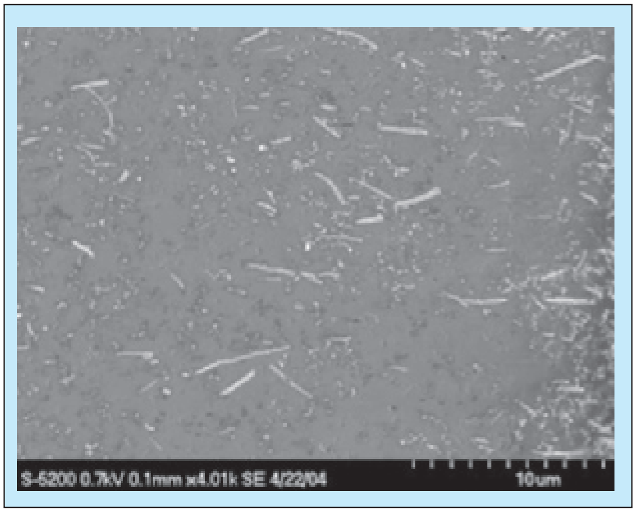
tions ≥20 weight- percent may not be melt-processable.

On the basis of the results from the foregoing characterizations, samples containing 10, 15, and 20 weight-percent of MWCNTs were scaled up to masses of ≈300 g and used to make specimens having dimensions of 10.2 by 15.2 by 0.32 cm. These specimens were molded by (1) injecting the mixtures, at temperatures between 260 and 280 °C, into a tool made of the low-thermal-expansion alloy Invar® and then (2) curing for 1 hour at 371°C. The tool was designed to impart shear during the injection process in an attempt to achieve some alignment of the MWCNTs in the flow direction.

Qualitatively, the moldings from the 10 and 15 weight-percent samples appeared to be good. The moldings were subsequently characterized with respect to thermal, mechanical, and electrical properties. However, as expected from the results of the extrusion experiments, the 20 weight-percent sample could not be injected because of its higher viscosity.

The hardness value of each molded PETI-330/MWCNT specimen was found to be lower than that of the neat resin in the sense that an indenter was found to penetrate to a greater depth or an enhanced plastic deformation of the material was observed. The neat resin specimen was found to be electrically insulating. For the other specimens, the electrical resistivity was found to decrease with increasing concentration of MWCNTs, ranging from 8.86×10^3 Ω/cm for the 10 weight-percent sample to 5.13×10^3 Ω/cm for the 15 weight-percent sample. The thermal conductivities were found to increase with the proportion of MWCNTs, ranging from 0.219 W/(m·K) for the neat resin specimen to 0.577 W/(m·K) for the 10 weight-percent specimen and 0.777 W/(m·K) for the 15 weight-percent specimen. This trend in thermal conductivity suggests that nanotubes form networks in the polymer matrices that conduct heat, but not to the extent expected based on the high thermal conductivity of the MWCNTs.



MWCNTs Were Substantially Aligned along the flow direction after extrusion, as shown in this high-resolution scanning electron micrograph.

Upon machining of the specimens to prepare them for mechanical tests, voids were observed. Unfortunately, these voids made the samples unsuitable for determination of mechanical properties. Notwithstanding the present lack of data on mechanical properties, the electrical and thermal properties and processing characteristics of these materials offer significant potential for applications in which multifunctionality may be required.

This work was done by John W. Connell, Joseph G. Smith, Emilie J. Siochi, and Dennis C. Working of Langley Research Center; Jim M. Criss of M&P Technologies; Kent A. Watson and Donavon M. Delozier of the National Institute of Aerospace; and Sayata Ghose of the National Research Council. Further information is contained in a TSP (see page 1).
LAR-17082-1

Multilayer Impregnated Fibrous Thermal Insulation Tiles

Temperature rises are limited by transpiration cooling.

Ames Research Center, Moffett Field, California

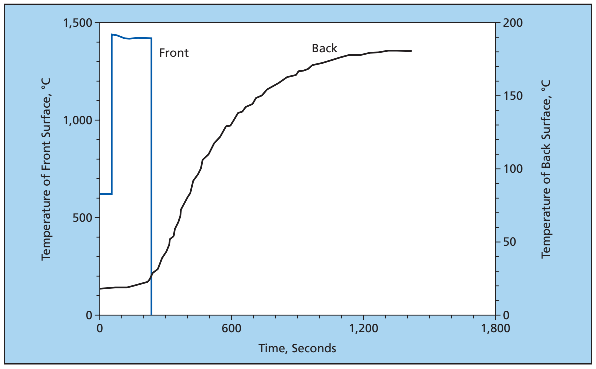
The term “secondary polymer layered impregnated tile” (“SPLIT”) denotes a type of ablative composite-material thermal-insulation tiles having engineered, spatially non-uniform compositions. The term “secondary” refers to the fact that each tile contains at least two polymer layers wherein endothermic reactions absorb considerable amounts of heat, thereby helping to prevent overheating of an underlying structure. These tiles were invented to afford lighter-weight alternatives to the reusable thermal-insulation materials heretofore variously used or considered for use in protecting the space shuttles and other spacecraft from intense atmospheric-entry heating. Tiles of this type could also be useful on Earth as relatively lightweight components of fire-retardant structures.

The SPLIT concept admits to so many different combinations of constituent materials, spatial distributions of the materials, and fabrication processes, that it is not possible to even list, much less summarize or describe all of them. Instead, a representative example must serve to illustrate the main principles. The starting material for fabricating a typical SPLIT is a porous substrate, having a void volume fraction of about 90 percent, that comprises a rigid tile or fabric made from any of a large variety of carbon fibers and/or ceramics fibers. The fiber composition can be the same throughout the thickness or can be graded: for example, it can differ among front, middle, and rear layers.

The front layer, which is the one to be exposed directly to intense heating, is typically impregnated with a thermosetting resin (e.g., a phenolic or a silicone). This layer becomes the first line of defense against intense heating: a large amount of heat is absorbed in the pyrolysis of the front polymer layer and is dissipated to the environment through a combination of outflow of the pyrolysis gas, and thermal radiation from the char layer formed in the pyrolysis. The outflow of the pyrolysis gas also provides further protection against heating by blocking the inflow of hot ambient gas.

The middle layer (if any) is typically not impregnated. The back layer is the one to be placed in contact or proximity to the structure to be protected. The back layer is initially impregnated with a thermoplastic polymer (the secondary polymer) in solution and the solvent is allowed to evaporate, so that the fibers in the back layer become coated with the thermoplastic but the layer retains substantial porosity. The secondary polymer is chosen to be one that pyrolizes completely or nearly completely to gaseous products (i.e., leaving little or no solid residue), at a temperature much lower than the pyrolysis temperature of the front layer. For example, polystyrene and poly (methyl methacrylate) decompose at temperatures between 350 and 450 °C.

Eventually, some heat penetrates the front layer and diffuses to the back layer, where the lower-temperature pyrolysis reaction of the secondary polymer retards the transfer of heat to the structure to be protected. The pyrolysis gas from the secondary polymer escapes through the middle (if any) and front layers, thereby effectively preventing excessive heating of the underlying structure through a combination of transpiration cooling and blockage of inflow of hot ambient gas. With suitable choice of materials and processing, it is possible to delay the onset of heating of the back surface and limit the temperature rise of the back surface (see figure) to a value low enough to protect the underlying structure.



A **Pulse of Heat** was applied, by an arc jet in a partial vacuum, to the front surface of a 2.73-cm-thick specimen impregnated with a silicone in the front layer and poly (methyl methacrylate) in the back layer. The rear-surface temperature rise, measured by use of thermocouples, was limited to a range that would be safe for an underlying aluminum structure or for most composite-material structures.

This work was done by Huy K. Tran, Daniel J. Rasky, and Christine E. Szalai of Ames Research Center; Ming-ta S. Hsu of HC Chem Research and Services Corp.; and Joseph A. Carroll of Tether Applications. Further information is contained in a TSP (see page 1).

This invention has been patented by NASA (U.S. Patent No. 6,955,853 B1).

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-14165-1.