## Materials & Coatings

## Polyurea-Based Aerogel Monoliths and Composites

These aerogels can be used in portable apparatus for warming, storing, and/or transporting food and medicine, and can be recycled for fillers for conventional plastics.

Lyndon B. Johnson Space Center, Houston, Texas

A flexible, organic polyurea-based aerogel insulation material was developed that will provide superior thermal insulation and inherent radiation protection for government and commercial applications. The rubbery polyureabased aerogel exhibits little dustiness, good flexibility and toughness, and durability typical of the parent polyurea polymer, yet with the low density and superior insulation properties associated with aerogels. The thermal conductivity values of polyurea-based aerogels at lower temperature under vacuum pressures are very low and better than that of silica aerogels.

Flexible, rubbery polyurea-based aerogels are able to overcome the weak and brittle nature of conventional inorganic and organic aerogels, including polyisocyanurate aerogels, which are generally prepared with the one similar component to polyurethane rubber aerogels. Additionally, with higher content of hydrogen in their structures, the polyurea rubber-based aerogels will also provide inherently better radiation protection than those of inorganic and carbon aerogels. The aerogel materials also demonstrate good hydrophobicity due to their hydrocarbon molecular structure.

There are several strategies to overcoming the drawbacks associated with the weakness and brittleness of silica aerogels. Development of the flexible fiber-reinforced silica aerogel composite blanket has proven to be one promising approach, providing a conveniently fielded form factor that is relatively robust in industrial environments compared to silica aerogel monoliths. However, the flexible, silica aerogel composites still have a brittle, dusty character that may be undesirable, or even intolerable, in certain application environments. Although the crosslinked organic aerogels, such as resorcinol-formaldehyde (RF), polyisocyanurate, and cellulose aerogels, show very high impact strength, they are also very brittle with little elongation (i.e., less rubbery). Also, silica and carbon aerogels are less efficient radiation shielding materials due to their lower content of hydrogen element.

The invention involves mixing at least one isocyanate resin in solvent along with a specific amount of at least one polyamine hardener. The hardener is selected from a group of polyoxyalkyleneamines, amine-based polyols, or a mixture thereof. Mixing is performed in the presence of a catalyst and reinforcing inorganic and/or organic materials, and the system is then subjected to gelation, aging, and supercritical drying. The aerogels will offer exceptional flexibility, excellent thermal and physical properties, and good hydrophobicity.

The rubbery polyurea-based aerogels are very flexible with no dust and hydrophobic organics that demonstrated the following ranges of typical properties: densities of 0.08 to 0.293 g/cm<sup>3</sup>, shrinkage factor (raerogel/rtarget) = 1.6 to 2.84, and thermal conductivity values of 15.2 to 20.3 mW/m K.

This work was done by Je Kyun Lee of Aspen Aerogels, Inc. for Johnson Space Center. 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:

Aspen Aerogels, Inc.

30 Forbes Road, Building B

Northborough, MA 01532

Phone No.: (508) 691-1111

Fax No.: (508) 691-1200

Refer to MSC-24214-1, volume and number of this NASA Tech Briefs issue, and the page number.

# Resin-Impregnated Carbon Ablator: A New Ablative Material for Hyperbolic Entry Speeds

From surface temperatures as high as  $\approx$ 3,000 °C, the measured back temperature is only 50 °C. *Goddard Space Flight Center, Greenbelt, Maryland* 

Ablative materials are required to protect a space vehicle from the extreme temperatures encountered during the most demanding (hyperbolic) atmospheric entry velocities, either for probes launched toward other celestial bodies, or coming back to Earth from deep space missions. To that effect, the resin-impregnated carbon ablator (RICA) is a high-temperature carbon/phenolic ablative thermal protection system (TPS) material designed to use modern and commercially viable components in its manufacture. Heritage carbon/phenolic ablators intended for this use rely on materials that are no longer in production (i.e., Galileo, Pioneer Venus); hence the development of alternatives such as RICA is necessary for future NASA planetary entry and Earth re-entry missions. RICA's capabilities were initially measured in air for Earth re-entry applications, where it was exposed to a heat



Figure 1. **RICA Sample** during plasma wind tunnel testing.

flux of 14  $MW/m^2$  for 22 seconds. Methane tests were also carried out for potential application in Saturn's moon Titan, with a nominal heat flux of 1.4  $MW/m^2$  for up to 478 seconds. Three slightly different material formulations

RICA	Phenolic Content (~%)	Carbon Content (~%)	Density (gm/ml)	Plasma Wind Tunnel Heat Flux (MW/m <sup>2</sup> )	Heat Duration (s)	Integrated Heat Input (J/m <sup>2</sup> )	Mass Loss (gm)	Average Recession (mm)	Average Surface Temp from Pyrometer(c)	Average Thermal Gradient (K/mm)	Heat of Ablation (J/kg)
5C	17	83	1.41	1.4	478	6.69E+08	7.84	4.218	1978.1	44.37	49E.+07
SA (1)	27	73	1.39	14	22	3.08E+08	3.33	1.96	3336.1	34.32	1.1E+08
3A	24	76	1.36	1.4	478	6.69E+08	3.32	0.342	1962.5	54.50	8.5E+07
5B	33	67	1.37	1.4	476	6.67E+08	3.73	1.217	1990.8	53.68	7.7E+07
ЗB	31	69	1.35	1.4	477	6.67E+08	3.70	1.143	1967.5	51.11	8.5E+07
(1) Tested in Air; all others tested in Methane											

Table. Material Properties and initial test results.

were manufactured and subsequently tested at the Plasma Wind Tunnel of the University of Stuttgart in Germany (PWK1) in the summer and fall of 2010. The TPS' integrity was well preserved in most cases, and results show great promise.

There are several major elements involved in the creation of a successful ablative TPS material: the choice of fabric and resin formulation is only the beginning. The actual processing involved in manufacturing involves a careful choice of temperature, pressure, and time. This manufacturing process must result in a material that survives heat loads with no de-lamination or spallation. Several techniques have been developed to achieve this robustness. Variants of RICA's material showed no delamination or spallation at intended heat flux levels, and their potential thermal protection capability was demonstrated. Three resin formulations were tested in two separate samples each manufactured under slightly different conditions. A total of six samples was eventually chosen for test at the PWK1. Material performance properties and results for five of those are shown in the table. In the most extreme case, the temperature dropped from  $\approx 3,000$  to 50 °C across 1.8 cm, demonstrating the material's effectiveness in protecting a spacecraft's structure from the searing heat of entry.

With a manufacturing process that can be easily re-created, RICA has proven to be a viable choice for highspeed hyperbolic entry trajectories, both in methane (Titan) as well as in air (Earth) atmospheres. Further assessment and characterization of spallation and an exact determination of its onset heat flux (if present for intended applications) still remain to be measured.

This work was done by Jaime Esper of Goddard Space Flight Center and Michael Lengowski of the University of Stuttgart. Further information is contained in a TSP (see page 1). GSC-16183-1

### Self-Cleaning Particulate Prefilter Media

This technology has application for air filter manufacturers for self-cleaning particulate prefilters.

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

A long-term space mission requires efficient air revitalization performance to sustain the crew. Prefilter and particulate air filter media are susceptible to rapid fouling that adversely affects their performance and can lead to catastrophic failure of the air revitalization system, which may result in mission failure. For a long-term voyage, it is impractical to carry replacement particulate prefilter and filter modules due to the usual limitations in size, volume, and weight. The only solution to this problem is to reagentlessly regenerate prefilter and filter media in place. A method was developed to modify the particulate prefilter media to allow them to regenerate reagentlessly, and in place, by the application of modest thermocycled transverse or reversed airflows. The innovation may allow NASA to close the breathing air loop more efficiently, thereby sustaining the vision for manned space exploration missions of the future.

A novel, self-cleaning coatings technology was developed for air filter media surfaces that allows reagentless in-place regeneration of the surface. The technology grafts thermoresponsive and nonspecific adhesion minimizing polymer nanolayer brush coatings from the prefilter media. These polymer nanolayer brush architectures can be triggered to contract and expand to generate a "pushing-off" force by the simple application of modestly thermocycled (i.e. cycling from ambient cabin temperature to 40 °C) air streams. The nonspecific adhesion-minimizing properties of the coatings do not allow the particulate foulants to adhere strongly to the filter