Materials

Optimizing a Laser Process for Making Carbon Nanotubes

Trends in process parameters for optimization and scale-up have been identified.

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A systematic experimental study has been performed to determine the effects of each of the operating conditions in a double-pulse laser ablation process that is used to produce single-wall carbon nanotubes (SWCNTs). The comprehensive data compiled in this study have been analyzed to recommend conditions for optimizing the process and scaling up the process for mass production.

The double-pulse laser ablation process for making SWCNTs was developed by Rice University researchers. Of all currently known nanotube-synthesizing processes (arc and chemical vapor deposition), this process yields the greatest proportion of SWCNTs in the product material. In the normal version of this process, one uses a green (wavelength 532 nm) laser pulse followed by an infrared (wavelength 1,064 nm) laser pulse within a few nanoseconds to ablate a metal-containing graphite target located in a flow of argon at 100 standard cubic centimeters per minute (sccm) at a pressure of 500 torr (≈66.7 kPa) in a flow tube that is maintained in an oven at a temperature of 1,473 K. The aforementioned process conditions are important for optimizing the production of SWCNTs and scaling up production. Reports of previous research (mostly at Rice University) toward optimization of process conditions mention effects of oven temperature and briefly mention effects of flow conditions, but no systematic, comprehensive study of the effects of process conditions was done prior to the study described here.

This was a parametric study, in which several production runs were carried out, changing one operating condition for each run. The study involved variation of a total of nine parameters: the sequence of the laser pulses, pulse-separation time, laser pulse energy density, buffer gas (helium or nitrogen instead of argon), oven temperature, pressure, flow speed, inner diameter of the flow tube, and flow-tube material. The same graphite target was used in all the runs. The nanotube-containing material produced in each run was collected and characterized by a variety of analytical techniques.

The results of the characterizations indicated trends in the effects of process parameters that could be used to optimize the process and increase the efficiency of the production process. Among the conclusions (see table) reached in this study is that SWCNT material of better quality can be produced by use of lower pressure and faster flow, relative to the normal version of the process. This conclusion could be useful in scaling up the process. This limited study could be extended by changing more than one parameter at a time in an effort to identify some of the intricate mutual effects of different process parameters.

This work was done by Sivaram Arepalli, Pavel Nikolaev, and William Holmes of GB Tech Inc. for Johnson Space Center. Further information is contained in a TSP (see page 1). MSC-23508-1

Parameter	Normal Condition	Conclusions
Temperature	1,473 K (1,200 °C)	Lower temperature gives narrower, weaker tubes.
Laser Energy Density	1.5 J/cm^2	Higher energy density produces C60 and narrower tubes that may contain less metal.
Pulse Sequence	Green Before Infrared	Green should be first. Green repeated (no infrared) is better.
Pulse Separation	50 nanoseconds	Slightly longer delay may be helpful.
Buffer Gas	Argon	Don't use helium. Nitrogen is probably acceptable.
Pressure	66.7 kPa (500 torr)	Lower pressure is preferable.
Flow Rate	100 sccm	Higher flow rate is better.
Diameter of Inner Tube	2.5 cm	Narrower is bad.
Inner Tube Material	Quartz	Alumina is better.

Some Conclusions concerning nine process parameters were reached in a parametric study.