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FINAL REPORT

on

AN EVALUATION OF UPGRADED BORON FIBERS IN EPOXY-MATRIX COMPOSITES

by

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prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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NASA Langley Research Center
Hampton, Virginia

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FOREWORD

The work described herein was conducted at Battelle's Columbus Laboratories. Upgrading of the boron fibers was performed at Hough Laboratory in Springfield, Ohio under the direction of Mr. Ralph Hough.

NASA Contract NAS 1-11589 was monitored by Mr. R. A. Pride, Head Composites Section, Materials Division, Langley Research Center.

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SUMMARY

This program was undertaken to perform an initial evaluation of upgraded boron fibers in an epoxy matrix. Data generated on the program show that fiber strength does increase as a consequence of the upgrading treatment. However, the interlaminar shear strength of upgraded fiber composites is lower than that for an untreated-fiber composite. In the limited tests performed, the increased fiber strength failed to translate into the composite.

INTRODUCTION

The development of boron fibers with high strength and high modulus has had a dramatic impact on the properties attainable in polymeric composites. Further improvements such as a reduction in coefficient of variation in strength properties and cost reductions via larger fiber diameters and the use of carbon substrates are anticipated in the near future.

Recently, Hough Laboratory developed an upgrading process which significantly increases the tensile strength of commercial boron fiber. This process, which is based on a coating only several microns thick, reproducibly increases fiber strength from the 350-500 ksi (2413-3447 MN/m^2) range to the 500-650 ksi (3447-4482 MN/m^2) range, while the modulus, density, and other physical properties are essentially unchanged.

This study was undertaken to evaluate the potential of these upgraded fibers in an epoxy matrix. In an iterative development, the effect of a number of processing variables on short-beam shear strength was evaluated. Tensile properties were then determined for the most promising system.

EXPERIMENTAL PROCEDURES AND RESULTS

Materials

The following boron fibers were used in this program:

AVCO	Lot 1623	Nominal 0.004 in. (0.0100 cm)
UAC	Lot 20147	Nominal 0.004 in. (0.010 cm).

The resin selected was BP-907. This was obtained in the form of 0.02 lb/ft^2 (0.098 kg/m^2) film containing 104 glass scrimcloth from Bloomingdale Section of American Cyanamid Company. The tensile strength is 40 and 15 lb/in. (7144 and 2679 g/cm) in the warp and fill directions, respectively. This contributes to the handling characteristics of the prepreg but has virtually no effect on composite properties.

Fiber Upgrading

High-strength fibers, such as boron, have already demonstrated unique potential as reinforcements for various matrices. It has recently been found* that upgraded fibers can be synthesized by coating existing fibers, such as boron, silicon carbide, carbon alloy, etc., to achieve fibers having superior strength compared to uncoated fibers. Subtle process modifications in the coatings, known as Process "A", "B", etc., are used to enhance interlaminar shear properties in conjunction with the organic matrices used in the program.

Tape Making

Monolayer tape was prepared at Battelle by a drum-winding technique. A 16 in. (40.64-cm) diameter drum was used for this program. After thorough cleaning, the drum was wrapped with BP-907 film adhesive, the length of which was cut to the circumference of the drum. The boron fibers were then wound over the prepreg at approximately 0.5 lb (0.23 kg) tension. The drum-winding operation is shown schematically in Figure 1. An interfiber spacing of 0.0007 in. (0.0018 cm) or 212 fibers/inch (84 fibers/cm) was used. This spacing was desinged to yield a laminate with 50 to 55 v/o fiber. After winding, the band was cut normal to the filaments and removed from the drum as a single sheet of boron-epoxy tape. Because of the limited amounts of each type of fiber available, most tapes were 1 in. (2.54 cm) or less in width.

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* R. L. Hough, "High Strength, Continuous Large Diameter Composite Fibers", Paper No. 47 presented at 2nd ASTM Conference on Composite Materials: Testing and Design, April 20-22, 1971, Anaheim, California

Fabrication of Composites

All composites used in this study were prepared by press-molding using flurocarbon-coated stainless steel tooling. Short-beam shear specimens were molded as 0.190 in. (0.483-cm) wide by a nominal thickness of 0.075 in. (0.191 cm) by 6 in. (15.2-cm) long strips which were then cut to a length of 0.75 in. (1.9 cm) for testing. Tensile specimens were molded as 0.25 or 0.5 in. (0.635 or 1.27 cm) wide by 5-in. (12.7-cm) long strips. Thicknesses varied from 4 to 6 plies, depending upon fiber availability. A schematic drawing of one such mold is shown in Figure 2. These molds were placed into electrically heated compression molding presses for curing. All specimens were heated to 250 F (121 C) under a pressure of 30 psi (0.21 MN/m^2), held for 60 min., and then heated to 350 F (177 C) and held for 2 hr. All tensile specimens were then tabbed with 4 plies of 1002 glass/epoxy.*

The fiber content of several randomly selected composite specimens was accurately established by extracting the matrix in hot-sulfuric acid and physically counting the remaining boron fibers, all of which were intact. In each case, the boron content was 55 volume percent.

Testing and Evaluation

Fiber tests were conducted using 0.035-in. (0.089-cm) diameter tungsten rod grips to which the filament was affixed with nominal 0.5-in. (1.27-cm) lap joints. The standard gage length was 1 in. (2.5 cm) and the cross head speed was 0.05 in./min (0.127 cm/min). Filament stress was monitored with a 10-lb (4.54-kg) Statham load cell.

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* 3-M Company.

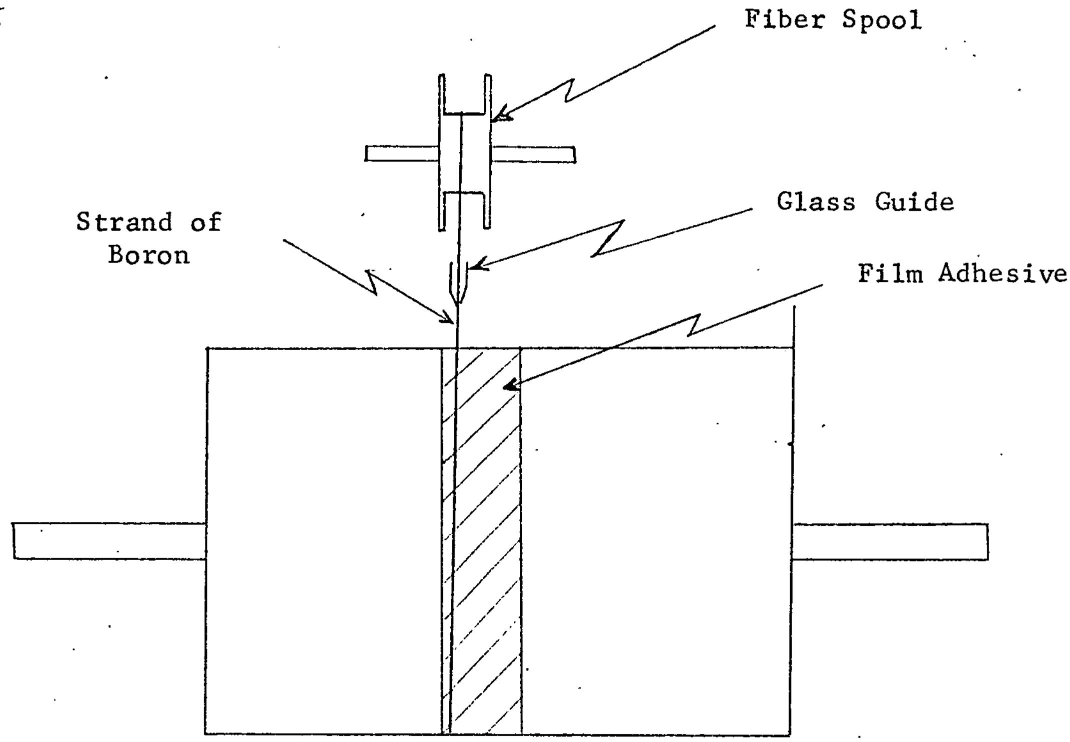

FIGURE 1. SCHEMATIC OF DRUM WINDING PROCESS

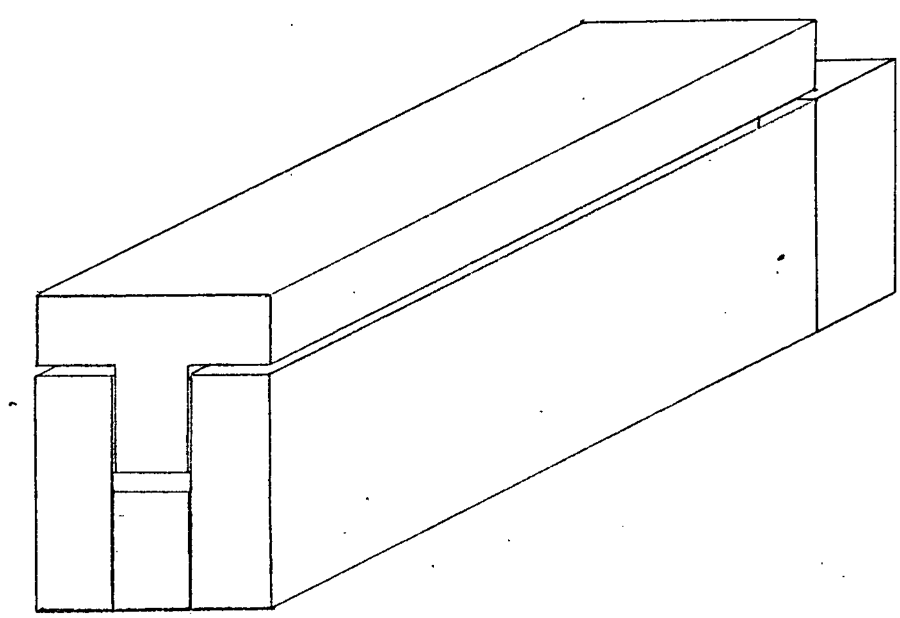

FIGURE 2. STAINLESS-STEEL MOLD FOR BORON-EPOXY TEST SPECIMEN

Note that front end plate is not shown in this sketch.

Several preliminary individual fiber tests were performed on samples taken from the beginning of the spool of each lot. These tests, which are presented in Table 1 indicated an as-received strength of 547 ksi (3771 MN/m^2) and 410 ksi (2827 MN/m^2) respectively for the AVCO and UAC fiber.

TABLE 1. PRELIMINARY AS-RECEIVED BORON FIBER STRENGTH

	AVCO Lot 1623		UAC Lot 20147
	526 ksi (3626 MN/m^2)		380 ksi (2620 MN/m^2)
	612 ksi (4219 MN/m^2)		385 ksi (2682 MN/m^2)
	590 ksi (4067 MN/m^2)		389 ksi (2682 MN/m^2)
	486 ksi (3350 MN/m^2)		470 ksi (3240 MN/m^2)
	551 ksi (3799 MN/m^2)		380 ksi (2620 MN/m^2)
			459 ksi (3165 MN/m^2)
Average	547 ksi (3771 MN/m^2)	Average	410 ksi (2827 MN/m^2)

Three upgrading processes, "A", "B", and "C", investigated. Table 2 presents the resulting strength of both fiber lots subsequent to upgrading. Process B appeared to yield the highest strength and most consistent results on both AVCO and UAC fiber.

Interlaminar shear strengths (ILSS) were measured using the short beam technique, shown schematically in Figure 3. The radius of the supports and loading nose was 0.125 in. (0.318 cm) and the span was fixed at 0.4 in. (1.116 cm); the specimen was nominally 0.2 in. (0.51 cm) wide by 0.075 in. (0.19 cm) thick.

The resulting shear strength data are presented in Table 3. Both the "A" and "C" process resulted in a substantial drop in ILSS whereas the decrease for the "B" process was slight and reproducible. No tensile failures were observed. The ILSS and previously presented fiber strength results indicated that the "B" process was superior to the other candidates.

TABLE 2. TENSILE STRENGTH OF UPGRADED BORON FIBER

Process	Fiber	Tensile Strength, ksi (MN/m^2)[a]
A	AVCO	592 (4082)
	UAC	507 (3496)
B	AVCO	
	Lot 1	594 (4096)
	Lot 2	658 (4537)
	UAC	
	Lot 1	542 (3737)
	Lot 2	496 (3420)
C	AVCO	462 (3185)

(a) Each value represents an average of three tests.

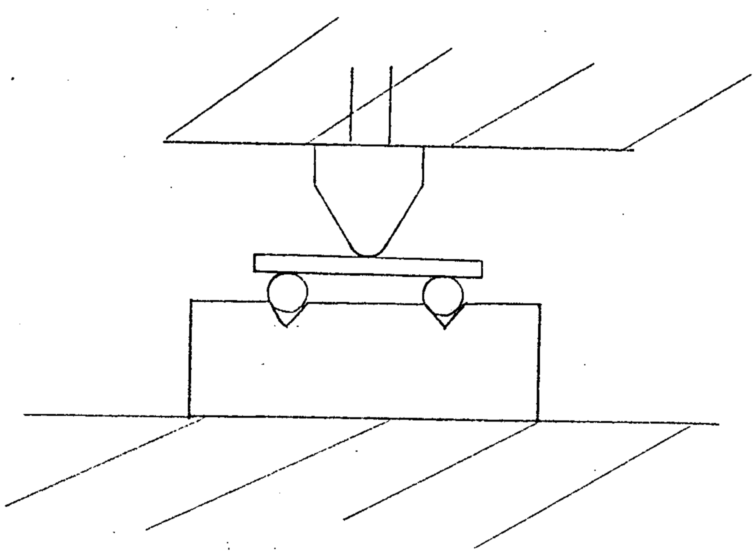

FIGURE 3. SCHEMATIC DRAWING OF SHORT BEAM SHEAR TEST

Fixture is positioned between compression platens in a standard test machine. Support radius is 0.125 in. (0.318 cm) and the span is 0.4 in. (1.116 cm).

TABLE 3. INTERLAMINAR SHEAR STRENGTH (ILSS) OF UPGRADED BORON-EPOXY COMPOSITE

	Fiber Lot			
	AVCO(a)		UAC(a)	
Process	Specimen L/d	ILSS, ksi (MN/m^2)	Specimen L/d	ILSS, ksi (MN/m^2)
As-received	5.33	13.4	4.71	10.4
		12.8		9.8 Average
		12.5 Average		11.3 10.5 (72.4)
		10.3 11.9 (82.1)		
		10.4	6.56	11.2
				11.3
				11.0 Average
				11.3 11.2 (77.2)
"A"	5.19	7.3	5.33	7.2
		7.6 Average		7.3
		7.7 7.8 (53.8)		7.6 Average
		8.8		7.8 7.5 (51.7)
				7.7
"B"	6.06	11.6	4.82	11.5
		11.0		9.4
		11.6		11.2
		11.2		10.3
		10.8 Average		10.1 Average
		11.2 (77.2)		10.2 10.4 (71.7)
				10.0
				10.6
				10.4
			5.56	11.1
				11.6
				10.9 Average
				11.7 11.1 (76.5)
				10.1
			6.45	9.5
				9.6
				11.1 Average
				10.9 10.3 (71.0)
"C"	5.33	9.5	Not evaluated	
		9.4		
		9.2 Average		
		9.7 9.5 (65.5)		
		9.8		

(a) Span distance, L, was 0.4-in. (1.02 cm).

A rather extensive series of individual fiber tensile tests were performed on UAC boron to determine the degree of variation in strength as a function of position in the spool. Tests on as-received and Process B upgraded material were performed. The results are summarized in Table 4. These data illustrate the strength upgrading effect of Process B. Strength was increased by 10 to 20 percent and the variation in strength after upgrading was noticably reduced.

A series of experiments were conducted to evaluate the effect of coating thickness on ILSS. AVCO fibers treated with the "B" process were used. The ILSS values were as follows:

Coating Thickness, mils (cm^{-3})	Specimen L/d	ILSS, psi (MN/m^2)
0.01 (0.025)	5.33	10,920 (75.28)
0.05 (0.127)	6.06	11,220 (77.35)
0.08 (0.203)	5.13	9,890 (68,18)

These data indicate that the coating thickness can influence ILSS in a composite and suggest that the coating thickness should be about 0.05 mil (0.00013 cm).

A series of 0.081 in. (0.21 cm) thick specimens were fabricated using AVCO fiber upgraded via the "B" process and then aluminized. The ILSS for these specimens was only 7850 psi (54.12 MN/m^2). No further work was performed with metallized fiber.

The effect of a 2-hour water boil on UAC-"B" process fiber composites was evaluated. Several ILSS specimens were cut from a single 0.067-in. (0.17-cm) thick panel. The shear strength of the control sample was 8,870 psi (61.16 MN/m^2). After the water boil, the shear strength dropped to 7100 psi (48.95 MN/m^2). This is not deemed excessive, as losses of approximately 25 percent after a 2-hr boil have been reported elsewhere.* The low ILSS value of the control sample is not readily explainable. The specimen L/d was 5.97 which was similar to those presented in Table 3. An ILSS value of 10 to 11 ksi was anticipated.

* e.g., Wawner, F. E., "Boron Filaments", in Modern Composite Materials, Addison-Wesley Publishing Company, pp 244-269 (1967).

TABLE 4. TENSILE STRENGTH OF UAC BORON FIBER

Sample No.	Diameter, mils (cm)	Strength, ksi (MN/m^2) As-Received	Upgraded
Start of Spool			
0	4.07 (0.01034)	504, 450, 345, (3474), (3102), (2378), 407, 288 (2806), (1985)	--
5	4.10 (0.01041)	459, 470, 380 (3163), (3240), (2620)	552, 480, 457 (3806), (3310), (3151)
14	4.08 (0.01036)	455, 490, 555 (3137), (3378), (3826)	584, 593, 493 (4027), (4089), (3399)
37	4.04 (0.01026)	476, 500, 510 (3281), (3447), (3516)	605, 480, 445 (4171), (3310), (3068)
65	4.08 (0.01036)	337, 383, 451 (2323), (2640), (3109)	555, 275, 500 (3827), (1896), (3448)
67	4.05 (0.01028)	380, 385, 389 (2620), (2654), (2682)	576, 525, 525 (3972), (3620), (3620)
174	4.05 (0.01028)	614, 590, 620 (4233), (3067), (4274)	726, 600, 480 (5006), (4137), (3310)
194	4.05 (0.01028)	513, 470, 505 (3537), (3240), (3481)	650, 641, 626 (4482), (4420), (4316)
195[a]	4.07 (0.01034)	423, 490, 508 (2916), (3378), (3507)	530, 585, 645 (3654), (4036), (4447)
Average		460 (3171)	543 (3744)
Coefficient of Variation		0.17	0.16
200	4.02 (0.0102)	497, 420, 430 (3426), (2895), (2964)	611, 530, 435 (4213), (3654), (2999)
201	4.06 (0.0103)	525, 656, 540, 465 (2619), (4522), (3723), (3206)	693, 671, 596, 515 (4778), (4627), (4109), (3551)
202[b]	4.02 (0.0102)	630, 670, 645, 474 (4343), (4619), (4447), (3268)	675, 667, 660, 660 (4654), (4599), (4551), (4551)
Average		541 (3730)	601 (4144)
Coefficient of Variation		0.17	0.13

(a) Samples 0 through 195 taken at 200 to 400 ft (60.9 to 122 m) intervals.
(b) Samples 200 through 202 taken at 1000 ft (305 m) intervals.

A series of four-ply subsize [nominally 0.250 x 0.020 in. (0.635 x 0.051 cm)] tensile specimens were prepared from "B" process upgraded UAC fibers from Runs 195, 201, and 202. The minimum strength of these fibers was 515,000 psi (3551 MN/m^2) and the average strength was 613,000 psi (4227 MN/m^2). The results of these tests are shown in Table 5. Although the moduli agree with published values, the tensile strengths are quite low. By way of comparison, the tensile strength of a control sample based on as-received UAC fiber was 173,000 psi (1193 MN/m^2).

A small quantity of "B" process upgraded AVCO fiber with an average tensile strength of 594,000 psi was also incorporated into 6-ply by 0.5-in. (1.27-cm) tensile specimen. The strength of this material was 180,000 psi (1241 MN/m^2).

DISCUSSION OF RESULTS

The data generated during this study and earlier* by Hough Laboratory show that there is a definite upgrading in the tensile strength of processed fiber. The exact mechanism of strengthening is not fully understood, but is believed to be associated with the healing of surface defects.

Because this upgrading process alters the surface of the fiber, this study was undertaken to determine whether the fiber-matrix bond strength might be adversely affected. Indeed, a decrease in interlaminar shear strength was noted for all three variations of the process. The drop is about 20 to 33 percent for the "A" and "C" processes but only about 5 percent for the "B" process. This is believed to be a tolerable amount. Furthermore, upgraded fiber-epoxy was shown to be as resistant, if not more so, to moisture degradation than untreated boron-epoxy.

The high tensile strengths of the upgraded fibers failed to translate into the composites. As noted earlier, strengths of even the composites containing the best upgraded fiber, AVCO process "B", exhibited a tensile strength at the lower end of the usual strength band of boron-epoxy. Further, the test on specimens with as-received fiber yielded values in the same range. The limited number of tests performed make it difficult to specify a cause or causes for below goal strength.

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* Hough, R. L., "High Strength, Continuous Large Diameter Composite Fibers", Paper No. 47, 2nd ASTM Conference on Composite Materials: Testing and Design (April, 1971).

TABLE 5. TYPICAL TENSILE PROPERTIES OF BORON-BP907 EPOXY LAMINATES

Fiber Type	Tensile Strength, psi (MN/m^2)			Tensile Strength, psi x 10^6 (GN/m^2)		
	RT (20 C)	165 F (74 C)	240 F (115 C)	RT (20 C)	165 F (74 C)	240 F (115 C)
Typical value*	212,000 (1468)	183,000 (1262)	--	30 (207)	28 (193)	--
As-received UAC fiber	173,000 (1193)	--	--	--	--	--
Upgraded UAC fiber fiber	155,000 (1069)	132,500 (910)	131,000 (903)	30 (207)	28 (193)	26.3 (179)
Upgraded AVCO fiber fiber	180,000 (1241)	--	--	--	--	--

*Structural Design Guide for Advanced Composite Application, Volume 1, Materials Characterization, Second Edition (January, 1971).

Possible experimental variation in loading axis, area measurement, and specimen fabrication could feasibly influence measured strength on the order of 10 to 15 percent. However, it was more difficult to reconcile the values obtained from upgraded UAC boron-bearing composite specimens strictly on the basis of experimental variation as the values were roughly 25 percent below goal at each of the test temperatures investigated. While such was not within the scope of the present investigation, it is apparent that additional tests are needed on well characterized panels prepared from comparable upgraded and nonupgraded boron fiber. The test specimen population should be of at least minimal statistical significance. The additional tests would tend not only to clear up the apparent tensile strength anomoly but the water boil ILSS anomoly as well.

CONCLUSIONS

(1) Fiber upgrading does increase the tensile strength of the fiber and, in the case of "B" process UAC fiber, decreases the coefficient of variation somewhat.

(2) The "A" and "C" processes were found to cause a significant loss in interlaminar shear strength in BP907 epoxy composites. However, for the "B" process, the decrease was very minor.

(3) The increased tensile strength developed in the fibers apparently failed to translate into the composites prepared from the fibers.