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NASA CONTRACTOR REPORT 17 **73** 97

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FABRICATION AND TESTING OF FIRE RESISTANT GRAPHITE COMPOSITE PANELS

W.D. ROPER

(NASA-CR-177397) EABRICATIGN AND TESTING OF N88- 12472 ElRE KESlSTBlT GfiLPHIlE CCEFCSITE PANELS (Hercules AerczFace CO-) 57 F CSCL OK

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NASA CONTRACTOR REPORT

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FABRICATION AND TESTING OF FIRE RESISTANT GRAPHITE COMPOSITE PANELS

W.D.ROPER HER CULES A EROSPACE COMPANY **BACCHU S WORKS MAGNA, UTAH, 84 04 4** - 0 098

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Space Administration

Ames Research Center Moffett Field. California 94035 **CONTENTS**

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Appendix

SUMMARY

This program was conducted to fabricate and supply NASA-Ames Research Center with a number of graphite fiber composite honeycomb panels which were prepared using several different resin prepreg constructions. It was hoped that these constructions would exhibit better fire resistance as compared with baseline (epoxy/fiberglass) panels. The tasks within the program were:

- **Fabrication of eight (8) selected panel constructions.**
- \bullet **Mechanical Testing of the selected panel constructions.**
- **Flammability testing of the selected panel constructions.**

The above Tasks were completed and the panels were supplied to NASA-Ames according to the specified delivery. This report includes the procedures used in making all the panels and the mechanical and flammability test results.

The work of this report showed that the best graphite fiber panel construction was one prepared from a resin fomulation of a modified vinylpolystyrylpyridine (VPSP) and bismaleimide (BWI) resin. This conclusion was based on the panel fire resistance and ease of fabrication. The panel fire resistance was also better than the baseline epoxy/fiberglass standard.

INTRODUCTIOU

Improvement in aircraft safety has been a strong concern within governmental agencies and those associated with the aircraft industry. This is particularly true of the incidence of interior aircraft fires and their associated toxic gas generation. The goal has been to improve the chances of passenger survival during an interior fire. Currently the Federal Aviation Administration has specifications pending for the improvement of the fire resistance of aircraft materials - **in particular, the interior paneling. This reported work was undertaken to support the materials development which is necessary for any specification change. The work was conducted by Hercules Inc., Aerospace Division for NASA-Ames Research Center under NASA contract NAS2-12039.**

The primary objective of this program was to fabricate and provide NASA-Ames with graphite composite panels constructed using several different high temperature resin systems. These resin systems were selected by UASA as the best candidates for reducing the fire hazard in aircraft paneling. These candidates should exhibit slower burning, reduced smoke and toxic gas emission, and less heat release during fire exposure. Since the panels were of graphite fiber construction, a secondary objective was their decided weight advantage over current fiberglass panels.

The specific tasks to be performed in this program included:

- **1. The fabrication of eight (8) panel constructions as outlined in Table 1.**
- **2. The mechanical testing of the specified panels.**
- **3. The flammability testing of the specified panels.**

PANEL MATERIALS

As shown in Table 1, all eight specified panels used a Womex/phenolic honeycomb core. All panel facesheets used Hercules AS4 graphite as either unidirectional tape or plain weave fabric. All graphite prepregs were prepared with a 42 3 percent resin content. Appendix A of this report contains a discussion of the development work performed for the preparation of the graphite prepregs for panels A, B, C, and D. After each panel was cured a 0.007 cm. (0.003 in.) film of polyetheretherketone (PEEK) was bonded to the panel facesheet. A pressure sensitive silicone adhesive (X3-5815) was used in this bonding. A listing of the materials used and their suppliers is shown in Table 2.

PANEL FABRICATIOW

Detailed procedures for each of the prepared panels are given in Appendix B of this report. Since some resin prepregs required high temperature cures and post cures (above 177°C., 350°F) these prepregs were first cured **as facesheets and then bonded to the lomex core in a second step. One step layup and bonding (co-cure) could not be used in these cases due to the temperature limitation of Nomex core. A co-cure was used in panel constructions not requiring high temperature curing.**

In the panel constructions using unidirectional graphite tape, all facesheets were layed up with 3 plies using a Oo, 90°, Oo, configuration. This was done to prevent possible ply distortion. All panel curing was performed within an autoclave.

For the PEEK film bonding, several adhesives were tried. These included: BR-34 (American Cyanamid), Chemlon 701 (Chem-Tronic Inc.) and 68070 adhesive (DuPont). None of these adhesives were found suitable for bonding of the PEEK. The most satisfactory PEEK adhesive was a new silicone pressure sensitive adhesive, X3-5815, manufactured by Dow Coning. This adhesive provided good PEEK bonding. In initial development of the PEEK bond it was thought that prior plasma treatment of the PEEK would enhance the PEEK bond. This was somewhat true in the initial trials which used the rigid adhesives (e.g. BR-34). However it was ultimately found that plasma treating of the PEEK was unnecessary with the Dow Corning X3-5815 adhesive.

TABLE 1

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PANEL CONSTRUCTIONS

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PANEL CONSTRUCTIONS

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TABLE 2

MATERIAL SOURCES

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PANELS A and B

At the start of this program it was planned that vinylpolystyryl pyridine (VPSP) resin from American Cyanamid would be used for the construction of these panels. However, the resin supplier had dif€iculty in supplying a suitable VPSP. VPSP was therefore substituted with a new Dow Chemical Co. resin, designated as XU-71775.01L (see appendix under "Prepreg Development **and Preparation"). This Dow resin was formulated at Hercules with the bismaleimide (BWI) resin Compimide H795. A glass transition temperature** of 193°C. (380°F.) was achieved with this formulation. Prepreg tape of **this formulation was produced by hot melt in both 7.6 cm. and 30 cm.** (**3 in., 12 in.) widths. The fabric prepreg was prepared in solvent coating equipment in 106 cm. (42 in.) widths. All prepregging was done at Hercules using production equipment. The prepregs performed very well during panel fabrication and a co-cure (no film adhesive) bonding technique was used.**

PANELS C AND D

Prepreg for these panel constructions, both fabric and tape, was manufactured on Hercules production equipment. Since the Compimide H795 required a 232°C. (450°F.) post cure, a co-cure was not possible with **these panels. The cured facesheets were bonded to the honeycomb core as a** second operation using the polyimide film adhesive FM-34 which was **specified by NASA-Ames.**

Satisfactory sandwich bonding of these panels proved to be very difficult. In initial attempts at making the tape panel D, at a 122 cm x 244 cm size (48 in. x 96 in.), the cured panels showed facesheet to core delamination at the panel center. This delamination problem did not occur with the fabrication of large fabric panels of type C construction. The cause of the delamination was ultimately traced to the high volatile content of the Fn-34 film adhesive. Considerable effort was expended in trying to overcome this problem with the type D panel. Limited success was obtained in making D panels when panel size was made smaller (approximately 60 cm. x 60 cm., 24 in. x 24 in.). A perforated core would have probably avoided this problem, however, the specified HRH-10 core is **not available with perforations. Despite several fabrication attempts, sufficient D type panel material could not be obtained for all the planned testing** .

PANELS E IWD F

Prepreg for these panels was prepregged by American Cyanamid on Hercules graphite fabric (A193P) and tape (AS4). These panels were prepared using a co-cure without film adhesive. The cure cycle used was specified by NASA-Ames. The panels fabricated readily without any problem.

PANELS G AND H

Prepreg for these panels was made by Composites Horizons Inc., Covina, CA. The prepregging was on Hercules graphite fabric (A193P) and tape (AS4). The cure cycle used €or preparing the panel facesheets was that specified by Composites Horizons Inc. The resin system required a 250°C. (482°F.) cure temperature so a co-cure was not possible. FM-34 film adhesive was used for the final sandwich bonding.

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The prepreg tape that was supplied for these panels was difficult to layup due to its lack of tack. This was overcome by methylene chloride misting of the tape prior to layup. The Fn-34 volatility was again a problem with these panels as previously discussed with panels C and D. Panel type G (fabric) could be prepared satisfactorily, however, panel type H (tape) consistantly showed delamination after several fabrication attempts. As with the D panel construction, the delamination occurred between the core and facesheet and was prodominant at the center of the panel. There was insufficient panel H material to perform all the planned testing of this panel construction.

MECHANICAL TESTING

The panels of Table 1 (excluding panel H, for the reasons previously discussed) were given the following testing:

1. Sandwich Panel Peel

The panels were tested according to the climbing drum method outlined in MIL-STD-401 at a cross head speed of 2.54 cm./min. (1 in./min.). Peel strength was determined by peeling the PEEK film side away from the honeycomb core of the panel. The 30.5 cm. (12 in.) specimen dimension was prepared parallel to the fabric warp direction.

2. Flatwise Tensile Strength

This test was conducted in accordance with HIL-STD-401.

3. Flexural Strength

This test was conducted in accordance with MIL-STD-401 with the specimens prepared with the 60.96 cm. (24 in.) dimension parallel to the core ribbon direction.

4. Panel Density

Panel density was determined from weight and volume measurements which included the weight and thickness of the PEEK decorative film.

FLAHHABILZTY TESTING

The following flammability tests were performed on the panel constructions of Table 1 plus an epoxy-fiberglass baseline panel supplied by NASA-Ames. For the reasons previously discussed, panels D and H could not be given all of these listed tests.

1. 60-Second Vertical Flammability Test

The tests were performed according to: Federal Aviation Regulation, Part 25, Amendment 25-32, paragraph 25-853, "Fire Protection Compartment Interiors" Hay 1, 1972.

2. Limiting Oxygen Index Test

This test was performed according to ASTH D 2863, "Flanunability of Plastics Using the Oxygen Index Method."

3. Flame Spread Test

This test was performed according to ASTH E 162, "Surface Flammability of Haterials Using a Radiant Heat Source". The test was performed with the panel PEEK film side facing the radiant heat source.

4. Smoke Density Test

This test was performed according to ASTM E 662, "Test Hethod For Specific Optical Density of Smoke Generated By Solid Materials." The test was conducted at 2.5 w/sq cm under flaming and nonflaming conditions. Under flaming conditions, analysis for HF, CO, HCN, UO, plus smoke density was obtained. The PEEK film side was placed facing the radiant heat source.

5. Heat Release Test

This test was performed according to the Ohio State University Heat and Smoke Release Test (ASTH E 906). The tests were conducted at 3.5 w/sq cm, with specimen mounted vertical, with piloted ignition, and with PEEK film side facing the heat source.

DISCUSSION OF TEST RESULTS

Detailed results of all testing are given in the appendix of this report.

TABLE 3

RELATIVE RANKING OF **PANEL CONSTRUCTIONS BASED ON** FLAHHABILITY **TESTS**

OHIO STATE CALORIMETER TEST *ASTU* **E-162**

Table 3 shows a relative ranking of the panel constructions based on three significant flanunability tests. This ranking was made taking into consideration the data spread that was obtained in these tests. It appears from this data that constructions A, B, and H were the better constructions from a flammability testing standpoint. Constructions A and B used the XU-71775/H795 formulation (fabric and tape) in the panel facesheet construction. Construction H used the PSP (polystyrylpyridine) resin system with a graphite tape facesheet.

It should be noted that all of graphite composite panels had a greatly reduced smoke emission as compared with the baseline (epoxy/fiberglass) panel **that was tested for comparison. Heat release was, in most cases, lower with the graphite composite panels as compared with the baseline panels. Panel construction A showed the lowest average heat release (73 kw. min./m2** @ **3 minutes). Flame spread for A, B, C and baseline panels was essentially equivalent. Unfortuantely, flame spread tests on H panel were not performed due to lack of test material.**

All graphite composite panels exhibited limiting oxygen indices significantly higher than the baseline panel. This should indicate that the graphite panels will exhibit lower relative flanunability. Panels A, B, and D showed the highest indices (44.3, 45.6, 45.0 respectively) and compared to 34.6 for the baseline panel.

For the fabrication of the test panels, the following mechanical property goals were set.

- **Climbing drum peel strength: 1.36 cm.-kg/cm. of width (9 in-lb./3** \bullet **in. of width).**
- **Flatwise tensile strength: 10.5 kg./sq. cm. (150 lb./sq. in.).**

Sandwich beam flexure test:

Compressive strength: *50* **kg./cm. (280 lb./in.). Modulus: 1.5 kg./sq. cm. x 104 (21.3 lb./sq. in. x 104).**

All panels met the flexural modulus goal, however, all panel fabric constructions fell below the compressive strength goal. This is attributed to the use of only a single ply in the fabric constructions. The compressive strengths of the tape constructions (3 plies of unidirectional tape) were well above the goal value. Panels C and D did not meet the goal value for flatwise tensile strength. These panels were difficult to fabricate due to adhesive outgassing.

Panels A, B, and G had peel strength values below the goal value. Panels A and B showed values of 0.99 and 0.6 em,-kg./cm. respectively. The reason for this is not apparent. It is felt that some modification in the panel fabrication procedure might result in higher peel strength values. Panel G also showed a low peel values. This may be due to the outgassing problem associated with the panel adhesive. Panel H could not be peel tested because of the panel adhesive outgassing problem.

CONCLUSIONS

- **1. Panels A and B (modified VPSP/BHI) and panel H (PSP), showed the better fire resistance of all the tested panels.**
- **2. From a fabrication standpoint, the panel A and B resin system offer the** advantages of a moderate cure temperature of 177°C. (350°F.) and a simple **one-step fabrication (co-cure) without panel adhesive. The panel H resin** requires a much higher cure and post-cure temperature (204/249°C., **(400/480°F.)). In addition, the H construction requires a separate panel bonding step using film adhesive.**
- **3. A vented honeycomb core is required in any panel fabrication requiring a polyimide type panel adhesive.**
- **4. Although the PEEK adhesive used in the work performed satisfactorily from a mechanical bond standpoint, it is believed that the panel fire resistance may be still further improved with a different adhesive. Further development in this area is needed.**
- *5.* **Adequate ventilation must be provided during handling of the resin systems of this report. This is particularly the case with the PSP resin (H and G type panels). This resin produces very strong irritating and toxic vapors when it is heated prior to cure.**

APPENDIX A

PREPREG DEVELOPMENT AND PREPARATIOU

Prepreg Development and Preparation

The following discussion describes the formulation and testing that was completed prior to the prepreg preparation for panels A, B, C, and D. Prepregs for panels A and B contained a Hercules VPSP/BHI formulation on unidirectional AS4 tape and on A193P fabric, respectively. Prepregs for panels C and D contained Technochemie's BHI resin, Compimide H795, on unidirectional AS4 tape and on A193P fabric, respectively.

Panels A and B

The Hercules proprietary fire-resistant resin formulation was used for panels A and B. This formulation is based on a formulation of a modified vinylpolystyrylpyridine (VPSP) and Technochemie's Compimide H795. Other reactive materials were added to allow hot-melt prepregging of the resin.

The modified VPSP resin was designated XU-71775.01L and was manufactured by Dow Chemical Company. This resin has the same oligomer backbone (polystyrylpyridine) as VPSP, but possesses different reactive end groups. Its odor was comparatively low, though somewhat irritating, and it was an easily handled granular solid. However, the granules would stick together on room temperature storage. Formulations of ambient stored and freezer (-17°C., 0°F.) stored 71775 indicated that 71775 had **some reactivity at ambient temperature and should be stored cold. Its volatile content was 7 percent.**

The 71775 was insoluble in the molten formulation components, as well as in methylene chloride, l,l,l-trichloroethylene, acetone, and methyl ethyl ketone. Thus, the formulation required roll milling to finely disperse the 71775 for prepregging. Mixing at lower temperature showed that the formulation must reach at least 75'C. (167'F.) to completely melt and disperse the Compimide H795.

A prepreg sample of the formulation on A193P was prepared by hand dipping the fabric into a suspension of the formulation in methyl ethyl ketone. This sample was sent to NASA-Ames for preliminary wet-out and flammability tests.

A number of tests, listed below, were performed on the formulation.

<u>DSC:</u> Major exotherms occurred at 209°C. (408°F.) and at 339°C.

(642 **tests** .

A number of tests, listed below, were performed on the formulation.

- **(642'F.).**
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<u>DSC:</u>
<u>TGA:</u> 5% volatiles loss occurred by 230°C. (446°F.) major decomposition occurred at 350°C. (662°F.), char yield was **48% at 700°C. (1292-F.).**
- **Gel Curve by Rheometrics: The following viscosities were observed:**

800,000 poise at 25°C. (75'F.I 1170 poise at 50°C. (122'F.I 6.2 poise at 100'C. (212°F.) 2.7 poise (minimum) at 127'C. (261OF.)

- Gel Times: **5** minutes at **177°C. (350°F.) 18** minutes, **40** second at **140°C. (300°F.)** > **1** hour, **15** minutes at **121OC. (250°F.)**
- Exotherm Tests: Exothermed in 10 hours at 80°C. (176°F.); in **3** hours at **100°C. (212°F.)**
- Solubilities: Soluble in tetrahydrofuran and dimethylformamide, dispersible in methyl ethyl ketone.

An attempt was made to make **7.6** cm **(3** in.) prepreg tape of this formulation on **AS4W-12K** without roll-milling. Prepregging was very difficult due to the large particle size of the **71775.** Roll-milling of the hot resin solved this problem and good tape was produced. A quantity of this tape was sent to NASA-Ames for evaluation. The same tape lot was used to layup panel type **B.**

^A15 ply, **7.6** cm. **x 25** em. **(3** in. **x 10** in.) panel was cured for **2** hours at **177'C. (350'F.).** Samples measuring **1.3** cm. x **6.4** cm. **(0.5** in. x *2.5* in.) were cut and given various post cures. The samples were then tested for TAG' by Rheometrics. From this study and from results of testing **by** NASA-Ames, the **following** cure/post cure schedule was developed.

The pre-cure step was found by NASA-Ames to aid in flow control on fabric prepreg. A TAG' of 193°C. (380°F.) was ultimately reached. Higher temperature post-cures (up to 232°C., 450°F.) produced the same TAG'.

Sixteen kilograms of the formulation were mixed. This was roll-milled in four batches for hot-melt prepregging onto **A193P** fabric using Hercules production equipment - **¹⁰⁷**cm. **(42** in.) wide prepreg was produced. The resin ran very well in the prepregger and the odors from the hot resin were only slightly objectionable. Samples of this prepreg were sent to NASA-Ames for evaluation. The same fabric prepreg lot was used to layup panel type **A** construction.

Prepregs for Panels **C** and **D**

H795 is a brittle, glassy solid which becomes tacky and flexible above 50°C. (122°F.). In order to obtain a room temperature drapeable and tacky prepreg, the resin requires formulation with some liquid diluent preferably a reactive diluent to avoid losing mechanical properties at elevated temperatures. Thus, **H795** was mixed at various percentages, with several reactive, unsaturated and fairly high boiling liquid monomers. The mixtures were tested for flexibility and tackiness at room temperature by pressing with a wooden spatula. In this way a **H79S** formulation possessing suitable tack and drape properties was found. Several tests

(i.e., - Differential Scanning Calorimetry, gel time at 177°C. (350°F.), **gel curve by Rheometrics) showed it to be fairly reactive. However, exotherm tests indicated it to be resistant to runaway exotherming at prepregging temperatures.**

This formulation was soluble to at least 70% w/w in methyl ethyl ketone. Hand-made fabric prepregs (A193P) from this solvent were successfully made. Resin contents were determined to be 68% and 56%. The prepreg lost its tackiness on standing at room temperature, probably due to increased reactivity of the reactive diluents.

In spite of these indications of reactivity, and in view of the reassuring exothetm tests, 7.6 cm. (3 in.) tape prepregging was attempted. The first 4000 g. batch gelled in the hopper after 60 meters (200 ft.) of tape had been made, and the second batch gelled during mixing. Neither batch exothermed.

Fabric prepregging of this formulation using the solvent coater (methyl ethyl ketone as solvent) was also unsuccessful. The machine malfunctioned and only 12 meters (40 ft.) of prepreg were produced.

It was decided to test free radical inhibitors to reduce the reactivity of this formulation. Thus, a selection of inhibitors were added to the formulation at several levels. The resulting formulation possessed physical and reactivity properties (listed below) which were suitable for hot-melt and solvent based prepregging.

Differential Scanning Calorimetry (DSC):

Small exotherm (4 J./g.) at 105' - **155"C., (221** - **311'F.I with major** exotherm (220-250 J./g.) at 214°C. (417°F.).

Thetmogravimetric Analysis (TGA):

About 5% weight loss in volatiles up to 160°C., (320°F.) with major **decomposition occuring at 415'C. (779'F.I. Char yield of** *55%* **at S2O'C. (968'F.I.**

Gel Curve by Rheometrics:

Viscosity at 78°C. (172°F.) of 1200 poise, minimum viscosity of 4.2 poise at 130'C. (266'F.I.

Gel Time at 177°C. (350°F.):

8 to 9 minutes

Ex0 therm Test :

Exotherm started at about 10 hours at 100°C. (212°F.).

Solubilities:

Soluble in acetone, methyl ethyl ketone, methylene chloride; insoluble in l,l,l-trichloroethane.

This inhibited formulation was successfully hot melt prepregged to yield 7.6 cm. (3 in.) tape. This prepreg was used for panel D. A 15 ply 7.6 cm. x 25 cm. (3 in. x 10 in.) panel was given a cure of 2 hours at 177OC. (3SOOF.). Pieces, 1.3 cm. x 6.4 cm. (0.5 in. x 2.5 in.), were cut from the cured panel and given various post cures. The TAG' (the temperature at which the composite modulus drastically drops) was over 300°C. (572°F.) with an autoclave cure of two hours at 177°C. (350°F.) and a post cure of one hour at 204°C. (400°F.) and four hours at 232°C. (450°F.).

Fabric prepreg on A193P was then successfully made using a solvent coater with the resin dissolved at 65% w/w in acetone. This prepreg was used €or panel C.

APPENDIX B

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FABRICATION PROCEDURES

FOR COMPOSITE PANELS

1.0 SCOPE

This process document covers materials and procedures for the fabrication of UASA-Ames Panel Types A, B, C, D, E, F, G, and H shown in Table 1.

- 2.0 APPLICABLE DOCUMENTS
- 2.1 **MILITARY DOCUMENTS**

MIL-STD-401 Sandwich constructions and core materials; general test methods

3.0 REQUIREMENTS

This process document establishes the fabrication techniques for manufacture of NASA-Ames panel types A, B, C, D, E, F, G, and H, fire resistant aircraft interior paneling. Included are materials, tooling, equipment, and fabrication procedures.

- **4 .O MATERIALS**
- **4.1 PRODUCTIVE MATERIALS**

Productive materials are those materials incorporated into the product during fabrication and shall be .limited to those described below:

DESCRIPTIOU

SOURCE

- **A. NOMEX HONEYCOMB TYPE HRH-10-1/8-3.0 BEXCEL CORP.**
6.35 mm (0.25 in.) THICK 6.35 LONG BEACH, CA. **6.35 mm (0.25 in.) THICK**
- B. XU71775/H795/A193P GRAPHITE PREPREG **HERCULES INC.**
(PANEL TYPE A) **HAGNA**, UT (PANEL TYPE A)
- C. XU71775/H795/AS4 GRAPHITE PREPREG **HERCULES INC.**
(PANEL TYPE B) **HAGNA**, UT (PANEL TYPE B)
- **D. H79S/A193P GRAPHITE PREPREG (PAUEL TYPE C)**
- **E. H795IAS4 GRAPHITE PREPREG (PAUEL TYPE D)**
- **F. CYCOM 6162/A193P GRAPHITE PREPREG AMERICAN CYANAMID (PAUEL TYPE E) CHARLOTTE, NC**
- **G. CYCOM 616 /AS4 GRAPHITE PREPREG** . **AMERICAN CYANAMID** (PANEL TYPE F) CHARLOTTE, NC

HERCULES INC. UAGUA, UT

HERCULES INC. UAGUA, UT

- **H. PSP 6022MIA193P GRAPHITE PREPREG (PANEL TYPE G)**
- **I. PSP 6022MIAS4 GRAPHITE PREPREG (PANEL TYPE H)**
- **J. FM 34-B-32 ADHESIVE FILM (PANEL TYPE C, D, G, AND H)**
- **K. X3-5815 ADHESIVE**

L. PEEK FILM

COMPOSITES HORIZONS INC COVINA, CA

COMPOSITES HORIZONS INC COVIUA, CA

AMERICAN CYANAMID HAVRE DEGRACE, HD

DOW CORNING MIDLAND, MI

Commercial

REDLANDS, CA

SANTA FE SPRINGS, CA

WESTLAKE PLASTICS LENNI, PA

4.2 NON-PRODUCTIVE HATERIALS

Non-productive materials are those materials not incorporated into the product, but are typical of those used and consumed during the fabrication process.

B. BLEEDERS AND BREATHERS 1. Airweave N10 **AIRTECH IIC. CARSON CITY, CA**

2. Hochburg

- **3. Glass Cloth, Style 120**
- **C. VACUUH BAGGING MATERIALS** 1. Vac-Pac HS-8171 RICHMOND
	- **2. Kapton (High Performance Bag)**
	- SM 5126-2 Vacuum Sealant SCHNEE-MORIHEAD INC.

D. MISCELLANEOUS MATERIALS

-
- 1. Silicon Daming
2. High Temperature Tape **Commercial** Commercial 2. High Temperature Tape **Commercial**
3. Methylene Chloride **Commercial**
- **3.** Methylene Chloride **Commercial**
4. 0.64 cm. (0.25 in.) Aluminum Caul Commercial
- 0.64 cm. (0.25 in.) Aluminum Caul Commercial **Plate**

5.0 EQUIPMENT AND FACILITIES

5.1 OVEN

A circulating air batch oven capable of controlling temperatures up to 522K. (480"F., 249°C.) is required for processing of the honeycomb core, postcuring of the skins, postcuring of sandwichs, and curing of adhesive for bonding of the decorative peek film. The oven must be equipped with a suction fan to vent the exhaust gases to the outside atmosphere.

5.2 AUTOCLAVE

An autoclave capable of the following minimum temperatures and pressures are required for cure of grahite sandwiches and/or graphite skins; 450 K. (350°F., 177°C.) and pressure of 172 KPa (25 psi), for panel types A and B; 450K. (350°F., 177OC.) and pressure of 690 KPa (100 psi), for panel types C and D; 405 K. (27OoF., 132°C.) and pressure of 172 KPa (25 psi) for panel types E and F; 478 K. (400°F.. 204°C.) and pressure 1034 KPa (150 psi) for panel types G and H.

5.3 TOOLS

^Aflat aluminum or graphite platen is required to layup graphite skins and sandwich construction. This platen is used in the cure cycle of the panels.

6 **.O MATERIALS STORAGE AND HAMDLING**

All graphite prepreg and Fn 34B film adhesive shall be stored at or below 255 K. (0°F, -18°C.). Those materials shall be allowed to warm at **room temperature in their sealed containers prior to removal to prevent moisture condensation. Honeycomb shall be stored flat in its original shipping container. X3-5815 adhesive will be stored in a non-flaxmnable cabinet when not in use.**

7.0 FABRICATION OF PRECURED GRAPHITE SKINS, PANEL TYPES C, D, G, AND H

- **A. Allow graphite prepreg to warn to room temperature prior to unspooling.**
- **B. Cut patterns for the following and layup on an aluminum or graphite platen.**

Type C and D panels: 1 mil Teflon Film TX 1040 1 ply Type C Graphite Prepreg 3 ply Type D Graphite Prepreg Tx 1040 1 mil Teflon Film 114" Aluminum Caul Plate Airweave N10

Type G and H panels:

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and and

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> **1 mil Teflon Film TX 1040 1 ply Type G Graphite Prepreg 3 ply Type H Graphite Prepreg TX 1040 Glass Cloth, Style 120 1 mil Teflon Film 114" Aluminum Caul Plate** Airweave N10

- **C. Bag layup for autoclave cure. For panel Type's C and D use Vac-Pac HS-8171 film and vacuum sealant. For panel Type's G and H use kapton film and vacuum sealant.**
- **D. Autoclave cure as follows:**

Type C and D Panels:

Apply vacuum 584 rma **Xercury (23 in. Mercury). Raise pressure to 690 KPa (100 psi) and temperature to 450°K. (350"F., 177°C.) at a rate of 1.5 K./min. (3"F./min., l.S'C./min.). Hold temperature, pressure, and vacuum for 2 hours. Cool to 339 K. (lSO"F., 66°C.) under pressure and vacuum.**

Type G and H Panels:

Raise temperature to 450K. (350°F., 177°C.) at a rate of 1.5 **K./min. (3"F./min., l.S'C./min.).** Hold 450 K. (350°F., 177°C.) for 1 hour. **Raise temperature to 478 K. (400°F.. 204°C.) at a rate of 1.5 K./min. (3'F./min., l.S"C./min.) dwell 15 minutes. Apply vacuum, 584 naa Mercury (23 in. Mercury), dwell 10 minutes. Apply 1034 Kea (150 psi) at a rate of 69 KPa/min. (10 psi'min.).** Vent vacuum at 139 KPa (20 psi). **Hold 478 K. (400"F., 204°C.) and 1034 KPa.(150 psi) for 9 hours. Cool to 339 K. (150"F., 66°C.) under pressure.**

E. Debag cured skin stock. Place skin stock (stack same size skins) on aluminum or graphite platen and cover skin stock with **0.64** cm. **(0.25** in.) aluminum caul plate. Place platen in a circulating air oven and post cure as follows:

Type **C** and D Panels:

Raise temperature rapidly to **477 K. (400°F.. 204°C.).** Hold **477 K. (400°F., 204°C.)** for **1** hour. Raise temperature to *505* **K. (450"F., 232°C.)** at **1.5** K./min. **(3"F./min., l.S'C./min.).** Hold **505 K. (450°F., 232°C.)** for **4** hours. **Cool** to **339 K. (150°F., 66°C.).**

Type *G* and H Panels:

Raise temperature to **522 K. (480°F., 249°C.).** Hold **522 K. (480°F., 249°C.)** for **2** hours. **Cool** to **339 K. (150°F.. 66°C.).**

F. Store precured **skin** stock flat until **ready** for use in the sandwich bonding operations.

7.1 FABRICATION OF CORE STOCK

- A. Cut the 6.3 mm (0.25 in.) thick Nomex core into the required sizes (same size as graphite skins).
- **B.** Place the Nomex in an air circulating oven and bake **as** follows:

Raise temperature to **394 K. (250°F., 121°C.).** Hold **394 K. (25OoF., 121°C.)** for **1/2** hour. **Cool** to **339°K. (150"F., 66°C.).**

7.2 BOUDING OF SANDWICH STOCK

- **A.** Tape corners of teflon film to platen with high performance tape. Teflon should be 25.4 mm. (1 in.) larger, on all sides, than graphite skin.
- **B.** Lay precured graphite skin on top of teflon film (leave **TX 1040** on bottom side only).

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- **C.** Position **a** ply of **Fn-34B** adhesive film onto the precured skin. If joints are required, they are to be butt joints.
- D. Position the trimm.ed Nomex core per engineering drawing requirements.
- **E. Position a ply of FH-34B adhesive film on to the Momex core. I€ joints are required, they are to be butt joints.**
- **I?. Position a precuced graphite skin on top of the FH-348 adhesive (leave TX 1040 on top side of graphite only).**
- **G. Apply a piece of mochburg over the TX 1040.**
- **H. Apply 0.64 cm. (0.25 in.) aluminum caul plate (same size as graphite skin) on top of the mochburg.**
- **I. Surround the layup with silicon edge dam 63 nun. x 25.4 nun. (0.25 in. x 1 in.). Stack darning until it is flush with or higher than the aluminum caul plate.**
- **J. Cover entire layup with airweave 110 leaving at least 25.4 mm. (1 in.) overhang around the perimeter.**
- **K. Apply a Vac-Pac HS-8171 bag over the layup with vacuum sealant and draw a minimum vacuum of 584 nun. (23 in.) Hg. Be sure all bridging is eliminated, and the bag is free of leaks.**
- **H. Autoclave cure as follows:**

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Apply a vacuum of 584 mm. (23 in.) Hg. and pressure of 173 KPa **(25 psi).** Raise temperature at 1.5 K./min. (3°F./min., 1.5°C./min.) to 394 K. (250°F., 121°C.). Hold 394 K. (250°F., 121°C.) for 30 minutes. **Raise at 1.5 K./min. (3"F./min., l.S"C./min.) to 450 K. (350°F** $177°C.$). **Hold at 450 K. (350"F., 177°C.) for 4 hours. Cool to 339 K. (150"F., 66°C.). Release pressure.**

- **1. Debag the layup and clean up as required.**
- **8.0 FABRICATION OF CO-CURED PANEL TYPES A, B, E, AMD F**
- **8.1 FABRICATION OF CORE STOCK (SEE STEP 7.1)**
- **8.2 FABRICATION OF PANELS ARE DESCRIBED BELOW:**
	- **A. Allow graphite prepreg to warm to room temperature prior to unspoo ling.**
- **B. Tape corners of teflon film to platen with high temperature tape.**
- **C. Lay uncured graphite prepreg (with Tx 1040 on bottom side) on teflon film. One graphite prepreg ply, Type A or E, 3 graphite prepreg plies, Type B or F.**
- **D. Position honeycomb core on prepreg.**
- **E. Lay uncured graphite prepreg (with TX 1040 on top side) on** honeycomb core. One graphite prepreg ply, Type A or E, or 3 **graphite prepreg plies, Type B or F.**
- **F. Lay a piece of teflon film over TX 1040.**
- *G.* **Apply high temperature tape from the teflon to platen on all edges.**
- **H. Position a 0.64 cm. (0.25 in.) aluminum caul plate over the tef lon.**
- **I. Surround the layup with silicon edge dam 6.3 nun. x 25.4 nun. (0.25** in. **x 1** in.). **Stack** damning **until it** is **flush with or higher than the aluminum caul plate.**
- **J. Cover entire layup with airweave NlO leaving at least 254** mm. **(1 in.) overhang around the perimeter.**
- **K. Apply a Vac-Pac HS-8171 bag over the layup with vacuum sealant and draw a minima! vacuum of 584 mm. (23 in.) Hg. Be sure all bridging is eliminated and the bag is free of leaks.**
- **L. Autoclave cure as follows:**

Type A and B panels:

Apply vacuum of 584 mm. (23 in.) Hg. and pressure of 173 KPa (25 psi). Raise temperature at 1.5 K./min. (l.S°C./min.) to 403 K. $(266°F., 130°C.).$ **Hold at 403 K. (266"F., 130°C.) for 20 minutes.** Raise temperature at 1.5 K./min. (3°F./min., 1.5°C./min.) to **450 K. (350°F., 177°C.).** Hold at 450 K. (350°F., 177°C.) for 6 hours. Cool to 339 K. (150°F., 66°C.). **Release pressure.**

Type E and F Panels:

Apply vacuum **of** 584 mm. (23 in.) **Hg.** and pressure **of** 173 KPa **(25** psi). Raise temperature at 1.5 K./min. (3"F./min., l.S"C./min.) to **405** K. (270°F.. 132°C.). **Hold** at **405** K. (270°F., 132°C.) for 1 hour. **Cool** to 339 K. **(150°F..** 66°C.). Release pressure.

M. Debag the layup and clean as required.

ti. Post cure panels A and **B** only as follows:

Place panels on aluminum **or** graphite platen and cover with **0.64** cm. (0.25 in.) aluminum caul plate. Place platen in circulating air oven and post cure 18 hours at *450* K. (350"F., 177°C.). Cool to 339 **K.** (150°F.. 66°C.).

9.0 APPLICATION **OF** PEEK FILM

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A. Clean panel skin with MeCl. Air dry.

B. Prepare xylene and benzoyl peroxide catalyst solution as follows:

> xylene - 4.70 **gm.** benzoyl peroxide - 0.52 **gm.**

> > 5.22 gm.

Mix solution with 350 gm. of X3-5815 adhesive. Several batches of catalized adhesive may be required depending on the number **of** panels to be processed.

C. Apply **a** 0.0025 cm. (0.001 in.) coating of the prepared adhesive **to the finished panel.**

D. Dry the adhesive at the following temperatures:

343 **K.** (158"F., 70°C.) for 15 minutes plus 423 K. (302"F., 150°C.) for *5* minutes

- E. Apply peek film to the panel and place under vacuum **584** nun. (23 in.) **Hg. for** 1 hour.
- **F.** Trim peek film to edge **of** panel.
- **G.** Cut panels to the desired size.

APPENDIX C

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MECHANICAL TEST DATA

SANDWICH BEAM FLEXURE

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Tested at room temperature Rate of Test: 3-6 minutes to failure Bottom Span: 55.9 cm Top Span: 10.2 cm

TEST METHOD: MIL-STD-401B and Spec. 2-31379

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TOTAL SANDWICH mxmn SPECIMEN THICKNESS WIDTH **(CUI (cn) (KG) TYPE OF COMPRESSIVE FAILURE STRESS MODULUS**
(KG/CM) KG/SO CI **(KGICM) KGISQ cn x 105 Haterial Type: D 7.72 7.75 7.72 7.57 7.47 7.65 46.5 C&S 36.6 S 46.3 C&S 50.0 C&S 49.1 C6S 46.3 C&S 104.8 82.5 104.8 112.7 110.5 104.1 1 0.709 2 0.737 3 0.739 4 0.737** *5* **0.739 6 0.739 8.9 8.9 9.1 8.8 8.7 8.9 Average** : **Standard Deviation: 92.5 10.7 8.9 0.1 Uaterial Type: E 1 0.690 2 0.690 3 0.688 4 0.688** *5* **0.688 6 0.686 7.67 7.67 7.67 7.67 7.67 7.67 20.4 C 20.5 C 20.7 C 20.7 C 21.6 C 20.6 C 46 .O 46.4 46.7 46.7 48.7 46.5 4.4 4.4 4.4 4.4 4.4 4.4 Average** : **Standard Deviation: 46.9 0.9 4.4 0.04 Material Type: F 1 0.709 2 0.709 3 0.709 4 0.711** *5* **0.711 6 0.709 7.67 7.67 7.67 7.67 7.67 7.67 35.4 C 37.7 C 38.1 C 35.1 C 37.4 C 36.3 C 79.8 85.0 85.9 79.3 84.5 81.9 6.5 6.5 6.5 6.4 6.6 6.5 Average** : **Standard Deviation: 82.7 2.8 6.5 0.1**

SANDWICH BEAX FIXTURE (Continued)

SANDWICH BEAM FIXTURE (Continued)

C = **Compressive facing failure**

S = **Shear failure in adhesive bond on tensile face**

- **MOTE** : **1. 2.54 cm x 10.2 cm x 0.318 cm Stainless Steel covered with Silicone Rubber of 60-70 Shore "A" Durometer Hardness were used as load pads.**
	- **2. The following dimensions were used in calculations (as listed in spec. 2-31379): Compression Face Thickness** = **0.0393 cm Tensile Face Thickness** = **0.0229 cm Sandwich Panel Thickness** = **0.6766 cm Core Thickness** = **0.635 cm Specimen Width** = **7.6 cm**
	- **3. All specimens were tested with the plastic coated facing in compression.**

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CLIMBING DRUM PEEL

Tested at room temperature Rate of Test: 2.54 cm/minute Drum Radius = **5.08 cm Flange Radius** = **6.40 cm Torque Am** = **1.32 cm**

CA = **Cohesive failure within adhesive ILF** = **Interlaminar failure of facing**

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CLIMBING ORUH PEEL (Continued)

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Haterial Type: G

Average : **Standard Deviation:** *0.7* **0.1**

AC = **Adhesive failure to core**

** = Adhesive bond on the non test facing debonded and the specimen could **not be tested,**

FLATWISE TENSILE STRENGTH

Tested at room temperature Rate of Test: 3-6 minutes to failure

TEST METHOD: MIL-STD-401B

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ILF = **Interlaminar failure of facing**

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FLATWISE TENSILE STRENGTH (Continued)

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Standard Deviation:

0.9

Haterial Type: G

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AC = **Adhesive failure to core**

C = **Core tensile failure**

CA = **Cohesive failure within adhesive**

NOTE: Plastic film was removed from facing before bonding the flatwise **tensile specimens.**

DENSITY Tested at Room Temperature

HATERIAL TYPE WEIGHT WIDTH LENGTH THICKNESS VOLUUE DENSITY GRAMS (C**H**) (C**H**) (C**H**) (C**H**³) **GHS /CM3 A 3.211 5.118 5.118 0.686 17.969 0.179 B 4.540 5.080 5.131 0.709 18.468 0.246 C 3.649 5.100 5.113 0.709 18.485 0.197 D 11.776 5.138 10.231 0.739 38.847 0.303 E 3.002 5.095 5.133 0.693 18.140 0.165 F 5.158 5.103 5.110 0.701 18.288 0.282 G 7.491 7.554 7.691 0.665 38.635 0.194**

TEST METHOD: Weight and Volume

APPENDIX D

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FLAMMABILITY TEST DATA

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FLAMMABILITY TEST DATA

Ohio State Calorimeter Testing

Test Method

Total heat r lease, peak heat release, and smoke density evolution was Total heat release, peak heat release, and smoke density evolution was
determined for each of the panel constructions, according to the Ohio State
2. antipater test (1977-1996 23), Testing use performed at 3.5 W/cm² by Calorimeter test (AST**H E906-83).** Testing was performed at 3.5 W/cm² by **Boeing Technical Services Company.**

Test Results

See Tables 4 and 5 for the results of this testing.

TABLE 4

OHIO STATE CALORIMETER TEST RESULTS

HEAT RELEASE DATA

 H_{EAT} RELEASE, Kw . $\min.7m$.² (Peak = Kw . $(m.2)$)

**OHIO STATE CALORIMETER TEST RESULTS
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FLAHHABILITY TEST DATA

ASTH E-162. FLAME SPREAD

Test Method

The samples were tested for surface flanunability in accordance with the procedures specified in ASTH E-162-83 "Surface Flammability of Materials Using a Radiant Heat Energy Source".

Specimens were pre-dried for 24 hours at 60°C. (140°F.) and then conditioned to equilibrium at a temperature of 22.7° C. \pm 3°C. (73 \pm 5°F.) and a relative humidity of $50 + 5\%$.

The specimens were supported with a 2.54 cm. (1 in.) hexagonal wire mesh in accordance with paragraph 5.9.2 of the test method.

The panels were tested with the film side facing the radiant heat source.

Test Results - **Panel Type A**

Observations:

Considerable melting, bubbling, and shrinking of the film facing. The panel core maintained good structural integrity with moderate charring on surface. Slight smoke evolution.

Conclusion :

The average flame spread index Is for the honeycomb panel material ^A type is 4.59.

Test Results - **Panel Type B**

NOTE: A disparity among the flame spread indices is indicative of a much greater heat rise in the case of specimens 3 and 4 and in the case of specimen 2, a flame front advance which did not extend to the first data point at 3".

Observations:

Considerable melting and shrinking away of the film facing. The panel core maintained good structural integrity. Very light smoke evolution.

Conclusion:

The average flame spread index Is for the honeycomb panel material B type is 12.77.

Test Results - **Panel Type C**

Observations:

Considerable melting, shrinking, and bubbling of the film facing. The panel core maintained good structural integrity with moderate charring and slight flaking. Slight smoke evolution.

Conclusion:

The average flame spread index Is for the honeycomb panel material C type is 5.26.

Test Results - **Panel Type E**

NOTE: The higher flame spread indices for specimens 3 and 4 are due to a greater heat rise in both cases and additionally, a flame spread advance which extended to the 6" data point on specimen four.

Observations:

Considerable melting, bubbling, and shrinking away of the film facing. The panel core maintained good structural integrity with slight charring and swelling. Slight smoke evolution.

Conclusion:

The average flame spread index Is for the honeycomb panel material E type is 13.41.

Test Results - **Panel Type F**

Observations:

Considerable melting and shrinking away of the film facing. The panel core maintained good structural integrity, with moderate charring, swelling and blistering. Moderate smoke evolution.

Conclusion:

The average flame spread index Is for the honeycomb panel material F type is 8.57.

Test Results - **Panel TyPe G**

NOTE: The higher flame spread index for specimens number one is due to a greater heat rise.

Observations:

Considerable bubbling of the film facing noted shortly after radiant heat exposure. Surface flaming was confined to the facing material. The panel core maintained good structural integrity.

Conclusion:

The average flame spread index Is for the honeycomb panel material ^G type is 17.70.

Test Results - **Baseline Panel**

Observations:

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Considerable charring, bubbling, and cracking on the specimen surface. The panel core maintained good structural integrity. Slight smoke evolution.

Conclusion:

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The average flame spread index Is for the honeycomb panel material Baseline type is 5.84.

FLAMMABILITY TEST DATA

ASTH E662-83lNFPA 258, Test for Evaluating the Smoke Generating Characteristics of Solid Haterials"

TEST METHOD

The test method ASTH E662-83 was used for this testing. The method defines smoke generation under flaming and nonflaming modes which are reported as average Maximum Specific Optical Density.

In addition to Specific Optical Density, the samples were sampled for carbon monoxide (CO), nitrogen oxide (NO_x), hydrogen fluoride (HF) and **hydrogen cyanide (HCN). These gases are measured as approximate parts per million (ppm) produced during the burning process in the flaming mode only. These values are not a valid measure of toxicity of the material under test but do give an idea of relative concentration of the gas produced.**

Test samples were conditioned at 60°C. (140°F.) for 24 hours followed by stabilization at 21°C. (70°F.) and 50-percent relative humidity prior to **testing** .

Test Results

See Table 6 for the results of this testing.

TABLE **6**

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Test for Evaluating the Smoke Generating Characteristics of Solid Materials

TEST RESULTS

ASTH **E662-83lNFPA 258,**

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 D_S = SPECIFIC OPTICAL DENSITY

TABLE 6 (cont'd)

MAXIMUM SPECIFIC OPTICAL DENSITY UNCORRECTED = D_m

HAXIMUM SPECIFIC OPTICAL DENSITY CORRECTED = D_m (Corr)

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GAS ANALYSES, TEST RESULTS

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FLAtMABILITY TEST RESULTS

ASTH D2863- 7 7 , **"Standard Method of Test for Flammability of Plastics Using the Oxygen Index Method"**

TEST METHOD

The procedures followed in this test are defined in ASTM D2863-77. The intent of this test method is to determine the relative flammability of plastics by measuring the minimum concentration of oxygen in a slowly rising mixture of oxygen and nitrogen that will just support combustion. This method is limited to the use of physically self-supporting plastic test specimen(s). Oxygen Index is defined as the minimum concentration of oxygen, expressed as volume percent, in a mixture of oxygen and nitrogen that will just support combustion of a material under the conditions of this method.

Seven specimens were tested. The average value for each panel construction is reported. The specimens were equilibrated at approximately 50% RH and 24'C (75'F) prior to testing.

Test Results

TABLE 7

ASTM D2863-77, Oxygen Index Test Results

FLAMMABILITY TEST RESULTS

FAR 25.853a. "Ignition Resistance of Aircraft Interior Materials, 60-Second Vertical"

TEST METHOD

The test method used was that described by FAR 25.853a. This method is intended for use in determining the resistance of material to flame and glow propagation and tendency to char.

The specimens were conditioned in accordance with the Standard. Each specimen tested was exposed to the test flame within 20 seconds after removal from the standard atmosphere.

The material undergoing test was evaluated for afterflame time, afterglow time and char length on each specimen as applicable.

The afterflame time and afterglow time of the specimens were recorded to the nearest 0.2 seconds and the char length to the nearest 2.5 $mm.$ (0.1 in.). **The test criteria for this test are as follows:**

Seven specimens were tested for the baseline panel and four each for the other panels.

Test Results

See Table 8 for the results of this testing.

TABLE 8

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IGNITION RESISTANCE OF AIRCRAFT INTERIOR MATERIALS

60 SECOND VERTICAL TEST RESULTS

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