Encapsulation of Fullerenes and Alkali Metals Inside Carbon Nanotubes Using Plasma Technology

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An original approach using plasma technology has been performed in order to develop fullerene-based materials with new functions. The fullerene feeding into an alkali-metal plasma results in the generation of the plasma of $A^+ - C_{60}^-$ due to electron attachment (A = Li, Na, K, Cs). Purified single-walled carbon nanotubes (SWNTs) were dispersed and deposited on substrates, which were immersed in this alkali-fullerene plasma.

A field emission gun transmission electron microscopy (FE-TEM), energy dispersive X-ray spectrometry, and Raman scattering spectroscopy are adapted for the characterization of the carbon nanotubes.

When DC bias voltages (ϕ_{ap}) are applied to the substrate with respect to the grounded plasma-source electrode, positively- and negatively-charged particles are substantially accelerated by plasma sheaths in front of the substrate for $\phi_{ap} < 0$ and $\phi_{ap} > 0$, respectively. This impact of the accelerated charged particles in the alkali-metal or alkali-fullerene plasmas eventually provokes structural deformation of SWNTs, such as tube bending or tube cutting.¹

Figure 1(a) shows a FE-TEM image of fullerene encapsulated SWNTs obtained by applying a positive bias $(\varphi_{ap} > 0)$ to the substrate in the A⁺-C₆₀⁻ plasma. We can clearly see the isolated SWNTs containing a selfassembled or set of fullerene molecules.² Considering the diameter difference between the C₆₀ (~ 0.71 nm) and six-membered ring (~ 0.25 nm) of the nanotube, C₆₀⁻ ions can be inserted inside the SWNT from its cut region when an open end is not plugged during the plasma-ion irradiation. In the case of the negative-bias ($\varphi_{ap} < 0$) application in the Cs⁺-C₆₀⁻ plasma, on the other hand, a Cs row inside the SWNT is observed as shown in Fig. 1(b). The spiral shape of Cs chain is very similar to the result of a recent report describing an atomic arrangement of iodine atoms.³

Finally, when we sequentially apply biases with polarity change between $\phi_{ap} = -100V$ and 20V in the $Cs^+ - C_{60}^-$ plasma, surprisingly in Fig. 1(c), the SWNT is found to encapsulate a junction of Cs atoms on one side and C_{60} molecules on the other.

More works, such as establishment of optimal intercalation conditions and more precise evaluations, are now progressing. This result, however, gives a confidence of high feasibility for the realization of new functional materials.

¹ R. Hatakeyama *et al.*, *The 20th Fullerene General Symposium*, 2001, **2A30P**.

² G. -H. Jeong *et al.*, Appl. Phys. Lett., 2001, **79**, 4213.

³ X. Fan *et al.*, Phys. Rev. Lett., 2000, **84**, 4621.



Fig. 1. (a) C_{60} (b) Cs, and (c) C_{60} / Cs encapsulated SWNTs.