

EVALUATION OF FIBROUS MATERIALS IN A CRYOGENIC STRUCTURAL ADHESIVE

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PREPARED BY: L. M. ROSELAND
SATURN PRODUCTION DESIGN BRANCH

C. A. Hedges

APPROVED BY: C. A. HEDGES
CHIEF, MATERIALS RESEARCH AND
PRODUCTION METHODS

DOUGLAS MISSILE & SPACE SYSTEMS DIVISION
SPACE SYSTEMS CENTER - HUNTINGTON BEACH, CALIFORNIA

A B S T R A C T

The results of a recent Douglas cryogenic structural adhesive program are presented and discussed. Twenty-three different fibrous constructions were placed in a urethane/diamine adhesive system and evaluated in T-peel and lap shear over a temperature range from -196°C (-320°F) to 82°C (180°F). One nonwoven glass construction dramatically improves the T-peel strength at all temperatures tested, improves the consistency of results, and produces a uniform thickness bond line.

D E S C R I P T O R S

Cryogenic Structural Adhesive
Polyurethane
Glass Fabrics
Organic Fabrics
T-peel Strength
Lap Shear Strength

P R E F A C E

SM-49202 is submitted by the Douglas Aircraft Company Inc., Missile and Space Systems Division, as a technical report under Contract NAS7-101, and covers data gathered from February through April 1966.

The purpose of this report is to provide the National Aeronautics and Space Administration and Douglas management with information concerning the improvement in the polyurethane adhesive system used for adhering S-IVB tank assembly attachments. Comments, techniques, tabulation of pertinent data, and photomicrographs have been included in this report.

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1. INTRODUCTION

Tank assembly attachments on the Saturn S-IVB stage are bonded with a structural cryogenic adhesive. This adhesive, a polyurethane paste type, produces bond lines of thicknesses ranging from 0.0004 inch to 0.012 inch. Laboratory tests on bonded lap shear and T-peel coupons show little, if any, strength reduction with very thin bond lines. However, when aluminum attachments, particularly star-shaped types, are bonded to the tank assembly, very thin bond lines have a tendency to produce debonds when stressed under cryogenic environment. The non-uniform and excessively thin cured bond line aggravates stress conditions caused by volume changes due to temperature fluctuation and differences in the coefficient of expansion between the aluminum adherend and the urethane adhesive system.

This report describes the results of a three-phase program to increase the reliability of the bonded attachments. Previous work had indicated that more reliability could be effected by embedding a glass fabric into the bond line of adhesively bonded aluminum specimens (reference 8.1 and 8.2).

To ensure more-uniform cured bond lines and to choose the optimum material to accomplish this uniformity, 23 different fibrous constructions were embedded in the adhesive and evaluated in three phases. Phase I consisted of the selection of five fabrics which exhibited the highest T-peel strengths in liquid nitrogen. Phase II consisted of the selection of three of these five fabrics on the basis of lap shear strengths at -196°C (-320°F) and at 24°C (75°F). In Phase III, the most suitable fabric of the three determined in Phase II was selected on the basis of T-peel and lap shear results. These results encompassed a temperature profile having a range of -196°C (-320°F) to 82°C (180°F).

2. MATERIAL

The materials used in this program are discussed below.

2.1 Adhesive System

The adhesive used was a two-part system as follows:

- a. EC 3515 B, Polyurethane Adhesive Base, Lot 6M5P - The 3M Company
- b. EC 3515 A, Diamine Curing Agent, Lot 2M5P - The 3M Company

2.2 Primer

XC 3901, Silane Primer, Lot 6M5P, manufactured by Minnesota Mining and Manufacturing 3M Company, was the primer used throughout this program.

2.3 Metal Test Specimens

The metal test specimens used in this program were as follows:

- a. Aluminum Peel Strips, 2014-T6 Bare, 1 inch x 10 inches x 0.032 inch
- b. Aluminum Shear Pads, 2014-T6 Bare, 3 inches x 5 inches x 0.064 inch

2.4 Fibrous Materials

2.4.1 Glass Fabrics

Additional information on the following fabrics is contained in Appendix 1.

- a. Woven, Glass, Type 104, Clark-Schwebel Corp.
- b. Woven, Glass, Type 108, Clark-Schwebel Corp.
- c. Woven, Glass, Type 1610, Clark-Schwebel Corp.

- d. Woven, Glass, Type 1562, Clark-Schwebel Corp.
- e. Nonwoven, Glass, Modiglass SSM, Modiglass Fibers
- f. Nonwoven, Glass, Modiglass Veil, Modiglass Fibers
- g. Nonwoven, Glass Mat 236, Thalco Corp.

2.4.2 Organic Fabrics

Additional information on the fabrics listed below is contained in Appendix 2.

- a. Woven, Dacron, 15041/1, Stern and Stern Textiles, Inc.
- b. Woven, Nylon, A-2827/4, Stern and Stern Textiles, Inc.
- c. Woven, Dacron, 15320, Stern and Stern Textiles, Inc.
- d. Woven, Nylon, 38068, J. P. Stevens
- e. Woven, Nomex, HT-65-30, E. I. DuPont Company
- f. Woven, Cotton, Producer Unknown
- g. Nonwoven, Dacron, Reemay 200, E. I. DuPont Company
- h. Nonwoven, Dacron, Reemay 201, E. I. DuPont Company
- i. Nonwoven, Nylon, XS-144415, The 3M Company

2.4.3 Miscellaneous Fibrous Materials

Additional information on the following materials is contained in Appendix 3.

- a. Cellulosic Paper, Disposable, Crown Zellerbach Corp.
- b. Nomex Paper, No.-51, Uncalendered, E. I. DuPont Company
- c. Asbestos Paper, Noramite, C10-V30, North American Asbestos Company
- d. Milled Glass Fiber, 1/4 inch, No. 701, Thalco Corp.
- e. Window Screen, Aluminum, Alcoa Aluminum Company
- f. Asbestos Fibers, Blue, North American Asbestos Company
- g. Glass Mat and Nylon Nonwoven Composite, 11-W, Douglas Aircraft Company

3. EQUIPMENT

3.1 Processing

The equipment listed was used in the preparation of specimen for testing.

- a. Blue Line Circulating Oven, Model 625, Range to 325°C (617°F)
- b. Bell Jar and Vacuum Pump
- c. Semco Sealant Gun

3.2 Testing

This program incorporated the use of the following test equipment:

- a. Baldwin Universal Testing Machines and Recorders
 1. Model 5K, Range 0.002 to 5,000 pounds
 2. Model 10K, Range 100 to 10,000 pounds
- b. Cryostat for liquid nitrogen, 5 liter capacity
- c. Thermatron, Model No. 2154, range -
- d. Micrometer

4. PROCEDURE

4.1 Specimen Preparation

T-peel and lap shear specimens for the program were prepared as outlined in the following paragraphs.

4.1.1 T-peel Specimens

The T-peel specimens were prepared from 2014-T6 bare 0.032 inch thick aluminum panels. The specimens were 1 inch wide and 12 inches long.

The T-peel panels were cleaned as follows:

- a. Each was thoroughly wiped with clean cotton cloths soaked in MEK (Methyl-ethyl-Ketone).

- b. Each was etched in hot British etch solution for 20 minutes at 155°F.
- c. Each was washed with deionized water until all acid was removed.

4.1.1.1 Phase I Specimens

In Phase I, the T-peel panels were air-dried for 2 to 4 hours prior to the application of the primer. Primer dry time was 2 hours before application of the adhesive.

4.1.1.2 Phase III Specimens

In Phase III, the T-peel panels were air-dried 2 to 4 hours prior to the application of the primer. Primer dry time was 2 hours before application of the adhesive.

4.1.2 Lap Shear Specimens

The lap shear specimens were prepared from 2014-T6 bare 0.064-inch thick aluminum finger panels cleaned as follows:

- a. Each was thoroughly wiped with clean cotton cloths soaked in MEK.
- b. Each was etched for 30 minutes in British paste etch which was applied by a brush. Every 5 minutes, the paste on the panels was agitated by rebrushing.
- c. Each was washed with deionized water until all paste etch was removed.

4.1.2.1 Phase II Specimens

In Phase II, the lap shear panels were air-dried for 2 to 4 hours prior to the application of the adhesive. No primer was used in Phase II.

4.1.2.2 Phase III Specimens

The Phase III lap shear panels were air-dried for 2 to 4 hours prior to the application of the primer. The panels were primed using a one-inch wide paint brush. Primer dry time was 2 hours before application of the adhesive.

4.1.3 Adhesive Preparation and Lay-Up

The adhesive was prepared as follows:

- a. The EC3515 B (base) was heated to 49°C (120°F) by placing it in a 49°C (120°F) oven for 2 hours.
- b. The EC3515 A (curing agent) was heated to 121°C (250°F) by placing it in a 121°C (250°F) oven for 2 hours.
- c. The heated EC3515 B and EC3515 A were thoroughly mixed by hand for 3 minutes. The mix ratio was 11 parts of EC3515 A to 100 parts of EC3515 B.
- d. The mixture was degassed by placing it in a vacuum chamber at a vacuum of over 26 inches of mercury until most of the bubbles disappeared. (About 5-8 minutes).
- e. The mixed adhesive was poured into a Semco polyethylene tube. The tube was inserted in the Semco gun and the adhesive was immediately extruded onto both surfaces of each coupon. The adhesive was spread evenly on the coupons with a spatula.

4.1.3.1 Organic Fabrics and Papers

All organic fabrics and papers were conditioned 24 hours at 60°C (140°F) in an oven before evaluation, in order to ensure low moisture content. The organic fabrics and papers were cooled to ambient temperature and used within 30 minutes.

4.1.3.2 Fibrous Constructions

In the case of fibrous constructions, (the fabrics and papers) each was placed in the adhesive on one panel surface. The other panel with

applied adhesive was then placed against this fabric. The aluminum screen was evaluated in the same manner.

4.1.3.3 Adhesive Formulations

The following adhesive formulations were used to evaluate the two short fibers:

a.	EC3515 B	100.0 gms
	Asbestos Fibers, Blue	1.3 gms
b.	EC3515 B	100.0 gms
	Milled Glass Fibers	2.0 gms

Each of the fibers was mixed into the EC3515 B previous to the addition of the EC3515 A. The EC3515 A was added at the ratio of 11 parts to 100 parts of EC3515 B.

4.1.3.4 Humidity

The maximum allowable relative humidity during the adhesive preparation was 65 percent.

4.1.4 Adhesive Cure

Vacuum was applied within 1 hour after the adhesive was mixed.

Total vacuum time was 24 hours at 22°-32°C (72°-90°F) under 9-15 inches of mercury.

After removal from the vacuum bag, specimens were placed in a circulating oven at 71° ± 2°C (160°F ± 5°F) for 24 hours.

Maximum allowable relative humidity during the adhesive cure cycle was 65 percent.

4.2 Testing

A summary of the lap shear and T-peel testing is given below.

4.2.1 Lap Shear

Lap shear specimens were tested per Federal Test Method Standard 175, Method 1033.1, except that the specimens were pin-loaded on the tensile testing machine.

4.2.2 T-peel

T-peel specimens were pin-loaded on the tensile testing machine with a jaw separation rate of 20 inches per minute.

4.2.3 Temperatures

Test temperatures were as follows:

- | | |
|--------------|--|
| a. Phase I | -196°C (-320°F) |
| b. Phase II | 24°C (75°F) and -196°C (-320°F) |
| c. Phase III | -196°C (-320°F), -73°C (-100°F)
-18°C (0°F), 24°C (75°F), and
82°C (180°F) |

The test temperature of -196°C (-320°F) was produced by liquid nitrogen. The test temperatures of -73°C (-100°F) and -18°C (0°F) were produced with carbon dioxide and the Thermatron.

4.2.4 Soak Time

Specimen soak time was 3 minutes at all temperatures except 24°C (75°F).

5. RESULTS

5.1 T-peel

The Phase I T-peel strengths at -196°C (-320°F) of the polyurethane adhesive system reinforced with different materials are reported in table 1. The Phase III T-peel strengths at -196°C (-320°F), -73°C (-100°F), -18°C (0°F), 24°C (75°F), and 82°C (180°F) on the best three reinforcing materials for the EC3515 B/A adhesive system as selected by Phase II are reported in table 4.

5.2 Lap Shear

Phase II lap shear strengths at -196°C (-320°F) and 24°C (75°F) of the best five reinforcing materials for the EC3515 B/A adhesive system as selected by Phase I are reported in table 2.

5.3 Porosity Index

The porosity index of glass and organic woven fabrics is reported in table 5.

5.4 Glass Content

The glass content of four woven fabrics is reported in table 6.

5.5 Consistency - T-peel and Lap Shear

The consistency index of T-peel values is found in table 7 and table 8 contains the consistency index of lap shear values.

6. DISCUSSION

The following paragraphs contain a discussion of significant events encountered in this three-phase program.

6.1 Phase I

6.1.1 Test Method

T-peel strength in liquid nitrogen was used as the initial selection method in Phase I. Since the urethane adhesive system used (EC3515 B/A) usually fails cohesively in T-peel tested in liquid nitrogen, this method reflects the internal strength of the system rather than the adhesive strength of the system.

6.1.2 Comparison of T-peel Strengths

Table 1 compares the T-peel strengths in liquid nitrogen, of all the fibrous materials tested. It is of interest to note that the best five fibrous materials all represent different and distinct materials rather than any single type or form. For example, the Modiglass SSM is a non-woven, continuous glass filament mat, the 1562 is a woven glass fabric, the Reemay 200 is a nonwoven Dacron fabric, the Mat 236 is a glass surfacing mat whose fibers are arranged in a veil-like (jack straw) manner, and the milled glass 701 fibers are short, 1/8 inch long. Of the 23 materials evaluated, 11 produced T-peel strengths of equal or improved values over the control. The Phase I results do not clearly indicate reasonable generalizations for fabric utilization in structural adhesives at cryogenic temperatures. One can conclude, however, that simple increase in the cured bond line thickness does not increase the T-peel values in liquid nitrogen.

6.1.3 Physical Structure of Woven Fabrics

Some insight into these different values can be seen by examining the photomicrographs (10 diameters magnification) of the fabrics themselves. In the case of woven glass fabrics, the 1562 glass fabric has markedly improved the T-peel strength over the control, and is far better than the other three woven glass materials. As can be seen in figure 1, the

porosity of 1562 is the highest of the four materials. For use in this program, the "porosity index" is an arbitrary number which represents the open area in square inches between the warp and fill threads of a woven structure. (See table 5 for relative differences in the glass and organic fabrics evaluated.) More properly, "porosity" is the ratio of the volume of air, or void, contained within the boundaries of a material to the total volume (solid material plus air or void) expressed as a percentage.

Fabric style 1562 is the thickest of the four woven fabrics, but it has the least number of threads in both the warp and fill directions. Fabric style 1562 is a leno weave rather than a plain weave, as are the other three fabrics. In a leno weave, adjacent warp yarns interweave alternately to the right and left of the adjacent fill. One set of yarns is firmly anchored to the other by this special type of yarn intersection. Leno weaves, especially open construction fabrics, are made more stable in this manner.

Glass fabric styles 104 and 108 contribute approximately the same T-peel strength to the adhesive, yet, style 108 has twice the thickness and over twice the amount of glass as style 104 (see table 6).

Figure 2 shows photomicrographs of the four organic, woven fabrics. Here we see that the material which results in the lowest T-peel strength for the adhesive system has the lowest porosity and has a plain weave. The best two fabrics (Styles 15320 and A-2827) are leno weaves and are quite porous.

6.1.4 Physical Structure of Nonwoven Fabrics

Figure 3 shows photomicrographs of three of the nonwoven fabrics evaluated. Of the four materials which exhibit the highest T-peel values with the adhesive at -196°C (-320°F), three of them were nonwoven fabrics. As can be seen, both the Modiglass SSM and the Mat 236 have the greatest

porosity and are light in weight. The Reemay 201 (a black version of Reemay 200), while not as open, is composed of fine webs of randomly arranged, continuous filament fibers. The finished structure is unlike any other nonwoven fabric.

6.1.5 Milled Glass Fibers

The milled glass fibers (No. 701), added to the adhesive, produced good T-peel values in liquid nitrogen. Since the object of this program was to control the cured bond-line thickness, Dacron fabric 15320, which exhibited similar T-peel values, was substituted in Phase II for the milled glass fibers. The milled fibers would probably not control the cured bond-line thickness as accurately as this woven Dacron fabric.

6.2 Phase II

6.2.1 Test Method

Lap shear strength in liquid nitrogen, and at 23°C (74°F), was used as the selection method in Phase II.

6.2.2 Comparison of Lap Shear Strengths

Table 2 shows the comparison of the lap shear strengths of EC3515 B/A adhesive reinforced with the five fabrics selected from Phase I. In liquid nitrogen, the control exhibited rather low values. The five variations containing fabrics produced metal failure (bearing) at the pin hole. At 23°C (74°F) the controls were normal. The Modiglass SSM exhibited the highest lap shear values, with Dacron fabric 15320 exhibiting the second highest. It is quite unusual that the inclusion of a scrim cloth or fabric actually improves the lap shear strength because it normally does not contribute to strength, being, in effect, an inert filler material.

6.3 Phase III

6.3.1 T-Peel Strength

T-peel strengths at five temperatures were obtained on EC3515 B/A adhesive reinforced with the three fabric materials which exhibited the highest T-peel strengths in Phase I and the highest lap shear values in Phase II. These T-peel strengths are reported in table 3. A graph comparing these strengths can be seen in figure 4. The Modiglass SSM reinforced adhesive again exhibits the highest strengths of all materials tested at -196°C (-320°F), and increases this superiority at -73°C (-100°F). At -18°C (0°F), 24°C (74°F) and at 82°C (180°F) the adhesive containing Modiglass SSM exhibited increased strengths over the control of 129 percent, 123 percent, and 80 percent respectively.

6.3.1.1 Reproducibility

In addition to this improvement of T-peel strength, the Modiglass SSM variation exhibits considerable improvement in reproducibility of T-peel values over the control. Numerical values, representing the relative consistency index value, changes at each temperature level evaluated. Over the temperature range of -196°C (-320°F) to 82°C (180°F), the variation containing Modiglass SSM shows an overall improvement of 44 percent in the consistency of strengths over the control (see table 7).

The T-peel value of any single specimen is an average value obtained from a continuously recorded peel chart. In this evaluation, the first inch of peel (or 2 inches of chart movement) is eliminated, and the average value of the next 6 inches of peel (or 12 inches of chart movement) is averaged. In figure 5, the T-peel chart of a control specimen is superimposed on the chart of a Modiglass SSM variation. The amount of change in a single control specimen is about twice as much as the Modiglass SSM variation specimen. This indicates that the Modiglass SSM

improves reproducibility within the specimen as well as reproducibility of a numerical average.

6.3.2 Lap Shear Strengths

Lap shear strengths at five temperatures were obtained on the adhesive reinforced with the three fabric materials which were selected by Phases I and II. These values are reported in table 4. Again the Modiglass SSM-containing variation shows superiority over the control at 24°C (75°F). In addition, this superiority is maintained at -18°C (0°F). All of the three fabrics tested exhibit metal pinhole failure (bearing) at -73°C (-100°F) and -196°C (-320°F). The Modiglass SSM variation shows some decrease in lap shear strength from the control at 82°C (180°F).

6.3.2.1 Reproducibility

The Modiglass SSM variation also exhibits an overall improvement of 31 percent in consistency of lap shear values over the control (see table 8).

6.4 Comments on Modiglass SSM Variation

The superior T-peel and lap shear values of EC3515 B/A adhesive reinforced with this fabric are probably due to several reasons. Modiglass SSM is a very porous and light weight mat. It is composed of single, continuous filaments rather than strands or threads of many filaments as are woven fabrics. The high porosity permits the adhesive to easily penetrate the fabric and wet out the single filaments. Microscopic examination of lap shear specimens prepared with Modiglass SSM shows that the fibers are completely surrounded with the adhesive. No loss of adhesion between the adhesive and filament is evident after testing (see figure 6A). The fabrics woven from multifilament glass or organic material exhibit a different appearance under microscopic examination. The exterior of the

thread bundle itself appears to have been wet with adhesive. The interior of the thread, however, appears to be dry; no adhesive is evident and the filaments are not in contact with each other and are, in fact, loose (see figure 6B). This partial wetting of the reinforcement filaments decreases the surface available for adhesion between the adhesive and the filament, and lowers the efficiency of the composite system. The surface area available for adhesion is obviously much higher in a multiple filament strand than in a single filament. However, if the major portions of the surface area is not available for adhesion because of poor adhesive wet-out, the nonadhered reinforcement then becomes a liability rather than an asset. The effective fiber surface area per fiber density would be an important physical property to know, to properly compound composite structures.

The Modiglass SSM uses a coupling agent, glycidoxy-propyl trimethoxysilane in the binder system. This is the same silane which was previously shown to have excellent utility with urethane adhesive systems at cryogenic temperatures (reference 8.1, 8.2). The woven glass fabrics all utilize gamma-aminopropyltriethoxysilane.

The Modiglass SSM variation produces uniform cured bond lines. Lap shear specimens exhibited bond lines ranging from 10 to 12 mils thick, with an average of 11 mils. The T-peel specimens exhibited bond lines ranging from 9 to 12 mils, with an average of 11 mils.

6.5 Comments on the Reemay 201

Reemay 200 was evaluated in Phases I and II. Reemay 201, black, was substituted for the 200 in Phase III since the 200 was not available locally. The 201 is almost identical to the 200 except for color.

The T-peel and lap shear results of the Reemay 201 reinforced adhesive are quite remarkable. The T-peel values at -18°C (0°F), 24°C (75°F) and 82°C (180°F) are the highest of all fabrics tested. The Reemay material

is made entirely of continuous filaments of polyester formed into a web and bonded in position. Microscopic examination reveals a truly three-dimensional structure. The high values of peel and lap shear would indicate that the adhesive has thoroughly wet the filaments and produced an effectively bonded structure. Microscopic examination of tested lap shear bonds surprisingly indicate that physical entanglement of the random fibers in the adhesive is probably responsible for the high values rather than any reinforcement due to adhesion between the fibers and the adhesive. Many of the fibers appear as if they have lost contact with the adhesive.

6.6 Comments on the Glass Woven Fabric 1562

Since woven fabrics are bidirectional, this fabric was tested in both the warp and fill directions in Phase III. The warp direction contains twice as much area of glass as the fill direction (table 6).

At some temperatures, the T-peel and lap shear results differ in the warp and fill direction. For example, in T-peel results, the fill direction values appear best at -196°C (-320°F) and at -73°C (-100°F). At -18°C (0°F), 24°C (75°F) and at 82°C (180°F), the warp and fill direction values appear identical. In the case of lap shear results, the fill direction values appear lowest at -18°C (0°F), 24°C (75°F) and at 82°C (180°F).

7. CONCLUSIONS

Since EC3515 B/A polyurethane adhesive, containing Modiglass SSM mat improves the average T-peel values over the range from -196°C (-320°F) to 82°C (180°F), exhibits good lap shear values over the same temperature range, and significantly increases the reproducibility of results, this mat has been selected as the most promising for cryogenic usage of those reinforcements tested.

8. REFERENCES

8.1 L. M. Roseland, Preliminary Evaluation of Narmco 7343/7139 Contain-
ing a Glass Scrim Cloth, Douglas MP Report No. 2191, Catalog No.
PDL 54783, July 1965.

8.2 L. M. Roseland, Investigation of Prepolymer Urethane Resin Systems
for Space Usage, Douglas S. M. Report No. SM-49146, August 1965.

9. DATA

Technical Record 03198 Pages 10-16, 24, 27, 28, 32, 33, 40-51.

EFFECT OF REINFORCING FILLERS ON T-PEEL STRENGTH
OF EC3515 B/A ADHESIVE AT -196°C (-320°F)

REINFORCEMENT*	BOND LINE THICKNESS (MILS)	AVERAGE T-PEEL, (PIW)	T-PEEL LOW/HIGH, (PIW)	TYPE OF BREAK
Control (no reinforcement)	3	54	41/69	C.
Modiglass SSM	10	96	95/100	C. Fibers on both surfaces
1562 Fabric, Glass	9	83	74/89	C. Fabric alternates from one side to other
Reemay 200, Dacron	15	78	71/85	C. Fabric on both surfaces
Mat 236, Surfacing	9	72	67/80	C. Fibers on both surfaces
Milled Glass Fibers, No. 701	4	72	65/81	C. Fibers on both surfaces
15320 Fabric, Dacron	6	66	64/68	C. Fabric on one side only
XS-144415 Fabric, Nylon	12	65	56/68	C. Fabric on both sides
Aluminum Screen	29	64	63/66	C. Screen on one side only
Modiglass Veil	10	64	35/89	C. Fibers on both surfaces
Douglas Composite 11-W	16	62	58/66	C. Fibers on both surfaces
A-2827 Fabric, Nylon	7	57	53/62	C. Fabric on both surfaces
108 Fabric, Glass	5	51	46/55	C. Fabric on one side only
104 Fabric, Glass	5	48	45/51	C. Fabric on both surfaces
1610 Fabric, Glass	9	38	35/41	C. Fabric on one side only
HT-102-42 Nomex	7	31	30/31	C. Fabric on one side only
38068 Fabric, Nylon	13	28	25/34	C. Fabric on one side only
Asbestos Fibers, Blue	5	28	26/31	C. Fibers on both surfaces
15041 Fabric, Dacron	8	26	19/40	C. Fabric on one side only
Fabric, Cotton	8	19	17/20	C. Fabric on both surfaces
HT-65-30 Nomex	11	11	10/11	C. Fabric on one side only
Cellulosic Paper, Disposable	5	11	6/24	C. Fibers on both surfaces
Asbestos Paper, Normite	12	5	2/13	C. Fibers on both surfaces
Nomex Paper, E-51	9	3	2/6	C. Fibers on both surfaces

- NOTE: 1. Each value represents the average of 5 specimens
 2. All are unprimed values
 3. C. is cohesive break
 * Warp direction was used in all cases of woven fabric

PHASE II

TABLE 2

EFFECT OF REINFORCING FILLERS ON LAP SHEAR STRENGTH OF EC3515 B/A ADHESIVE AT -196°C (-320°F) AND 23°C (74°F)

REINFORCEMENT	BOND LINE THICKNESS (MILS)	AVERAGE LAP SHEAR AT -196°C (-320°F) (PSI)	AVERAGE LAP SHEAR AT 23°C (74°F) (PSI)	LAP SHEAR, LOW/HIGH AT 23°C (74°F) (PSI)	TYPE OF FAILURE
Control (no re-inforcement)	1.5	4660 (4 specimens) Metal Failure (1 specimen)	1330	$\frac{1265}{1450}$	A
Modiglass SSM	9.5	5830 (1 specimen) Metal Failure (4 specimens)	1655	$\frac{1585}{1735}$	A
*Dacron Fabric 15320	6.5	Metal Failure	1365	$\frac{1345}{1400}$	A
Reemay 200, White	11.0	Metal Failure	1185	$\frac{1155}{1225}$	A
Mat 236	6.5	Metal Failure	1155	$\frac{1100}{1210}$	A
*Glass Fabric 1562	8.5	Metal Failure	1130	$\frac{1080}{1195}$	A

- NOTE: 1. Each value represents the average of 5 specimens unless noted.
 2. All are unprimed values.
 3. A is adhesive failure.
 * Warp direction

PHASE III

TABLE 3

T-PEEL STRENGTHS OF REINFORCED EC3515 B/A ADHESIVE
AS A FUNCTION OF TEMPERATURE

RESULTS IN PIW

REINFORCEMENT	BOND LINE THICKNESS (MILS)	-196°C (-320°F)			-73°C (-100°F)			-18°C (0°F)			24°C (75°F)			82°C (180°F)		
		AVE. T-PEEL	LOW/HIGH	TYPE OF BREAK	AVE. T-PEEL	LOW/HIGH	TYPE OF BREAK	AVE. T-PEEL	LOW/HIGH	TYPE OF BREAK	AVE. T-PEEL	LOW/HIGH	TYPE OF BREAK	AVE. T-PEEL	LOW/HIGH	TYPE OF BREAK
Control (no reinforcement)	1-5	77.6	$\frac{67}{90}$	100%C	35.9	$\frac{31}{39}$	100% AP	51.7	$\frac{42}{58}$	100% AP	27.7	$\frac{17}{35}$	100% AP	21.4	$\frac{18}{24}$	100% AP
Modiglass SSM	8-12	97.3	$\frac{90}{105}$	100%C	145.0	$\frac{140}{150}$	100% C	118.8	$\frac{101}{128}$	50% AP 50% AP	61.9	$\frac{54}{67}$	100% AP	38.7	$\frac{31}{43}$	50% AP 50% C
Reemay 201, Black	10-14	55.4	$\frac{15}{80}$	10% AP 90% C	106.9	$\frac{65}{125}$	100% C	117.6	$\frac{86}{131}$	100% C	84.5	$\frac{70}{92}$	100% AP	71.9	$\frac{65}{78}$	10% AP 90% C
Glass Fabric 1562, Warp Direction	6-8	71.3	$\frac{39}{85}$	100% C	78.5	$\frac{65}{95}$	100% AP	111.2	$\frac{100}{121}$	100% AP	66.3	$\frac{50}{74}$	100% AP	42.6	$\frac{33}{45}$	85% AP 15% C
Glass Fabric 1562, Fill Direction	7-8	87.8	$\frac{83}{92}$	100% C	103.0	$\frac{95}{115}$	60% AP	111.4	$\frac{105}{124}$	75% AP 25% C	72.4	$\frac{63}{84}$	100% AP	42.8	$\frac{34}{45}$	70% AP 30% C

Each value represents the average of 10 specimens except the 1562, fill direction with represents 7 values.
 AP - Adhesive Failure to Primer
 C - Cohesive Failure
 All Aluminum panels primed with XC 3901

PHASE III

TABLE 4

LAP SHEAR STRENGTHS OF REINFORCED EC3515 B/A ADHESIVE
AS A FUNCTION OF TEMPERATURE

RESULTS IN PSI

REINFORCEMENT	BOND LINE THICKNESS (MILS)	LAP SHEAR AT -196°C (-320°F)	LAP SHEAR AT -73°C (-100 F)	-18°C (0°F)			24°C (75°F)			82°C (180°F)		
				AVERAGE LAP SHEAR	LOW/HIGH	TYPE OF BREAK	AVERAGE LAP SHEAR	LOW/HIGH	TYPE OF BREAK	AVERAGE LAP SHEAR	LOW/HIGH	TYPE OF BREAK
Control (no reinforcement)	5	*	*	4187 3468 5221	50%AP 50%AP	2097	1686 2755	75%AP 25%AP	1622	1247 2010	75%AP 25%AP	
Modiglass SSM	11	*	*	4501 3773 5102	15%AP 85%AP	2292	1890 2849	90%AP 10%AP	1300	1133 1460	70%AP 30%AP	
Reemay 201, Black	11	*	*	4443 3940 4740	100%AP	2347	1990 2861	25%AP 75%AP	1174	990 1296	15%AP 85%AP	
1562 Fabric, Glass, Warp Direction	10	*	*	3503 3182 3900	100%AP	1653	1422 1802	100%AP	1249	1066 1490	50%AP 50%AP	
1562, Fabric, Glass, Fill Direction	10	*	*	2729 2603 2951	100%AP	1455	1257 1713	100%AP	1012	907 1102	100%AP	

Each value represents the average of 10 specimens except control average at 180°F which represents 5 values.

*PIN HOLE IN METAL PANEL FAILS IN BEARING

AP - Adhesive Failure to Primer

C - Cohesive Failure

All panels primed with XC 3901

TABLE 5
POROSITY INDEX

FORMULA:
$$\frac{\text{PORE SIZE* IN SQUARE INCHES X NO. OF PORES/3 INCH WIDTH}}{10^{**}} = \text{POROSITY INDEX}$$

GLASS FABRICS

1562	0.055
104	0.016
1610	0.009
108	0.008

ORGANIC FABRICS

A-2827 (Nylon)	0.104
38068 (Nylon)	0.096
15320 (Dacron)	0.026
15041 (Dacron)	0.009

*Open area between threads measured from photomicrographs
**Magnification

TABLE 6
GLASS CONTENT OF FOUR WOVEN FABRICS*

TYPE	YARN DESCRIPTION		INDIVIDUAL FILAMENT AREA, IN. ²	AREA OF GLASS IN SQ. IN./IN., WARP	AREA OF GLASS IN SQ. IN./IN., FILL
	WARP	FILL			
104	90-1/0	1800-1/0	0.379×10^{-7}	2.31×10^{-4}	1.00×10^{-4}
108	900-1/2	900-1/2	0.379×10^{-7}	4.62×10^{-4}	3.61×10^{-4}
1610	150-1/0	150-1/0	0.96×10^{-7}	6.27×10^{-7}	5.48×10^{-4}
1562	150-1/0	150-1/0	0.96×10^{-7}	6.08×10^{-4}	3.04×10^{-4}

*Calculated from vendor data

TABLE 7
 EFFECT OF REINFORCEMENTS IN EC3515 B/A ADHESIVE
 ON CONSISTENCY INDEX (T-PEEL)

FORMULA: $\frac{\text{HIGH VALUE} - \text{LOW VALUE}}{\text{AVERAGE VALUE}} = \text{CONSISTENCY INDEX}$

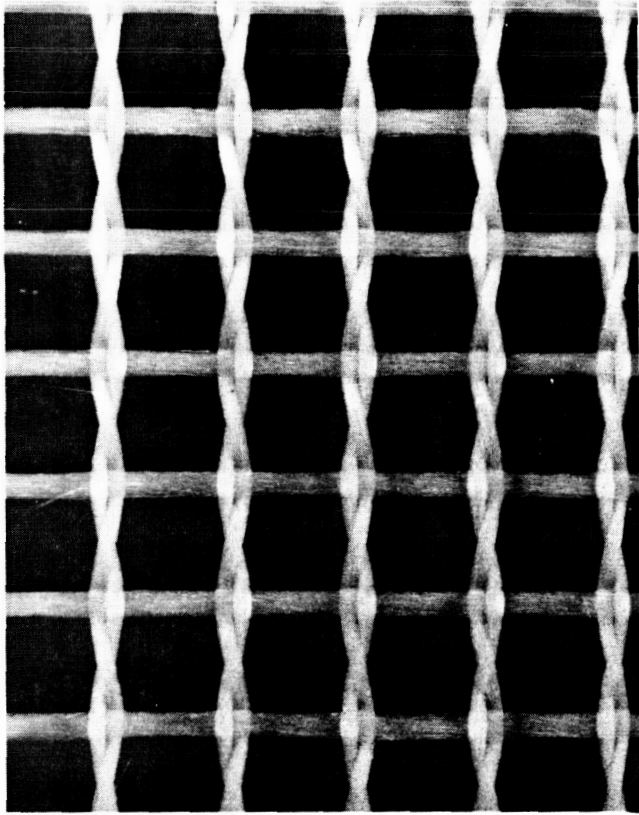
	-196°C (-320°F)	-73°C (-100°F)	-18°C (0°F)	24°C (72°F)	82°C (180°F)	AVERAGE TOTAL IMPROVEMENT OVER CONTROL
Control (no reinforcement)	0.29	0.22	0.31	0.65	0.28	--
Modiglass SSM	0.15	0.07	0.23	0.21	0.31	+44%
Reemay 201, Black	1.17	0.56	0.38	0.26	0.18	-44%
Glass Fabric, 1562 Warp	0.64	0.38	0.18	0.36	0.19	0
Glass Fabric, 1562 Fill	0.09	0.19	0.17	0.29	0.25	+43%

TABLE 8

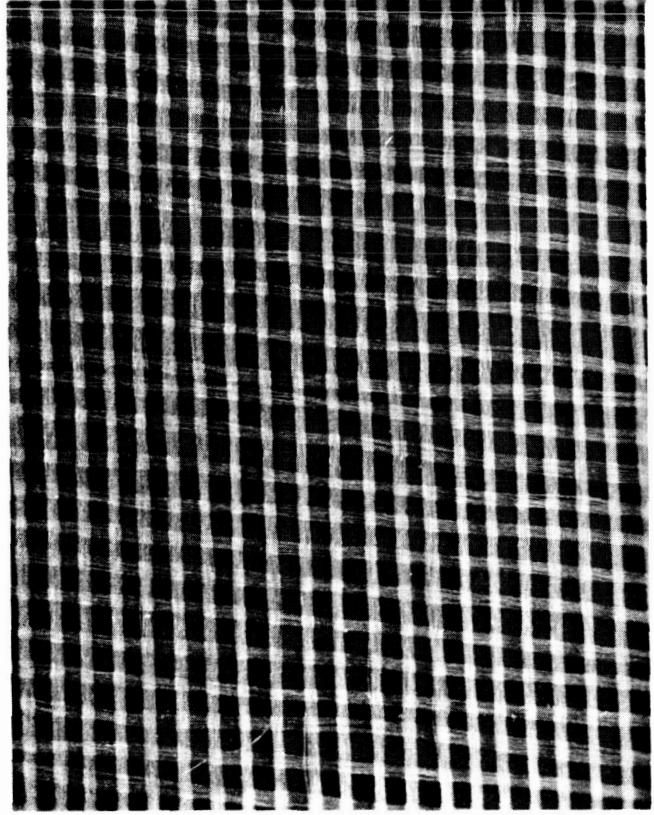
EFFECT OF REINFORCEMENT IN EC3515 B/A ADHESIVE
ON CONSISTENCY INDEX (LAP SHEAR)

FORMULA: $\frac{\text{HIGH VALUE} - \text{LOW VALUE}}{\text{AVERAGE VALUE}} = \text{CONSISTENCY INDEX}$

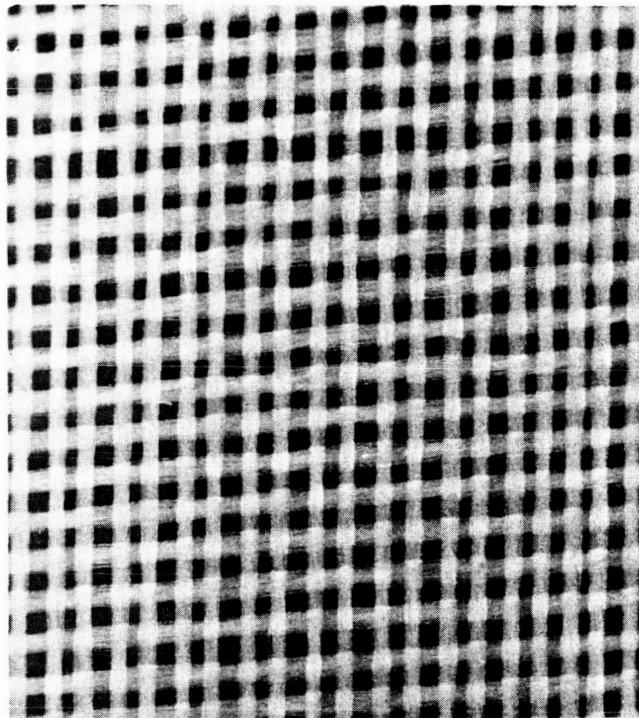
	-18°C (0°F)	24°C (75°F)	82°C (180°F)	AVERAGE TOTAL IMPROVEMENT OVER CONTROL
Control (no rein- forcement)	0.41	0.51	0.57	--
Modiglass SSM	0.29	0.41	0.25	31%
Reemay 201, Black	0.18	0.37	0.20	46%
Glass Fabric, 1562 Warp	0.20	0.23	0.34	46%
Glass Fabric, 1562 Fill	0.12	0.31	0.19	55%



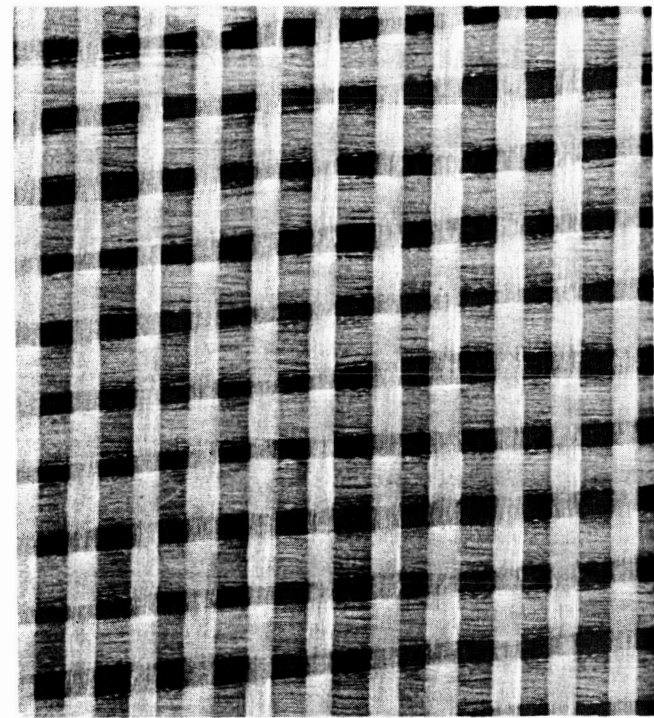
1562 FABRIC



104 FABRIC

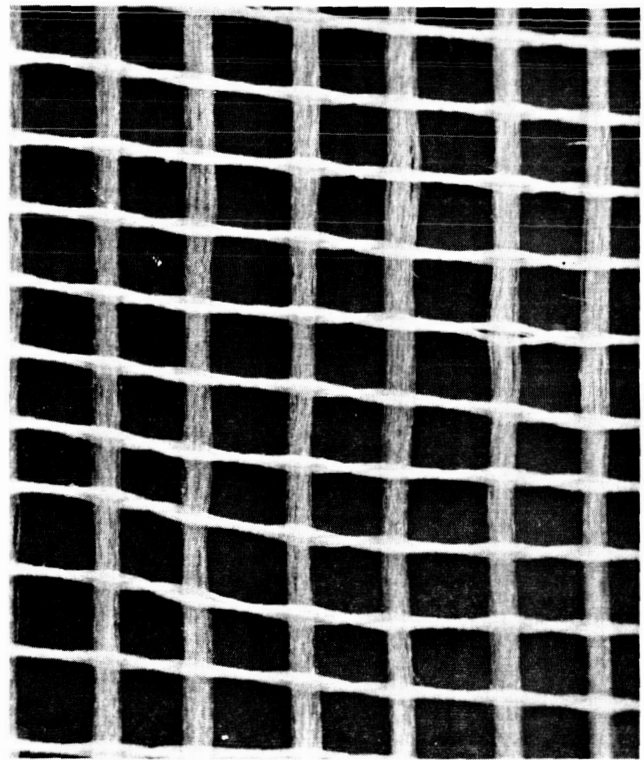
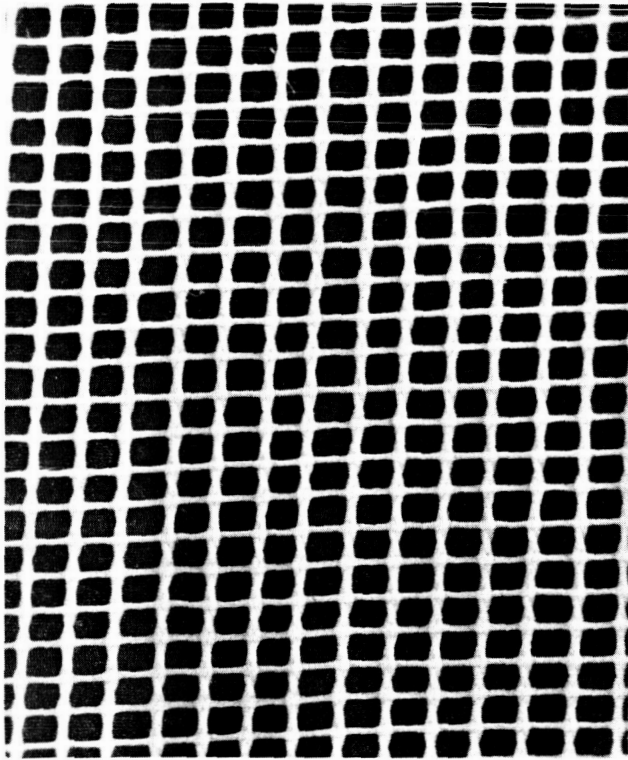


108 FABRIC



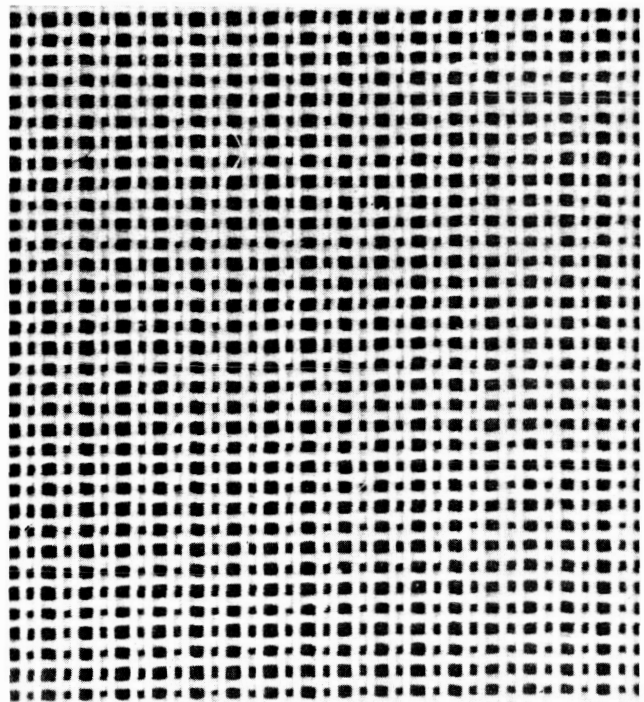
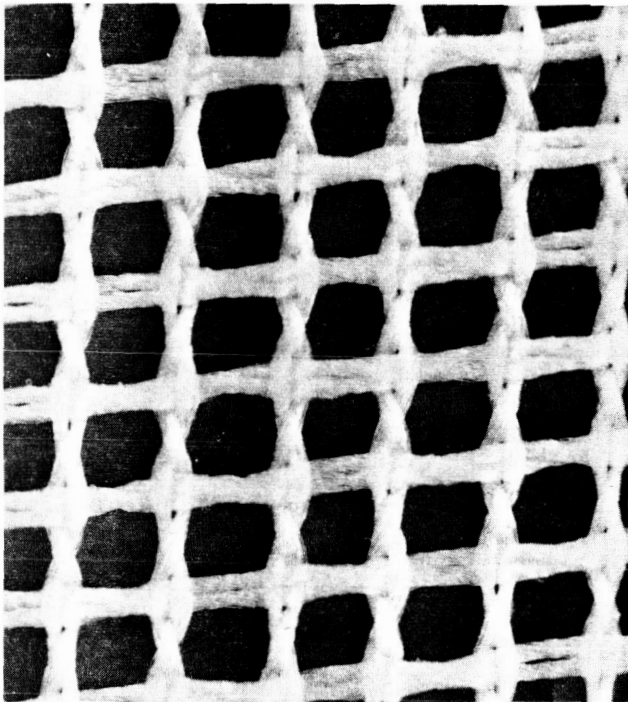
1610 FABRIC

FIGURE 1. Photomicrographs of Four Woven Glass Fabrics



15320 DACRON

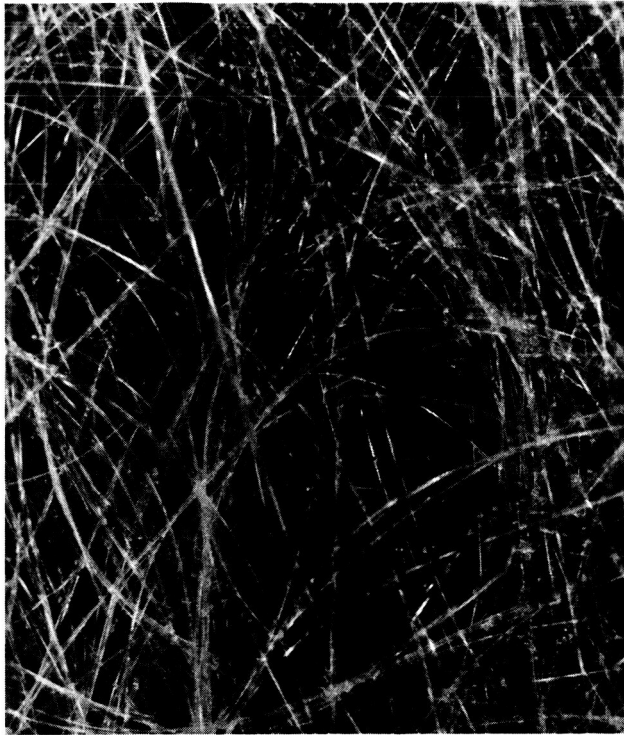
A-2827 NYLON



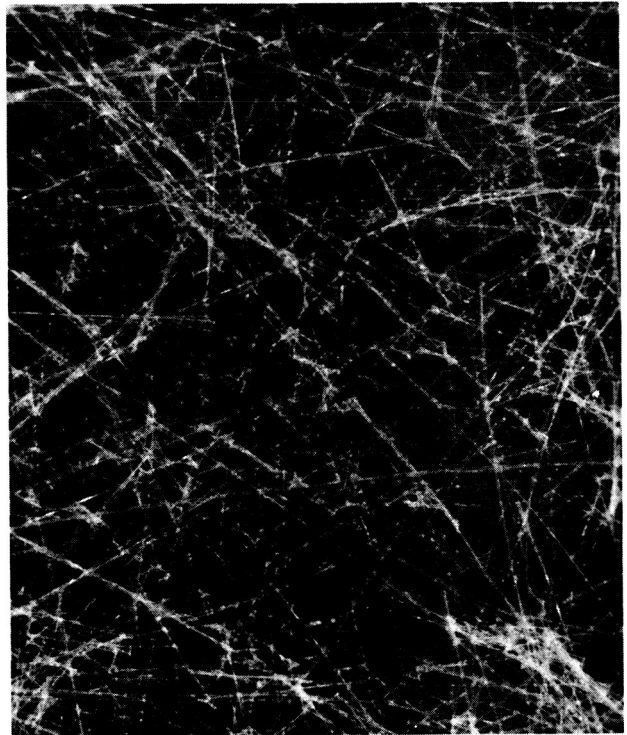
38068 NYLON

15041 DACRON

FIGURE 2. Photomicrographs of Four Woven Organic Fabrics



MODIGLASS SSM (GLASS)



MAT 236 (GLASS)

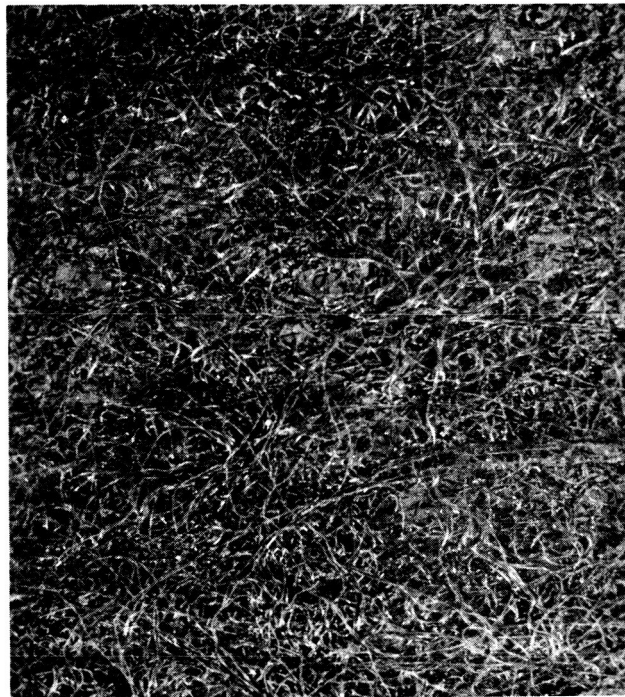


FIGURE 3. Photomicrographs of Three Nonwoven Mats

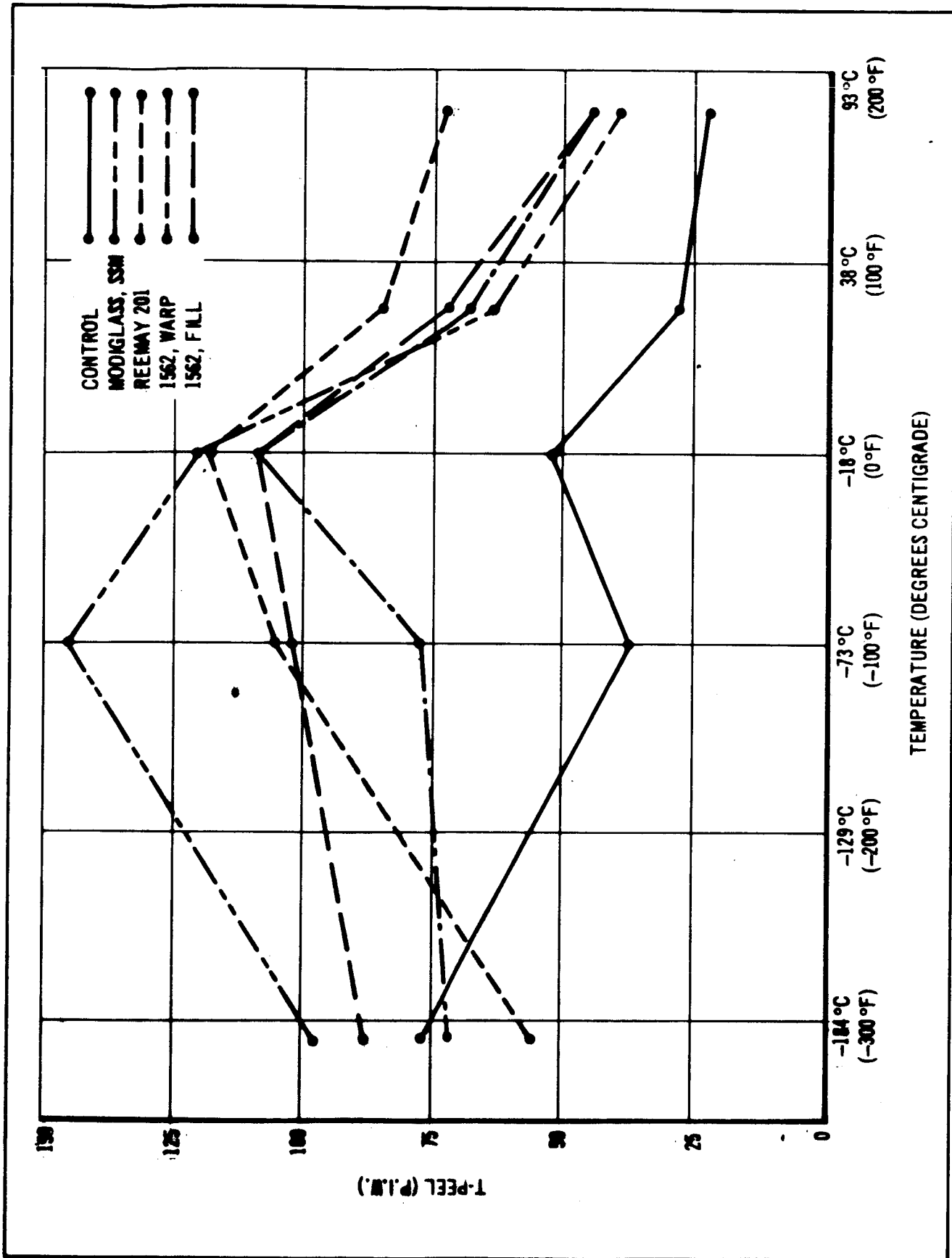
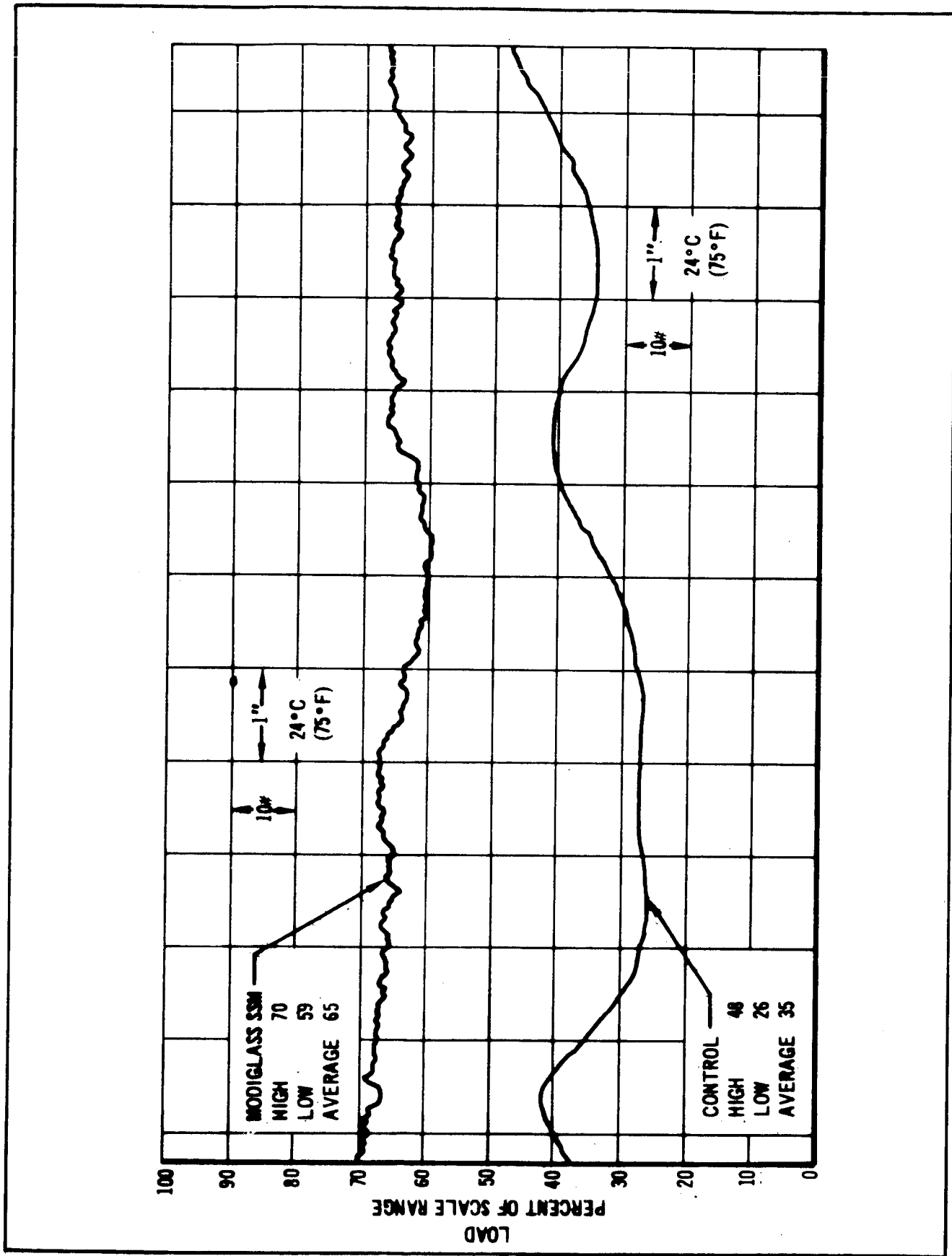
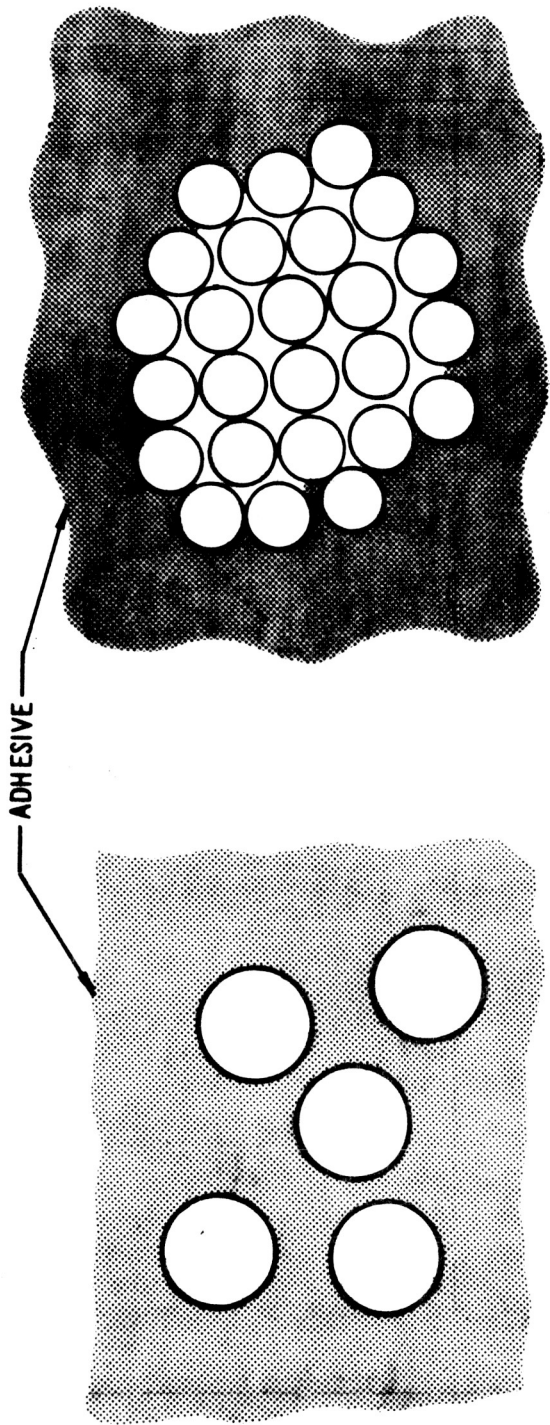


FIGURE 4. Comparison of T-Peel Values at Five Temperatures



..FIGURE 5. Comparison of T-Peel Charts on Control and Modiglass SSM Variation



DIAGRAMATIC CROSS-SECTION OF
RANDOM SINGLE FILAMENTS

A

DIAGRAMATIC CROSS-SECTION OF
MULTIFILAMENT THREAD

B

FIGURE 6. Comparison of Single Filament vs. Multifilament Composites

APPENDICES

APPENDIX 1

GLASS FABRICS*

TYPE (WOVEN)	THICKNESS IN MILLS.	WT. IN GMS/SQ. FT.	INDIVIDUAL FIBER DIAM. IN INCHES	THREAD WARP	COUNT FILL	NO. OF FIBERS PER STRAND WARP FILL	WEAVE	FINISH
104	1	1.8	0.00020-0.00025	60	52	102 51	Plain	A-1100
108	2	4.5	0.00020-0.00025	60	47	102 102	Plain	A-1100
1610	4	7.2	0.00035-0.00040	32	28	204 204	Plain	A-1100
1562	5	6.0	0.00035-0.00040	32	16	204 204	Leno	A-1100
(NON-WOVEN)								
Modiglass SSM	10	3.8	0.00070	--	--	-- --	Non- Woven	A-137
Modiglass Veil	Variable	5.2	0.00070	--	--	-- --	Non- Woven	A-137
Mat 236	10	2.8	0.00060	--	--	-- --	Non- Woven	Silane

*Vendor Data

APPENDIX 2

ORGANIC FABRICS*

TYPE (WOVEN)	THICKNESS IN MILS.	WT. IN GMS/SQ.FT.	THREAD WARP	COUNT FILL	WEAVE	FINISH	YARN DENIER WARP	YARN DENIER FILL
Dacron, 15041/1	3.8	3.3	106	92	Plain	None	40	40
Nylon, A-2827/4`	4.1	1.9	50	19	Leno	None	40	100
Dacron, 15320	4.6	2.6	86	55	Leno	None	40	40
Nylon, 38068	15.0	8.5	35	19	Leno	None	260	260/2
Nomex, HT-102-42	4.6	6.6	80	80	Plain	--	--	--
Nomex, HT-65-30	9.0	6.9	50	30	Plain	--	--	--
Cotton	6.0	4.9	72	72	Plain	Starch		
(NON-WOVEN)								
Reemay 200, Dacron, White	12.0	4.0			--	None	Fiber Denier not available	
Reemay 201, Dacron, Black	10.0	4.0			--	None	Fiber Denier not available	
Nylon, XS-144415	10.0	15.0			--	Trace, Oil	5 Denier Fibers	

*Vendor Data

APPENDIX 3

MISCELLANEOUS FIBROUS MATERIALS*

	THICKNESS IN MILLS.	WT. IN GMS/SQ.FT.	COMMENTS
Cellulosic Paper, Disposable	3	3.0	Low porosity
Nomex Paper, E51, Uncalendered	5	3.8	Low porosity
Asbestos Paper, Noramite C10-V30	10	--	Low porosity
Milled Glass Fibers, 1/8" No. 701	0.00035-0.00040 inches (Av. Fiber Diam.)	--	E Type glass, trace of Cationic finish
Screen, Window, Aluminum	26	19.6	Warp 17 - Fill 19
Asbestos, Blue, Fibers	1×10^{-5} inches (Av. Fiber Diam.)	--	Crocidolite type, African $NA_2O_3 \cdot FeO \cdot Fe_2O_3 \cdot 8SiO_2 \cdot H_2O$
Douglas Composite 11-W	15	16.4	One layer of 5 mil Mat 236 (Glass); and One layer of XS-144415 (Nylon)

*Vendor Data