

tion. In order to raise its operational temperature limit, a proprietary oxidation barrier coating was developed. TZM was selected as the attach mechanism material because of its manufacturability and structural performance as well as its ability to withstand the plasma environment when coated with the oxidation barrier coating.

Prior art involved square, inflexible plugs that were too small to provide complete WLE coverage and would also create protuberances into the hypersonic flow that would overheat during operation. This innovation is an evolution of the prior art in that the current

plug is a thin, flexible double-curved shell with rounded edges, which facilitates a deformed shape that conforms to the WLE geometry with minimal disturbance of the flow.

The repair plug is designed to capitalize on flexibility and curved shape in order to minimize the number of cover plates required. The current design reduced the number of unique plug shapes required to cover critical WLE regions from over 1,300 to less than 20. Non-linear finite element analysis, including contacting surfaces, was used to model the plug during installation and operation. Computa-

tional fluid dynamics and thermal analysis were used to predict plug temperatures during entry.

This work was done by Charles J. Camarda, Joseph Sikora, Russel Smith, H. Rivers, and Stephen J. Scotti of NASA; Alan M. Fuller of United Space Alliance; Robert Klacka of General Electric Ceramic Composites, LLC; Martin Reinders of The Boeing Company; Francis Schwind of Carbon-Carbon Advanced Technologies, Inc.; Brian Sullivan of Materials Research and Design, Inc.; and Dean Lester of ATK Thiokol for Johnson Space Center. Further information is contained in a TSP (see page 1). MSC-24347-1

Three Dimensionally Interlinked, Dense, Solid Form of Single-Walled CNT Ropes

The nanotube block has high mechanical strength and resistance to indentation.

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A 3D networked, dense form of single-walled carbon nanotubes (SWNT) has been made through isotropic shrinking of a gel-like SWNT-water paste by very slow evaporation. Approximately 35 g of Raw HiPco nanotubes were cleaned by the method of soft baking (250 °C for 15 hours in air saturated with water vapor) in a glass beaker followed by leaching with concentrated hydrochloric acid. Typically, one liter of concentrated hydrochloric acid was added to the soft-baked voluminous mass in the same large beaker, and allowed to digest at room temperature with stirring overnight. The acid-digested SWNT slurry was filtered through a large porcelain Buchner funnel under atmospheric pressure.

The slurry was continuously flushed, while still in the funnel, with a very slow but steady stream of deionized water employing a peristaltic pump. This process, referred to as “washing,” continued until the filtrate water dripping from the Buchner funnel was clear, colorless, and neutral to a pH paper. This took about 15 liters of water to flow through the slurry over a day. At this point, the water pump was stopped and the SWNT-water slurry was allowed to drain the excess water for about 10 hours. The resulting thick paste of SWNT-neutral water was transferred to a beaker. The beaker was covered with aluminum foil with few holes and allowed to dry very slowly in a hood at room temperature. In about eight weeks, the sam-

ple gradually dried isotropically to a cylindrical dense mass referred to as a carbon nanotube block (CNB).

There was no carbonaceous matter sticking to any of the glass surface where the SWNT-water paste made contact. The approximate dimensions of the cylindrical SWNT block that weighed 28 g were 1.5 in. (≈ 3.8 cm) in diameter and 1.25 in. (≈ 3.2 cm) in height. The bottom portion of the cylinder that was in contact with the beaker surface was slightly wider, indicating some resistance to shrinking. The cylindrical mass also consisted of several pores. The cylindrical mass was very tough and could not be broken with a small hammer using considerable force. The mass of the solid could be polished over a fine grain emery paper or even a smooth, stainless steel surface indicative of alignment at finer levels.

When attempting to cut with a sharp knife edge, the mass showed extreme resistance to the back-and-forth movement of the blade and indentation. Small portions were cut out of the solid block using a hacksaw and experimented. A small piece of the block was placed in water. It floated initially for a few minutes, but sank subsequently, indicative of a density >1 g/cm³.

Two small portions (total volume approximately 1 cm³) were placed in a small, conical flask, and 25 ml of 100-percent H₂SO₄ was added to that and closed with a ground-glass stopper. The

small pieces floated in 100-percent H₂SO₄. The SWNT block gradually swelled in volume and occupied the whole of the liquid volume (≈ 25 cm³) in two days. The SWNT/100-percent H₂SO₄ paste was very rigid and did not show fluidity. Scanning electron microscopy of the block showed evidence for the block to be constituted by packing of densely placed SWNT ropes. The polished surface showed evidence of a very high degree of smoothness up to 200 nm. The novelty and significance of this solid is the clear presence of a three dimensionally connected dense network of SWNT (with high mechanical strength) in a bulk form.

One immediate problem this new form of SWNT solves is handling. The dense form of SWNT is much easier to handle than the pristine forms of SWNT that could easily form potentially hazardous aerogels. The other bulk form of SWNT, known as buckypaper, needs ample time, equipment, and manpower to process; lacks mechanical strength; mostly retains the surfactants used in the process as organic impurities; and poses problems in redispersion to single-tube levels. The CNB, on the other hand, is made in a single step from the water-SWNT gel-like paste without recourse to any surfactant, possesses extreme mechanical strength, and can be redissolved in strong acids.

This work was done by Richard E. Smalley of Rice University 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:

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