

that most of the interior of the material is accessible for intercalation of lithium. Moreover, the greater accessibility of SWCNT structures can be expected to translate to greater mobilities for ion-exchange processes and, hence, an ability to sustain greater charge and discharge current densities.

For the experiments, soot containing carbon nanotubes was produced by laser vaporization of a graphite target containing 1.2 atomic percent Ni/Co in an argon atmosphere at a pressure of 500 torr (≈ 67 kPa) at a temperature of 1,200 °C. The soot was purified by refluxing in nitric acid at a temperature of 125 °C followed by annealing in oxygen at 550 °C for 30 minutes. By means of scanning electron microscopy, transmission electron microscopy, ultraviolet-visible spectrophotometry, and thermogravimetric analysis, 99 weight percent of the purified soot was found to consist of SWCNTs. The specific surface area of the pu-

rified material, as measured by use of the Brunauer, Emmett, and Teller (BET) technique based on adsorption of nitrogen, was found to be 1,200 m²/g — much greater than the specific surface areas of several other carbonaceous anode materials that were also subjected to the BET test.

Anodes were fabricated by casting, onto copper foils, thin films of the purified SWCNT material dispersed at a concentration of 5 weight percent in poly(vinylidene fluoride). The anodes were incorporated into standard three-electrode test cells along with lithium-foil counter and reference electrodes and an electrolyte comprising 1.0 M LiPF₆ in a solution of ethylene carbonate (2 parts by volume) and dimethyl carbonate (1 part by volume). Over-potentials for interaction of the anodes with lithium and lithium capacities of the anodes were measured by use of cyclic voltammetry and galvanostatic cycling, respectively.

The figure shows representative charge and discharge capacities determined from the measurements. The plotted values show a high degree of reversibility after the initial cycle. The specific capacity after 30 cycles was found to be about 1.33 Ah/g — almost 3 times the generally accepted value (0.45 Ah/g) for graphite.

This work was done by Aloysius F. Hepp of Glenn Research Center; Ryne Raffaele and Tom Gennett of Rochester Institute of Technology; Prashant Kumta and Jeff Maranchi of Carnegie-Mellon University; and Mike Heben of National Renewable Energy Laboratory. Further information is contained in a TSP (see page 1).

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Tantalum-Based Ceramics for Refractory Composites

Compositions can be graded from porous substrates to impervious outer layers.

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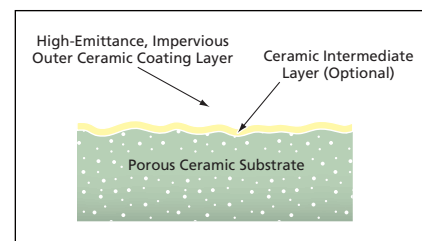
A family of tantalum-based ceramics has been invented as ingredients of high-temperature composite insulating tiles. These materials are suitable for coating and/or permeating the outer layers of rigid porous (foamlike or fibrous) ceramic substrates to (1) render the resulting composite ceramic tiles impervious to hot gases and (2) enable the tiles to survive high heat fluxes at temperatures that can exceed 3,000 °F ($\approx 1,600$ °C). Originally intended for use on the future space exploration vehicles, insulating tiles made with these materials may also be useful in terrestrial applications (e.g., some industrial processes) in which there are requirements to protect against flows of hot, oxidizing gases.

The starting ingredients of a ceramic of this invention are the following:

- Tantalum disilicide (TaSi₂), which can act as either an emittance agent or a matrix material, depending upon the overall composition;
- Molybdenum disilicide (MoSi₂), which, depending on the overall composition, acts as a secondary emittance agent or as an oxygen getter (to inhibit the oxidation of tantalum within the finished composite);

- A borosilicate glass (B₂O₃-SiO₂), which acts as a source of boron and may, depending on the overall composition, act as an alternative matrix; and
- Silicon hexaboride (SiB₆), which acts as a processing aid by facilitating sintering of the aforementioned ingredients.

These ingredients are milled together in ethanol, and the resulting slurry is sprayed or painted onto a porous ceramic substrate. The underlying layers (sub-layers) of different compositions are used to integrate the outer layer (coating) with a wide variety of porous substrate materials. For this purpose, infiltration of these compositions into the substrate results in a functional gradient system that accounts for the difference in the coefficient of thermal expansion (CTE) between coating and substrate. The preferred composition for such underlying layers are in the approximate range of 20 to 60 weight percent of MoSi₂, 0 to 60 weight percent of TaSi₂, 40 to 80 weight percent of borosilicate glass, and 1 to 5 percent of SiB₆. The ingredients are then sintered by heating the treated substrate at atmospheric pressure at either a temperature of 2,225 °F ($\approx 1,218$ °C) for 90 minutes or a temperature of 2,400 °F ($\approx 1,316$ °C) for 10 min-



A Porous Ceramic Substrate Is Coated with one or more ceramic layers to render it impervious to hot gas and increase its ability to withstand high heat flux.

utes. The milling, coating, and sintering process conditions are chosen to minimize the undesired oxidation of tantalum compounds.

For an outer coating layer, the preferred composition is between 20 and 45 weight percent of borosilicate glass, between 10 and 65 weight percent of TaSi₂, between 5 and 30 weight percent of MoSi₂, and between 1 and 5 weight percent of SiB₆. An underlying layer or sublayer of different composition can be used to integrate the outer layer with the substrate and, for this purpose, may be allowed to infiltrate to some small depth below the surface of the substrate. The preferred composition for such an underlying layer lies in the approximate range of 20 to 60 weight percent of

MoSi₂, 40 to 80 weight percent of B₂O₃·SiO₂, and 1 to 5 percent of SiB₆. One or more intervening layer(s) of intermediate composition(s) could also be included (see figure). The precise composition of the sublayer should be chosen to match the CTE of the substrate, while the composi-

tions of intermediate layers should be chosen to grade the transition from the CTE and porosity of the substrate to the CTE and full density of the outer coating layer.

*This work was done by David A. Stewart, Daniel Leiser, Robert DiFiore, and Victor Katvala of **Ames Research Center**.*

This invention is owned by NASA, and a patent application has been filed. Inquiries concerning rights for the commercial use of this invention should be addressed to the Ames Technology Partnerships Division at (650) 604-2954. Refer to ARC-14743-1.