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HYBRIDIZED POLYMER MATRIX COMPOSITES

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16. Abstract

Methods of improving the fire resistance of graphite epoxy composite laminates were investigated with the objective of reducing the volume of loose graphite fibers disseminated into the airstream as the result of a high intensity aircraft fuel fire. Improvements were sought by modifying the standard graphite epoxy systems without significantly negating their structural effectiveness. The modifications consisted primarily of an addition of a third constituent material such as glass fibers, glass flakes, carbon black in a glassy resin. These additions were designed to encourage coalescense of the graphite fibers and thereby reduce their aerodynamic float characteristics. A total of 38 fire tests were conducted on "thin" (1.0 mm) and "thick" (6.0 mm) hybrid panels.

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FOREWORD

This report summarizes work performed at Avco Specialty Materials Division, Lowell, Massachusetts under contract NAS3-21385. The project reported herein consisted of evaluating hybrid graphite epoxy laminate concepts for fire resistance.

This program was conducted under the direction of Dr. T.T. Serafini of the NASA Lewis Research Center. The principal investigators were Mr. C. Mullen and Mr. J. Henshaw. Mr. J.G. Alexander conducted the fire tests and Dr. J. A. McElman was responsible for the structural analysis, and also, in conjunction with Mr. C. Mullen, the concept formulation.

1.0 Introduction

The most promising use of the carbon/graphite fibers involves their use as reinforcing fibers in resin matrix composite materials. Composite materials, utilizing epoxy resins, provide low weight, high strength, high stiffness materials which can be tailored to meet structural requirements. The graphite epoxy materials in the composite form do not pose any known problem. However, if the carbon/graphite fiber composite were exposed to severe thermal oxidation environments (fire and/or explosion) then the matrix material would decompose and oxidize and no longer be capable of serving as a binder for the fibers.

Carbon graphite fibers are very small (typically 6 to 10 microns diameter), lightweight (1.7 to 1.8 g/cc) and have a low electrical resistivity (90 to 10,000 ohm/cm). Because of these unique properties, the separated filaments can be floated and transported large distances by normal atmospheric motion.

In recent years it was believed and documented that the release of free carbon graphite fibers into the environment represented a potential hazard.

There are two fundamental approaches to solving a graphite fiber electrical hazard and migration problem:

1) Alteration of the graphite fiber itself to change its density, aerodynamic behavior or electrical conductivity, or
2) Devising methods of preventing the fibers from escaping the matrix material during thermal degradation.

Because of the current and projected wide spread application of currently available graphite fiber resin matrix composites, methods to prevent the release of graphite fibers appears to be the most advantageous approach. The development of hybridized polymer matrix composite techniques to retain the graphite fibers is the thrust of this research program.

The objective of this program was to identify different materials concepts which could be fabricated of hybridized composites which demonstrated improved graphite fiber retention capability in severe oxidative environments without significant reduction to the composite properties. Additional requirements imposed on the hybridized polymer matrix composite concepts were minimum impact on processability, fabrication costs or composite material properties.

2.0 Hybrid Material Concepts

Hybrid graphite epoxy composite materials proposed for investigation in this program are based upon the concept of mechanically entrapping the individual graphite fibers, and thereby preventing their release into the atmosphere. Three methods of entrapment were proposed for investigation: (1), the use of woven graphite fabric, (2) the addition of a second fiber that would interlock the graphite fibers, and (3) the addition of filler into the resin to form a strong high char yield matrix during oxidation. The concept of adding a second fiber also included the addition of a low melting fiber which would melt during oxidation and serve to coalesce the graphite into large clumps of material that would exhibit less aerodynamic float.

Fibers used for evaluation of interlocking the graphite fibers included boron, quartz and glass. Char promoting additives included carbon black, silica and siloxane resins. A general summary of the material concepts are presented below:

- A 8 harness satin weave graphite fabric.
- A unidirectional graphite fabric collimated with a cross weave of glass.
- A unidirectional graphite fabric collimated with a cross weave of glass with each graphite tow "served" with a glass yarn.
- A graphite harness satin weave fabric with each graphite yarn served with a glass yarn.
- A conventional graphite prepreg, with each graphite yarn served with a glass yarn.
- Siloxane glass resin and glass flake additives.
- Carbon black filler.

- 104 glass scrim.
- Glass chopped fiber veil.
- Unidirectional boron fabric collimated with a polyester cross weave.
- Quartz fabric.

The term "serving" describes a process by which a bundle of fibers (in this case the graphite tow) is overwrapped with a helical winding of another fiber to hold the graphite tow together. (See figure 1.) The serving is accomplished in a braiding machine which can vary the spacing of the serving and also the number of clockwise and counter clockwise layers of served fibers.

At the inception of this program, it was thought that the evaluation of hybridized composite materials for fire resistance could be conducted on unidirectional material graphite epoxy, because the analysis of these simply constructed materials would prove to be less cumbersome as compared to cross-plied laminates. However, during the early stages of fire testing in the Avco Model 25 fire test facility, the standard unidirectional laminates exhibited excellent fiber retention compared to cross-plied laminates. Unidirectional materials tend to embed each layer into its neighbor, such that there is a lack of stratification between the individual layers. In contrast, the cross-plied materials are highly stratified and delaminated very easily during the fire test. As a result, a quasi-isotropic construction (0°/90°/±45°) was selected as the reference graphite epoxy laminate. All of the hybrid concepts were also to have the same configuration.

The thickness considerations were dramatized in some of the Avco preliminary fire tests where it was observed that "thin" panels (thickness > 1.3mm) exhibited a dramatic collapse and burn-through in a proportionally much shorter time than that required for thicker panels. It appeared from these tests

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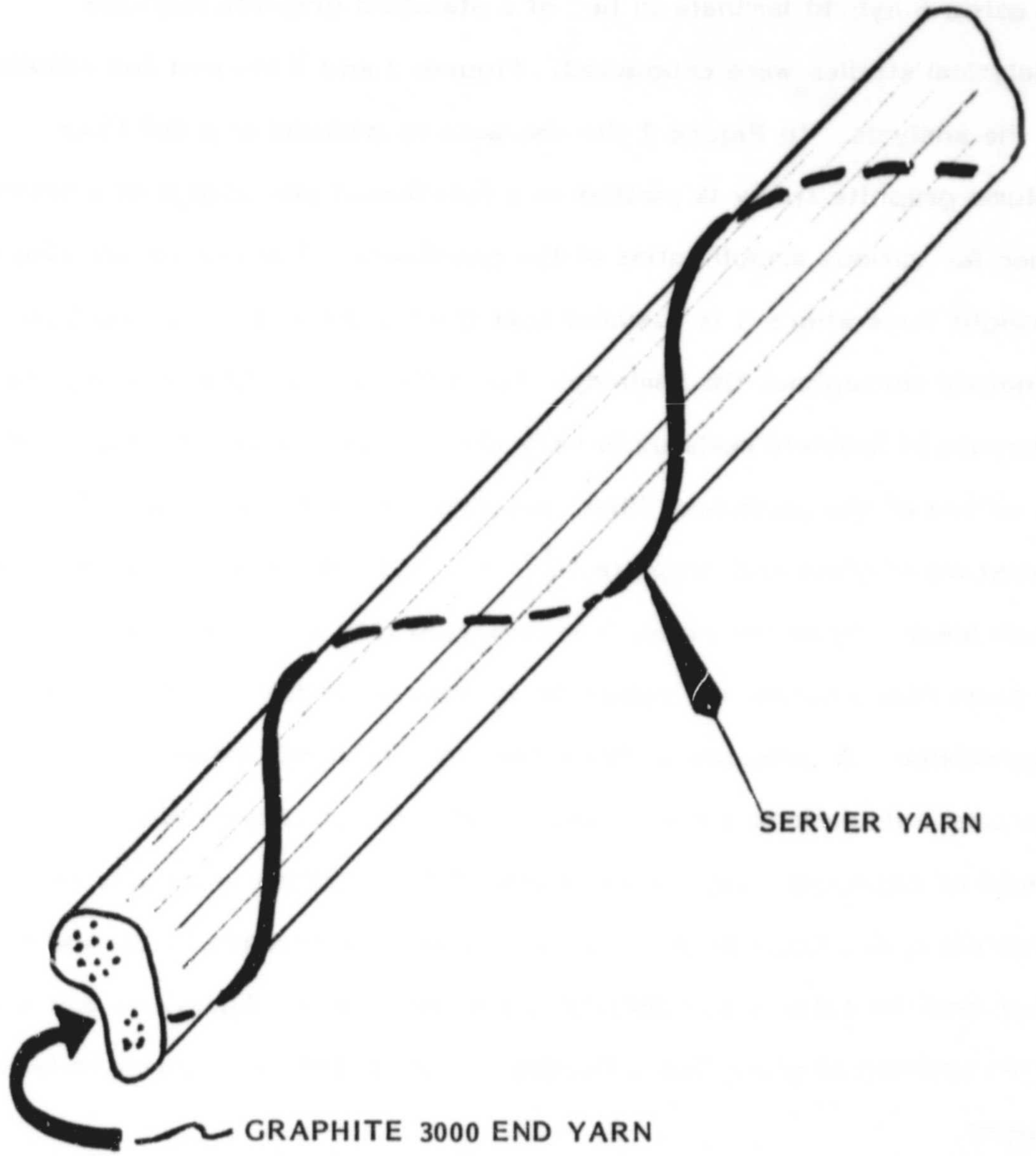

Figure 1 Sketch of Graphite Fiber With Server Yarn

that "thin" panels would require a proportionally greater addition of "protection" materials than thicker material, and hence would suffer a greater reduction in structural efficiency.

To provide a better understanding of the effects and penalties of using a hybrid laminate in lieu of a standard graphite laminate, analytical studies were conducted. Figures 2 and 3 present the results of the analysis. In Figure 2 the decrease in modulus of a 50% fiber volume graphite epoxy is plotted as a function of percentage of a secondary fiber, for various moduli ratios of the two fibers. The curves are simple straight lines where it is assumed that the two materials are mixed homogenously throughout the laminate. For a low ratio of fiber moduli, the decrease in laminate modulus is very close to being directly proportional to volume of the secondary fiber, however, for a modulus ratio that reflects a mixture of glass and graphite ($\frac{Es}{Eg} = .33$) the reduction in modulus is much lower. From the plots, it is indicated that we can add up to 10% of glass fibers before the reduction in modulus of the hybrid laminate is appreciable. A strength analysis was not conducted, however if rule of mixtures calculations are acceptable, then an equivalent reduction in strength would be expected. Figure 3 is a plot of the increase in density of a graphite epoxy laminate as a function of the percentage of a secondary fiber and the ratio of the density of the two fibers. Again, we can see that a 10% addition of glass fibers results in a very minimal increase in laminate weight.

Various other analyses were conducted with the purpose of assessing the mechanical property and density penalties of hybrid laminates. The analyses were performed on "thick" and "thin" laminates where for a given

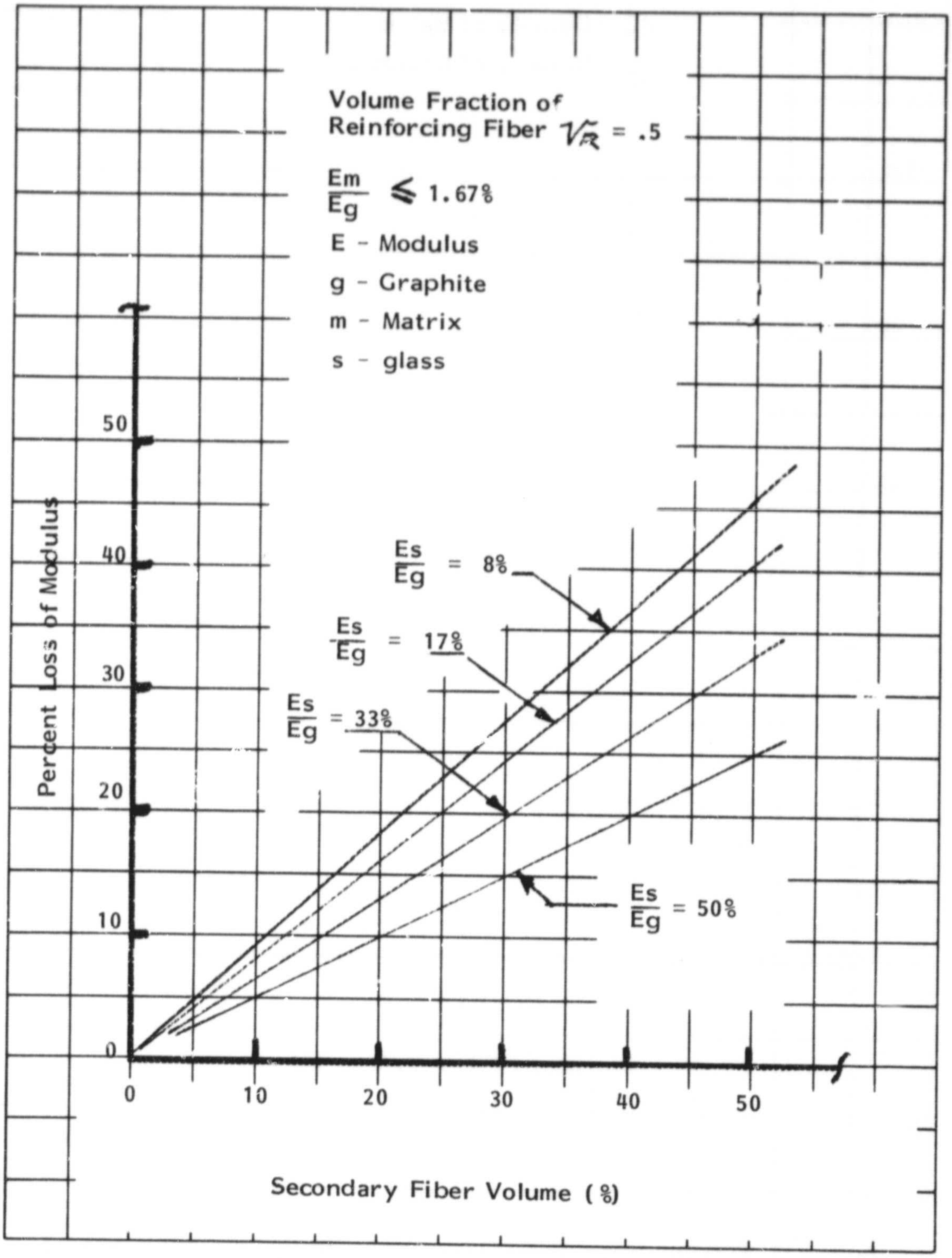

Figure 2 Percent Loss of Modulus vs. Secondary Fiber Volume

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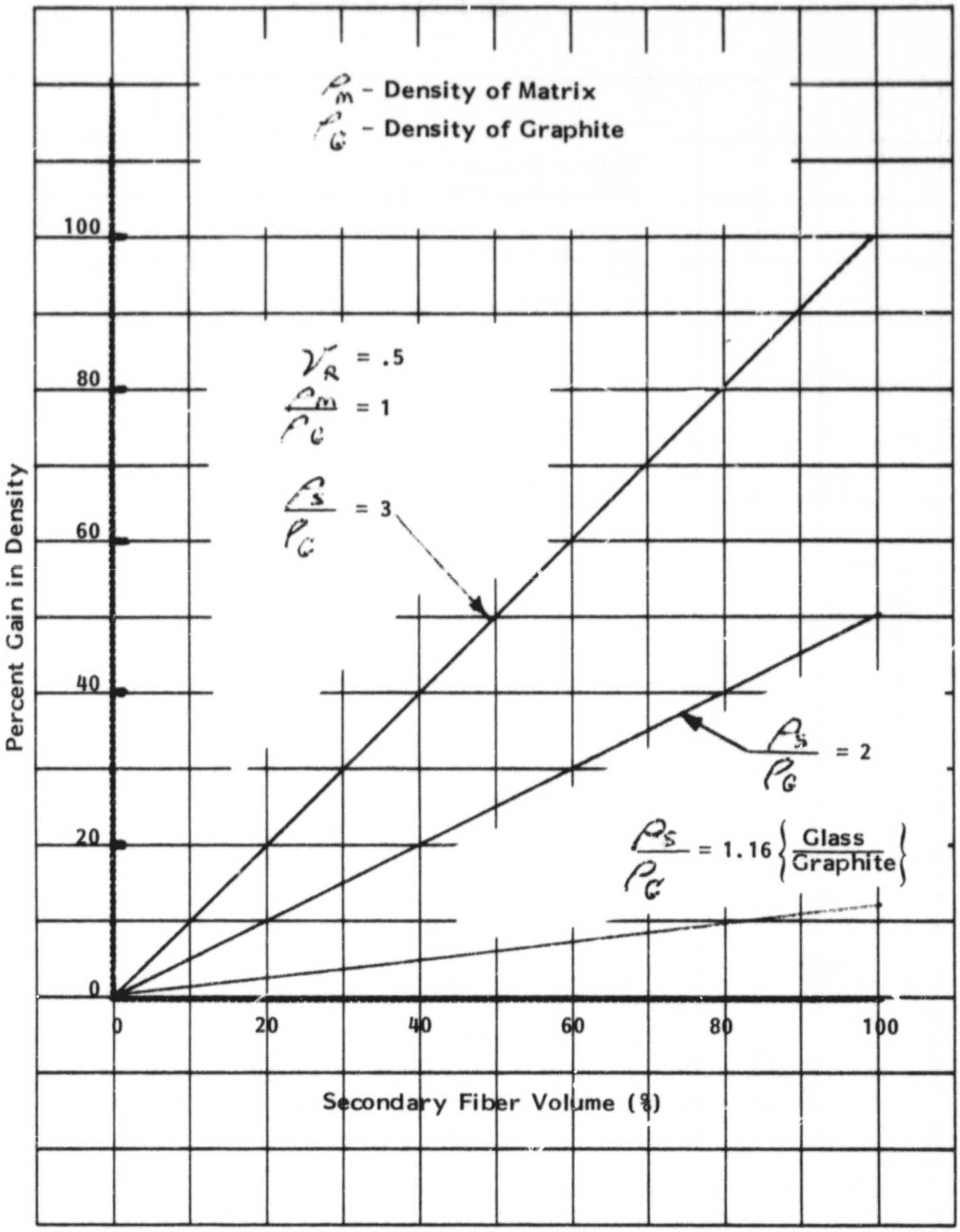

Figure 3 Percent Increase in Density vs. Secondary Fiber Volume

thermal exposure, a outer layer of the "thick" and "thin" panels was modified to provide fire protection. In figure 4 a 0.13 mm outer layer of a "thin" (1.0mm) and a "thick" (6mm) graphite epoxy panel is presumed to be modified (for fire protection) and as a result the stiffness of these layers is changed. The effect of modifying the outer layer upon the extensional stiffness (Et) of the tri-layer hybrid composite laminates is plotted in Figure 4 as a function of the ratio of the moduli of the outer and inner layers layers and as a ratio of the thickness of these layers. Modifying the outer layer of the two laminates is seen to effect the thin laminate to a much larger extent than the thick laminate. A similar analysis was conducted for strength and is shown in Figure 5. Here, non-dimensional stress is plotted for the graphite core and the coating as a function of various ratios of coating modulus to graphite modulus. Inspection of Figure 5 shows that for a given ratio of moduli the stress in the protective coating is nearly the same for laminates having a thick or thin coating. Again, it also can be seen that the core stress in the "thin" panel is affected significantly more so than the "thick" panel.

Based upon the analyses discussed above and the hybrid concepts presented earlier, the following test panels were planned. As the resin composition was a factor in the thermal degradation response of the laminate, it was decided to use an epoxy resin that was readily available and representative of typical aerospace 450°K curing systems being used in the industry. The system designated as Avco 5535 was used for all of the baseline and hybrid constructions.

2.1 Thin Panels

All of the "thin" panels were designed to be as close as possible to the reference panel thickness, $\sim$ 1.2 mm.

Reference Panel - A conventional eight ply quasi-isotropic panel $(0°, \pm 45°, 90°)_s$ was used as reference test material. The graphite

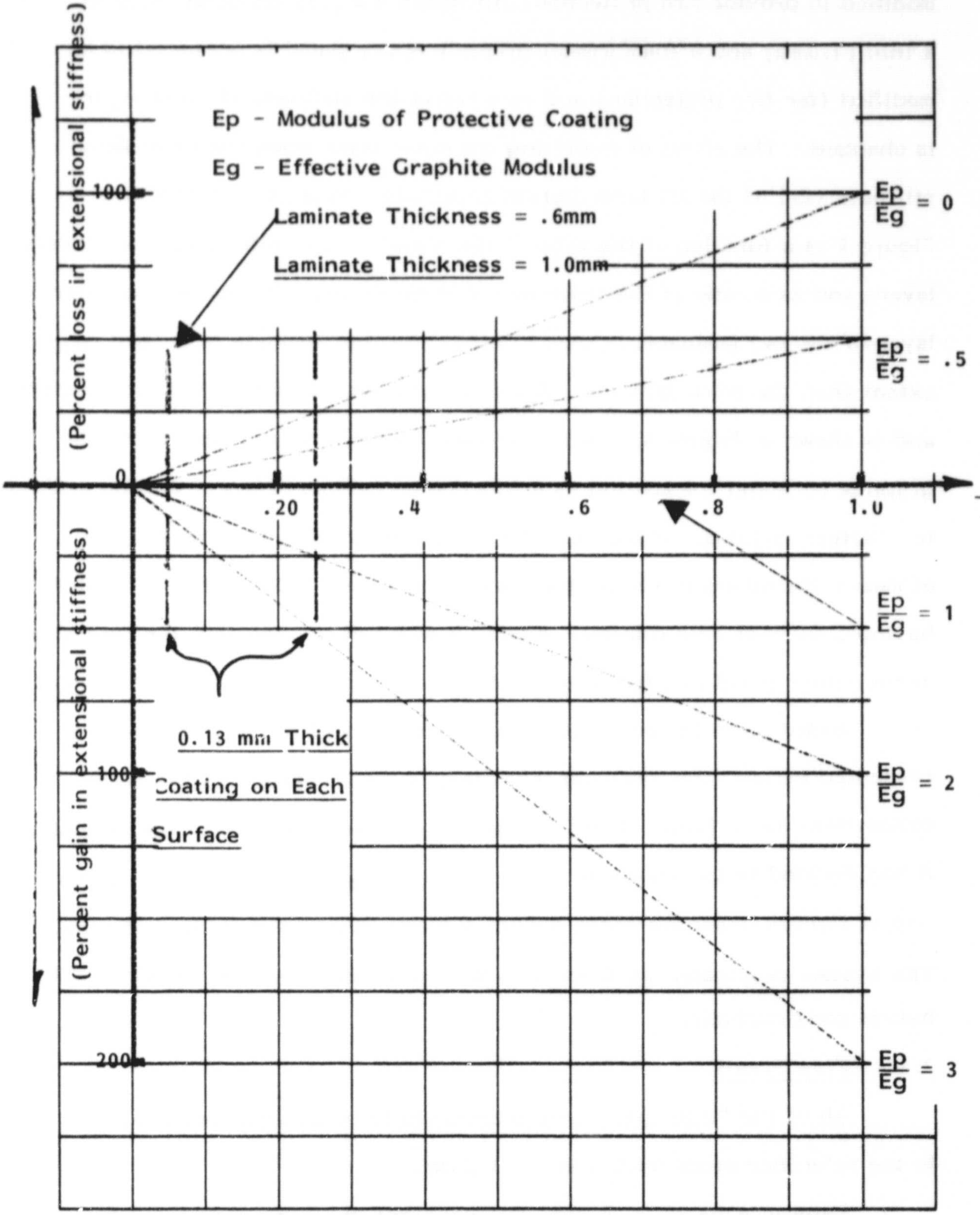

Figure 4 Change in Extensional Stiffness vs. Coating Thickness

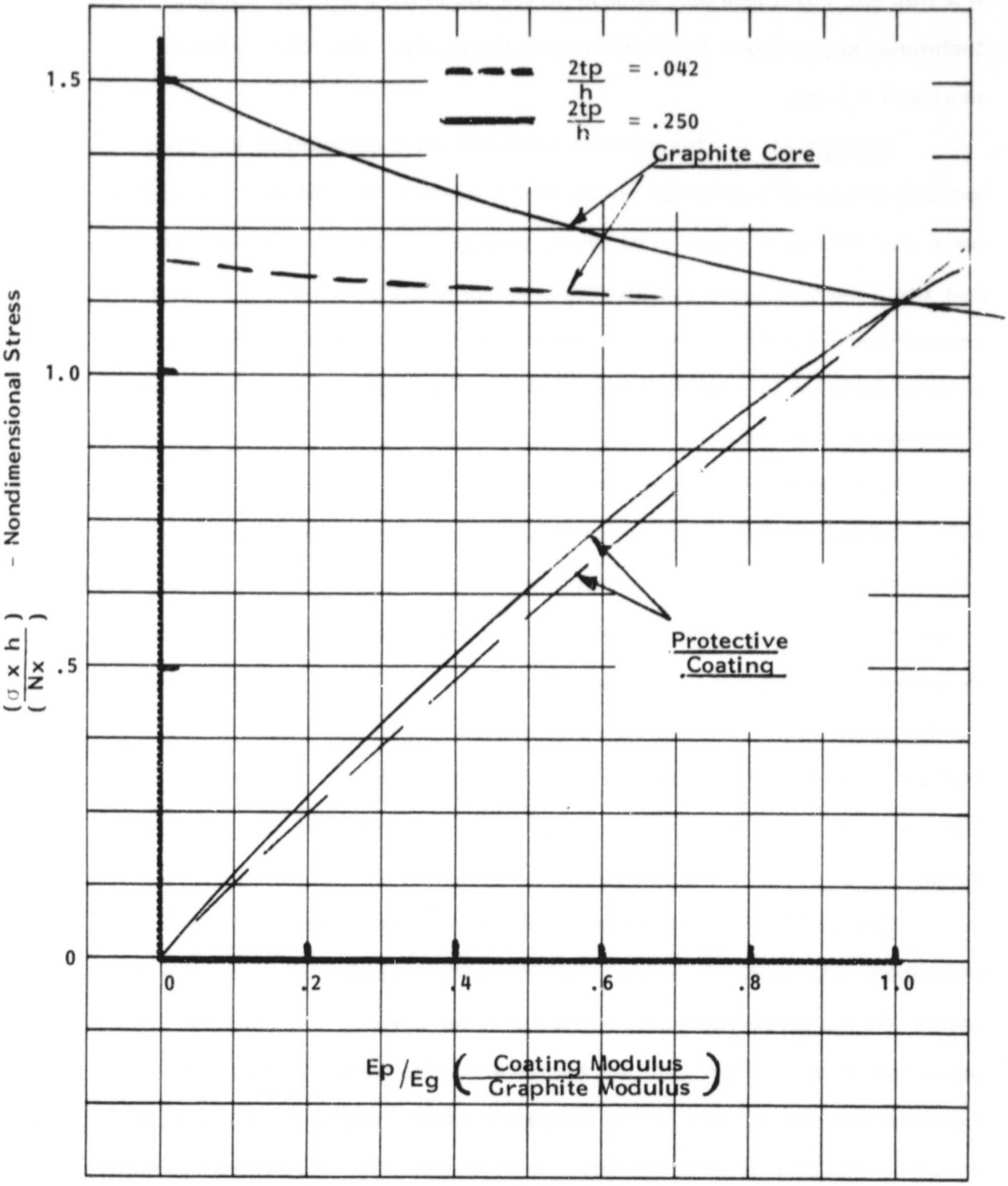

Figure 5 Nondimensional Stress vs. Ratio of Coating to Graphite Modulus

tow was the T300/3000 end material prepregged by the drum winding technique to provide a 60% fiber volume composite. The design thickness was 1.2 mm.

Concept #1 - This concept involved the evaluating of the interlocking effects of a graphite woven fabric using T300/3000 end tow in the warp and fill weave axes. The fabric selected for use in this program was the 8 harness satin configuration utilizing 9.5 tows per cm in both the warp and the fill. It was expected that a lay-up of 4 layers of fabric in an orthogonal (0°/±45°/0°) orientation would produce a test specimen thickness of 1.32 mm.

Concept #2 - This concept utilized a uniweave fabric construction with 8.26 tows per cm in the warp axis, collimated by 3.93 glass yarns per cm in the fill direction. The molded thickness of the uniweave was expected to be 0.20 mm and an eight layer quasi-isotropic (0°/±45°/90°) layer was designed with a total thickness of 1.62 mm. It was expected that these panels would exhibit a small degradation in terms of mechanical properties and weight in comparison to the woven fabric concept - #1. The graphite fiber was the T300/3000 end tow. The glass fiber was the 150 1/0 E glass style.

Concept #3 - This concept is similar to the uniweave fabric described in Concept #2, except that the warp graphite tows were served with a 900 ½ E glass yarns; this time the lower density yarn was 900 ½ E glass spaced at 11.8 per cm. As compared to the woven fabric, it was expected that the density of the composite would increase by 4% and the specific modulus and strength would decrease by 14%. As before (Concept #2) a quasi-isotropic (0°/±45°/90°) eight ply 1.62 mm thick test panel was configured.

Concept #4 - This concept is a modification of the baseline test panels where glassy materials are added to melt during heating and coalesce the fibers. The additives took the form of a glass resin that was impregnated into the 3000 end T300 graphite tow prior to the addition of the standard epoxy resin, and an addition of a glass flake into the epoxy resin itself. It was thought that the build up of the glass resin on the fiber would be minimal and the addition of the glass flakes would encompass 10% of the resin volume. As a result, it was expected that an eight ply quasi-isotropic (0°/±45°/90°) test panel would mold to a thickness of 1.16 mm and would realize only a 4% increase in density.

Concept #5 - This concept is again a modification of the baseline reference test panels where in this case an oxidation resistant carbon black filler was added to the standard resin. Here it was assumed that the total volume of graphite will remain the same and the filler will appear in the composite at the expense of resin material. A 15% by volume replacement of resin was designated and the composite density and thickness was not expected to change measurably. The graphite was the T300/3000 end tow.

Concept #6 - In this case, the concept was to increase the volume of glass serving that would be available for coalescing during the heating cycle. The glass was to be added as a double serving to the T300/3000 tow and used in the conventional lay-up as used in the reference panel. However, it proved to be impossible to maintain the required thickness of the individual layers. The problem appeared to be the large quantity of serving that restrained the bundles of graphite fibers from spreading during the pressing operation. Hence, for the "thin" panels at least, it was impossible to obtain the nominal 0.13 mm per layer that is required for a quasi-isotropic laminate. (See later discussion.)

Concept #7 - This concept provided further modification to the baseline reference material where in this case a style 104 glass scrim was interleaved between each graphite layer. The scrim was expected to mold to a 0.25 mm thickness composite, increasing the thickness of an eight ply composite to 1.34 mm. The major penalty here would be a reduction in modulus. The composite modulus will decrease by approximately 14% with the specific modulus reflecting the same decrease. The density and strength of the composite would be relatively unaffected. The addition of a silica and/or carbon filler was to be added to stabilize the char residue formed after themal degradation. As before, the graphite tow was the T300/3000 end product.

Concept #8 - This concept is similar to Concept #7 with a thin glass veil (0.25 mm thick) replacing the glass scrim in the otherwise identical to the baseline reference design. The glass veil was evaluated because it provided a more random fiber distribution. In addition, glass flake was added to replace 5% of the resin. This concept was expected to exhibit the largest penalties of the concepts considered. The glass veil is assumed to contribute nothing to modulus or strength and the glass flake will increase the overall density of the composite. The modulus and strength of the composite would be reduced by approximately 15.0% while the density increases by 2%. A summary of the recommended "thin" panels is shown in Table I.

2.2 Thick Panels

Reference Panel - A conventional 48 ply quasi-isotropic panel (0°/±45°/90°/90°/±45°/90°) was designated as the reference test material. The graphite tow was the T300/3000 end product prepregged by the drum winding technique to provide a 60% by volume laminate. The design thickness was 6.09 mm.

TABLE i

RECOMMENDED THIN PANEL CONCEPTS

Design Concept	Panel Construction: No. of Plies	Panel Construction: Lay-Up Sequence	Panel Thickness (mm): Design	Panel Thickness (mm): As Fabricated
Reference Quasi-isotropic GR/epoxy Thornel 300/Avco 5535	8	$[0/90/\pm45]_s$	1.11	1.06
1. Graphite Fabric - 8 H/S Weave 24 x 24 warp/fill	4	$[0/+45/-45/90]$ 0°-warp direction	1.32	1.37
2. Unidirectional weave with glass tie fill - 22 warp/10 fill (glass)	8	$[0/90/\pm45]_s$	1.62	1.47
3. Server yarn of glass on unidirectional weave with glass tie fill	8	$[0/90/\pm45]_s$	1.62	1.57
4. Reference plus glass resin (Siloxane) fiber coating with milled glass/glass flake resin additive	8	$[0/90/\pm45]_s$	1.16	1.14
5. Reference plus oxidation resistant carbon black resin filler additive (15% addition)	8	$[0/90/\pm45]_s$	1.11	1.06
6. Reference plus double server (braided) glass yarns on the graphite tow	8	$[0/90/\pm45]_s$	1.21	
7. Reference with an interleaf of glass scrim cloth between graphite fiber layers with Silica and/or carbon filler in resin (5% - 10%)	8	$[0/90/\pm45]_s$	1.34	1.32
8. Reference with an interleaf of E-glass surface veil and milled glass/glass flake resin additives (5% - 10%)	8	$[0/90/\pm45]_s$	1.34	1.34

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Concept #1 - This concept is similar to concept #1 in the "thin" category (8 H.S. fabric) with the exception that a glass scrim cloth was added between each layer of the fabric for improved fiber retention capability. The scrim addition was expected to decrease the modulus by approximately 7% but should not to have an appreciable effect on the strength and density. A 20 ply laminate in a $(0°/45°/90°/45°/45°/0°/90°/45°/45°/0°)_s$ would provide a near quasi-isotropic response with a thickness of 7.11 mm.

Concept #2 - This concept is identical to concept #1 above, except a glass veil replaced the glass scrim cloth and included the addition of carbon/silica resin fillers to increase and stabilize the char residue. The composite properties were expected to be reduced by approximately 15% with a small (3%) increase in density.

Concept #3 - This concept consisted of winding a glass server yarn on the warp and fill graphite yarns prior to weaving the 8 harness satin 24 x 24 (warp/fill) fabric. The serving was a double contro-rotating helix of 1/0 E glass. It was anticipated that the presence of the server would reduce the effective yarn count (number per mm of ply thickness). This would reduce the modulus and strength of the composite by an estimated 10%--the density in turn would also be increased by 3%-4% due to the glass addition. As estimated, thickness was 6.85 mm for a construction that was identical to concept #1 above.

Concept #4 - This concept is similar to the uniweave concept #3 (in the "thin" category) with the addition of milled and/or glass flake. The technique investigated the effect of melt type hybrid fibers coupled with the same type of resin additives for additional retention capability. The composite strength and modulus were expected to be decreased by

14% when compared to a conventional woven composite and a 32 ply, 6.5 mm thick $(0°, \pm 45°, 90°)_s$ panel was calculated.

Concept #5 - This is the same as concept #5 in the thin category, being a modification of the baseline reference panel where the graphite tows were to be double served with 450 1/0 E glass. A 48 ply thick $(0°, \pm 45°, 90°)_s$ construction of a thickness of 6.70 mm was calculated

Concept #6 - This concept utilizes a fire protection coating of Avco Flamarest™ 2600B. This material is an intumescent type coating material which supplies thermal protection to the substrate material. The paint layer was to be applied approximately 0.76 mm thick to both sides of the reference composite material. The protective layer amounts to a weight penalty of approximately 9%.

Concept #7 - This concept employs a panel surface coating of unidirectional boron fibers in the warp direction and glass tie yarns in the fill direction added to the fabric construction used in concept #1 above. A total thickness of 7.62 mm was calculated. This concept would not reduce the strength and modulus of the composite but would increase the density by 2.5%. This concept would evaluate the effectiveness of structural surface layers.

Concept #8 - The concept also evaluated a surface layer material. The approach consisted of adding a non-melt layer of a silica or quartz fabric to the surface of the graphite fabric used in concept #1 above. The use of the fabric layer penalizes the composite by increasing the density. This effect will be less than 10% for composites at this thickness. A total thickness of 7.77 mm was calculated.

A summary of recommended "thick" panels is shown in Table 2.

TABLE 2

RECOMMENDED THICK PANEL CONCEPTS

Design Concept	Panel Construction		Panel Thickness (mm)	
	No. of Plies	Lay-Up Sequence	Design	As Fabricated
Reference Quasi-isotropic GR/Epoxy Thornel 300/Avco [illegible]35	48	$[0/90/\pm45]_s$	6.7	6.85
1. Graphite Fabric - 8 H/S weave with glass scrim (104) interleaf	20	$[90/45/90/\pm45/0/90/\pm45/0]_s$	7.11	7.21
2. Graphite Fabric - E-glass veil interleaf and (carbon/silica) resin additives	20	Same as No. 1	7.11	7.21
3. Graphite Fabric with glass served graphite yarns	20	Same as No. 1	6.85	[illegible].65
4. Unidirectional fabric with glass server yarns and glass fill yarns and glass additives	32	$[0/90/\pm45]_s$	6.50	5.13
5. Reference with graphite double glass served (braided)	See Note (1) 48 (24)	$[0/90/\pm45]_s$	7.31	6.73
6. Fire Protection Intumescent coating sprayed on panel - Flamarest 1400	48	0.030 Surface Coating of Avco Flamarest 1600B applied to reference.	8.22	
7. Unidirectional boron/glass surface layer over graphite fabric	20	0.010 Molded Surface Layers on Concept #1	7.62	8.89
8. Silica/quartz surface fabric over graphite fabric	20	0.013 Molded Fabric Layers on Concept #1	7.77	7.69

(1) See Text (Section 3)

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3.0 Laminate Fabrication

A total of more than thirty, 23.0 x 23.0 cm, hybrid panels were fabricated for test purposes. The fabrication technique used was press molding to stops, where the stop thicknesses were based upon the theoretically required fiber volumes and prepreg thickness.

In general, the fabrication procedures worked well, except for some of the served material where it appears that the 3000 end tow, when served with the glass, retained its bulk and resisted flattening during the prepregging and molding operations. By adjusting the tow spacing and the per ply thickness, satisfactory laminates were fabricated for all panels except the 8 ply double served graphite panel (originally concept "thin 6") where the per ply thickness increased to a level where a satisfactory quasi-isotropic panel could not be fabricated. Also, the number of plies for the "thick 5" panel had to be reduced by a half. Figure 6 compares a cross section of the reference panel, Figure 62, showing adequate flattening of the 3000 end tow, to the double served tow used in "thick 5" which has remained in discrete bundles within each layer (Figure 6b). The addition of the carbon black and glass additives to the resin was accomplished without any problem and the glassy resin (Siloxane) was impregnated into the tow by drawing the tow through a resin bath and exiting through a controlled orifice. The impregnating of the continuous tow prepregs was accomplished by the standard wet drum winding technique. For the fabric material the resin was impregnated using the squeegee technique on a flat table. All woven materials were purchased from Fabric Development, Pennsylvania.

Table 3 is a presentation of the result of the x-ray analyses, gross density measurements and panel thickness.

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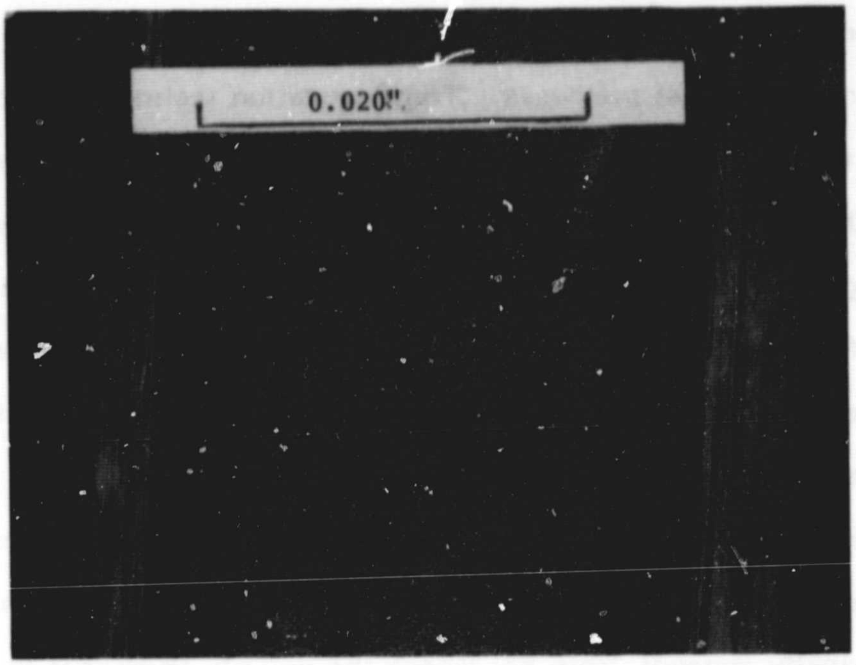
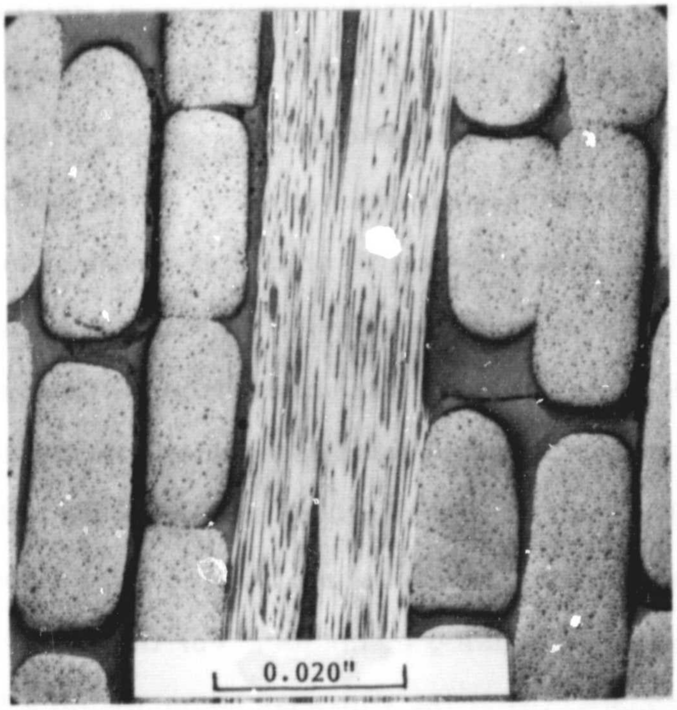


a- Thin Ref



b- Thick 5

Figure 6 Cross Section of "Thin" Ref and Thick 5

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Table 3
X-Ray And Physical Observations
Of
Hybrid Panels As Fabricated

Panel No.	High Density Inclusions (Dia)	Low Density Bands	Geometric Distortions	Bulk Density (grm/cc)	Average Thickness (mm)
TNRA	15-20 > 0.8mm	1.2 to 2.5 cm Long	Minor Waviness	1.529	1.06
TNRB	10 < 0.8mm	---	---	1.537	1.09
TN1A	21 < 0.25 mm	3 Small Regions	---	1.536	1.32
TN1B	12 < 0.25 mm	---	---	1.549	1.37
TN2A	---	---	45° Bowed	1.507	1.32
TN2B	1 ∿ 0.8mm	---	Some Bowing	1.429	1.47
TN3A	1 ∿ 3.2mm	---	Some Bowing	---	1.57
TN3B	2 < 0.8mm	---	Some Bowing	1.607	1.21
TN4A	Extensive	Minor	---	1.44	1.11
TN4B	12-15 < 0.8mm	Minor	---	1.545	1.14
TN5A	---	Extensive @ 90° & 45°	---	1.527	1.06
TN5B	1 @ 1.6mm 10 < 0.8mm	---	---	1.613	1.11
TN7A	---	---	---	1.528	1.32
TN7B	10 < 0.40mm	---	Minor Waviness	1.582	1.42
TN8A	---	---	---	1.433	1.34
TN8B	3 - .40 mm	---	---	1.521	1.34

Table 3 - Cont'd.

X-Ray And Physical Observations Of Hybrid Panels As-Fabricated

Panel No	High Density Inclusions (Dia)	Low Density Bands	Geometric Distortions	Bulk Density (grm/cc)	Average Thickness (mm)
TKRB1	1 - 0.8mm	Minor	---	1.549	7.39
TKRB2	14 - 0.40mm	---	---	1.524	6.85
TK1A	2 < 0.25mm	---	---	1.579	7.23
TK1B	6 ∿ 0.25mm	---	---	---	7.21
TK2A	15 ∿ 0.12 - 0.63 mm	---	---	1.621	7.21
TK2B	15 ∿ 0.12 - 0.9 mm	---	---	1.610	7.51
TK3A	---	---	---	1.472	9.42
TK3B	---	---	---	---	9.65
TK4A	1 - 0.25mm	Intermittent	---	---	5.13
TK4B	2 - 0.12mm	Intermittent	---	---	5.13
TK5A	2 ∿ 0.12mm	Several @ 45°	---	1.565	6.73
TK5B	---	Minor	---	1.417	6.73
TK7A	12 - < 0.25 mm	One Area 2.5 cm Wide in Center	---	1.511	8.89
TK7B	35 - 0.12 - 0.9mm	---	Slight Waviness	1.519	7.72
TK8A	5 - 0.25mm	---	---	---	7.69
TK8B	6 - 0.12 - 0.5mm	---	---	---	9.11

Figures 6 through 13 are photomicrographs of various panels illustrating different types of cross sections, varying from highly stratified material separated by the glass scrim and veils to the very discrete construction of served bundles of tow.

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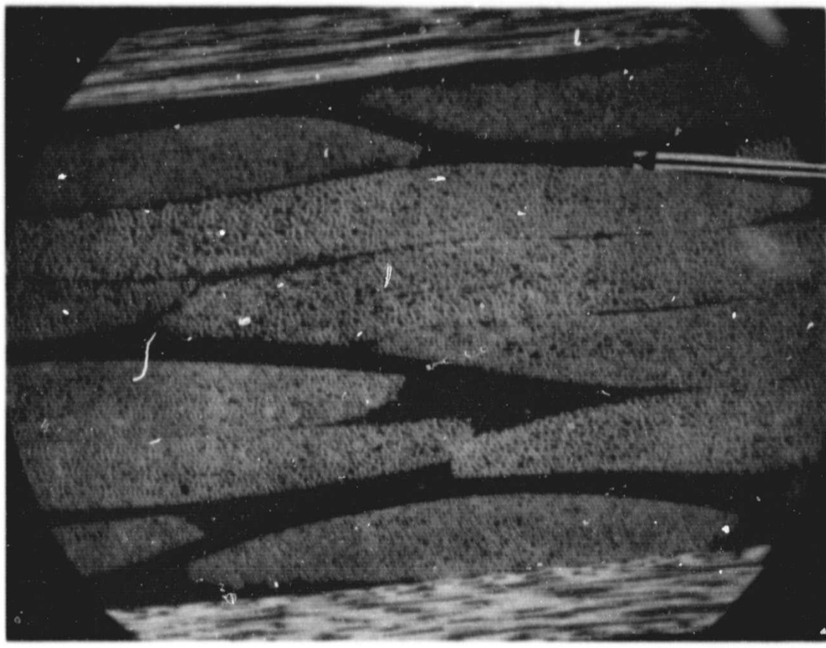

Glass Tie Yarns

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Figure 7 Cross Section Thin 2B

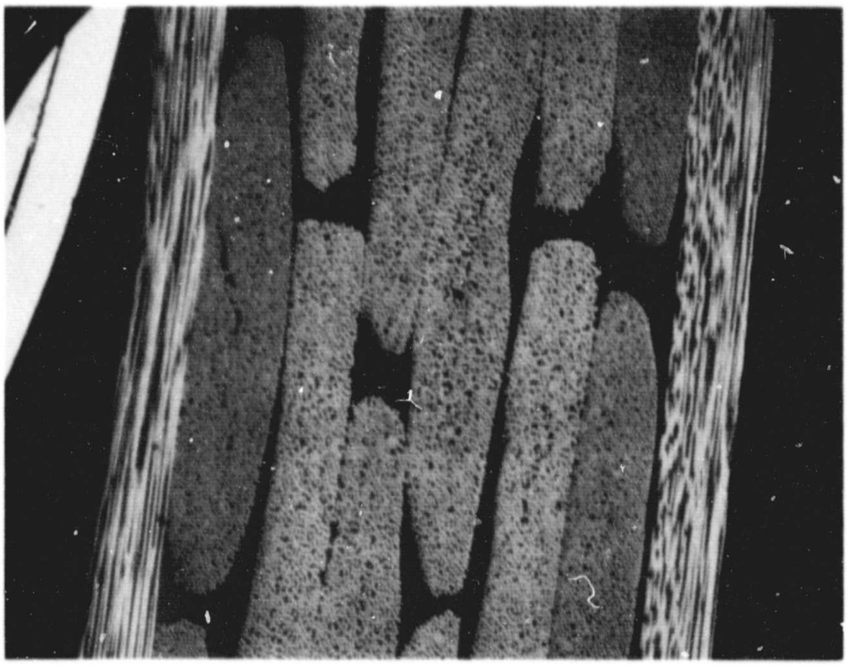

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Figure 8 Cross Section Thin 3B

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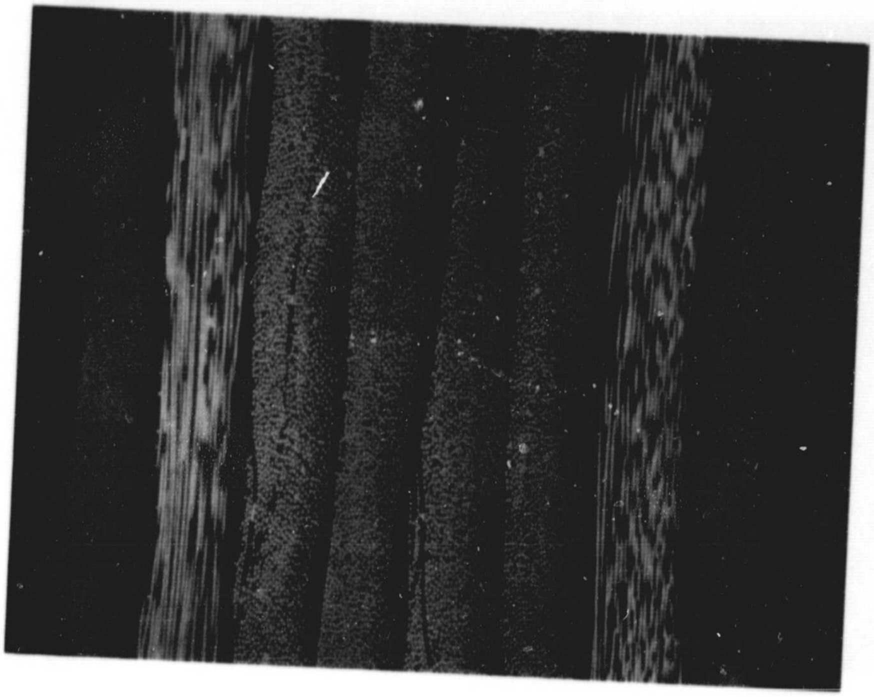

Figure 9 Cross Section Thin 7B 70X

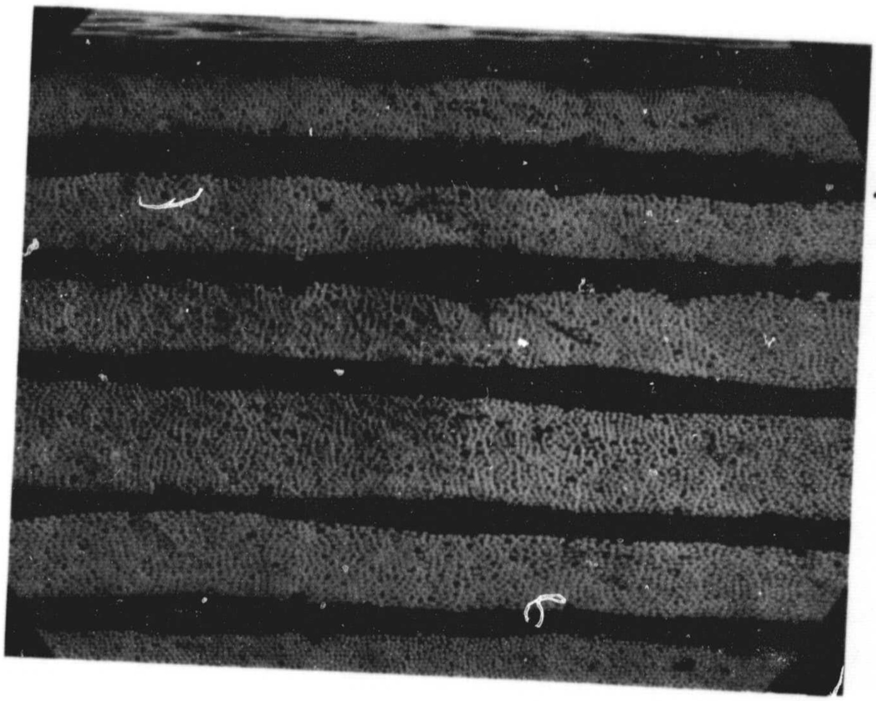

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Figure 10 Cross Section Thin 8B

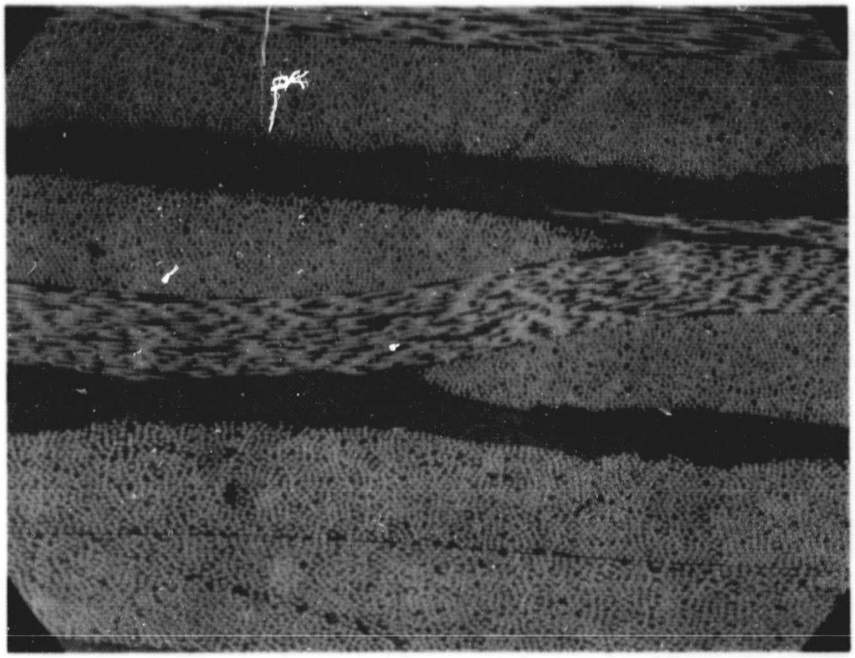

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Figure 11 Cross Section Thick 2A

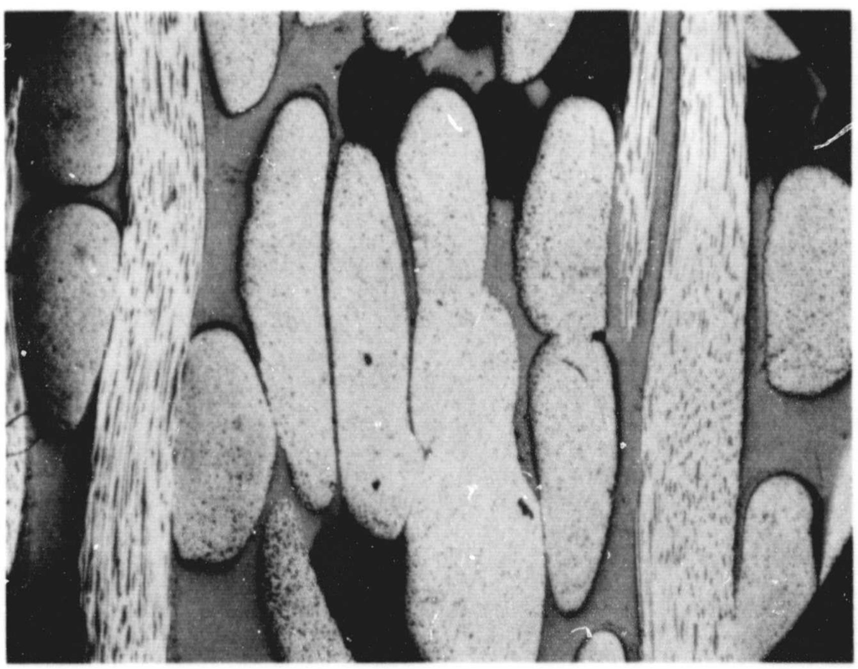

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Figure 12 Cross Section Thick 3A

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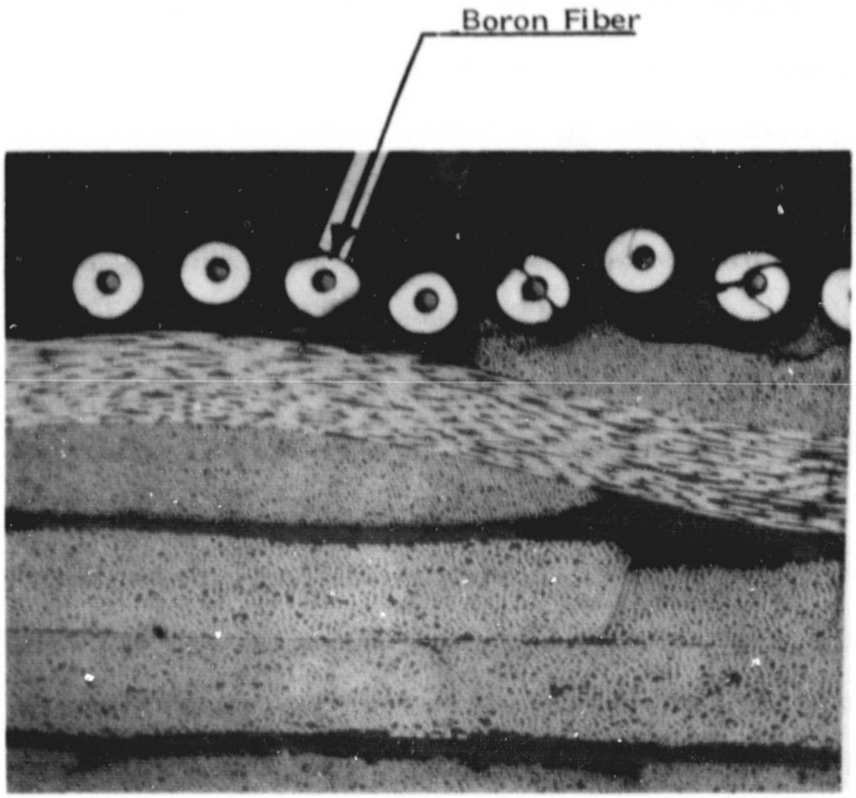

Figure 13 Cross Section Thick 7B

4.0 Material Tests

Material tests of the baseline and the hybrid concepts were conducted in the area of fire testing, mechanical testing and physical testing.

4.1 Fire Tests

Fire Test Facility - Interest in the evaluation of composite material and the assessment of their resistance to aircraft crash fire environments has led to a requirement to define a suitable test to quantitatively evaluate their performance. The test method requires stimulation of the thermal parameters and combustion chemistry of aircraft crash fires and sufficient structural disturbance of the sample to cause dissemination of fiber material. Recovery of substantially all solid residue is desired, particularly that component consisting of small single fibers which would be readily distributed by air currents in a actual fire.

The Avco Model 25 fire simulation facility, located at Lowell, Massachusetts, is applicable to this type of activity. It has been in operation for nearly ten years in support of the development of fire protection materials and has recently, under contract from NASA (NAS1-15511), been modified to support this and other composite test programs.

The basic Avco Model 25 fire facility as it was originally configured is illustrated in Figure 14. It consisted of an electrically heated ceramic hood to provide a radiation heat source, a natural gas supply system to provide convective heating and combustion gases, and an opening in the floor for insertion of a test specimen mounted flush to the floor of the test section. Under sponsorship of NASA, the facility was extensively modified to provide the capabilities necessary for evaluation of the fiber release

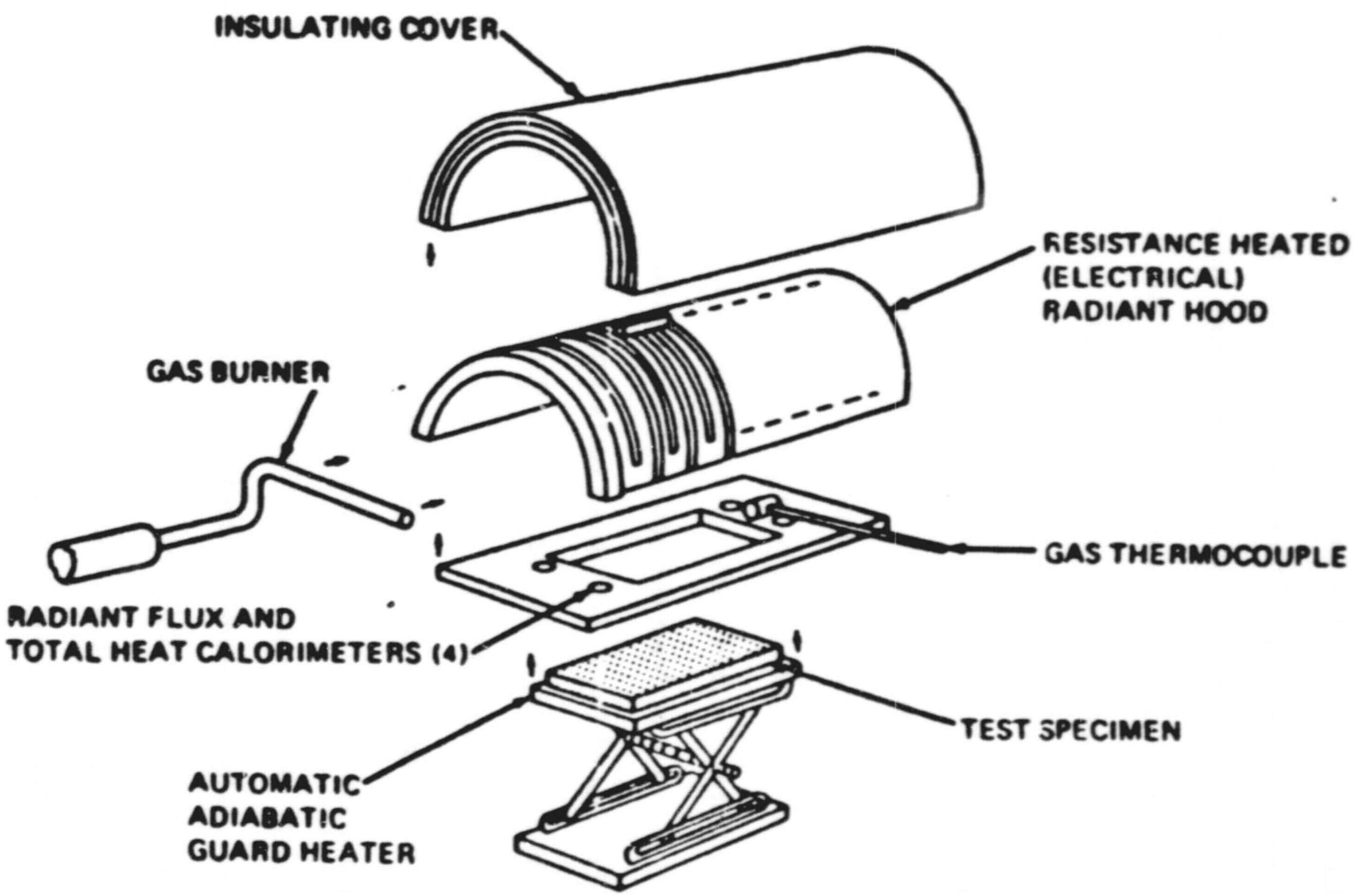

Figure 14 Original Model 25 Fire Facility Configuration

characteristics of graphite-reinforced composites. The principal modifications included a forced draft exhaust system, a fiber trap, glass flow instrumentation, and a specimen agitation device.

The final configuration is illustrated schematically in Figure 15 and is shown in Figure 16. The exhaust is provided by a ½ H.P. centrifugal blower and is sufficient to draw 85 m^3/hr. air through the entire system. The fiber trap is essentially a small water-bath air-scrubber made of stainless steel and operating at a water flow rate of 20 liters per minute. The combustion gases are passed through a water spray which is effective for cooling the gases to about 93°C. The gases are expanded into a plenum chamber and passed through a vertical water curtain which removes the fibers from the combustion gas. The fibers are then collected on a cellulosic filter through which the scrubber water is passed.

The water curtain effectively removes not only the graphite fiber but quantities of soot from composite resin products and from combustion of rich mixtures of fuel. For those tests where a soot-fiber mixture was collected on the filter, it was found that the soot could be made to pass through the filter by adding a moderate quantity of detergent to the mixture. Fiber samples collected in this manner from graphite-epoxy burns were quite clean. The fiber collected in the water trap included single fibers, lint or small clumps of fiber, and perhaps even a few very small fragments. Weight of fibers is deduced by weighing the fiber-loaded filter after drying and comparing with the pre-test weight of the filter. Fiber samples from typical tests range from 1/20 to 1/4 gram and are measured to the fourth decimal place with a Mettler micro-balance.

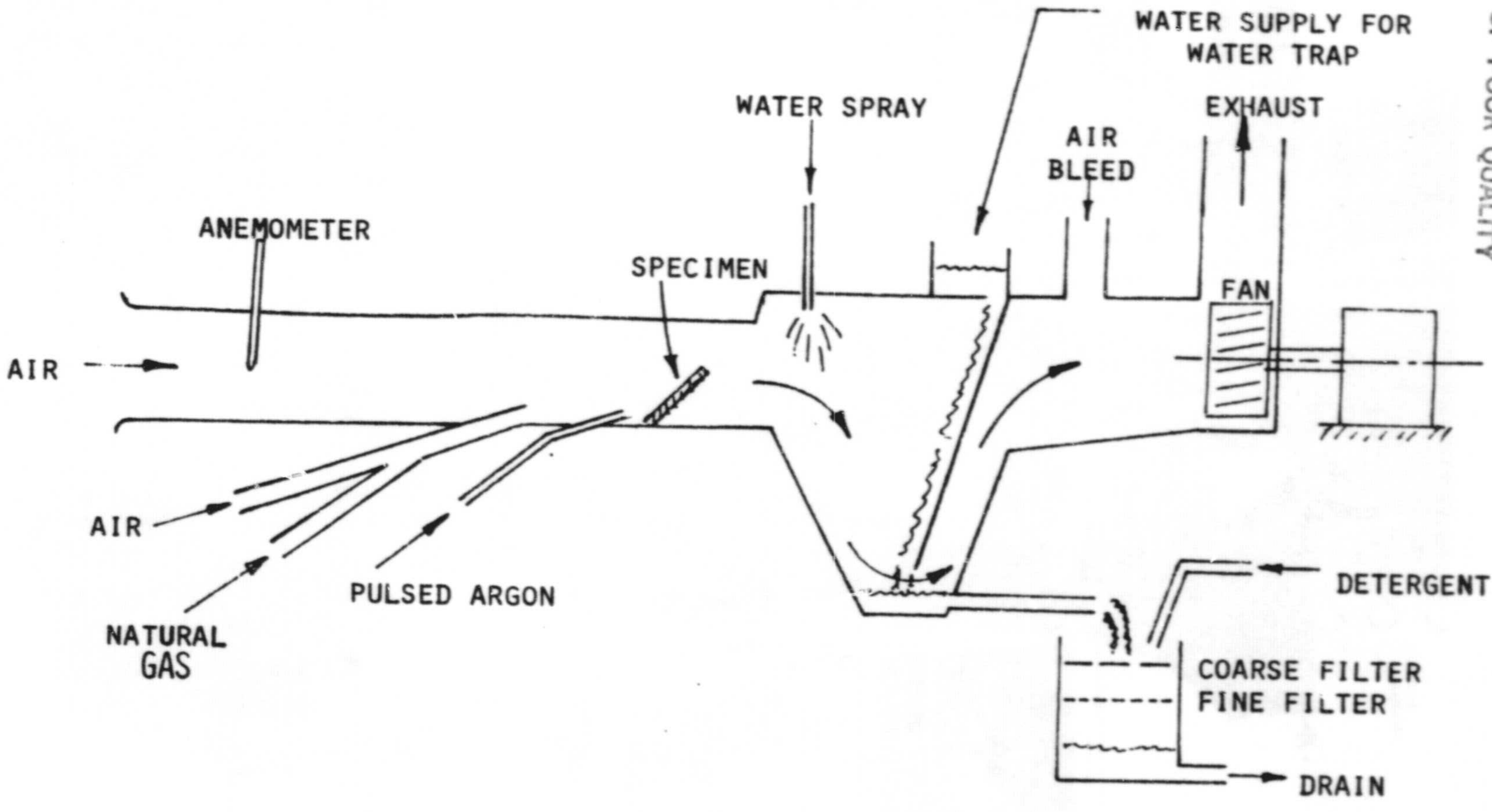

Figure 15 Facility Schematic

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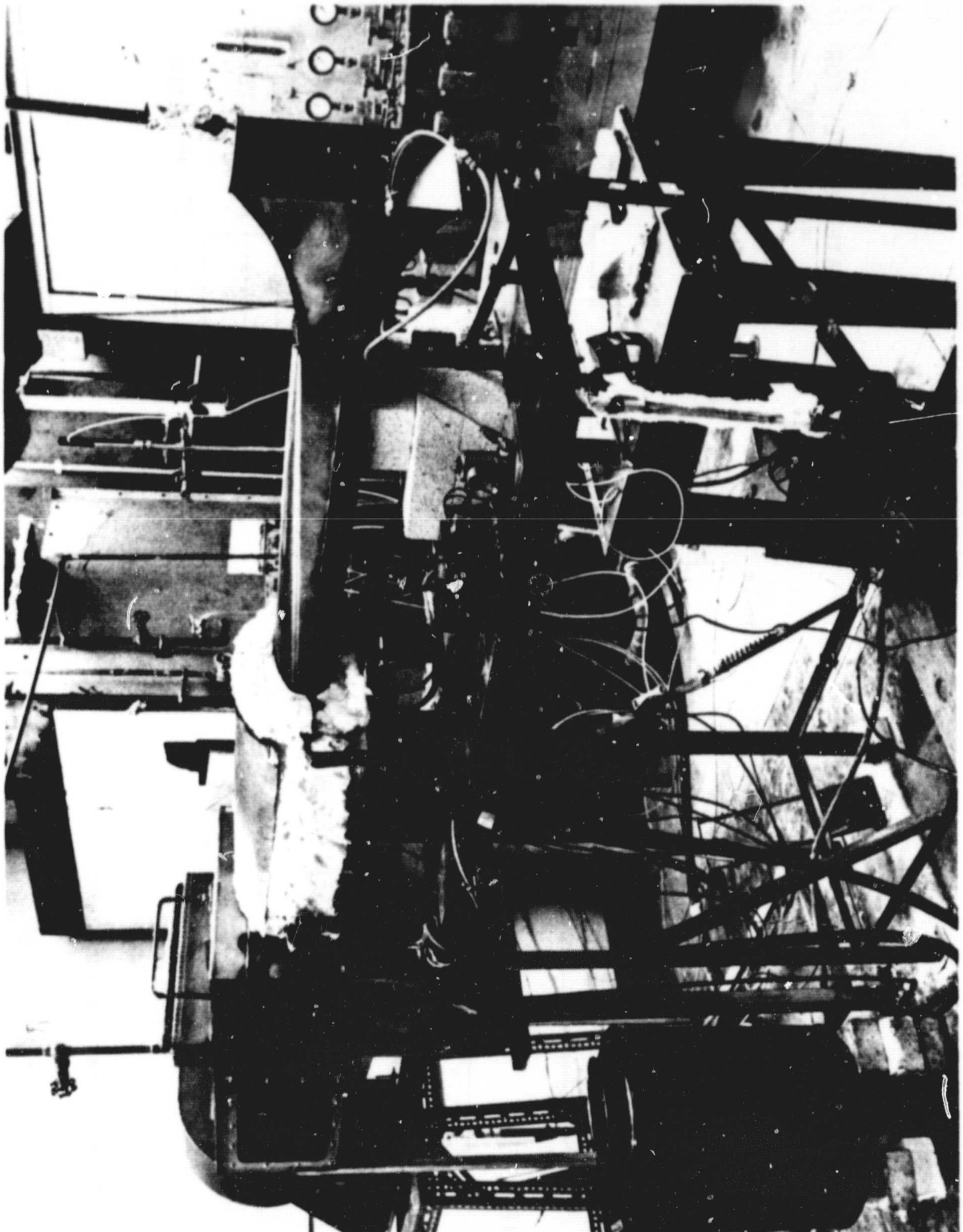

Figure 16 Modified Model 25 Facility

Experiments were performed to verify the effectiveness of the fiber trap by operating the system cold with a dry cellulosic filter installed at the exhaust blower inlet. Chopped graphite fibers 2 mm long, were introduced into the fiber trap with a paint-sprayer using a pre-mix of fibers and water. No fibers were observed reaching the dry filter while substantial quantities were collected on the wet filter.

A number of flat-panel specimen configurations and orientations were evaluated in the course of developing a standardized test procedure. These included the following: (1) flush-mounted in the floor of the test section, (2) perpendicular mounted to the floor but parallel to the flow direction, (3) mounted perpendicular to the floor and oriented perpendicular to flow, (4) and oriented at 45 degrees to both the floor and the flow direction.

The recommended orientation is with the specimen at 45 degrees to both the floor and the flow direction and with the unrestrained edge exposed to the combustion gas flow (Figure 17). This configuration provides several advantages over the others evaluated:

a) The specimen can be readily observed and photographed from the upstream furnace inlet.

b) Calorimeters can be installed for direct calibration of radiative and convective flux. (This is not possible for vertically mounted specimens because the calorimeter sides and backside cannot be properly protected.)

c) Fibers released from the heated surface are readily swept off the surface by the gas flow.

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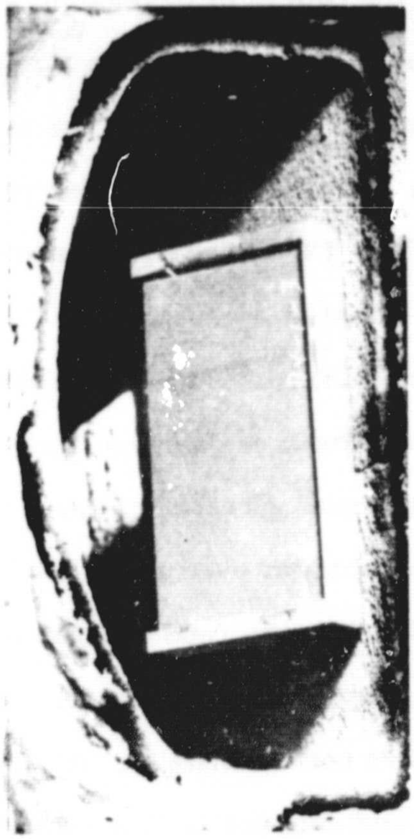

Figure 17 - Specimen mounted 45° to Floor

d) Convective heat flux is maximized; levels of 57 kw/m^2 are readily achieved. (However, radiative fluxes are reduced to 2/3 that received in the floor mount location, because of a reduced view factor between the specimen and the radiative hood.)

Heat flux measurement is accomplished with Hi-Cal Corporation Model C-1300 and R-2040 Asymptotic^R calorimeters and radiometers. The design of the two instruments differs only in that the radiometer has a transparent window over the sensing element which eliminates sensitivity to convective heating. The calorimeter element is sensitive to both the radiative and convective environments. The convective flux is deduced from the difference in the outputs of the two instruments. The manufacturer provides emissivity corrections for these instruments. Heat flux measurements are obtained by substituting the calorimeter block at the specimen location. Calorimeter measurements are not possible during actual sample testing, but repeatability of test conditions is excellent and the substitution method is not considered a significant source of error.

The fuel source is commercial natural gas (methane) supplied through a Selas Corporation Model 20-CA combustion controller and a Model SH-4-FF burner nozzle (5 x 80 mm gas flow opening). Fuel and air are metered and pre-mixed by the controller. The burner nozzle mixture is injected through the floor of the furnace inlet duct where it is mixed with the air supplied by the forced draft system. The latter airflow rate is deduced from hot-wire anemometer measurement of the air velocity just downstream of the bell mouth in the inlet duct.

A range of gas flow and airflow rates was investigated during early testing in the facility and much of the early composite burn data were obtained with widely varying flow rates. The standard "lean" fire environment is achieved with $5m^3$/hr air and 180 scfh gas supplied from the Selas controller and mixed with 66 m^3/hr air flow from the inlet duct supply. The latter corresponds to a duct velocity of 1 m/s as measured by the anemometer and results in a lean air/fuel mixture ratio of 14 in the furnace test section. (Stoichiometric ratio is 9.8.)

The standard "rich" fire environment is achieved with the same fuel and air rates from the Selas, but with a reduced inlet duct velocity of 0.3 m/s. This provides 20 m^3/hr flow in the inlet duct and an air fuel ratio of 5. This mixture ratio results in a very sooty fire and much soot deposited on the fiber trap wet filter along with the fibers. This soot is readily washed through the filter with a detergent. The variation in inlet duct air flow is achieved by varying the opening of bleed ports in the fiber trap chamber.

It should be noted that the duct velocities of 1 m/s and 0.3 m/s indictated are those of the room temperature air at the metering location. At the specimen location the velocities are estimated at 7.6 and 3.0 m/3, considering the blockage caused by the specimen and the expansion of the combustion gases upon heating to 1089°K (1800°F).

A flow agitation system is provided by injecting a small, pulsating flow (5 pulses per second) of argon parallel to the main combustion gas flow. This flow is directed at the center of the specimen heated surface with the intent of increasing the breakage and release of fibers which have lifted from the surface. However, the argon flow also imparts an overall pulsation to the combustion gas flow which is judged (subjectively) to be similar to the turbulence present in a large fire. The agitation flow velocity at the specimen location is estimated at 4.6 to 6.0 m/s based on an anemometer survey of the jet.

Use of the agitator greatly increases the fiber release rate for those materials which permit fibers to lift from the surface. Without agitation, the lifted fibers are consumed by combustion in a few seconds and relatively little material is collected downstream. With agitation, the fibers tend to fracture more readily and are carried out of the furnace before they are burned.

The orientation of the specimen at a 45 degree angle to the flow also enhances fiber release by the combustion gases sweeping the surface. For specimens oriented perpendicular to the flow, fractured fibers tend to remain trapped on the surface and are consumed by combustion.

Fire Test Results - Fire testing of the hybrid panel and reference panels were completed in the Avco code 25 facility, modified as discussed previously for evaluation of the fiber release problem. Under the NASA Langley contract, a test procedure for thick and thin panels was established. Two Air/Fuel ratios were selected namely 15:1 and 6:1 (lean and rich). The specimen was mounted at a 45° angle as discussed previously and a total of 38 tests conducted on the "Thick" and "Thin" panels. The standard test parameters are outlined in Table 4. The results of the "Thick" panel tests are shown in Table 5 which gives the test log book number, the specimen identification number (e.g., 2B-15 is concept #2, panel B, test specimen identification number 15), specimen

TABLE 4

RECOMMENDED STANDARD TEST

Specimen Configuration

Size 114.3 x 63.5 mm (4½ x 2½ inches), thickness variable.

Specimen mounted 45° to floor

Three edges restrained in steel frame

Nominal Test Condition

Lean air/fuel mixture, 15:1

Rich air/fuel mixture, 6:1

Radiation source temperature, 1256°K (1800°F).

Radiation flux at specimen, 102 Kw/m^2 (9 Btu/ft^2-sec).

Convective flux at specimen, 57 Kw/m^2 (5 Btu/ft^2-sec).

Local velocity at specimen, 7.62 m/s (25 ft/sec) in lean environment, and 3.05 m/s (10 ft/sec) in rich environment.

Pulsed gas (argon) agitation of specimens.

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TABLE 5 - FIRE TEST RESULTS - THIN SPECIMENS

Test No	Specimen Identification	Orientation	Initial Spec Wt (Gm)	Post Test Weight (Gm)	Collected Fiber Wt (Gm)	Air/Fuel Ratio	Test Time Min	Comments
111	TN-RA-45	V	11.5	2.69	0.111	15	3.0	1st layer delaminated and lost at 40 sec. Layer by layer material loss-thru at 3 min
119	-43	H	11.4	2.17	0.0586	15	3.5	1st layer gone at 50 sec-thru at 2½ min
83	-42	H	11.4	2.13	0.204	6	3.2	1st layer lost at 10 sec-2nd layer at 1.2 min thru at 3¼ min
112	TN-1A-50	V	14.5	4.11	0.219	15	3.2	1st layer damage at 1.5 min-thru at 3.2 min
120		H	14.7	3.09	0.2550	15	3.8	Surface damage at 2 min-1st layer gone at 2.5 min-thru at 3.8 min
84	-47	H	14.31	4.08	0.198	6	3.5	1st layer gone at 20 min-thru at 3.5 min
113	TN-2A-55	V	14.2	3.84	0.127	15	3.5	1st layer damage at 1.0 min-thru at 3.5 min
121	-52	H	14.1	3.68	0.1029	15	2.5	1st layer gone in less than 10 sec-thru in 2½ min
114	TN-3A-60	V	16.8	4.41	0.022	15	4.0	1st layer damage at 1.0 min-spec looks good fire - no burn thru
76	-56	H	16.4	4.58	0.061	15	4.0	Spec looks good-no delamination in fire
85	-57	H	16.36	6.14	0.043	6	4.0	1st layer damage at 2.2 min-looks good in fire. No burn thru
115	TN-4A-65	V	12.0	2.43	0.001(?)	15	4.0	1st layer damage at less than ½ min - burned thru in 4 min
77	-61	H	11.4	1.85	0.178	15	4.0	Coming apart at 15 sec-no burn thru
116	TN-5A-70	V	12.0	3.54	0.069	15	4.0	1st layer damage at 1 min-no burn thru
78	-66	H	11.8	2.11	0.137	15	4.0	Early delamination-looks poor in fire
117	TN-7A-75	V	15.0	4.92	0.004	15	4.0	Delaminated in 10 sec-much fiber fluffing No burn thru
79	-71	H	15.0	5.13	0.114	15	3.0	Early delamination-less than a min-thru in 3 min
118	TN-8A-80	V	14.0	5.21	0.010	15	4.0	No burn thru
80	-76	H	14.0	4.12	0.101	15	4.0	Looks poor in fire-no burn thru

were placed vertically), initial specimen weight, post test weight of specimens; weight of fibers collected in filter; air/fuel mixture ratio; time of test and comments.

The "thick" test series was extremely successful and provided positive guidance for further examination of hybrid concepts. The reference quasi-isotropic panel exhibited major surface erosion and extensive delamination throughout. If a large test panel had been evaluated, there is no doubt that the turbulent forces would have completely destroyed the material--Figure 18 is a photograph of the specimen remains. In dramatic contrast, Figure 19 shows the remains of the best performer of the "thick" panel concepts. (Concept # 3 - the 8 Harness Satin fabric material that has each graphite tow double served with an E glass yarn prior to weaving). The test specimen (#3) was of course charred completely throughout, however the section was completely intact and it was impossible to deform the section by hand. Each layer was observed to be attached to its neighbor by the melted glass yarn. One further observation of note was the tendency to grow in thickness---presumably by pressures resulting from the decomposition gases.

The performance of specimen #1 with a layer of 104 glass scrim between each layer of fabric was somewhat inferior, exhibiting some surface erosion and in-depth delamination. (See Figure 20.) There was some evidence of unmelted glass scrim on the surface and when broken apart by hand the scrim was observed to be un-melted. The 104 scrim was constructed from S glass which has a softening point of 1204°K compared to 1116°K for the E glass used in the other panels.

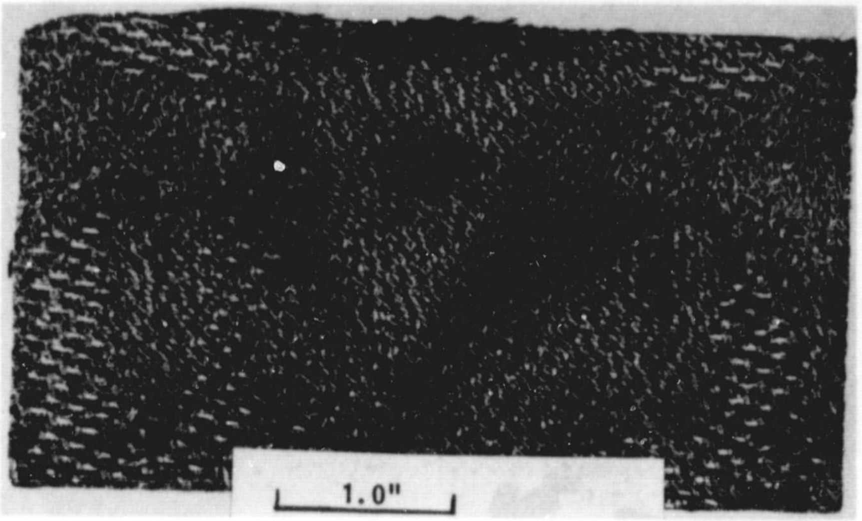


Figure 19 Post Test - Thick 3

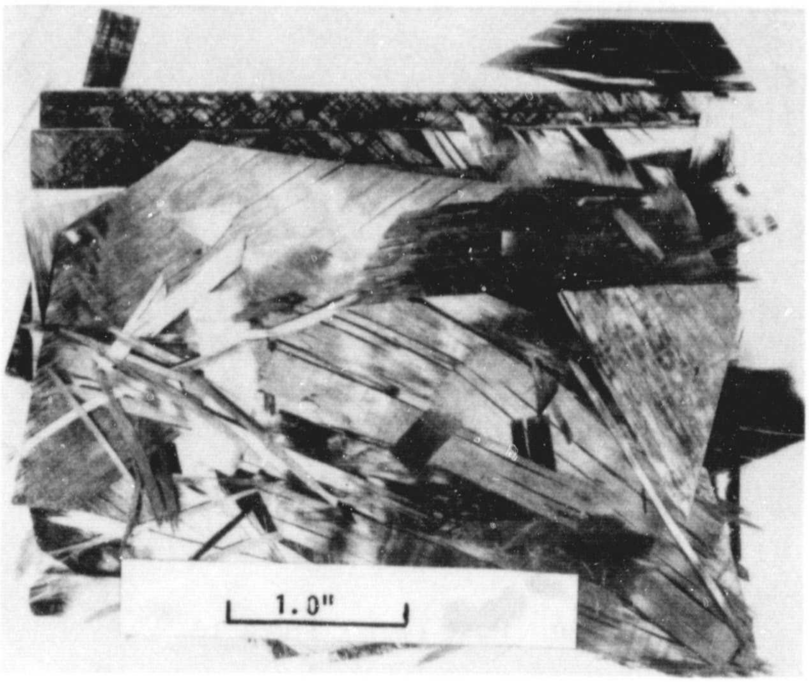
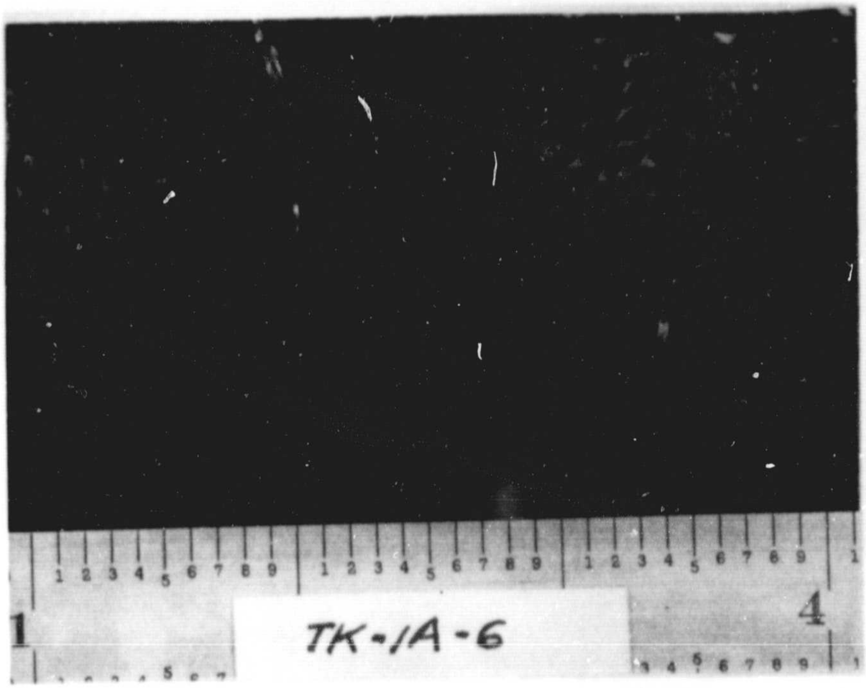


Figure 18 Post Test - Thick Ref



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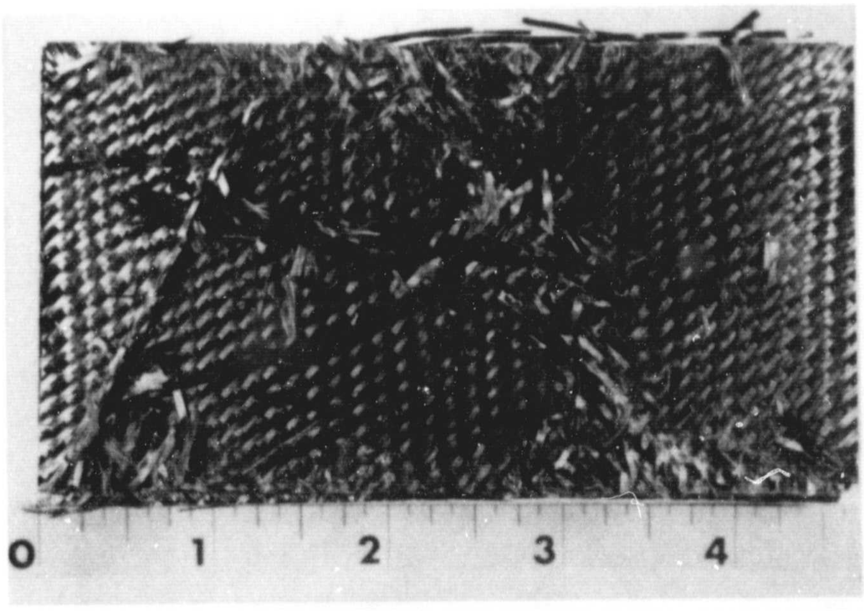

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Figure 20 Post Test - Thick 1A

Specimen #2 performed nearly as well as #3 in terms of physical appearance and in fact was superior to #3 in terms of weight loss. However, #2 produced more than double the amount of collected fibers. The post test specimen also exhibited less expansion than #3. It also appeared that the E glass veil had melted and held the panel together. (See Figure 21.)

Specimen #4 was constructed from the uni-fabric with glass fill yarns and siloxane resin additive. The specimen exhibited severe surface delamination and could easily be picked apart. It appeared that each layer of material was in itself "integritized" but not "bonded" to its neighbor. The few graphite yarns that were picked from the edge of the specimen were securely held together by the charred resin. (See Figure 22.)

Specimen #5 exhibited severe delamination. However the bundles of graphite tow were securely and uniformly held together by the melted glass serving. Specimen #7 used the boron layer on the outer surface only. The boron layer was totally disturbed and it failed to protect the sublayers. (See Figure 23.) The remainder of the panel were damaged to an extent similar to #1. The outer layers of quartz fabric on #8 was also ineffective in protecting the graphite sub-layer. (See Figure 24.)

The trend in the weight of the collected fibers generally followed the physical assessment, indicating less fibers collected for increased post test integrity. A number of analyses, such as weight loss ratios and collected weight to initial weight, were conducted, however no trend was evident in the results. Also, it appeared that the two air/fuel test ratios utilized were equally damaging.

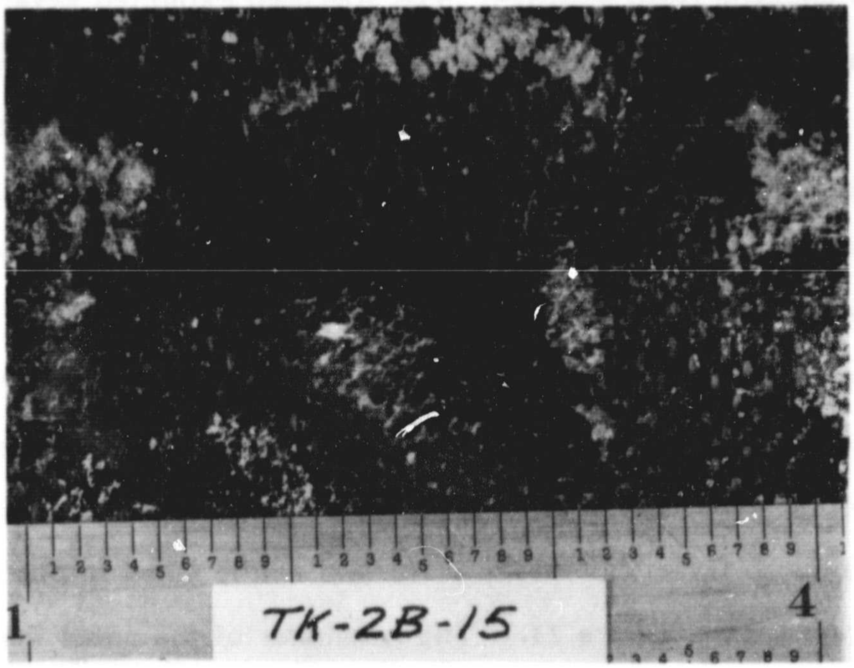
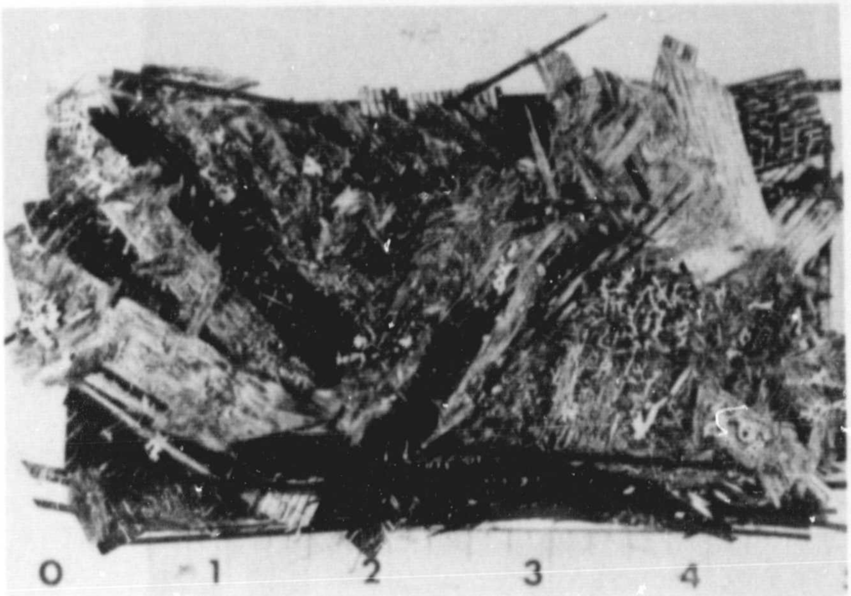
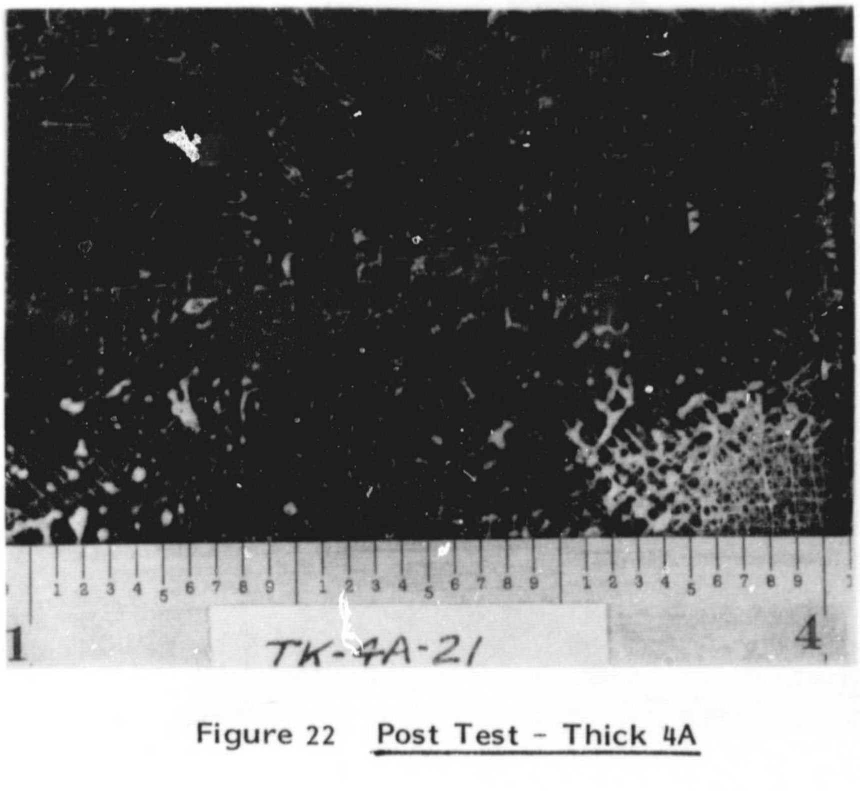


Figure 21 Post Test - Thick 2B



Thick 4B25



Thick 4A21

Figure 22 Post Test - Thick 4A

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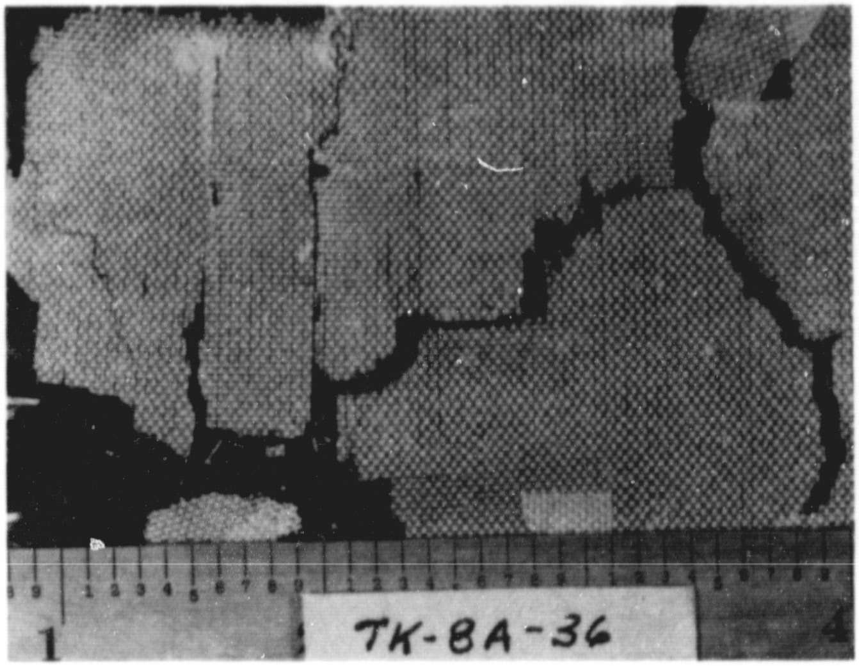

Figure 24 Post Test - Thick 8A

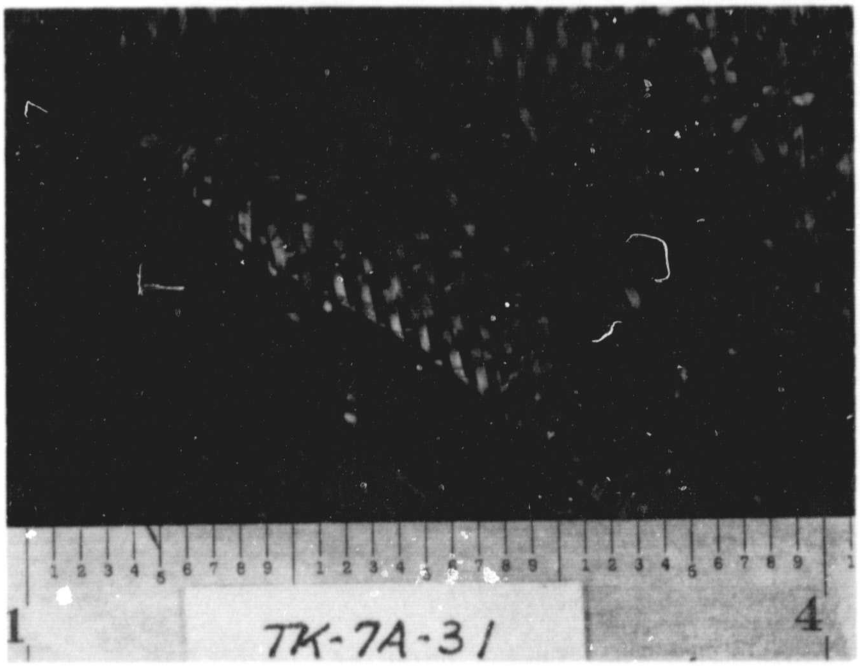

Figure 23 Post Test - Thick 7A

The "thin" panel tests exhibited severe damage compared to panels. Table 6 illustrates the results of the fire tests, the format being the same as before, however, more test comments are included due to the larger number of observable incidents. All of the test panels (except #3) broke up into many layers. In some cases individual layers and whole sections were destroyed. Some tests had to be terminated early due to extensive damage. Specimen #3 remained intact with obvious benefits attributed to the glass serving on the uniweave. Two layers were removed from the front face over a small area. (See Figure 25.)

The remainder of "thin" specimens were severely damaged, as indicated by the test comments in Table 5. Specimens 4, 7 and 8 were also severely damaged, but there was evidence in these specimens of the glass materials coalescing around the fibers. Some of the remaining materials were held together in bundles by a fused layer of silicon. Figures 26, 27 and 28 are some photographs of specimens 5, 7 and 8.

Further Fire Tests - The main set of fire tests reported above were conducted without concern for the condition of the material collected downstream other than to establish the weight of the material for comparison with the panel weight. In a separate set of fire tests, a detailed analyses of the collected fiber was conducted. Table 7 is a presentation of the test conditions. Figure 29 is a full scale photocopy of the fiber residue on the filter, and Figure 30 and 31 are results of microscopic analysis. A total of 150 particles were examined on the filter and measurement of the length and diameter of the particles were recorded. Figure 30 shows a histogram of the

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TABLE 6 FIRE TEST RESULTS (THICK)

Test No	Specimen Identification	Specimen Orientation	Initial Spec Wt (Gm)	Post Test Weight (Gm)	Collected Fiber Wt (Gm)	Air/Fuel Ratio	Test Time (Min)	Comments
122	TK-RB-5	V	78.3	48.70	0.068	15	6.0	Major Surface Delamination
67	TK-RB-1	H	80.0	44.50	0.173	15	6.0	Delaminations Throughout
	-2	H	81.0	47.44	0.109	6	6.0	
123	TK-1A-10	V	80.1	52.50	0.186	15	6.0	Minor Surface Delamination
68	TK-1A-6	H	69.5	52.05	0.119	15	6.0	Reduced In Depth Delamination
87	-7	H	79.9	52.78	0.194	6	6.0	
124	TK-2B-15	V	85.9	60.20	0.141	15	6.0	Minor Surface Delamination
70	TK-2B-11	H	85.0	56.63	0.106	15	6.0	High Interlamina integrity
126	TK-3B-20	V	101.4	60.00	0.035	15	6.0	Virtually No Surface Delamination
75	Tk-3B-16	H	101.2	57.27	0.061	15	6.0	Very High Interlamina Integrity
89	-17	H	105.6	59.65	0.042	6	6.0	
125	TK-4B-25	V	56.7	37.70	0.073	15	6.0	Severe Surface Delamination
69	TK-4A-21	H	57.0	36.16	0.052	15	6.0	Delamination Throughout
127	TK-5B-30	V	69.7	45.30	0.034	15	6.0	Severe Surface Delamination
74	TK-5B-26	H	69.0	43.86	0.083	15	6.0	Major Delamination
128	TK-7A-35	V	83.5	57.70	0.257	15	6.0	Loose Boron Fibers
71	TK-7A-31	H	83.2	53.62	0.123	15	6.0	Delamination Throughout
129	TK-8A-40	V	82.0	56.70	0.015	15	6.0	Surface Intact
72	TK-8A-36	H	82.0	56.02	0.016	15	6.0	Delaminations Throughout

(1) V - Surface Fibers Vertical
H - Surface Fibers Horizontal

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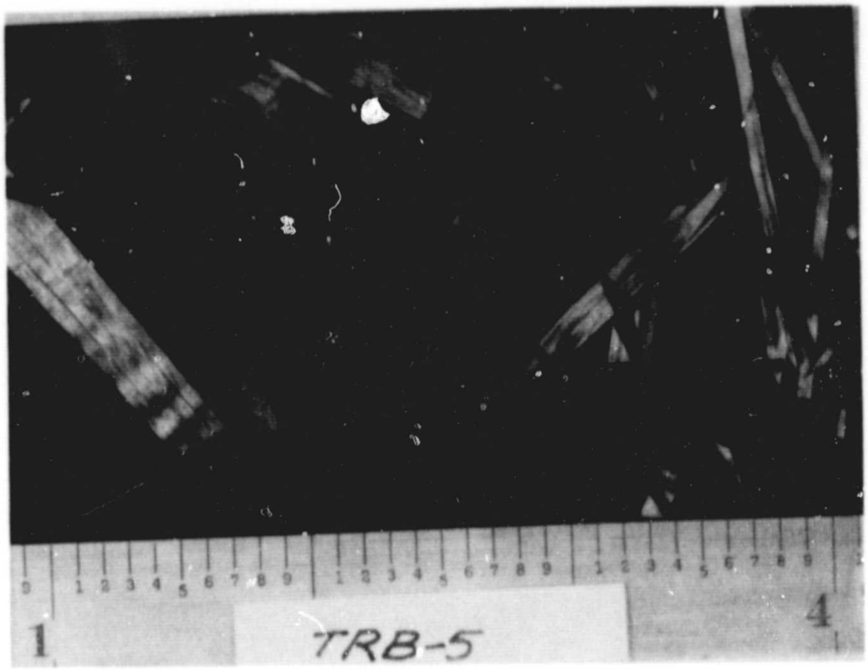

Figure 26 Post Test - Thin Ref

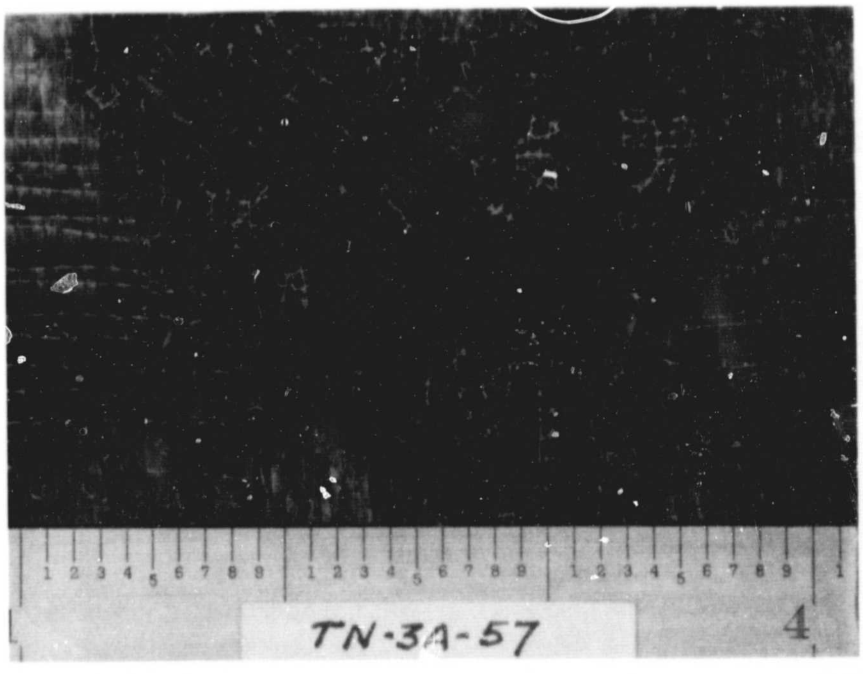

Figure25 Post Test - Thin 3A

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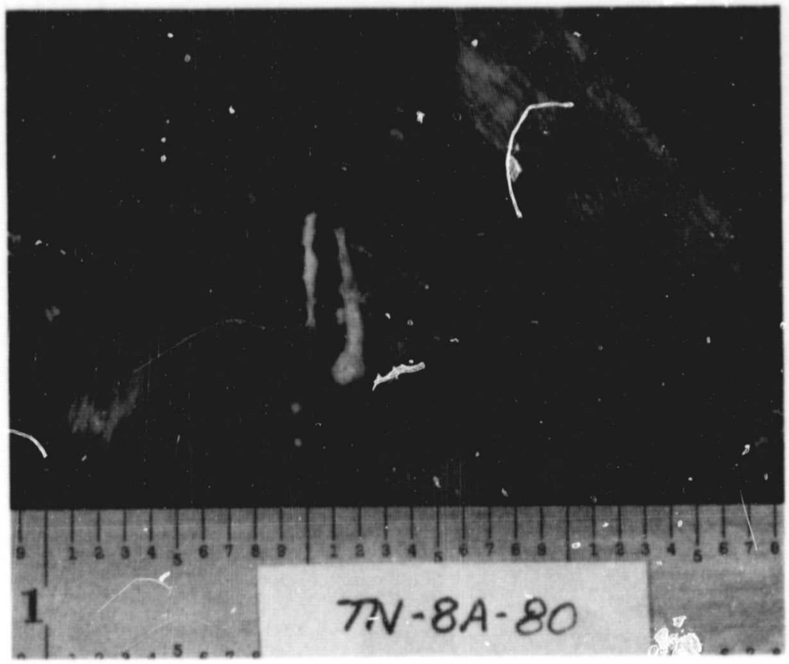


Figure 28 Post Test - Thin 8A

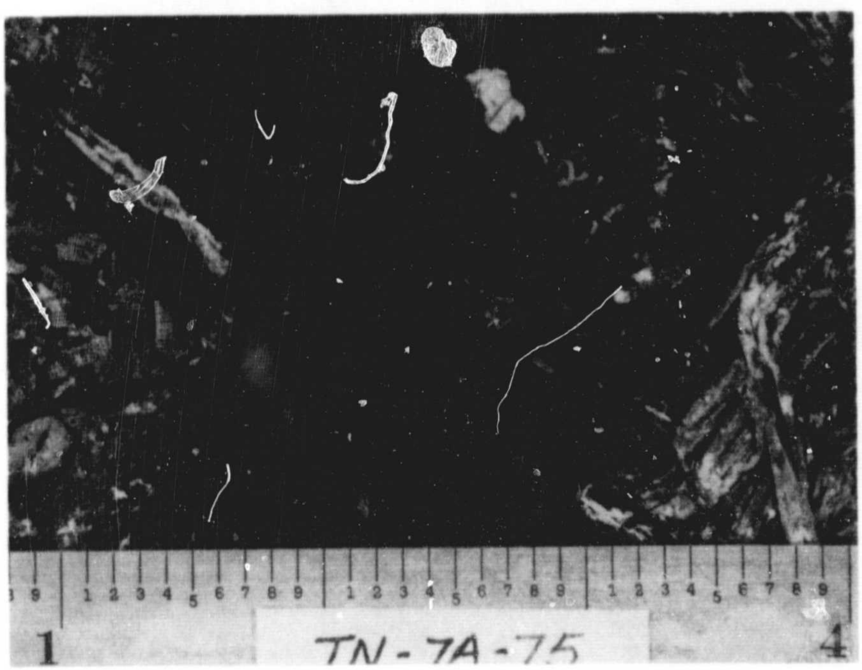


Figure 27 Post Test - Thin 7A

TABLE 7

PRELIMINARY FIRE TEST CONDITIONS

Test No.	Concept Defin.	Initial Spec. Weight (gm)	Post Test Weight (gm)	Collected Fiber Weight (gm)	QG Gas Flow (SCMH)	V_D Duct Velocity (ft.min.)	Q_D Duct Air Flow (SCMH)	Test Time (min.)	Specimen Orientation (Note 1)	Gas Temp. (° F)	Hood Temp. (° F)
45	(Prelim). for Thin Ref. A	12.1	3.6	.027	5.38	450	105.3	5	V	1450	1242
46	(Prelim). for Thin Ref. A	11.5	3.4	.022	5.66	450	105.3	4½	H	1377	1242
48	(Prelim). for Thick 5B	21.4	9.4	.038	5.66	500	117.0	5	V	1338	1255
50	(Prelim). for Thick 5B	21.9	8.7	.024	5.66	500	117.0	5	H	1338	1255
47	(Prelim). for Thin 7C	14.0	4.5	.003	5.66	500	117.0	5	V	1339	1255
49	(Prelim). for Thin 7C	14.0	4.6	.002	5.66	500	117.0	5	H	1338	1255

Note 1 - Test Specimen is 11.9 cm long by 6.1 cm wide and is placed in test fixture with the major dimension in the horizontal plane. Specimen is cut from panel so that the surface fibers are vertical (V), or the surface fibers are horizontal (H).

2 - QB (Burner Air) = 7.36 SCMH

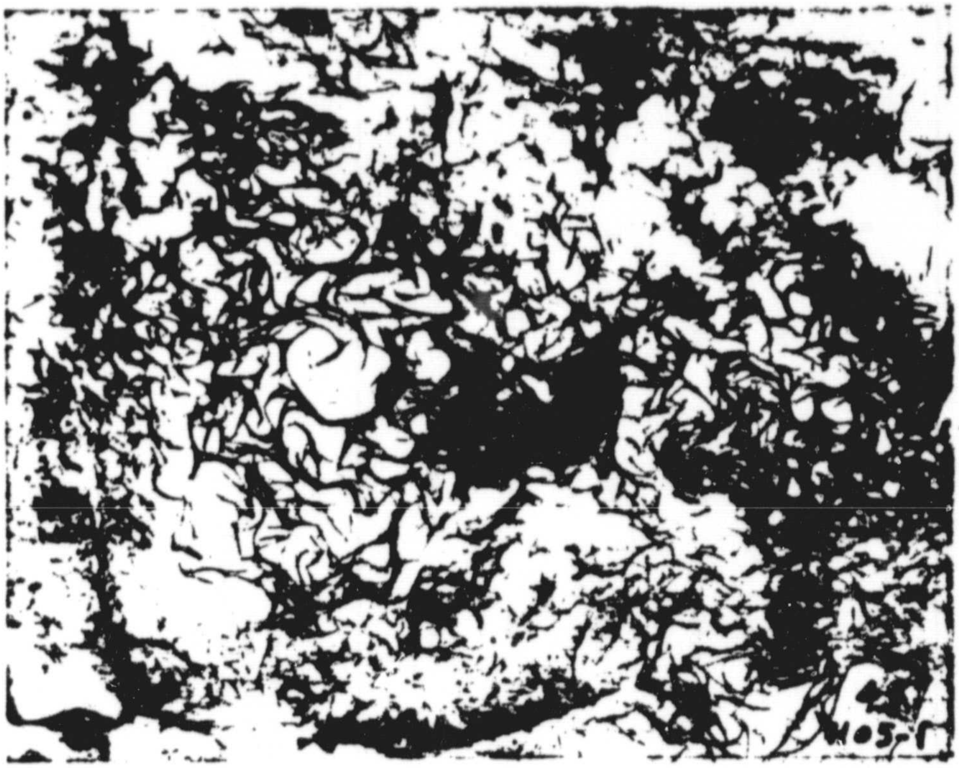
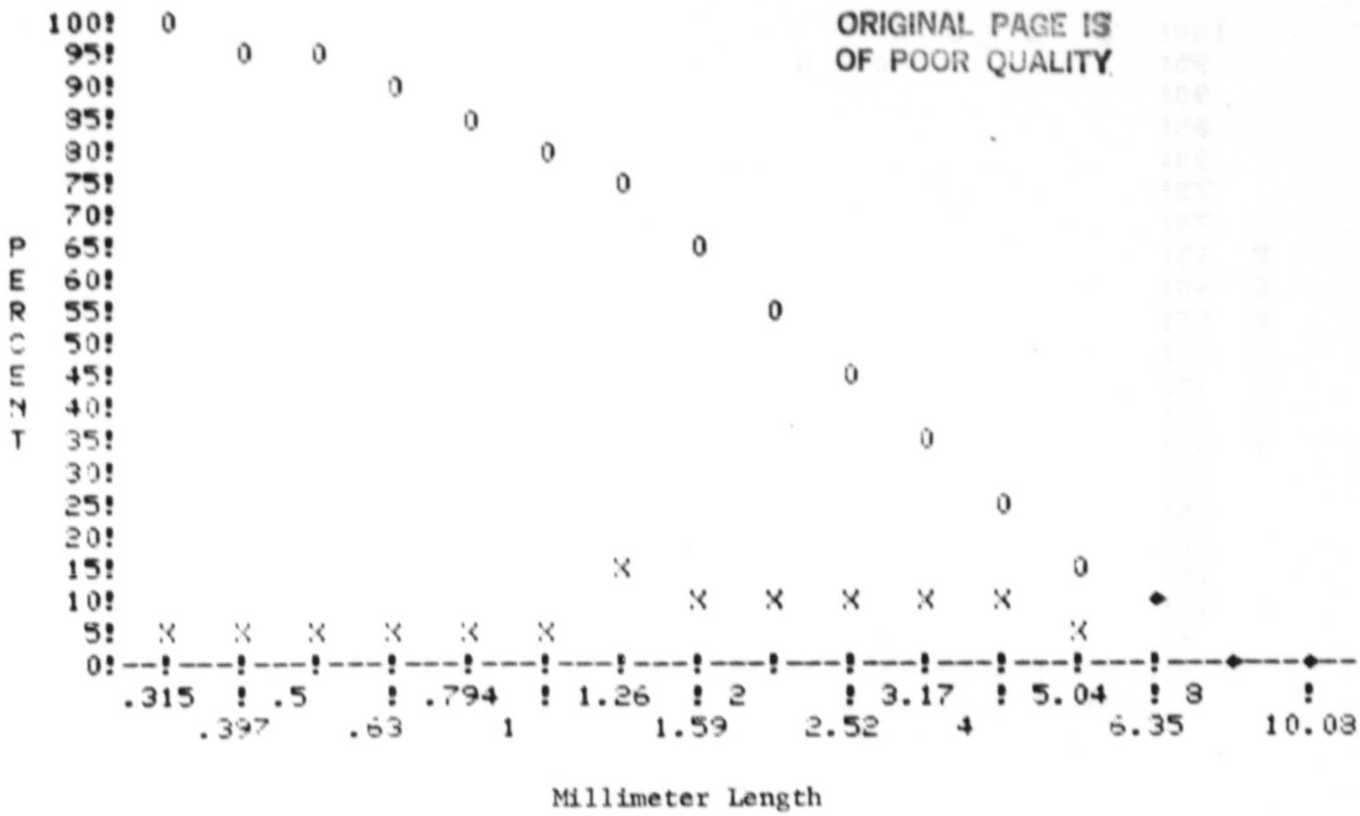


Figure 29 Sample of Residue Collected from Test No. 45

••••• ANALYSIS BY NUMBER •••••

MEAN	MEDIAN	MODE	STANDARD DEVIATION	SKEWNESS
2.57	2.25	1.26	1.91	.98

TOTAL NUMBER : 149 PARTICLES



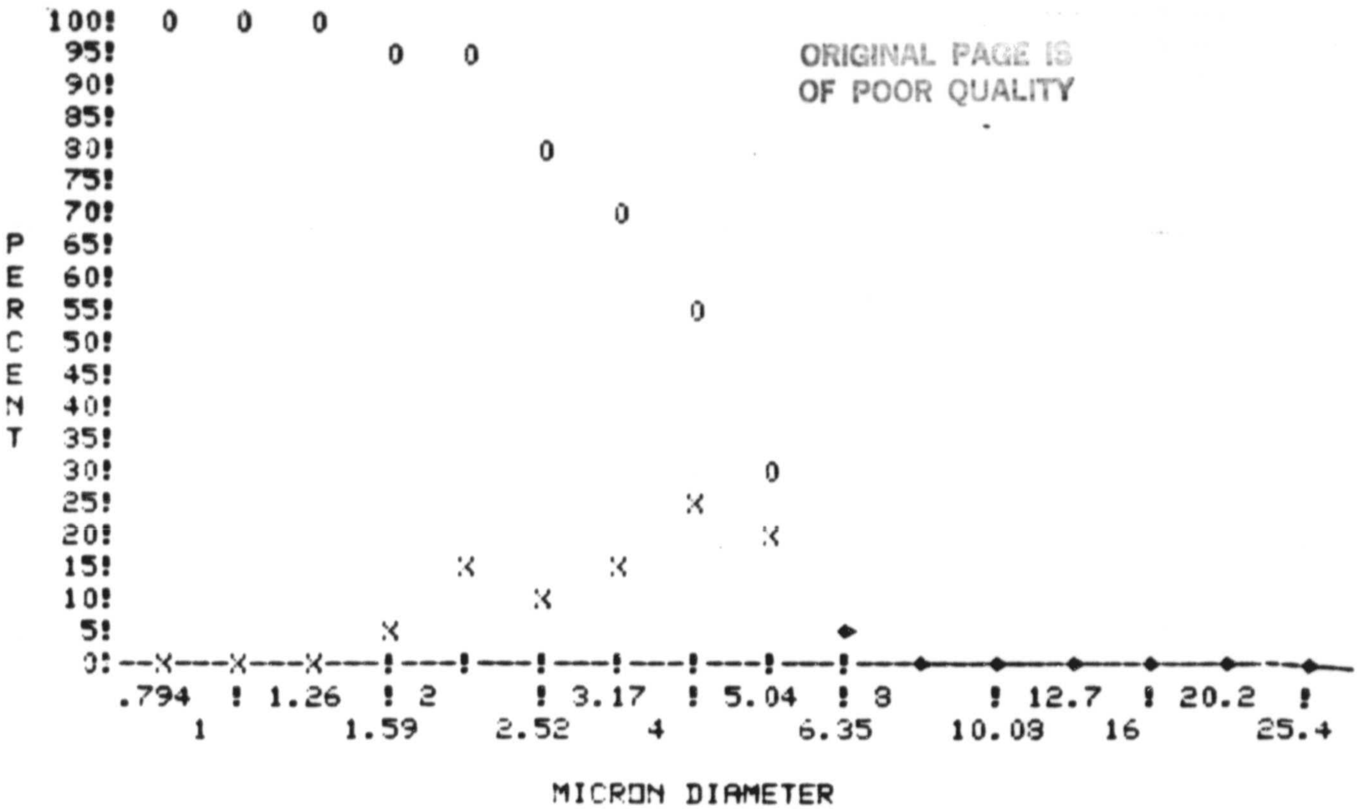
CHANNEL NO.	MILLIMETER LENGTH	NUMBER	FREQUENCY	CUMULATIVE %
1	.315	5	3.4	100
2	.397	4	3	96.6
3	.5	7	4.7	93.7
4	.63	6	3.9	89
5	.794	7	4.6	85.2
6	1	6	3.3	80.6
7	1.26	19	12.5	76.8
8	1.59	14	9.5	64.3
9	2	15	10	54.8
10	2.52	14	9.2	44.8
11	3.17	14	9.4	35.6
12	4	15	10	26.1
13	5.04	8	5.3	16.2
14	6.35	13	8.9	10.8
15	8	3	1.9	1.9
16	10.08	0	0	0

Figure 30 Particle Size Data Reduction Analysis - Length
Test No. 45

◆◆◆◆◆ ANALYSIS BY NUMBER ◆◆◆◆◆

MEAN	MEDIAN	MODE	STANDARD DEVIATION	SKEWNESS
3.63	4.12	4	1.35	.11

TOTAL NUMBER : 150 PARTICLES



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CHANNEL NO.	MICRON DIAMETER	NUMBER	FREQUENCY	CUMULATIVE %
1	.794	1	.6	100
2	1	2	1.1	99.4
3	1.26	2	1.3	98.3
4	1.59	5	3.5	97
5	2	23	15.2	93.5
6	2.52	14	9	78.3
7	3.17	25	16.6	69.3
8	4	36	24.3	52.7
9	5.04	33	22.2	28.4
10	6.35	9	6.2	6.2
11	8	0	0	0
12	10.08	0	0	0
13	12.7	0	0	0
14	16	0	0	0
15	20.2	0	0	0
16	25.4	0	0	0

Figure 31 <u>Particle Size Data Reduction Analysis - Diameter Test No. 45</u>

fiber length with a mean of 2.57 millimeters and a standard deviation of 2.91 millimeters. Figure 31 is a similar presentation of filament diameter, exhibiting a mean of 3.63 microns to be compared to the 23-fabricated diameter of 7-8 microns.

4.2 Mechanical Testing

Flexural tests were conducted on 1.27 cm wide specimens to ASTM standard D790. These were conducted at RT and 344°K on as fabricated material, and on specimens subjected to a 95% relative humidity at 344°K for two weeks minimum or longer as necessary to absorb ½% moisture. Exposed specimens were maintained in the wet condition to within a minute of initiating the 344°K flexural test.

Table 8 presents the detailed test results for the "Thin" hybrid panels where sufficient material was available to achieve the desired span to depth ratio of 32. For the "Thick" panels sufficient material was available only for a span to depth ratio of ten and although the majority of test specimens exhibited the conventional tension side failure mode, the failure stresses were significantly less than observed for the "Thin" specimens. For the "Thin" panels, the RT data shows that there is minimum reduction in strength if fiber volume differences are included in the assessment. The exception appears to be those samples which included resin additives, where a 20% reduction of strength is observed.

Similar degradations are observed in the as-fabricated and environmentally exposed specimens (tested at 344°K) that included a second fiber (glass), all showed reductions in strength--up to approximately 25%. Significant differences in modulus values were determined. The fabric materials, even at lower fiber volumes, showed an increase in stiffness. However, the served uni-material (Thin 3) shows comparable

Table 8 - Flexural Strength and Stiffness of Thin Hybrid Panels (4)

Panel Designation	Specimen Thickness (ins)	Fiber Vol (%)	As Fabricated RT(1) Str (MN/n$_2$)	As Fabricated RT(1) Mod (MN/n$_2$)	As Fabricated RT(1) Str MN/n$_2$)	As Fabricated RT(1) Mod (MN/n$_2$)	Exposed, 344°K(3) Std (MN/n$_2$)	Exposed, 344°K(3) Mod (MN/n$_2$)
TN-R-B	1.04	62	951	59.1	983	54.2	899	54.3
			944	58.6	936	52.3	930	52.6
			972	59.3	941	54.2	958	53.7
TN-1-B	1.37	59	1006	67.2	981	62.3	906	60.2
			896	70.8	920	58.9	931	62.9
			929	69.1	914	58.9	824	59.4
TN-2-B	1.45	53	1032	77.8	903	68.3	887	65.7
			925	74.4	906	67.9	762	65.1
			1078	73.2	864	65.4	748	70.9
TN-3-B	1.24	64	862	51.5	888	49.8	710	45.6
			998	54.8	793	50.5	662	48.8
			872	53.7	807	48.4	727	48.6
TN-4-B	1.11	68	913	50.8	779	47.9	550	48.3
			805	54.5	896	48.9	605	51.2
			892	53.0	938	50.3	696	47
TN-5-B	1.09	63	867	59.5	809	52.6	827	52.6
			910	55.9	793	49.8	802	49.1
			879	57.5	854	52.2	860	49.6
TN-7-B	1.40	48	695	65.9	582	61.3	567	59.7
			578	71.1	545	64.2	524	63.2
			644	74.8	603	66.5	464	62.9
TN-8-B	1.34	49	1122	73.9	1059	63.3	676	60.3
			1115	77.3	847	78.7	693	61.6
			1082	76.05	992	66.3	847	75.7

1) As Fabricated Tested at RT
2) As Fabricated Tested at 344°K
3) Exposed at 344°K at 95% RH for ½% Moisture Pick-up
4) Load Axis Normal to 0° Surface Layer

stiffness to the reference panel.

Interlaminar shear tests were conducted on the thick laminates at a span to depth ratio of 3:1 and the results are summarized in Table 9. Stress levels achieved for the baseline material were approximately two thirds that normally achieved for graphite epoxy. The RT tests identified the "thick"3, 5 and 7 hybrid specimens as having significant reductions in strength (50%, 40% and 40%, respectively) as compared to the baseline.

Reductions in shear strength as a function of elevated temperature (160°F) were significant. However, when exposed to the humidity environment and tested at 160°F, hybrid specimens No. 2 and 4 exhibited reduced strength. The baseline and No. 8 were unaffected and Nos. 3, 5 and 7 retained their low RT strength, as noted above. Evidently the resin additives used in Nos. 2 and 4 are to be considered suspect.

Physical Testing - Table 10 shows the results of tests conducted on sections removed from the test panel for specific gravity and fiber volume. In each case, the test data is considered typical of conventional graphite epoxy laminates, except perhaps for some of the low fiber volumes which can be explained by the peculiar construction of this hybrid material and some lack of accuracy of the "stop" dimensions. Table 11 illustrates the apparent porosity of specimens determined in a 930 Beckman air comparison pycnometer. These data points are suspect because even the reference panels showed an extremely high value.

Table 9

Interlaminar Shear Strength Of Thick Panels (MN/n$_2$)

Panel No.	R.T.	344°K	344°K(1)
TK-R-A	65.5	62.4	59.4
	65.7	64.3	61.5
	64.31	67.9	63.9
TK-1-B	54.8	53.0	47.6
	62.8	51.1	54.8
	58.7	53.9	50.6
	63.5(1)	56.1(1)	53.2(1)
	62.2(1)	59.2(1)	50.4(1)
TK-2-B	52.2	58.1	31.3
	57.3	58.1	48.9
	56.7	59.2	48.1
	59.7(1)	52.8(1)	49.3(1)
	59.1(1)	45.7(1)	48.0(1)
TK-3-A	28.5	32.9	31.8
	31.0	35.4	34.0
	29.4	36.0	33.7
	29.33(1)	36.5(1)	35.3(1)
	28.5(1)	34.7(1)	33.8(1)
TK-4-B	57.8	59.8	36.5
	57.3	58.0	39.3
	54.9	56.8	
TK-5-A	39.9	39.8	32.3
	37.8	40.3	35.4
	38.3	40.6	30.6
TK-7-B	37.3	44.6	36.8
	39.8	43.2	36.1
	36.2	42.7	36.6
	44.0(1)	51.2(1)	45.4(1)
	40.0(1)	51.9(1)	44.3(1)
TI-8-B	47.0	47.5	43.7
	56.2	55.9	55.32
	51.4	48.9	46.2
	54.3(1)	52.0(1)	50.7(1)
	58.0(1)	61.5(1)	59.2(1)

(1) Exposed to 344°K @ 95% RH and Tested at 344°K

TABLE 10 - SPECIFIC GRAVITY AND FIBER VOLUME - THIN SPECIMENS

Panel No	Fiber Volume (%)[1]	Specific Gravity[2]
TN-R	62.12	1.557 1.544 1.563 Avg 1.555
TN-1	59.44	1.531 1.522 1.539 Avg 1.531
TN-2	53.42	1.499 1.495 1.511 Avg 1.502
TN-3	64.36	1.599 1.591 1.579 Avg 1.590
TN-4	68.32	1.545 1.591 1.563 Avg 1.578
TN-7	48.32	1.546 1.561 1.541 Avg 1.549
TN-8	69.15	1.576 1.591 1.595Avg 1.587

1) Determined on flexure test specimens

2) Determined from three different areas of 23 cm square test panel

TABLE 10 - SPECIFIC GRAVITY AND FIBER VOLUME - THICK SPECIMENS - CONT'D.

Panel No	Fiber Volume (%)(1)	Specific Gravity(2)
TK-R-A	59.88	1.557 1.544 1.563 Avg 1.555
TK-1-B	64.39(3)	1.561 1.559 1.554 Avg 1.558
TK-2-A	66.03	1.616 1.626 1.611 Avg 1.618
TK-3-A	57.81	1.466 1.487 1.467 Avg 1.473
TK-4-B	55.47	1.621 1.625 1.621 Avg 1.622
TK-5-A	67.72	1.592 1.595 1.559 Avg 1.582
TK-7-B	67.08(3)	1.530 1.533 1.499 Avg 1.521
TK-8-B	60.92(3)	1.547 1.558 1.518 Avg 1.541

1) Determined on flexure test specimen
2) Determined from three different areas
3) Sample contained small amount of scrim cloth

TABLE 11 - APPARENT POROSITY

Sample No	Apparent Porosity (%)*
TN-R-B	9.20
TN-1-B	9.39
TN-2-B	6.05
TN-3-B	9.97
TN-4-B	7.54
TN-5-B	7.54
TN-7-B	7.03
TN-8-B	5.66
TK-R-A	7.30
TK-1-B	2.06
TK-2-A	6.88
TK-3-A	4.95
TK-4-B	10.70
TK-5-A	9.03
TK-7-B	5.69
TK-8-B	3.88

* Determinations made with a mode 930 Beckman air comparison pycnometer

5.0 Material Delivery

The following hybrid laminates were fabricated for delivery to NASA for further testing. All panels were 8 inches square.

Hybrid Description	No. of Plys	Reason for Selection
Thick 1	(20)&(4)	Least costly material and high potential use in civil aircraft
Thick 2	(20)	Minimal material modification/ high performance in fire test
Thick 3	(20)	Excellent fire test performance
Thin 3	(40)&(8)	Good fire test performance
Thin 8	(40)&(8)	Possible high fire resistance in thick material then tested here

6.0 Conclusions

The "fire" performance of the panels tested were excellent although statistical data is not available to quantify the results. The "thick" panels provided the most satisfactory evidence of success where most of the concepts illustrated a marked improvement over the baseline graphite/epoxy laminate. Compared to the baseline panel, which delaminated severely, the concept using fabric performed very well with very minimal delaminations. Glass serving on the graphite maintained the tow integrity which by itself was a significant improvement. However, a synergistic effect was evident when the served tow was woven into fabric. Here, total laminate integrity was maintained. Other additives such as the siloxane resins demonstrated some beneficial coalescing effects, however the interleaving of glass veils and scrim cloth were less satisfactory. "Thin" panel test results exhibited severe damage due to the severity of the test, however, there was some evidence that the glass fibers had melted and served to prevent release of the graphite fibers.