



## Materials & Coatings

### Use of Functionalized Carbon Nanotubes for Covalent Attachment of Nanotubes to Silicon

**This method enables the introduction of carbon nanotubes onto all types of silicon-based devices and silicon surfaces.**

*Lyndon B. Johnson Space Center, Houston, Texas*

The purpose of the invention is to covalently attach functionalized carbon nanotubes to silicon. This step allows for the introduction of carbon nanotubes onto all manner of silicon surfaces, and thereby introduction of carbon nanotubes covalently into silicon-based devices, onto silicon particles, and onto silicon surfaces.

Single-walled carbon nanotubes (SWNTs) dispersed as individuals in surfactant were functionalized. The nanotube was first treated with 4-t-butylbenzenediazonium tetrafluoroborate to give increased solubility to the carbon nanotube; the second group attached to the sidewall of the nanotube has a silyl-protected terminal alkyne that is de-protected *in situ*. This gives a soluble carbon nanotube that has functional groups appended to the sidewall that can be attached covalently to silicon. This reaction was monitored by UV/vis/NJR to assure direct covalent functionalization.

Once the reaction to form the appropriately functionalized carbon nanotube was complete, the nanotube solution was passed through a plug of glass wool to

remove particulates. This filtered solution was then flocced by diluting with acetone, and filtered through a Teflon membrane. The collected solid was dispersed in dimethylformamide (DMF) with sonication and filtered once again through a Teflon membrane. The functionalized material was then dispersed in dry DMF and assembled onto silicon by hydrosilation. The assembly was conducted by treating the nanotube solution with a catalytic amount of triphenylcarbonium tetrafluoroborate and submersing a hydrogen-passivated silicon sample in the solution. The assembly mixture was agitated with warming for 12 hours. After that time, the silicon sample was rinsed with organic solvent and dried with a stream of nitrogen. The assembly was characterized by AFM (atomic force microscopy).

The most immediate and obvious use of this procedure is the covalent attachment of carbon nanotubes onto silicon. This method allows for the attachment of individual (not bundles) carbon nanotubes. With this methodology, the highest temperature required to regen-

erate the pristine carbon nanotube is 450 °C.

Although other methods exist to introduce carbon nanotubes into silicon-based devices, this methodology is selective for silicon and allows for the generation of working devices at a much lower temperature.

*This work was done by James M. Tour, Christopher A. Dyke, Francisco Maya, Michael P. Stewart, Bo Chen, and Austen K. Flatt of Rice University for Johnson Space Center. Further information is contained in a TSP (see page 1).*

*In accordance with Public Law 96-517, the contractor has elected to retain title to this invention. Inquiries concerning rights for its commercial use should be addressed to:*

*Krystina Baez, Project Coordinator  
Office of Technology Transfer  
Rice University*

*6100 Main Street*

*Houston, TX 77005*

*Phone No.: (713) 348-6188*

*E-mail: [kbaez@rice.edu](mailto:kbaez@rice.edu)*

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### Flexible Plug Repair for Shuttle Wing Leading Edge

**Thin, flexible plugs conform to surfaces.**

*Lyndon B. Johnson Space Center, Houston, Texas*

In response to the Columbia Accident Investigation Board report, a plug repair kit has been developed to enable astronauts to repair the space shuttle's wing leading edge (WLE) during orbit. The plug repair kit consists of several 17.78-cm-diameter carbon/silicon carbide (C/SiC) cover plates of various curvatures that can be attached to the refractory carbon-carbon WLE panels using a TZM refractory metal attach mechanism. The attach mechanism is inserted

through the damage in the WLE panel and, as it is tightened, the cover plate flexes to conform to the curvature of the WLE panel within 0.050 mm. An astronaut installs the repair during an extravehicular activity (EVA). After installing the plug repair, edge gaps are checked and the perimeter of the repair is sealed using a proprietary material, developed to fill cracks and small holes in the WLE.

In developing the plug repair concept, several issues had to be addressed

including material, design, performance, and operability. An oxyacetylene torch was calibrated to heat a specimen to WLE entry temperatures and was used to screen candidate repair materials. Promising materials were then tested in the Johnson Space Center arc-jet test facility to determine their resistance to oxidation in a hypersonic environment. C/SiC was selected as the cover plate material because of its superior strength and resistance to oxida-