Nanometrology Sees Progress in Synthesis, Optics, and Microscopy

The Second International Workshop on Metrology, Standardization and Industrial Quality of Nanotubes (MSIN08) took place on June 28, 2008, in Montpellier, France, as a satellite of the Ninth International Conference on the Science and Application of Nanotubes (NT08). The MSIN08 satellite conference built upon the foundation laid by the first workshop (the MSIN07, held in Rio de Janeiro, on June 22, 2007). Some findings related to the important challenges and fundamental characteristics of nanometrology, as discussed in the December 2007 issue of the *MRS Bulletin*, were revisited due to the large number of new attendees, but the focus was mostly directed toward improvements achieved in synthesis, optics, and microscopy during the past year. Besides the further development of analytical techniques for metrological studies of carbon nanotubes, also included in the program were the applications of carbon nanotubes as sensors for developing nanometrology and two presentations on the formal and legal aspects of standards.

Recent developments occurring in the synthesis of single-walled carbon nanotubes (SWNTs) have been impressive. From one extreme, a scalable and flexible technique for sorting SWNTs by their physical and electronic structure has been achieved using density gradient ultracentrifugation (DGU) in aqueous solutions. From another extreme, industrial-scale mass production of high-purity SWNTs has been achieved to establish a carbon nanotube-based industry (10 tons/year promised for 2010). With well-established and well-characterized SWNT material, metrology can develop consistently, forming the basis for the transition from nanotube-based science to technology. Confirmation of the DGU sorting methods by various groups has already occurred and mass production techniques that would decrease the price of SWNTs by several orders of magnitude are now expected.

The impressive development of SWNT synthesis could only have happened because of the equally impressive development of SWNT characterization techniques. Optical absorption and spectroscopies (mostly photoluminescence and Raman scattering), and high-resolution transmission electron microscopy have achieved the level of accuracy to characterize, unequivocally, the SWNT structure [(*n*,*m*), tube type and handedness]. Combining different methods for (*n*,*m*) characterization has given strong support to the credibility of the methods. The metrology of carbon nanotubes is rapidly moving forward, now addressing length-dependent effects and defects. These issues are important for the development of readily available techniques for accurate (*n*,*m*) population analysis.

Moreover, SWNTs are not only a prototype for the development of characterization protocols. Carbon nanotubes are also a material that can be used for the development of nanometrology tools. At the MSIN08, the successful developments of gas sensors and electromechanical devices were demonstrated. Besides being compact and potentially cheap, the gas sensors were shown to be very sensitive (measuring 100 ppt of NO_2), able to differentiate chemicals (NO_2, NH_3, NO, NO_2, CO, CO_2), effective at room temperature, fast (some seconds), lower power consumption, and potentially cheap. The nanotube-based electromechanical devices were shown to be orders of magnitude faster than usual NEMs (tunable in the MHz and GHz ranges) and technically competitive, even with a low quality factor Q, because of their high Young modulus, light weight, and natural nanoscale size. One remarkable metrological advance reported by several different groups at NT08 was the measurement of the mass of a single atom sitting on a vibrating carbon nanotube. It is interesting also to stress the good performance of the nanotube even at high temperatures. Here the big challenge is, of course, the control of the fabrication process. Many fabrication challenges still have to be overcome before these nanometrological devices become a practical possibility.

The legal and practical issues related to the production of carbon nanotube standards were also addressed. Standards are needed for any real product, and the development of standards for nanotechnology is a big challenge. A nanotube standard reference material would certainly help to strengthen the synthesis, characterization, and applications of nanotubes from a commercial standpoint. To deliver such a standard reference material, several exploratory steps are needed, such as: (1) Testing how an exploratory reference material could be produced and how it could be calibrated (the U.S. National Institute of Standards and Technology is on the way to produce the first carbon nanotube standard reference material, that will be available in 2009); (2) Determination of which physical properties should be investigated and determination of reproducible measurement techniques for each property; (3) A whole suite of standard reference materials will be needed for different properties as well as standards for measurement and analysis techniques; (4) Standards should be developed that are specific to various applications; and (5) Demand from the application side has to determine priority. It is not yet clear which applications are ready to be tested against standards. It is clear that the research community will need to get engaged in various aspects of the development of standard reference materials as well as legal standards developments.

In addition, promising applications of carbon nanotubes in the field of medicine were discussed but, at the present stage, it is not possible to answer whether carbon nanotubes are toxic and under which conditions. Too many parameters have yet to be considered, and thorough characterization of the carbon nanotube samples is mandatory for correct analysis of data. Therefore, standard protocols should be developed to protect researchers, production workers, and the environment. For the moment, the principles of precaution are the same as in the case of any chemical: wear gloves, a laboratory coat, and an appropriate dust mask (FFP3). These issues were addressed separately in the First Carbon Nanotube, Biology, Medicine and Toxicology Satellite Symposium. The discussion on metrology of carbon nanotubes will continue a satellite conference attached to the Tenth International Conference on Science and Application of Carbon Nanotubes, which will take place in Beijing in 2009. Contributions are welcome for the development of this important research field.

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