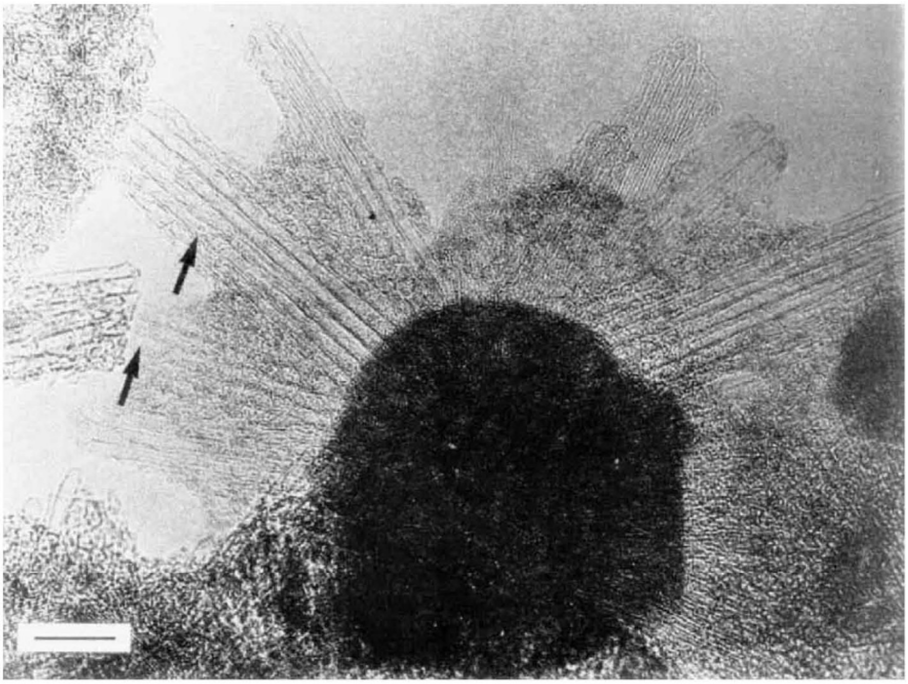
Radial single-layer nanotubes

SIR — Several novel forms of carbon have been discovered since the mid-1980s, including fullerenes[1], nanometre-sized nested graphitic tubes and polyhedra[2], and single-layer carbon nanotubes[3,4]. These single-layer tubes have so far been observed only in the primary soots from arc-discharge experiments where the anodes were packed with the ferromagnetic elements Fe (ref. 3), Co (ref. 4) and Ni (which was observed by our group). Here we report the discovery of clusters of single-layer tubes arranged around nuclei of $\mathrm{Gd}_x\mathrm{C}_y$ in patterns similar to sea-urchin spines. They are present in large quantities in the primary soots from arc-discharge experiments using either $\mathrm{Gd_2O_3}$ or pure metal Gd in the anode.



High-resolution transmission electron microscope image of bundles of short predominantly single-layer carbon tubes encapsulating an amorphous core of $\mathrm{Gd}_x\mathrm{C}_y$. Scale bar, 10 nm. Arrows, possible multi-wall tubes. The microscope is a Phillips CM20 ultra-high resolution TEM.

The soot samples described here were prepared in a manner similar to that described earlier[5]. The graphite anode was drilled out and packed with either $\mathrm{Gd_2O_3}$ or Gd such that the Gd/C ratio was between 3 and 6 atom %. The cathode is a 12.7-mm diameter graphite rod, and the anode a 8-mm diameter graphite rod with a 3.2-mm bore drilled out in the centre. The arc gap was typically 3–6 mm, and the current and pressure were 85 amp and 500 torr ($\mathrm{Gd_2O_3}$) or 75 amp and 1,000 torr (pure Gd). The primary soot obtained was dispersed ultrasonically in ethanol and placed on holey carbon-coated Cu grids for transmission electron microscopy.

The soot morphology can be broadly classified into two types — pure amorphous carbon particles; and amorphous carbon regions containing substantial quantities of high electron contrast metal nuclei. Energy dispersive spectra confirm that these nuclei are Gd compounds. Careful observation of the regions containing the $\mathrm{Gd}_x\mathrm{C}_y$ particles shows the prevalence of a 'sea-urchin' type morphology, with the $\mathrm{Gd}_x\mathrm{C}_y$ particles forming the nuclei of these radial structures.

The figure shows a high resolution image of one of the 'sea urchin' particles, clearly illustrating the bundles of rather short (<75 nm) single-layer tubes radiating out from a $\mathrm{Gd}_x\mathrm{C}_y$ core, which appears to be amorphous. Some of the bundles also appear to contain what look like multi-layered tubes, as indicated by arrows in the figure. We have also observed similar structures (not shown) around single crystal nuclei, which are partially encapsulated with turbostratic carbon layers. Analysis of the *d*-spacings of the crystalline nuclei prove that the single crystal cores are α-$\mathrm{GdC_2}$. Note that the bundles of single layer tubes are not present on those facets where layers of turbostratic carbon cover the α-$\mathrm{GdC_2}$ crystals (data not shown).

Observation of 'hybrid' encapsulates, which have $\mathrm{GdC_2}$ single-crystal cores and are partially coated with single-walled nanotubes, and in other regions by incomplete polyhedral shells, suggests that the thermal history of the encapsulate may determine its final form. Complete encapsulation by (nested) polyhedral shells has never been observed by us in primary soot samples, but rather only in the 'boule' growth on the cathode, such as for α-$\mathrm{LaC_2}$ (ref 5) and α-$\mathrm{GdC_2}$ (ref 6). The 'sea urchin' encapsulates reported here demonstrate that dramatically different morphologies can result even though the encapsulate is the same (in this case, single crystal α-$\mathrm{GdC_2}$). This opens the possibility of studying the encapsulates which are formed in the primary soot.

We have also observed that it is possible to significantly enhance the fraction of these novel types of carbon coated encapsulates in the primary soots by oxidizing the soot to about 50% (carbon) mass loss, which shows that the bundles of single-layer tubes are substantially more resistant to oxidation than amorphous carbon.

We have prepared various primary soots produced with other rare earth oxides. We find that Nd is the only other candidate producing the structure described here, and that La, Ce, Pr, Sm, Sc, and Er do not. Further experiments are needed to understand these differences.

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Waxy tea

SIR — Major components of the scum on tea[1] are high-melting-point lipids, epidermal waxes which are standard waterproofing equipment on leaves of land plants such as *Camellia* (*Thea*). Boiling water takes them off, and they float to the surface in tea cups, where they solidify as the surface cools. After that, disturbances (such as insertion of a spoon) crack the thin surface layer into floes, easily visible by obliquely reflected light. As the tea is drunk, the waxy layers stick to the sides of the cup. They stain with tea phenolics, which go brown on oxidation. Milk components, casein and butter-fat, thicken them. One can liquefy, saponify and solubilize them with very hot water and detergent. (I do this every day.)

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