



## Materials

### Improved Small-Particle Powders for Plasma Spraying

Minimum layer thicknesses needed for complete coverage are reduced.

*John H. Glenn Research Center, Cleveland, Ohio*

Improved small-particle powders and powder-processing conditions have been developed for use in plasma spray deposition of thermal-barrier and environmental-barrier coatings. Heretofore, plasma-sprayed coatings have typically ranged in thickness from 125 to 1,800  $\mu\text{m}$ . As explained below, the improved powders make it possible to ensure complete coverage of substrates at unprecedentedly small thicknesses — of the order of 25  $\mu\text{m}$ .

Plasma spraying involves feeding a powder into a hot, high-velocity plasma jet. The individual powder particles melt in the plasma jet as they are propelled towards a substrate, upon which they splat to build up a coating. In some cases, multiple coating layers are required. The size range of the powder particles necessarily dictates the minimum thickness of a coating layer

needed to obtain uniform or complete coverage. Heretofore, powder particle sizes have typically ranged from 40 to 70  $\mu\text{m}$ ; as a result, the minimum thickness of a coating layer for complete coverage has been about 75  $\mu\text{m}$ .

In some applications, thinner coatings or thinner coating layers are desirable. In principle, one can reduce the minimum complete-coverage thickness of a layer by using smaller powder particles. However, until now, when powder particle sizes have been reduced, the powders have exhibited a tendency to cake, clogging powder-feeder mechanisms and feed lines.

Hence, the main problem is one of synthesizing smaller-particle powders having desirable flow properties. The problem is solved by use of a process that begins with a spray-drying subprocess to produce

spherical powder particles having diameters of  $<30 \mu\text{m}$ . (Spherical-particle powders have the best flow properties.) The powder is then passed several times through a commercial sifter with a mesh to separate particles having diameters  $<15 \mu\text{m}$ . The resulting fine, flowable powder is passed through a commercial fluidized-bed powder feeder into a plasma spray jet.

*This work was done by QuynhGiao N. Nguyen and Robert A. Miller of Glenn Research Center and George W. Leissler of QSS Group, Inc. Further information is contained in a TSP (see page 1).*

*Inquiries concerning rights for the commercial use of this invention should be addressed to NASA Glenn Research Center, Commercial Technology Office, Attn: Steve Fedor, Mail Stop 4-8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-17433-1.*

### Bonding-Compatible Corrosion Inhibitor for Rinsing Metals

Strong adhesive bonds can be made after rinsing with corrosion-inhibiting solutions.

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A corrosion-inhibiting mixture of compounds has been developed for addition to the water used to rinse metal parts that have been cleaned with aqueous solutions in preparation for adhesive bonding of the metals to rubber and rubberlike materials. Prior to the development of this corrosion inhibitor, the parts (made, variously, of D6AC steel and 7075-T73 aluminum) were rinsed by deionized water, which caused corrosion in some places on the steel parts — especially in such occluded places as sealing surfaces and threaded blind holes.

An integral part of the particular cleaning process is the deposition of a thin layer of silicates and silane primers that increase the strength of the adhesive bond. The corrosion inhibitor is formulated, not only to inhibit corrosion of both D6AC steel and 7075-T73 aluminum, but also to either increase or at least not reduce the strength of the adhesive bond to be formed subsequently. The corrosion inhibitor is a mixture of sodium silicate and sodium tetraborate. The sodium

silicate functions as both a corrosion inhibitor and a bond-strength promoter in association with the silane primers. The sodium tetraborate buffers the rinse solution at the optimum pH and functions as a secondary corrosion inhibitor for the steel.

For a given application, the concentrations of sodium silicate and sodium tetraborate must be chosen in a com-

promise among the needs to inhibit corrosion of steel, inhibit corrosion of aluminum, and minimize cosmetic staining of both steel and aluminum. Concentrations of sodium silicate in excess of 150 parts of silicon per million parts of solution (ppm Si) have been determined to enhance inhibition of corrosion; unfortunately, because of



**In a Blind Hole in a D6AC Steel Specimen** washed with deionized water, corrosion products can be seen on the thread (a). There is no visible sign of corrosion in a similar specimen rinsed with the corrosion-inhibited solution (b).

the alkalinity of sodium silicate, even a small concentration can raise the pH of the rinse solution to such a level that aluminum becomes corroded despite the inhibiting effect. The pH of a solution that contains a high concentration of sodium silicate can be decreased by adding sodium tetraborate. On the other hand, the addition of sodium tetraborate increases the concentration of dissolved solids to such a high level that cosmetic staining becomes an issue.

One suitable compromise calls for establishing the pH of the rinse solution at 9.5 and including sodium silicate at a concentration of 50 ppm Si. This

choice is justified by the observation that at a pH of 9.5, the rates of corrosion of both steel and aluminum are relatively low, and, therefore, only a minimal concentration of corrosion inhibitor is needed. By reducing the concentration of silicate, one reduces the amount of sodium tetraborate needed to buffer the rinse solution, thereby also reducing the level of cosmetic staining.

The figure depicts the effectiveness of the corrosion inhibitor in blind holes in D6AC steel specimens. In other tests on D6AC steel specimens, rates of corrosion in water containing this corrosion inhibitor were found to be approximately

$10^{-5}$ × those in deionized water. In similar tests on 7075-T73 aluminum specimens, rates of corrosion in water containing this corrosion inhibitor were found to be only slightly greater than those in deionized water. In still other tests, the strengths of adhesive bonds on steel and aluminum specimens rinsed with corrosion-inhibited solutions were found to be approximately equal to those on specimens rinsed with deionized water.

*This work was done by C. R. Saunders, L. A. Wurth, and A. Radar of ATK Thiokol, Inc., for Marshall Space Flight Center. For further information, contact businessdevelopment@atk.com.*

*MFS-31542*

## Wipes, Coatings, and Patches for Detecting Hydrazines

With suitable reformulation, other hazardous substances could also be detected.

*John F. Kennedy Space Center, Florida*

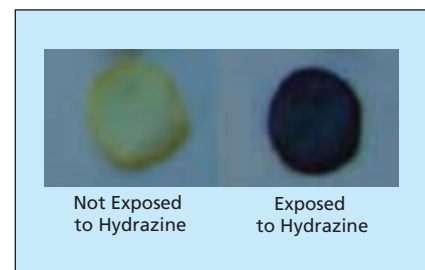
Three color-indicating devices have been conceived as simple, rapid, inexpensive means of detecting hazardous liquid and gaseous substances in settings in which safety is of paramount concern and it would be too time-consuming or otherwise impractical to perform detection by use of such instruments as mass spectrometers. More specifically, these devices are designed for detecting hypergolic fuels (in particular, hydrazines) and hypergolic oxidizers in spacecraft settings, where occasional leakage of these substances in liquid or vapor form occurs and it is imperative to take early corrective action to minimize adverse health effects. With suitable redesign, including reformulation of their color indicator chemicals, these devices could be adapted to detection of other hazardous substances in terrestrial settings (e.g., industrial and military ones).

One of the devices is a pad of a commercially available absorbent material doped with a color indicator. The absorbent material is made from 70 percent polyester and 30 percent nylon and can absorb about eight times its own weight of liquid. The color indicator is a mixture of conventional pH color indicator chemicals. Hydrazine and monomethyl hydrazine, which are basic, cause the color indicator to turn green. In the original intended application, the

pad is wiped on a space suit that is suspected of having been exposed to leaking monomethyl hydrazine during a space walk, before the wearer returns to the interior of the spacecraft. If the wiped surface is contaminated with hydrazine, the pad turns green. In addition, the pad absorbs hydrazine from the wiped surface, thereby reducing or eliminating the hazard. Used pads, including ones that show contamination by hydrazine, can be stored in a sealed plastic bag for subsequent disposal.

The second device, which has been proposed but not yet developed, would comprise a color indicator material in the form of either a coating on a space suit (or other protective garment) or a coating on a sheet that could be easily attached to and detached from the protective garment. The coating material would be a hydrogel doped with a suitable pH indicator. The hydrogel would also serve to maintain a level of moisture needed to support the chemical reaction mentioned in the next sentence. In addition to changing color to indicate the presence of any hypergolic fuel (which is basic) or hypergolic oxidizer (which is acidic) that might splash on the space suit, the pH indicator would also react with the hypergolic fuel or oxidizer and thereby bind it.

The third device is a color dosimeter for detecting hydrazine liquid or vapor



A Porous Cellulose Disk Loaded With  $\text{KAuCl}_4$  changes color upon exposure to hydrazine.

coming from microscopic leaks. This device is designed to satisfy several requirements specific to its original intended use in the auxiliary power unit of the space shuttle. These requirements include stability under vacuum, stability at moderate temperature, fast and irreversible change in color upon exposure to hydrazine, and visibility of the color change through polyimide tape. The device is a patch comprising a porous glass-fiber or cellulose patch loaded with potassium tetrachloroaurate (III) [ $\text{KAuCl}_4$ ], which changes color from yellow to purplish gray upon exposure to hydrazine (see figure).

*This work was done by Rebecca Young of Kennedy Space Center and William Buttner of Dynacs, Inc. For further information, contact the Kennedy Innovative Partnerships Office at (321) 867-1463. KSC-12400/342/552*