Rice Refining Production of Pure Nanotube Fibers

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Rice University scientists are refining pioneering chemical production methods used to make pure carbon nanotube fibers. Research appearing in tomorrow's issue of the journal Science describes the scalable production techniques, which yield highly aligned, continuous macroscopic fibers composed solely of single-walled carbon nanotubes (SWNTs), the type of carbon nanotubes with the best mechanical and transport properties.

Chemist Richard Smalley, director of Rice's Carbon Nanotechnology Laboratory (CNL), said the production methods CNL is pioneering for single-walled carbon nanotube fibers are similar to those used in making two of the world's strongest commercially available fibers, Kevlar and Zylon. CNL's fiber research team expects the development path of pure nanotube fibers to follow a similar track to those two as well, with several years of refinement in processing and a significant investment needed for research prior to commercial availability.

The Air Force and its industrial partners spent a decade and more than $100 million perfecting Zylon, the strongest fiber on the market today. Zylon is about twice as strong as Kevlar, the material used to make much of the world's bulletproof body armor. Ultimately, CNL researchers believe pure nanotube fibers hold the promise of being 10 times stronger than Zylon.

"The early results are auspicious," said Smalley, University Professor, the Gene and Norman Hackerman Professor of Chemistry and professor of physics at Rice. "We've got no impurities, our densities are about 77 percent of what's theoretically possible and we're confident that the strength and conductance will improve as we refine the heat treatment, spinning and other elements of production."

In 2000, a team of researchers centered at Rice in Smalley's research group began in earnest to spin a fiber from SWNTs dispersed at high concentration in a strong acid. Ongoing work at that time showed that SWNTs interact strongly with sulfuric acid and assemble into endless spaghetti-like domains composed of myriad highly aligned, mobile SWNTs. This Science article is the fifth paper reporting the four-year journey that resulted in the current discovery. Researchers at the University of Pennsylvania's Department of Materials Science and Engineering helped determine the structure of the nanotube acid dispersion.

"The SWNT fiber project is one of the 'holy grails' of nanotechnology -- spin a pure single-walled nanotube fiber with the highest strength of any fiber possible," said paper co-author Wade Adams, director of Rice's Center for Nanoscale Science and Technology (CNST).

Adams said Rice's fiber project was one of the factors that enticed him to join CNST in 2002 following his retirement after 32 years of service at the Air Force Research Laboratory at Wright-Patterson Air Force Base in Dayton, Ohio.

" The Air Force developed what is now called Zylon fiber from a rod-like polymer, with tensile properties twice that of Kevlar fiber," said Adams. "SWNTs are predicted to have tensile strengths many times that of Zylon or Kevlar, based upon the much greater theoretical strength of the single molecule carbon nanotube. However, unlike Zylon and Kevlar, SWNTs are also excellent conductors of electricity and heat. This unique multifunctionality makes them candidates for many critical applications beyond structural ones."

Nanotubes are hollow cylinders of pure carbon that are just one atom thick. In addition to being very strong, nanotubes can also be either metals or semiconductors, which means they could be used to manufacture materials that are both "smart" and ultra-strong. NASA, for example, is researching how composite materials containing nanotubes could be used to build lighter, stronger aircraft and spacecraft.

Chemically, carbon nanotubes are difficult to work with. They are strongly attracted to one another and tend to stick together in hairball-like clumps. Scientists have developed ways to untangle and sort small amounts of nanotubes but have not found a satisfactory way to achieve stable dispersions suitable for processing. To date, the medium of choice has been detergent and water solutions that contain less than 1 percent of dispersed nanotubes by volume and are processed with polymer solutions. Such concentrations are too low to support industrial processes aimed at making large nanotube fibers. Moreover, it is difficult to remove all the soap and polymer and convert the nanotubes back into their pure form.

Rice's team believes they have overcome the major hurdle to industrial production of macroscale SWNT objects -- finding a way to store large amounts of nanotubes in liquid form. By using strong sulfuric acid, a team of chemists and chemical engineers was able to disperse up to 10 percent by weight of pure carbon nanotubes -- more than 10 times the highest concentrations previously achieved. This new processing route uses no polymeric additives or detergents, which were used in previous processing methods and are known to be an obstacle to commercial scalability and final product purity.

The research paper is titled "Macroscopic, Neat, Single-walled Carbon Nanotube Fibers." Smalley's and Adams' co-authors at Rice were Robert

H. Hauge, distinguished faculty fellow; Matteo Pasquali, assistant professor of chemical engineering; W.E. Billups, professor of chemistry; Howard Schmidt, CNL executive director; research scientists Wen-Fang Hwang, Carter Kittrell, Sivarajan Ramesh and Rajesh K. Saini; postdoctoral research associate Haiqing Peng; graduate students Richard Booker, Virginia A. Davis, Lars M. Ericson, Myung Jong Kim, A. Nicholas G. Parra-Vasquez, Hua Fan and Yuhuang Wang; former undergraduate student Joseph Sulpizio; and Gerry Lavin, visiting scientist from Carbon Consultations. Co-authors at the University of Pennsylvania were John E. Fischer, professor of materials science and engineering, and graduate students Csaba Guthy, Juraj Vavro and Wei Zhou.

Source: Rice University

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