DDF annual report

Title of Investigation:

Low-cost carbon nanotubes (CNTs) from coal

Principal Investigator:

Dr. Jeannette Benavides (Code 562)

Other In-house Members of Team:

Darryl Mitchell (Code 504) and Harry Shaw (Code 562)

Other External Collaborators:

Rodney Travis (NAVSEAS)

Initiation Year:

FY 2005

Funding Authorized for FY 2005:

$20,000

Actual or Expected Expenditure of FY 2005 Funding:

$13,750 to Loomis Products Co. for (rods); $3,250 to NAC Carbon (bake coal); $2500 to Jet Pulverizer Co. (pulverize coal); $3,100 to Bruker Optics (service to Raman Spectroscope); $2400; Fisher (22-200C oven), $1500; National Electric Co. (graphite cathodes), $1,000

Status of Investigation at End of FY 2005:

To be continued

Expected Completion Date:

January 2006

Purpose of Investigation:

In the future, large quantities of carbon nanotubes will be needed for different applications. Because the price of making pure carbon nanotubes using other processes is about $300 per gram, the purpose of this investigation was to determine whether carbon nanotubes (CNTs) could be produced from carbon rods made from coal using a GSFC-patented process. If the rods could be made directly from coal, the cost of making carbon nanotubes would decrease, although they would be less pure. If this process worked, the technology could result in a new industry in Appalachia, where coal and electricity are cheap and where new industries are needed.

Accomplishments to Date:

The carbon rods that we used in the past were made using a proprietary process that we were unable to obtain. By thoroughly studying the properties of coal and understanding the changes that occur when it is baked at different temperatures, however, we were able to come up with a method to make the carbon rods. The University of Kentucky (UKY) advised us and recommended that we use cornstarch, as opposed to tar pitch, as the binder because it was safer and less messy to use. For this investigation, we also used a special kind of cornstarch recommended by UKY. The manufacturer gave us 1 kg for free.

In April 2005, we collected nine coal samples from several mines in Appalachia and one from Pennsylvania.

The first step was to pulverize the coal. We have a ball mill in the laboratory and thought that it would work. It did not; so we contacted Jet Pulverizer Co., which agreed to pulverize the coal, first grinding it coarsely.

The second step before pulverizing was to bake the coal to 350°C under nitrogen gas to eliminate the volatiles. We sent samples to the NAC Carbon Co. in June. Due to a series of delays, the work was not completed until December. In the beginning, we planned to have 10 different samples from which we could make 20 rods; however, because of the delays, we combined the Appalachian samples into two and left the one from Pennsylvania alone. (The Appalachian coal is bituminous and fairly homogeneous. The coal from Pennsylvania also is fairly homogeneous, but it is anthracite.)

Meanwhile, we began studying the possibility of using coke (already-baked and pulverized coal) and carbon black to make rods that we could use for comparison. We also asked the Loomis Products Co. (responsible for extruding the rods from the pulverized coal) to try different amounts of cornstarch and water. We obtained free samples of coke and carbon black from U.S. Steel and the Carbon Black Co., respectively.

NAC Carbon sent the samples to the Jet Pulverizer Co., which then sent the samples to the Loomis Products Co. after pulverizing them. The Loomis Products Co. also experienced delays. First, the company president (to whom I had spoken extensively and in detail about the project) left the company and I was not informed until a month later. I already had sent the practice samples and assumed that everything was on schedule. The company did not start until the end of November and then discovered that the extrusion press they used provided very little pres-

sure (about 4000 psi). The samples would not extrude. The company ordered another machine (14,000 psi) and assured me that work would begin shortly.

The method for making the Goddard-designed rods is:

1. Grind coal to about 1 centimeter in diameter using a hammer.
2. Bake at 350°C for five hours under nitrogen.
3. Pulverize the coal to 5-µm particle size.
4. Add 2 percent cornstarch and 10 percent water.
5. Extrude into 1/8-inch, 12-inch long rods.
6. Bake at 1,000°C for 5 hours under nitrogen.

Once I get the rods, I will make the CNTs—three from coal, one from carbon black, and one from coke. I do not need to make large quantities of each. I only need enough to determine by Raman Spectroscopy (in house) and scanning electron microscope whether the rods worked. I hope to finish everything by the end of January 2006 and make an addendum to this report.

A scientific paper by Jeannette Benavides, et al., will be published soon. The results of the DDF will add additional information. In addition, we will publish the results separately in *Tech Briefs* if the project is successful.

Summary:

The importance of having a known process to make low-cost carbon rods from coal to produce CNTs is well documented. It is very important to learn if the process works with the different carbon rods. Raman spectroscopy will answer these questions.

If it produces suitable rods, this method is innovative and will benefit NASA by lowering the cost of the CNTs and giving NASA control over the source material. In addition, a private industry that will mass produce CNTs using our process needs to know that the source material is inexpensive, easy to make, reproducible, and available.