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Thermally Stable Polyimides from Solutions of Monomeric Reactants

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NASA TECH BRIEF

Lewis Research Center

Polyimides synthesized from a mixture of three types of monomers in a suitable solvent overcome the problems of limited solubility and poor shelf-life. Polyimide resins, noted for their thermal stability and high temperature strength were previously synthesized utilizing solutions of low molecular weight polyamide-acid prepolymers, end-capped with reactive alicyclic rings. These prepolymers polymerize at elevated temperatures into thermally stable polyimides, with a minimal evolution of volatile by-products. However, such solutions exhibited limited solubility and poor shelf-life, as evidenced by precipitation and/or gel formation. In contrast, the monomer solutions have excellent shelf-life: increased solubility (solutions containing 60% to 70% are easily obtainable): lower cost because the monomers can be shipped in powder form and the reaction to form the polyamide-acid prepolymer is omitted: lower solution viscosity: and easier handling.

The monomers are (a) the dialkyl ester of an aromatic tetrecarboxylic acid (synthesized from the corresponding dianhydride of the tetracarboxylic acid). (b) an aromatic diamine, and (c) the monoalkyl ester of an alicyclic dicarboxylic acid (synthesized from the corresponding anhydride of the dicarboxylic acid). The molar ratio of a: b: c: is n: (n+1): 2. The monomers are nonreactive at room temperature but react at elevated temperatures to form prepolymers, and cure at higher temperatures forming thermally stable polyimides.

Fiber-reinforced composites are made by impregnating fibers with solutions of the monomers in commonly used solvents such as N,N-dimethylformamide with a solids content of 60% to 70%. Following the removal of excess solvent at 120°C, the prepreg is Bstaged at 200°C for 1 to 2 hours. The prepreg is then stacked and molded at 325°C under relatively low pressure to form a thermally stable, essentially void-free composite. Composites made from these monomeric solutions have properties essentially identical to composites made from the corresponding freshly prepared alicyclic terminated polyamide-acid prepolymer.

Notes:

- 1. This synthesis process has potential application to other high temperature resin systems.
- 2. Such thermally stable polyimides can be used for constructing aircraft components, such as compressor blades, and other composite structures.
- The following documentation may be obtained from: National Technical Information Service Springfield, Virginia 22151 Single document price \$3.00 (or microfiche \$0.95) Reference: NASA TM-X-67803 (N71-23367),

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4. Technical questions may be directed to: Technology Utilization Officer Lewis Research Center 21000 Brookpark Road Cleveland, Ohio 44135 Reference: B71-10442

Patent status:

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> Patent Counsel Mail Stop 500-311 Lewis Research Center 21000 Brookpark Road Cleveland, Ohio 44135

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