The Importance of Lower Voltages for the Application of Aberration-Corrected TEM to Nanomaterials

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There are several benefits in using medium-to-high voltage transmission electron microscopy (TEM) to characterize materials. These include better spatial resolution and higher penetration power, which allows thicker specimens to be observed and thereby retaining their bulk nature. With the implementation of aberration correctors in TEMs and increasing interest in nanomaterials, the field of low-voltage, aberration-corrected TEM is rapidly gaining importance. In this talk, we will highlight a few examples in which the use of lower voltages is important when studying nanomaterials using aberration-corrected TEM.

Since their discovery in 1991 carbon nanotubes (CNTs) [1] have found an increasing number of applications, most notably as field emission electron sources in X-ray tubes for medical applications [2, 3]. Under less stringent vacuum conditions, the field emission current and lifetimes of CNTs are found to decrease [4, 5]. To study the underlying mechanism of carbon nanotube oxidation, we observed structural changes in CNTs as they were oxidized *in situ* using an aberration-corrected environmental TEM (ETEM). An 80 kV incident electron beam energy (which is below the threshold energy for knock-on damage in single-walled carbon nanotubes [6]) was utilized in this study. Contrary to earlier reports that CNT oxidation initiates at the end of the tube and proceeds along its length, our findings show that only the outside graphene layer is being removed and, on occasion, the interior inner wall is oxidized, presumably due to oxygen infiltrating into the hollow nanotube through an open end or breaks in the tube [7]. The CNT caps are not observed to oxidize preferentially [8].

The study of nanomaterials using TEM typically requires the use of thin supporting substrates such as amorphous carbon or SiO_2. At higher accelerating voltages (200 kV and above), Cherenkov radiation alters the low-loss electron energy-loss (EEL) spectrum of SiO_2, but this phenomenon disappears when the voltage is reduced to 80 kV. Using STEM-EELS with an 80 kV electron beam energy, we show band gap variations within dome-shaped PbS quantum dots that have been dispersed on a SiO_2 support film [9]. We also report the first direct measurement of hydrogen absorption and desorption in individual palladium nanocrystals on a SiO_2 substrate using *in situ* environmental STEM-EELS at 80 kV [10].

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Acknowledgements
This work is supported by funding from the National Cancer Institute through a CCNE grant, the Center on Nanostructuring for Efficient Energy Conversion, an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under Award Number DE-SC0001060. Use of the Stanford Nano Shared Facilities is appreciated.