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Polyarylacetylene-Matrix Composites for Solid Rocket Motor Components

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ABSTRACT

The fabrication process for an ablative material for solid rocket motor applications, carbon-fabric-reinforced polyarylacetylene (PAA) resin composite, is detailed. Data concerning the physical, mechanical, and ablative performance of this material are presented. PAA is a highly cross-linked aromatic polymer that contains only carbon and hydrogen. Its principal advantage is its high char yield (~ 90 percent). For the resin synthesis, a controllable, low-temperature technique that produces a soluble, easy-to-process prepolymer was developed. Polymer chain modifications incorporated during prepolymer synthesis to improve ductility and toughness of the resin were demonstrated. Utilizing these modifications, T300/PAA composites with high char yield and good mechanical properties were fabricated using standard prepregging techniques. Comparative ablation tests of T300/PAA and the standard phenolic ablative insulator material (FM 5055) were performed with a CO_2 *plasma jet at NASA Marshall Space Flight Center. The results indicate that T300/PAA undergoes less weight loss and erosion than FM 5055. Also, whereas many of the FM 5055 samples exhibited unstable ablation resulting in pocketing, there was no pocketing in any of the T300/PAA samples.*

INTRODUCTION

Currently, carbon-phenolic, graphite-phenolic, and silica-phenolic materials are used for ablative and insulative nozzle components of solid rocket motors. Ideally, these materials should exhibit repeatable, predictable, uniform recession rates at the flame surface and should thermally insulate the backside structure as well. In operation, however, the ablation behavior of phenolic matrix materials has been variable and unstable, posing doubts about their reliability. Although no system failures have been attributed to these phenolic materials, some of the more serious incidents have led to costly redesign and recovery efforts.

The best known example of unstable ablation is the "pocketing" discovered during post-flight inspection of the carbon-phenolic nozzle inlet recovered from the STS-8A booster.[1] Localized deep pockets in a regular array were found. In the worst region, only a few seconds of burn-through life margin remained. NASA conducted a major materials investigation and redesign effort to avoid recurrence and initiated a number of raw material and component process controls. However, incidents of less severe pocketing have occurred in subsequent STS flights. Other examples of unexpected and unpredictable ablation are incidents of erosion pits, shallow craters, and grooves. Some solid motor test anomalies have been attributed to the large amounts of evolved pyrolysis gases generated by the phenolic matrix materials. Inadequate management of pyrolysis gas can lead to extremely high pressures within the composite materials, and the forces exerted by such pressures can exceed the design capability of nozzle components.

These problems partially result from the hydrophilic nature of phenolic resins, which readily absorb moisture. In addition, the resins cure by a condensation reaction in which water is a by-product. The condensation curing process results in diffusion paths that allow for the rapid reabsorption of moisture. The absorbed moisture acts as a plasticizer, which significantly reduces the glass transition temperature (Tg) of the resin and lowers its interlaminar load-carrying capability. During ablative heating, the moisture is vaporized and causes pressure to build up within the composite, resulting in an array of internal delaminations (ply lifts) that extend to the surface, causing local sloughing and uneven ablation.[2] That pressure is in addition to the pressure caused by the large amount of volatile hydrocarbons from pyrolysis of the resin itself, which has a char yield of only about 50 percent (Figure 1). These hydrocarbons can be thermally cracked, resulting in carbon deposition and plugging of gas-diffusion paths, and hence greater internal pressure buildup. Pressure in the pores can also cause tensile failure of the yarns, leading to ejection of chunks of material and formation of localized pockets.

This paper details the fabrication process for an alternate ablative material, carbon-fabric-reinforced polyarylacetylene (PAA) resin, and presents data concerning the physical, mechanical, and ablative performance of this material. PAA is a highly cross-linked aromatic polymer that contains only carbon and hydrogen. Its principal advantage is that only about 10 wt percent is volatilized when the polymer is heated to high temperatures in an inert environment; the remaining 90 percent is carbon char (Figure 1). PAA resin absorbs much less moisture (0.1 - 0.2 wt percent) than phenolic (5 - 10 wt per-

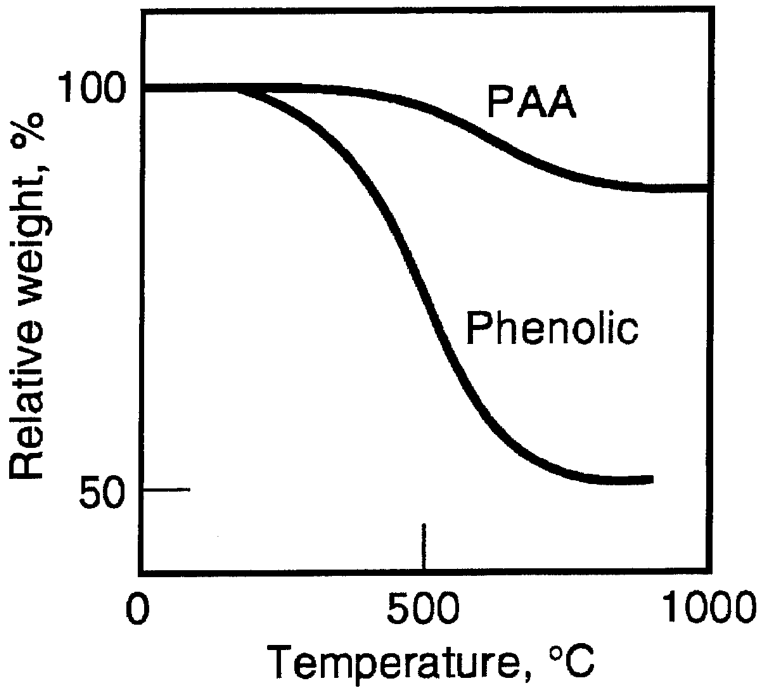

Figure 1. Weight loss in a nitrogen environment of neat PAA and phenolic resin as a function of temperature (heating rate: 5°C/minute).

cent) and cures by an addition reaction that has no by-products. Perhaps even more important is the fact that PAA has a very high Tg (above the temperature of decomposition) that is unaffected by moisture. Its char yield of 90 percent means that far less volatile material is generated and that minimal shrinkage is associated with pyrolysis.

The composition and relative amounts of gases released during pyrolysis of PAA neat resins has been measured using gas chromatography mass spectrometry.[3] The dominant gas evolved by PAA is hydrogen, the amount of which peaks at 800°C (Figure 2a). In contrast, the gaseous pyrolysis products of phenolic resin are dominated by high-molecular-weight hydrocarbons, as well as oxygen-containing hydrocarbons, that peak at the lower temperature of 500°C[4] (Figure 2b). Since most of the PAA pyrolysis gas is hydrogen, hydrocarbon cracking and pore plugging are greatly diminished.

PAA is a polyphenylene[5-6] that results from the cyclotrimerization reaction of diethynylbenzene (DEB), as shown in Figure 3. DEB was first polymerized in the late 1950s as part of a broad search for polymers with high char yield.[7-8] During cure, that early formulation underwent severe shrinkage and released large amounts of heat (exothermic reaction). Such processing difficulties limited its practical application. In the early 1960s, advances in catalyst technology[9] made possible techniques for reducing both the exotherm and extent of shrinkage during polymer curing. However, those techniques were not applied to PAA until Hercules patented a process for the production of PAA from DEB for use as a high temperature molding compound.[10-12] This resin was designated HA 43 by Hercules and was made available in the late 1970s for evaluation by the technical community. Researchers at The Aerospace Corp. used it to produce a carbon-fabric-reinforced ablative thermal protection system. Although these composites ablated satisfactorily, they were brittle and had poor structural integrity.

We found that these deficiencies could be overcome by a controllable, low-temperature prepolymerization technique that produces a soluble, easy-to-process prepolymer. Polymer chain modifications incorporated during prepolymer synthesis to improve ductility and decrease brittleness were demonstrated, and these modifications resulted in composites with high char yield and good mechanical properties. This paper presents the fabrication procedures for these materials, thermophysical characterization at Southern Research Institute, and comparative arc jet ablation testing at NASA Marshall Space Flight Center (MSFC).

EXPERIMENTAL

Figure 3 shows the technique for fabrication of carbon-fiber-reinforced PAA composites. First, meta-DEB, para-DEB, and phenylacetylene are cyclotrimerized in a methyl ethyl ketone (MEK) solution to produce a low-molecular-weight prepolymer. During cyclotrimerization, three actylene groups (from three different monomer molecules) react together in the presence of a catalyst to form a benzene ring. Woven carbon fabric is then coated with the prepolymer to produce a prepreg. This prepreg can be wrapped, wound, or stacked and formed into the desired shape by conventional lay-up procedures, and the polymer can be cured under moderate pressure (0.7 - 1.4 MPa) at a relatively low temperature (150-200°C).

The cyclotrimerization step is designed to produce a soluble solid that softens at a temperature lower than the onset of cure. Cyclotrimerization is important because it produces resonance-stabilized aromatic rings that enhance both the thermal stability of the system and the high char yield on pyrolysis. The overall reaction is exothermic; however, part of the heat of polymerization is liberated during this step, resulting in a milder and more controllable exotherm during final cure. In addition, part of the shrinkage that accompanies curing occurs during cyclotrimerization. Successful PAA composite processing requires that the prepolymerization be stopped when the reaction has proceeded to the required point. Too little reaction negates the benefits of the cyclotrimerization step; on the other hand, a reaction permitted to proceed too far forms a prepolymer that is too viscous to process.

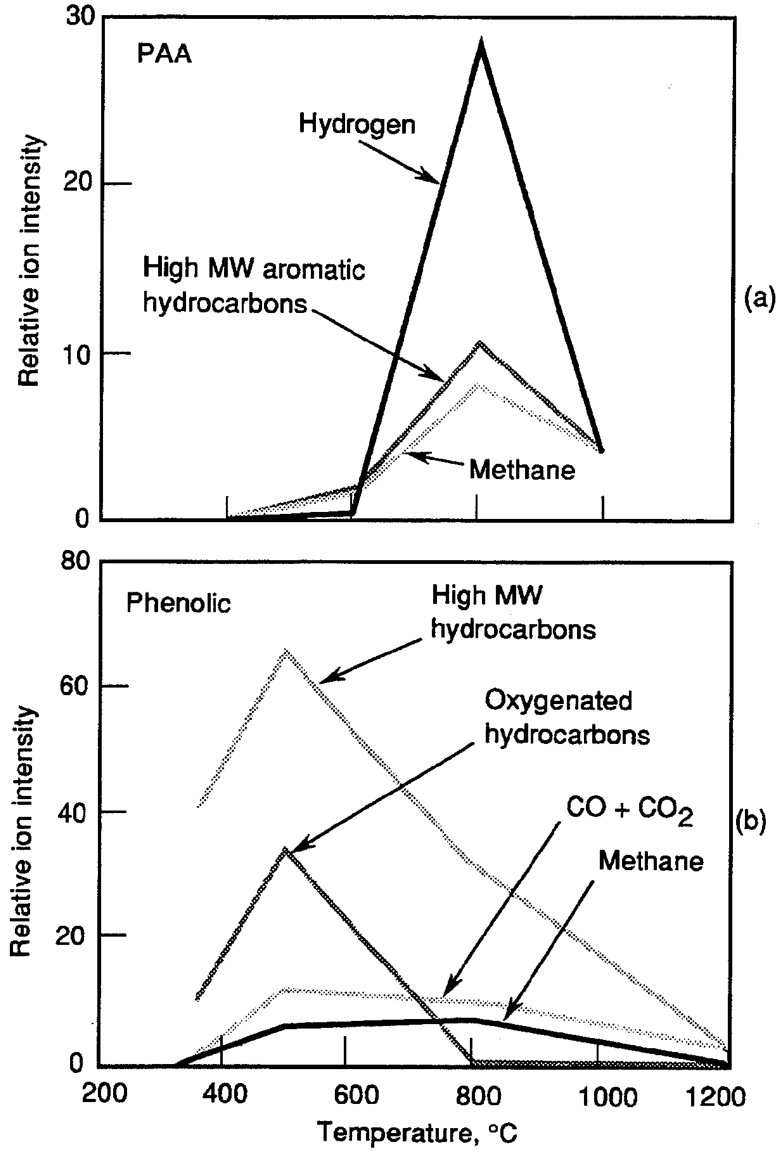

Figure 2. Composition and relative amounts of gases released during pyrolysis of neat resin, measured by gas chromatography mass spectrometry: a) PAA (Ref. 4) and b) phenolic (Ref. 3).

In the next step of the composite fabrication, woven fabric is impregnated with the prepolymer solution, and the MEK solvent is evaporated, leaving a prepreg fabric. For good composite fabrication, this prepreg must be easy to handle: it must be easy to drape over curved surfaces and be tacky enough to keep the prepreg layers from moving in relationship to each other. Also, the resin must soften and flow before curing to completely impregnate and consolidate the laminated preform. We have successfully developed a procedure for controlling drape, tackiness, and flow of the prepreg. This is most effectively accomplished by copolymerizing phenylacetylene with DEB. Since phenylacetylene has only a single active acetylene group, it acts as a chain terminator. The resulting prepolymer is less branched and, therefore, less viscous when softened. To further control the flow properties, a small amount of DEB monomer can be blended with the prepolymer, compensating for any monomer or polymer variation. We have found that such blending does not decrease the char yield of the cured resin.

Resin Synthesis

The starting material for the resin synthesis was DEB obtained from Hercules, Inc., from their previous production of HA 43. (Recently, new synthesis routes for DEB have been reported.[13-14]) According to information provided by Hercules, Inc., the composition of the DEB was as follows:

m-diethynylbenzene	57.0 -60.0 wt percent
p-diethynylbenzene	22.0 - 25.0 wt percent
3-ethylphenylacetylene	2.7 - 3.0 wt percent
4-ethylphenylacetylene	0.6 - 0.9 wt percent
high boiling constituents	6.5 - 7.5 wt percent
residual methyl isobutyl ketone	5.0 - 6.0 wt percent

Phenylacetylene was obtained from Farchan Laboratories, Inc., and used as received. Nickel acetylacetonate, triphenylphosphine, and MEK were reagent grade and also used as received.

The catalyst used to cyclotrimerize the DEB was prepared by dissolving 51.4 g of nickel acetylacetonate hydrate and 157.4 g of triphenylphosphine in 500 ml of MEK. The solution was heated to reflux, and 350 ml of MEK was distilled off at atmospheric pressure. The remaining MEK was removed under aspirator vacuum to yield a green solid that was dried under vacuum and ground to a powder before use.

The following example of prepolymer preparation illustrates two methods for controlling highly exothermic reactions in large batches: 1) the use of small aliquots of catalyst to control reaction rates and 2) the use of a temperature-limiting solvent. For very large batches, further dilution should also be considered. The prepolymer reaction was carried out in a 12 liter, 4-neck, round-bottomed flask equipped with two condensers and a thermometer that measured the pot temperature (not vapor temperature). We found that MEK is the best solvent for the cyclotrimerization

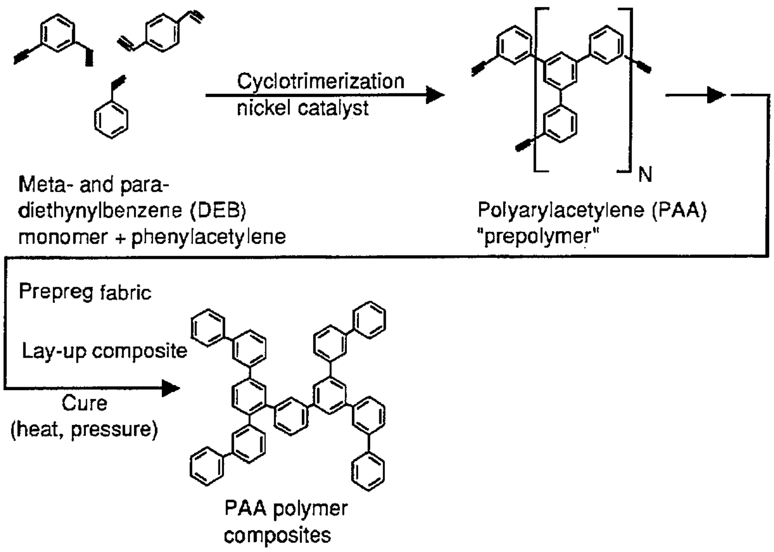

Figure 3. Schematic of fabrication of polyarylacetylene composites.

reaction because its use results in rapid reaction, and its low boiling point (80°C) enables good process control. Nine grams of catalyst were dissolved in 4,250 ml of MEK, and the flask was heated to distill off 500 ml of solvent, azeotropically drying the MEK. After the addition of 2,250 g of DEB and 750 g of phenylacetylene, the flask was slowly heated. When the pot temperature reached 93°C, the heat was turned off. The temperature continued to rise from the exothermic reaction until the onset of reflux at 98°C. As the reflux subsided, an additional 3 g catalyst were added, resulting in an increased rate of reflux. Thereafter, 6 g aliquots of catalyst were added periodically to maintain the rate of reflux of the polymer solution. No external heat was supplied to the system during the reaction, so the reflux was driven by the heat of reaction.

As the reaction proceeded, the pot temperature eventually decreased to 90°C, at which time the reaction was stopped by applying towels soaked in cold water to the exterior of the flask. (An alternate cooling technique is the use of a light vacuum to strip off part of the solvent.) A total of 48 g of catalyst was added by the end of the reaction, so the catalyst comprised 1.6 percent of the solids (nonsolvent components) in the reaction mixture. The final solution contained 46 wt percent of prepolymer in MEK, measured by weighing the residue remaining after solvent evaporation at 130°C for 30 minutes. (For optimum prepregging, a final prepolymer concentration of 60 to 65 wt percent is recommended.) This solution had a shelf life at 20 to 25°C of more than a year, as determined by a lack of change in its IR spectrum and viscosity. However, storage at 0°C is recommended.

The endpoint of the reaction was determined by following the drop in pot temperature (about 8°C) and by checking the consistency of the resin on a Fisher-Johns melting point apparatus held at 95°C. After evaporation of the solvent on the melting point apparatus, the proper end-point was judged to have occurred when one could draw fibers from the resin with a spatula. Although we monitored the extent of reaction by following pot temperature and resin viscosity, IR spectroscopy of the prepolymer reaction can also be used. In this technique, one can monitor the ratio of the intensities of an IR peak associated with the synthesized prepolymer (stretching due to the carbon-carbon bond between aromatic rings at 1,600 cm^{-1}) to that of an IR peak from the acetylenic groups that are being consumed (≡ C-H stretch at 3,256 cm^{-1}). The ratio of intensities of these two peaks at various reaction times can be correlated with the capability of the resulting prepolymer to be processed. Thereby, an optimum ratio can be determined.

Four batches of resin with a total mass of about 24 kg were prepared by the above procedure. The four batches were mixed together, and the resin for the prepregging operation was taken from this 24 kg "master batch." Rheometry and differential scanning calorimetry (DSC) were performed on powdered samples of resin prepared by drying the resin several days under vacuum. For the rheometry measurement, the powder was compressed into a small (~ 2 mm thick) pellet and heated on a temperature-controlled platen. A 5 g stainless steel weight was placed on the pellet. The distance between this weight and the platen was monitored with a linear variable differential transformer. As the pellet was heated, it softened, and its viscosity decreased. The weight fell toward the platen, and the displacement was noted. The rheometry data (displacement versus temperature, Figure 4) indicate that the resin softens at about 50°C and reaches a minimum viscosity at about 75°C. Figure 4 also shows a DSC scan of the dried resin taken at a heating rate of 1°C/minute, which is the same as the platen heating rate. The scan was obtained utilizing a DuPont Instruments thermal analyzer, model 1090, with a nitrogen purge. The scan shows that the cure exotherm begins at about 90°C and reaches a peak at about 120°C. It is important that the resin flows adequately to infiltrate the fiber tows before the cure begins. Since the resin reaches minimum viscosity at 75°C, but does not begin to cure until 90°C, good impregnation occurs.

Fabric Prepregging

T300 eight-harness satin (HS) weave carbon fabric purchased from Ferro Corp. (Los Angeles, CA) was used to fabricate a composite panel. The fabric was washed, first with acetone, then MEK, to remove the sizing. The prepregging operation was carried out at Programmed Composites, Inc. (Brea, CA). The prepregging equipment carries the fabric through the resin bath and squeeze-out rollers, then winds the fabric onto a large drum with a 6.7 m circumference. DEB monomer was added to the resin to increase the flow. The amount of DEB added was 10 percent

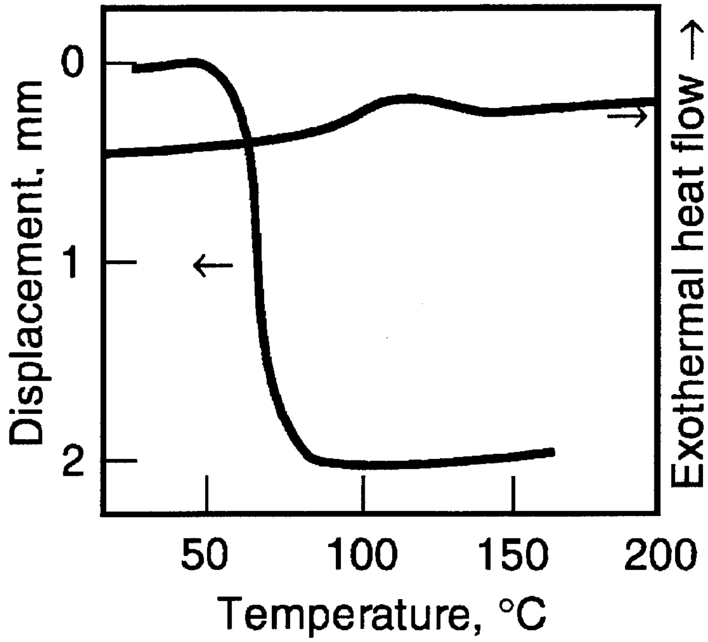

Figure 4. Rheometry and differential scanning calorimetry (heating rate: 1°C/minute) of PAA.

of the solids content of the final resin. The resin solution was prepregged onto the fabric with a gap of 0.5 mm on the squeeze-out rollers. After prepregging, the MEK solvent was allowed to evaporate in air for 36 hours. At that point, the residual solvent content was less than 3 percent. The resin content, as determined by hot MEK extraction, averaged 30 wt percent. This prepreg was stored at -20 to -30°C.

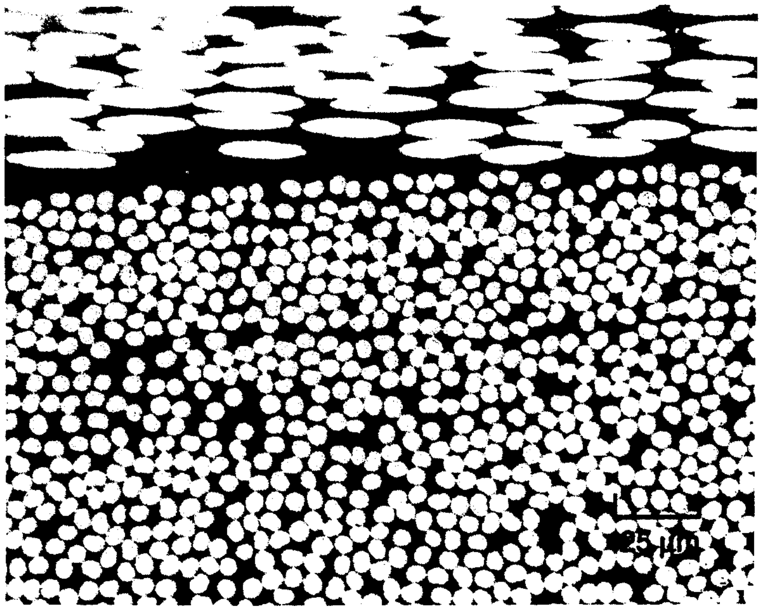

Figure 5. Optical photomicrograph of T300/PAA composite.

Composite Fabrication

About 200 plies (28 cm x 30.5 cm) were stacked and vacuum bagged. A pair of thermocouples were inserted near the center of the stack. The platens of a press were preheated to 100°C. The vacuum-bagged lay-up was then placed into the press, and the ram pressure was increased to 0.7 MPa. The internal thermocouple temperature was monitored. When the internal temperature reached 80°C (about 40 minutes), the platen temperature was increased to 110°C. The internal temperature was kept at about 100 - 105°C for about 15 minutes; then the platen temperature was increased to 200°C at a rate of 1°C/minute. At an internal temperature of 125°C the cure exotherm began, and the panel temperature changed from 125°C to 230 - 240°C in about eight minutes, yielding a heating rate of about 13-14°C/minute. The platen temperature was about 135°C when the panel temperature reached its maximum, so the internal panel temperature was about 100°C higher than the platen temperature at the peak of the cure exotherm. After eight hours at 200°C, the platen heat was turned off, and the panel was cooled to 60°C under pressure before removal from the press.

PROPERTIES

The fabricated panel was removed from the press, and its edges were trimmed with a diamond-coated bandsaw blade. The final dimensions were 28 cm x 30.5 cm x 5.1 cm thick. A density of 1.46 g/cm^3 was determined by helium pycnometry. Acid digestion indicated that the resin content of the panel was ~ 29 wt percent or ~ 41 vol percent.

Figure 5 shows an optical photomicrograph of a polished cross section of the composite. Microscopic examination revealed that the composite was well densified and that the fabric was uniformly impregnated. Ply spacing was approximately constant through the thickness. Some microcracks were visible throughout the matrix; most were oriented perpendicular to the plies. Such microcracks are commonly seen in lay-

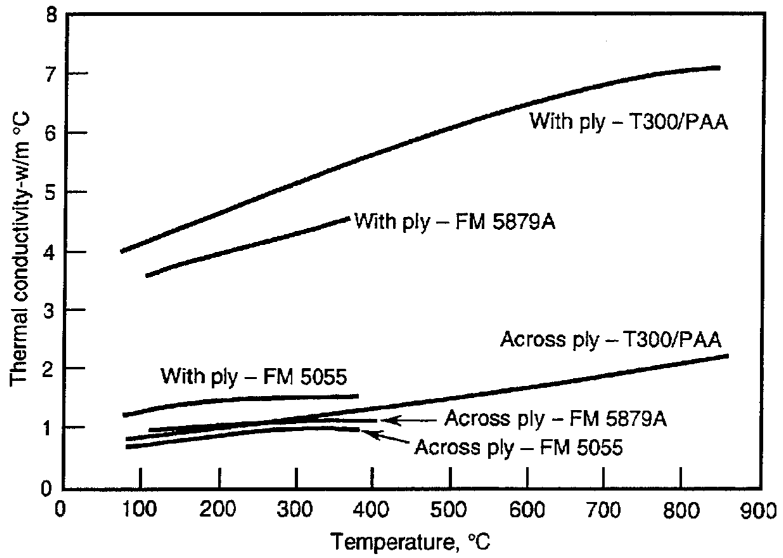

Figure 6. Thermal conductivity of T300/PAA, FM 5879A, and FM 5055 as a function of temperature.

ups of this type and are thought to provide some stress relief during ablation.

The with-ply and across-ply thermal conductivity of T300/PAA were measured at Southern Research Institute, Birmingham, AL.[15] A plot of thermal conductivity versus temperature is shown in Figure 6. For comparison, the conductivities of FM 5879A and FM 5055 carbon phenolic are shown. FM 5879A is a SHS T300 composite made with phenolic resin. FM 5055 utilizes rayon-precursor CCA-3 fabric. Since the across-ply thermal conductivity is dominated by the resin, the across-ply values are equivalent. The with-ply values are dominated by the fabric and reflect the slightly lower reported heat treatment of the T300 for the FM 5879A and the much lower thermal conductivity of CCA-3.

The across-ply tensile properties of T300/PAA were measured at room temperature (RT) and 400°C. The strength at RT was 5.3 MPa, with a stiffness of 10.2 GPa and a strain to failure of 0.6 percent. At 400°C, these values had dropped to 1.4 MPa, 3.0 GPa, and 0.5 percent, respectively. These values compare quite favorably with those for the T300/phenolic resin FM 5879A, discussed above. At RT, the FM5879A had a strength of 4.2 MPa and a strain to failure of 0.6 percent. By 260°C, the strength dropped to 0.3 MPa.

ABLATION TESTING

NASA MSFC performed comparative ablation tests[16] of T300/PAA and the standard phenolic ablative insulator material (FM 5055) with a CO_2 plasma jet. Specimens were exposed under identical conditions for 10 seconds. The results (Figure 7) indicate that T300/PAA is markedly superior – it undergoes less weight loss and erosion – and that T300/PAA data display less variance than data of FM 5055. Also, whereas a large fraction of the FM 5055 samples exhibited pocketing, there was no pocketing in any of the T300/PAA samples.

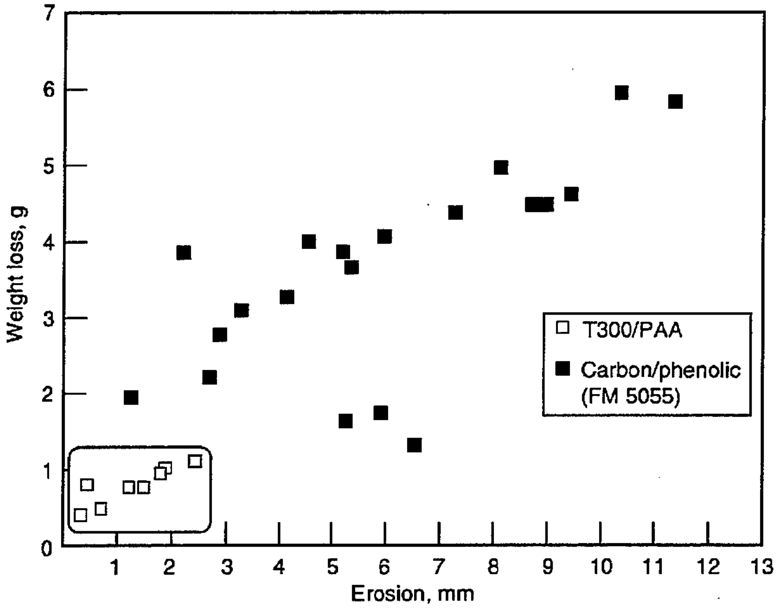

Figure 7. Weight loss and erosion of T300/PAA and carbon-phenolic (FM 5055) resulting from comparative ablation testing at NASA MSFC.

SUMMARY/CONCLUSIONS

A successful technique for the fabrication of polyarylacetylene resin composites has been developed. The technique consists of synthesizing a soluble processible prepolymer followed by standard prepregging and composite fabrication methods.

These composites were demonstrated in CO_2 plasma jet testing to have superior ablation performance when compared to the currently used carbon phenolic material.

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