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WEAVABILITY OF DRY POLYMER POWDER TOWPREG

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ABSTRACT

Carbon fiber yarns (3k, 6k, 12k) were impregnated with LARC™ thermoplastic polyimide dry powder. Parameters for weaving these yarns were established. Eight-harness satin fabrics were successfully woven from each of the three classes of yarns and consolidated into test specimens to determine mechanical properties. It was observed that for optimum results warp yarns should have flexural rigidities between 10,000 and 100,000 mg-cm. Tow handling minimization, low tensioning, and tow bundle twisting were used to reduce fiber breakage, the separation of filaments, and tow-to-tow abrasion. No apparent effect of tow size or twist was observed on either tension or compression modulus. However, fiber damage and processing costs favor the use of 12k yarn bundles versus 3k or 6k yarn bundles in the weaving of powder-coated towpreg.

INTRODUCTION

In order 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) has developed a method of prepregging carbon fiber with dry thermoplastic and thermosetting polymer powder.

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These efforts at NASA LaRC have focused on two established manufacturing technologies - textiles and robotics. In order to be used in these automated applications, powder-coated towpreg must be produced in the form of either a textile quality yarn or an advanced tow placement (ATP) quality ribbon. This study deals with the former, namely, with textile applications such as powder-coated preforms and broad goods.

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 an alternative to resin transfer molding (RTM) of dry preforms in cases where complex mold geometries and tightly fabricated preforms pose wet-out problems. 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.

One of the objectives of the present study was to develop the weaving protocol for powder-coated yarns. 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 larger tow bundles were used. Offsetting the advantage of using large tow bundles are factors such as potential difficulty in consolidation and possible reduction in composite properties.

In 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 composites made from resultant woven cloth were determined. G30-500* (BASF) and AS-4* (Hercules) carbon fibers in tow bundles of 3k, 6k, and 12k filaments were used. Each was impregnated with a thermoplastic polyimide, LARC™TPI* 1500 medium flow powder (Mitsui Toatsu Chemicals). Weaving was performed on towpreg yarns that had twist levels of zero twist or 15 twists per meter (tpm). After establishing a weaving protocol, an experimental epoxy (AMD-0029*, produced by 3M) was fabricated into towpreg and woven into eight-harness satin fabric.

POWDER PREPREGGING PROCESS

The dry powder prepregging process involves three steps: tow spreading, polymer deposition, and polymer fusion onto the fibers (3). The carbon fiber tow bundle was first pneumatically spread to approximately 8 centimeters in width, then impregnated with powder by means of a dry, recirculating,

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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 10-15 meters/min and over 20,000 meters of towpreg were produced for the current study.

WEAVING CONDITIONS AND PARAMETERS

The primary objective of the weaving study was to learn how to convert powder-coated yarn into quality fabric. The parameters considered in order to establish a weaving protocol for powder-coated towpreg are listed in table I.

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. In this investigation, only tow bundle twisting and shaping were used to reduce the separation of filaments, decrease tow-to-tow abrasion, and minimize fiber loss.

Towpreg flexural rigidity 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.

OBSERVATIONS AND WEAVING PROTOCOL

Towpreg was woven into eight-harness satin fabric under NASA Contract NAS1-18358 by Textile Technologies, Incorporated (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 10.2 cm (4") wide fabric with 394 picks per meter (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 a rapier-type loom (Iwer 1200), which was used instead of a shuttle-type loom in order to minimize damage on the fill yarn. Initial weaving efforts revealed problems with the surface 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 15 twists per meter (tpm). Use of twisted towpreg greatly improved the weaving operation.

Findings from these initial studies can be summarized as follows: the combination of careful rewinding, use of a rapier-type loom, reduction of tension on the warp yarns, and minimizing turns and bends at the loom provided an appropriate protocol for weaving both twisted and untwisted towpreg.

As shown in figure 1, noticeable fiber damage was observed in the woven material. While twisting improves weavability, the action of twisting was found to impart damage to the prepregged 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.

An analysis is given in table III 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.

CONSOLIDATION OF WOVEN FABRIC

Several parameters for consolidating woven powder-coated towpreg fabric were investigated. Special consideration in consolidating woven goods, as distinct from consolidating unidirectional tape, had to be given to the elimination of intra- and inter-tow voids within fabric, as well as the elimination of inter-ply voids that are also of concern in conventional tape processing.

The work of Van West et al. (5) on a consolidation model for commingled fiber yarns stitched and woven into drapeable broadgoods and preforms, and the studies of Iyer and Drzal (6) on powder-impregnated thermoplastic composite consolidation as a two-step process provided guidelines for the consolidation studies. The general steps in the consolidation of woven materials established by these studies are illustrated in figure 2. They are intimate contact of the polymer-polymer interface at numerous sites across the composite, followed by deformation and autohesion, or interdiffusion, of polymer chains to cause the interface to disappear. Resin flow, wetting of fibers, and fiber movement are necessary to eliminate voids and fill the intra- and inter-tow spaces.

In order to follow the cycle shown in figure 2, a vacuum press was used to remove air from void spaces in the LARC™TPI/carbon fiber specimens. At maximum temperature, pressure was applied at 0.05 to 0.15 MPa/min to 4.2 MPa 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 consolidation and stress release at 350°C or 370°C for the unidirectional laminates and 370°C for the woven eight-harness satin prepreg cloth. The part was cooled below Tg at a rate sufficient to stop consolidation before the thickness curve flattened. This avoided resin squeeze-out and resulting dry spots.

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. First, 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 (by ASTM method D790-84a), the transverse flexural strength (7), and the short beam shear strength (ASTM method D2344-84). The mechanical properties generated from these tests for untwisted tow are compared to the tow bundle size in figures 3-5.

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 15 tpm. Flexural strength and modulus (ASTM method D790-84a) values were obtained and compared to untwisted towpreg (table IV). In addition, compression tests were performed by the IITRI method (ASTM method D3410-87, procedure B), where the specimens were 14.0 cm long, 0.64 cm wide, an average of 0.279 cm thick (5.5 in x 0.25 in x 0.110 in), and had a gage length of 1.27 cm (0.50 in). The values for compressive strength, modulus, and Poisson's ratio for twisted tow and untwisted tow are also listed in table IV.

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 20.3 cm long, 2.54 cm wide, an average of 0.374 cm thick (8 in x 1 in x 0.147 in), and had a gage length of 10.2 cm (4 in). These 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 4.45 cm long, 3.81 cm wide, an average of 0.635 cm thick (1.75 in x 1.50 in x 0.250 in), and had a gage length of 2.54 cm (1 in). 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 figures 6 and 7.

DISCUSSION

Learning to use powder-coated tow to make composite materials is an ongoing process. This study has dealt with textile applications, 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.

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. 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. This is 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.

A consolidation cycle for woven towpreg, such as that shown in figure 2, must account for the inter-bundle crimp of the weave. Since a higher resin content is needed to fill the interstitial spaces, in general, composites made from woven material will have a lower fiber volume fraction than those made from unidirectional tape. Further studies will be required to establish the optimum fiber volume fraction for powder-coated preforms. Because of the initial bulk associated with woven materials, vacuum should be applied 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 to stop consolidation, yielded composite specimens of woven material that were void-free, as determined by ultrasonic C-scans.

For unidirectional laminates, if the panels are well consolidated and the fiber and matrix are well distributed within each, 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 3 and 4) with a possible exception in the transverse flexural strength values (figure 5). Unidirectional composites made only with 6k towpreg showed a high flexural strength. This difference may be due to the fact that the 6k material was consolidated at 370°C, whereas the 3k and 12k materials were consolidated at 350°C. 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 "as expected" 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 (figures 6 and 7) 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 figures 6 and 7 represented an averaged 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 displayed little difference from those properties obtained on specimens with untwisted tow (table IV). At 15 tpm the non-alignment effect of fibers in a twisted yarn is negligible (8). 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.

CONCLUDING REMARKS

The weavability of dry, polymer powder-coated towpreg depends upon a number of material properties and equipment parameters. An optimal weaving protocol requires tow handling minimization, low tensioning, and tow bundle twisting. These textile techniques are important factors for automating the production of quality composite parts from powder-coated towpreg preforms.

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 composites. In addition, no effect of twist or tow size was found in the eight-harness satin fabric composites. The lower compression strength that the unidirectional composites made with twisted towpreg displayed was due to apparent fiber damage that occurred during the twisting operation to the tow bundle.

The matter of optimum tow bundle size remains unresolved when comparing the mechanical properties. 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 larger tows are favored, especially in regard to powder processing and weaving costs.

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REFERENCES

1. Marchello, J. M.; and Baucom, R. M.: LARC™ Dry Powder Towpreg Process. *Science of Advanced Materials and Process Engineering Series*, vol. 36, April 1991, pp. 68-80.

2. Hugh, M. K.; Marchello, J. M.; Baucom, R. M.; and Johnston, N. J.: Composites from Powder-Coated Towpreg: Studies with Variable Tow Sizes. *Science of Advanced Materials and Process Engineering Series*, vol. 37, March 1992, pp. 1040-1051.

3. Baucom, R. M.; and Marchello, J. M.: LARC™ Powder Prepreg System. *SAMPE Q.*, vol. 21, no. 4, July 1990, pp. 14-19.

4. Hirt, D. E.; Marchello, J. M.; and Baucom, R. M.: Study of Flexural Rigidity of Weavable Powder-Coated Towpreg. 22nd International SAMPE Technical Conference, November 1990, pp. 360-369.

5. Van West, B. P.; Pipes, R. B.; Keefe, M.; and Advani, S. G.: The Draping and Consolidation of Commingled Fabrics. *Composites Manufacturing*, vol. 2, no. 1, March 1991, pp. 10-22.

a-3.

6. Iyer, S. R.; and Drzal, L. T.: Manufacture of Powder-Impregnated Thermoplastic Composites. *J. Thermoplastic Composite Materials*, vol. 3, October 1990, pp. 325-355.

7. Adams, D. F.; King, T. R.; and Blackketter, D. M.: Evaluation of the Transverse Flexure Test Method for Composite Materials. *Composites Science and Technology*, vol. 39, 1990, pp. 341-353.

8. Zweben, C.; Smith, W. S.; and Wardle, M. W.: Test Methods for Fiber Tensile Strength, Composite Flexural Modulus, and Properties of Fabric-Reinforced Laminates. Fifth Conference on Composite Materials: Testing and Design, ASTM STP 674, 1979, pp. 228-262.

Table I. Weaving Parameters

Towpreg Characteristics

- Yarn Shape
- Amount of Twist
- Yarn Flexibility
- Degree of Yarn Damage

Weaving Characteristics

- Passing Yarn Through Eyelets
- Minimizing Turns and Bends
- Proper Tensioning
- Heddles and Reed Action

Final Parts

- Optimal Resin Content
- Bulk Factor

Table II. Flexural Rigidity

Description	Overhang (cm)	Areal Weight (mg/cm^2)	Rigidity (mg cm)
Twisted tow, 6k LARC™TPI	10.16	8.27	1,100
Twisted woven cloth, 6k LARC™TPI	8.26	45.02	3,200
Twisted tow, 6k LARC™TPI	22.86	5.62	8,400
Twisted woven cloth, 6k LARC™TPI	17.78	43.35	30,500
Twisted tow, 12k LARC™TPI	13.97	20.85	7,100
Twisted woven cloth, 12k LARC™TPI	10.16	87.64	11,500
Untwisted tow, 12k LARC™TPI, 34.6% w/w resin	17.15	20.15	12,700
Untwisted tow, 12k 3M epoxy, 32% w/w resin	12.70	19.84	5,100

Table III. Towpreg 8HS Fabric

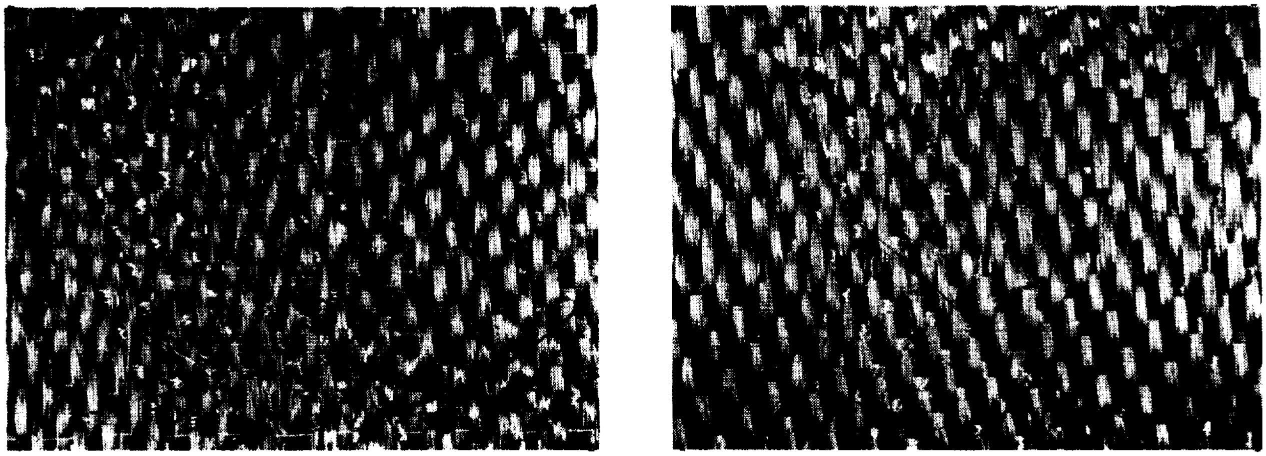
Towpreg Specification	Weave Count (ppi)	Weave Count (ppm)	Weight (g/m^2)	Thickness (cm)
6k (G30-500) / LARC™TPI, No Twist	10.2 x 9.8	402 x 386	478.4	.170
6k (G30-500) / LARC™TPI, Twisted Tow	10.2 x 9.8	402 x 386	483.7	.180
6k (G30-500) / LARC™TPI, No Twist	10.1 x 10.0	398 x 394	448.2	.196
6k (G30-500) / LARC™TPI, Twisted Tow	10.2 x 9.3	402 x 366	499.4	.262
12k (AS-4) / LARC™TPI, Twisted Tow	8.2 x 8.2	323 x 323	810.5	.320
3k (G30-500) / LARC™TPI, Twisted Tow	20.0 x 19.8	787 x 780	428.1	.147

Table IV. Twisted and Untwisted Towpreg Properties in Unidirectional 12k AS-4 Carbon Fiber / LARC™ Thermoplastic Polyimide (TPI)

Mechanical Properties	Non-twisted towpreg	Twisted towpreg (15 tpm)
Flexural Strength† (MPa)	1760 ± 97	1713 ± 110
Flexural Modulus† (GPa)	107.5 ± 1.7	111.9 ± 2.8
Compression Strength (MPa)	1140 ± 84	968 ± 67
Compression Modulus (GPa)	118.2 ± 5.5	108.4 ± 6.3
Poisson's Ratio††	0.345 ± 0.023	0.382 ± 0.030

† Values have been normalized for 60% fiber volume fraction.

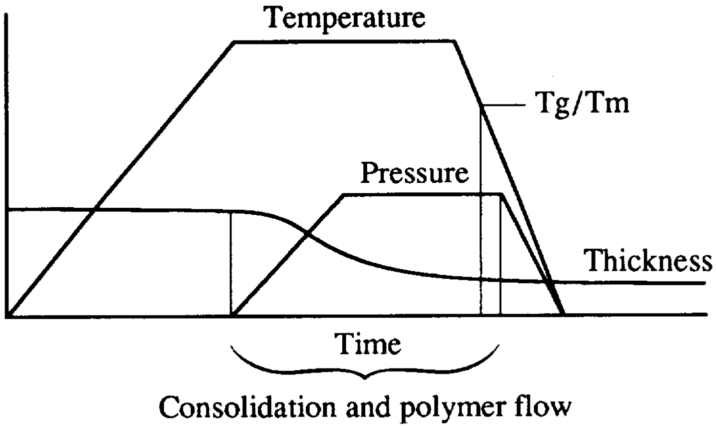
†† Based on IITRI compression data (by ASTM method D3410-87, procedure B).



Using twisted towpreg

Using non-twisted towpreg

Figure 1. Photographs of eight-harness satin fabric from 6k carbon fibers and LARC™TPI powder.



- Vacuum is used to eliminate air voids.
- Pressure ramp allows time for fiber movement into a compact arrangement with minimum fiber crimping and breakage.
- Pressure ramp also provides time for resin flow and adhesion.
- Holding temperature above Tg or Tm anneals the composite and relieves internal stresses.
- Cooling below Tg or Tm stops consolidation before thickness curve flattens and avoids resin squeeze out and resulting dry spots.

Figure 2. Woven towpreg cure cycle.

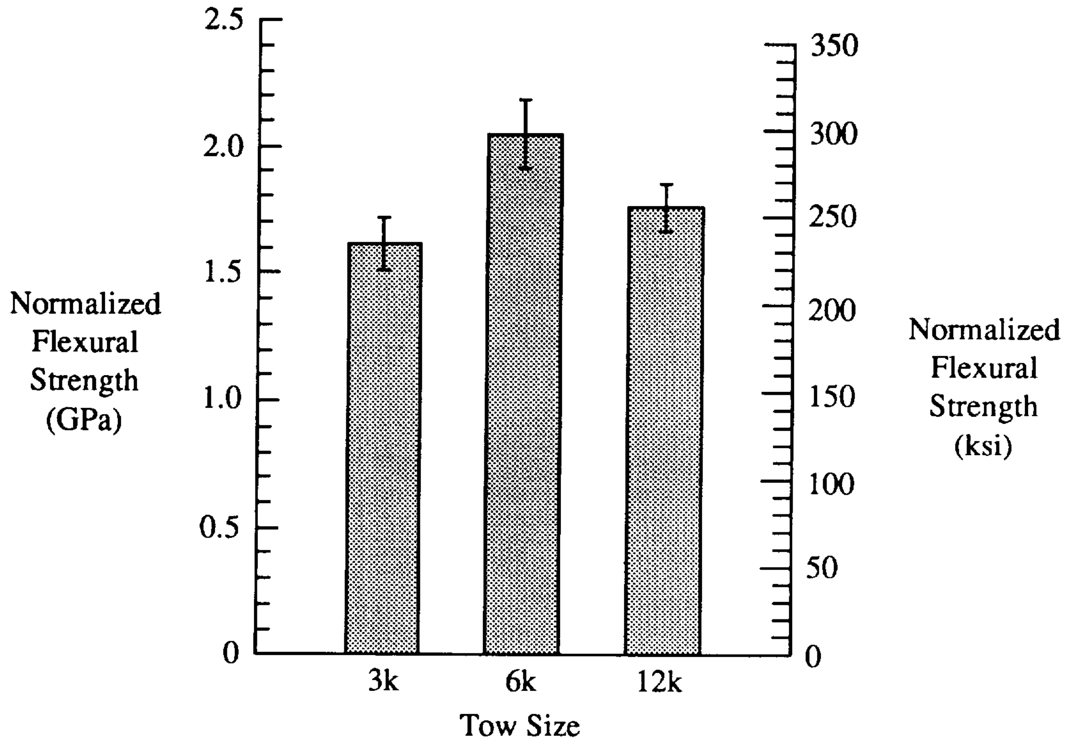


Figure 3a. Normalized flexural strength vs. tow size in untwisted, unidirectional composites of LARC™TPI/AS-4 or G30-500 carbon fibers.

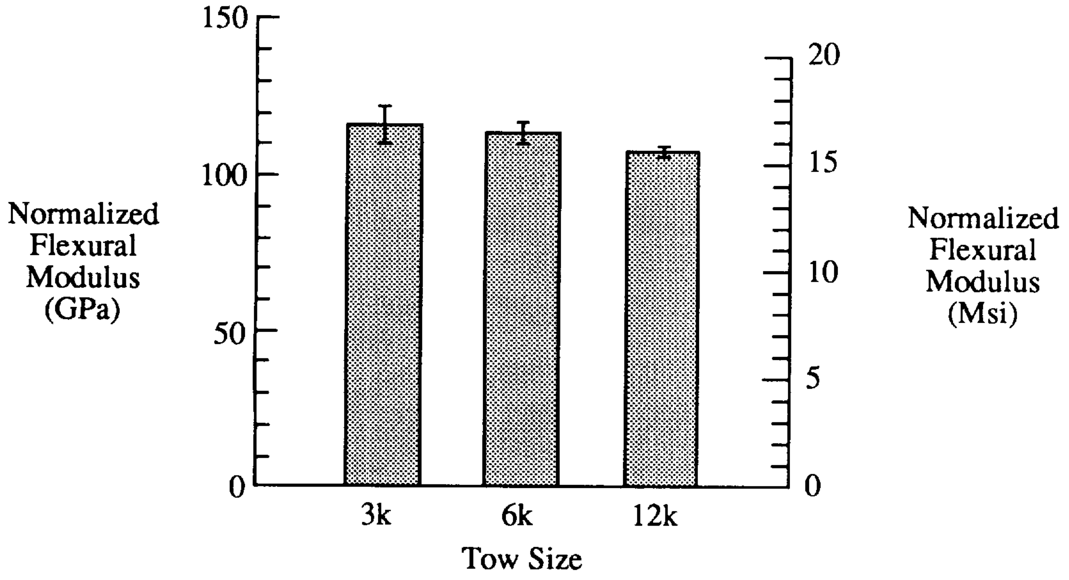


Figure 3b. Normalized flexural modulus vs. tow size in untwisted, unidirectional composites of LARC™TPI/AS-4 or G30-500 carbon fibers.

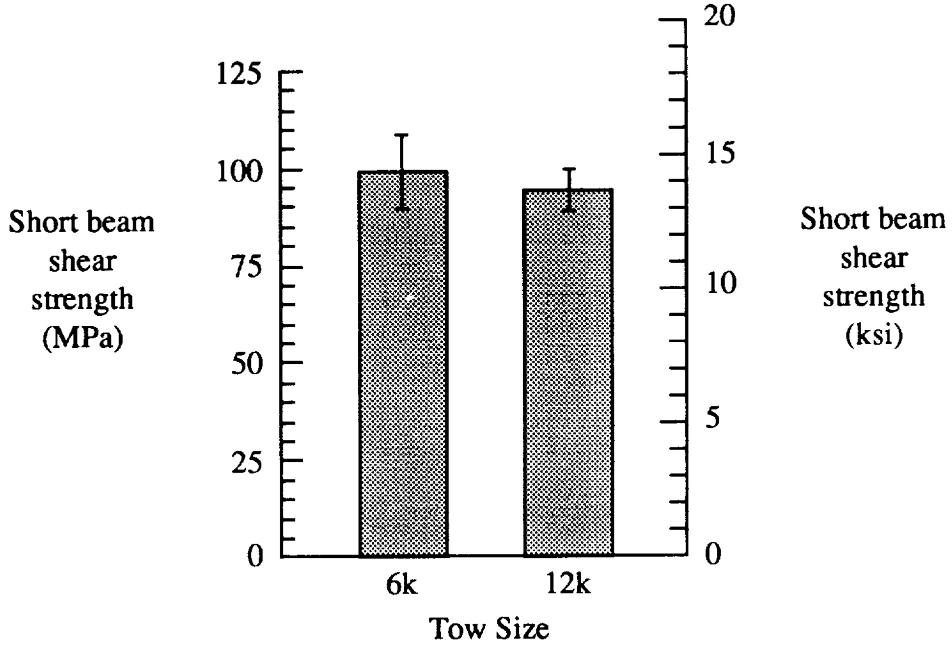


Figure 4. Short beam shear strength vs. tow size in untwisted, unidirectional composites of LARC^{TM}TPI / AS - 4 or G30 - 500 carbon fibers.

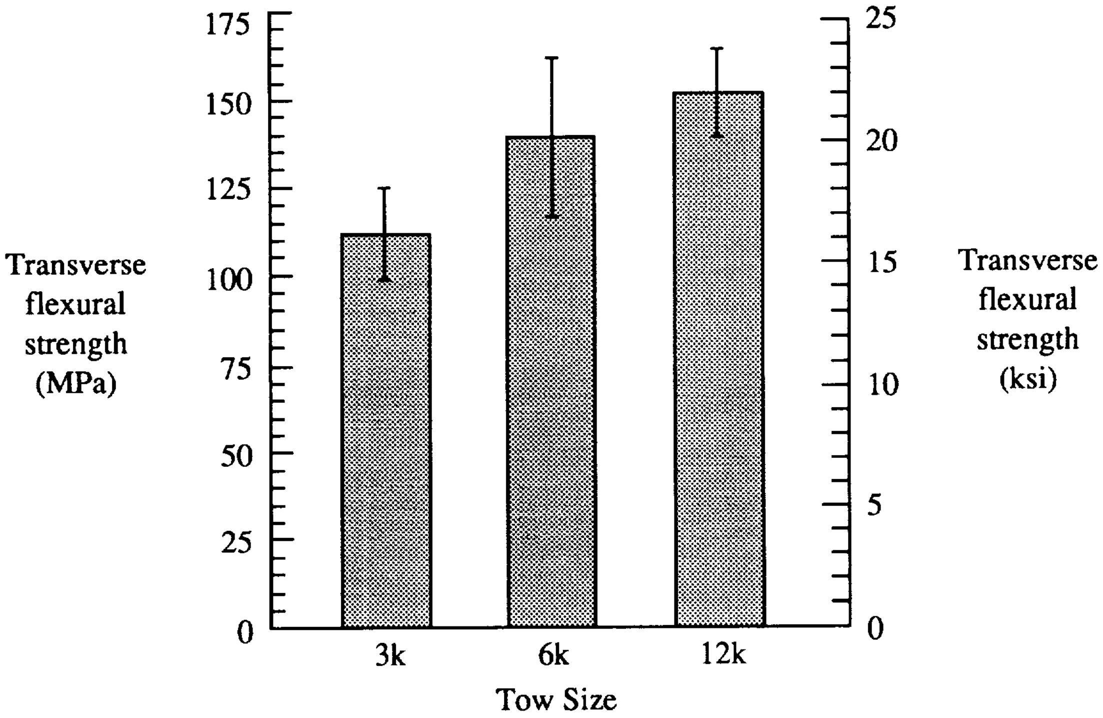


Figure 5. Transverse flexural strength vs. tow size in untwisted, unidirectional composites of LARC^{TM}TPI / AS - 4 or G30 - 500 carbon fibers.

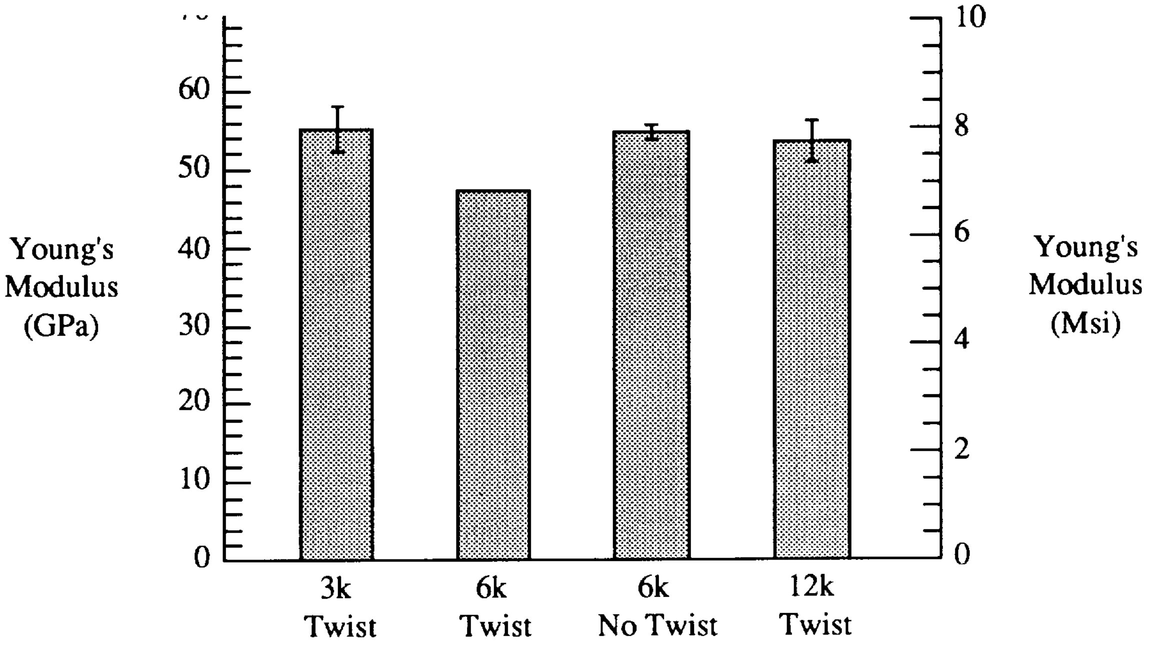


Figure 6. Young's modulus vs. tow size for eight-harness satin woven composites of LARC™TPI/AS-4 or G30-500 carbon fibers. Data collected from tension tests performed in the warp direction.

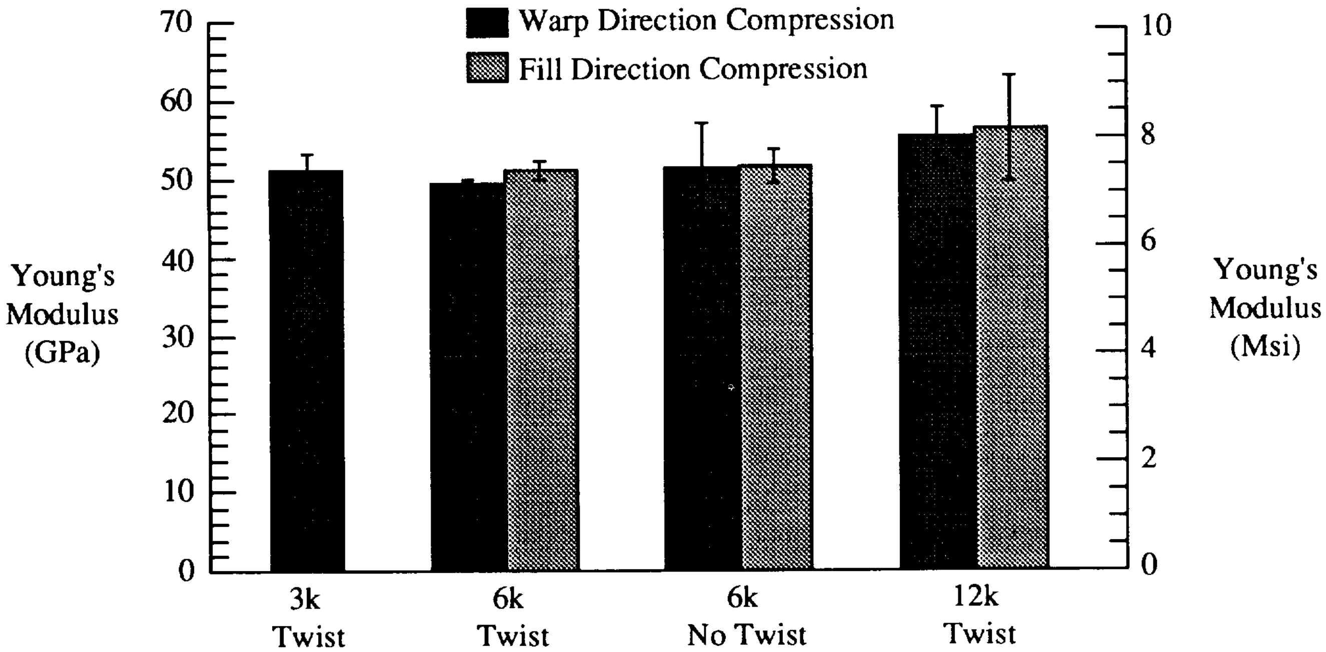


Figure 7. Young's modulus vs. tow size for eight-harness satin woven composites of LARC™TPI/AS-4 or G30-500 carbon fibers. Data collected from short block compression tests performed in the warp and fill directions.