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Processing , Properties and Applications of Composites Using Powder-Coated Epoxy Towpreg Technology

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ABSTRACT

Composite manufacturing using the current prepregging technology of impregnating liquid resin into three-dimensionally reinforced textile preforms can be a costly and difficult operation. Alternatively, using polymer in the solid form, grinding it into a powder, and then depositing it onto a carbon fiber tow prior to making a textile preform is a viable method for the production of complex textile shapes. The powder-coated towpreg yarn is stable, needs no refrigeration, contains no solvents and is easy to process into various woven and braided preforms for later consolidation into composite structures.

NASA's Advanced Composites Technology (ACT) program has provided an avenue for developing the technology by which advanced resins and their powder-coated preforms may be used in aircraft structures. Two-dimensional braiding and weaving studies using powder-coated towpreg have been conducted to determine the effect of resin content, towpreg size and twist on textile composite properties. Studies have been made to customize the towpreg to reduce friction and bulk factor. Processing parameters have been determined for three epoxy resin systems on eight-harness satin fabric, and on more advanced 3-D preform architectures for the downselected resin system. Processing effects and the resultant mechanical properties of these textile composites will be presented and compared.

INTRODUCTION

For composite materials to be utilized as primary structures in subsonic and supersonic aircraft applications, the total production costs of the composite parts must be decreased from their present levels. Developments in the fabrication of composite parts point toward cost reduction through increased automation. In conjunction with the development of automated fabrication techniques, NASA Langley Research Center (LaRC) and BASF Structural Materials, Inc. have developed methods of prepregging carbon fiber with thermoplastic and thermosetting polymer powders.

Under NASA's Advanced Composites Technology (ACT) Contract, NAS1-18888, Lockheed Aeronautical Systems Company (LASC) is investigating the use of textile composites for primary aircraft structures. By coupling powder-coated towpreg with existing, highly automated textile processes, the resulting impregnated fabrics, broad goods and preforms can be easily molded into parts. These combined fabrication processes may be a low cost alternative to resin transfer molding (RTM) of dry preforms in cases where complex mold geometries and tightly fabricated preforms pose wet-out problems. Additionally, the physical size of aircraft primary structures makes tooling for RTM an expensive manufacturing option. The powder-coated process may offer the only viable method of part fabrication if high melt viscosity polymers are required to obtain improved composite properties, such as thermal stability and/or fracture toughness.

The following work has been conducted to generate fabrication technology for 3-D composite structures from powder coated 3-D textile preforms. Issues related to the weaving of towpreg yarns, debulking of 2-D and 3-D preforms, and processing and properties of eight-harness satin fabric flat panels have been addressed in detail. In the weaving study, the size, level of twist , and the rigidity of powder-coated tow were systematically varied to determine the effects of each process variable on resulting fabric quality. Debulking trails were performed to determine how effective various pressure/temperature combinations were on reducing bulk in as-fabricated 2-D and 3-D textile preforms. A layup and consolidation scheme was adopted which produced good quality flat laminates from 3M AMD 0036[1] epoxy powder coated preforms. Mechanical property tests were performed on these laminates to compare with identical laminates fabricated using the 3M PR-500 RTM epoxy resin system.

2-D WEAVING STUDIES

Towpreg Variables vs. Weavability

The primary objective of the 2-D weaving study was to learn how to convert powder-coated yarn into quality fabric. In earlier studies [1,2], the process of powder-coating tow and its weaving or braiding into preforms for part fabrication was found generally to be less expensive and inflicted less damage to the fibers when large (12,000 filament and more) tow bundles are used. Offsetting the advantage of using large tow bundles are factors such as

potential difficulty in consolidation and possible reduction in composite properties. Yarn splitting and loose fibers on the yarn surface cause difficulties in weaving. To overcome this, yarn shaping, twisting, serving, wetting, and sizing are common practices used to reduce the separation of filaments, decrease tow-to-tow abrasion, and minimize fiber loss.

All towpreg used in the 2-D weaving portion of this study was fabricated using a dry powder process developed by NASA [3]. The dry powder prepregging process involves three steps: tow spreading, polymer deposition, and polymer fusion onto the fibers. The carbon fiber tow bundle was first pneumatically spread to approximately 3 inches in width, then impregnated with powder by means of a dry, recirculating, fluidized powder chamber. Radiant heating was used to sinter or fuse the polymer powder particles to the tow. The powder line was upgraded to speeds of 11-16 yards/min and over 21,000 yards of towpreg were produced for this study.

The effects of varying yarn bundle sizes and yarn twist on the weavability of dry polymer powder-coated fibers were studied in detail. The mechanical properties of composite made from the resultant woven cloth were determined. G30-500[1] (BASF) and AS-4[1] (Hercules) carbon fibers in tow bundles of 3k, 6k and 12k filaments were used. Each was impregnated with a thermoplastic polyimide, LARC™-TPI[1] 1500 medium flow powder (Mitsu Toatsu Chemicals). Weaving was performed on towpreg yarns that had twist levels of zero twist or 0.4 twists per inch (tpi). After establishing a weaving protocol, an experiment epoxy (3M AMD-0029[1]), was fabricated into towpreg and woven into eight-harness satin fabric.

Towpreg was woven into eight-harness satin fabric under NASA Contract NAS1-18358 by Textile Technologies, Incorporated[1] (TTI) in Hatboro, PA. The initial work was performed on yarns containing 6k filaments. During this phase, the set-up of the loom and the weaving of the towpreg were examined for ways to minimize damage imparted to the yarn. The towpreg yarn prepared at LaRC was rewound at TTI onto 40 separate spools in order to produce a balanced 4 inch wide fabric with 10 picks per inch (ppi). Two rewinding machines were used to determine how best to rewind towpreg. A rewinder that yielded a parallel winding pattern was found to cause less fiber damage than a rewinder that gave a cross winding pattern.

The spools of rewound towpreg were loaded into an Iwer 1200 rapier-type loom. Weaving powder-coated tow in a conventional rapier loom results in less fiber damage on the fill yarns, since the fill yarn is taken directly from the manufacturer's spool. Initial weaving efforts revealed problems with the surface of some of the warp towpreg yarns having loose filaments, which accumulated in the heddles and reed. To alleviate this problem, the towpreg was twisted to a carbon fiber manufacturer's standard of 0.4 tpi. Use of twisted towpreg greatly improved the weaving operation.

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[1]Use of trade names or manufacturers does not constitute an official endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

An analysis is given in Table 1 of the powder-coated fabric produced for this study from both twisted and untwisted yarn. The weave counts, linear weights and fabric thicknesses are presented for 3k, 6k, and 12k towpreg made with LARC™-TPI. Noticeable fiber damage was observed in the woven material. While twisting improves weavability, the action of twisting was found to impart damage to the towpreg yarn. The method of tow twisting at LaRC was performed off-line after the prepregging had been completed, and required additional fiber handling. It is likely that improvements in the twisting equipment and on-line twisting can reduce fiber damage.

Table 1. Towpreg 8-harness Satin Fabric Parameters for 2-D Weaving Study.

Towpreg Specification	Weave Count (ppi)	Areal Weight (oz/yd^2)	Thickness (inches)
6k (G30-500) / LARC™-TPI, Untwisted	10.2 x 9.8	14.10	0.067
6k (G30-500) / LARC™-TPI, Twisted	10.2 x 9.8	14.27	0.071
6k (G30-500) / LARC™-TPI, Untwisted	10.1 x 10.0	13.22	0.077
6k (G30-500) / LARC™-TPI, Twisted	10.2 x 9.3	14.73	0.103
12k (AS-4) / LARC™-TPI, Twisted	8.2 x 8.2	23.91	0.126
3k (G30-500) / LARC™-TPI, Twisted	20.0 x 19.8	12.63	0.058

Towpreg flexural rigidly was also systematically varied since a previous study indicated that yarn flexural rigidity was an important parameter in successfully weaving towpreg [4]. Powder-coated yarn rigidity is a function of percent resin content, oven temperature, and yarn residence time in the oven. These parameters were appropriately altered to furnish the required rigidity variations. Samples were taken from each lot of towpreg yarn, and flexural rigidity was measured by ASTM method D1388-64. Towpreg flexural rigidity values utilized in this study are listed in Table II.

Towpreg Variables vs. Mechanical Properties

A mechanical testing program was developed to determine the effects of tow bundle size and twist on the mechanical properties of unidirectional and eight-harness satin fabric laminates. To investigate the effects of tow bundle size, powder-coated towpreg made from LARC™-TPI and 3k and 6k G30-500, and 12k AS-4 carbon filaments were frame-wrapped into unidirectional panels to obtain the flexural strength and modulus, and the transverse flexural strength [5].

A vacuum press was used to remove air from void spaces in the LARC™-TPI/carbon fiber specimens consolidated for the weaving study. At maximum temperature, pressure was applied at 1.5 psi/min to 60 psi in order to allow sufficient time for resin flow, adhesion, and fiber movement. The pressure ramp was followed by a hold period of one hour for final

Table 2. Flexural Rigidity Results for Powder Coated Tow and Fabrics Used in 2-D Weaving Study

Tow Description	Overhang (in)	Areal Weight (oz/yd^2)	Rigidity (oz-yd)
6k LARC™-TPI, Twisted Tow	4.0	2.44	1.54×10^{-2}
6k LARC™-TPI, Twisted Woven Cloth	3.25	13.28	4.45×10^{-2}
6k LARC™-TPI, Twisted Tow	9.0	1.66	1.17×10^{-1}
6k LARC™-TPI, Twisted Woven Cloth	7.0	12.79	4.24×10^{-1}
12k LARC™-TPI, Twisted Tow	5.5	6.15	9.87×10^{-2}
12k LARC™-TPI, Twisted Woven Cloth	4.0	25.85	1.60×10^{-1}
12k LARC™-TPI, Untwisted Tow (34.6 ʷ/o resin)	6.75	5.94	1.76×10^{-1}
12k 3M epoxy, Untwisted Tow (32 ʷ/o resin)	5.0	5.85	7.09×10^{-2}

consolidation and stress release at 662°F or 698°F for the unidirectional laminates and 698°F for the woven eight-harness satin prepreg cloth. The part was cooled below Tg at a rate sufficient to stop consolidation before final part thickness was reached. This avoided resin squeeze-out and resulting dry spots. The mechanical properties generated from these tests for untwisted tow are compared to the tow bundle size in Figures 1 and 2.

To determine the effects of twisted tow on mechanical properties, tests were conducted on unidirectional composites specifically, 12k carbon fiber (AS-4) towpreg of LARC™-TPI with a twist level of 0.4 tpi. Flexural strength and modulus (ASTM D790-84a) values were obtained and compared to untwisted towpreg (Table III). In addition, compression tests were performed tests were performed by the IITRI method (ASTM method D3410-87, procedure B). The values for compressive strength, modulus, and Poisson's ratio for twisted tow and untwisted tow are also listed in Table III.

The eight-harness satin fabric, woven from powder-coated towpreg, was consolidated into panels, and then cut into tension and short block compression specimens. The tension specimens were tested untabbed using hydraulic grips. A tensile load was applied only in the warp direction. Insufficient material was available for testing in the fill direction. The short block compression specimens were tested in both the warp and fill directions. Tension and compression moduli for eight-harness satin fabric composites are shown as a function of tow bundle size and twist in Figure 3.
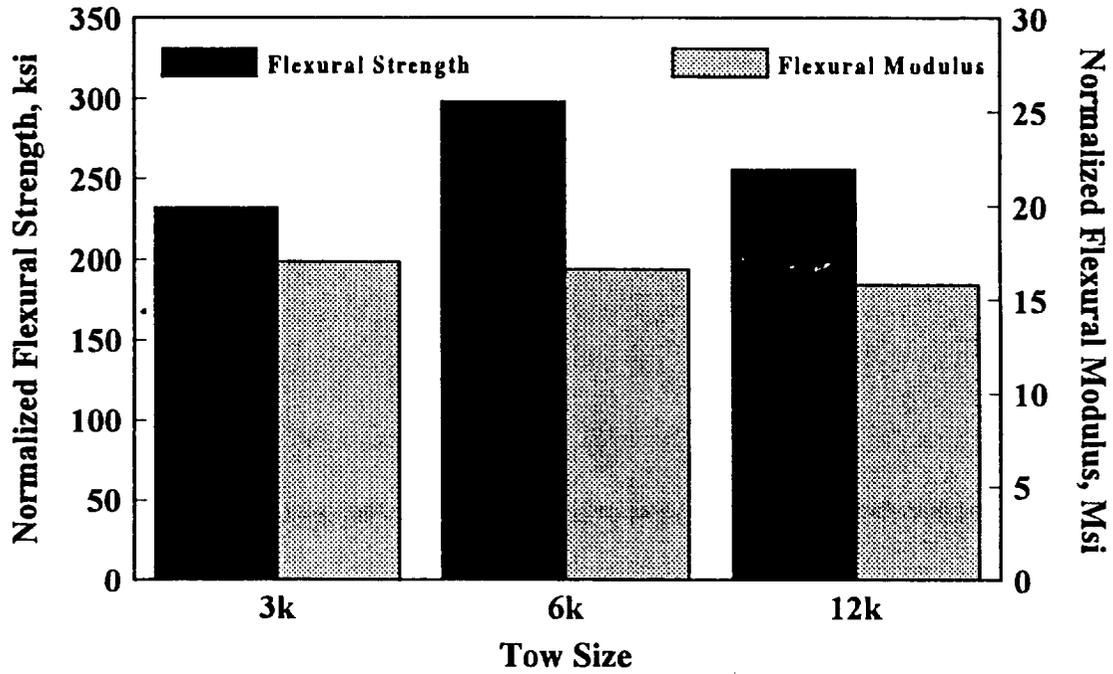
Figure 1. Flexural Properties vs. Tow Size in Unidirectional LARC™-TPI/AS-4 or G30-500 Composites (Normalized to 60 volume percent fiber)
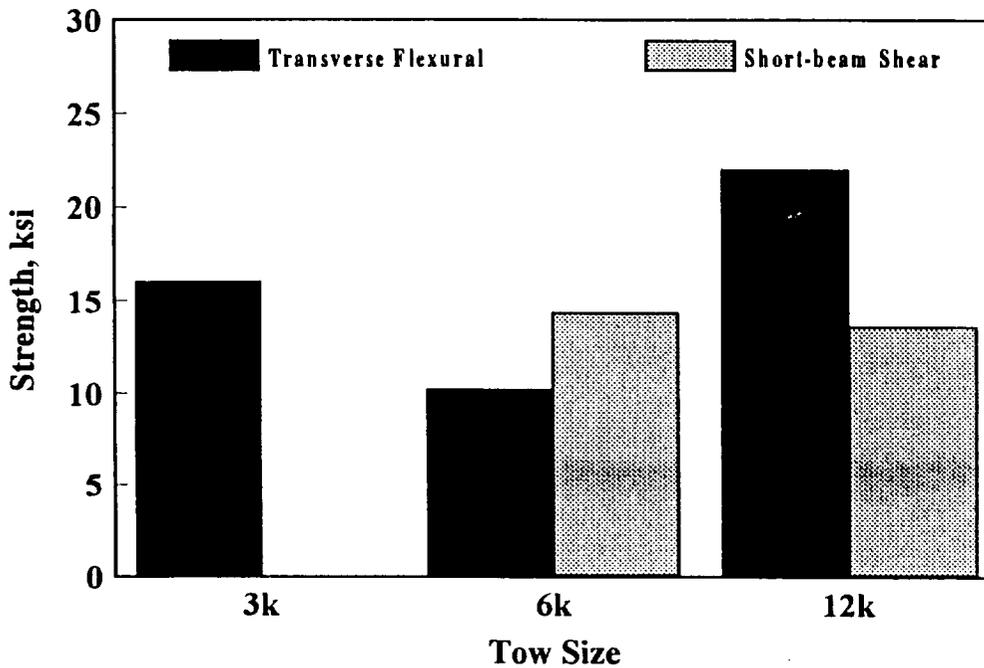

Figure 2. Mechnaical Properties vs. Tow Size in Unidirectional LARC™-TPI AS-4 or G30-500 Composites.

Table 3. Twisted and Untwisted Towpreg Mechanical Properties in Unidirectional 12k AS-4/LARC™- Thermoplastic Polyimide (TPI) Composites.

Mechanical Properties	UnTwisted Towpreg	Twisted Towpreg (0.4 tpi)
Flexural Strength[1], ksi	255.3 ± 14.1	248.4 ± 15.9
Flexural Modulus[1], Msi	15.6 ± 0.2	16.2 ± 0.4
Compression Strength, ksi	165.3 ± 1 2.2	140.4 ± 9.7
Compression Modulus, ksi	17.1 ± 0.8	15.7 ± 0.9
Poisson's Ratio[2]	0.35 ± 0.02	0.38 ± 0.03

[1] Values have been normalized for 60% fiber volume fraction.
[2] Based on IITRI compression data (by ASTM Method D3410-87, procedure B).
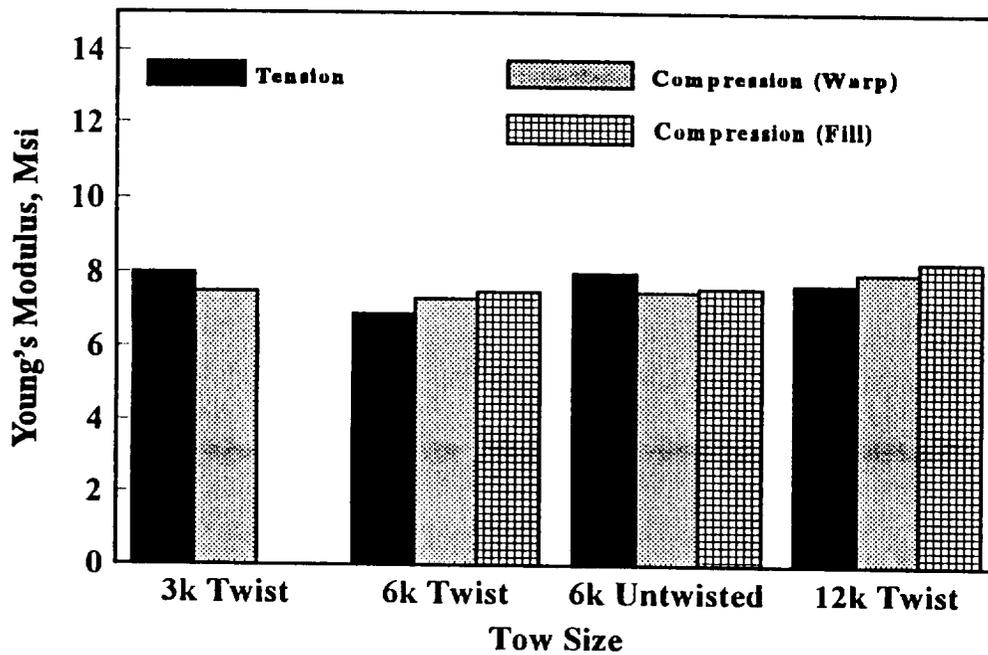
Figure 3. Young's Modulus (Short Block Compression) vs. Tow Size for 8-Harness Satin Woven LARC™-TPI/AS-4 or G30-500 Composites.

Discussion of Weaving Study Results

All weaving operations require that care be taken to minimize fiber damage. There should be as few as possible eyelets, bends and other tow touch points. Tensioning should be kept low. Rewinding and other handling activities should be minimized.

An important observation regarding weaving and tow size selection is the relation between fiber damage and tow size. During both powder prepregging and towpreg weaving, fiber damage is greater for the smaller tow bundles, because damage occurs primarily to the fibers that are at the bundle surface. For a given total amount of fiber, the use of small tows results in larger tow surface area and correspondingly higher fiber damage.

For well consolidated unidirectional laminates, the tow bundle size should have no effect on mechanical properties. No apparent pattern was found in the mechanical properties of the unidirectional laminates as a result of tow bundle size (Figures 1 and 2) with a possible exception in the transverse flexural strength values (Figure 2). Unidirectional composites made only with 6k material was consolidated at 698°F, whereas the 3k and 12k materials were consolidated at 662°F. The increase in temperature for the consolidation cycle may have resulted in an increase in consolidation due to a decrease in resin viscosity. As the processing cycles for LARC™-TPI fiber reinforced composites are improved, more mechanical property data will be generated.

In contrast to the unidirectional mechanical properties, the fabric composites were expected to exhibit increased mechanical property values with decreasing tow bundle size due to the contribution of crimp, which increases with increasing tow bundle size. The limited data obtained for composites made with eight-harness satin woven cloth (Figure 3) show no apparent effect of tow size or twist on tension and compression modulus. Because of a lack of material, each data point shown in Figure 3 presented an average value taken from testing three to five specimens. A large scatterband was observed for the strength data, consequently, more tests will be required to develop statistically significant strength values.

Mechanical properties of composites made of twisted towpreg exhibited lower properties, except flexural modulus, than specimens with untwisted tow (Table 3). At 0.4 tpi the non-alignment effect of fibers in a twisted yarn is negligible [6]. This is illustrated in the compression and flexural modulus values. Composites made with twisted towpreg had a 15 percent lower compression strength than those made with untwisted towpreg. Apparently, the additional fiber damage that resulted from the current method of twisting caused the reduction in strength. Twisting the towpreg improved weavability, since it caused the yarn to take on a cross-section that was round and compact. In order to create a suitable yarn for weaving, either the current method of twisting must be improved or the towpreg must be shaped with heated dies or rollers to achieve the same cross-section with less damage.

PROCESSING SCIENCE FOR POWDER-COATED PREFORMS

Special consideration in consolidating woven goods, as distinct from consolidating unidirectional tape, must be given to the elimination of intra- and inter-tow voids within fabric, as well as the elimination of inter-ply voids which are also of concern in conventional tape processing. The established steps [7] in the consolidation of woven materials are illustrated in Figure 4. They are: intimate contact of the polymer-polymer interface at numerous sites across the composite; followed by deformation and interdiffusion of polymer chains to cause the interface to disappear.
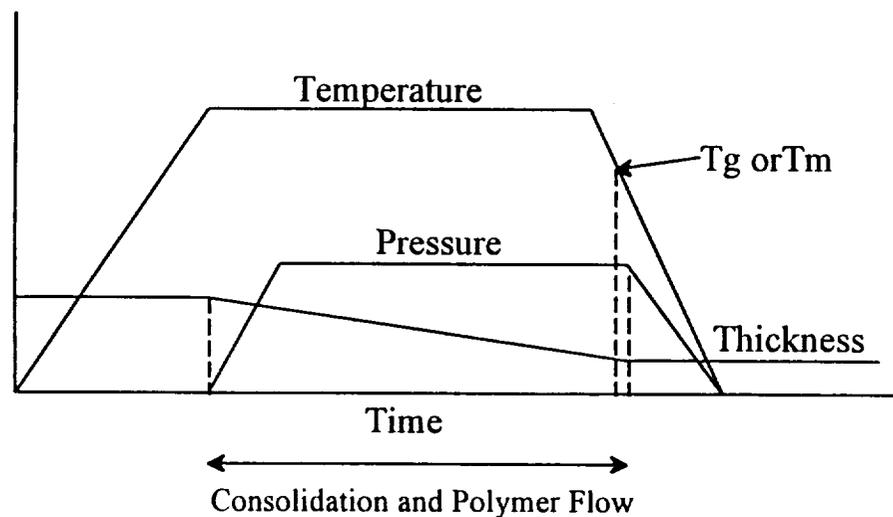
Figure 4. Towpreg Textile Preform Consolidation Cycle

Vacuum and autoclave pressure is applied to induce the resin flow, wetting of fibers, and fiber movement necessary to eliminate voids and fill the intra- and inter-tow spaces. The change in thickness during consolidation is about 4 to 1 for textile preforms. The debulking of preforms is part of the ongoing investigation. The ramping of the pressure allow the fibers time to move into a compact arrangemet,with minimum fiber crimping and breakage, and provides time for resin flow and adhesion.

The composite panel is held above Tg or Tm to anneal the part and releive internal stress which are generated duing the cooling down portion of the cure cycle. Once the material is cooled below Tg, pressure is released so that consolidation is stopped before resin can squeeze out of the layup, causing resin poor areas with lowered mechanical properties.

Flat test panels have been manufactured from woven 8-harness satin fabric from towpreg produced in both wet and dry powder impregnation processes. These methods have been previously described [8]. Early work was done using towpreg having 35 ± 2 weight percent resin. After analysis of the resulting parts, the resin content was raised to 38 ± 2

weight percent for subsequent material. It was evident that the textile operations were causing some powder losses during preform fabrication.

Bulk in Powder Coated Textile Preforms

A bulk factor is inherent in the powder towpreg manufacturing process. Figure 5 is a schematic of the acquisition of bulk during the impregnation of the tow with powder particles. This occurs regardless of the method of towpreg production, although towpreg processed by a dry (for example, electrostatic) technique generally has a slightly higher bulk than that produced using a wet (slurry) technique. This occurs when the tows are spread before the impregnation step. The process of debulking a composite prior to final consolidation is standard throughout the industry. Experience has shown that debulking increases the quality of a thermoset laminate. The debulk cycle removes air that becomes trapped between plies as they are laid down, with the result being both improved handling of the preform and ease of insertion into the tooling. High bulk factors are becoming a very challenging technical problem working with powder towpreg preforms. Typically, bulk factors run about 4:1 in powder towpreg preforms.
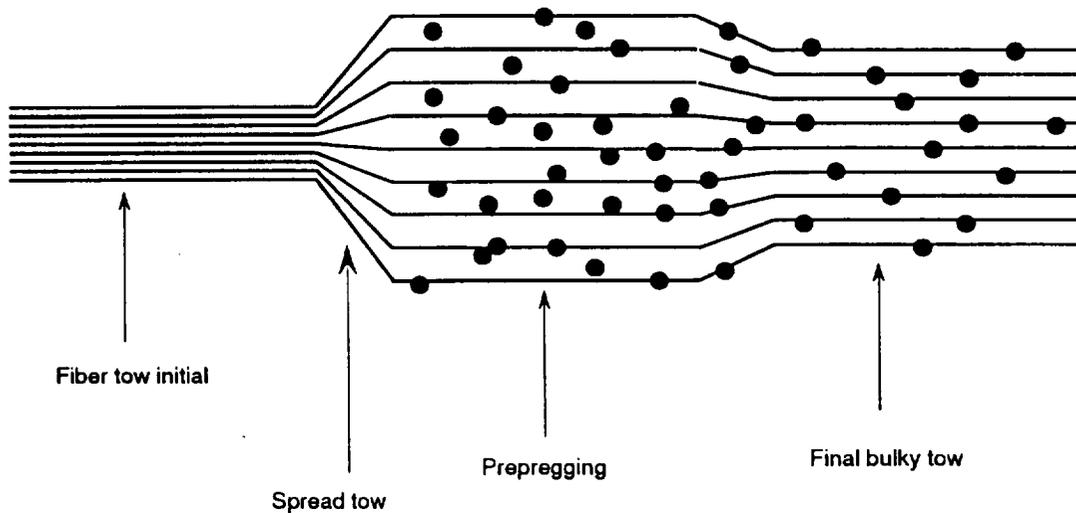

Figure 5. The Origination of Bulk in Powder Towpreg.

Bulk is not a great problem in flat laminates, however, the problem requires special attention whenever complex contours, angles, webs, or dropoffs are produced, if dimensional tolerances are to be maintained. When 3-D weaves or braids are used as preforms, a priority is to minimize fiber bulk in the z-direction. This is critical because z-directional tows restrict the ability of the preform to be compressed (and thus more difficult to consolidate), and then if the preform does compress, the excess fiber will buckle inside the part. Figure 6 shows the effect of bulk on the consolidation characteristics of a 3-D preform.

Area for high probability
of fiber buckling
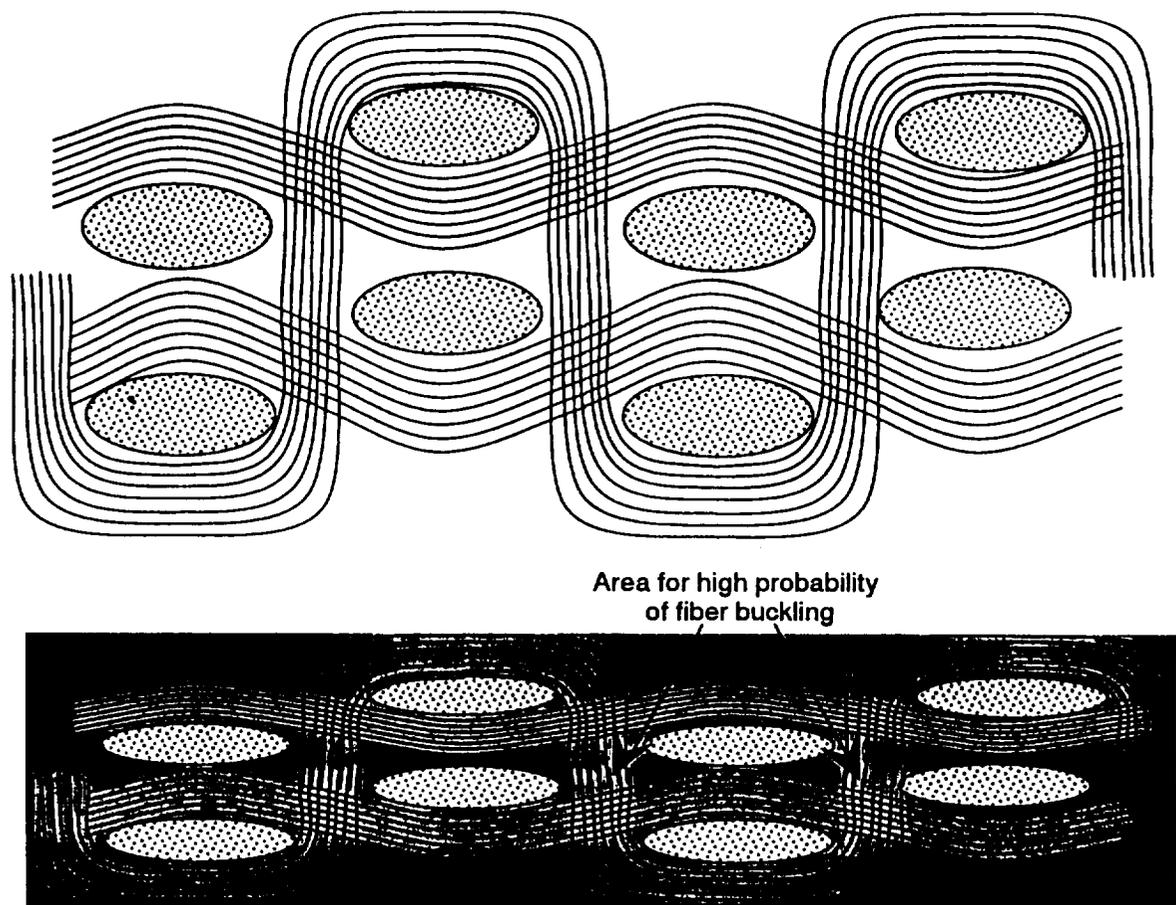
Figure 6. The Effect of Bulk on the Consolidation Characteristics of a 3-D Preform.

Debulking of 2-D and 3-D Textile Preforms

A debulking study has been initiated to address the issues related to consolidation of advanced textile preforms. The preform types which have been, or will be, under consideration are: 1) 2-D braid (supplied by Fiber Innovations[1]); 2) 3-D through the thickness weave (Techniweave[1]); 3) 3-D Multi-axial knit (Tech Textiles[1]); 4) 3-D weave (Fiberite[1], Fabric Development[1]). Initially, the study will establish the time, temperature, and pressure parameters for debulking flat samples of several advanced textile preform types, using towpreg produced at BASF (resin content: 38 ± 2 % by weight). Upon completion of the debulking study and establishment of process parameters, the verification of stiffened panel preform quality, tooling and manufacturing approaches shall be demonstrated. The manufacturing demonstrations will consist of debulking and processing representative sections of a side panel, such as an integrally stiffened window belt.

Prior to embarking on the debulking study, a short aging study was conducted on the AMD 0036 neat resin. Samples of the resin were held at 200°F for varying times, then analyzed rheometrically and by Differential Scanning Calorimeter (DSC). These analyses were done on samples which had been held at temperature for 15 minutes, 45 minutes, and 60

minutes at a heating rate of 4°F per minute. Viscosity data in Table 4 show that the minimum viscosity is virtually unchanged after aging at 200°F for 30 minutes. The DSC was run at a heating rate of approximately 18°F/minute. The results, also shown in Table 4, were compared with an as-received sample of the same batch, as well as with a sample from a second batch of powdered AMD 0036. The results show little difference between unaged and aged resin, for both the onset and peak exotherm temperature.

Table 4. Results of Differential Scanning Calorimetry and Rheometric Studies of Aged AMD 0036 Epoxy Resin.

Sample (Batch #IM2P)	1st Peak (J/oz)	2nd Peak (J/oz)	Total (J/oz)	Peak Exotherm (°F)	Minimum Viscosity (Poise)
#1--As Received	1700.2	430.4	2130.6	456.3	1300
#2--15 min. at 392°F	1632.4	498.6	2131.0	452.7	
#3--30 min. at 392°F					1360
#4--45 min. at 392°F	1666.7	404.9	2071.6	452.3	
#5--60 min. at 392°F	1486.8	315.6	1802.4	456.4	
#6--As Received Batch # 8126			2280.7	455	

2-D braided preforms manufactured by Fiber Innovations, Inc. were fabricated using BASF[1] produced AS-4/RSS-1952 towpreg, at a resin content of 39 ± 2% by weight. All towpreg was 6k, and samples 10.5" x 6" having 10 ends/inch were braided using 72 carriers, and a 3.25-inch mandrel diameter. Preforms containing 6 plies were braided; a dry lubricant was applied to the axial tows to reduce friction during the braiding of the towpreg. A silicone rubber vacuum table, with heat on the top only, was used, and the debulk cycle was as follows: 1) apply full vacuum; 2) heat from room temperature to 200°F; 3) hold for 15 minutes; and 4) turn the ply stack over, and repeat steps 1-3.

The typical initial thickness of the 6-ply braided preforms was 0.50". After the debulk cycle, the preform thickness was 0.22". After curing, the thickness of the sample was 0.124". This represents a debulk from 4:1 to 1.8:1. While the 2-D braided preforms react well to a pre-consolidation debulk cycle, issues relating to the incorporation of the dry lubricant must still be resolved before these materials will be considered for test panels. These issues relate to the reactivitiy of the dry lubricants under consideration and the effects of their presence on the mechanical properties of the resulting laminate.

Initial efforts at debulking 3-D preforms centered on the 3-D through the thickness woven preform manufactured by Techniweave, Inc. The nature of the z-direction fibers in these preforms is such that it is advantageous to try to debulk during preform fabrication.

On-line debulking studies will begin in the early summer at Techniweave. Currently, post-fabrication debulking of 3-D woven shaped preforms is being studied by Lockheed.

Two methods of debulking the Techniweave preforms have been undertaken. In the first method, hard tooling was made to debulk the T-stiffened preform . Three debulking cycles were conducted on the preforms. First, the preform was heated under vacuum pressure to 200°F, and held for 20 minutes. The bulk factor of the skin portion was reduced from 2.5 to 2.0, and the stiffener from 3.2 to 3.1. The second cycle was also done at 200°F, using autoclave pressure of 100 psi for 20 minutes. This time, the bulk factor of the skin was reduced from 2.0 to 1.2, and in the stiffener from 3.1 to 2.3. To reduce the bulk of the stiffening element further, a third cycle was conducted using moveable plates, rather than angle tools, to apply pressure to the perpendicular members of the preform individually. Under autoclave pressure of 100 psi for 20 minutes at 200°F, the bulk factor of the stiffener was reduced to 2.0. The step-wise debulking which occurred using this method is shown in Figure 7. The large surface area of the skin portion of the preform restricted the tool from sliding inward to the stiffener, preventing an even application of tool pressure, which caused an uneven debulk of the vertical stiffening member. After final consolidation of the part, it was evident that the cross-section of the skin/stiffener intersection was not symmetric about a neutral axis, however, the thickness of the respective horizontal and vertical members of the part were within specification.
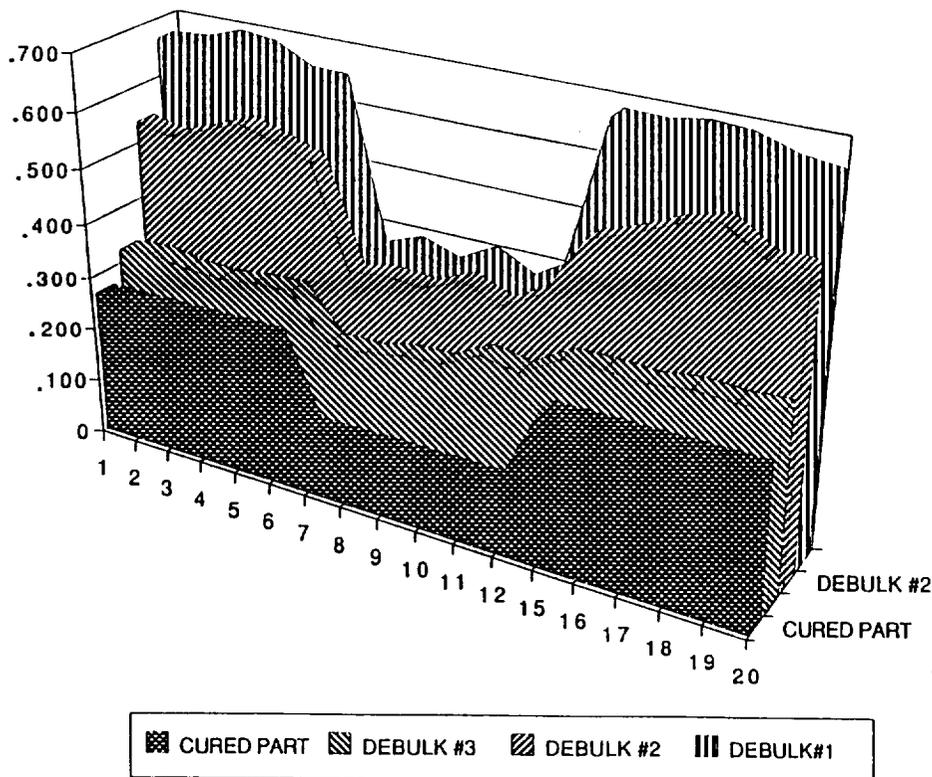

Figure 7. Results of Debulking Trial on 3-D Woven T-Stiffened Preform.

The results of the first debulking effort led to a redesign of the tooling for the T-stiffened Techniweave preform. In this approach, the part is vacuum bagged, placed in an autoclave and debulked using full vacuum pressure plus autoclave pressure of 100 psi. This is a two step approach where the stiffener will be debulked before debulking the skin. The preform will be hot debulked from both sides towards the center line of the blade using a combination of hard and rubber tooling material. The rubber is sized to provide approximately 150 psi on the stiffeners. Again, the part is heated to 200°F, and held for 20 minutes. Preliminary results show this approach to be successful in providing an even debulk of the preform. Both the vertical and horizontal members were debulked from approximately 4:1 to less than 2:1, however, there was a small amount of fiber bridging in the skin/stiffener intersection. The results of this trial are currently being further analyzed.

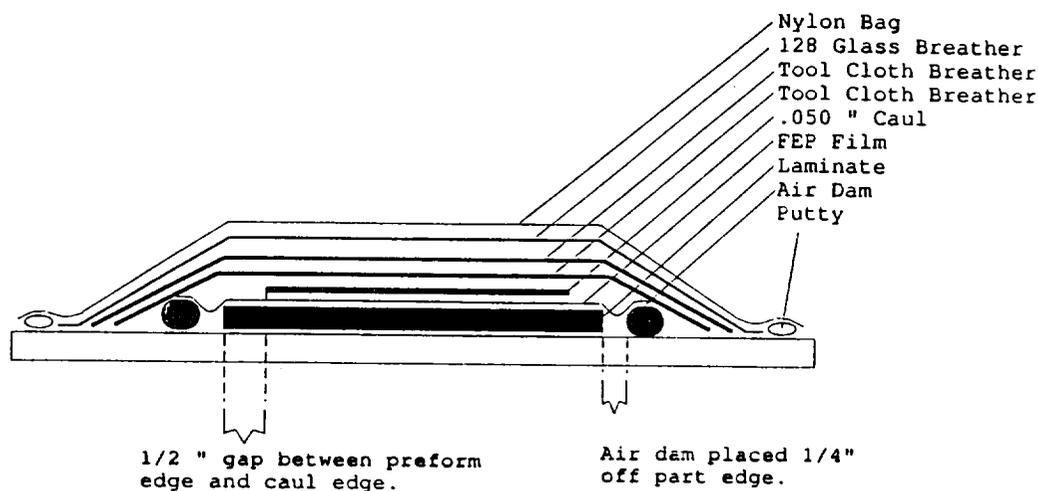**Consolidation of 2-D Woven Fabric**

Epoxy powder coated eight-harness satin fabric was laid-up, bagged and autoclave processed into flat panels for mechanical property testing for three powdered epoxy resin systems: AMD 0036[1] (3M), RSS-1952[1] (Shell), and CET-3[1] (Dow). Powder towpreg manufactured by Custom Composites Materials, Inc.[1] (CCMI) of the 3M resin system was woven into 8-harness satin fabric by Textile Technologies, Inc.[1] (TTI). The resin content of this material was only about 27 weight percent; powder was added by hand to bring the panels up to 35 ± 2%. The other two resin systems were evaluated using towpreg produced by BASF[1], and woven into 8-harness satin fabric by Fabric Development, Inc[1]. Flat panels having fiber volume fraction of 58% to 60% were fabricated and evaluated by Lockheed [8].

Early attempts at consolidating the RSS-1952 and AMD 0036 coated powder fabrics were not successful due to the inherent hydroscopic tendencies of the powdered resins. Since the towpreg will be stored at room temperature, long term exposure to a high humidity environment may be experienced. Some moisture may also be used by the textile operators for lubrication during fabric production.

During the first few trials, water droplets formed between the part and the caul plate during the cure cycle. Some drying cycle tests were performed to determine the time required to dry a given number of plies in an oven/autoclave. The results resulted in a one hour drying cycle at 120°F under full vacuum pressure, during the autoclave cure cycle to insure that trapped moisture was eliminated before consolidation in the autoclave. High quality laminates were made using this approach, and a study to evaluate the necessary drying segment length for particular 3-D preforms will be initiated at Lockheed in the future.

Towpreg flat panel laminates were cured using a caul plate with 0.050" thickness after early trials showed that 0.080" thick cauls were too thick, given the low viscosity of the resin systems. The center regions of these panels were porous due to lack of pressure caused by the bowing of the caul, from edge bleeding of the resin. When resin extruded from the perimeter of the panel, the edge of the caul was unsupported and the caul subsequently bent. A more uniform pressure distribution having only local edge deformation was delivered using the thinner caul.

The low viscosity of the resin systems used in the program prompted excessive resin bleed during consolidation, therefore, an air dam around the perimeter of the parts was incorporated into the bagging scheme. The air dam was placed roughly 0.5" away from the apart to minimize bleed, yet maximize breather paths. The resulting layup bagging scheme is shown in Figure 8. A noticeable gap (0.5") is seen in Figure 8 between the edge of the caul and the edge of the part. This configuration is intended to allow for better breathing of the laminate during cure. Porosity tends to occur when adequate breathing channels are not available, even with the low viscosity of the resin. This is more than likely a result of high bulk factors which are inherent in the powder towpreg preforms. Future work is palnned to determine whiy this caul plate configuration must be used to fabricate quality laminates.

Nylon Bag
128 Glass Breather
Tool Cloth Breather
Tool Cloth Breather
.050 " Caul
FEP Film
Laminate
Air Dam
Putty

1/2 " gap between preform edge and caul edge.

Air dam placed 1/4" off part edge.

Figure 8. Layup Bagging Scheme for Powder Coated Flat Panel Laminates.

Some alterations to these generalized consolidation guidelines have been incorporated because of resin specifics. Initial cycles using the Lockheed standard heating rate of 5°F/min produced porous panels, however, well consolidated panels resulted when the heating rate was lowered to 3.5°F/min. The AMD 0036 epoxy resin requires a lower heating rate than standard epoxies in the cure cycle to minimize porosity. The CET-3 system required a thicker caul plate, since this system had a higher melt viscosity than the 3M resin. The lack of edge thinning due to bowing of the caul was reduced, and less bleeding was observed about the perimeter of the CET-3 parts.

Mechanical Properties of AS-4/AMD 0036 Flat Panels

The quality of the flat panel laminates met Lockheed ultrasonic specification. Self-calibration of 65% reflected through ultrasonic signal is considered acceptable. C-scans are

performed using a 0.5-inch diameter transducer, at a frequency of 5 MHz, gain of 16 db, and a focal length of 4.0 inches.

Mechanical property testing of powder coated 8-harness satin fabric panels fabricated at Lockheed was conducted at Delsen Testing Laboratories, Inc. Test results normalized for fiber volume fraction for the PS-501 (3M) and RSS-1952 (Shell) systems are presented in Table 5, and compared to similar results for the PR-500 RTM resin [8]. PS-501 data represents the earlier lab scale version of the AMD 0036 epoxy system. Testing of the Dow CET-3 flat panels is currently underway.

Test results indicate that the PS-501 system is superior to the RSS-1952 material in all tests, with the exception of open hole tension and compression, both at room temperature (dry) and 180°F (wet). In the open hole tests, RSS-1952 outperforms the PS-510 resin system by 15% in compression; in tension the difference is only 2.7 ksi (6.4%). The PS-501 compression after impact (CAI) results are 40% higher than RSS-1952, for the NASA CAI test specimens. A photomicrograph from a CAI specimen is presented in Figure 9. The micrograph shows a classic compression failure, with a shear line across the failed region, when viewed from the panel profile. No global bending or buckling deformation was observed. Some splintering of the plies was observed on the back side of the panel, due to the impact.
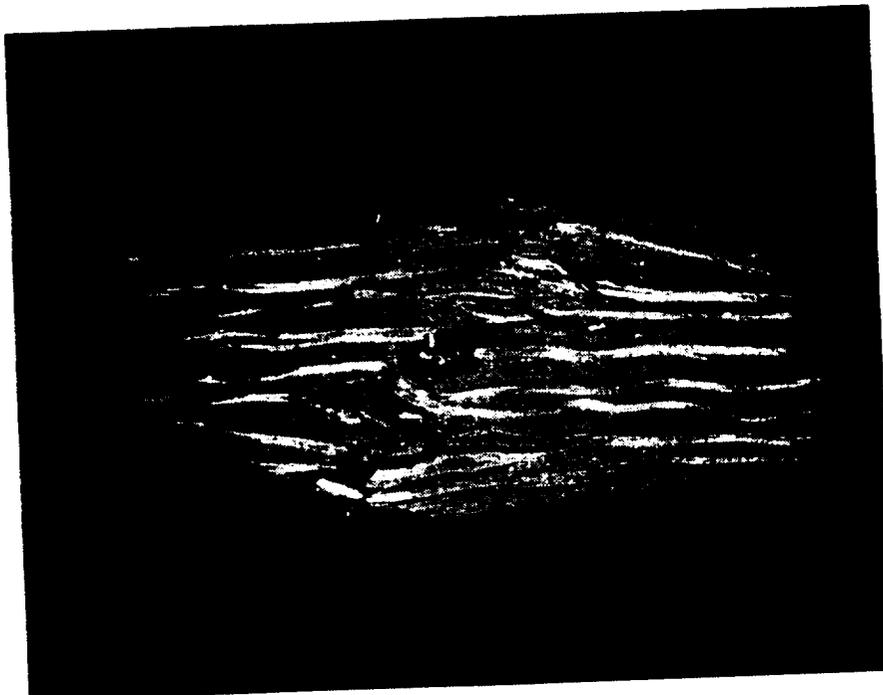
Figure 9. Photomicrograph from PS-501 CAI Specimen, Showing Compression Failure.

The PS-501 flat panel test results compare favorably with panels fabricated from the PR-500 RTM resin, except in the case of the hot-wet results, where the RTM panels were higher. The strength loss in these tests was from 21% to 28%. 3M Company has indicated

Table 5. Normalized Mechanical Test Results for Powder Coated 8-harness Satin Fabric Flat Panels

Test Type	PS-501 3M	RSS-1952 Shell	CET-3 Dow	PR-500 RTM Resin
0° Tension				
RT				
Ultimate Strength, ksi	99.2	91.8		94.7
Modulus, Msi	10.04	8.77		9.17
Poison's Ratio	0.042	0.042		0.079
Strain To Failure, %	1.17	0.00		1.10
-65°F				
Ultimate Strength, ksi	104.4	93.2		99.3
Modulus, Msi	9.92	8.96		8.92
Poison's Ratio	0.048	0.048·		0.065
Strain to Failure, %	1.08	1.01		1.15
Fiber Volume, %	52.5	54.8		57.0
Orientation / Ply	0° / 4	0° / 4		0° / 3
0° Compression				
RT				
Ultimate Strength, ksi	103.1	81.1		100.9
Modulus, Msi	9.13	8.91		8.98
Poisson's Ratio	0.053	0.021		0.028
180°F, wet				
Ultimate Strength, ksi	70.8	50.3		87.8
Modulus, Msi	8.96	8.08		8.93
Poisson's Ratio	0.064	0.065		0.040
Fiber Volume, %	53.98	53.3		56.1
Orientation / Ply	0° / 8	0° / 8		0° / 8
45° Tension				
Ultimate Strength, ksi		10.6		16.1
Modulus, Msi		0.59		0.75
Fiber Volume, %		54.6		58.7
Orientation / Ply		45° / 6		45
Unnotched Tension				
Ultimate Strength,ksi	65.3	64.4		
Modulus, Msi	6.84	5.84		7.1
Strain To Failure, %	1.21	1.07		.6.47
				1.09
Fiber Volume, %	61.5	54.4		
Orientation / Ply	quasi / 8	quasi / 8		56.2
				quasi / 8

Test Type	PS-501 3M	RSS-1952 Shell	CET-3 Dow	PR-500 RTM Resin
Open Hole Tension, ksi				
RT	41.9	44.6		47.8
-65°F	41.6	43.0		45.4
Fiber Volume, %	54.3	52.3		53.6
Orientation / Ply	quasi / 8	quasi / 8		quasi / 8
Unnotched Compression				
RT				
Ultimate Strength, ksi	70.3	59.3		72.4
Modulus, Msi	6.24	6.09		6.30
Strain to Failure, %	1.34	1.07		1.35
180°F, wet				
Ultimate Strength, ksi	50.3	37.5		64.3
Modulus, Msi	6.14	5.89		6.30
Strain To Failure, %	0.86	0.66		1.22
Fiber Volume, %	54.4	52.7		54.6
Orientation / Ply	quasi / 8	quasi / 8		quasi / 8
Open Hole Compression, ksi				
RT	38.1	44.3		41.4
180°F, wet	30.5	29.4		34.1
Fiber Volume,%	58.9	46.6		54.7
Orientation / Ply	quasi / 8	quasi / 8		quasi / 8
Compression After Impact (SACMA)				
Ultimate Strength, ksi		23.9	30.9	34.1
Modulus, Msi		6.36	5.82	6.64
Strain to Failure, %		0.39	0.50	0.51
Fiber Volume, %		55.6	60.4	59.8
Orientation / Ply		quasi / 12	quasi / 12	quasi / 12
Compression After Impact (NASA)				
Ultimate Strength, ksi	40.5	28.3		39.3
Modulus, Msi	6.40	6.52		6.54
Strain to Failure, %	0.67	0.44		0.63
Fiber Volume, %	55.6	55.5		57.5
Orientation / Ply	quasi / 18	quasi / 18		quasi / 18

that the hot-wet properties will improve in the scaled-up (AMD 0036) version of the powder. Some of the tests in Table 5 will be repeated using CCMI produced AMD 0036 towpreg, to evaluate any mechanical property differences between the electrostatic method and the slurry method of producing towpreg. All of the hot-wet tests will also be repeated.

SUMMARY

This study has dealt with textile applications of powder towpreg, focusing on weaving and consolidation. Some of the operating and design issues in these processes have been resolved while others have been highlighted for further attention. Making textile preforms from polymer powder-coated towpreg depends upon a number of material properties and equipment parameters. An optimal weaving protocol requires tow handling minimization, and tow bundle twisting of about 0.4 twists per inch. These textile techniques are important factors for automating the production of quality composite parts from powder-coated towpreg preforms.

The consolidation cycle for woven towpreg must account for the inter-bundle crimp of the weave. Since a higher towpreg resin content is needed to fill the interstitial spaces in woven materials than for unidirectional tape, in general, composites made from woven material will have a lower fiber volume fraction. Further studies are required to establish the optimum fiber volume fraction for powder-coated preforms. Because of the initial bulk associated with woven materials, vacuum pressure must be applied during the cure cycle to prevent the formation of air voids. During consolidation, the fibers in woven materials must move and realign, while resin must flow to fill the interstitial spaces. A gradual increase in pressure over time provides for greater ease of fiber movement and resin flow before the fibers align in a tight, compact arrangement. Attention to these factors, together with the general practice of holding for a period of time at maximum pressure and temperature followed by cooling at a rate which minimizes internal stresses, yielded composite specimens of woven material that were void-free, as determined by ultrasonic C-scans.

Mechanical properties were determined from composite specimens made with carbon fiber tow bundles of 3k, 6k and 12k that were coated with LARC™-TPI 1500 medium flow grade powder. Testing was performed on unidirectional and eight-harness satin fabric composite specimens. No apparent effect of tow size was found in the unidirectional or the eight-harness satin fabric composite specimens. The lower compression strength dispalyed by the unidirectional composites made with twisted towpreg was due to apparent fiber damage that occurred during the twisting of the tow bundle.

The matter of optimum tow bundle size when comparing the mechanical properties remains unresolved. Fiber damage has been observed to be less when larger tows are used. Weaving equipment capabilities are somewhat independent of tow size. It appears that the choice of tow bundle size is an open one in regard to properties, but that tows containing at least 12,000 or more fibers are favored, especially in regard to powder processing and weaving costs.

An aging study on AMD 0036 resin system showed that exposure to 200°F for up to one hour has little effect on the rheological characteristics of the material. Debulking studies on 2-D textile preforms showed that 200°F and vacuum pressure was sufficient to reduce the bulk factor from 4:1 to less than 2:1. Autoclave pressure is necessary to debulk 3-D woven preforms, due to the presence of the z-directional fibers.

The NASA ACT Program has selected 3M AMD 0036 as the powder system to be carried throughout the rest of the contract. The mechanical properties of flat panel laminates fabricated using towpreg coated with the lab scale formulation of AMD 0036 are superior to other powder-resin systems investigated, and comparable to the PR-500 RTM resin, except in hot-wet tests. It is anticipated that the hot-wet properties will be improved during the scale-up production, and tests to confirm this are planned.

ACKNOWLEDGEMENTS

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Ceramic Preconsolidation Tool for Thermoplastic Powder Towpreg

The ceramic preconsolidation tool facilitates isolation of hot and cool contact zones which are required for the conversion of bulky powder coated carbon fibers into preconsolidated towpreg ribbons.

Langley Research Center, Hampton, Virginia

The potential use of various high-temperature performance thermoplastics as carbon fiber composite matrix systems has prompted research to identify novel methodology in the production of advanced composite prepreg systems Many of these polymers are not amenable to solvation in environmentally friendly solvents. When these polymers melt they typically exhibit high viscosity and are difficult to utilize in conventional prepreg operations due to reduced polymer flow during impregnation. Several techniques have been developed to plasticize these polymers but the plasticizers tend to reduce the mechanical properties of the bulk thermoplastic.

Current research at NASA LaRC has led to the development of novel tooling and prepregging techniques which convert powder-coated towpreg into preconsolidated ribbon without the use of any plasticizers or solvents of any kind. Relying on melt-flow and proper application of tension, friction and cooling, this processing method converts powder towpreg into ribbon with consistent polymer distribution and cross-section.

This innovative debulking technique draws on the advantages of pulling unidirectional filaments through a heated / cooled open-faced die. The ceramic tool was constructed at NASA LaRC, specifically for this purpose. After the bulky towpreg leaves the pay-out creel, it is warmed by a flow of heated gas. Initial contact between the melted towpreg and the tool occurs on a curved surface which is channeled into the tool. As the bundle is drawn over the tool. it spreads out to fill the channel. This accomplishes two things: first, it allows for melt squeeze flow to distribute the polymer more evenly throughout the fibers and secondly, it facilitates fiber nesting under tension which provides debulking. The heated towpreg now has the shape of a wide and flat ribbon. An air insulated region divides the two sections of the tool which provides sufficient thermal barrier necessary to separate the actively cooled zone of the tool from the heated zone. The ribbon next enters the cool zone of the tool and cools to a solid under controlled conditions. Shrinkage of the polymer and its reduced stickiness allow it to separate from the tool surface.

CERAMIC PRECONSOLIDATION TOOL

HEAT SHIELD

PRECONSOLIDATED TOWPREG RIBBON

HEAT

COOL AIR
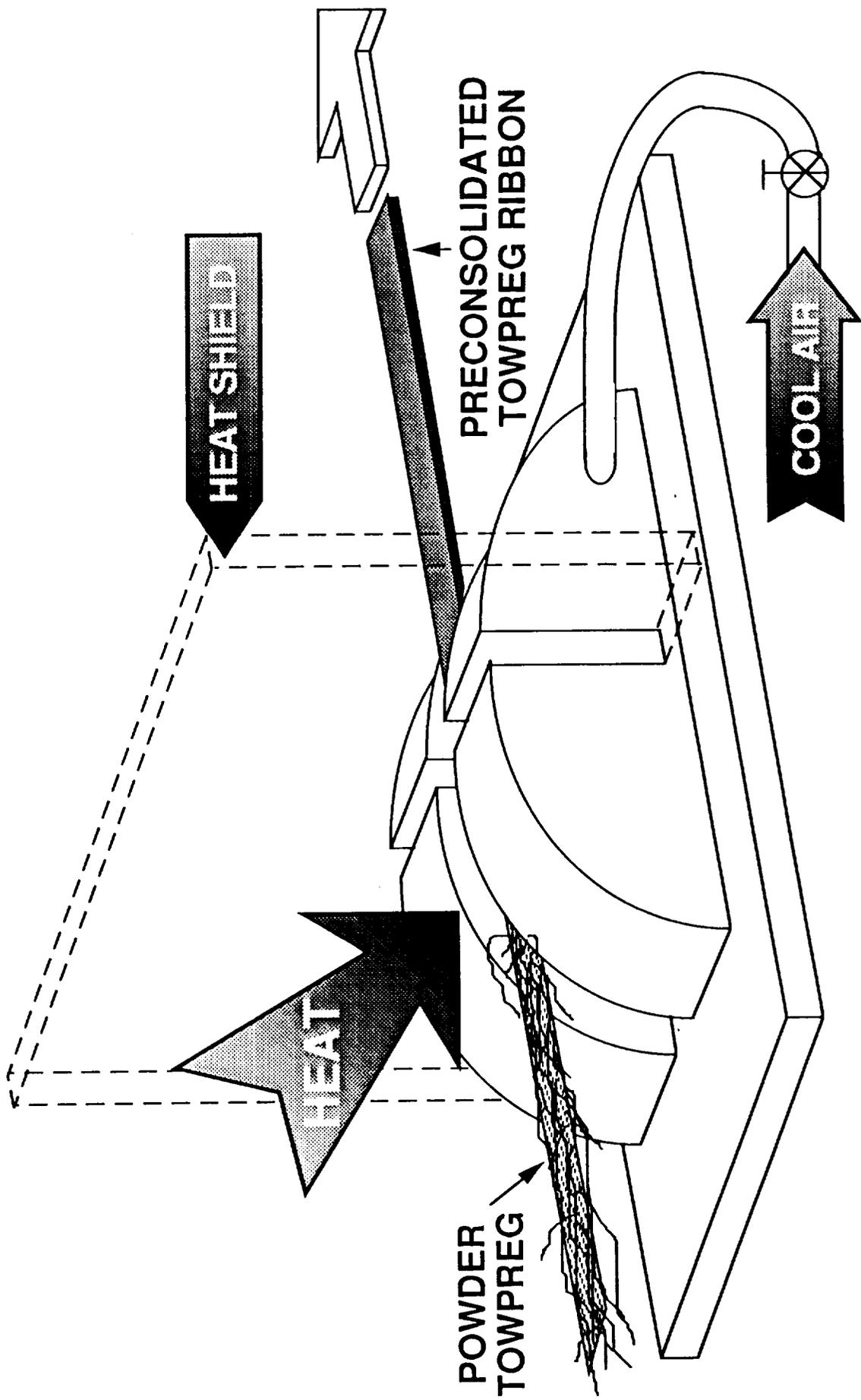
POWDER TOWPREG

April 15, 1993

Memorandum

To: N. Johnston, R. Baucom, M. Hugh, J. Hinkley,
         D. Sandusky, and S. Wilkinson

From: J.M. Marchello   *mum*

Subject: Composite Part Fabrication

Towpreg, polymer powder impregnated fiber tow, is now being produced at semi-commercial levels (over 100 pounds per month) by several companies (Custom Composites, BASF/Hexcel, EniChem and perhaps others). In the period ahead, these processes, and NASA-LARC's process, will continue to be developed while significant quantities of towpreg are made for test panels and part fabrication studies.

In conjunction with the increased availability of towpreg, our thoughts have shifted more toward composite processing and part fabrication. Some recent discussions have dealt with the issues involved in making different aircraft parts. The purpose of this memorandum is to summarize and share this information, and to obtain further comments.

The development of methods for using towpreg in the fabrication of composite parts for commercial aircraft is an important goal of the ACT and HSR (HSCT) programs. A knowledge about, and acceptance of, newly developed methods is vital to the Trade Studies and to the Design-Build team deliberations, which are concerned with increased use of composites in commercial aircraft.

Starting with towpreg yarn, there are several approaches to making composites. These are generally considered to be the use of textile technology, thermoforming, and automated tow/tape placement.

Textile technology may be used to weave or braid the towpreg into broadgoods and preforms for part fabrication. An important goal of the textile studies is to develop a powdered preform molding alternative to the RTM process for producing net shape parts. Having the polymer powder dispersed in the fiber bundle reduces melt-flow requirements and allows composites to be made from high melt viscosity polymers. Major developmental concerns with textile applications are tow friction (abrasion) during braiding and debulking of preforms

during consolidation. These issues are being addressed in studies of towpreg surface treatment for braiding, debulking (cold) during weaving, and special tooling for debulking (hot) preforms during consolidation and part fabrication.

Thermoforming, primarily pultrusion and die forming, using towpreg offers a means of making parts directly and of making customized ribbon for ATP. Development of automated tow placement methods focuses on reduction of labor costs, beyond those of automated tape placement, through the development of special towpreg ribbons and robotic heads that can handle complex tool surfaces with sharp turns, ribbon add and drop, honeycomb cores, and in-situ consolidation. These issues are being addressed by studies of towpreg ribbonizing, autohesion bonding kinetics, and robot head modifications.

Direct part pultrusion is limited to making long fairly uniform parts, such as floor boards and support members. Reduction in the cross section of a part along the length may be achieved to some extent by using a contracting die and tow dropping methods. For example, a J bar of varying height might be pulled starting with the die fully extended. As pulling proceeds, the die J height is reduced and internal tows are dropped to accomodate the reduced die area along the part length.

With high temperature polymers, the intermediate composite products, from which parts are to be fabricated, are much stiffer, or boardier, and more difficult to work with than conventional prepreg or aluminium. This requires new approaches to fabrication and suggests possible utilization of carpentry and cabinet making techniques. There is a precedence for this. In 1940, when he could not get aluminum to build a protype of his planned 800 passenger flying boat, Howard Hughes used wood and made the Spruce Goose. For the HSCT aluminum is inadequate, so stiff, wood like, composites will need to be used. Instead of SST, the next plane might be called SSG for Son of Spruce Goose.

Current composite aircraft part fabrication studies deal with wing and fuselage sections and their supporting stringer members, window frames and window belt sections, air ducts, and floor boards. As a final thought topic, in the quest to make an all-composite airplane, consider the following possible way of making an aircraft landing gear column.

Landing gear columns contain hydraulic fluid and are made of high strength steel to handle high compression, impact, and flexural loads. The objective, then is to

make a thick walled composite pipe that can handle the environment and loads of aircraft landing.

Composite are generally thought as being strong in tension and weak in compression. The reason for this is that the fibers are thin and they buckle readily under compression loading. If fiber buckling can be prevented, the composite might exhibit high compressive and flexural strength.

A composite landing gear column might be fabricated as follows. Filament wind several layers of towpreg on a metal pipe, having the diameter needed for the internal diameter of the cylindrical column, and consolidate. Cool and remove the pipe tool. (Alternately, the pipe tool might be left in and removed later.) This step produces a composite pipe with properties like an NOL ring. Attach on the outside of this composite pipe a large number of towpregs aligned in the axial direction. To ensure that they remain oriented in the axial direction these towpregs should be held with some tension. Consolidate so that the linear towpreg layer is debulked and resin bonded to the composite pipe. Tightly filament wind several towpreg layers around this tube and consolidate. Repeat the process by adding another layer of linear towpregs, consolidate, filament wind with towpreg and consolidate. Continue this buildup process to the external diameter of the part.

The result of the above process is a hollow composite cylinder consisting of a series of rings of linear (about 90%) and wrapping (about 10%) tows. The wrapping tows would serve to prevent buckling of the linear tows, which would provide the strength properties needed for a light weight landing gear column.

April 13, 1993

Memorandum

To: N. Johnston, R. Baucom, D. Sandusky, M. Hugh,
           J. Hinkley, and S. Wilkinson

From;   J. Marchello

Subject: Multi-tow Ribbon for ATP

     There are several important aspects of customized
towpreg ribbon that we have been discussing in recent
weeks. The purpose of this memorandum is to attempt to
summarized and share these thoughts, and to obtain
additional comments.

     Ribbon architecture concepts, that we have
developed and published during the past year or so, deal
with elimination of tow-tow gaps, improved bonding, rapid
laydown, the ability to handle sharp placement turns and
concave tool surfaces, and in-situ consolidation. In this
regard we have proposed non-rectangular ribbon cross-
sections (triangle, trapezoid, and parallelogram) that
provide for ribbon nesting during placement, and the use
of narrow ribbon for small radius laydown turns. These
considerations have led to studies of towpreg
ribbonizing, autohesion bonding kinetics, and robot head
design modifications.

     The standard of comparision for ATP ribbon is
APC-2. It is 5.6 mills (142 microns) thick and 250 mills
(6350 microns) wide. That corresponds to a cross-
sectional area of 901,700 square microns. With 65% fiber
volume, the fiber area is 586,105 sq. microns. With AS4
fibers (7 micron diameter or 38.5 sq. micron area) the
fiber count of the ribbon is 15k. With IM7 (5 micron
diameter or 19.6 sq. micron area) the fiber count is 30k.

     Since the HSR/HSCT program will use IM7 fiber, to
make a ribbon having the same area as APC-2, we would
need to use either one 6k and two 12k tows, or five 6k
tows. This is why we want to acquire a multi-tow creel
for the ribbonizing studies.

     An important feature of our customized ATP ribbon
effort rests on the goal of developing automated
(robotic) tow/fiber placement to the point where the
robot head can do anything that can be done by human
hands. This means handling complex tool surfaces that
require placement during sharp turns, in confined highly
curved areas. Add to this the desire for high

(commerical) laydown rates and in-situ (on-the-fly) consolidation. Also, in many cases the part will consist of a honeycomb core with a composite skin. To avoid damage to the core/tool, ATP placement of such skins will require using less than 50 psi of pressure, and good adhesive bonding to the honeycomb.

To place tow ribbon without buckling during sharp turns, it is necessary to use narrow ribbons. For example, 1/8 inch (125 mill) wide ribbon can be placed at a turning radius of 5 inches. Sharper turns would require narrower ribbon.

To achieve tow gap reduction, or elimination, by nesting, the ribbon sides must be at angle other than 90° (say 45°) so that the ribbons will slide together while under the roller pressure during laydown. Consideration of the requirements for nesting and bonding of shaped ribbon indicate that the ribbon should be thicker than APC-2, perhaps about 20 mills, to provide adequate surface for lateral tow movement and bonding.

The above suggests development of customized ribbon having an average width of about 100 mills and a thickness of around 20 mills. Such a ribbon would have a cross sectional area of 1,290,320 sq. microns. With 65% fiber volume, the ribbon would have a fiber count of 43k. It could be made from seven 6k tow, or perhaps four 12k tows.

Using this thicker and narrower ribbon provides the potential to laydown at turning radii less than 5 inches and, since it is larger, to increase laydown rates. However, it may give rise to concerns about achieving adequate isotropic properties and about interlaminer microcracking.

With APC-2 ribbon placement, the ply thickness is 5.6 mills, subquent plys are laid at different angles (0,±45,90 degrees) which provides for isotropic composite properties. For a given composite thickness, using 20 mill ribbon would reduce the degree of isotropy since there would be fewer plies. However, braiding the powdered tows prior to ribbonizing would give some degree of isotropy, but only within the width of the ribbon. A braided and consolidated 20 mill thick ribbon, laid at different angles (0,±45,90), would result in improved isotropy in the composite over unbraided ribbon, but it would have less isotropy than that obtained using APC-2. This could result in somewhat reduced composite mechanical properties, which must be balanced against the potential benefits of automated sharp turn laydown, reduced gaps, and higher placement rates (less labor).

It has been observed that microcracking increases with tow bundle size. That is, when the ply consists of unidirectional fibers, increased ply (ribbon) thickness results in more microcracks. Thus, a 20 mill thick ribbon of unidirectional fibers would be expected to exhibit more microcracks than a 5.6 mill ribbon. If the 20 mill ribbon were made from braided tows, the possibility of microcracking would be reduced.

In view of the above considerations regarding microcracks and isotropy, it seems that we should direct some of our ribbonizing efforts toward shaping (customizing) braided towpreg. We might braid six 12k towpreg yarns and pull the braid through a die to make a 20 mill ribbon. For comparsion, we could pull six 12k towpreg yarns, unbraided, throught the die to make a 20 mill ribbon. These ribbons could be made into test panels to study mechanical properties and microcracks, and compared to thinner ribbons.