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DEVELOPMENT OF PRESTRESSED GRAPHITE PROCESSING TECHNIQUES

FINAL REPORT

7 JULY 1976

Prepared by:

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Space Division
North American Rockwell

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ABSTRACT

Preliminary experimentation involving selective breaking of graphite and boron filaments in prepreg tape by prestressing has demonstrated a significant increase in the tensile strength and a corresponding sizeable reduction in the dispersion of test valves in the cured composite as compared to unprocessed or unstressed prepreg cured laminate test data. These observations resulted from preliminary testing of laminates fabricated from prestress processed graphite prepreg tape and compared to as-received unprocessed material.

The purpose of this contract was to conduct a comprehensive evaluation of the concept of prestressing graphite fibers by fabricating laminates from as-received and prestressed prepreg and testing statistically significant sample sizes for unidirectional tension, angle ply tension, short beam shear, and compression strength.

Commercially available high strength graphite/epoxy prepreg and pitch type graphite/epoxy prepreg were prestressed by pulling the material under constant tensile load in a reduced temperature environment over various stressing roller diameters in order to selectively break weak fiber sites within the fiber bundles prior to lamination and cure.

Mechanical properties of laminates fabricated from prestressed and as-received material determine what improvements in mechanical strengths and coefficients of variation occurred as a result of the prestressing operation.

Test data generated by this program indicate an improvement in some mechanical properties due to prestressing while others are unaffected. Specifically, prestressing shows a distinct improvement in unidirectional tension and short beam shear as well as in angle ply tension, and no changes in compression or transverse properties.

FOREWORD

The work described in this report was performed by Mr. G. Brown and Mr. D. Waterman under Contract No. N62269-75-C-0374, "Development of Prestressed Graphite Processing Techniques", and administered by the Structures Research Branch, Structures Division, of the Naval Air Development Center, with Mr. E. Deska acting as contract monitor.

This is a Final Report covering work conducted from May 1975 to February 1976.

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SUMMARY

Commercially available graphite/epoxy prepreg (AS/3501) was purchased from Hercules Corporation. Pitch type graphite fiber was purchased from Union Carbide, and prepreged with the 3501 resin system into three inch wide tape by Hercules Corporation.

Laminates of the as-received materials were fabricated and mechanically tested to provide baseline data for comparison with prestressed laminate test data.

Prestressing of the prepreg tapes was performed by drawing the tape under constant tensile load through a double set of stressing rollers in a reduced temperature chamber. Variation in the level of stress induced in the prepreg tape was accomplished by utilization of different stress roller diameters and exposure temperatures. A number of stressing conditions were evaluated to establish the optimum conditions for providing the best overall improvement in mechanical strength properties as compared to the initial baseline laminate property data.

The prestress condition selected for processing the prepreg tape for the mechanical test specimens consisted of 0.50 inch diameter stress rollers and an exposure temperature of $0^{o}F$. The laminate test data for graphite/epoxy (AS/3501) shows an improvement in the "B" design allowable values for unidirectional tension of 32%, angle ply tension of 15%, and unidirectional short beam shear of 5% due to prestressing. Test data for unidirectional and angle ply transverse tension, angle ply short beam shear, and unidirectional and angle ply compression show no change within a normal data spread due to prestressing.

A preliminary examination of the effect of prestressing on unidirectional tension for high modulus pitch type fiber prepreg was also performed. "B" design allowable values for prestressed laminates were in the 67,000 psi range, with a modulus at 29 million psi compared to a 48,000 psi "B" design allowable for the as-received material. This represents a 39% improvement in "B" design values due to prestressing.

Since only a preliminary examination was performed on this material, it is possible that with a comprehensive effort on prestressed pitch prepreg, that the "B" design allowable tensile strength could be improved to 100,000 psi with a 29×10^{6} psi modulus, thus providing an inexpensive structural material.

TECHNICAL DISCUSSION

Typical static test data on graphite/epoxy laminates demonstrate a spread in the test values with a coefficient of variation of approximately 10%. The coefficient of variation and average strength of a composite material determine the "A" or "B" design allowable which the structural engineer can use for a specific design application.

By selectively breaking weak sites through prestress conditioning of graphite prepreg tape, the failure mode within the cured laminate is altered. Elimination of weak sites precludes failure within the laminate at low load levels, increasing the average strength and reducing the coefficient of variation, or spread of test data from certain mechanical tests. Structures fabricated from prestressed material will provide a higher level of load carrying capability.

Acceptance of incoming AS/3501 prepreg was based upon unidirectional and transverse flexural strength and modulus. Table 1 lists the test results. Flexural strength and modulus values were well within acceptable limits.

TABLE 1

Acceptance Test Values of AS/3501 Prepreg

Orientation	Flexural Strength	Flexural Modulus
0^{o}	259,600 psi	16.5×10^{6}
90^{o}	14,200 psi	1.2×10^{6}

Resin Content %/Wt. 32.3
Fiber Content %/Vol. 64.6

Ten specimens from laminates fabricated of as-received AS/3501 material were tested to provide a data base for each of the following fiber orientation and mechanical tests: unidirectional tension and modulus; $(0^{o} \pm 45^{o})_s$ angle ply tension and modulus; transverse angle ply tension; transverse unidirectional tension; unidirectional and $(0^{o} \pm 45^{o})_s$ angle ply short beam shear; and unidirectional and $(0^{o} \pm 45^{o})_s$ angle ply compression and modulus. Test methods used were AMS-3894, 4.5.6 for tension, 4.5.7 for compression, and 4.5.9 for short beam shear strength. Tables 2 and 3 present the data, coefficient of variation, and the resulting "A" and "B" design allowable values for each property tested.

TABLE 2

3501-AS

Unstressed Unidirectional & Angle Ply Tension & Modulus, Transverse Unidirectional & Transverse Angle Tension & Modulus

Orientation	No. of Specimens	Avg. Tensile Psi	Avg. Tensile Modulus	Coefficient of Variation	"A" Design	"B" Design
Unidirectional 0^o	27	189,000	18.2×10^6	9.4%	133,900	157,000
Angle Ply $(0^o \pm 45^o)_s$	30	93,000	9.8×10^6	17.3%	43,800	64,500
Unidirectional 0^o Transverse	28	6,490	1.4×10^6	30%	503	3,015
Angle Ply $(0^o \pm 45^o)_s$ Transverse	30	26,700	2.6×10^6	7.5%	20,500	23,100

TABLE 3

3501-AS

Unstressed Unidirectional & Angle Ply Short Beam Shear, Unidirectional & Angle Ply Compression & Modulus

Orientation	No. of Specimens	Avg. Strength Psi	Avg. Modulus	Coefficient of Variation	"A" Design	"B" Design
Unidirectional 0^o Short Beam Shear	30	17,900		3.4%	16,020	16,800
Angle Ply $(0^o \pm 45^o)_s$ Short Beam Shear	30	14,400		7.1%	11,260	12,590
Unidirectional 0^o Compression	20	129,900	16.1×10^6	4.7%	109,700	118,100
Angle Ply $(0^o \pm 45^o)_s$ Compression	20	80,900	9.0×10^6	5.7%	65,800	72,100

Acceptance of 3501 pitch type prepreg was based on quantity and the prepreger's best effort only. Typical test values for pitch laminates had not been established at the start of this contract.

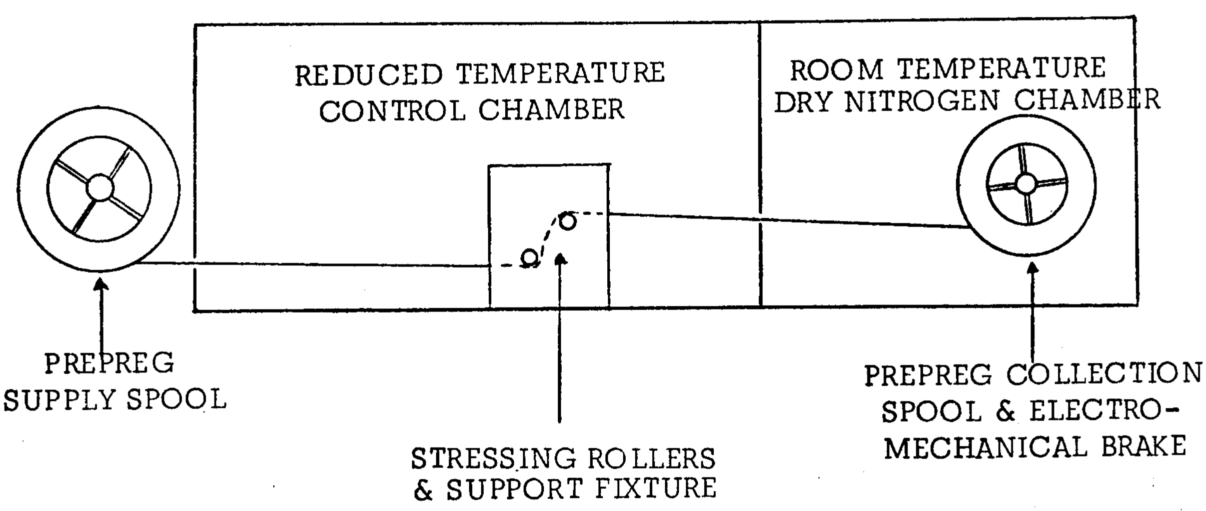
Unidirectional tension and modulus was the only property determination made for pitch graphite laminates. Table 4 lists the test data derived on as-received and prestressed pitch fiber laminates. Prestressed data shows a 39.7% improvement in the "B" design allowable value.

Prestressing Technique

Prestressing of the three inch wide graphite prepreg tape was accomplished by pulling the material over a double set of rollers contained in a temperature controlled chamber capable of maintaining an environment of -50 to 50°F. A constant load was applied to the tape by means of an electromechanical brake. A diagram of the prestressing equipment is shown in Figure 1.

FIGURE 1

PRESTRESSING CHAMBER
SIDE VIEW



Prior to the tape exiting to the atmosphere, it is passed through a dry nitrogen chamber maintained at room temperature to prevent any condensation of moisture on the surface of the tape.

TABLE 4

Unstressed & Prestressed Unidirectional Tension & Modulus for 3501 Pitch Prepreg

Condition	No. of Specimens	Avg. Tensile Strength	Avg. Tensile Modulus	Coefficient of Variation	"A" Design	"B" Design
Unstressed	10	69,100 psi	29.1×10^6	12.9%	33,900	48,400
Stressed at 50°F Over 1.50" Rollers	10	78,000	29.0×10^6	9.2%	49,600	61,300
Stressed at 40°F Over 1.50" Rollers	19	80,900	30.0×10^6	8.5%	58,100	67,600

Pitch fiber prepreg stressed at 40°F over 1.50" diameter rollers shows a 39.7% in "B" design allowable over the unstressed pitch laminate.

Selection of Prestressing Condition

Five different stressing conditions were investigated for the AS/3501 prepreg material. Table 5 lists the stressing conditions. For each condition a minimum of ten unidirectional tension specimens were fabricated and tested from the prestressed prepreg. The average tensile strength and coefficient of variation of test results were calculated for each of the five prestressing conditions.

The prestressing condition selected for development of a comprehensive prestressed mechanical data base was condition number 1, 0°F over .500" diameter rollers. This condition was selected based upon the high average tensile strength, low coefficient of variation, and least severe stressing conditions as compared to the other stressing conditions investigated.

Two different stressing conditions were investigated for the Pitch/3501 prepreg material. The stressing conditions for the pitch type prepreg are also listed in Table 5.

Because of the small quantity of prepreg received from the manufacturer, only two stressing conditions were investigated. The second condition, (2) to 40°F over 1.5" rollers, was selected for further evaluation based upon a higher average tensile strength and a lower dispersion of test values.

TABLE 5

Prestressing Conditions for AS/3501 Prepreg

Condition	Chamber Temp. ($^{\circ}$F)	Roller Size (in.dia.)
1	0	.500"
2	-25	.310"
3	-50	.500"
4	0	.310"
5	-40	.500"

Prestressing Conditions for Pitch Graphite/3501

Condition	Chamber Temp. ($^{\circ}$F)	Roller Size
1	50	1.5"
2	40	1.5"

The prepreger encountered considerable difficulty in producing useable prepreg tape from the pitch fiber. The weak tow strength of the fiber caused excessive tow breakage resulting in only a little over 2-1/2 pounds of useable prepreg for the pitch prestressing evaluation.

Tables 6 and 7 show the average strengths, "A" and "B" design allowable and coefficient of variation for the prestressed AS/3501 laminate properties. Tables 8 thru 16 are as-received and prestressed data presented as histograms of failure frequency versus strength at failure. All individual test values, and test values from the prestress conditions listed in Table 5, are presented in Appendix A.

TABLE 6

Prestressed Unidirectional & Angle Ply Tension & Modulus, Transverse Unidirectional & Angle Ply Tension & Modulus Stressed at $0^{o}F$ Over .500" Rollers

Orientation	No. of Specimens	Avg. Tensile Strength	Avg. Tensile Modulus	Coefficient of Variation	"A" Design	"B" Design
Unidirectional 0^{o}	28	221,500	18.3×10^{6}	6.5%	177,200	195,800
Angle Ply $(0^{o} \pm 45^{o})_s$	20	93,060	9.5×10^{6}	10.1%	62,200	75,060
Unidirectional 0^{o} Transverse Tension	20	5,312	1.4×10^{6}	17.2%	2,314	3,562
Angle Ply $(0^{o} \pm 45^{o})_s$ Transverse Tension	20	25,700	2.6×10^{6}	6.8%	19,950	22,340

TABLE 7

Prestressed Unidirectional & Angle Ply Short Beam Shear, Prestressed Unidirectional & Angle Ply Compression & Modulus Stressed at $0^{o}F$ Over .500" Rollers

Orientation	No. of Specimens	Avg. Shear & Compression Strength	Avg. Compression Modulus	Coefficient of Variation	"A" Design	"B" Design
Unidirectional 0^{o} Short Beam Shear	30	19,070		4.3%	16,530	17,600
Angle Ply $(0^{o} \pm 45^{o})_s$ Short Beam Shear	20	14,820		8.0%	10,940	12,560
Unidirectional 0^{o} Compression	20	127,000	15.4×10^{6}	7.8%	94,600	108,115
Angle Ply $(0^{o} \pm 45^{o})_s$ Compression	20	80,900	8.4×10^{6}	5.0%	67,500	73,100

TABLE 8

UNIDIRECTIONAL 0° TENSION
AS/3501

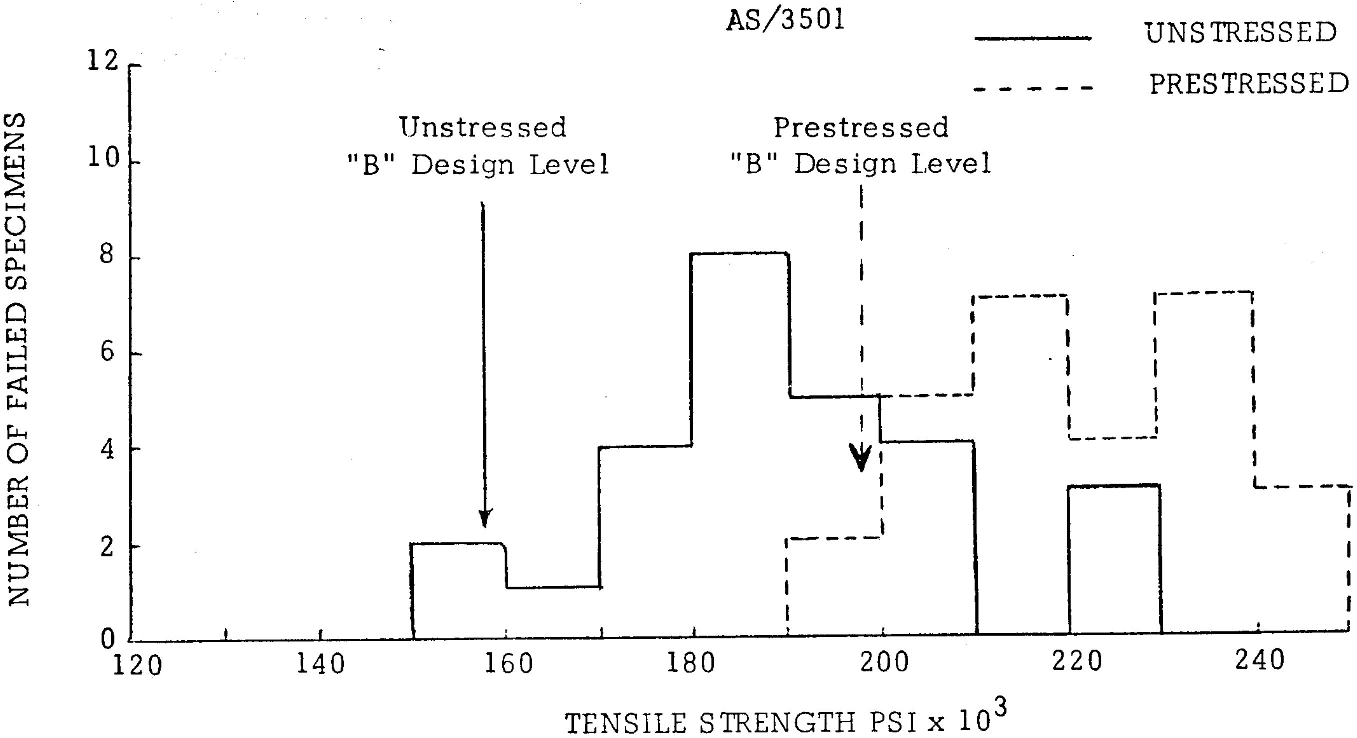


TABLE 9

ANGLE PLY $(0^{\circ} \pm 45^{\circ})_s$ TENSION
AS/3501

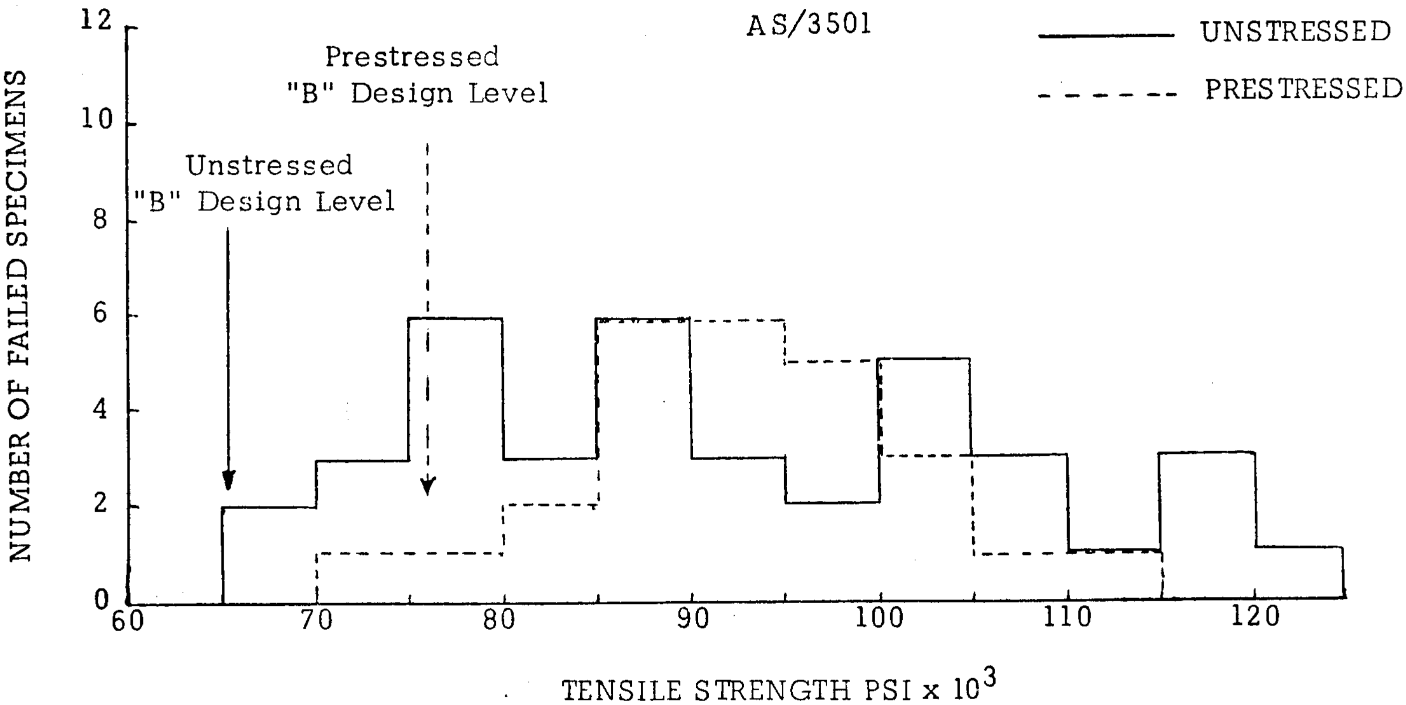


TABLE 10

UNIDIRECTIONAL 0° COMPRESSION

AS/3501

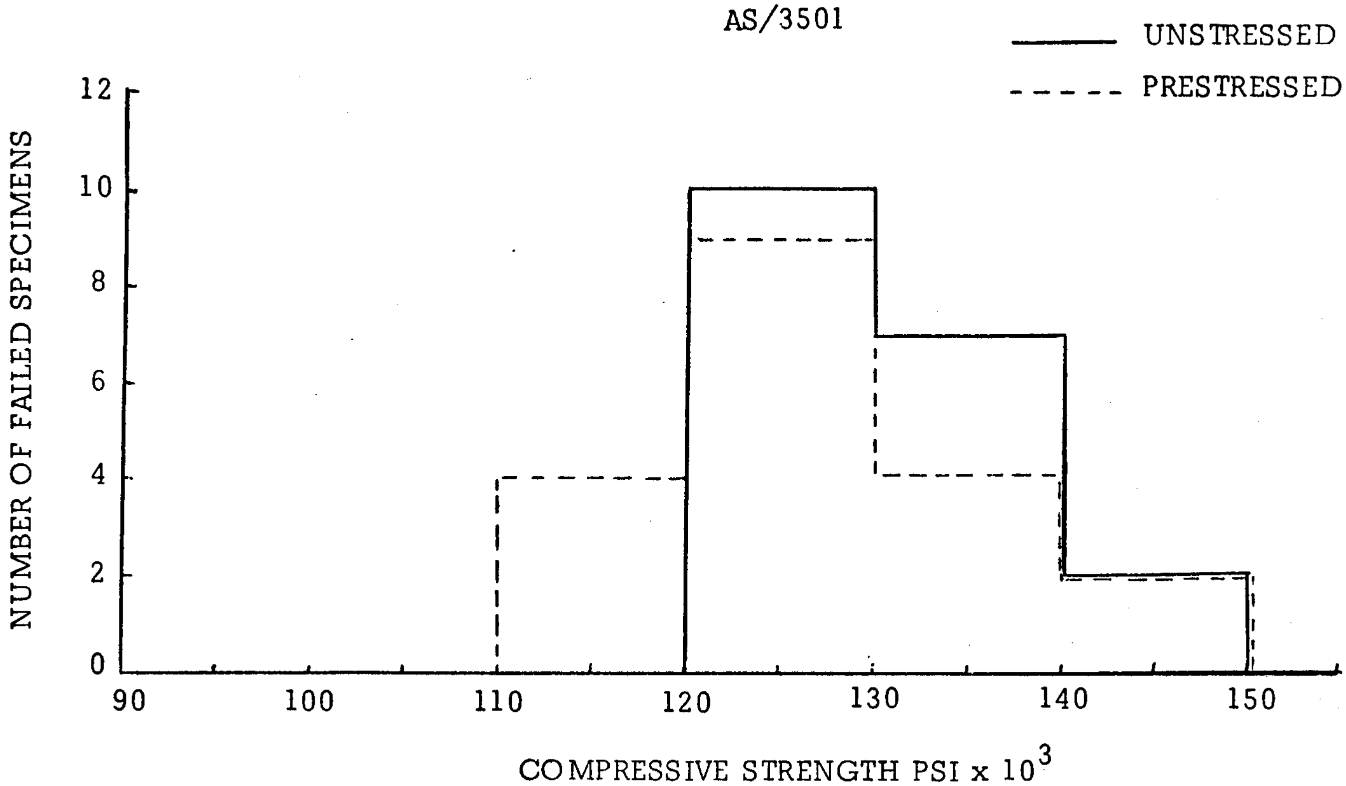


TABLE 11

ANGLE PLY $(0^{\circ} \pm 45^{\circ})_S$ COMPRESSION

AS/3501

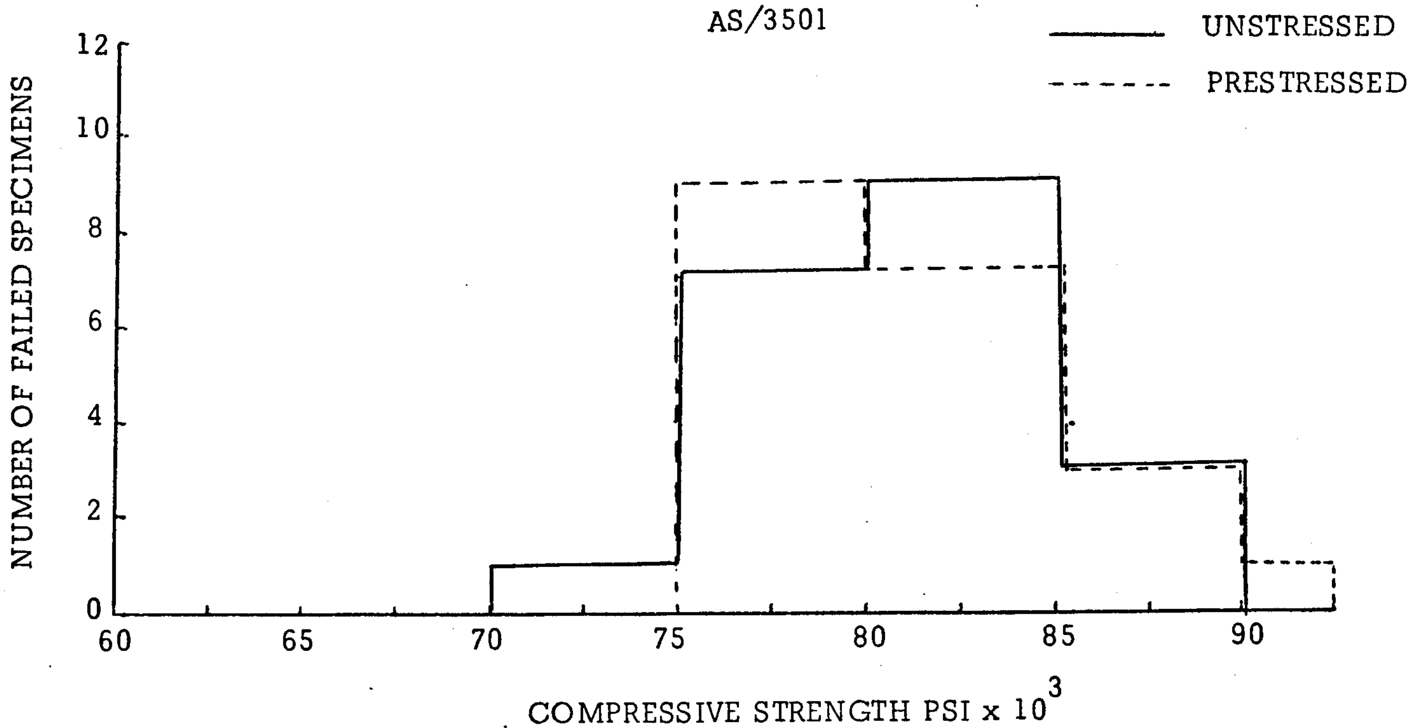


TABLE 12

UNIDIRECTIONAL 0°
AS/3501
TRANSVERSE TENSION

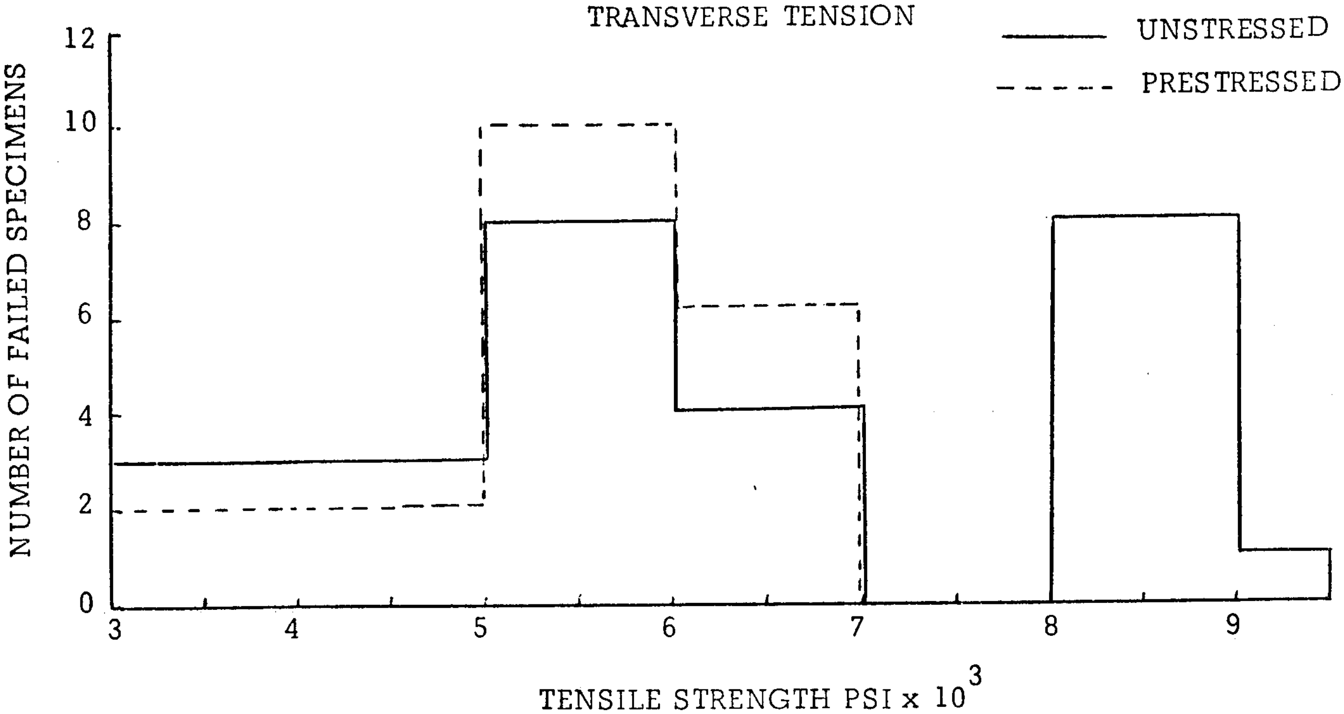


TABLE 13

ANGLE PLY $(0^{\circ} \pm 45^{\circ})_s$
AS/3501
TRANSVERSE TENSION

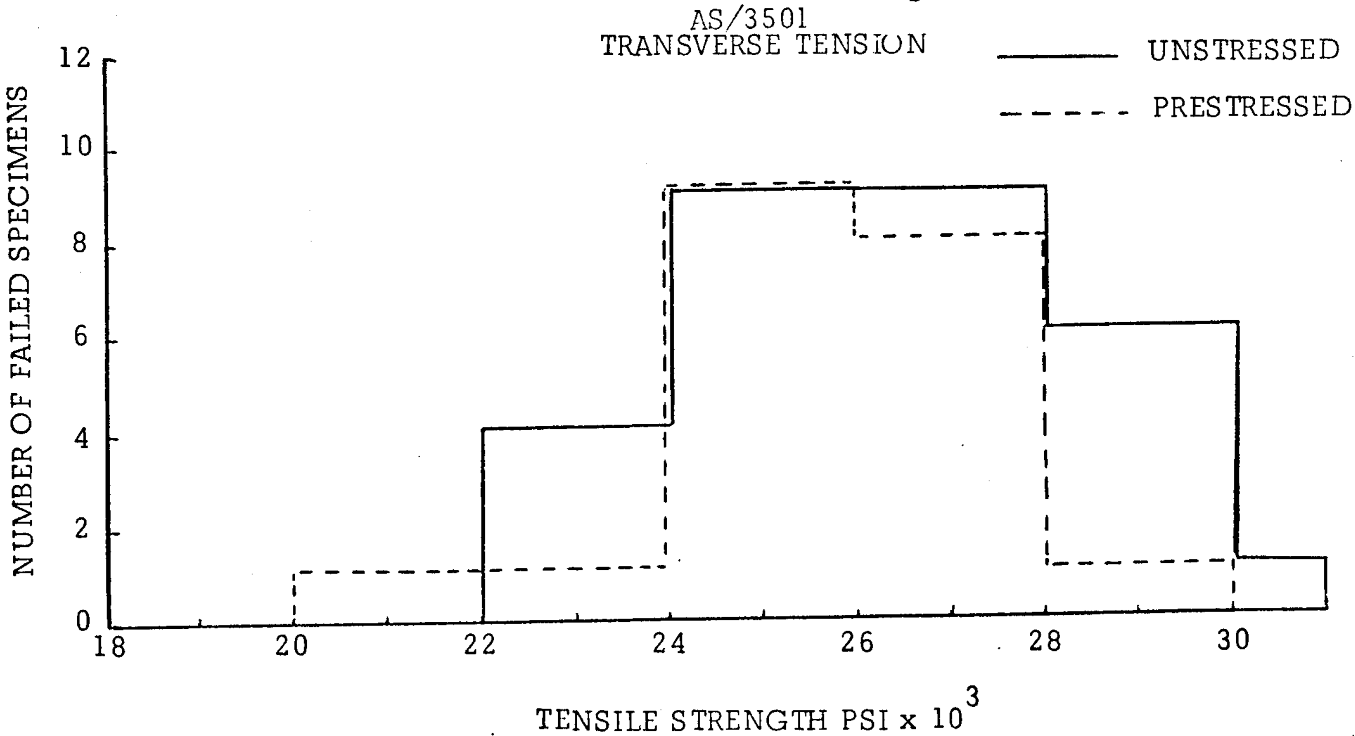


TABLE 14

UNIDIRECTIONAL 0°
AS/3501
SHORT BEAM SHEAR

——— UNSTRESSED
- - - - PRESTRESSED

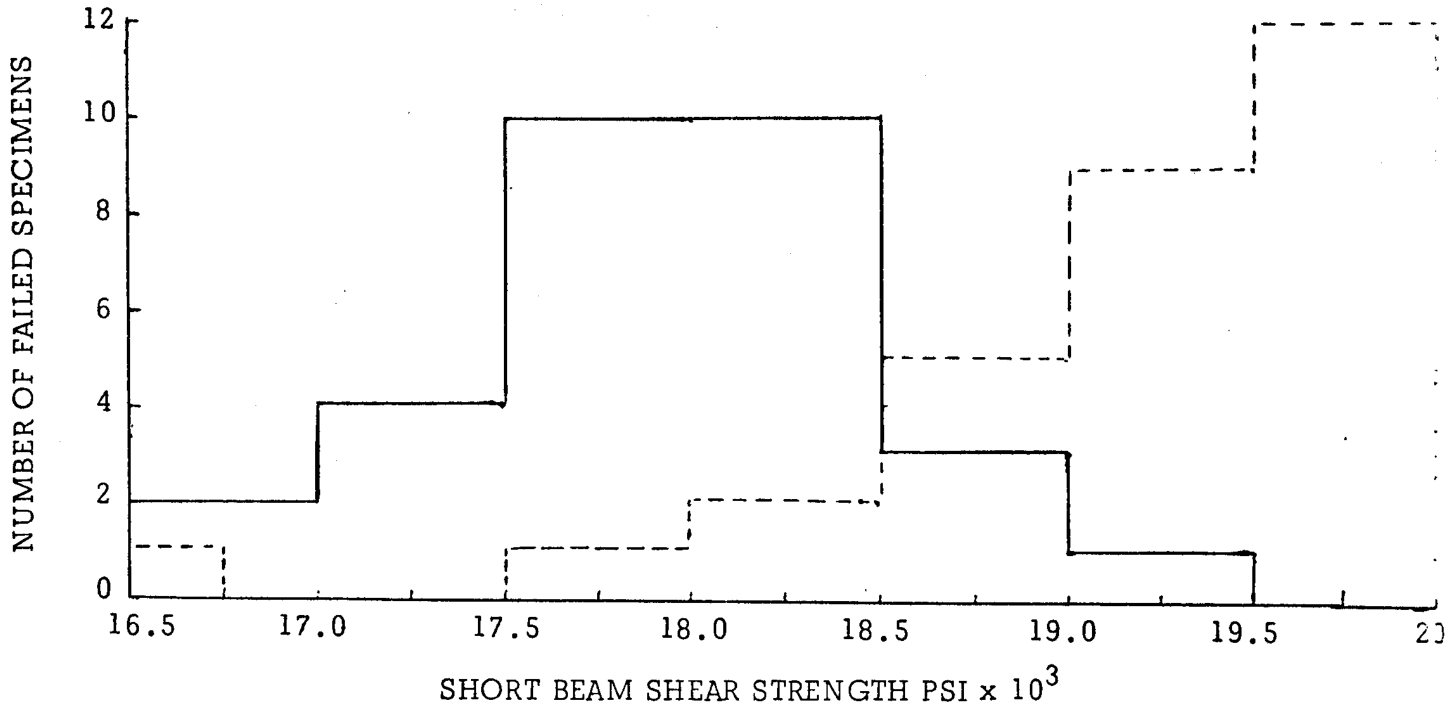


TABLE 15

ANGLE PLY $(0^{\circ} \pm 45^{\circ})_s$
AS/3501
SHORT BEAM SHEAR

——— UNSTRESSED
- - - - PRESTRESSED

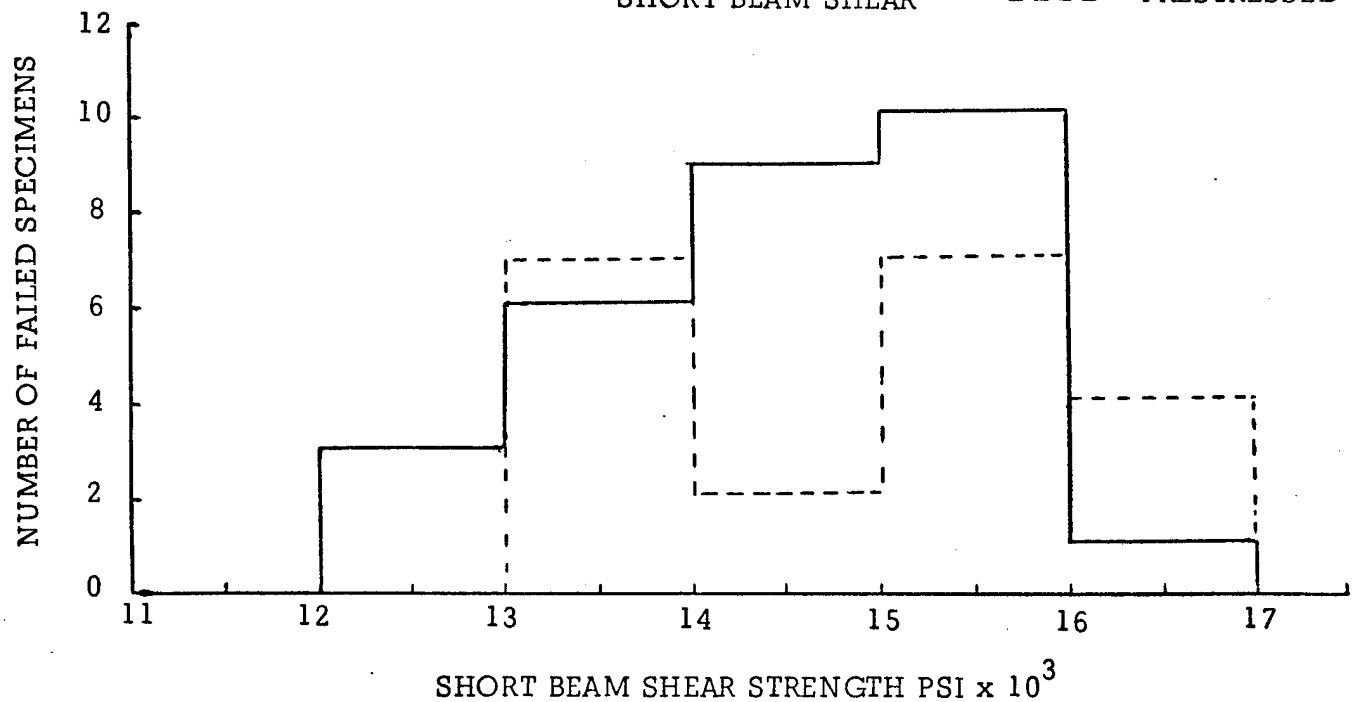
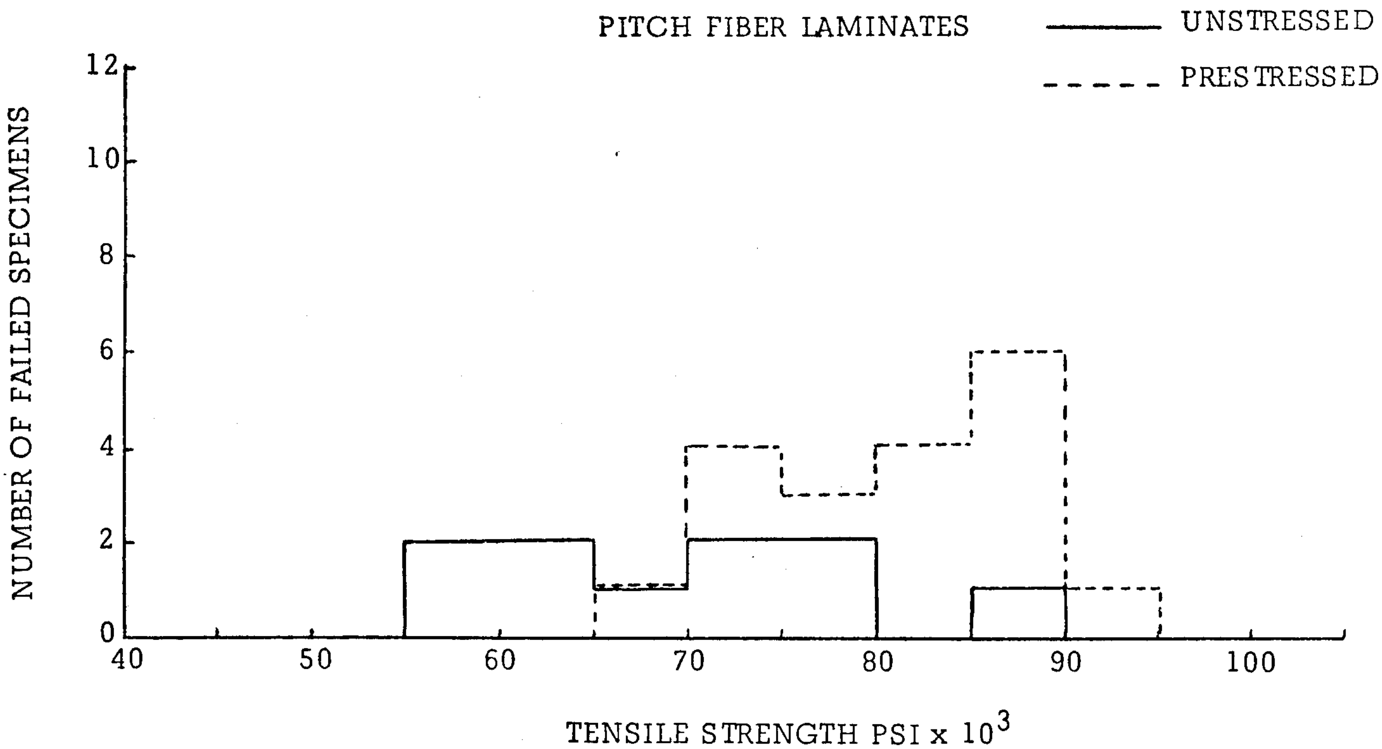


TABLE 16
UNIDIRECTIONAL 0° TENSION



CONCLUSIONS & RECOMMENDATIONS

Conclusions

Based on the test results presented in this final report on the as-received and prestressed AS/3501 prepreg system from Hercules Corporation, it is apparent that prestressing provided a significant increase (32%) in the "A" and "B" design allowables, higher loads at failure and a higher confidence level for most of the unidirectional tensile properties tested. Prestressed angle ply tension test results show an improvement of approximately 15% in design values, while compression test results indicate little or no improvement due to prestressing. Apparently the effect that prestressing demonstrated in the unidirectional laminate properties does not translate completely into the angle ply laminates with the same affect for this particular prepreg system.

Only a small data base was generated for pitch unidirectional tension, however, test results for 3501 pitch laminates demonstrate a 40% improvement in the "A" and "B" unidirectional tension design allowable.

In a comprehensive evaluation of prestressed pitch laminates, it is conceivable that an even greater increase in mechanical properties could be generated; i.e., tension properties approaching 100,000 psi. Whether or not the effect of prestressing would be seen in angle ply properties would also be determined in such an evaluation.

Recommendations

Because of the potential of prestressed pitch prepreg yielding a low cost, viable structural material, a comprehensive investigation should be undertaken to establish what improvements could be obtained by determining the optimum stressing conditions for unidirectional pitch epoxy prepreg laminates. This investigation should also include an evaluation of prestressed angle ply laminate properties for as-received pitch prepreg to determine if the improvement in unidirectional pitch laminate properties translates to prestressed angle ply laminate properties.

TABLE A-1

STRESSED AT $-40^{\circ}F$ OVER .500 INCH DIAMETER ROLLERS

Test Specimen	Unidirectional Tensile Strength Psi	Modulus Psi x 10^6
1	224,000	19
2	248,000	18
3	217,000	18
4	222,000	18
5	215,000	18
6	222,000	19
7	225,000	18
8	229,000	18
9	241,000	19
10	240,000	18
Average	228,000	18

Standard Deviation 4.8%
"A" Design Allowable 184,700
"B" Design Allowable 202,600

TABLE A-2

HERCULES AS/3501 GRAPHITE EPOXY TENSILE STRENGTH & MODULUS

STRESSED AT $0^{o}F$ OVER .310 INCH DIAMETER ROLLERS

Test Specimen	Unidirectional Tensile Strength Psi	Modulus Psi x 10^{6}
1	217,000	18
2	222,000	17
3	214,000	18
4	198,000	18
5	224,000	18
6	213,000	18
7	227,000	18
8	222,000	19
9	237,000	18
10	224,000	18
Average	220,000	18

Standard Deviation 4.7%
"A" Design Allowable 179,200
"B" Design Allowable 195,900

TABLE A-3

STRESSED AT -25°F OVER .310 INCH DIAMETER ROLLERS

Test Specimen	Unidirectional Tensile Strength Psi	Modulus Psi x 10^6
1	226,000	18
2	223,000	18
3	215,000	17
4	225,000	19
5	228,000	19
6	227,000	17
7	229,000	18
8	226,000	18
9	206,000	19
10	214,000	17
Average	222,000	18

Standard Deviation 3.4%
"A" Design Allowable 191,900
"B" Design Allowable 204,200

TABLE A-4

HERCULES AS/3501 PREPREG

STRESSED AT -50°F & .500" DIA. ROLLERS

Test Specimen	Unidirectional Tensile Strength Ksi
1	169.9
2	182.7
3	184.0
4	186.6
5	188.2
6	189.0
7	189.7
8	193.4
9	197.9
10	200.0
11	200.4
Mean	200.3 Ksi

Standard Deviation 4.7%
"A" Design Allowable 155.5 Ksi
"B" Design Allowable 169.4 Ksi

TABLE A-5

HERCULES AS/3501 PREPREG

STRESSED AT $-25^{o}F$ & .310" DIA. ROLLERS

Test Specimen	Unidirectional Tensile Strength Ksi
1	178.4
2	181.4
3	188.2
4	195.9
5	198.0
6	205.1
7	207.6
8	211.3
9	217.2
10	225.1
Mean	200.8

Standard Deviation 7.6%
"A" Design Allowable 140.6 Ksi
"B" Design Allowable 165.3 Ksi

TABLE A-6

HERCULES AS/3501 GRAPHITE EPOXY
SHORT BEAM SHEAR STRENGTH

STRESSED AT 0°F OVER .310 INCH DIAMETER ROLLERS

Test Specimen	Horizontal Shear Strength Psi	
1	19,600	
2	19,700	
3	18,900	
4	19,400	
5	19,600	Average 19,500 Psi
6	19,900	
7	19,900	
8	19,500	
9	19,500	
10	19,100	

TABLE A-7

HERCULES AS/3501 GRAPHITE EPOXY
SHORT BEAM SHEAR STRENGTH

STRESSED AT -25°F OVER .310 INCH DIAMETER ROLLERS

Test Specimen	Horizontal Shear Strength Psi	
1	17,700	
2	18,400	
3	18,500	
4	17,300	
5	18,500	Average 18,300 Psi
6	18,100	
7	18,100	
8	18,700	
9	18,600	
10	18,700	

TABLE A-8

AS/3501
Prestressed Unidirectional Short Beam Shear

Stress Condition	No. of Specimens	Avg. Shear Strength	Coefficient of Variation	"A" Design	"B" Design
40^{o}F Over .500" Dia. Rollers	10	18,500 psi	5.0%	14,830	16,330
0^{o}F Over .500" Dia. Rollers	10	19,100 psi	2.3%	17,300	18,040

TABLE A-9

AS/3501
Prestressed Unidirectional Tensile Strength
& Modulus - Pitch Fiber Laminate

Stress Condition	No. of Specimens	Avg. Tensile Strength	Avg. Modulus	Coefficient of Variation	"A" Design	"B" Design
40^{o}F Over 1.50" Dia. Rollers	9	80,900 psi	31×10^{6}	7.5%	55,850	66,100

TABLE A-10

HERCULES 3501 EPOXY PITCH BLEND GRAPHITE
TENSILE STRENGTH & MODULUS

STRESSED AT 50°F OVER 1.50 INCH DIAMETER ROLLERS

Test Specimen	Unidirectional Tensile Strength Psi	Modulus Psi x 10^6
1	86,700	30
2	86,300	30
3	68,600	27
4	68,400	27
5	70,800	28
6	79,400	27
7	80,700	30
8	86,400	30
9	77,100	30
10	75,200	29
Average	78,000	29

Standard Deviation 9.2%
"A" Design Allowable 49,600
"B" Design Allowable 61,300

TABLE A-11

UNSTRESSED HERCULES AS/3501
ANGLE PLY COMPRESSION

Test Specimen	Compressive Strength Psi	Modulus Psi x 10^6
1	65,500	11.5
2	67,500	8.4
3	67,800	8.5
4	68,400	10.7
5	68,600	10.6
6	69,000	8.6
7	70,100	8.4
8	73,900	9.9
9	74,800	8.1
10	79,900	9.5
Mean	70,550	9.4

Standard Deviation	6.2%
"A" Design Allowable	53,420
"B" Design Allowable	60,460

TABLE A-12

UNSTRESSED HERCULES AS/3501
UNIDIRECTIONAL COMPRESSION

Test Specimen	Compressive Strength Psi	Modulus Psi x 10^6
1	103,000	14.8
2	103,600	17.5
3	104,000	17.0
4	106,500	14.8
5	108,900	18.1
6	110,100	16.8
7	110,900	16.0
8	112,000	16.5
9	113,400	16.5
10	121,200	15.5
Mean	109,360	16.4

Standard Deviation	5.1%
"A" Design Allowable	87,500
"B" Design Allowable	96,480

TABLE A-13

HERCULES AS/3501 PREPREG
STRESSED AT 0°F & .500" DIA. ROLLERS

Test Specimen	Unidirectional Tensile Strength Ksi
1	168.5
2	180.3
3	191.5
4	195.7
5	196.6
6	202.4
7	212.9
8	214.8
9	231.3
10	239.3
Mean	203.3 Ksi

Standard Deviation	10.7%
"A" Design Allowable	117.3 Ksi
"B" Design Allowable	152.7 Ksi

TABLE A-14

HERCULES AS/3501 STRESSED AT
-25°F OVER .310" DIA. ROLLERS

Test Specimen	Unidirectional Tensile Strength Ksi
1	208
2	209
3	227
4	236
5	248
Mean	228.5

Standard Deviation	7.4%
"A" Design Allowable	143.0 Ksi
"B" Design Allowable	178.0 Ksi

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