In-situ Observations with High Spatial and Temporal Resolution

N. D. Browning[1], I. Arslan[1], M. A. Bonds[2], J. E. Evans[1], K. L. Jungjohann[2], J. McKeown[3], S. Mehraeen[2], L. R. Parent[2], W. D. Ristenpart[2], W. A. Schroeder[4], T. J. Woehl[2], H. Yang[2]

[1]Fundamental and Computational Sciences Directorate, Pacific Northwest National Laboratory, P.O. Box 999, Richland, WA 99352 USA

[2]Chemical Engineering and Materials Science Department, University of California-Davis, One Shields Ave, Davis, CA 95616 USA

[3]Physical and Life Sciences Directorate, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

[4]Department of Physics, University of Illinois at Chicago, 845 W. Taylor St, Chicago, IL 60607 USA

The last few years have seen a paradigm change in (scanning) transmission electron microscopy with unprecedented improvements in spatial, spectroscopic and temporal resolution being realized by aberration correctors, monochromators and pulsed photoemission sources. Spatial resolution now extends to the sub-angstrom level, spectroscopic resolution into the sub-100meV regime and temporal resolution for single shot imaging is now on the nanosecond scale. However, to fully utilize these instrumentation developments to study both structures and processes, a full understanding of the variability in the initial structure must be obtained and in-situ stages to control the environment around the sample must also be employed.

In this presentation, the development and implementation of two environmental stages will be discussed. An in-situ gas stage has been developed in collaboration with Fischione Instruments that allows atmospheric pressure in a range of reactive gases to be maintained around the sample while atomic resolution images are obtained. By utilizing a novel laser heating source, temperatures up to 2000^{o}C can also be obtained in small areas of the sample. Such capabilities allow for direct imaging of oxidation and reduction processes in metals, ceramics and catalytic systems. An in-situ liquid stage has been developed in collaboration with Hummingbird that allows atomic scale images and electron energy loss spectra to be obtained from samples suspended in solution [1]. This has a wide range of applications to studying corrosion in materials science and also to studying live biological systems [2]. These stages have been designed to be incorporated into both high spatial resolution aberration corrected (S)TEM as well as into high temporal resolution Dynamic TEM (DTEM) [3].

An example of the use of the liquid stage is shown in Figure 1, where purified ferritin molecules within a liquid environment are imaged using aberration corrected STEM. Of course Brownian motion or fluid flow can both negatively affect the imaging of any type of purified biomolecule or nanoparticle during an acquisition period ranging from milliseconds to seconds (this effect is negated by the DTEM). Such effects can be seen in Figure 1b where the white arrowhead indicates a ferritin molecule that was moving through solution while the image was recorded. However, other ferritin molecules were imaged without such artifacts and confirm the presence of both the less dense protein shell and more dense nanoparticle core with equivalent dimensions to the known

structure. The unambiguous detection of the protein shell surrounding the nanoparticle core indicates the spatial resolution of this approach is better than 2 nanometers. Additionally, apoferritin molecules were visualized in the absence of the nanoparticle core and these combined results prove that this integrated approach can visualize individual proteins through a fluid thickness up to 100 nm [4].

References

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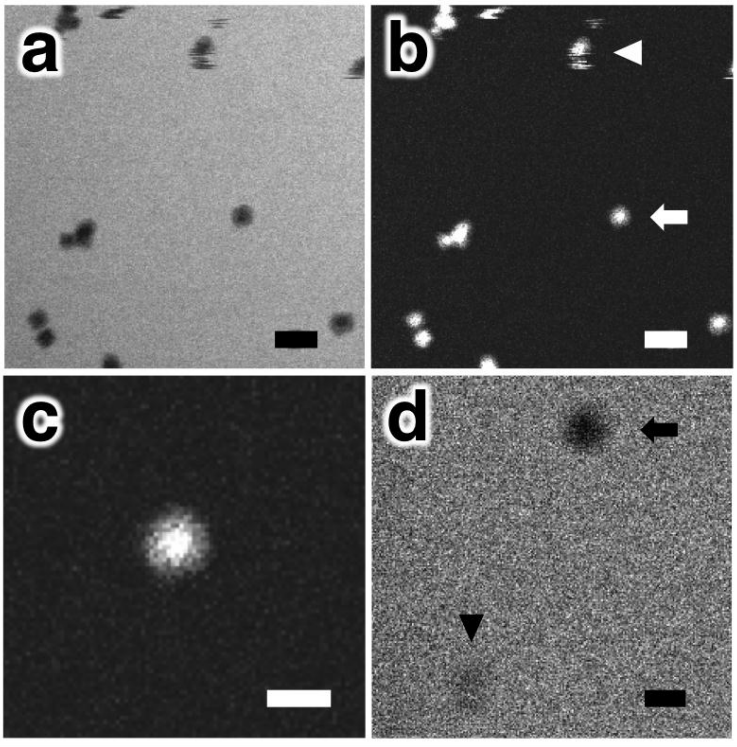

Figure 1 (a&b) *In situ* bright field and dark field STEM images of a suspension of ferritin molecules in a buffered saline solution. The white arrowhead points to a ferritin molecule that moved during acquisition. c) Magnified view of ferritin molecule indicated by white arrow in (b) depicting both the outer protein shell and more dense (brighter) inner iron oxide nanoparticle. d) *In situ* bright field STEM image of ferritin and apoferritin. Scale bars are 10 nm.