Graphite Electrodes Containing Nanometer-Sized Metal Particles and Their Use in the Synthesis of Single-Walled Carbon Nanotube Composites

Alan M. Cassell, Walter A. Scrivens, and James M. Tour*

Department of Chemistry and Biochemistry, University of South Carolina, Columbia, South Carolina 29208

Received March 20, 1996⊗

Composite graphite/metal(0) electrodes were prepared by the absorbtion of organic solutions of metal carbonyls or aqueous solutions of metal salts into high-purity porous graphite rods. The metal carbonyls were converted to the CO-free metal(0) species by heating the composites under an atmosphere of N_2 and then under reduced pressure at 1000 °C. The metal-salt-containing graphite rods were heated under an atmosphere of H_2 at 1000 °C to reduce the salts to the corresponding metal(0) species. This procedure permitted dispersion of metal(0) throughout the graphite rod with average metal particle sizes in the range 6.2−11.6 nm by transmission electron microscopy (TEM) analysis. Metals used were Ag, Co, Cu, Fe, La, Ni, and Pt. The composite graphite/metal(0) electrodes were vaporized in a plasma discharge apparatus under a helium atmosphere. The Co, Fe, Ni, and Pt all catalyzed single-walled nanotubule (bucky tube) growth. The soot material from the Co, Fe, and Ni-containing rods had a foam-rubber-like texture. All the metal-containing soots, except for the La-derived material, could be press molded into pellets, without the use of a binder. Data from the trace remaining ligand analyses, powder X-ray diffraction, TEM, conductivity measurements, and surface area analyses are presented. The properties of the arc-derived soots prepared by this method are compared to the soots prepared by standard cored rod/metal(0) methods.

New carbon structures from the arc vaporization of graphite and composite graphite electrodes include fullerenes,[1] multilayered nanotubes,[2] single-layered nanotubes,[3] endohedral fullerenes,[4] and nanoparticles.[5] Interest exhibited in these materials results, in part, from the numerous potential applications in areas such as electrochemistry,[6] nanotechnology,[7] and catalysis.[8] There are various methods known for introducing metals into a carbon arc including packing of a cored graphite rod with a powdered mixture of metal(0) or metal oxide,[3] use of a metal block placed inside a graphite crucible as the anode with a tungsten cathode,[9] and use of a composite rod that is pressed and heated from a mixture of graphite, metal oxide, and a graphite cement binder.[10] A solution-based preparation of graphite electrode/metal(0) composites is described here. The method permits the use of a wide variety of metals and the formation of well-dispersed nanometer-sized metal(0) in the electrodes. Little or no exogenous ligands, oxides, or binders are present that can interfere or inhibit subsequent electrode use. Moreover, this technique avoids the use of large amounts of metal(0) that often extrude from the rod during discharge preparation of nanotubes by the cored-rod method.

Composite graphite/metal(0) electrodes were prepared by the absorbtion of organic solutions of metal carbonyls or aqueous solutions of metal salts into high-purity porous graphite rods. The metal carbonyls were converted to the CO-free metal(0) species by heating the composites under an atmosphere of N_2 and then under reduced pressure at 1000 °C. The metal-salt-containing

---

⊗ Abstract published in *Advance ACS Abstracts,* May 1, 1996.

(1) (a) Krätschmer, W.; Lamb, L. D.; Fostiropoulos, K.; Huffman, D. R. *Nature* **1990**, *347*, 354. (b) Constable, E. C. *Angew. Chem., Int. Ed. Engl.* **1994**, *33*, 2269.

(2) (a) Iijima, S. *Nature* **1991**, *354*, 56. (b) Iijima, S. *Mater. Res. Soc. Bull.* **1994**, *19* (11), 43. (c) Ebbesen, T. W.; Ajayan, P. M. *Nature* **1992**, *358*, 220. (d) Ruoff, R. S. *Nature* **1994**, *372*, 731.

(3) (a) Bethune, D. S.; Kiang, C. H.; de Vries, M. S.; Gorman, G.; Savoy, R.; Vazquez, J.; Beyers, R. *Nature* **1993**, *363*, 605. (b) Seraphin, S.; Zhou, D. *Appl. Phys. Lett.* **1994**, *64*, 2087. (c) Lin, X.; Wang, X. K.; Dravid, V. P.; Chang, R. P. H.; Ketterson, J. B. *Appl. Phys. Lett.* **1994**, *64*, 181. (d) Ajayan, P. M.; Lambert, J. M.; Bernier, P.; Barbedette, L.; Colliex, C.; Planeix, J. M. *Chem. Phys. Lett.* **1993**, *215*, 509. (e) Iijima, S.; Ichihashi, T. *Nature* **1993**, *363*, 603. (f) Saito, Y. *Recent Advances in the Chemistry and Physics of Fullerenes and Related Materials*; Kadish, K. M., Ruoff, R. S., Eds.; Electrochemical Society: Pennington, NJ, 1994; p 1419. (g) Seraphin, S. Ibid., p 1433. (h) Guo, T.; Nikolaev, P.; Thess, A.; Colbert, D. T.; Smalley, R. E. *Chem. Phys. Lett.* **1995**, *243*, 49.

(4) (a) Bethune, D. S.; Johnson, R. D.; Salem, J. R.; de Vries, M. S.; Yannoni, C. S. *Nature* **1993**, *366*, 123. (b) Edelmann, F. T. *Angew. Chem., Int. Ed. Engl.* **1995**, *34*, 981.

(5) (a) Diggs, B.; Zhou, A.; Silva, C.; Kirkpatrick, S.; Nuhfer, N. T.; McHenry, M. E.; Petasis, D.; Majetich, S. A.; Brunett, B.; Artman, J. O.; Staley, S. W. *J. Appl. Phys.* **1994**, *75*, 5879. (b) Brunsman, E. M.; Sutton, R.; Bortz, E.; Kirkpatrick, S.; Midelfort, K.; Williams, J.; Smith, P.; McHenry, M. E.; Majetich, S. A.; Artman, J. O.; De Graef, M.; Staley, S. W. *J. Appl. Phys.* **1994**, *75*, 5882. (c) Rao, C. N. R.; Seshardi, R. *Mater. Res. Soc. Bull.* **1994**, *19* (11), 28. (d) Ruoff, R. S.; Lorents, D. C.; Chan, B.; Malhotra, R.; Subramoney, S. *Science* **1993**, *259*, 346.

(6) (a) Dohzono, M.; Katsuki, H.; Egashira, M. *J. Electrochem. Soc.* **1989**, *136*, 1255. (b) Matsumura, Y.; Wang, S.; Kasuh, T.; Maeda, T. *Synth. Met.* **1995**, *71*, 1755. (c) Yun, M.-S.; Moon, S.-I.; Doh, C.-H.; Lee, K.-H. *Synth. Met.* **1995**, *71*, 1761.

(7) (a) Ajayan, P. M.; Stephan, O.; Colliex, C.; Trauth, D. *Science* **1994**, *265*, 1212. (b) Rinzler, A. G.; Hafner, J. H.; Nikolaev, P.; Lou, L.; Kim, S. G.; Tománek, D.; Nordlander, P.; Colbert, D. T.; Smalley, R. E. *Science* **1995**, *269*, 1550.

(8) Planeix, J. M.; Coustel, N.; Coq, B.; Brotons, V.; Kumbhar, P. S.; Dutartre, R.; Geneste, P.; Bernier, P.; Ajayan, P. M. *J. Am. Chem. Soc.* **1994**, *116*, 7935.

(9) Dravid, V. P.; Host, J. J.; Teng, M. H.; Elliott, B.; Hwang, J.; Johnson, D. L.; Mason, T. O.; Weertman, J. R. *Nature* **1995**, *374*, 602.

(10) (a) Chai, Y.; Guo, T.; Jin, C.; Haufler, R. E.; Chibante, L. P. F.; Fure, J.; Wang, L.; Alford, J. M.; Smalley, R. E. *J. Phys. Chem.* **1991**, *95*, 7564. (b) Wang, Y. *J. Am. Chem. Soc.* **1994**, *116*, 397.

S0897-4756(96)00199-8 CCC: $12.00

Table 1. Starting Metal Complex, Composite Electrode Properties, and Soot Properties

metal complex	trace elemental composition of remaining ligand in graphite/ metal(0) electrodes[a]	metal(0) particle size in graphite/ metal(0) electrodes by XRD (nm)[c]	metal(0) particle size in graphite/ metal(0) electrodes by TEM, average, range, (nm)[e]	single-walled nanotubes observed in soot? (size in nm)[g]	metal(0) particle size in soots by XRD[c] (nm)	metal(0) particle size in soots by TEM, average, range, (nm)[e]	conductivity of soot (Ω^{-1} cm^{-1})[h]
$Co_2(CO)_8$	*b*	13	7.9, 1.4−24.1	yes (1.0−1.4)	10	7.5, 0.6−21.3	1.4−30
$Ag(NO)_3$	N = 338 ppm	41	7.6, 1.8−24.4	no	29	11.5, 1.3−58.3	4.4−7.9
$Fe(NO_3)_3{\cdot}9H_2O$	N = 415 ppm	36	11.6, 1.7−31.0	yes (1.0−1.4)	*d*	3.4, 0.7−7.8	0.02
$CoCl_2{\cdot}6H_2O$	Cl = 88 ppm	*d*	8.9, 1.1−58.3	yes (1.0−1.4)	*d*	8.9, 1.2−32.9	4.0−6.7
$CuCl_2{\cdot}2H_2O$	Cl = 1.36%	39	8.8, 1.7−37.7	no	*d*	9.0, 1.1−35.9	0.9−1.2
$LaCl_3{\cdot}7H_2O$	Cl = 2.74%	*d*	*f*	no	*d*	*f*	*i*
$NiCl_2{\cdot}6H_2O$	Cl = 456 ppm	33	6.2, 1.9−23.3	yes (1.4−1.6)	*d*	4.3, 1.5−14.9	3.6−4.9
$PtCl_4$	Cl = 0.46%	36	11.1, 2.2−35.0	yes (1.6−1.8)	*d*	5.9, 2.2−25.5	2.8−2.9

[a] Composition after annealing. [b] The C, H, and Co content was 83.21, <0.5, and 16.83%, respectively. [c] Average particle size was determined by powder XRD from the most intense Bragg peaks (θ_B); the Debye−Scherrer analysis method was used to determine the average particle size (t) where $t = 0.9\ \lambda/\cos\theta_B\ \Delta\theta_B$, λ = the X-ray wavelength (1.54 Å for Cu Kα), and $\Delta\theta_B$ = the peak width at half-height. Since smaller particles contribute less to the X-ray pattern due to the short repeat distances,[11] corroboration with the transmission electron microscopy (TEM) analysis is essential. The TEM sample preparation process would not exclude the larger particles. [d] No metal-derived diffractions were observed. [e] A representative 50-particle sample area was used for the determination. [f] Unable to image by TEM. [g] This represents the presence of single-walled nanotubes within the soot of the reaction flask. We never observed multiwalled tubes except on the electrode slag. [h] Conductivities measured on pressed pellets by a four-point probe. [i] Unable to form a free-standing pellet.

graphite rods were heated under an atmosphere of H_2 at 1000 °C to reduce the salts to the corresponding metal(0) species. The metal salt procedure has advantages over the metal carbonyl procedure in that it requires fewer impregnations, inert atmosphere conditions are unnecessary, and the procedure is more general.

Table 1 summarizes the graphite/metal composites that have been prepared. The trace elemental analyses indicate that both the carbonyl (oxygen content) and nitrates (nitrogen content) can be efficiently removed during the annealing/reduction process. For reasons that are presently unclear to us, chloride can be effectively removed in only the Co and Ni cases; Cu, La, and Pt residual chloride contents remained somewhat higher. Hence, for some applications, use of the corresponding Cu, La, and Pt nitrates may be preferred. Therefore, this solution-based impregnation method provides a superb route for the formation of graphite/metal(0) composites where the metal is nanometer-sized and well-dispersed throughout the graphite matrix.

The efficacy of the process for tubule formation is also outlined in Table 1. With all metals tested, multiwalled tube structures could be seen by transmission electron microscopy (TEM) on the slag generated on the unconsumed (cathodic) rod; however, no multiwalled structures were found in any of the soots. In the cases of Co, Fe, Ni, and Pt, single-walled tubes filled the reaction flask. In the case of the Co reaction, the toluene-soluble fullerene content was 5.5% C_{60} and 3.3% C_{70}. The vast majority of the material (~70%) was the metal-containing tubule material with pyrolitic carbon sheaths. Scanning electron micrographs (SEM) of the cobalt-containing soot, produced at an arc gap of 2−4 mm, showed a weblike material that filled the reaction flask.[3] Closer examination by TEM revealed single-walled nanotubes with 1.0−1.4 nm diameters and lengths of >1 μm encased in pyrolitic carbon sheaths. In addition, small metal particles in the 2−6 nm range buttressing the tubes were present (Figure 1). The larger arc gaps (2−4 mm) often resulted in this buttressing interaction[3d] of metal particles and tubules. By contrast, cobalt-containing soot produced at an arc gap of <1 mm showed bundles of single-walled nanotubes with much less pyrolitic carbon deposits (Figure 2). Figure 3 shows bundles of single-walled nanotubes that appear to be emanating in two directions from a Pt particle, while Figure 4 shows Ni-derived naked single-walled tubules that were peeled from the reactor wall.

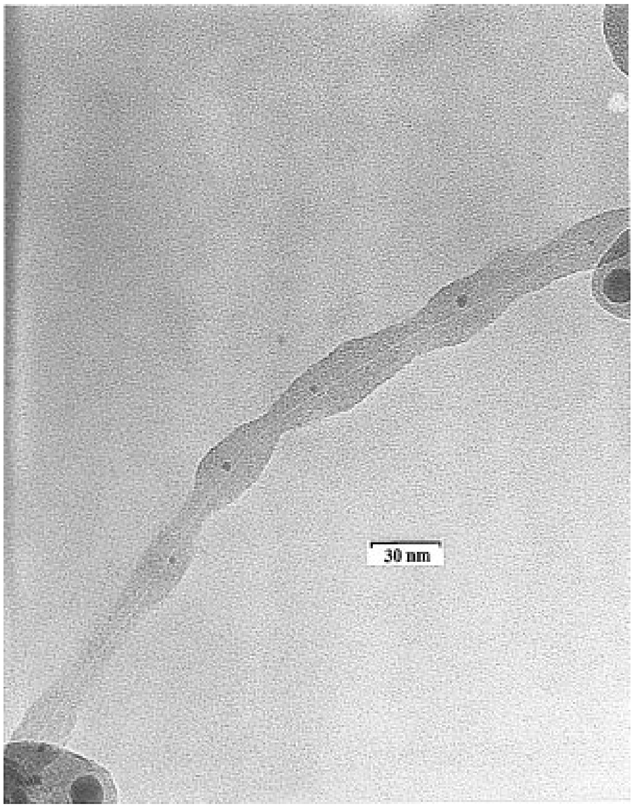

Figure 1. TEM image of the Co-containing soot prepared at an arc gap of 2−4 mm showing single-walled nanotubes with 1.0−1.4 nm diameters with pyrolitic carbon sheaths and small metal particles in the 2−6 nm range buttressing the tubes.

Soots produced from Co, and to a lesser degree by Fe and Ni, had foam-rubber-like mechanical properties. All the soots, except for the La-derived material, could be press molded into pellets without the use of an exogenous binder. The cobalt-containing soot was unique; when removed from the reactor walls, it remained in one large piece.[3] Bulk conductivity measurements (four-point probe) on pressed pellets are listed in Table 1. For

---

(11) Cullity, B. D. *Elements of X-Ray Diffraction*; Addison-Wesley: London, 1959.

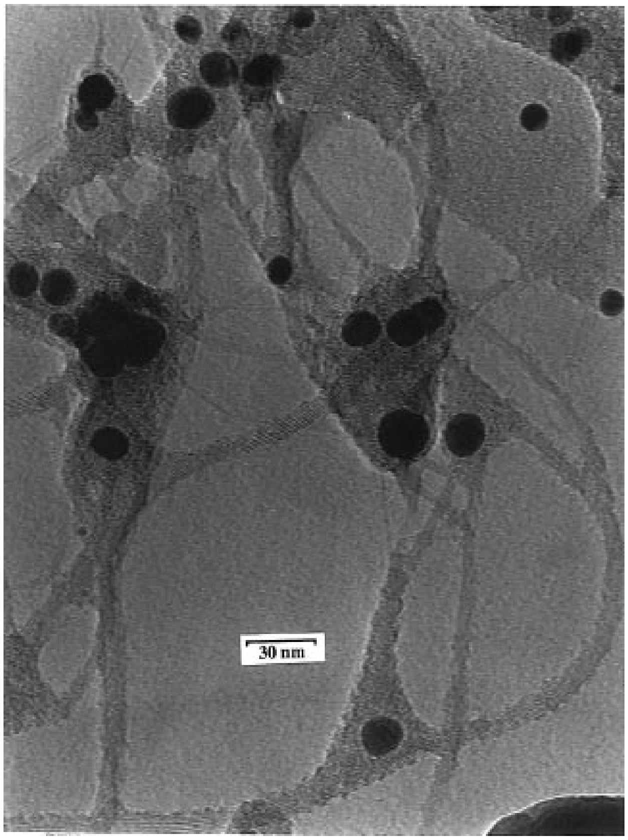

Figure 2. TEM image of Co-containing soot produced at an arc gap of <1 mm showing bundles of single-walled nanotubes.

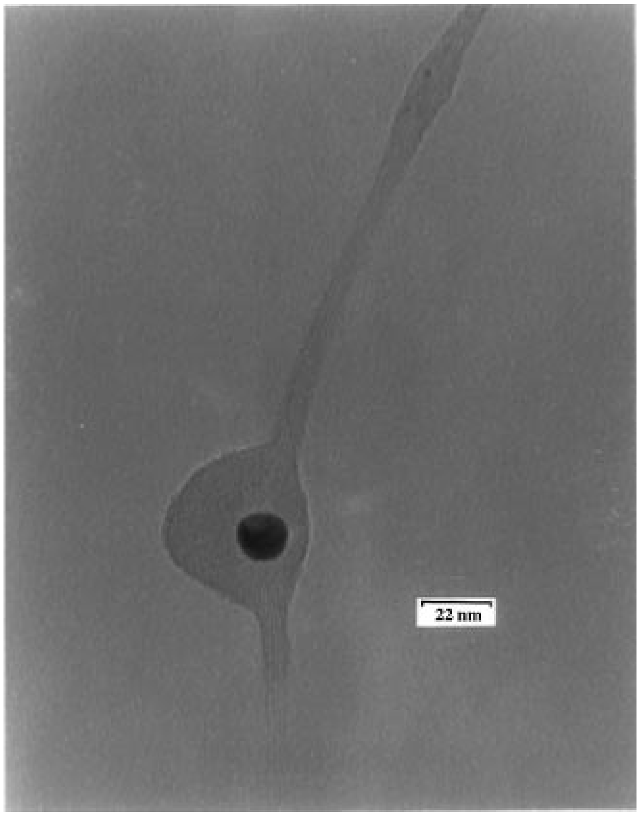

Figure 3. TEM image of a Pt particle from which bundles of single-walled nanotubes appear to have grown.

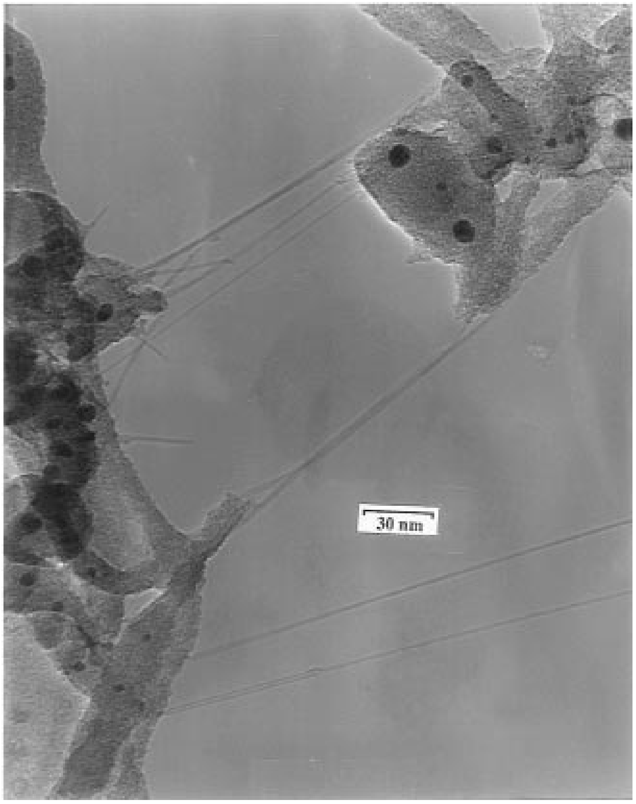

Figure 4. TEM image of nickel-containing soot prepared at an arc gap between 2 and 4 mm. This sample had peeled from the reactor wall and the naked single-walled tubes were evident.

the Co-derived sample, a higher conductivity of 150 Ω^{-1} cm^{-1} was obtained for samples pressed from an unground sheet of the film. This result suggests conduction is greater through the plane of the film than between layers of the film. The material exhibited a typical semiconductive behavior; the resistivity increased with a decline in temperature (studied down to 75 K). Though the conduction mechanism is speculative, I_2 doping[12] of the Co soots prior to pellet formation caused a 10-fold increase in the conductivity. This may be due to an increase in the amount of charge carriers.[12a] The collaret (ring around the slag),[3d] when ground and pressed into a pellet, had an I_2-doped conductivity of 450 Ω^{-1} cm^{-1} for the Co-derived material. Surface area measurements (BET) on both the Co-containing films and pressed pellets were $\sim$165 m^2 g^{-1}, indicating a fairly high surface area. The pressed pellet had a density of 1.41 g cm^{-3}.

The soot materials derived from the solution-based cobalt nanoparticle impregnated rods were compared to the soots obtained from the more common cobalt metal/cored rod (17.8 wt % Co, prepared according to ref 3a). Both rods were arc-consumed under identical electrical conditions. The cored-rod derived soot[3a] provided Co particles averaging 22 nm in size with the range 5.7–84 nm. Therefore, the metal particles from the standard cored-rod method were larger in magnitude and distribution than those obtained from the solution deposition method described here. Additionally, far more cathodic slag deposits formed during arcing of the cobalt metal/cored-rod sample as opposed to the solution-based cobalt nanoparticle impregnated rods. A conductivity of only 0.15 Ω^{-1} cm^{-1} was obtained on the soot-derived pellet from the cobalt metal/cored-rod sample. Thus, the

---

(12) (a) *Handbook of Conducting Polymers*; Skotheim, T. J., Ed.; Dekker: New York, 1986. (b) Klimenko, I. V.; Shchegolikhin, A. N.; Zhuravleva, T. S. *Synth. Met.* **1995**, *71*, 1773.

Table 2

starting metal salt (g, mmol)	water used for solution (mL)	graphite rod starting weight (g)	graphite/ salt rod weight after drying (g)	graphite/M(0) rod weight after reduction	weight % metal	atomic % metal
$Ag(NO)_3$ (10.00 g, 58.9 mmol)	15	1.333	1.772	1.546	13.8	1.75
$Fe(NO_3)_3{\cdot}9H_2O$ (10.00 g, 24.8 mmol)	10	1.042	1.114	1.090	4.59	0.98
$CuCl_2{\cdot}2H_2O$ (7.01 g, 41.1 mmol)	10	1.285	1.715	1.413	9.06	1.85
$LaCl_3{\cdot}7H_2O$ (10.09 g, 27.2 mmol)	10	1.476	1.781	1.646	10.30	0.99
$NiCl_2{\cdot}6H_2O$ (15.00 g, 63.1 mmol)	15	1.054	1.210	1.101	4.27	0.90
$PtCl_4$ (1.00 g, 2.97 mmol)	7	1.125	1.434	1.235	8.91	0.60

solution-based deposition method described here provided unique conductivity properties to the soots. Finally, there was a severe problem with metal(0) extrusion and graphite binder expulsion during the arcing process when using the cored-rod samples. The solution-derived metal-containing rods showed none of those difficulties.

In summary, a method to disperse nanometer-sized metal(0) throughout graphite electrodes was developed. Their uses for nanotube growth and generation of processable conductive composites were demonstrated, while other uses in electrochemistry and catalysis are envisioned.

Experimental Procedures

General Techniques. PS1 grade, porous graphite rods, 40% porosity/total volume, $^3/_{16}$ in. × 6 in., were purchased from Poco Graphite, Inc., Decatur, TX.[13] $Co_2(CO)_8$ was purchased from Strem Chemicals. $CoCl_2{\cdot}6H_2O$ and $CuCl_2{\cdot}2H_2O$ were purchased from Mallinckrodt Inc. $Ag(NO)_3$ was purchased from Goldsmith Inc. $NiCl_2{\cdot}6H_2O$ and $PtCl_4$ were purchased from Aldrich Chemical Co. $Fe(NO_3)_3{\cdot}9H_2O$, $LaCl_3{\cdot}7H_2O$, and cobalt metal were purchased from Fisher Scientific. The metal salts were used without purification. High-purity graphite was purchased from Union Carbide Corp. During the impregnation of the metal carbonyls, all operations were performed under a dry, oxygen-free, N_2 atmosphere. Samples for SEM analysis were prepared by sonicating a small amount of soot in methanol for 0.5 h, after which 10 μL of the resulting suspension was placed on a small piece of glass that was mounted on an aluminum sample disk with graphite adhesive (Electron Microscopy Sciences). The samples were coated with gold, then loaded into a Hitachi S-2500Δ scanning electron microscope. All images were obtained at 25 keV accelerating voltage. Samples for TEM analysis were sonicated in water for 0.5 h, and 10 μL of the suspension was placed onto carbon-film coated, copper TEM grids. The sample grids were dried overnight at 60 °C and 1 mmHg and then placed in a Hitachi H-8000 transmission electron microscope. All images were performed at 200 keV accelerating voltage. Pellets for electrical analysis were prepared by pressing 150 mg samples of the soots into a 13 mm die (Wilmad Glass Co., Inc.) that contained high-gloss weighing paper (Baxter S/P B2040-1) inserts on both faces of the die. The soots were pressed first at 5000 psi (5 min), then 10 000 psi (5 min) and finally at 15 000 psi (10 min). The pellet was removed, the paper inserts were peeled from the pellet with a razor blade, and the four-point probe conductivity measurements were performed. The four-point probe measurements[12a] were performed using an Alessi CPS-05 contact probe station with a Keithly 224 programmable current source and a Keithly 169 digital multimeter.

Impregnation of $Co_2(CO)_8$ into Porous Graphite. A porous graphite rod (3.712 g, 309.0 mmol, $^3/_{16}$ in. × 6 in.) was placed in a 280 mm × 25 mm Pyrex reaction tube with a 24/40 joint at the open end that was then septum-capped. A pentane solution of $Co_2(CO)_8$ (1 mL, 0.058 M, purified by passage through a plug of silica gel) was added to the tube. Rotating the tube horizontally allowed the porous rod to absorb the solution along its length. The excess pentane was evaporated by a stream of N_2, and the glass tube was then placed in a 100 °C oil bath and heated for 1 h under a constant N_2 stream to facilitate the loss of the CO. The entire process was repeated, and each iteration gave an approximately 0.03 g mass gain of cobalt. The final total mass of the cobalt-impregnated rod was 4.323 g after 20 iterations. The cobalt-impregnated graphite rod was placed in a quartz tube and heated to 1000 °C for 4 h under vacuum (<1 mmHg) to minimize the residual solvent or CO ligand content, affording a final rod mass of 4.253 g (12.7 wt % Co). $Fe(CO)_5$, $Cr(CO)_6$, and $Mo(CO)_6$ partially sublimed out of the rod during the annealing process; therefore, for these metals, the salt process must be used for impregnation. The cobalt complex may be optimal in the series because it decomposes (52 °C) immediately after melting (51 °C), and its decomposition temperature is 60−100 °C lower than that of the other three carbonyl complexes.

Impregnation of $CoCl_2{\cdot}6H_2O$ into Porous Graphite. $CoCl_2{\cdot}6H_2O$ (10.00 g, 42.0 mmol) and water (20 mL) were added to a 50 mL round-bottomed flask that contained a magnetic stir bar. The solution was heated to 95 °C and stirred for 30 min. The solution (∼7 mL) was placed in a 16 mm × 100 mm reaction tube that contained a magnetic stir bar, and the solution was reheated to 95 °C with stirring. A porous graphite rod (1.890 g, 157.4 mmol, $^3/_{16}$ in. × 3 in.) was then introduced into the reaction tube and allowed to soak in the solution for 12 h. As the water in the solution evaporated during the soaking process, more water was added to maintain a level that covered the entire graphite rod. After the soaking was complete, the rod was removed and placed in an empty 16 mm × 100 mm reaction tube. The tube was evacuated (<1 mmHg) while gently heating the tube with a Bunsen burner flame to remove water present in the rod. After one soaking and drying, the porous rod gained 0.375 g, giving the rod a mass of 2.265 g. (The number of soakings can be increased to achieve an increased metal loading level.) The $CoCl_2$-impregnated graphite rod was placed in a quartz tube, and the tube was evacuated (<1 mmHg) for 1 h and then purged with forming gas (5% H_2, 95% Ar) for 10 min. While under a slow stream of forming gas, the tube was placed in a furnace, gradually heated at 2 °C min^{-1} to 1000 °C and held at that temperature for 12 h. The final rod mass was 1.974 g (4.25 wt % Co).

Impregnation of other Metal Salts into Porous Graphite. The procedure described above was used with the other metal salts. The quantities used and weights recorded are shown in Table 2.

Plasma Discharge of the Metal-Impregnated Graphite Electrodes.[2,3] The metal-impregnated graphite rods, prepared from both the metal carbonyls and metal salts, were placed in the anodic end of a dc plasma discharge reactor[14] and arc-consumed to near completion to form gram quantities of composite soot materials. The arc was ignited at 27 V and a dc current of 55 A. Higher currents gave a less stable arc. Helium pressure was maintained at 450 mmHg with a differential flow of helium through the reactor of 3−5 mm (measured by the oil U-tube).[14] The rate of helium flow across the arc will undoubtedly affect the tubule formation; much like we previously observed for soluble fullerene formation.[14] The method of measuring the flow across the arc is unique to this apparatus (the details were described previously),[14] and all

---

(13) Scrivens, W. A.; Tour, J. M.; Creek, K. E.; Pirisi, L. *J. Am. Chem. Soc.* **1994**, *116*, 4517.

(14) Scrivens, W. A.; Tour, J. M. *J. Org. Chem.* **1992**, *57*, 6932.

attempts to put flow meters in-line affected the flows, thus limiting our ability to quantitate the flow for nonsimilar apparatus use. The problem with flow recording occurs because there is a split manifold with a positive helium pressure on the inlet side and a vacuum being drawn on the outlet side. In addition, the arc gap between the electrodes was typically maintained between 2 and 4 mm; however, we later discovered that soots produced at arc gaps of less than 1 mm afforded more single-walled nanotubes.

Preparation of Cored Graphite Rod/Co(0).[4a] A nonporous high-purity graphite rod (3 in. × $^{3}/_{16}$ in., 1.734 g, 144.4 mmol) that was cored (0.118 in. × 2 in.), was packed with a mixture of cobalt metal and high purity graphite in a 54:46 wt % ratio, respectively. A total of 0.667 g of cobalt/graphite mixture was packed into the rod. The rod was then arc-consumed under the same conditions used for the solution-based metal-impregnated porous graphite rods.

Acknowledgment. We are grateful for support from the Office of Naval Research, the National Science Foundation (DMR-9158315), and the Department of Energy by grant DE-FCO2-91ER75666 ammendment number A004. We are indebted to Professor S. A. Majetich for numerous suggestions and Professor T. Datta for the low-temperature conductivity measurement.

CM9601998