

the genomic instability and subsequent disease symptoms caused by both Bloom's and Werner's mutations may be due to a loss of a function other than the helicase activity, just as is the case for *SGS1*.

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## Purifying single-walled nanotubes

**SIR** — Single-walled carbon nanotubes<sup>1–3</sup> hold great promise for both fundamental understanding and potential applications of carbon materials<sup>4,5</sup>. Recently, they have been successfully synthesized in large quantities<sup>6</sup> using composite consumable carbon electrodes mixed with metallic species, such as Fe/Ni and Co/Ni. The single-walled nanotubes synthesized by arc discharge<sup>1–3</sup> are found to coexist with metals and with various other forms of carbon, and such impurities are a serious impediment to detailed characterization of the properties of the nanotubes. Various purification methods proposed for multi-walled nanotubes have been applied to the single-walled versions<sup>7</sup>, but so far without clear success. We present here a new purification method for single-walled nanotubes that incorporates a hydrothermal treatment<sup>8</sup> for easy removal of metal particles and carbonaceous materials.

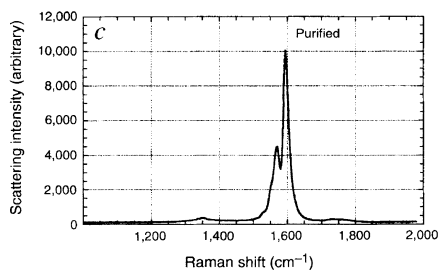
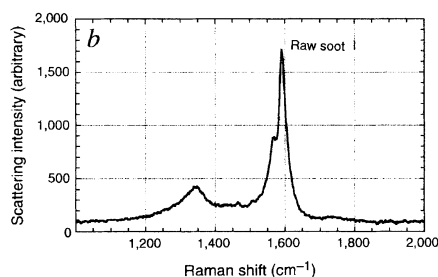
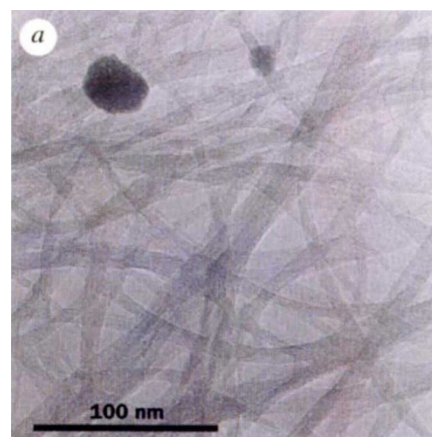
We used the method proposed by Seraphin and Zhou<sup>6</sup> to produce single-walled nanotubes. The mixing ratio of Ni, Fe and graphite powder was 1:1:3 by weight. The total metal content in the anode is approximately 6 at.%. The arc discharge was carried out under a He pressure of 100 torr. The discharge current was 70 A, and the gap between the

electrodes was maintained at 1 mm by manually advancing the consumed anode.

We retrieved soot produced by d.c. arc discharge from the upper wall and roof of an arc-discharge chamber and homogenized it. Single-walled nanotubes coexist with many by-products in the raw soot, such as metal particles, fullerenes and amorphous carbon. The HIDE treatment<sup>8</sup> is the initial step of the purification process. We introduced 100 mg of the soot containing the nanotubes into a flask with reflux attachment, sonicated it in 50 ml of distilled water and heated it at 373 K for 12 h. The soot disintegrated to sub-micrometre-sized particles. We filtered out the processed soot and dried it at 333 K for 12 h. Then we washed out fullerenes using toluene Soxhlet extraction. We heated the residual soot at 743 K for 20 min in air to burn out the amorphous carbon. In the final step of the purification process, we washed out almost all metal complexes from the soot using 6 M hydrochloric acid. We obtained at least 20 mg of single-walled nanotubes (with a purity of 95 wt%) per g of raw soot. One gram of raw soot contains roughly 50 mg of unpurified single-walled nanotubes. The purified nanotubes (*a* in the figure) are almost totally free of impurities and are separated in large quantities.

We then qualitatively analysed bulk samples of purified single-walled nanotubes for residual amorphous carbon using Raman scattering. The nanotubes show multiple-split Raman peaks of crystalline graphite centred at 1,580 cm<sup>-1</sup> because of their cylindrical symmetry, whereas amorphous or fine graphitic particles exhibit broad peaks around 1,350 and 1,580 cm<sup>-1</sup>. Our Raman spectrum of raw soot, showing a very broad peak near 1,350 cm<sup>-1</sup> together with weak structures between 1,400 and 1,500 cm<sup>-1</sup> of fullerene, is shown in *b* in the figure. The Raman profile matches the spectrum reported by Holden *et al.*<sup>9</sup> very well. Purified single-walled nanotubes are shown in *c*, with a spectrum around 1,580 cm<sup>-1</sup> showing multiple-split peaks (1,570 and 1,590 cm<sup>-1</sup>) that are five times more intense than in soot, without any change in their positions and intensity ratios, and clear shoulders (1,530 and 1,550 cm<sup>-1</sup>). These results suggest that we have successfully purified undamaged, single-walled nanotubes, and that only trace amounts of other carbonaceous materials remain in our purified sample.

To confirm the role of the HIDE treatment in the purification process, we processed the nanotubes without hydrothermal treatment. In this case, large amounts of graphite and metal particles are associated with them. Further, the surfaces of the bundles are covered with amorphous carbon. This confirms that without hydrothermal treatment ultrafine graphite particles, nanocapsules and amorphous carbon adsorbed on the single-



*a*, Transmission electron micrograph of purified single-walled nanotubes. *b*, *c*, Raman spectra of raw soot and purified single-walled nanotubes, respectively.

walled nanotubes are not removed from the soot. This may be the reason why the baking technique is not successful. We believe that, during the HIDE treatment, water molecules break the network between single-walled nanotubes, amorphous carbon and metal particles, and also attack the graphite layer encapsulating the metal particles. Consequently, almost all graphite nanoparticles and nanocapsules are washed out from the soot.

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