NOVEL DEVELOPMENTS IN HIGH RESOLUTION ELECTRON MICROSCOPY FOR ULTIMATE NANOTUBE METROLOGY

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Measurements are the backbone of any scientific research and development and any industrial commercialization. Nanotubes, specifically Carbon Nanotubes (CNTs) are a case in point: development of standards for measurement, characterization and test methods are needed and being developed, taking into consideration needs for metrology and reference materials [1]. Measurements include assessment of morphology, dimensionality, purity, physical properties, and chemical modifications.

In this contribution, we present the latest electron microscopy advancements (imaging and calibration), which may prove powerful for developing and implementing HR-TEM characterization techniques for CNTs.

The method of High Resolution Transmission Electron Microscopy (HR-TEM) is seen powerful in measuring the above mentioned first three parameters, leading to information on wall structure, amorphous carbon, metal coating, surface cleanliness, and tube dimensions. Conventional HR-TEM is well-accepted in the field of nanostructure characterization. Atomically resolved images can be obtained routinely. However, the interaction of the electrons with the material of investigation may cause break-down or damage. It is an empirical fact that Carbon Nanotubes exhibit limited life times at the operating energies in conventional HR-TEM. Typically, 200keV or more energy is used to obtain atomically resolved images. Some structures (SWCNT) show life times in the order of seconds or less. This limits the applicability of such traditional method for CNT measurements. To extend life times, the energy of the beam in HR-TEM is lowered to values at or below 120keV. In conventional microscopy, this lowering typically results in a loss of the atomic resolving power. However, advancements in HR-TEM technology with the advent of aberration corrector technology have allowed permitting high resolution imaging with improved contrast and sample stability at these lower beam energies.

In this contribution we would like to discuss these latest developments in the context of CNT imaging and atomic detail measurements. We will demonstrate that measurement times on a single Carbon Nanotube can now be extended to minutes, while still resolving this delicate structure atomically. We will show that sub-Angstrom information transfer is possible at 80keV. A series of CNT measurements will be discussed to proof the applicability of the new HR-TEM

method for ultimate imaging and analysis. Even, we will report the result of a focus series reconstruction, delivering quantitative imaging with atomic detail. Such measurements, typically needing 10-30 seconds of experimental time, can only be performed when the life time is prolonged. We anticipate that aberration corrected HR-TEM will be a powerful method for ultimate imaging on CNTs and may represent the benchmark for complementary measurements. The improved performance over the full range of accelerating voltages allows beam energy to be freely selected to optimize performance for a particular material or analysis mode. This will allow further application of this new technology for metrology questions for various nanoparticle classes also in the context of Environment, Health and Safety [2].

The methods for reliable and reproducible characterization of nanoparticles not only call for the appropriate microscopy techniques, they also require protocols and standards for microscopy calibration. In this paper we will describe a novel approach for calibration of the TEM, to precisions and trueness within <2%. It relies upon commonly accepted calibration samples and is based on innovative automatic image processing and spans a TEM magnification range over 5 decades, starting from the highest level of detail - atomic spacing of the calibration sample. This is the first time this approach is discussed in the context of metrology of Nanotubes.

References

[1] ISO TC229, Nanotechnologies: WG 2: Metrology & Characterization. Focus on Carbon Nanotubes.

[2] “The National Nanotechnology Initiative: Environmental, Health and Safety Research Needs for Engineered Nanoscale Materials,” (September 2006), NS&T Council, USA.