France. It was stated in the same editorial that these authors have also obtained statistically significant results from Sardinia, but this is not true. In effect, the result remains to be replicated in independent case control and family studies, as is the case for most genetic associations in type-2 diabetes published so far.

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Controlled-size nanocapsules

SIR — Encapsulation of second phases inside graphite shells/nanotubes[1–7] is of considerable significance not only because it offers an opportunity to investigate dimensionally confined systems, but also because the encapsulated species are likely to be immune to environmental effects or degradation owing to the protective graphite sheets around them[8]. It has been difficult to encapsulate ferromagnetic transition metals using the traditional graphite arc process, however, because of poor and irreproducible yield, no particle size control and the unavoidable presence of unwanted carbides, graphite flakes, nanotubes and particulates.

Here we report a novel, large-scale synthesis method to encapsulate controlled-size ferromagnetic transition metals, such as Fe, Co and Ni, inside protective graphite shells. The experimental set-up consists of an arc chamber with a vertical tungsten cathode (about 3.2 mm diameter), and an anode composed of a metal block placed inside a graphite crucible with an inner diameter of about 38 mm. A transverse helium gas jet passes through the arc plasma and provides quenching of the metal vapour. The metal block (such as nickel) is usually thermally isolated from the graphite crucible by a thin *c*-axis-oriented graphite foil. Under arc evaporation conditions, the metal block forms a molten pool and the spatial distribution of the arc plasma is wide enough to engulf part of the surrounding graphite crucible and foil, both of which provide just enough supply of carbon for subsequent graphitic encapsulation.

A well-defined log-normal size distribution with mean particle size ranging from 7 to 14 nm has been obtained by controlling the helium jet velocity between 56 and 20 $m\,s^{-1}$ (ref. 9). The resultant product consists mainly of nanocrystalline metal particles, graphite-encapsulated nanocrystalline metal with a minority of amorphous carbonaceous debris. It is important to note that the synthesis product in our method is virtually devoid of any graphitic flakes, nanotubes or coarse nanoparticles, which commonly occur in the traditional graphite–graphite arc process and are very difficult to eliminate.

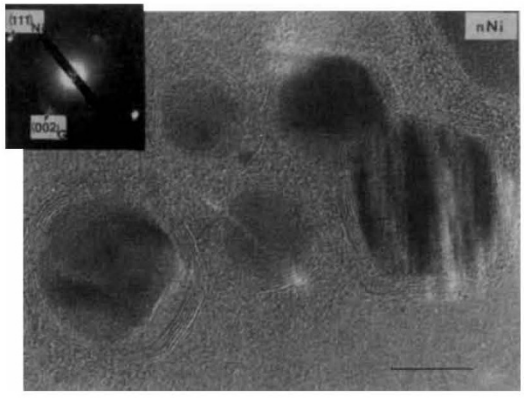

FIG. 1 High-resolution electron microscope image of graphite-encapsulated nanocrystalline nickel particles. Inset, nanodiffraction pattern indicating graphite (002) and nickel (111) reflections. Scale bar, 10 nm.

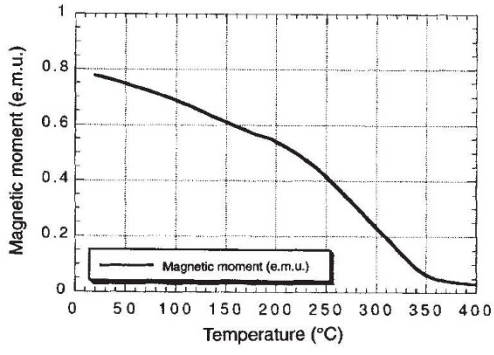

FIG. 2 Magnetization versus temperature plot of graphite-encapsulated nanocrystalline nickel. The Curie temperature determined from the plot (~358 °C) is consistent with that of metallic nickel.

The condensed product is collected and washed (under exposure to ultrasound) in a strong solvent (such as HCl or aqua regia: a 3:1 mixture of HCl and HNO_3), which dissolves unprotected metallic particles, leaving behind a large amount (about 20–40%, but sometimes as large as 70%) of residue containing graphite-encapsulated nanocrystals. The acid-washed residue consists of graphite-encapsulated nanocrystals and amorphous carbonaceous debris, with no trace of graphite nanotubes or graphite nanoparticles. The minor amount of carbonaceous debris present may be oxidized and removed using a method similar to that reported by Ebbesen *et al.*[10] for the purification of carbon nanotubes. The final yield depends on the type of metal species and critically on some operating variables such as the geometry of the graphite crucible–metal anode assembly (for example, inner diameter of the graphite crucible).

Figure 1 shows a dispersed ensemble of nanocrystalline nickel particles completely encapsulated with graphite. X-ray microanalysis, electron nanodiffraction and electron energy loss spectroscopy fine-structure analysis confirmed that the encapsulated product is metallic nickel and that the coating is graphite. The intimate and contiguous graphite fringes around the nanocrystal particles is evidence for complete encapsulation by graphite shells.

Measurements of the magnetic properties of the encapsulated nanocrystalline nickel indicate that the intrinsic properties of ferromagnetic nickel are retained even with encapsulation in graphite shells. Saturation magnetization, Curie temperature, and magnetization versus field loops are consistent with those of nanocrystalline nickel (which also shows superparamagnetic behaviour below about 4–6 nm particle size). Figure 2 shows a plot of magnetization against temperature for graphite-encapsulated nickel. The Curie temperature is 358 °C, with an error of about 10 °C.

Our results demonstrate successful encapsulation of controlled-size ferromagnetic nanocrystalline metals which retain their intrinsic nanocrystal properties. Graphite encapsulation makes the nanocrystalline metal particles immune to environmental degradation: they are insoluble and undamaged even when exposed to aqua regia. Some encapsulated nanocrystals were kept in aqua regia for months, but did not dissolve, and the protective graphite coating remained intact.

The approach presented here not only results in a much higher yield of controlled-size encapsulated species, but also the occurrence of carbides and carbonaceous debris (for example, graphite flakes, buckytubes and their particulate derivatives), which is very difficult to eliminate in subsequent processes, is minimized. The synthesis approach reported here may be extended to other materials that catalyse nucleation of the graphite form of carbon on their surfaces.

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