

Pt-Ru Catalytic Powder Is Made from organic salts of Pt and Ru in a sol-gel process that involves fewer steps and less time than does a process based on chloride salts of Pt and Ru.

In this sol-gel process (see figure), the starting materials are platinum(II) acetylacetonate $[Pt(C_5H_7O_2)_2$, also denoted Pt-acac] and ruthenium(III) acetylacetonate $[Ru(C_5H_7O_2)_3, also denoted Ru$ acac]. First, Pt-acac and Ru-acac are dissolved in acetone at the desired concentrations (typically, 0.00338 moles of each salt per 100 mL of acetone) at a temperature of 50 °C. A solution of 25 percent tetramethylammonium hydroxide [(CH₃)₄NOH, also denoted TMAH] in methanol is added to the Pt-acac/Ruacac/acetone solution to act as a highmolecular-weight hydrolyzing agent. The addition of the TMAH counteracts the undesired tendency of Pt-acac and Ru-acac to precipitate as separate phases during the subsequent evaporation of the solvent, thereby helping to yield a desired homogeneous amorphous gel. The solution is stirred for 10 minutes, then the solvent is evaporated until the solution becomes viscous, eventually transforming into a gel. The viscous gel is dried in air at a temperature of 170 °C for about 10 hours. The dried gel is crushed to make a powder that is the immediate precursor of the final catalytic product.

The precursor powder is converted to the final product in a controlled-atmosphere heat treatment. Desirably, the final product is a phase-pure (Pt phase only) Pt-Ru powder with a high specific surface area. The conditions of the controlled-atmosphere heat are critical for obtaining the aforementioned desired properties. A typical heat treatment that yields best results for a catalytic alloy of equimolar amounts of Pt and Ru consists of at least two cycles of heating to a temperature of 300 °C and holding at 300 °C for several hours, all carried out in an atmosphere of 1 percent O2 and 99 percent N₂. The resulting powder consists of crystallites with typical linear dimensions of <10 nm. Tests have shown that the powder is highly effective in catalyzing electro-oxidation of the methanol.

This work was done by Sekharipuram Narayanan and Thomas Valdez of Caltech, and Prashant Kumta and Y. Kim of Carnegie-Mellon University 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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Making Activated Carbon for Storing Gas

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Solid disks of microporous activated carbon, produced by a method that enables optimization of pore structure, have been investigated as means of storing gas (especially hydrogen for use as a fuel) at relatively low pressure through adsorption on pore surfaces. For hydrogen and other gases of practical interest, a narrow distribution of pore sizes <2 nm is preferable. The present method is a variant of a previously patented method of cyclic chemisorption and desorption in which a piece of carbon

is alternately (1) heated to the lower of two elevated temperatures in air or other oxidizing gas, causing the formation of stable carbon/oxygen surface complexes; then (2) heated to the higher of the two elevated temperatures in flowing helium or other inert gas, causing the desorption of the surface complexes in the form of carbon monoxide. In the present method, pore structure is optimized partly by heating to a temperature of 1,100 °C during carbonization. Another aspect of the method exploits the finding that for each gas-storage pressure, gas-storage capacity can be maximized by burning off a specific proportion (typically between 10 and 20 weight percent) of the carbon during the cyclic chemisorption/desorption process.

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