Thermogravimetric Analysis of Single-Wall Carbon Nanotubes

An improved protocol yields greater consistency.

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An improved protocol for thermogravimetric analysis (TGA) of samples of single-wall carbon nanotube (SWCNT) material has been developed to increase the degree of consistency among results so that meaningful comparisons can be made among different samples. This improved TGA protocol is suitable for incorporation into the protocol for characterization of carbon nanotube material as described in the preceding article.

TGA has been used extensively to characterize carbon nanotube materials during the past decade. In most cases, TGA of carbon nanotube materials is performed in gas mixtures that contain oxygen at various concentrations. The data acquired in TGA following this approach provide information on ash content and on oxidation temperature (which is usually described as the temperature of the maximum rate of loss of weight). In many cases, rates of loss of weight have several maxima at temperatures ranging from 300 to 800 °C. These peaks have been attributed to various components in the nanotube material, including amorphous carbon, nanotubes, and graphitic particles.

Metal particles are always present in nanotube materials because the metals are used as catalysts in the production of the nanotubes. The position of each oxidation peak is strongly affected by the amounts and microstructures of the metal particles because these particles also catalyze oxidation of all carbon forms present in the nanotube material.

The ash content is used to determine the amount of metal catalyst in the material. It is usually assumed that upon completion of TGA, all carbon has been removed in the forms of CO and CO_2 and that all remaining material consists of metal oxides.

It has been observed that different TGA results can be obtained from the same nanotube material on different TGA apparatuses and even in different runs on the same apparatus. These inconsistencies can be attributed to the use of a wide variety of sample-preparation practices, instruments, protocols, heating rates, and carrier gases and to inhomogeneities within nanotube batches. In the improved TGA protocol, knowledge gained in a study of the effects of TGA experimental parameters on TGA results is applied to reduce the inconsistencies substantially. In all of the experiments in the study, air was used as the gas mixture and the flow rate was 100 standard cubic centimeters per minute.

The improved protocol is summarized as follows:

1. Use a heating rate of 5 °C/min up to the maximum temperature. The exact value of maximum temperature can be varied; 800 °C accommodates most samples.
2. The mass of the sample should be between 2 and 4 mg.
3. Three separate TGA runs should be performed on each sample.
4. In addition to the TGA weight measurement, an independent weight measurement should be performed on a microbalance, after TGA, to increase the precision of determination of the ash content.
5. Results from the three runs should be processed to obtain the mean values of oxidation temperature(s) and ash content.
6. Results from the three runs should also be processed to obtain standard deviations of the oxidation temperature(s) and ash content. These standard deviations are representative of inhomogeneity in the sample.

This work was done by Sivaram Arepalli, Pavel Nikolaev, and Olga Gorelik of GB Tech Inc. for Johnson Space Center. Further information is contained in a TSP (see page 1). MSC-23507-1.