

new here is the selection of  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  particles as candidate opacifiers that, in comparison with some prior opacifiers (carbon black and metal nanoparticles), are more thermally stable.

The preparation of a composite material of the present type includes synthesis of the silica-aerogel component in a sol-gel process. The  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  particles, made previously in a separate process, are mixed into the sol, which is then cast onto fused-quartz-fiber batting. Then the aerogel-casting solution is poured into the mold, where it permeates the silica fiber felt. After the sol has gelled, the casting is aged and then subjected to supercritical drying to convert the gel to the final aerogel form.

The separate process for making the  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  particles begins with the slow addition of corresponding proportions of  $\text{La}(\text{CH}_3\text{COOH})_3$ ,  $\text{Mn}(\text{CH}_3\text{COOH})_3$ , and  $\text{Sr}(\text{NO}_3)_2$  to a solution of  $\text{H}_2\text{O}_2$  in  $\text{H}_2\text{O}$ . The solution is then peptized by drop-wise addition of  $\text{NH}_4\text{OH}$  to obtain a sol. Next, the sol is dried in an oven at a temperature of  $120^\circ\text{C}$  to obtain a glassy solid. The solid is calcined at  $700^\circ\text{C}$  to convert it to  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ . Then  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  particles are made by ball-milling the calcined solid.

The effectiveness of  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  particles as opacifiers and thermal-conductivity reducers depends on the statistical distribution of particle sizes as well as the relative proportions of  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  and aerogel. For experiments performed thus far, samples of

aerogel/fiber composites were formulated to have, variously, silica target density of  $0.07$  or  $0.14\text{ g/cm}^3$  and to contain 30 percent of  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  in average particle size of  $0.3$  or  $3\text{ }\mu\text{m}$ . The thermal conductivities of the samples containing the  $3\text{-}\mu\text{m}$   $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  particles were found to be lower than those of the samples containing the  $0.3\text{-}\mu\text{m}$   $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  particles. The optimum particle size is believed to be between  $1$  and  $5\text{ }\mu\text{m}$ .

*This work was done by Wendell Rhine, Andrew Polli, and Kiranmayi Deshpande of Aspen Aerogels, Inc. for Marshall Space Flight Center. For further information, contact Sammy Nabors, MSFC Commercialization Assistance Lead, at [sammy.a.nabors@nasa.gov](mailto:sammy.a.nabors@nasa.gov). Refer to MFS-32587-1.*

## Cyclic Oxidation Behavior of CuCrAl Cold-Sprayed Coatings for Reusable Launch Vehicles

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

The next generation of reusable launch vehicles is likely to use GRCop-84 [Cu-8(at.%)Cr-4%Nb] copper alloy combustion liners. The application of protective coatings on GRCop-84 liners can minimize or eliminate many of the environmental problems experienced by uncoated liners and significantly extend their operational lives and lower operational cost. A newly developed Cu-23 (wt.%) Cr-5% Al (CuCrAl) coating, shown to resist hydrogen attack and oxidation in an as-cast form, is currently being considered as a protective coating for GRCop-84. The coating was deposited on GRCop-84 substrates by the cold spray deposition technique, where the CuCrAl was procured as gas-atom-

ized powders. Cyclic oxidation tests were conducted between  $773$  and  $1,073\text{ K}$  to characterize the coated substrates.

The coating proved to be effective in preventing the cyclic oxidation of the substrate for up to  $1,000$  cycles. The coated substrates showed no significant weight loss in comparison to uncoated specimens, which lost between  $60$  to  $80$  percent of its original weight with much lower lives. The coating was adherent to the substrate at all temperatures, whereas the uncoated GRCop-84 showed excessive spallation of the oxide scale. It is anticipated that the use of this alloy can extend the operational life of the liner, which translates to increased component reliability,

shorter depot maintenance turnaround time, and lower operational cost. Additionally, engines using Cu-CrAl-coated GRCop-84 combustion liners could operate at higher temperatures, thereby resulting in its increased thermal efficiency.

*This work was done by Sai Raj of Glenn Research Center and J. Karthikeyan of ASB Industries. 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, Innovative Partnerships Office, Attn: Steve Fedor, Mail Stop 4-8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-18330-1.*

## Ceramic Fiber Structures for Cryogenic Load-Bearing Applications

**Woven or braided fibers resist embrittlement under cryogenic conditions, enabling ultralow-temperature applications.**

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

This invention is intended for use as a load-bearing device under cryogenic temperatures and/or abrasive conditions (i.e., during missions to the Moon). The innovation consists of small-

diameter, ceramic fibers that are woven or braided into devices like ropes, belts, tracks, or cables. The fibers can be formed from a variety of ceramic materials like silicon carbide, carbon, alumi-

nosilicate, or aluminum oxide. The fiber architecture of the weave or braid is determined by both the fiber properties and the mechanical requirements of the application. A variety of weave or braid

architectures is possible for this application. Thickness of load-bearing devices can be achieved by using either a 3D woven structure, or a layered, 2D structure. For the prototype device, a belt approximately 0.10 in. (0.25 cm) thick, and 3.0 in. (7.6 cm) wide was formed by layering and stitching a 2D aluminosilicate fiber weave. The circumferential length of the 2D, layered belt was approximately 36 in. (91 cm).

To demonstrate the resistance to abrasion while under load, the ceramic fiber belt was installed on two aluminum spools that were mounted in an Instron load frame. Both spools were completely enclosed in a Lexan box within the load frame. Bearings were used at each end of the spool shafts to allow the spools to spin freely while a load was applied. The lower spool, which was secured to the stationary head of the load frame, was also attached to a small motor to drive the rollers. The upper spool was attached to the movable crosshead of the

Instron frame to apply a load to the belt while it was rolling. JSC1a, a highly abrasive lunar regolith simulant, was added to the Lexan box. Enough JSC1a was placed in the bottom of the box to allow the belt to contact and pick up the dust as it traveled around the lower spool. The track was exposed to the dust while rolling under load for several hours to simulate relevant rover mission duration. After 12.5 hours of exposure to the lunar simulant, under loads varying between 50 and 100 N, no elongation or mechanical creep of the belt was measured. Under these loads, which were estimated to be comparable to those required for tracks on the lunar rover, there was no deformation or loss of load carrying ability.

To demonstrate flexibility under cryogenic conditions, individual fibers and fiber tows were exposed to cryogenic temperatures by being submerged in liquid nitrogen for 4 minutes, and then were flexure tested. Immediately upon

removal from the liquid nitrogen, the fibers and tows were wrapped around mandrels of progressively smaller diameters. Both the fibers and the tows were successfully wrapped around wire mandrels with a diameter of approximately 0.02 in. (0.5 mm) without any breakage. The continuous ceramic belt that is envisioned for the lunar rover would be a closed-edge, multilayer weave with through thickness holes woven in place. The holes in the weave would engage the sprockets on the drive mechanism of the track device.

*This work was done by Martha H. Jaskowiak and Andrew J. Eckel of Glenn Research Center. 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, Innovative Partnerships Office, Attn: Steve Fedor, Mail Stop 4-8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-18364-1.*

## Elastomer Reinforced With Carbon Nanotubes

*Lyndon B. Johnson Space Center, Houston, Texas*

Elastomers are reinforced with functionalized, single-walled carbon nanotubes (SWNTs) giving them high-breaking strain levels and low densities. Cross-linked elastomers are prepared using amine-terminated, poly(dimethylsiloxane) (PDMS), with an average molecular weight of 5,000 daltons, and a functionalized SWNT.

Cross-link densities, estimated on the basis of swelling data in toluene (a dispersing solvent) indicated that the polymer underwent cross-linking at the ends of the chains. This thermally initiated cross-linking was found to occur

only in the presence of the aryl alcohol functionalized SWNTs. The cross-link could have been via a hydrogen-bonding mechanism between the amine and the free hydroxyl group, or via attack of the amine on the ester linkage to form an amide.

Tensile properties examined at room temperature indicate a three-fold increase in the tensile modulus of the elastomer, with rupture and failure of the elastomer occurring at a strain of 6.5.

*This work was done by James M. Tour and Jared L. Hudson of Rice University and Ramanan Krishnamoorti of University of Hous-*

*ton for Johnson Space Center. For further information, contact the JSC Innovation Partnerships Office at (281) 483-3809.*

*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:*

*William M. Rice University  
Office of Technology Transfer  
6100 Main Street  
Houston, TX 77005  
Phone No.: (713) 348-6188*

*Refer to MSC-24071-1, volume and number of this NASA Tech Briefs issue, and the page number.*

## Biologically Inspired Purification and Dispersion of SWCNTs

*Lyndon B. Johnson Space Center, Houston, Texas*

A biologically inspired method has been developed for (1) separating single-wall carbon nanotubes (SWCNTs) from other materials (principally, amorphous carbon and metal catalysts) in raw production batches and (2) dispersing the SWCNTs as individual particles (in contradistinction to ropes and bundles) in suspension, as required for a number of applications.

Prior methods of purification and dispersal of SWCNTs involve, variously, harsh physical processes (e.g., sonication) or harsh chemical processes (e.g., acid reflux). These processes do not completely remove the undesired materials and do not disperse bundles and ropes into individual suspended SWCNTs. Moreover, these processes cut long SWCNTs into shorter pieces,

yielding typical nanotube lengths between 150 and 250 nm.

In contrast, the present method does not involve harsh physical or chemical processes. The method involves the use of biologically derived dispersal agents (BDDAs) in an aqueous solution that is mechanically homogenized (but not sonicated) and centrifuged. The dense solid material remaining after centrifuga-