



Corrosion-Prevention Capabilities of a Water-Borne, Silicone-Based, Primerless Coating

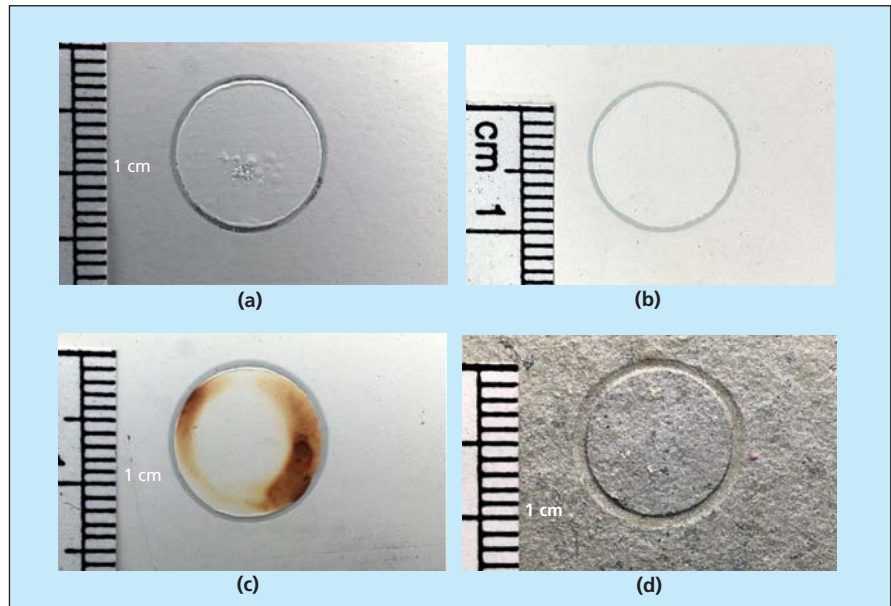
Some formulations are better for steel, some for aluminum.

John F. Kennedy Space Center, Florida

Comparative tests have been performed to evaluate the corrosion-prevention capabilities of an experimental paint of the type described in "Water-Borne, Silicone-Based, Primerless Paints," *NASA Tech Briefs*, Vol. 26, No. 11 (November 2002), page 30. To recapitulate: these paints contain relatively small amounts of volatile organic solvents and were developed as substitutes for traditional anti-corrosion paints that contain large amounts of such solvents. An additional desirable feature of these paints is that they can be applied without need for prior application of primers to ensure adhesion.

The test specimens included panels of cold-rolled steel, stainless steel 316, and aluminum 2024-T3. Some panels of each of these alloys were left bare and some were coated with the experimental water-borne, silicone-based, primerless paint. In addition, some panels of aluminum 2024-T3 and some panels of a fourth alloy (stainless steel 304) were coated with a commercial solvent-borne paint containing aluminum and zinc flakes in a nitrile rubber matrix. In the tests, the specimens were immersed in an aerated 3.5-weight-percent aqueous solution of NaCl for 168 hours. At intervals of 24 hours, the specimens were characterized by electrochemical impedance spectroscopy (EIS) and measurements of corrosion potentials. The specimens were also observed visually.

As indicated by photographs of specimens taken after the 168-hour immersion



Blistering of an Experimental Silicone Paint is manifest on two alloy specimens after immersion for a week in an aerated saltwater solution: (a) silicone-coated aluminum 2024-T3 panel, (b) silicone-coated 316 stainless-steel panel, (c) silicone-coated cold-rolled-steel panel, and (d) aluminum 2024-T3 panel coated with aluminum- and zinc-filled nitrile rubber.

(see figure), the experimental primerless silicone paint was effective in preventing corrosion of stainless steel 316, but failed to protect aluminum 2024-T3 and cold-rolled steel. The degree of failure was greater in the case of the cold-rolled steel. On the basis of visual observations, EIS, and corrosion-potential measurements, it was concluded that the commercial aluminum- and zinc-filled nitrile rubber coating affords

superior corrosion protection to aluminum 2024-T3 and is somewhat less effective in protecting stainless steel 304.

This work was done by Luz Marina Calle and Louis G. MacDowell of Kennedy Space Center, and Rubie D. Vinje of ASRC Aerospace. For further information, contact the Kennedy Innovative Partnerships Office at (321) 867-1463. KSC-12520

Sol-Gel Process for Making Pt-Ru Fuel-Cell Catalysts

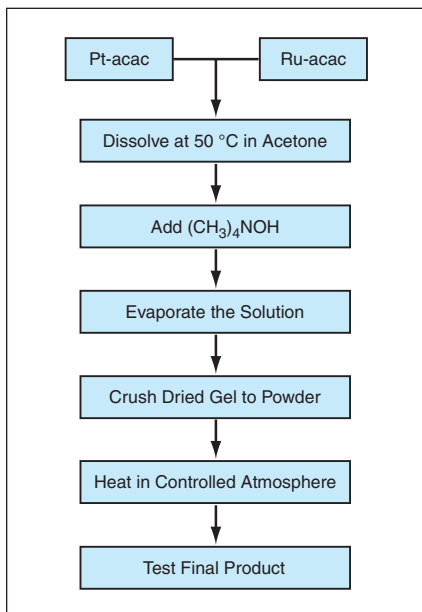
Relative to another process, this one takes less time and yields better results.

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A sol-gel process has been developed as a superior alternative to a prior process for making platinum-ruthenium alloy catalysts for electro-oxidation of methanol in fuel cells. The starting materials in the prior process are chloride salts of platinum and ruthenium. The

process involves multiple steps, is time-consuming, and yields a Pt-Ru product that has relatively low specific surface area and contains some chloride residue. Low specific surface area translates to incomplete utilization of the catalytic activity that might otherwise be available,

while chloride residue further reduces catalytic activity ("poisons" the catalyst). In contrast, the sol-gel process involves fewer steps and less time, does not leave chloride residue, and yields a product of greater specific area and, hence, greater catalytic activity.



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(C₅H₇O₂)₂, also denoted Pt-acac] and ruthenium(III) acetylacetonate [Ru(C₅H₇O₂)₃, also denoted Ru-acac]. First, Pt-acac and Ru-acac are dis-

solved 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/Ru-acac/acetone solution to act as a high-molecular-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 the electro-oxidation of 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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Refer to NPO-30500, volume and number of this NASA Tech Briefs issue, and the page number.

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.

This work was done by Marek A. Wójtowicz and Michael A. Serio of Advanced Fuels Research, Inc., and Eric M. Suuberg (consultant) for Johnson Space Center. For further information, contact the Johnson Innovative Partnerships Office at (281) 483-3809. MSC-23233