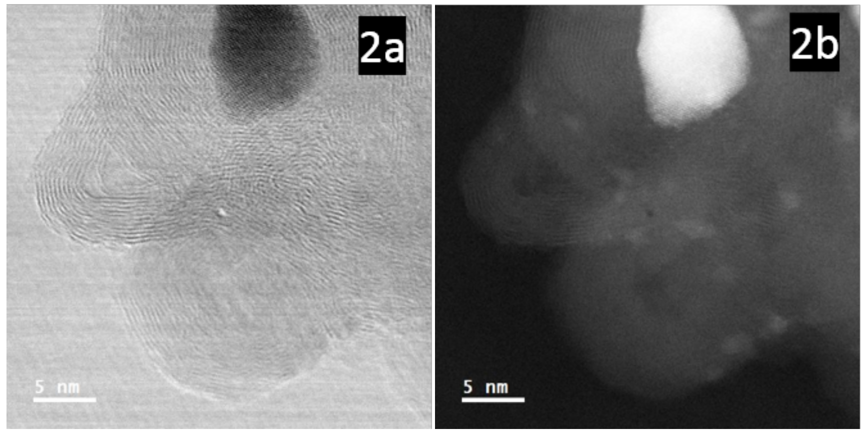
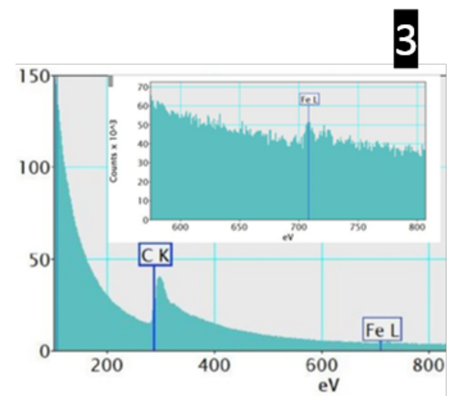
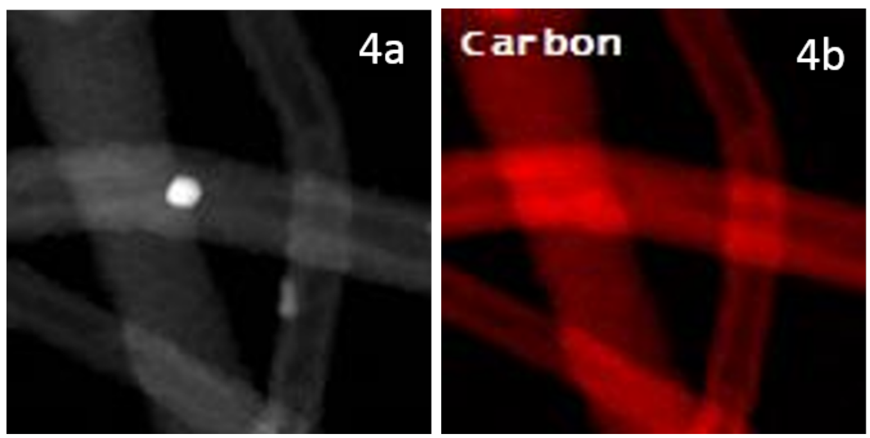
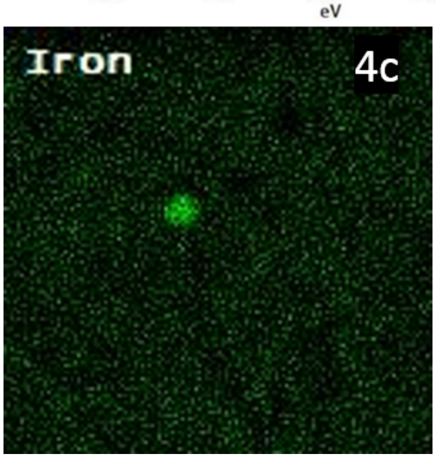


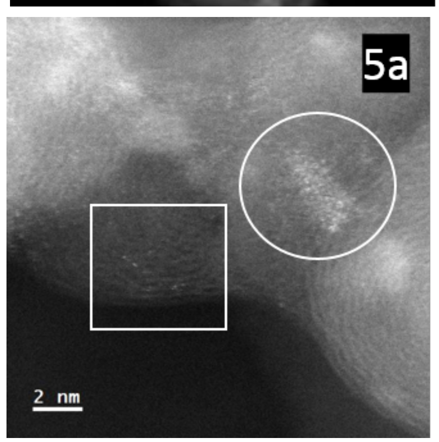
Figure 1. HAADF STEM image showing the morphology of the catalyst sample.

Figure 2. a) Bright-field STEM image and b) HAADF STEM image of the sample at high magnification.









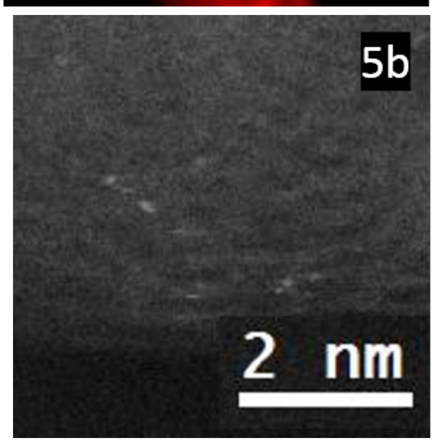


Figure 3. a) An EELS spectrum from one nanoparticle supported on carbon nanotube. The inset shows an enlargement of the EELS spectrum in the 600-800 eV range.

Figure 4. a) HAADF STEM image of a small area, b-c) carbon and iron maps of the area using EELS.

Figure 5. a) HAADF STEM image showing individual Fe atoms in the circle and the square, b) enlargement of the area in the square of a).

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