



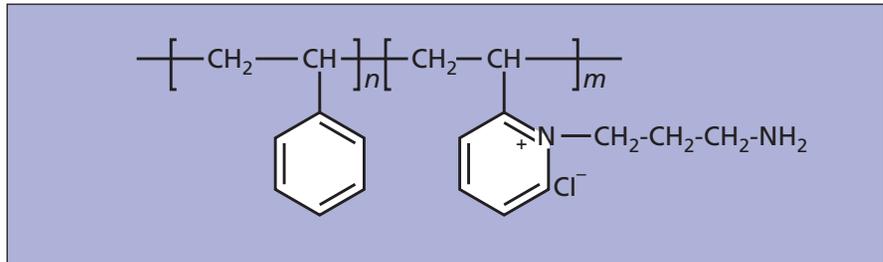
## Special Polymer/Carbon Composite Films for Detecting SO<sub>2</sub>

These films offer distinct advantages over prior SO<sub>2</sub>-sensor materials.

NASA's Jet Propulsion Laboratory, Pasadena, California

A family of polymer/carbon films has been developed for use as sensory films in electronic noses for detecting SO<sub>2</sub> gas at concentrations as low as 1 part per million (ppm). Most previously reported SO<sub>2</sub> sensors cannot detect SO<sub>2</sub> at concentrations below tens of ppm; only a few can detect SO<sub>2</sub> at 1 ppm. Most of the sensory materials used in those sensors (especially inorganic ones that include solid oxide electrolytes, metal oxides, and cadmium sulfide) must be used under relatively harsh conditions that include operation and regeneration at temperatures >100 °C. In contrast, the present films can be used to detect 1 ppm of SO<sub>2</sub> at typical operating temperatures between 28 and 32 °C and can be regenerated at temperatures between 36 and 40 °C.

The basic concept of making sensing films from polymer/carbon composites is not new. The novelty of the present family of polymer/carbon composites lies in formulating the polymer components of these composites specifically to optimize their properties for detecting SO<sub>2</sub>. First-principles quantum-mechanical calculations of the energies of binding of SO<sub>2</sub> molecules to various polymer functionalities are used as a guide for selecting polymers and understanding the role of polymer functionalities in sensing.



This Copolymer is a member of the family of polymers suitable for use as components of polymer/carbon composite films for sensing SO<sub>2</sub>. The integers  $m$  and  $n$  can be chosen by formulation to be in a desired ratio: a typical ratio for the molecular structure shown here is  $nm = 3/7$ .

The polymer used in the polymer-carbon composite is a copolymer of styrene derivative units with vinyl pyridine or substituted vinyl pyridine derivative units (see figure). To make a substituted vinyl pyridine for use in synthesizing such a polymer, poly(2-vinyl pyridine) that has been dissolved in methanol is reacted with 3-chloropropylamine that has been dissolved in a solution of methanol. The methanol is then removed to obtain the copolymer. Later, the copolymer can be dissolved in an appropriate solvent with a suspension of carbon black to obtain a mixture that can be cast and then dried to obtain a sensory film.

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*Jewell, Abhijit Shevade, Kenneth Manatt, Charles Taylor, Mario Blanco, and William Goddard 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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## Nickel-Based Superalloy Resists Embrittlement by Hydrogen

This alloy also exhibits high strength and ductility.

Marshall Space Flight Center, Alabama

A nickel-based superalloy that resists embrittlement by hydrogen more strongly than does nickel alloy 718 has been developed. Nickel alloy 718 is the most widely used superalloy. It has excellent strength and resistance to corrosion as well as acceptably high ductility, and is recognized as the best alloy for many high-temperature applications. However, nickel alloy 718 is susceptible to embrittlement by hydrogen and to delayed failure and reduced tensile properties in gaseous hydrogen. The

greater resistance of the present nickel-based superalloy to adverse effects of hydrogen makes this alloy a superior alternative to nickel alloy 718 for applications that involve production, transfer, and storage of hydrogen, thereby potentially contributing to the commercial viability of hydrogen as a clean-burning fuel.

The table shows the composition of the present improved nickel-based superalloy in comparison with that of nickel alloy 718. This composition was

chosen to obtain high resistance to embrittlement by hydrogen while maintaining high strength and exceptional resistance to oxidation and corrosion. The alloy-design approach followed to arrive at this composition was based on accounting for the simultaneous effects of several additions. The approach included systematic modification of  $\gamma$ -matrix compositions for increased resistance to embrittlement by hydrogen, increasing the volume fraction of the

Element	Proportion in the Present Superalloy	Proportion in Nickel Alloy 718
Ni	Balance	Balance
Fe	24 to 34	17 to 19
Cr	17 to 19	16 to 18
Mo	3.0 to 5.0	2.5 to 3.5
Co	3.0 to 5.0	0.1 to 1.0
V	0.1 to 1.0	—
W	3.0 to 6.0	—
Ti	2.0 to 3.5	0.5 to 1.5
Nb	0.5 to 2.0	4.5 to 6.0
Al	0.1 to 0.5	0.2 to 0.8

Proportions of Chemical Elements in the two alloys are given in weight percentages.

$\gamma'$  phase, adding  $\gamma$ -matrix-strengthening elements to increase strength, and obtaining precipitate-free grain boundaries. Substantial amounts of chromium and nickel were also included to obtain excellent resistance to oxidation and corrosion. Microstructural stability was maintained through improved solid solubility of the  $\gamma$  ma-

trix along with the addition of alloying elements that retard  $\eta$ -phase precipitation. This alloy represents a material system that greatly extends ranges of composition beyond those of prior nickel-base superalloys that resist embrittlement by hydrogen.

This alloy is first processed by a combination of vacuum induction melting and vac-

uum arc remelting. Typically, the resulting alloy ingot is homogenized at a temperature of 2,100 °C for 24 hours and then hot-rolled in the range of 927 to 1,093 °C into 1.6-cm-thick plates. The plates are subjected to a solution heat treatment at 1,050 °C for 1 hour, followed by aging at 718 °C for 8 hours, then 621 °C for 8 hours.

The most novel property of this alloy is that it resists embrittlement by hydrogen while retaining tensile strength >175 kpsi (>1.2 GPa). This alloy exhibits a tensile elongation of more than 20 percent in hydrogen at a pressure of 5 kpsi ( $\approx$ 34 MPa) without loss of ductility. This amount of elongation corresponds to 50 percent more ductility than that exhibited by nickel alloy 718 under the same test conditions.

*This work was done by Jonathan Lee of Marshall Space Flight Center and Po-Shou Chen of Illinois Institute of Technology Research Institute. For more information, contact Sammy Nabors, MSFC Commercialization Assistance Lead at sammy.a.nabors@nasa.gov. Refer to MFS -31781-1*

## Chemical Passivation of Li<sup>+</sup>-Conducting Solid Electrolytes

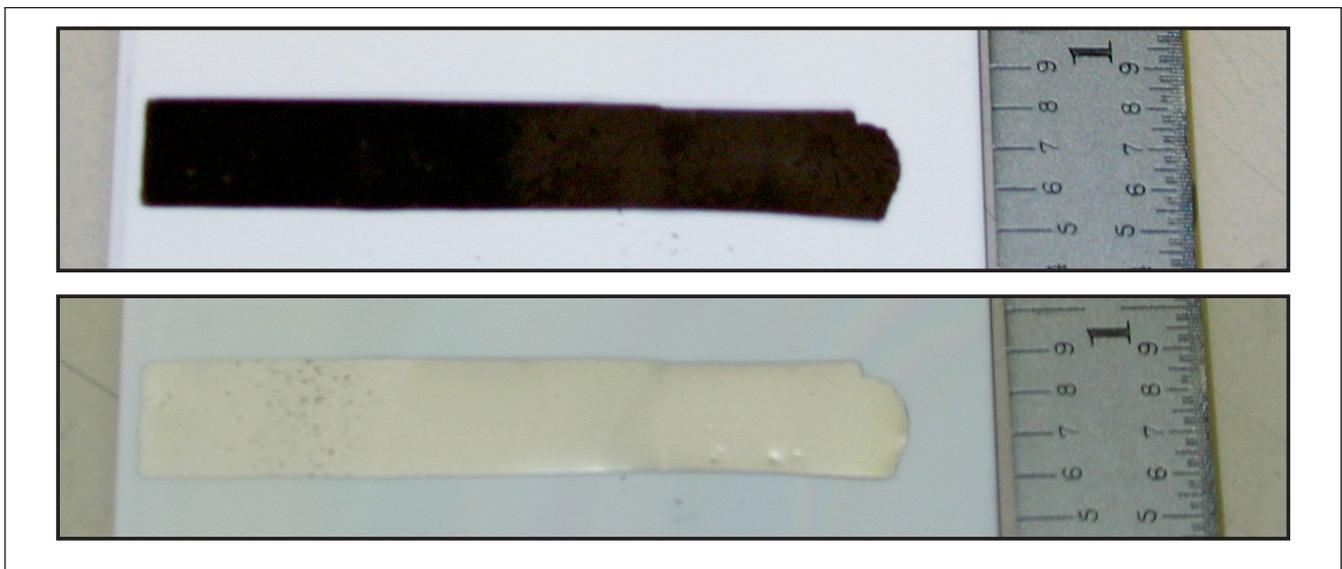
Such passivation could enable long-life lithium rechargeable cells.

NASA's Jet Propulsion Laboratory, Pasadena, California

Plates of a solid electrolyte that exhibits high conductivity for positive lithium ions can now be passivated to prevent them from reacting with metallic lithium. Such passivation could en-

able the construction and operation of high-performance, long-life lithium-based rechargeable electrochemical cells containing metallic lithium anodes. The advantage of this approach, in com-

parison with a possible alternative approach utilizing lithium-ion graphitic anodes, is that metallic lithium anodes could afford significantly greater energy-storage densities.



The Dark Strip in the Upper Photograph is the product of the chemical reaction of a deposited lithium film and an underlying solid-electrolyte plate. The corresponding somewhat shiny strip in the lower photograph is a lithium film deposited on a solid-electrolyte plate coated with LiPON. The dark spots in the Li film contain the reaction product formed at pinholes in the LiPON, where the Li film came into contact with the underlying solid electrolyte. This illustrates the importance of depositing pinhole-free LiPON films to protect solid-electrolyte plates.