

etch masks to define the 50- $\mu\text{m}$  apertures on a silicon substrate. In the second step, which is part of the previously reported process, the non-masked silicon area between the apertures is subjected to reactive ion etching (RIE) under a special combination of conditions that results in the growth of fluorine-based compounds in randomly distributed formations, known in the art as "polymer RIE grass," that have dimensions of the order of microns.

The polymer RIE grass formations serve as microscopic etch masks during the next step, in which deep reactive ion etching (DRIE) is performed. What remains after DRIE is the carpet of nano-

tips, which are high-aspect-ratio peaks, the tips of which have radii of the order of nanometers. Next, the nanotip array is evaporatively coated with Cr/Au to enhance the absorption of light (more specifically, infrared light in the Sun-sensor application). The photoresist etch masks protecting the apertures are then removed by dipping the substrate into acetone. Finally, for the Sun-sensor application, the back surface of the substrate is coated with a 57-nm-thick layer of Cr for attenuation of sunlight.

*This work was done by Youngsam Bae, Sohrab Mobasser, Harish Manohara, and Choonsup Lee of Caltech for NASA's Jet*

*Propulsion Laboratory. 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:*

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## Nano-Engineered Catalysts for Direct Methanol Fuel Cells

**Small particle sizes and large surface areas can be produced economically and consistently.**

*NASA's Jet Propulsion Laboratory, Pasadena, California*

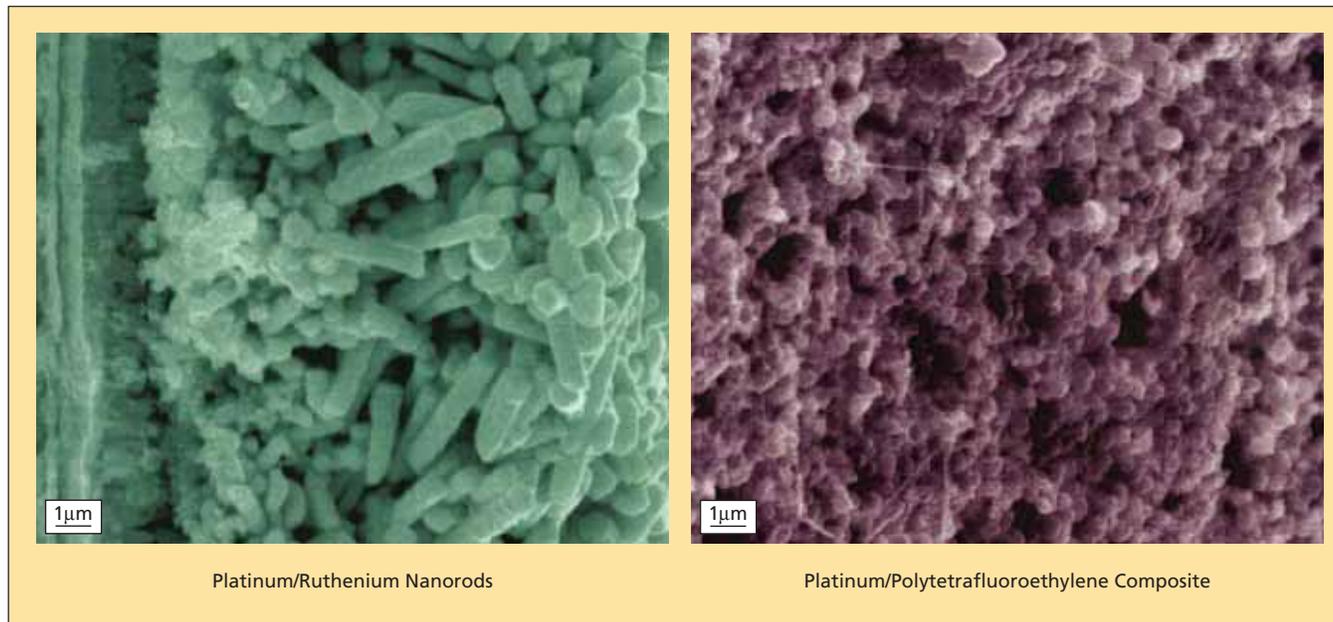
Nano-engineered catalysts, and a method of fabricating them, have been developed in a continuing effort to improve the performances of direct methanol fuel cells as candidate power sources to supplant primary and secondary batteries in a variety of portable electronic products. In order to realize the potential for high energy densities (as much as 1.5 W·h/g) of direct methanol fuel cells, it will be necessary to optimize the chemical compositions and geometric configurations of catalyst layers and electrode structures. High performance

can be achieved when catalyst particles and electrode structures have the necessary small feature sizes (typically of the order of nanometers), large surface areas, optimal metal compositions, high porosity, and hydrophobicity.

The present method involves electrodeposition of one or more catalytic metal(s) or a catalytic-metal/polytetrafluoroethylene nanocomposite on an alumina nanotemplate. The alumina nanotemplate is then dissolved, leaving the desired metal or metal/polytetrafluoroethylene-composite catalyst layer. Unlike some prior methods

of making fine metal catalysts, this method does not involve processing at elevated temperature; all processing can be done at room temperature. In addition, this method involves fewer steps and is more amenable to scaling up for mass production.

Alumina nanotemplates are porous alumina membranes that have been fabricated, variously, by anodizing either pure aluminum or aluminum that has been deposited on silicon by electron-beam evaporation. The diameters of the pores (7 to 300 nm), areal densities of



**These Are Scanning Electron Micrographs** of catalysts fabricated by the method described in the text. The platinum/ruthenium rods, about 0.2  $\mu\text{m}$  wide and 1  $\mu\text{m}$  long, were electrodeposited from a plating bath containing  $\text{H}_2\text{PtCl}_6$ ,  $\text{K}_2\text{RuCl}_5$ , and  $\text{H}_2\text{SO}_4$ . The platinum/polytetrafluoroethylene composite was electrodeposited from a solution containing  $\text{H}_2\text{PtCl}_6$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{CH}_3\text{OH}$ , and suspended polytetrafluoroethylene nanoparticles having sizes from 0.1 to 0.2  $\mu\text{m}$ .

pores (as much as  $7 \times 10^{10} \text{ cm}^{-2}$ ), and lengths of pores (up to about 100 nm) can be tailored by selection of fabrication conditions.

In a given case, the catalytic metal, catalytic metal alloy, or catalytic-metal/polytetrafluoroethylene composite is electrodeposited in the pores of the alumina nanotemplate. The dimensions of the pores, together with the electrodeposition conditions, determine the sizes and surface areas of the catalytic particles. Hence, the small features and large surface areas of the porosity translate to the desired small particle size and large surface area of the catalyst (see figure).

When polytetrafluoroethylene is included, it is for the purpose of imparting hydrophobicity in order to prevent water from impeding the desired diffusion of gases through the catalyst layer. To incorporate polytetrafluoroethylene into a catalytic-metal/polytetrafluoroethylene nanocomposite, one suspends polytetrafluoroethylene nanoparticles in the electrodeposition solution. The polytetrafluoroethylene content can be varied to obtain the desired degree of hydrophobicity and permeability by gas.

*This work was done by Nosang Myung, Sekharipuram Narayanan, and Dean Wiberg of Caltech for NASA's Jet Propulsion Labora-*

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## Capillography of Mats of Nanofibers

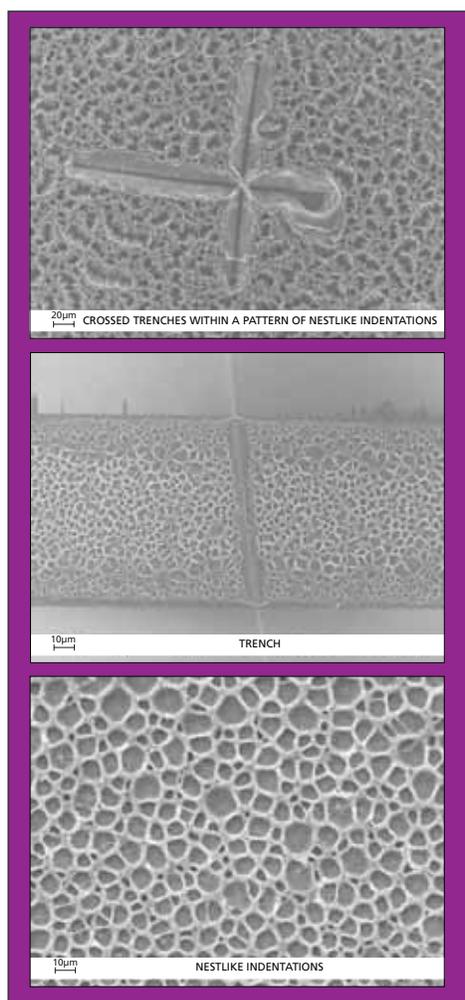
**These mats can be the basis of small devices and instruments.**

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Capillography (from the Latin capillus, "hair", and the Greek graphein, "to write") is a recently conceived technique for forming mats of nanofibers into useful patterns. The concept was inspired by experiments on carpetlike mats of multiwalled carbon nanotubes. Capillography may have the potential to be a less-expensive, less-time-consuming alternative to electron-beam lithography as a means of nanoscale patterning for the fabrication of small devices and instruments.

In capillography, one exploits the lateral capillary forces exerted on small objects that pierce the surface of a liquid. If the small objects are identical, then the forces are always attractive. Two examples of the effects of such forces are the agglomeration of small particles floating on the surface of a pond and the drawing together of hairs of a wet paintbrush upon removal of the brush from water. Because nanoscale objects brought into contact remain stuck together indefinitely due to Van der Waals forces, patterns formed by capillography remain even upon removal of the liquid.

For the experiments on the mats of carbon nanotubes, a surfactant solution capable of wetting carbon nanotubes (which are ultra-hydrophobic) was prepared. The mats were wetted with the solution, then dried. Once the mats were dry, it was found that the nanotubes had become ordered into various patterns, including nestlike in-



These **Scanning Electron Micrographs** show representative patterns formed in mats of multiwalled carbon nanotubes that were wetted with a surfactant solution and then dried.

dentations, trenches, and various combinations thereof (see figure).

It may be possible to exploit such ordering effects through controlled wetting and drying of designated portions of mats of carbon nanotubes (and, perhaps, mats of nanofibers of other materials) to obtain patterns similar to those heretofore formed by use of electron-beam lithography. For making patterns that include nestlike indentations, it has been conjectured that it could be possible to control the nesting processes by use of electrostatic fields. Further research is needed to understand the physics of the patterning processes in order to develop capabilities to control patterns formed in capillography.

*This work was done by Flavio Noca, Elijah Sansom, Jijie Zhou, and Mory Gharib of Caltech for NASA's Jet Propulsion Laboratory. For further information is contained in a TSP (see page 1).*

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