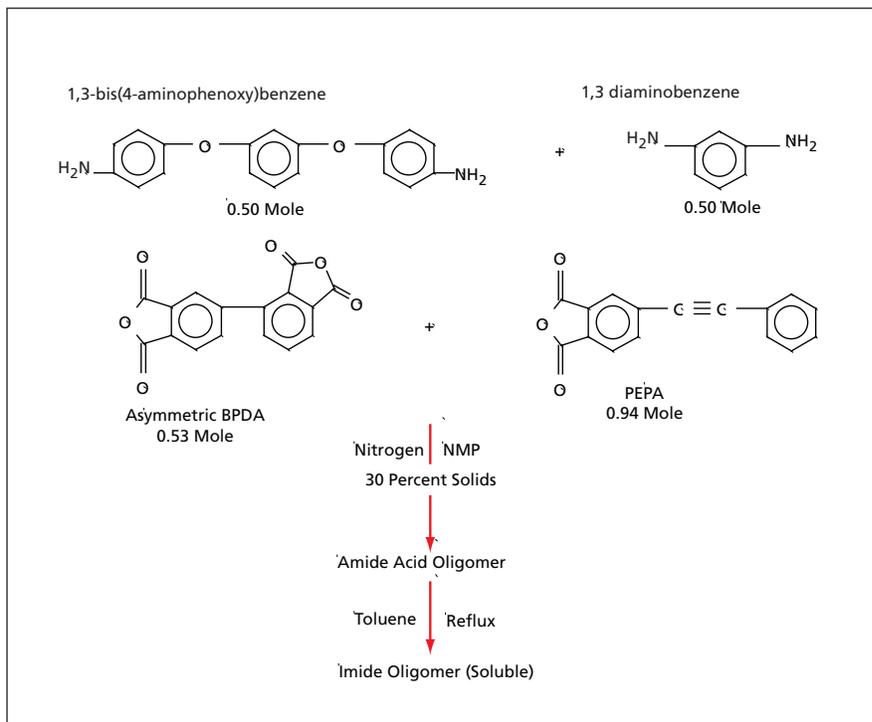


shown in the figure. First, 0.53 mole of 2,3,3',4'-biphenyltetracarboxylic dianhydride (BPDA) and 0.94 mole of PEPA were reacted with 0.50 mole of 1,3-bis(4-aminophenoxy)benzene and 0.50 mole of 1,3-diaminobenzene in NMP to form the phenylethynyl-terminated amide acid oligomer. Then, by azeotropic distillation using toluene, the phenylethynyl-terminated amide acid oligomer was converted to the PETI oligomer having a calculated repeat unit molecular weight of 750 g/mole. The imide oligomer was isolated, washed well with water, and dried. The melt viscosity of the imide oligomer was found to be 0.6 poise initially at a temperature of 280 °C and 0.9 poise after 2 hours at that temperature.

The imide oligomer was degassed, then injected, at a temperature of 280 °C, into a preform made of a fabric of T650 carbon fibers in 8-harness satin weave. The oligomer was then cured for 1 hour at a temperature of 371 °C and a pressure of 200 psi (≈ 1.4 MPa). The resulting quasi-isotropic composite was found to have an open-hole compressive strength of 39 kpsi (≈ 0.27 GPa) at a temperature of 23 °C and 29 kpsi (≈ 0.2 GPa) at a temperature of 288 °C. After aging for 1,000 hours at 288 °C in air, the open-hole compressive strength was found to be 32 kpsi (≈ 0.22 GPa) at 23 °C. The unnotched compressive strength was found to be 75 kpsi (≈ 0.52 GPa) at 23 °C. The composite exhibited no microcracking, either



PETI-330 is Synthesized in this sequence of reactions.

immediately after cure or after 200 subsequent thermal cycles from -55 °C to $+288$ °C. The T_g of the composite was found to be 330 °C.

Another such PETI oligomer, denoted PETI-375, was synthesized and used to make an RTM composite with a carbon-fiber cloth of the type described above. The open-hole compressive strength of this composite was found to be 43.5 kpsi

(≈ 0.3 GPa) at 23 °C, 34.8 kpsi (≈ 0.24 GPa) at 288 °C, and 32.2 kpsi (≈ 0.22 GPa) at 316 °C. The T_g of the composite was found to be 370 °C.

This work was done by John W. Connell, Joseph G. Smith, Jr., and Paul M. Hergenrother of Langley Research Center. Further information is contained in a TSP (see page 1). LAR-15834-1

Stable Polyimides for Terrestrial and Space Uses

Low-color films exhibit high resistance to space environment.

Langley Research Center, Hampton, Virginia

Polyimides of a recently developed type have an attractive combination of properties, including low solar absorptivity (manifested as low color) when cast into thin films, resistance to atomic oxygen and ultraviolet radiation, solubility in organic solvents, high glass-transition temperatures (T_g s), and high thermal stability. The focus of the development work was on polymers that can endure the space environment and that have specific combinations of properties for use on Gossamer spacecraft. Because of their unique combination of properties, these polymers are also expected to find use in a variety of other applications on Earth as well as in space.

Examples of other space applications include membranes on antennas, second-surface mirrors, thermal optical coatings, and multilayer thermal insulation. For both terrestrial and space applications, these polyimides can be processed into various forms, including films, fibers, foams, threads, adhesives, and coatings.

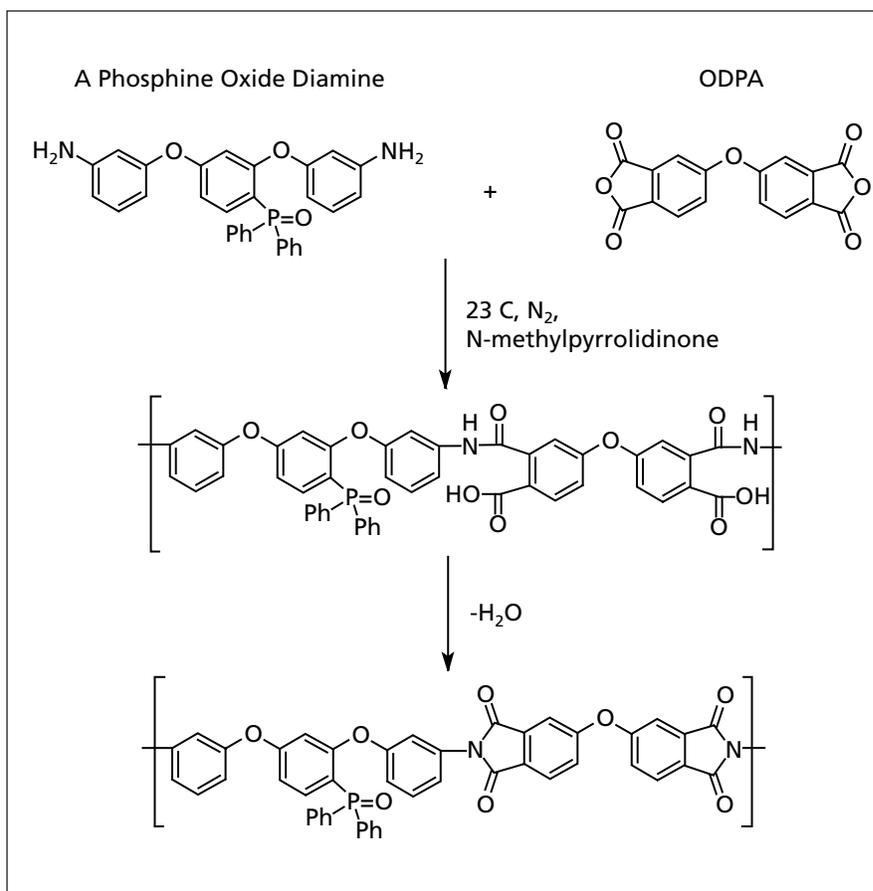
In preparation for synthesizing a polyimide of this type, one makes [2,4-bis(3-aminophenoxy)phenyl]diphosphine oxide (hereafter denoted "the phosphine oxide diamine"), which is a novel aromatic diamine. In an experiment, this aromatic diamine was made from commercially available starting ma-

terials in a two-step process in relatively high yield. Then, in experimental syntheses of polyimides of this type, the phosphine oxide diamine was reacted with aromatic dianhydrides in polar aprotic solvents. In other experiments, copolyimides were synthesized by use of the phosphine oxide diamine in combination with other commercially available aromatic diamines. These various syntheses made it possible to tailor the polymers to reduce costs and obtain hitherto unavailable combinations of properties.

The figure illustrates the example of one synthesis, in which the phosphine oxide diamine was reacted with oxydiphthalic anhydride (ODPA) in N-methyl-

pyrrolidinone (a polar aprotic solvent) at 20 weight percent to form a polyamide acid having an inherent viscosity of 1.01 dL/g. The polyamide acid was converted to the polyimide by reaction with a mixture of acetic anhydride and pyridine. The polyimide was isolated, dried, and dissolved in N,N-dimethylacetamide, then thin films were cast and stage-dried at temperatures up to 250 °C for one hour. A 1.6-mil (≈ 0.04 -mm)-thick film was found to have 85-percent transparency at a wavelength of 500 nm, solar absorptivity of 0.06, thermal emissivity of 0.56, T_g of 212 °C, 5-percent loss of weight upon heating in air at a rate of 2.5 °C/minute up to 461 °C, and the following tensile properties at a temperature of 23 °C: strength of 14.7 kpsi (≈ 101 MPa), modulus of 410 kpsi (≈ 2.8 GPa), and elongation of 4.7 percent. A film subjected to atomic oxygen in a ground-based experiment that simulated 6 months in a low orbit around the Earth exhibited 81-percent transmission at 500 nm, only 1.74-percent of mass loss, and excellent retention of properties in general.

In another example, a copolyimide was made from the reaction of ODPA with 75 mole percent of the phosphine oxide diamine and 25 mole percent of 3,4'-oxydianiline. A 1-mil (≈ 0.025 -mm)-thick film of this copolyimide was found to have 88-percent transparency at a wavelength of 500 nm, solar absorptivity of 0.06, thermal emissivity of 0.38, T_g of 218 °C, and the following tensile properties at a temperature of 23 °C: strength of 14.7 kpsi (≈ 101 MPa), modulus of 460 kpsi (≈ 3.2 GPa), and elongation of 6.3 percent.



A Phosphine Oxide Diamine Was Reacted With ODPA to obtain a polyimide that strongly resists degradation under space conditions.

This work was done by John W. Connell, Joseph G. Smith, Jr., and Paul M. Hergenrother of Langley Research Center, and Kent A. Watson and Craig M. Thompson of the National Institute of Aerospace. The technology is covered in Patent Application No. US

2003/0045670 A1 and NASA Case No. LAR 16176-1 and was exclusively licensed to Triton Systems, Inc. Requests for information on this technology should be directed to Norm Rice at nrice@tritonsys.com. LAR-16176-1

Low-Density, Aerogel-Filled Thermal-Insulation Tiles

Lyndon B. Johnson Space Center, Houston, Texas

Aerogel fillings have been investigated in a continuing effort to develop low-density thermal-insulation tiles that, relative to prior such tiles, have greater dimensional stability (especially less shrinkage), equal or lower thermal conductivity, and greater strength and durability. In preparation for laboratory tests of dimensional and thermal stability, prototypes of aerogel-filled versions of recently developed low-density tiles have been fabricated by impregnating such tiles to various depths with aerogel formations ranging in density from 1.5 to 5.6 lb/ft³ (about 53 to 200 kg/m³). Results available at the time of report-

ing the information for this article showed that the thermal-insulation properties of the partially or fully aerogel-impregnated tiles were equivalent or superior to those of the corresponding non-impregnated tiles and that the partially impregnated tiles exhibited minimal (<1.5 percent) shrinkage after multiple exposures at a temperature of 2,300 °F (1,260 °C). Latest developments have shown that tiles containing aerogels at the higher end of the density range are stable after multiple exposures at the said temperature.

This work was done by Maryann Santos, Vann Heng, Alfred Zinn, Andrea Barney,

Kris Oka, and Michael Droege of the Boeing Co. for Johnson Space Center. For further information, contact the Johnson Technology Transfer Office at (281) 483-3809.

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act {42 U.S.C. 2457(f)} to The Boeing Company. Inquiries concerning licenses for its commercial development should be addressed to:

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Huntington Beach, CA 92657-2099

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