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NASA TECH BRIEF

Ames Research Center

Polyimide Fiber-Glass Composite Resists High Temperatures

Composites of fiber glass and a polyimide resin synthesized from bismaleimide have superior strength and oxidation resistance at elevated temperatures when compared with similar composites prepared with epoxy or silicone polymers of similar cost. The polyimide is obtained from the easily-controlled thermal polymerization of simple bismaleimide derivatives of aromatic diamines. The polyimide synthesis technique and the processing method yield essentially void-free fiber-glass reinforced composites.

The monomer, 4,4′-bismaleimido-diphenylmethane, is synthesized by placing 2 moles of methylene dianiline, 2 moles of sodium acetate, and one liter of N,N-dimethyl formamide in a reaction flask. Four moles of maleic anhydride are added slowly to the mixture; the flask is placed in an ice bath during the addition of maleic anhydride to maintain a temperature of 50° to 60°C. Four moles of acetic anhydride are then added to the mixture and the temperature is held at 50° to 60°C for 45 minutes after the addition. The product is then precipitated by pouring the contents of the reaction flask into 20 liters of distilled water; after filtration, the precipitate is dried overnight in a circulating-air oven at 50°C. The yield of monomer is 92 to 93 percent.

The monomer is gradually polymerized by raising the temperature in stages as the molecular weight increases because of reaction at the double bond; the degree of polymerization is determined by acetone extraction. Molded parts of neat resin samples of 4, 4′-bismaleimido-diphenylmethane are made by staging the monomer at 150°C for 1½ hours. The staged powder is molded at 34 MN/m^2 for 2 hours at 240°C.

Test laminates may be prepared by coating woven glass cloth with a varnish prepared by dissolving monomeric 4,4′-bismaleimido-diphenylmethane in 1-methyl-2-pyrrolidone solvent at 40% resin solids (viscosity 50 centipoises). After coating, the glass cloth is dried for 15 minutes at 79°C and staged 20 minutes at 150°C; after staging, almost no solvent is retained and resin flow under pressure is about 15 percent.

After the prepreg is prepared, it is cut, stacked, and pressed between aluminum plates and a polytetrafluoroethylene film release sheet. The "buildup" is placed in a cold press and heated at a rate of 3°C/min at 0.689 MN/m^2 to 240°C and held at 240°C for two hours. Laminate thicknesses of 8 to 50 plies have been prepared; resin content varies from 21 to 40 percent (wt.) with a corresponding range in flexural strength of 199 MN/m^2 to 377 MN/m^2.

In comparative tests for retention of flexural strength after 15 minutes at 260°C, the polyimide/glass laminate retained 78.5% of flexural strength, but an epoxy/glass retained only 15.3%, and a silicone/glass 24.6%. Sixty percent of flexural strength is retained by the polyimide/glass laminate after 100 hours in air at 260°C, but the laminate undergoes oxidative degradation (as shown by weight-loss measurements) so that only 26 percent of the original flexural strength is retained after 500 hours.

(continued overleaf)

Note:

Requests for further information may be directed to:

Technology Utilization Officer
Ames Research Center
Moffett Field, California 94035
Reference: TSP 73-10505

Patent status:

NASA has decided not to apply for a patent.

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