



## Composite Silica Aerogels Opacified With Titania

Thermal insulation is enhanced by reducing the radiative contribution to heat transfer.

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A further improvement has been made to reduce the high-temperature thermal conductivities of the aerogel-matrix composite materials described in "Improved Silica Aerogel Composite Materials" (NPO-44287), *NASA Tech Briefs*, Vol. 32, No. 9 (September 2008), page 50. Because the contribution of infrared radiation to heat transfer increases sharply with temperature, the effective high-temperature thermal conductivity of a thermal-insulation material can be reduced by opacifying the material to reduce the radiative contribution. Therefore, the essence of the present improvement is to add an opacifying constituent material (specifically,  $\text{TiO}_2$  powder) to the aerogel-matrix composites.

To recapitulate from the cited prior article: A material of the type to which this improvement applies consists of a silica aerogel matrix reinforced with silica fibers and silica powder. The advantage of an aerogel-matrix composite material of this type, relative to neat

aerogels and prior aerogel-matrix composites, lies in formulations and processes that result in superior properties, which include (1) much less shrinkage during a supercritical-drying process employed in producing a typical aerogel, (2) much less shrinkage during exposure to high temperature, and (3) as a result of the reduction in shrinkage, much less or even no cracking.

An opacified aerogel-matrix/silica-powder/silica-fiber composite is synthesized by means of a sol-gel process. Except for the addition of the  $\text{TiO}_2$  powder, the process is almost identical to that used to make the prior, non-opacified version. The first step is to make a silica sol composed of tetramethylorthosilicate, methanol, acetonitrile, and nitric acid through refluxing. The second step is to prepare a solution for casting the composite aerogel: Fumed silica (325-mesh powder having specific surface area of about  $200 \text{ m}^2/\text{g}$ ), silica powder

(particle sizes between 1 and  $2 \mu\text{m}$ ) and  $\text{TiO}_2$  powder (also in particle sizes between 1 and  $2 \mu\text{m}$ ) are suspended in acetonitrile and then the silica sol, water, and ammonium hydroxide base are added to the acetonitrile/powder suspension. After thus preparing the aerogel-casting solution, a piece of silica fiber felt (destined to become the fiber reinforcement in the composite) is placed in a mold. Then the aerogel-casting solution is poured into the mold, where it permeates the silica fiber felt. After the solution has gelled, the casting is transferred to an autoclave filled with acetonitrile, wherein the casting is subjected to supercritical drying at a temperature of  $295 \text{ }^\circ\text{C}$  and pressure of  $5.5 \text{ MPa}$ .

The proportions of the various constituents can be chosen to minimize shrinkage or otherwise adjusted to suit a specific application. In determining the proportion of  $\text{TiO}_2$  to use in a given application, it is necessary to consider that as the concentration of  $\text{TiO}_2$  increases, the opacity increases (and thus the radiative contribution to heat transfer decreases) while the conductive contribution to heat transfer increases. The optimum proportion of  $\text{TiO}_2$  would be the one that minimizes the effective thermal conductivity (the total of the conductive plus radiative contributions), as shown by example in the figure.

*This work was done by Jong-Ah Paik, Jeffrey Sakamoto, Steven Jones, Jean-Pierre Fleurial, Salvador DiStefano, and Bill Nesmith of Caltech for NASA's Jet Propulsion Laboratory. Further information is contained in a TSP (see page 1).*

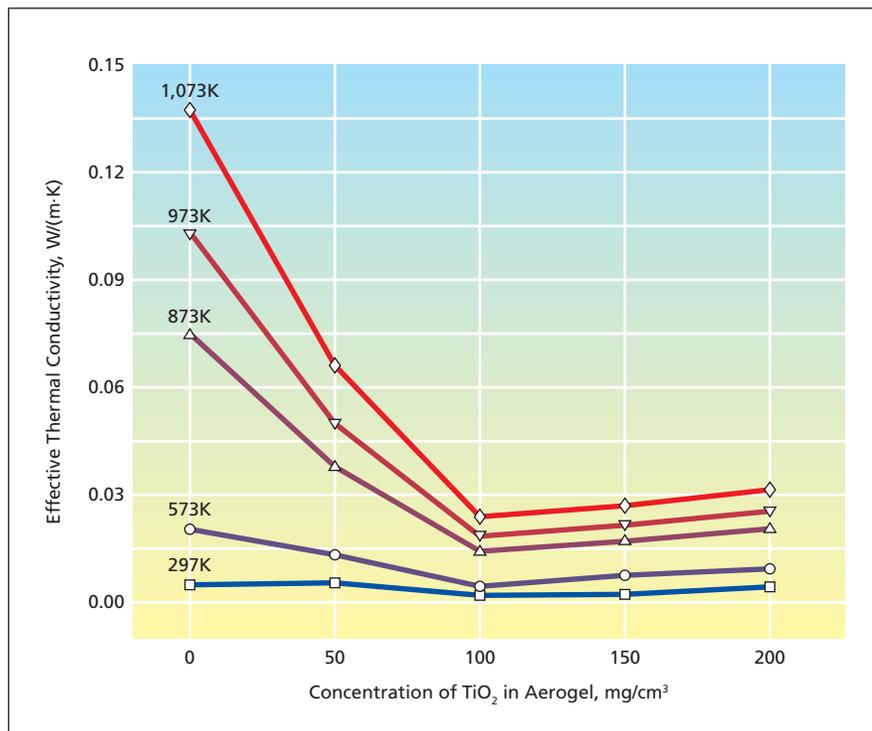
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Effective Thermal Conductivities of silica aerogel samples containing five different proportions of  $\text{SiO}_2$  were measured in a vacuum at temperatures from 297 to 1,073 K. The sample containing  $\text{TiO}_2$  at a concentration of  $100 \text{ mg}/\text{cm}^3$  exhibited the lowest conductivities.