INTRODUCTION

Single-walled carbon nanotubes (SWNTs) are the most promising of all nanomaterials, with unique electronic and mechanical properties which lend themselves to a variety of applications, such as field-emission displays, nanostructured composite materials, nanoscale sensors, and elements of new nanoscale logic circuits.[1-5] In all cases the quality of the SWNT material is important, and for some applications it is paramount. Despite sustained efforts, all currently known SWNT synthetic techniques, generate significant quantities of impurities, such as amorphous and graphitic forms of carbon and carbon encapsulated catalytic metal nanoparticles. The presence of such impurities necessitates the application of vigorous purification procedures, which adds to the high cost of production and limits the application of these materials.

Improvements in SWNT quality by optimization of the synthesis, as well as subsequent purification steps is a multi-parametric task which requires the accurate and efficient evaluation of small changes in the purity and yield of bulk quantities of the SWNT product as a function of the synthetic parameters and purification protocol. Ideally, such a procedure should provide a rapid, convenient, and unambiguous measure of the bulk purity of standard SWNT samples with instrumentation that is routinely available in research laboratories. For customers and end users of SWNTs such an evaluation procedure is essential for the development of quality assurance measures which allow a comparison of commercial materials with customer determined specifications.

While the reader may believe that this is a settled issue because many suppliers label their SWNT products with percentage purities, evaluation of such samples shows that in many cases the purity is overstated - some to a high degree.[6] Progress in chemistry and material science has been marked by the ability to obtain pure substances: the isolation of the pure elements and the refinements that have occurred in electronics grade silicon are obvious examples. While it may not be possible to obtain analytically pure SWNTs, it is high time for the

SWNT industry to make a serious attempt to divulge to its customers the quality of the products that are offered for commercial sale.

In this Practice Guide we present a number of analytical techniques which are relevant to the evaluation of the purity of SWNT materials. There are two major forms of impurities in current SWNT preparations: the metal catalyst residue and the carbonaceous component which usually takes the form of amorphous carbon and nanographitic structures. Most of the analytical techniques discussed in this Guide are suited to the detection of only one of those fractions, and the choice of an analytical technique is critical.[7] In special cases, such as the comprehensive characterization of the highest purity reference materials for standards development, the application of a number of analytical techniques is required.[8]

In Section 2 we discuss Thermo-Gravimetric Analysis (TGA) which is the most efficient tool for determining the metallic impurities in SWNT material. In certain special cases the TGA technique is also capable of providing information on the carbonaceous impurities as a result of differences in combustion temperatures.

The evaluation of the purity of the carbonaceous component of SWNT material is a long-standing problem, which has traditionally been addressed at a local scale by SEM and TEM techniques.[7]. Recently, NIR spectroscopy has been advanced as an efficient tool to measure the carbonaceous purity of bulk SWNT samples and is discussed in Section 3. The NIR technique takes advantage of the unique spectroscopic features resulting from the interband electronic transitions in SWNTs and it is most efficient for SWNT samples with a narrow distribution of diameters; it is distinguished from other methods because it is quantitative and readily applicable to bulk samples. The power of this method is already established by published studies that document the improvements in synthetic parameters and purification protocols that may be achieved by its application.

Chapter 4 discusses the application of resonantly enhanced Raman spectroscopy for SWNT purity evaluation. The two most prominent features observed in the first-order resonant Raman spectrum of SWNTs are the low frequency radial breathing mode (RBM) and the high frequency G-band; the D-band associated with disordered carbon provides a measure of the contribution of carbonaceous impurities. A comparison of the intensities of the RBM- or G-bands to the intensity and shape of the D-band provides information on the SWNT content of the sample. Due to the resonant nature of the SWNT Raman response, multiple excitation wavelengths must be utilized in order to allow for the contributions of SWNTs of different diameters.

Chapter 5 discusses the application of optical, electron and scanning probe microscopy (SPM) for SWNT quality evaluation. TEM and SEM have traditionally been the most important techniques for the characterization of carbon nanotubes. Indeed, SWNTs were discovered in 1993 using TEM, and high-resolution TEM remains a major analytical tool for the study of SWNT nucleation and growth. The SEM technique has been extensively used to monitor the efficiency of the bulk scale production of SWNTs by a variety of synthetic techniques, and it has traditionally been the most popular tool to evaluate the quality of as-prepared SWNT soot.[7] Because a typical SEM frame visualizes less than 10^{-12} g of an inhomogeneous sample for which there is no published algorithm with which to quantify the SWNT content, the use of these techniques to assess the purity of bulk SWNT samples is not recommended.

References

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