# Materials

## New Material for Surface-Enhanced Raman Spectroscopy

**Reproducible measurements can be made quickly, without preparation of samples.**

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

A chemical method of synthesis and application of coating materials that are especially suitable for surface-enhanced Raman spectroscopy (SERS) has been developed. The purpose of this development is to facilitate the utilization of the inherently high sensitivity of SERS to detect chemicals of interest (analytes) in trace amounts, without need for lengthy sample preparation.

Up to now, the use of SERS has not become routine because the methods available have not been able to reproduce sampling conditions and provide quantitative measurements. In contrast, the coating materials of the present method enable analysis with minimum preparation of samples, and SERS measurements made using these materials are reproducible and reversible. Moreover, unlike in methods investigated in prior efforts to implement SERS, sampling is not restricted to such specific environments as electrolytes or specific solvents.

The coating materials of this method are porous glasses, formed in sol-gel processes, that contain small particles of gold or silver metal. Materials of this type can be applied to the sample-contact surfaces of a variety of sampling and sensing devices, including glass slides, glass vials, fiber-optic probes, and glass tubes. Glass vials with their in-

sides coated according to this method are particularly convenient for SERS to detect trace chemicals in solutions: One simply puts a sample solution containing the analyte(s) into a vial, then puts the vial into a Raman spectrometer for analysis.

The chemical ingredients and the physical conditions of the sol-gel process have been selected so that the porous glass formed incorporates particles of the desired metal with size(s) to match the wavelength(s) of the SERS excitation laser in order to optimize the generation of surface plasmons. The ingredients and processing conditions have further been chosen to tailor the porosity and polarity of the glass to optimize the sample flow and the interaction between the analyte(s) and the plasmon field that generates Raman photons.

The porous silica network of a sol-gel glass creates a unique environment for stabilizing SERS-active metal particles. Relative to other material structures that could be considered for SERS, the porous silica network offers higher specific surface area and thus greater interaction between analyte molecules and metal particles.

Efforts to perform SERS measurements with the help of sampling devices

coated by this method have been successful. In tests, numerous organic and inorganic chemicals were analyzed in several solvents, including water. The results of the tests indicate that the SERS measurements were reproducible within 10 percent and linear over five orders of magnitude. One measure of the limits of detectability of chemicals in these tests was found to be a concentration of 300 parts per billion. Further development may eventually make it possible to realize the full potential sensitivity of SERS for detecting some analytes in quantities as small as a single molecule.

## Treated Carbon Nanofibers for Storing Energy in Aqueous KOH

**Treatment can increase specific capacitance by as much as 400 percent.**

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A surface treatment has been found to enhance the performances of carbon nanofibers as electrode materials for electrochemical capacitors in which aqueous solutions of potassium hydroxide are used as the electrolytes. In the treatment, sulfonic acid groups are attached to edge plane sites on carbon atoms.

The treatment is applicable to a variety of carbon nanofibers, including fib-

rils and both single- and multiple-wall nanotubes. The reason for choosing nanofibers over powders and other forms of carbon is that nanofibers offer greater power features.

In previous research, it was found that the surface treatment of carbon nanofibers increased energy-storage densities in the presence of acid electrolytes. Now, it has been found that the same treatment increases energy-

storage densities of carbon nanofibers in the presence of alkaline electrolytes when the carbon is paired with a NiOOH electrode. This beneficial effect varies depending on the variety of carbon substrate to which it is applied.

It has been conjectured that the sulfonic acid groups, which exist in a deprotonated state in aqueous KOH solutions, undergo reversible electro-