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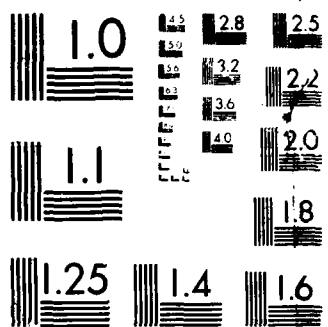
A STUDY OF THE CRITICAL FACTORS CONTROLLING THE
SYNTHESIS OF CERAMIC MATR. (U) UNITED TECHNOLOGIES
RESEARCH CENTER EAST HARTFORD CT J R STRIFE ET AL.
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A STUDY OF THE CRITICAL FACTORS CONTROLLING THE SYNTHESIS OF
CERAMIC MATRIX COMPOSITES FROM PRECERAMIC POLYMERS

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BACKGROUND

Several DOD technology thrusts such as the Integrated High Performance Integrated Turbine Engine Technology Program and the National Aerospace Plane depend on the development of high temperature low density materials. The development of fiber reinforced ceramics is projected to play a major role in this endeavor because of their high temperature capacity and superior mechanical properties on a density normalized basis at elevated temperature. Fiber reinforced ceramics have been processed to date primarily by hot pressing tape materials or by the chemical vapor infiltration of fibrous preforms. More recently, the conversion of preceramic polymers as a matrix synthesis process is being considered. This approach offers several advantages relative to other processes for the synthesis of ceramic composites. First, a range of useful composite matrices can be conceived based on carbide, oxide, and nitride precursors. Secondly, by using a polymer approach, fillers and other additives may be easily incorporated into the composite enabling the synthesis of multi-phase compositions. Thirdly, reproducible matrix characteristics can be anticipated by controlling polymer chemistry and composite fabrication cycles. Finally, the utilization of preceramic polymers is adaptable to complex shape configurations, and is less dependent on bulk thickness considerations.

RESEARCH OBJECTIVES

There are two critical science issues which must be addressed to successfully synthesize useful fiber reinforced ceramics from preceramic polymers. The first issue concerns establishing conversion methodology which is adaptable to composite synthesis. UTRC's approach in attacking this issue is to dissect an overall composite fabrication approach into a series of steps which can then be addressed by performing fundamental studies of the changes in polymer structure and physical properties as a function of temperature and time. The second key issue concerns the optimization of the interface characteristics between fiber and matrix to provide the necessary thermochemical stability at high temperature, and to influence the composite failure process so that strengthening and toughening are achieved simultaneously. The approach addressing this issue involves coupling microstructural observations and measurements of fiber/matrix bond strength with observed composite failure modes.

The specific objectives in the first year of the program are to (1) perform fundamental studies of the changes in polymer structure and physical properties of a polycarbosilane preceramic polymer as a function of temperature to derive synthesis methodology for SiC matrix composites, (2) investigate the role of interface modification in creating tough carbon fiber reinforced SiC matrix composites.

RESEARCH PROGRESS

Preceramic Polymer Characterization

Composite synthesis via polymer conversion has been structured as a three step process involving (1) preform fabrication, (2) pyrolysis and heat treatment, (3) densification. The fabrication of dense, well bonded preforms requires detailed knowledge of changes in polymer structure and viscoelastic characteristics as a function of time and temperature. The kinetics of subsequent pyrolysis and associated evolution of gaseous species must be well characterized to provide a structurally coherent composite body for further densification. Successful densification is judged by the relationship of physical and structural changes with

mechanical performance. Research in the first six months has focused on characterization of a polycarbosilane resin synthesized from mixtures of vinyl and methyl silanes as a precursor for SiC matrix composites. This resin was selected since it contains carbosilane ($\text{SiCH}_2\text{CH}_2\text{-Si}$) linkages as processed, thus requiring less thermal rearrangement and volatile loss prior to pyrolysis. This reduces the complexity of processing parameters and provides better reproducibility than for traditional polysilane precursors which contain high percentages of Si-Si linkages which must undergo rearrangement to carbosilanes prior to pyrolysis.

Two lots of the vinyl derived polycarbosilane resin supplied by Union Carbide have been characterized with regard to their viscoelastic behavior and polymer structure as a function of time, temperature, and environment. Activation energies for polymer rearrangement and pyrolysis initiation have been calculated and correlated with changes in the polymer structure as a function of heating rate and environment. It has been found that the temperature dependence of the resin viscosity varies significantly from lot to lot, but that thermal treatments can be defined to normalize resin viscosity for composite processing. Polymer rearrangement is not strongly affected by heating rate and occurs over a well-defined temperature range. Exposure of the resin to laboratory air environment as opposed to inert gas atmosphere during processing results in significant changes in viscoelastic properties and polymer rearrangement by absorption of oxygen and/or water vapor into the polymer structure. This effect can be minimized by rapid heating in air to the polymer rearrangement temperature.

Preliminary pyrolysis studies on specimens heated between 800°C and 1500°C indicate formation of a near amorphous SiC after heating to 800°C with the degree of crystallinity steadily increasing with increased heat treatment temperature. Heating at 1500°C produces a disordered β -SiC with some residual glass phases. Weight loss is approximately 35% after slow heating to 800°C with additional 6-7% loss on heating to 1500°C.

The implication from these studies is that a reproducible composite synthesis procedure can be defined for the vinyl-derived polycarbosilane resin with appropriate resin characterization and control of environmental factors.

Composite Synthesis Studies

A carbon fiber reinforced SiC composite synthesis cycle was defined based on the preliminary resin characterization data. The cycle consisted of thermally treating T-300 carbon fiber fabric impregnated with the resin for 1 hr at 120°C, press molding in a vacuum bag while heating to 180°C, and pyrolyzing the free standing panels by heating to 800°C over a 48 hr period. Following this cycle, the composite density was 1.28 gm/cm³. Panels were pressure reimpregnated at 100 psi, cured to 250°C, heated to 800°C over 20 hrs, and then to 1500°C. An average density of 1.37 gm/cm³ was obtained. Porosity determination by mercury intrusion indicated a residual pore size distribution of 10 μm to <0.01 μm . The interlaminar shear strength of these panels was only 170 psi indicating that a fiber surface treatment coupled with higher impregnation pressures will be required to increase the composite density and mechanical performance. Experiments have shown that addition of a wetting agent can improve the fiber/matrix bond strength and increase the interlaminar shear strength to 2000 psi.

FUTURE PLAN

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In the remainder of the first year of the research program, polymer characterization studies will focus on differences associated with air or inert gas processing on the pyrolysis and conversion process to SiC. The net result of these studies coupled with the work reported here will provide a general characterization methodology for processing preceramic polymers as well as a specific approach for optimizing the processing of the vinyl derived polycarbosilane resin.

Concurrent with the polymer characterization studies, composite synthesis will proceed with emphasis on varying the fiber/matrix interface character. This will be accomplished utilizing fiber wetting agents, fiber coatings, and through reactive particulate additions to the resin. Assessments of fiber/matrix bond strength will be made and correlated with a measure of the composite strength and toughness characteristics. These composites will be processed utilizing the simple procedure outlined previously.

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The proposed plan for the second year research program incorporates further investigation of the C/SiC composite system as well as application of the characterization methodology to a different composite system. C/SiC composites will be fabricated which couple the optimized resin processing procedures and optimum fiber surface treatments as defined in the first year of the program. The composites will be evaluated so as to allow comparison with baseline materials processed in the first year. Research will then commence to evaluate a second composite system incorporating a fiber other than carbon in an oxide matrix. Potential fiber/matrix combinations will be considered based on availability of preceramic polymers and an assessment of fiber/matrix thermodynamic stability. The combination of alumina fiber with a silica matrix where the silica is derived from a silicone precursor represents a candidate composite system. An assessment of the generic nature of the synthesis methodology determined for the SiC precursor as compared to the oxide precursor will be made.

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