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ASD TECHNICAL REPORT 61-300

THE PROPERTIES OF COMPRESSION MOLDED, GLASS FLAKE REINFORCED, RESIN COMPOSITES

Albert J. Luirette

Narmco Industries, Inc.
Research & Development Division

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JUNE 1961

AERONAUTICAL SYSTEMS DIVISION

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Albert J. Luirette

Narmco Industries, Inc.
Research & Development Division

JUNE 1961

Directorate of Materials and Processes
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AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

FOREWORD

This report was prepared by Narmco Industries, Inc., Research & Development Division, San Diego, California, under Air Force Contract No. AF 33(616)-7195. This contract was initiated under Project No. 7381, "Molded Glass Flake Reinforced Composites," Task No. 738103. The work was administered under the direction of the Directorate of Materials and Processes, Aeronautical Systems Division, with Marvin Knight as Project Engineer.

This report covers work conducted from 1 April 1960 to 1 June 1961.

ABSTRACT

Four molding compositions using glass flake as reinforcement were selected for study by means of a series of screening tests. The physical, electrical and mechanical properties of the compositions after compression molding into flat laminates were determined. Simple geometric shapes such as a hemisphere were compression molded from these compositions. The properties of the simple shapes were compared to the properties of the flat laminates.

It was observed that movement of the resin-flake mixture in the molds resulted in very poor alignment of the flakes within the molding. This improper alignment resulted in significantly reduced mechanical properties. It was concluded that this problem is inherent to presently available glass flake reinforced molding compounds and will prohibit the satisfactory compression molding of everything but extremely simple shapes such as flat plates using available molding compounds and molding techniques.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

W. P. Conrardy

W. P. CONRARDY
Chief, Materials Engineering Branch
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INTRODUCTION

The ultimate goal of this program was to supply, in a convenient form, sufficient data on the physical, mechanical, and electrical properties of compression molded resin-glass flake composites to allow design engineers to incorporate such moldings in the design of a weapon system.

The program was divided into two main phases. In the first phase, the effect of changes in processing parameters and of environmental exposures on the properties of flat laminates was determined. In the second phase, simple geometric shapes were compression molded and the properties of these shapes were compared to the properties of flat laminates.

SECTION I SCREENING TESTS

1.0 SCOPE

The screening tests described below were intended primarily to select the molding compounds for further evaluation. In addition, these tests provided an opportunity to evaluate the effect of changes in processing parameters such as molding temperature, time and pressure. This evaluation was useful in selecting a set of satisfactory molding conditions for each material.

Molding compounds were evaluated by comparing the results of flexural strength tests (modulus of rupture in bending tests).

2.0 DESCRIPTION OF THE MOLDING COMPOUNDS

Six compounds were selected for evaluation by this program from a series of Narmco Industries, Inc's. proprietary molding compounds. All of these compounds were based on the glass flake produced by Owens-Corning Fiberglas Corporation, New York, New York and marketed under the trade name, Filmglas.

Although compositions that contain both the two and four microns thick flakes were eventually evaluated, the screening tests were limited to those compositions which contained 70 percent by weight, random size, two microns thick, untreated Composition E, Filmglas.

The six composites selected for the screening tests were based on the types of resins normally associated with fiberglass fabric laminates. These are listed below:

1. X270 - A composition containing an anhydride-cured, elevated temperature resistant modified epoxy resin, supplied by Union Carbide Chemical Co. under the trade name of Unox Polyepoxide P-71.

2. X271 - A composition containing an aromatic amine-cured, elevated temperature resistant modified epoxy resin, supplied by Narmco Materials Div., Telecomputing Corp. under the designation of NRC 1174/3 Resin solution.

3. X272 - A composition based on a low cost, general purpose epoxy resin compounded from Shell Chemical Co's Epon 1002 containing 5.3 part per hundred by weight of pyromellitic dianhydride.

4. X273 - A composition using Resinox RI 4009, an elevated temperature resistant silicone based resin supplied by Dow Corning Chemical Co.

5. X274 - A composition containing R-5071, an elevated temperature resistant silicone based resin supplied by Dow Corning Chemical Co.

6. X275 - A composition based on Hetron 19, a general purpose polyester resin from Hooker Electrochemical Co. The resin was catalyzed with 6 percent by weight of Cadox BCP Benzoyl peroxide catalyst no. 55. Cadox, a powdered mixture of 35 percent by weight of benzoyl peroxide in calcium phosphate, is supplied by the Cadet Chemical Co.

These resins, except for the NRC 1174/3, are normally solids at room temperature. The NRC 1174/3 was reduced to a solid by evaporating the solvent solution from a water-bath at 200°F. The solids were ground and classified through a U. S. standard 100 mesh screen.

An intimate mixture between the resin and flakes was prepared by tumbling 70 parts by weight of random size flakes with 30 parts by weight of the powdered resin in conventional ball milling equipment for 30 minutes. Tumbling was followed by twenty minutes on a conventional paint shaker. Although the ratio of flakes to resin and the size of flakes used varied from the above description for some of the subsequent phases of this program, resin composition and mixing techniques remained essentially as described above.

3.0 DESCRIPTION OF MOLDING EQUIPMENT

3.1 Press

All molding was performed in the 75 ton, double acting, hydraulic press shown in Figure 1. This press is a modification of the Model H-8, 70 ton hydraulic press manufactured by Pasadena Hydraulics, Inc.

The press was equipped with 18-1/2" x 18-1/2" electrically heated and water cooled platens for use to 500°F.

3.2 Molds

Two molds were employed for the screening tests. The first, an 8" x 8" case-hardened mild steel mold, was an item of Narmco's equipment that was pressed into use to avoid delays at the beginning of this program (see Figure 2). Approximately 0.015 inches clearance per side was present between the plunger and the sides of this mold. The mold was only used until the second mold became available. The second mold was a 6" x 6" heat treated S.A.E. 4340 steel mold designed specifically for this task (see Figure 3). The plunger was fitted to have 0.003 to 0.005 inches clearance per side from the mold body. Neither mold was equipped with knock-out pins or similar devices to facilitate the removal of moldings and both molds required disassembling for removal of the finished molding.

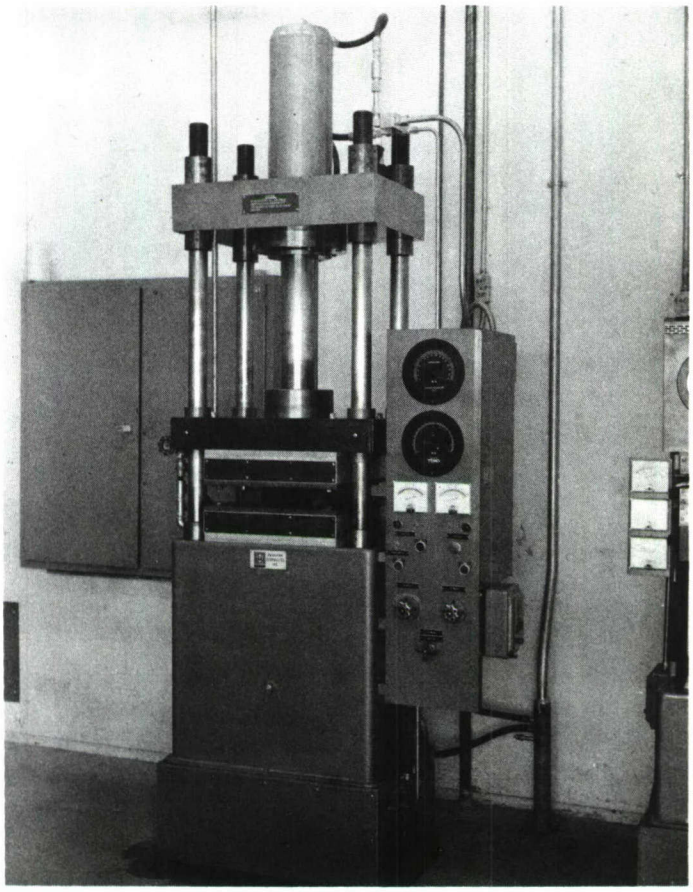


Figure 1 - The 75 Ton Hydraulic
Molding Press

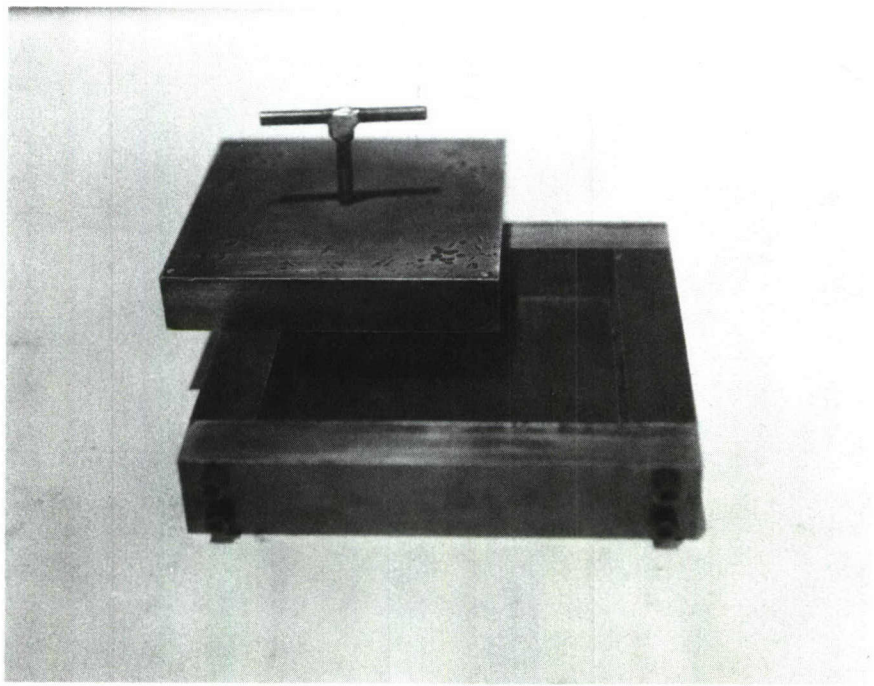


Figure 2 - Eight Inch Mold

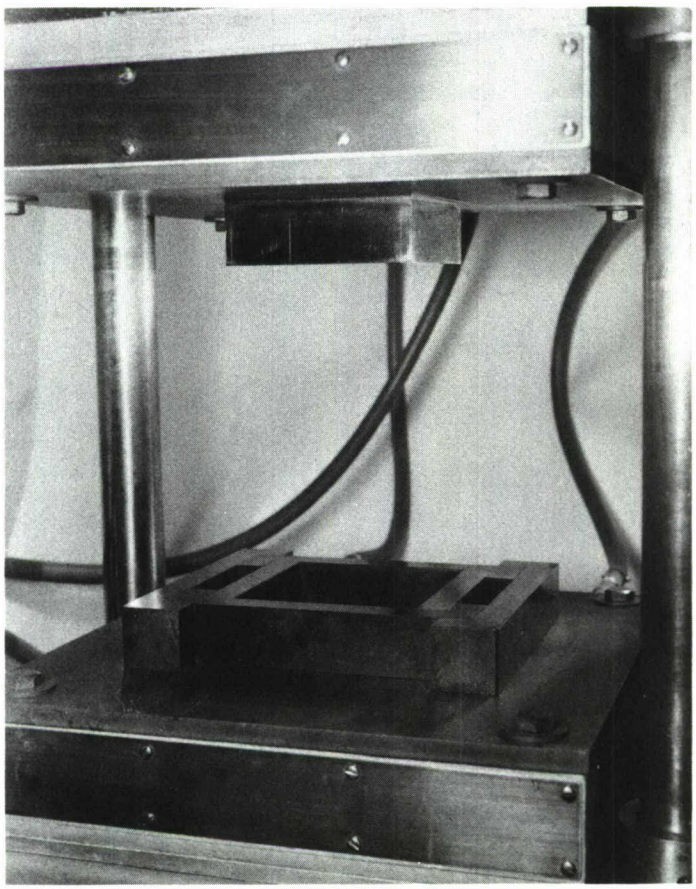


Figure 3 - Six Inch Mold Mounted
In The Molding Press

4.0 GENERAL MOLDING PROCEDURES

Certain portions of the molding sequence remained the same regardless of the type of resin that was used in the molding compound. So that these processes can be conveniently referred to throughout the balance of this report, and to insure that all of the terms used in this report are well defined, a discussion of the molding processes is presented below.

1. The mold was heated to the temperature required for the first step of the cure cycle.

2. The molding composition was charged into the mold. The compound was either charged directly into the mold or preformed into a bricket and thus charged into the mold. The term "preform" refers to a process for reducing the bulk of the molding composition by compressing the composition into a shape similar to that of the finished molded article.

   This operation was performed in the actual compression mold or in a similar mold at 200°F. This temperature was sufficiently high to reduce the viscosity of the resin but was well below the curing temperature. A pressure of 100 to 300 psi was used.

3. The mold was closed and a pressure of 10 psi was applied. The low pressure and the cure temperature were maintained for a measured period of time to advance the polymerization of the resin and to prevent excessive flow. (The period of time at low pressure and cure temperature is subsequently referred to as contact time.)

4. Full molding pressure was applied and the cure cycle was completed.

5. If required, the mold was cooled before the molded article was removed. DC 20, a silicone based releasing agent produced by Dow Corning Corp., Midland, Michigan, was used and performed very well with the 6" moldings except for some of the long cure cycles at 500°F.

5.0 SPECIFIC MOLDING PROCEDURES FOR THE INDIVIDUAL MOLDING COMPOUNDS

The description presented above applies to the general techniques that were used for all of the compositions under consideration. Specific procedures for the individual molding composition necessarily varied with each resin system. Details of these procedures are presented below:

5.1 X270 Molding Compound

Previous independent work at Narmco showed that satisfactory moldings could be made with this composition if a cure cycle of 2 hours at 350°F and 800 psi was used following a contact time of 2 minutes. Preforms were not required. This procedure was selected as standard and two 8" x 8" laminates were prepared.

Nine additional 8" x 8" laminates were prepared using various pressures, lengths of cure, contact times, and preform sizes. These laminates were compared with the original two. (See Table 2 for details of the process changes.)

A new set of standard conditions was selected on the basis of these results, namely, preformed material, cured for 2 hours at 350°F and 800 psi, using no contact time (see Table 1).

Five additional laminates were prepared using these conditions. Two of these were post cured to 400°F.

5.2 X271 Molding Compound

A standard cure cycle of 1 hour at 330°F and 800 psi with no contact time and using preformed material was selected on the basis of previous independent development work. Four laminates were prepared. Contact time, preform size, pressure, and cure time were varied and ten additional laminates were prepared for comparison with the original four (see Tables 3 and 4).

It was apparent from this comparison that more satisfactory laminates were prepared from preforms cured for 2 hours at 330°F and 800 psi after a contact time of 2 minutes. Four additional laminates were prepared to confirm these results. Two of these were post cured at 400°F.

5.3 X272 Molding Compound

A cure cycle for non-preformed material of 30 minutes at 350°F and 800 psi after a 2 minute contact time was selected as standard and three laminates were prepared.

In addition, three laminates were prepared to determine the effect of contact time and two laminates were prepared to evaluate the effect of preforming and of cure time (see Tables 5 and 6).

It was necessary to cool all of these laminates to 120°F or less before the release of pressure to prevent blowing or delamination.

5.4 X273 Molding Compound

A three-laminate series of standards was prepared using a cure cycle of 1 hour each at 250°F, 350°F, and 400°F and a pressure of 1600 psi without the use of preforms or contact time. Eleven laminates which were molded using various pressures, temperatures, contact times, cure times, and preform sizes were compared with the standards (see Tables 7 and 8). A problem of delamination or blowing during post cure was noted that seemed to be associated with the amount of volatile materials present in the molding composition. Seven laminates were prepared from three batches of molding compounds containing various volatile contents. Four laminates were prepared using the set of conditions that the original twenty laminates had indicated were most satisfactory, namely, 1 hour at 250°F, 1 hour at 350°F and 1 hour at 400°F at a pressure of 100 psi. Two

laminates were made with preforms and two without preforms, as the original data did not give sufficient indication as to which condition was more satisfactory.

All of the phenolic moldings were cooled to 120°F or less before the pressure was released.

5.5 X274 Molding Compound

Eight laminates, which were made using various contact times and cure times (see Tables 9 and 10), were compared with four laminates made under conditions which had been shown to yield satisfactory laminates, namely, a cure of preformed material for four hours at 500°F and 1000 psi after 10 minutes contact time at 500°F. This comparison indicated that the following was a more satisfactory cure:

30 minutes at 350°F and contact pressure
30 minutes at 350°F and 1000 psi
12 hours at 500°F and 1000 psi

Four laminates using this cycle were prepared and tested.

5.6 X275 Molding Compound

Little previous experience was available for this system. Therefore a cure cycle of 1 hour at 250°F and 1600 psi for unpreformed material was somewhat arbitrarily selected. The first laminate produced with this cycle delaminated badly when the molding pressure was released. All further laminates were cooled to 120°F or less before molding pressure was released. Only two laminates were prepared and as testing indicated a low flexural strength for this material, no attempt was made to find a more satisfactory cycle.

6.0 TEST PROCEDURES

6.1 Sample Preparation

Flexural test specimens, 1/2" x 4", were cut from the laminate samples with a diamond abrasive cut-off wheel on a table saw. Water was used as a coolant to prevent overheating during the sawing operation.

6.2 Flexural Testing

Ultimate flexural strength and the modulus of elasticity in bending were determined in accordance with Federal Specification L-P-406b, Method 1031, except that a nominal specimen width of 1/2" was used. Samples were tested on the 200 pound range of a 20,000 pound Tinius-Olsen Testing Machine. Calculation of flexural strength or maximum fiber stress was from the formula:

$$S = \frac{3\,PL}{2\,bd^2}$$

S = maximum fiber stress (psi)
P = load, pounds
L = distance between points of support, inches
b = width of beam, inches
d = depth of beam, inches

and the flexural modulus or modulus of elasticity in bending was from the formula:

$$E_B = \frac{L^3}{4\,bd^3} \quad (P/Y)$$

E_B = modulus of elasticity in bending (psi)
L = distance between points of support, inches
b = width of beam, inches
d = depth of beam, inches
P/Y = slope of the straight line portion of the load-deflection curve in pounds/inch

The estimated standard deviation for both flexural strength and modulus was calculated from the formula:

$$\sigma^2 \text{ estimated} = \frac{\sum_{i=1}^{N} (X_i)^2 - \frac{\left(\sum_{i=1}^{N} X_i\right)^2}{N}}{N - 1}$$

σ = standard deviation
N = number of samples

6.3 Resin Content

Resin content was determined by observation of the weight loss that occurred when a weighed sample was exposed in air to 1050± 50°F for two hours. Resin content was then calculated from:

$$\% \text{ Resin} = \frac{WT_{original} - WT_{\text{after burnout}}}{WT_{original}} \times 100$$

The silicone based resin leaves a residue after ignition. An attempt was made to correct weight loss by multiplication with a factor determined from the

weight of the residue which remained when a sample of pure resin was ignited. This technique did not prove reliable, as abnormally low results were obtained. For this reason, density measurements were substituted for the resin content determinations on the X274 system in subsequent work. The nominal composition of the original silicone resin-flake was reported for laminates tested during the screening tests.

7.0 TEST RESULTS

The results of the screening tests appear in Tables 1 - 10

TABLE I THE EFFECT OF PROCESSING VARIABLES ON THE ULTIMATE FLEXURAL STRENGTH AND MODULUS OF X270 EPOXY MOLDINGS

Sample No.	Process Variables	No. of Specimens	Average Ultimate Flexural Strength psi	Standard Deviation of Ultimate Flexural Strength psi	Average Flexural Modulus psi x 10^{-6}	Standard Deviation of Flexural Modulus psi x 10^{-6}
6	4 minute contact time	6	44,400	1,200	6.3	0.2
9	3 minute contact time	6	42,800	800	6.6	0.1
12	600 psi	6	42,700	1,800	5.5	0.1
7	Standard	6	42,000	2,200	5.4	0.2
10	No contact time	6	41,900	900	5.8	0.2
15	1000 psi	6	40,400	2,900	6.2	0.1
17	30 minute cure	6	38,900	2,400	5.9	0.2
11,13	Full size preform	12	37,300	4,500	5.5	0.4
14	Under size preform	6	37,000	1,100	5.5	0.4
Z,20R, 21	6" x 6" mold new standard conditions	26	36,400	1,242	5.2	0.3

TABLE 2 A DESCRIPTION OF THE MOLDINGS MADE WITH X270 EPOXY MOLDING COMPOUND

Sample No.	Final Resin Content % by Wt.	Molding Parameters			Cure Cycle			Appearance	Comments
		Contact Time	Preforms	Mold	Temp.	Time	Pressure psi		
6	22.3	4 min	None	8"x8"	350°F	2 hrs	800	Poor; wrinkles and dry areas at the corners	
7	28.6	2 min	None	"	"	"	"	Fair; many wrinkles	
8	-	2 min	None	"	"	"	"	Poor; many wrinkles, cloudy areas, dry corners	Incorrectly molded sample discarded
9	25.2	3 min	None	"	"	"	"	Poor; dry spots throughout, wrinkles, freckles	
10	28.4	None	None	"	"	"	"	Fair, cloudy spots	
11	29.5	2 min	Full size	"	"	"	"	Fair; freckles, large cloudy areas	
12	30.4	None	None	"	"	"	600	Fair; small blown areas, small dry corners	
13	29.8	2 min	Full size	"	"	"	"	Good; many freckles, dry corners	
14	26.7	"	Undersize	"	"	"	"	Good; many freckles, many wrinkles	
15	26.4	"	None	"	"	"	1000	Poor; dry flake scattered throughout	
16	-	None	Full size	"	"	1/2 hr	800	Poor; many wrinkles and cloudy areas	Incorrectly tested, results discarded

(Continued on next page)

TABLE 2 A DESCRIPTION OF THE MOLDINGS MADE WITH X270 EPOXY MOLDING COMPOUND (Continued)

Sample No.	Final Resin Content % by Wt.	Molding Parameters			Cure Cycle			Appearance	Comments
		Contact Time	Preforms	Mold	Temp.	Time	Pressure psi		
17	26.0	None	Full size	8"x8"	350°F	1/2 hr	800	Fair; many freckles and wrinkles	
Z	34.5	"	"	6"x6"	"	2 hrs	"	Very good; dry corners	
19	21.1	2 min	"	"	"	"	"	Fair; many wrinkles	
20	N.D.	None	"	"	"	"	"	Good; few wrinkles	Incorrectly post cured, sample discarded
21	22.6	"	"	"	"	"	"	Good; few wrinkles	
22	N.D.	"	"	"	"	"	"	Good; few wrinkles	Delaminated when post cured 1 hour each at 200°F, 250°F, 300°F, 350°F, and 400°F. Discarded
23	N.D.	"	"	"	"	"	"	Good; few wrinkles	Incorrectly post cured, sample discarded
20R	31.07	"	"	"	"	"	"	Very good	
23R	N.D.	"	"	"	"	"	"	Very good	Same as #22

NOTE:

1. A formulation error in the resin used to prepare samples 1-5 was noted and these samples were discarded.
2. Numbers 20R and 23R were replacement for laminates which were post cured incorrectly.

TABLE 3 THE EFFECT OF PROCESSING VARIABLES ON THE ULTIMATE FLEXURAL STRENGTH AND MODULUS OF X271 EPOXY MOLDINGS

Sample No.	Process Variables	No. of Specimens	Average Ultimate Flexural Strength psi	Standard Deviation of Ultimate Flexural Strength psi	Average Flexural Modulus psi x 10^{-6}	Standard Deviation of Flexural Modulus psi x 10^{-6}
1	Full size preform	10	37,800	2600	5.0	0.3
6	2 min. contact time	9	36,700	940	5.0	0.1
7	4 min. contact time	6	35,000	2400	5.0	0.2
3	No preforms	10	34,900	3400	5.1	0.2
8	1600 psi	10	34,800	2700	4.5	0.1
12	2 hour cure	10	34,400	1400	4.7	0.04
10	1/2 hour cure	10	33,700	5400	5.2	0.1
4,5,13, 14	Standard	31	33,700	2900	4.6	0.4
9	400 psi	10	32,400	3100	5.1	0.1
11	1/4 hour cure	10	30,900	1800	5.4	0.2
2	1/4" undersize preforms	10	23,700	6300	4.7	0.6
15,16	New standard with post cure	18	27,900	3600	4.2	0.3
17,18	New standard for 6" x 6" mold	20	34,000	2400	5.6	0.2

TABLE 4 A DESCRIPTION OF THE MOLDINGS MADE WITH X271 EPOXY MOLDING COMPOUND

Sample No.	Final Resin Content % by Wt.	Molding Parameters			Cure Cycle			Appearance
		Contact Time	Preforms	Mold	Temp.	Time	Pressure psi	
1	28.5	None	Yes	8"x8"	330°F	1 hr	800	Good; a few dry flakes
2	28.2	"	"	"	"	"	"	Poor; many large wrinkles
3	28.2	"	None	"	"	"	"	Fair; freckled
4	30.1	"	Yes	"	"	"	"	Good; small wrinkles
5	29.8	"	"	"	"	"	"	Good; dry corners
6	29.1	2 min	"	"	"	"	"	Good; dry corners
7	28.6	4 min	"	"	"	"	"	Poor; large dry corners
8	28.8	None	"	"	"	"	1600	Fair; large wrinkles and freckles
9	27.7	"	"	"	"	"	400	Poor; dry corners and edges, freckled
10	28.8	"	"	"	"	1/2 hr	800	Fair; few wrinkles
11	27.4	"	"	"	"	1/4 hr	"	Poor; dry edges and corners, freckles, wrinkles
12	29.6	"	"	"	"	2 hrs	"	Good; some cloudy areas, no freckles
13	29.6	"	"	"	"	1 hr	"	Good; center very clear, corners cloudy
14	29.0	"	"	"	"	"	"	Good; center very clear, corners cloudy
15	N.D.	2 min	"	6"x6"	"	2 hrs	"	Very good; center clear, few wrinkles on edge
16	N.D.	"	"	"	"	"	"	" " " "
17	27.9	"	"	"	"	"	"	" " " "
18	27.0	"	"	"	"	"	"	" " " "

TABLE 5 THE EFFECT OF PROCESSING VARIABLES ON THE ULTIMATE FLEXURAL STRENGTH AND MODULUS OF X272 EPOXY MOLDINGS

Sample No.	Process Variables	No. of Specimens	Average Ultimate Flexural Strength psi	Standard Deviation of Ultimate Flexural Strength psi	Average Flexural Modulus psi $\times 10^{-6}$	Standard Deviation of Flexural Modulus psi $\times 10^{-6}$
8	Preforms	6	43,200	1400	5.3	0.2
6	4 minute contact time	5	39,600	2600	5.3	0.5
5,11	Standard 2 minute contact time	12	37,200	2400	5.3	0.2
9	3 minute contact time	6	35,400	1800	5.5	0.1
7	1 hour cure	-	Discarded	-	-	-
12	1 minute contact time	-	Discarded	-	-	-

TABLE 6 A DESCRIPTION OF THE MOLDINGS MADE WITH X272 EPOXY MOLDING COMPOUND

Sample No.	Final Resin Content % by wt.	Molding Parameters			Cure Cycle			Appearance	Comments
		Contact Time	Preforms	Mold	Temp.	Time	Pressure psi		
5	31.0	2 min	None	8"x8"	350°F	1/2 hr	800	Poor; many wrinkles, general cloudy condition	
6	31.0	4 min	"	"	"	"	"	Fair; some dry flake areas, some wrinkles	
7	31.0	None	Yes	"	"	1 hr	"	Poor; many large wrinkles throughout	Laminate was too poor to test
8	26.9	2 min	"	"	"	1/2 hr	"	Good; small wrinkles at edges	
9	28.5	3 min	None	"	"	"	"	Very good; small dry flake areas and a few wrinkles	
10	N.D.	2 min	"	"	"	"	"	Poor; blown areas, wrinkles, dry flake areas	Laminate was too poor to test
11	29.9	"	"	"	"	"	"	Fair; relatively few wrinkles and few dry flake areas	
12	N.D.	1 min	"	"	"	"	"	Poor; blown areas and wrinkles throughout	Laminate was too poor to test

NOTE: Laminates 1-4 delaminated when molding pressure was released while mold was at 350°F. All other moldings were cooled to 120°F or less before the pressure was released.

TABLE 7 THE EFFECT OF PROCESSING VARIABLES ON THE ULTIMATE FLEXURAL STRENGTH AND MODULUS OF X273 PHENOLIC MOLDINGS

Sample No.	Process Variables	No. of Specimens	Average Ultimate Flexural Strength psi	Standard Deviation of Ultimate Flexural Strength psi	Average Flexural Modulus psix10^{-6}	Standard Deviation of Flexural Modulus psix10^{-6}	Average Ultimate Flexural Strength at 300°F psi	Average Ultimate Flexural Strength at 350°F psi	Average Ultimate Flexural Strength at Room Temp. After 1 Hr. at 350°F, psi
5	1000 psi	6	38,500	768	5.2	0.02	13,900	7,000	21,400
6	Cure cycle #3, 1000 psi	6	37,400	1600	-	-	12,600	8,000	18,900
9	Cure cycle #3	6	37,000	2500	5.1	0.3	14,700	8,700	17,100
7	Cure cycle #4, 1000 psi	6	35,800	6300	5.2	0.1	-	10,300	-
12, 13	800 psi	12	35,500	4300	5.3	0.2	14,100	19,500	15,300
11	Undersize preform	6	32,000	4900	7.9	0.7	11,400	9,000	15,200
1, 3, 4	Initial standard	18	30,100	4600	4.8	0.6	9,500	7,400	21,700
10	Full size preform	6	28,700	2900	4.8	0.2	7,900	5,400	13,200
14	10 minute contact time	6	27,300	2100	4.8	0.2	18,900	10,700	9,500
22, 23	New standard without preforms	20	27,100	9980	3.6	1.5	No post cured specimens		
24, 25	New standard with preforms	20	25,900	3500	3.9	0.5	No post cured specimens		
8	Cure cycle #5	6	25,800	5100	4.8	0.1	-	6,700	-
2	Cure cycle #2	6	23,300	3600	4.3	0.2	13,500	11,500	21,400
18, 19	Molding compound with a low volatile content	8	20,800	3800	3.5	2.4	Blown during post cure		
15, 17	Molding compound with a low volatile content	12	20,600	1600	4.5	0.2	Blown during post cure		
20	Dried molding compound *	6	17,600	268	2.7	0.1	Blown during post cure		
21	Dried molding compound**	6	15,100	1800	2.6	0.1	Blown during post cure		

NOTE: See Table 8 for a description of the cure cycles.

* See Note 2, Table 8

** See Note 3, Table 8

TABLE 8 A DESCRIPTION OF THE MOLDINGS MADE WITH X273 PHENOLIC MOLDING COMPOUND

Sample No.	Final Resin Content % by Wt.	Molding Parameters				Cure Cycle		Appearance
		Contact Time	Preforms	Mold	Volatile Content NOTE 1	Temp/Time	Pressure psi	
1	28.4	None	None	8"x8"	0.93	Cure #1	1600	Good; no freckles, some cloudy areas, few wrinkles
2	28.9	"	"	"	"	Cure #2	"	Good; no freckles, some dark areas, few wrinkles
3	26.9	"	"	"	1.26	Cure #1	"	Fair; freckles, small wrinkles
4	30.5	"	"	"	"	Cure #1	"	Poor; freckles, wrinkles
5	29.1	"	"	"	"	Cure #1	1000	Poor; wrinkles and many blown areas
6	29.2	"	"	"	"	Cure #3	"	Fair; small wrinkles
7	29.6	"	"	"	"	Cure #4	"	Fair; small wrinkles blown areas
8	29.1	"	"	"	"	Cure #5	1600	Poor; wrinkles, dry corners
9	29.7	"	"	"	"	Cure #3	"	Fair; many wrinkles
10	29.6	"	Yes	"	"	Cure #1	"	Good; few wrinkles
11	29.0	"	"	"	"	Cure #1	"	Good; few wrinkles
12	29.5	"	None	"	"	Cure #1	800	Fair; some wrinkles, dry corners
13	32.2	"	"	"	"	Cure #1	"	Fair; wrinkles and cloudy areas
14	29.4	10 min	"	"	"	Cure #1	1600	Poor; blown areas and wrinkles

(Continued on next page)

TABLE 8 A DESCRIPTION OF THE MOLDINGS MADE WITH X273 PHENOLIC MOLDING COMPOUND (Continued)

Sample No.	Final Resin Content % by Wt.	Molding Parameters				Cure Cycle		Appearance
		Contact Time	Preforms	Mold	Volatile Content NOTE 1	Temp/Time	Pressure psi	
15		None	None	6"x6"	0.84	Cure #1	1000	Good; few wrinkles
16	32.6	"	Yes	"	"	Cure #1	"	Good; few wrinkles
17	32.2	"	"	"	"	Cure #1	"	Good; few wrinkles
18	30.7	"	"	"	0.75	Cure #1	"	Fair; wrinkles
19	28.1	"	"	"	"	Cure #1	"	Fair; wrinkles
20	28.7	"	"	"	(NOTE 2)	Cure #1	1600	Poor; many wrinkles
21	27.9	"	"	"	(NOTE 3)	Cure #1	1000	Poor; many wrinkles
22	N.D.	"	None	"	1.26	Cure #1	"	Poor; many dry corners
23	N.D.	"	"	"	"	Cure #1	"	Poor; wrinkles, cloudy, dry corners
24	N.D.	"	Yes	"	"	Cure #1	"	Fair; wrinkles, cloudy
25	N.D.	"	"	"	"	Cure #1	"	Good; cloudy in spots, wrinkles at edge

NOTES:
1. Volatile content was percent weight loss when the molding compound was exposed to 350°F for 15 minutes.
2. Molding compound containing 1.26% volatiles was dried 1.0 hours at 230°-240°F.
3. Molding compound containing 1.26% volatiles was dried 1.5 hours at 230°-240°F.

Cure #1 - Stepped cure - 1 hour at 250°F, 1 hour at 300°F, and 1 hour at 400°F.
Cure #2 - Stepped cure - 1 hour at 250°F, 1 hour at 300°F, and 2 hours at 400°F.
Cure #3 - Stepped cure - 1 hour at 250°F, 2 hours at 300°F, and 1 hour at 400°F.
Cure #4 - Stepped cure - 1 hour at 250°F, 2 hours at 300°F, 1 hour at 400°F, and 1/2 hour at 450°F.
Cure #5 - Stepped cure - 2 hours at 250°F, 2 hours at 300°F, 1 hour at 400°F, 1 hour at 450°F, and 1 hour at 500°F.

TABLE 9 THE EFFECT OF PROCESSING VARIABLES ON THE ULTIMATE FLEXURAL STRENGTH AND MODULUS OF X274 SILICONE MOLDINGS

Sample No.	Process Variables	No. of Specimens	Average Ultimate Flexural Strength psi	Standard Deviation of Ultimate Flexural Strength psi	Average Flexural Modulus psix10^{-6}	Standard Deviation of Flexural Modulus psix10^{-6}	Average Ultimate Flexural Strength at 300°F psi	Average Ultimate Flexural Strength at 350°F psi	Average Ultimate Flexural Strength at Room Temperature After 1 Hr. at 350°F, psi
15	Step cure cycle #1	4	31,100	900	5.2	0.2	-	-	-
4	800 psi	6	27,500	2600	3.4	0.1	10,900	7000	9,100
16	Step cure cycle #2	4	26,600	1500	4.3	0.2	-	-	-
11, 6, 13	Step cure cycle #3	15	26,500	3300	4.8	0.5	5,670	5090	7,780
12,14	Mechanically vibrated to improve flake alignment	12	25,300	2200	5.7	1.2	10,800	5400	12,700
2,8,9	Standard	21	24,700	2900	4.4	0.4	11,500	7400	21,100
10	5 minute contact	6	24,500	1800	4.8	0.2	9,200	6200	11,600
5	20 minute contact	5	19,500	3400	4.7	0.4	11,200	7900	14,400

Cure #1 - Pressure 1000 psi, temperature 350°F for 1 hour, 500°F for 6 hours.
Cure #2 - Pressure 1000 psi, temperature 350°F for 1 hour, 500°F for 10 hours.
Cure #3 - Pressure 1000 psi, temperature 350°F for 30 minutes, 500°F for 2 hours.

TABLE 10 A DESCRIPTION OF THE MOLDINGS MADE WITH X274 SILICONE MOLDING COMPOUND

Sample	Final Resin Content % by Wt.	Molding Parameters			Cure Cycle			Appearance	Comments
		Contact Time	Preforms	Mold	Temp.	Time	Pressure psi		
2	30.0*	10 min	Yes	8"x8"	500°F	4 hrs	1000	Fair; wrinkles and dry flake	
3	"	15 min	"	"	"	"	"	Poor; wrinkles and dry flake	This laminate was too poor to test
4	"	"	None	"	"	"	800	Poor; wrinkles and dry flake	
5	"	20 min	"	"	"	"	1000	Poor; wrinkles and dry flake	
6	"	30 min	"	"	350°F 500°F	30 min 2 hrs	"	Good; few wrinkles and freckles	
7	"	10 min	Yes	"	500°F	4 hrs	"	Poor; wrinkles and dry flake	This laminate was too poor to test
8	"	10 min	"	"	"	"	"	Fair; wrinkles and dry flake	
9	"	"	"	6"x6"	"	"	"	Good; wrinkles at edges one blown area	
10	"	5 min	"	8"x8"	"	"	"	Poor; wrinkles	
11	"	30 min	None	"	350°F 500°F	30 min 2 hrs	"	Good; a few wrinkles and freckles	
12	"	20 min	"	"	500°F	4 hrs	"	Good; some dry flake and freckles	
13	"	30 min	Yes	6"x6"	350°F	30 min	"	Good; a few wrinkles and freckles	
14	"	15 min	None	8"x8"	500°F	8 hrs	"	Fair; dry flake and wrinkles	
15	"	1 hr	Yes	6"x6"	350°F 500°F	1 hr 6 hrs	" "	Good; a few wrinkles and freckles	
16	"	2 hrs	Yes	6"x6"	350°F 500°F	1 hr 10 hrs	"	Very good; very few wrinkles and freckles	

* Nominal composition of resin glass mixture

8.0 A DISCUSSION OF THE TEST RESULTS

Several phenomena were observed for all of the molding compounds and were not associated with any particular resin system. The first of these phenomena which was observed was the recurrence of flaws in the molded composites which resembled the wrinkles that are commonly seen in poorly fabricated fiberglass laminates. In general, these wrinkles were concentrated around the periphery of the laminates and were more numerous in laminates which were prepared in the 8" mold. This mold allowed a greater degree of flow of the molding composition during the cure cycle than did the more precisely machined 6" mold; hence, the occurrence of wrinkles seemed to be related to the degree of flow during the molding cycles. As these wrinkles continued to be a problem throughout the balance of this project, a more complete description of these flaws is in order at this time. Sections were cut from areas in a laminate that contained wrinkles and from a so-called "good" section of the same laminate. Photomicrographs of these cross sections appear in Figures 4 and 5. It is evident from these micrographs that the wrinkles are areas in which the flakes are very poorly aligned.

One other phenomena which was observed was that of blowing or delamination of the molded composite when the composites were exposed to elevated temperatures. This phenomena was first observed when an attempt was made to remove the moldings from their molds without cooling these molds below the molding temperature. The elevated temperature resistance of these moldings was one of the properties that was investigated at a later stage of this program, and so the blowing or delamination was cause for concern. To evaluate the extent of this problem, a short investigation of the room temperature strength of the composites after exposure to elevated temperatures was included in the screening tests.

8.1 The Epoxy System

A review of the data presented in Tables 1 through 6 indicated that essentially comparable ultimate flexural strengths were obtained with each of the epoxy compounds. There was some indication that higher strengths were attainable with either the X270 or X272 materials than were observed for the X271 material. However, upon closer examination, it was noted that these higher values were obtained from specimens prepared in the 8" mold. The 8" mold generally produced laminates which contained more flaws than did the 6" mold, but these wrinkles are concentrated around the periphery of the moldings. Thus, in the normal cutting of specimens from these laminates, the edges were removed and the effect of the flows were minimized. Another factor caused by the excessive flow in the 8" mold that influenced strengths was variations in resin contents of the finished laminates. In the 8" mold, more resin was allowed to flow from the composite, and significantly higher glass contents resulted. In a later section of this report, it is demonstrated that higher glass contents result in higher ultimate flexural strengths and moduli.

Thus the strength values alone were not sufficient criteria on which a selection of a molding system could be based and it was necessary to consider the over all appearance of the laminates. In addition, the handling characteristics of the various systems were considered. On this basis, the X270 system was selected as the epoxy resin which was used throughout the balance of this program. The tendency to

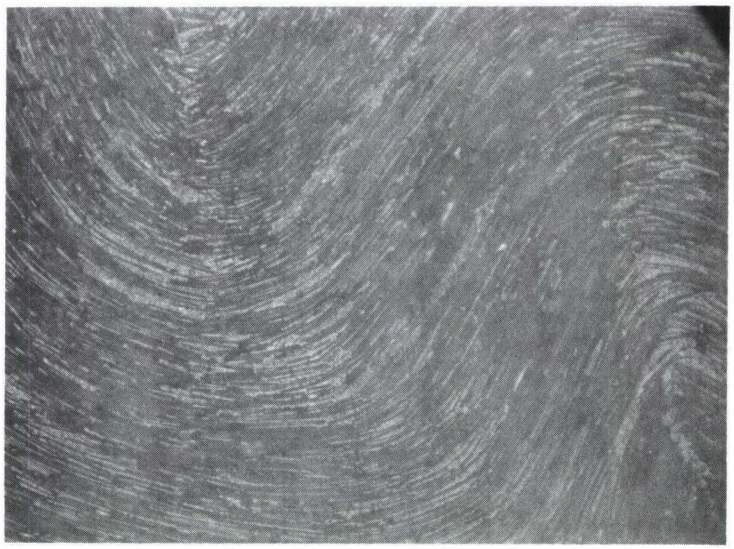


Figure 4 - Photomicrograph Of A Cross Section Of A Glass Flake Resin Composite Showing A "Wrinkled" Area

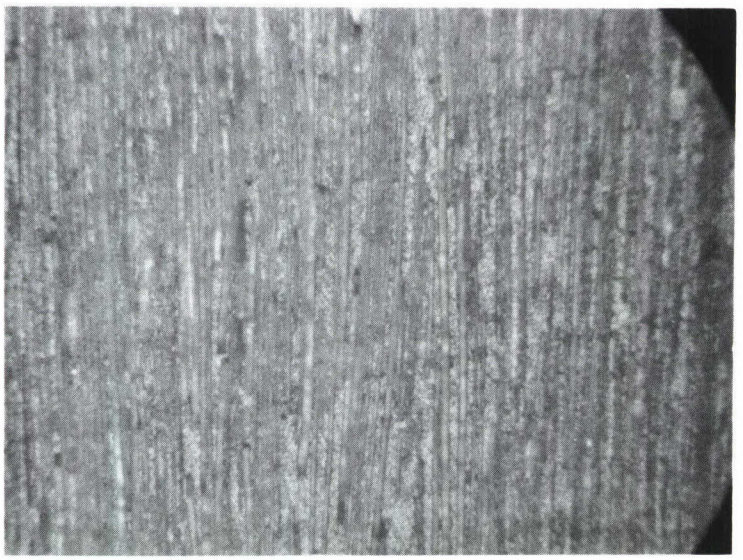


Figure 5 - Photomicrograph Of A Cross Section Of A Glass Flake - Resin Composite Showing Normal Flake Alignment

delaminate at elevated temperatures was more pronounced for the X270 system than for the X271 system. For this reason, the X271 system was not discarded at this time, but instead, was carried through the post cure and elevated temperature testing phases of this project.

It is interesting to note that the X272 system was not discarded because of any particular fault, but rather because it is less convenient to use than either of the other systems. As the three epoxy resins were selected as representatives of three very different classes of epoxies, this fact would indicate that the resin properties contribute only slightly to the mechanical strength of the composites.

8.2 The Phenolic Molding Compound

The problem of post cure as noted above, led to several additional tests of the X273 phenolic system; a flexural test at 300°F, at 350°F, and at room temperature, after a post cure of 1 hour at 350°F. The results of these tests are presented in Table 7 and indicate that the anticipated problem had occurred, i.e., the phenolic moldings tend to delaminate when exposed to elevated temperatures.

During the testing of the first fourteen laminates in this series (Table 8), it was noted that there seemed to be a relationship between the volatile content of the molding compound and the "blowing" problem; that is, laminates 1 and 2 which were made from a molding compound containing 0.9% volatiles did not show an appreciable change when subjected to a post cure of 1 hour at 350°F, while the best laminate made from a molding compound with a volatile content of 1.3% retained only approximately two-thirds of its original strength when subjected to the same conditions.

A short investigation was made of moldings prepared from molding compounds with very low volatile contents, but as the test results in Table 7 show (laminates 15-21 inclusive), the delamination problem was not a simple one to be solved merely by lowering the volatile content of the molding compound. This problem was more thoroughly investigated in a later phase of this program.

8.3 The Silicone Molding Compound

A series of tests similar to those used for the phenolic molding compound was performed on laminates molded with the X274 material. As shown in Table 9, similar results were obtained; that is, a definite reduction in flexural strength was observed when the X274 material was exposed to 350°F for 1 hour. It should be noted that as a matter of convenience, because of the large number of specimens required for the screening tests, the X274 moldings were cured for a relatively short time at 500°F, even though it was known that a longer cure at 500°F is normally required to develop the high temperature resistance of silicone resin.

Sufficiently high room temperature flexural strengths were developed using these shortened cures, warranting the adoption of the X274 material for the balance of this program.

8.4 X275, A Polyester Molding Compound

Although several attempts were made to produce the X275 moldings, only two of these moldings were sufficiently successful to warrant testing. Since the average room temperature flexural strength of these two moldings was only 7960 psi and because of the difficulties experienced to obtain even these values, it was apparent that the X275 was not a satisfactory material. This material was eliminated from further consideration.

SECTION II THE EFFECTS OF VARIATIONS IN COMPOSITION ON THE MECHANICAL PROPERTIES OF GLASS FLAKE REINFORCED COMPRESSION MOLDED COMPOSITES

1.0 SCOPE

This section of the report deals with results of a study to determine the effect of changes in size, thickness and concentration of glass flakes on the mechanical properties of glass flake reinforced compression molded composites. To reduce the number of samples required for this test, the assumption was made that the effect of the reinforcement would be independent of the resin used in the composite, and hence, the study was limited to the X270 system.

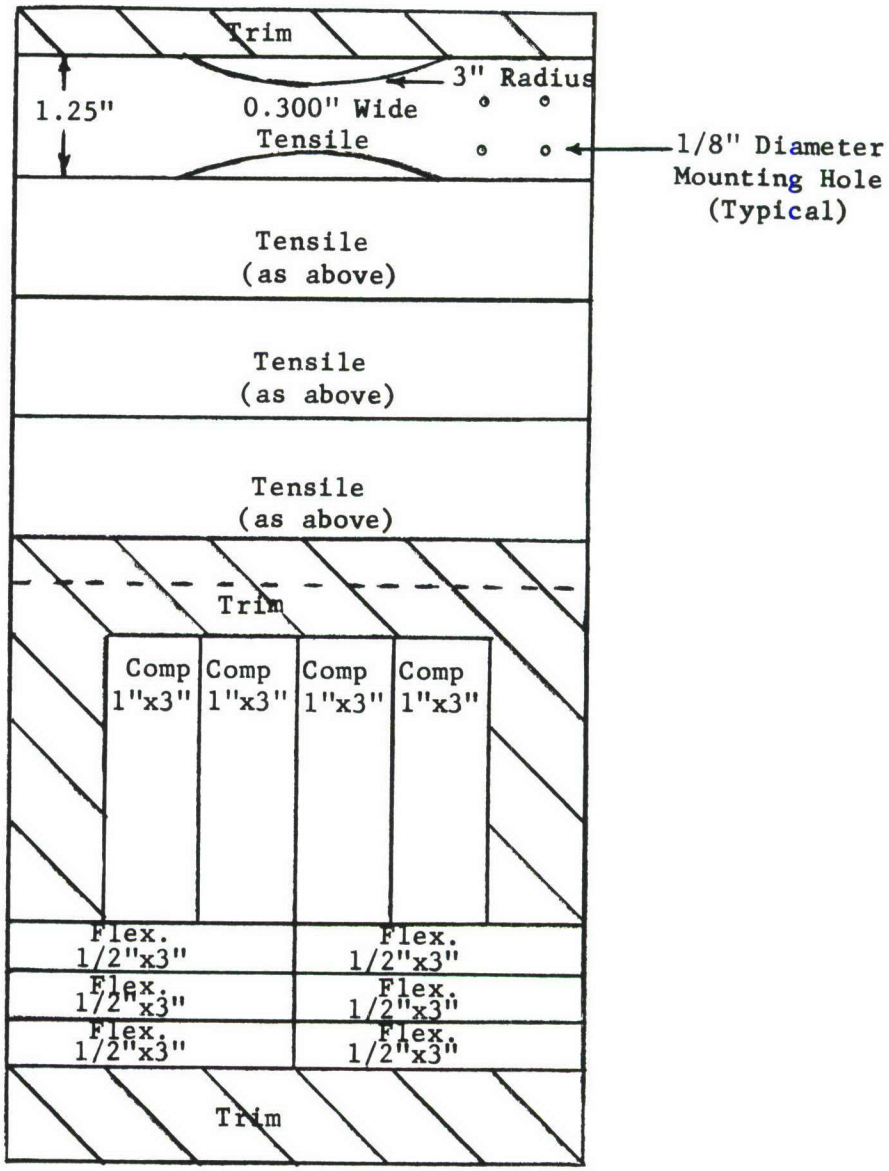
2.0 PREPARATION OF SAMPLES FOR THE STUDY OF FLAKE SIZE AND RESIN CONTENT

The effects of flake size and resin content on the mechanical properties of glass flake reinforced composites were based on the results of tensile, flexural, and compressive tests of specimens cut from 6" x 6" x 0.100" laminates in accordance with Figure 6. These laminates were prepared in accordance with the procedure described in the first section of this report, except that flakes were reduced to definite sizes by screening before they were incorporated into the molding composition. Sizing to within definite limits was accomplished by passing the flake through a screen of the desired mesh size and retaining the flake on a screen of the next smaller mesh size. For example, the flake referred to in this report as 5 mesh is actually that flake which will pass through a U. S. Standard 5 mesh screen but not through a 6 mesh screen. The following is a list of the size limits for the various assortments of flake that were used:

Nominal Mesh Size	Upper Size Limit		Lower Size Limit	
10 to 40	2000	(10 mesh)	420	(40 mesh)
35	500	(35 mesh)	420	(40 mesh)
18	1000	(18 mesh)	840	(20 mesh)
10	2000	(10 mesh)	1680	(12 mesh)
5	4000	(5 mesh)	3360	(6 mesh)

Both the 2 and 4 micron flakes were investigated. To compare the moldability of the various compositions, all were molded by the standard cycle for the X270 system, which was established during the screening tests, namely, cure of preformed material for two hours at 350°F and 800 psi following a contact time of 2.0 to 2.5 minutes. If, after two attempts to mold each composition by this cycle, it became apparent that satisfactory laminates could not be produced, this cycle was varied until good laminates were produced, or until it became apparent that the composition in question could not be conveniently molded.

Two laminates of each composition were prepared. One was used for flexural and compression samples and the other for the tensile tests. In addition, several duplicate laminates were prepared to demonstrate the reproducibility of the testing techniques. A description of each of these laminates appears in Tables 11 and 12.



NOTE: **When 6" x 6" laminates were used a separate laminate was required for tensiles and for compressions and flexures as indicated by the dotted line.**

Figure 6 - A Diagram Of Specimens Used For Studies Of The Effect Of Composition On The Mechanical Properties Of Flake Reinforced Laminates

3.0 TEST PROCEDURES

Mechanical testing with two exceptions, was performed in accordance with Federal Specification L-P-406b. The first exception was the use of non-standard tensile specimens, in that the 6" laminates were not sufficiently large to enable standard length specimens to be cut. The second exception was that a non-standard edgewise compression test of a supported 1" x 3" x 0.100" specimen was employed. This technique is described in detail in the control test section of this report. In addition to the mechanical tests, a standard burnout of a sample cut from each laminate was performed to determine the weight percentage of resin. The weight percentage of resin was converted into volumetric percentage of glass in the laminate from the graph shown in Figure 7. This graph was prepared from the theoretical relationships that exist between the weight percentage of resin, the laminate density, and the volumetric percentage of glass, if the assumption is made that the laminate consists only of resin and glass. Glass flake that remained after the resin was removed by ignition was carefully sorted through a set of analytical standard mesh screens, and the quantity of flake of each size found on these screens was compared with the original flake. The results of these comparisons appear in Table 13.

After the results of the tests to determine the ultimate tensile, flexural, and compressive strength were established, these data were graphed as a function of the volumetric percentage of resin in each specimen. As it appeared that a direct linear relationship existed between these properties and the glass content, a least-squares technique was employed to determine the parameters for the best possible straight lines through these points. The results of these calculations appear in Figures 8 through 17. An estimate of the standard deviations for these values was made, and the 95% confidence limits, which appear on the graphs, were calculated.

4.0 TEST RESULTS

The results of these tests appear either in Tables 11 through 13, or are presented graphically in Figures 9 through 17.

5.0 DISCUSSION OF THE TEST RESULTS

As would be anticipated, a direct linear relationship appears to exist between the amount of glass that is present in a glass flake reinforced laminate and the ultimate tensile and flexural strengths of the laminate. This relationship does not hold true for the compressive strength of the composite, and it appears that the compressive test is actually a measure of the properties of the resin itself rather than that of the composite. Although the ultimate compressive strength appears to be independent of the glass content of the laminates over the range under investigation, the modulus of elasticity in compression behaves in the same manner as the flexural modulus. This modulus appears to be directly related to the amount of glass in the composite. In all cases investigated, laminates reinforced with the 2 micron flakes showed mechanical properties superior to the properties of laminates reinforced with the 4 micron flakes. These results were not surprising, as it is generally accepted that thinner flakes possess superior physical properties.

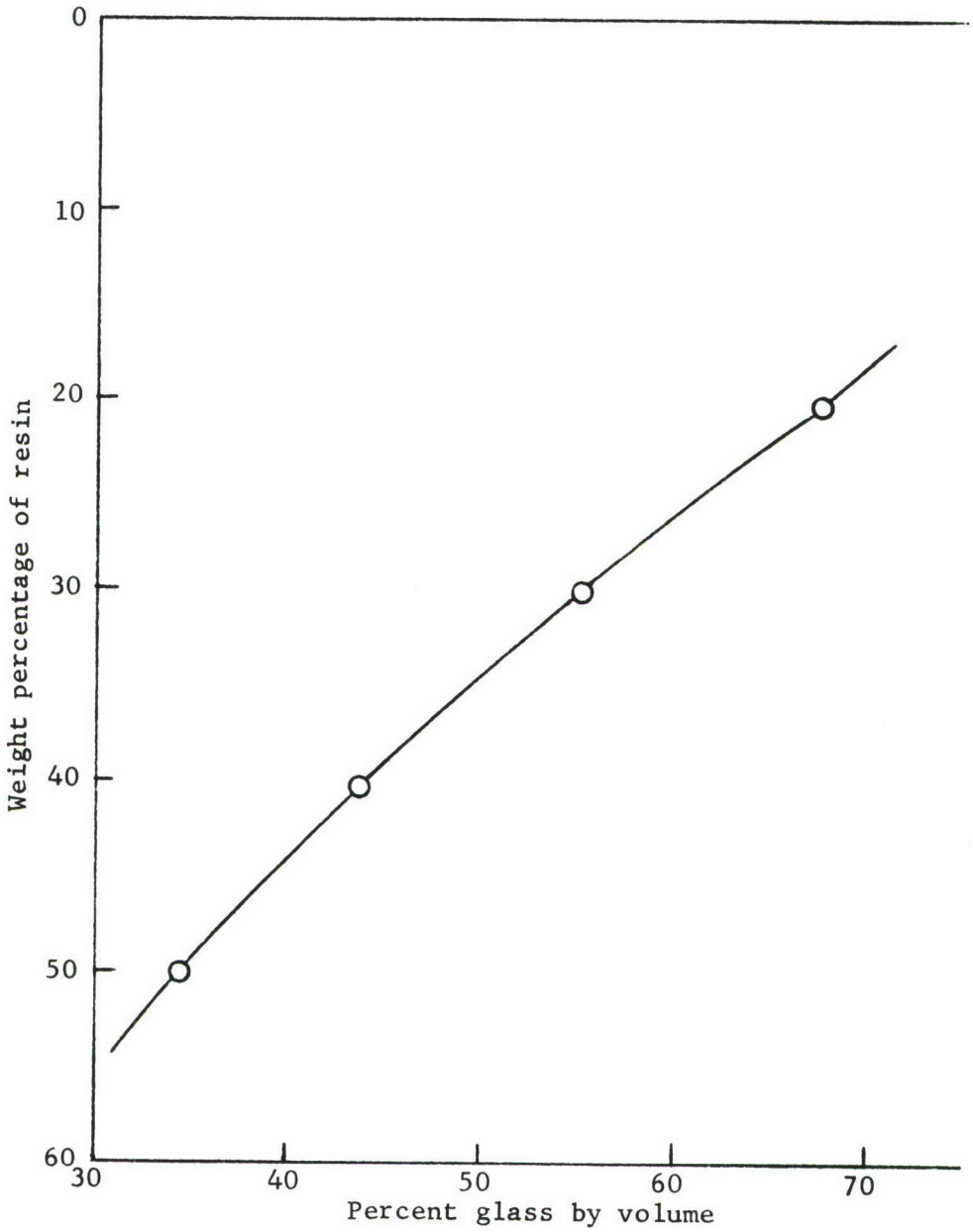


Figure 7 - The Relationship Between The Weight Of Resin And The Volume of Glass In X270 Molding Compound

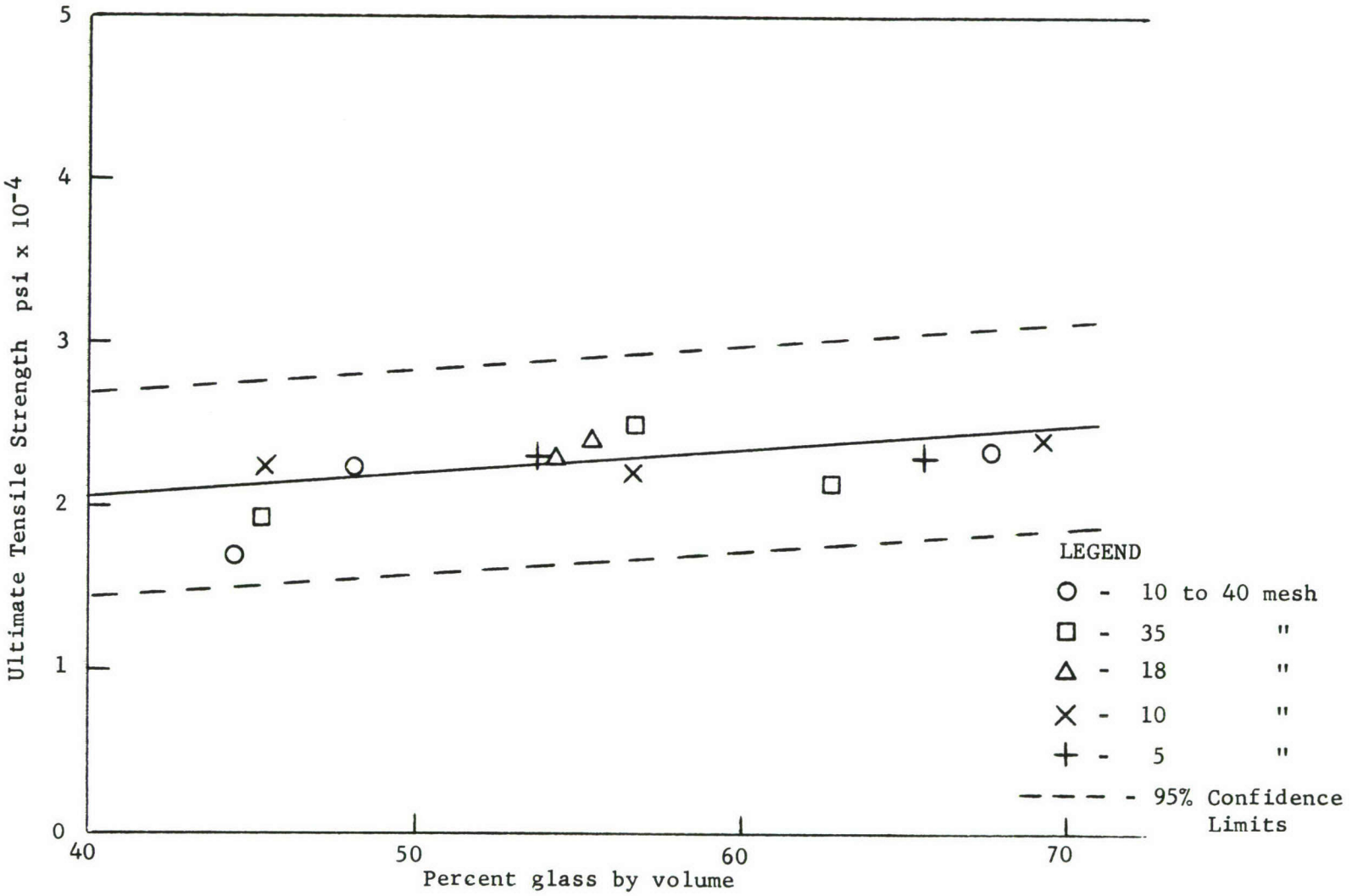


Figure 8 - Ultimate Tensile Strength As A Function Of Volumetric Glass Content For X270 Molding Compound Containing 2 Micron Glass Flakes

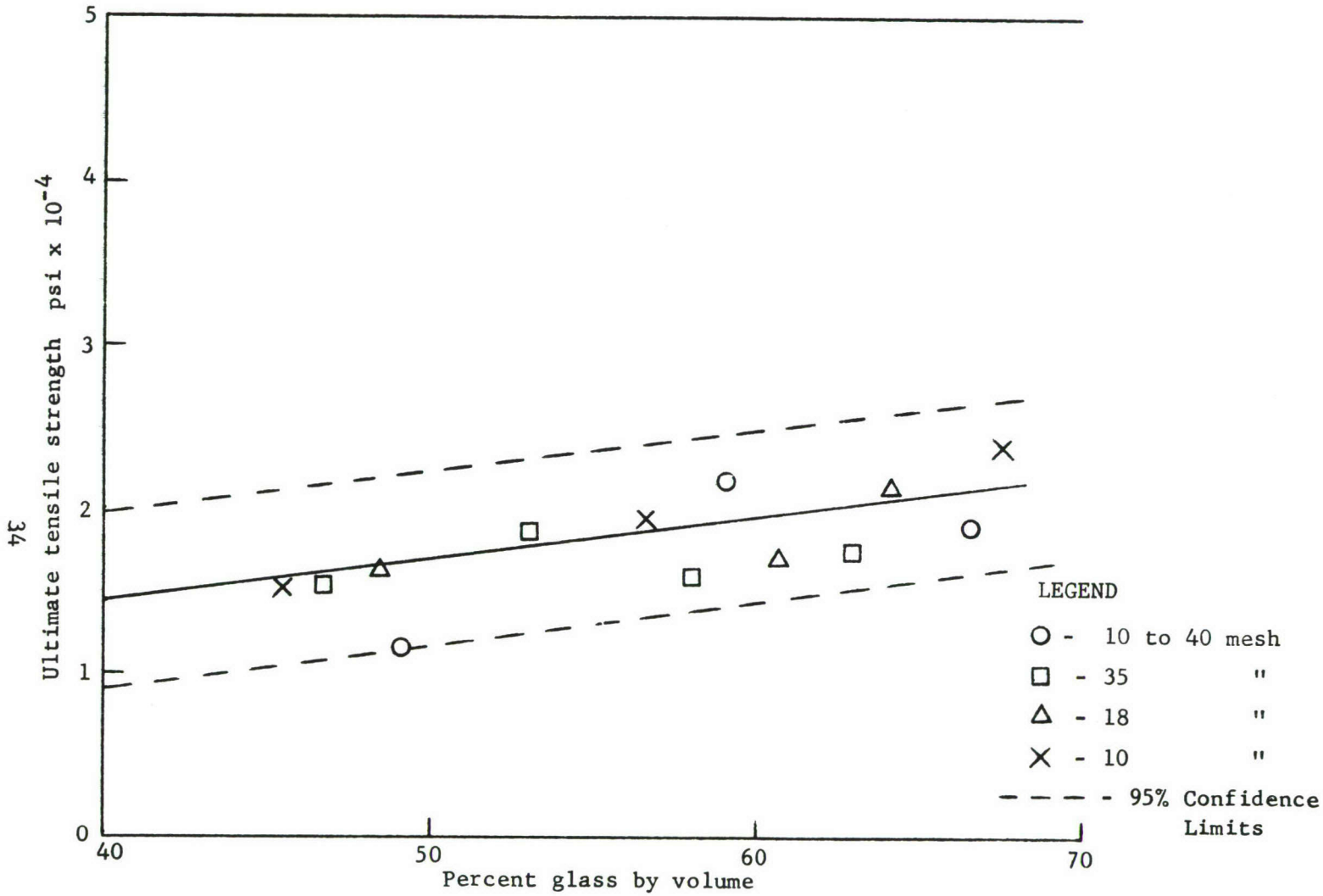


Figure 9 - Ultimate Tensile Strength As A Function Of The Volumetric Glass Content For X270 Molding Compound Containing 4 Micron Flakes

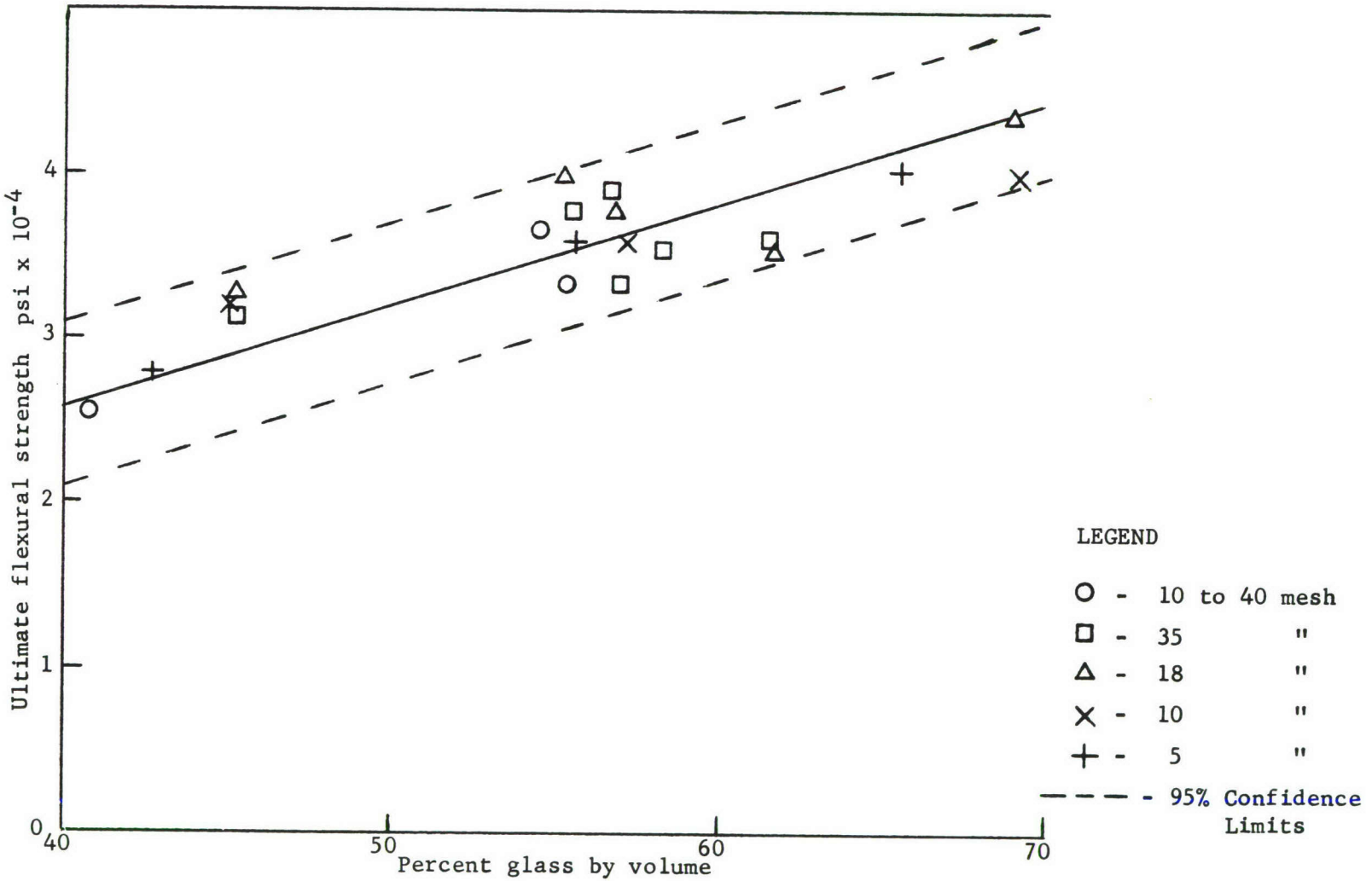


Figure 10 - Ultimate Flexural Strength As A Function Of The Volumetric Percentage Of Glass For X270 Molding Compound Containing 2 Micron Flakes

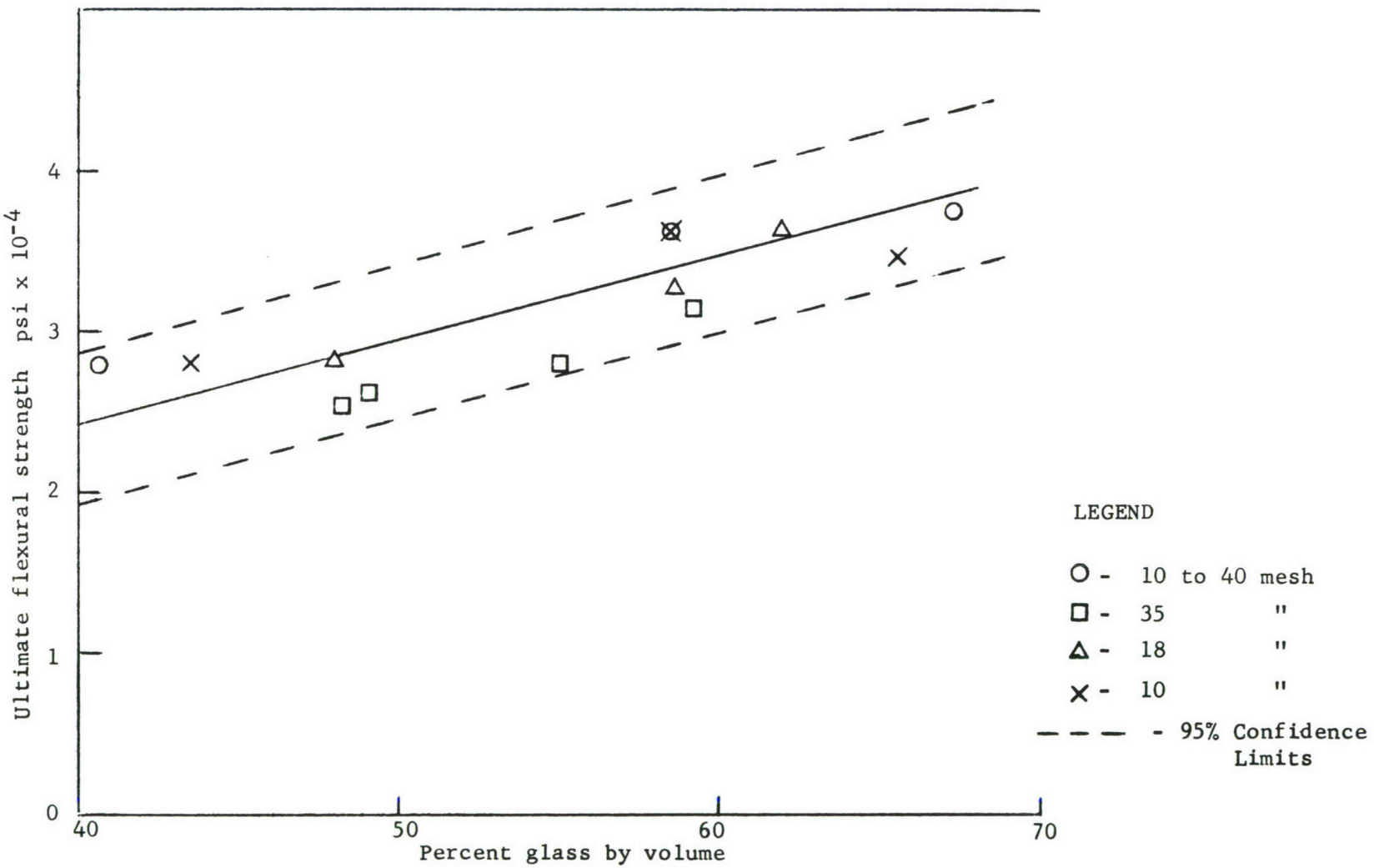


Figure 11 - Ultimate Flexural Strength As A Function Of The Volumetric Glass Content For X270 Molding Compound Containing 4 Micron Flakes

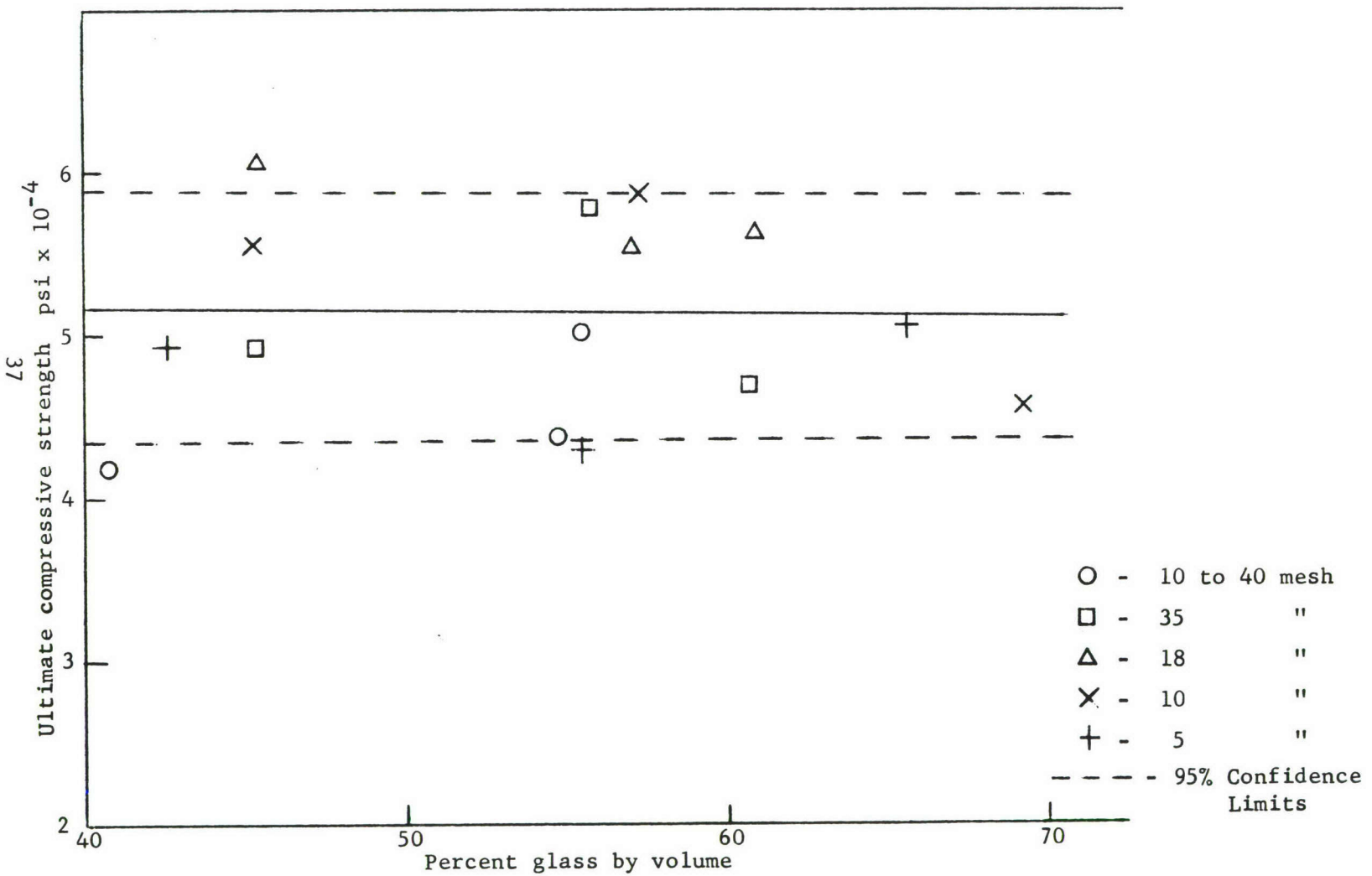


Figure 12 - Ultimate Compressive Strength As A Function Of The Volumetric Glass Content Of X270 Molding Compound Containing 2 Micron Flakes

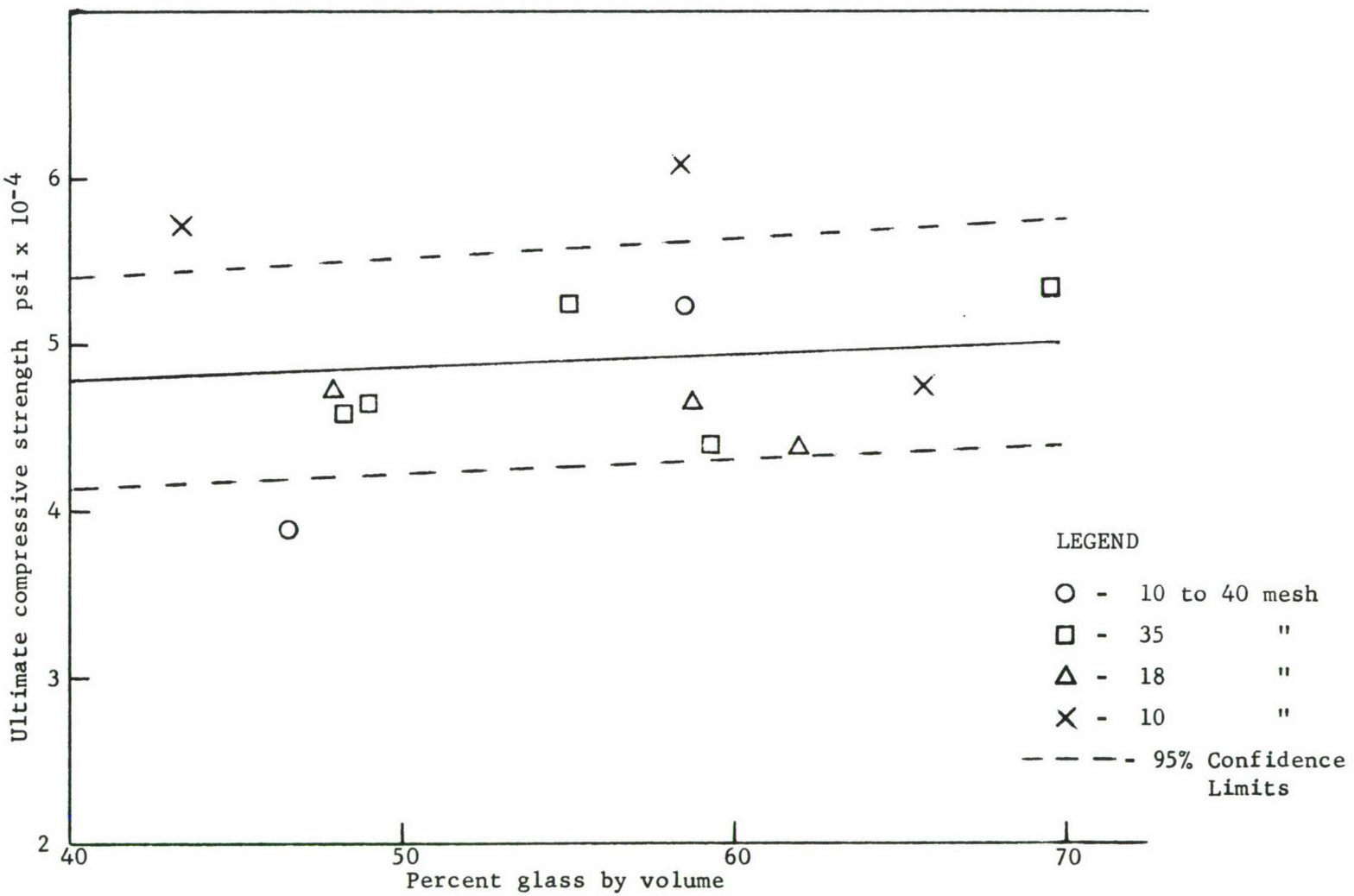


Figure 13 - Ultimate Compressive Strength As A Function Of The Volumetric Glass Content Of X270 Molding Compound Containing 4 Micron Flakes

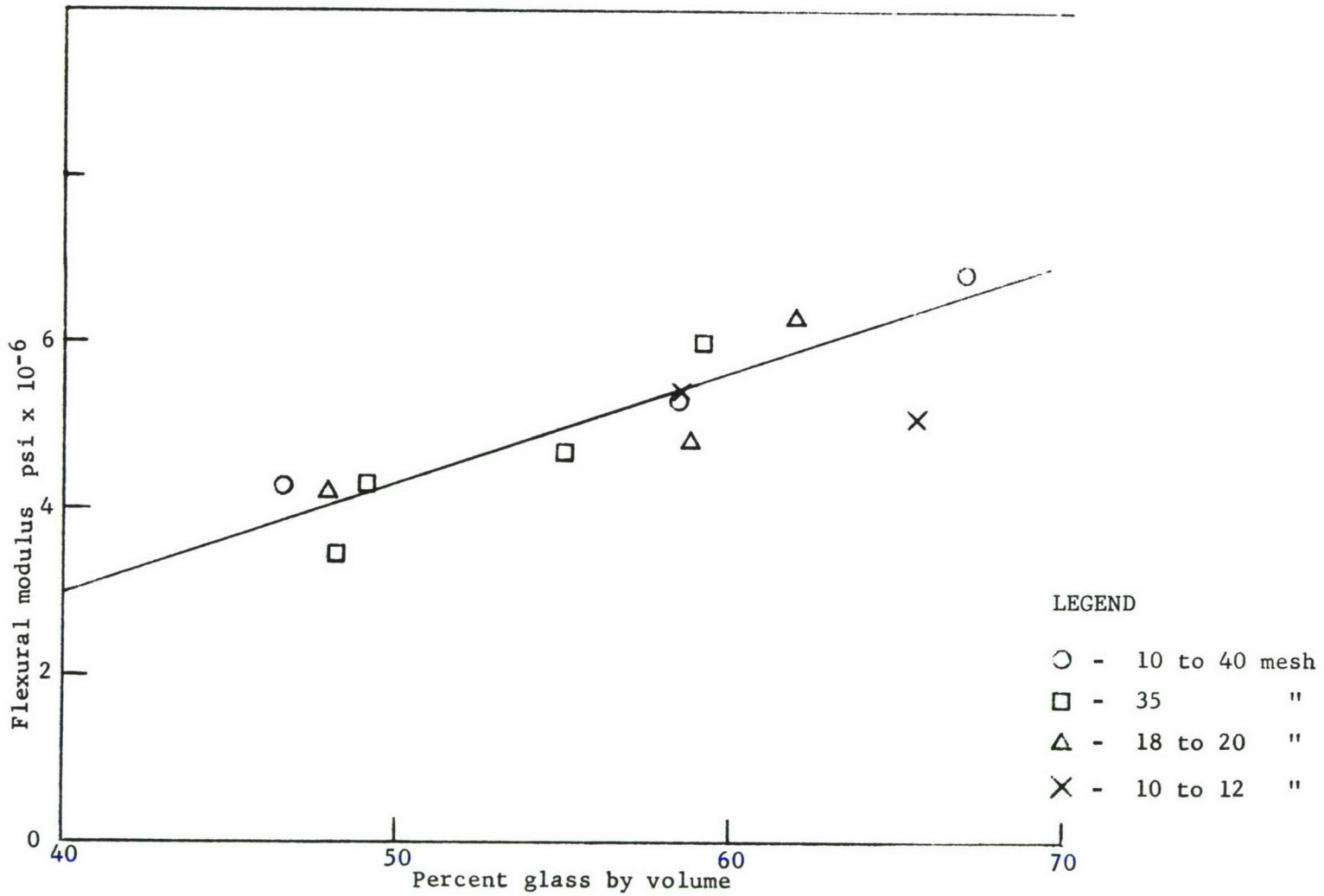


Figure 14 - Flexural Modulus As A Function Of Glass Content By Volume For 4 Micron Glass Flake In X270 Molding Compound

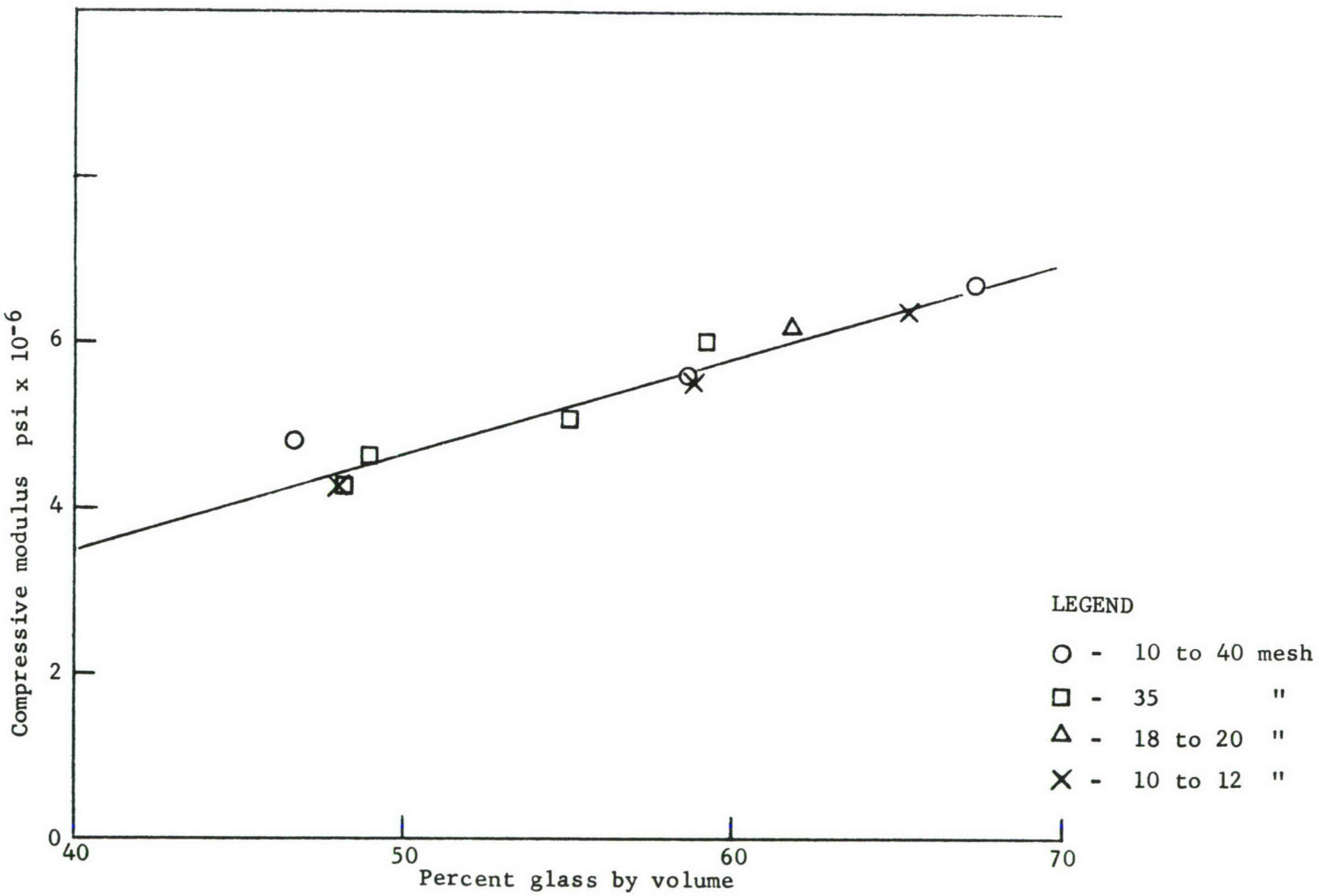


Figure 15 - Compressive Modulus As A Function Of Glass Content By Volume For 4 Micron Flake

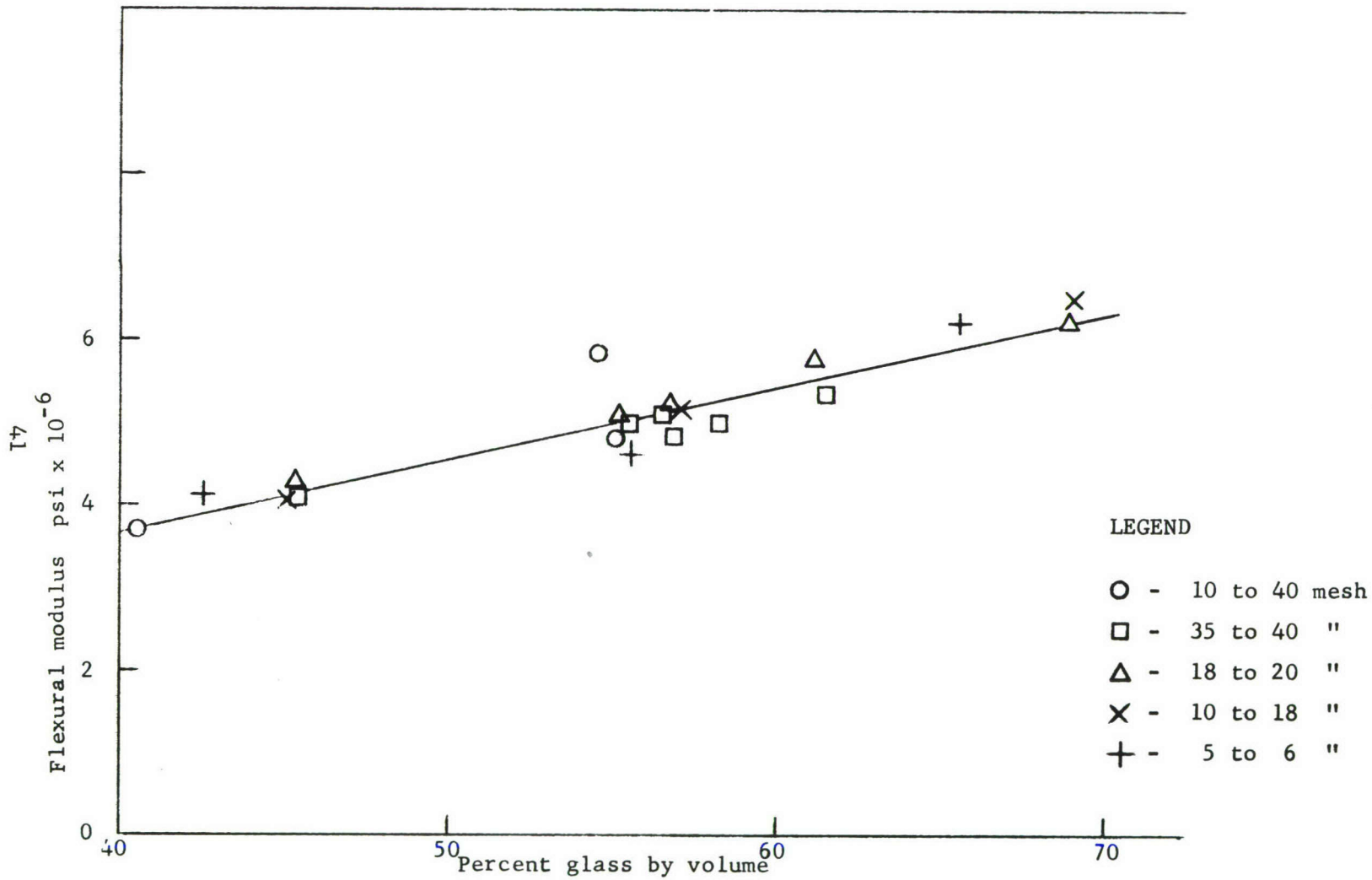


Figure 16 - Flexural Modulus As a Function of The Glass Content by Volume of 2 Micron Glass Flakes in X270 Molding Compound

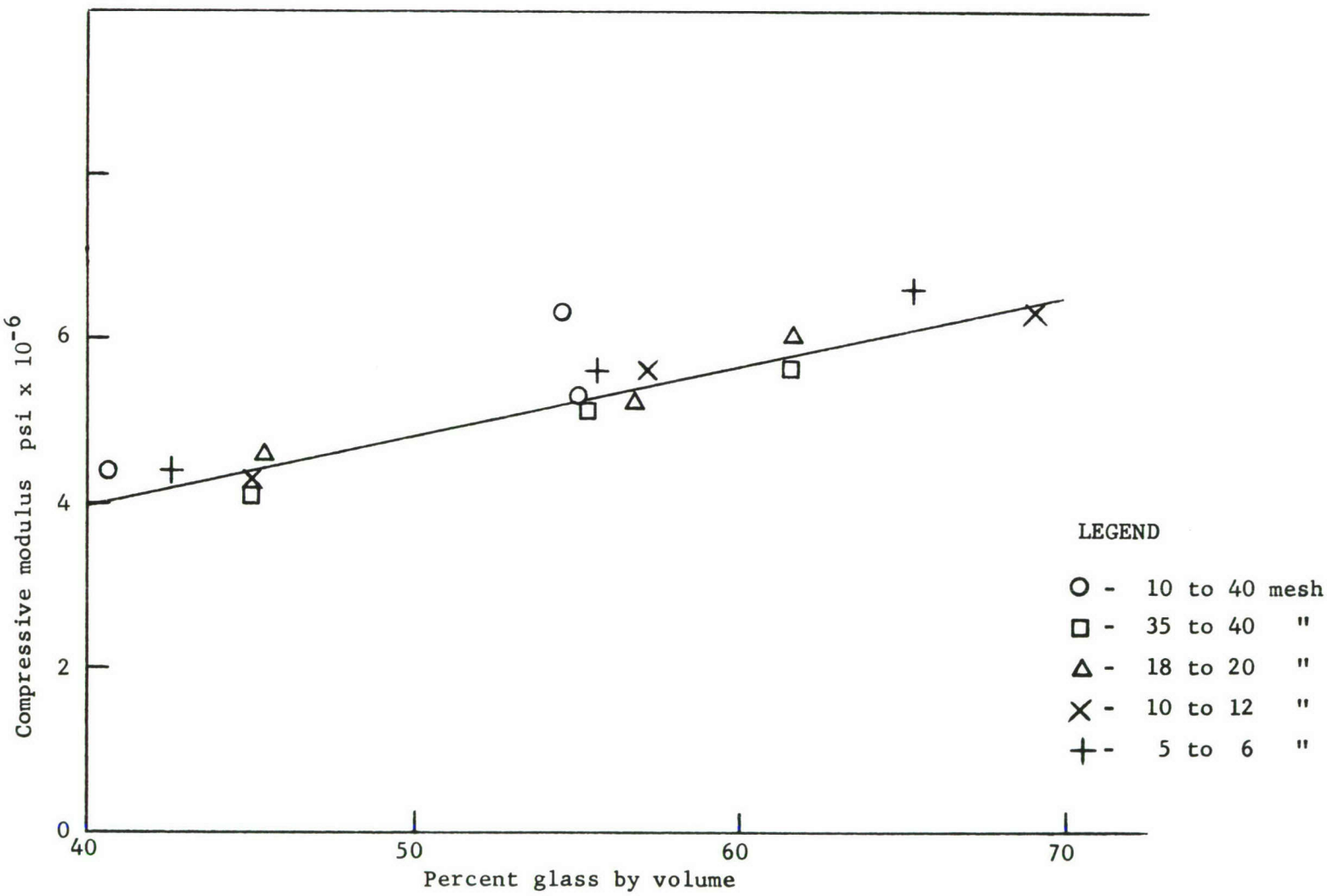


Figure 17 - Compressive Modulus As A Function Of The Glass Content By Volume For 2 Micron Glass Flakes in X270 Molding Compound

TABLE 11 DESCRIPTION OF LAMINATES MADE FROM X270 AND 2 MICRON FLAKES

Spec. No.	Wt Ratio of glass to resin (Nominal)	Flake Size (U.S. STD Mesh)	Wt % of Resin	Volume % of Glass	Mold-ability (Note 1)	Bulk Factor	Test	CURE CYCLE				
								Preforms	Contact Time min	Pres. psi	Temp °F	Cure Time hrs
Z-18 Z-19	60 : 40	10 to 40	43.1	40.5	B		Flex Tensile	Yes	2.5	800	350	2
A-2-7 A-2-8	60 : 40	35	39.4 39.1	45.2 45.0	A	17	Flex Tensile	Yes No	2.0	800	"	"
A-2-5 A-2-6	60 : 40	18	38.8 38.8	45.3 45.3	A	17	Flex Tensile	Yes	2.0	800	"	"
A-2-3 A-2-4	60 : 40	10	39.2 38.8	45.0 45.3	B	23	Flex Tensile	Yes	2.0	800	"	"
A-2-1 A-2-2	60 : 40	5	41.7 39.2	42.5 45.0		25	Flex Tensile	Yes	2.0 2.5	800	"	"
Z-20 Z-21	70 : 30	10 to 40	30.2 36.2	55.0 48.0	B	20	Flex Tensile	Yes	2.5	800	"	"
A-2-15 A-2-16 Z-3 Z-4 Z-6	70 : 30	35	30.1 29.2 29.0 27.8 29.2	55.3 56.5 56.7 58.2 56.4	C	15	Flex Tensile Flex " "	Yes Yes No No Yes	2.0 2.0 No No 2.0	800 800 800 1600 600	"	"
A-2-13 A-2-14 Z-7	70 : 30	18	29.0 30.0 30.2	56.7 55.3 55.1	A	20	Flex Tensile Flex	Yes Yes Yes	2.0 2.0 2.0	800 800 800	"	"
A-2-11 A-2-12	70 : 30	10	28.6 29.2	57.0 56.5	A	25	Flex Tensile	Yes	2.0	800	"	"
A-2-9 A-2-10	70 : 30	5	29.7 31.3	55.5 53.7	D	29	Flex Tensile	Yes	2.5	800	"	"
Z-22 Z-23	80 : 20	10 to 40	30.8 20.0	54.4 67.5	D	24	Flex Tensile	Yes	2.5	3500	350	2

(Continued on next page)

TABLE 11 DESCRIPTION OF LAMINATES MADE FROM X270 AND 2 MICRON FLAKES (Continued)

Spec. No.	Wt. Ratio of Glass to Resin (Nominal)	Flake Size (U.S. STD Mesh)	Wt % of Resin	Volume % of Glass	Mold-ability (Note 1)	Bulk Factor	Test	CURE CYCLE				
								Preforms	Contact Time min.	Pres. psi	Temp °F	Cure Time hrs
A-2-23 A-2-24	75 : 25	35	25.1 24.0	61.4 62.7	D	17	Flex Tensile	Yes	2.5	3500	350	2
A-2-21 A-2-22 Z-8	75 : 25 75 : 25 80 : 20	18	25.4 25.3 19.4	61.6 61.7 68.8	B B C	20	Flex Tensile Flex	Yes	2.5 2.5 2.0	800	" "	" "
A-2-19 A-2-20	80 : 20	10	19.2	69.0	C	31	Flex Tensile	Yes	2.0	800	" "	" "
A-2-17 A-2-18	80 : 20	5	21.8	65.3	D	30	Flex Tensile	Yes	2.5	4000	" "	" "

NOTE 1. Moldability is coded as follows:

1. "A" or very good indicates that good laminates were consistently obtained using the standard molding cycle.
2. "B" or good indicates that good laminates were usually obtained using the standard molding cycle.
3. "C" or poor indicates that good laminates could usually be obtained with a modified molding cycle.
4. "D" or very poor indicates that good laminates could not consistently be obtained.

TABLE 12 DESCRIPTION OF LAMINATES MADE FROM X270 AND 4 MICRON FLAKES

Spec. No.	Wt Ratio of Glass to Resin (Nominal)	Flake Size (U.S. STD Mesh)	Wt % of Resin	Volume % of Glass	Mold-ability (Note 1)	Bulk Factor	Test	CURE CYCLE				
								Preforms	Contact Time min	Pres. psi	Temp °F	Cure Time hrs
Z-12 Z-13	60 : 40	10 to 40	37.7 35.4	46.5 49.0	B		Flex Tensile	Yes	2.5	800	350	2
A-4-15 A-4-16 A-4-7	60 : 40	35	36.3 37.5 35.4	48.2 46.5 49.0	A	13	Flex Tensile Flex	Yes	2.5	800	"	"
A-4-5 A-4-6	60 : 80	18	36.4 35.8	48.0 48.5	A		Flex Tensile	Yes	2.5	800	"	"
A-4-3 A-4-4	60 : 40	10	40.4 38.5	43.5 45.5	A		Flex Tensile	Yes	2.5	800	"	"
Z-14 Z-15	70 : 30	10 to 40	27.6 27.0	58.4 59.0	B		Flex Tensile	Yes	2.5	800	"	"
A-4-15R A-4-16R A-4-8	70 : 30	35	30.4 28.2 31.7	55.0 57.7 53.2	A	12	Flex Tensile Tensile	Yes	2.5	800	"	"
A-4-13 A-4-14	70 : 30	18	27.2 25.5	58.7 60.7	A		Flex Tensile	Yes	2.5	800	"	"
A-4-11 A-4-12	70 : 30	10	27.4 29.0	58.5 56.7	A	20	Flex Tensile	Yes	2.5	800	"	"
Z-16 Z-17	80 : 20	10 to 40	20.4 21.0	67.2 66.4	D	19	Flex Tensile	Yes	2.5	3500	"	"
A-4-23 A-4-24	75 : 25	35	22.7 24.0	59.3 62.8	D	13	Flex Tensile	Yes	2.5	3500	"	"
A-4-21 A-4-22	80 : 20	18	24.8 22.8	61.8 64.2	C	15	Flex Tensile	Yes	2.5	3500 3000	"	"
A-4-19 A-4-20	80 : 20	10	21.6 20.1	65.5 67.5	C	22	Flex Tensile	Yes	2.5	3500	"	"

TABLE 13 FLAKE SIZE AFTER MOLDING AND IGNITION

ORIGINAL FLAKE SIZE	% OF EACH FLAKE SIZE FOUND AFTER MOLDING				
	5 mesh (4000to3360 μ)	10 mesh (2000to1680 μ)	18 mesh (1000to840 μ)	35 mesh (500to420 μ)	less than 40 mesh (less than 420 μ)
5 mesh (4000to3360 μ)	0.0	39.0	33.0	18.0	10.0
10 mesh (2000to1680 μ)	-	35.0	37.0	18.0	10.0
18 mesh (1000to840 μ)	-	-	61.0	27.0	12.0
35 mesh (500to420 μ)	-	-	-	64	36

Results that were somewhat surprising and probably the most significant in this phase of the program, were the findings showing that within the limits under investigation and using available molding techniques the mechanical properties of the glass flake laminates appear to be independent of the "diameter" of the flakes used for reinforcement. Specifically, equivalent results were obtained with flakes that ranges from the 35 mesh flakes, which are approximately 200 microns in diameter, to the 5 mesh flakes, which are approximately 4000 microns in diameter. The probable reason for this phenomenon becomes apparent when the results of the screening of the flake after ignition are considered (Table 13). As is shown in this table, the flakes are broken during the molding process, and the resulting reinforcement in the composite is reduced in size to approximately the same as the smallest flake that was considered in this test series. Therefore, regardless of the size of the flake that was initially mixed into the molding compound, the final results were the same as if the smaller flake had been used.

In addition to the mechanical properties of the various mixtures of flake, the moldability of each of these mixtures was considered. As in Tables 11 and 12, the most readily moldable compositions were those containing the 10 and 18 mesh, or medium sized flakes. This factor, when considered in conjunction with the results shown above, (which indicate that the strength of glass flake reinforced composites is not affected by variations in the size of the flakes within the limits under consideration), demonstrated that it was not necessary to investigate two separate compositions with each resin system to provide both a high strength molding composition and a readily moldable composition. Instead, it indicates that it was sufficient to investigate one formulation which would provide both of these characteristics. As the 10-40 mesh mixture results in the least amount of waste material, this mixture was used exclusively throughout the balance of this program.

SECTION III CONTROL TESTS

1.0 SCOPE

The control tests portion of this program was designed to establish the basic physical, mechanical and electrical properties of flat moldings made with each of the four molding compositions. It was intended that these values would serve two purposes:

1. To establish design allowables for flake reinforced composites.

2. To form the basis for evaluation of the properties of the compression molded geometric shapes.

2.0 PREPARATION OF SAMPLES FOR TESTING

2.1 Composition

All the laminates prepared were formulated to contain 70 percent by weight of two micron, composition E, untreated Filmglas flakes. Limits on the size or "diameter" of these flakes were established by using only those flakes which would pass through a U. S. Standard 10 mesh screen, but which were retained by a U. S. Standard 40 mesh screen. (Openings in these screens are 4000 microns and 480 microns respectively).

2.2 Molding Parameters

Wherever possible, the conditions which were established as most satisfactory for molding, during the screening tests of this project, were used to mold the laminates for the control tests. These conditions, together with any exceptions which were required to expedite the molding of good quality laminates, are shown in Table 12. Photographic comparison of a good quality X270 epoxy laminate and of an X270 laminate, which was rejected because of appearance, is shown in Figure 18.

A similar comparison for laminates made with X274 silicone is shown in Figure 19.

The term "preform", which is used in Table 14, refers to a process by which the bulk of the molding compounds was reduced by pressing the material into a biscuit by means of the application of 100 to 300 psi while the material was maintained at 200°F. A photograph showing such a preform and an X270 laminate made from a similar preform appears in Figure 20.

To conform to specifications for testing reinforced plastics, it was necessary to produce specimens up to 10" long. The 6" square laminates which had previously been used in the program were not sufficiently large to do this, therefore, a 12" x 12" mold was fabricated so that larger laminates could be molded. The use of the 12" mold introduced a problem in that 12" square, 1/8" thick laminates

TABLE 14

MOLDING PARAMETERS FOR CONTROL TEST LAMINATES

Molding Compound	Laminate Number	Molding Conditions			Cure Cycle			Remarks
		Contact Time, min.	No. of Preforms	Size	hrs	°F	psi	
X-270 Epoxy	F-13	1.5	2	12x12	2	350	1000	Good
	F-14	1.5	2	12x12	2	350	1000	Good
	F-15	2	2	12x12	2	350	1000	Good
	F-16	2	2	12x12	2	350	1000	Several short wrinkles
	F-17	1.7	2	12x12	2	350	1000	Wrinkles in one corner
	F-18	1.7	2	12x12	2	350	1000	Several very small dry spots
	F-19	2	2	12x12	2	350	1000	Numerous freckles
	F-20	2	2	12x12	2	350	1000	Numerous freckles
	F-21	2	2	12x12	2	350	1000	Numerous freckles
	F-22	2	8	6x6	2	350	1000	Wrinkles along edges Numerous freckles
	F-23	2	8	6x6	2	350	1000	Wrinkles along edges Numerous freckles
	F-24	2	8	6x6	2	350	1000	Wrinkles along edges Numerous freckles
X-271 Epoxy	G-13	5	2	12x12	2	350	800	Several small dry spots
	G-14	5	2	12x12	2	350	800	Several small dry spots
	G-15	5	2	12x12	2	350	800	Good
	G-16	5	2	12x12	2	350	800	Several small dry spots
	G-17	-	2	12x12	2	350	800	Good
	G-18	-	2	12x12	2	350	800	Good
	G-19	-	2	12x12	2	350	800	Good
	G-20	-	2	12x12	2	350	800	Good - several very small dry spots
	G-21	-	2	12x12	2	350	800	Several very small dry spots
	G-22	-	8	6x6	2	350	800	Laminate discarded because of delamination in approx. center of lam.
	G-22R	-	8	6x6	2	350	800	Good
	G-23	-	8	6x6	2	350	800	Several small dry spots and wrinkles
	G-24	-	8	6x6	2	350	800	Several small dry spots and winkles
X-273 Phenolic	H-13	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Slightly cloudy appearance
	H-14	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Several wrinkles and dry spots

TABLE 14

MOLDING PARAMETERS FOR CONTROL TEST LAMINATES (Continued)

Molding Compound	Laminate Number	Molding Conditions			Cure Cycle			Remarks
		Contact Time, min	No. of Preforms	Size	hrs	°F	psi	
X-273 Phenolic	H-15	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Several wrinkles and two dry corners
	H-16	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Several wrinkles Dry along edges
	H-17	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Dry corners and some wrinkles
	H-18	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Small wrinkles Slightly cloudy
	H-19	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Several small wrinkles - dry along edges
	H-20	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Slightly dry along edges
	H-21	-	2	12x12	1 1 1	250 300 400	1000 1000 1000	Slightly dry along edges
	H-22	-	8	6x6	1 1 1	250 300 400	1000 1000 1000	Wrinkles along edges
	H-23	-	8	6x6	1 1 1	250 300 400	1000 1000 1000	Wrinkles along edges
	H-24	-	8	6x6	1 1 1	250 300 400	1000 1000 1000	Wrinkles along edges
X-274 Silicone	J-13	1	*	12x12	1 8	350 500	1000	Cloudy appearance Two very small blows
	J-14	1	*	12x12	1 8	350 500	1000 1000	Short wrinkles and cloudy appearance
	J-15	1	*	12x12	1 8	350 500	1000	Cloudy appearance wrinkles in two corners
	J-16	1	*	12x12	1 8	350 500	1000	Cloudy and has short wrinkles
	J-17	1	*	12x12	1 8	350 500	1000	Cloudy appearance
	J-18	1	*	12x12	1 8	350 500	1000	Cloudy appearance

TABLE 14

MOLDING PARAMETERS FOR CONTROL TEST LAMINATES (Continued)

Molding Compound	Laminate Number	Molding Conditions			Cure Cycle			Remarks
		Contact Time, min	No. of Preforms	Size	hrs	°F	psi	
X-274 Silicone	J-19	1	*	12x12	1 8	350 500	1000	Cloudy appearance
	J-20	1	*	12x12	1 8	350 500	1000	Cloudy appearance
	J-21	1	*	12x12	1 8	350 500	1000	Cloudy appearance
	J-22	1	1	6x6	1 8	350 500	1000	Several wrinkles
	J-23	1	1	6x6	1 8	350 500	1000	Several wrinkles
	J-24	1	1	6x6	1 8	350 500	1000	Several wrinkles

* Loose (no preform)

NOTE: Contact time was varied to compensate for variations in gel time of the resins used in the molding compounds.

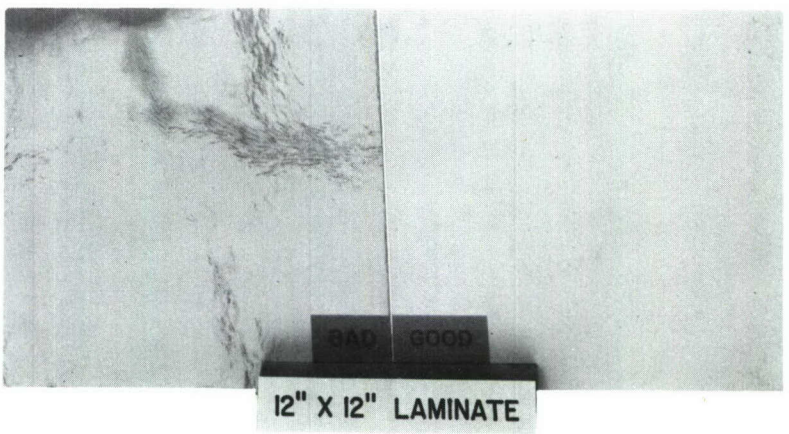


Figure 18 - A Comparison Of Good Quality And Poor Quality X270, Epoxy Moldings. The Laminate On The Left Was Not Tested

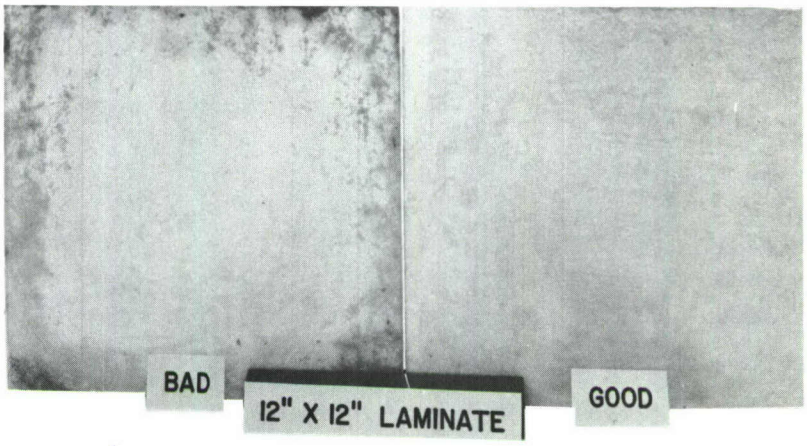


Figure 19 - A Comparison Of Good Quality And Poor Quality X274 Silicone Molding. The Laminate On The Left Was Discarded

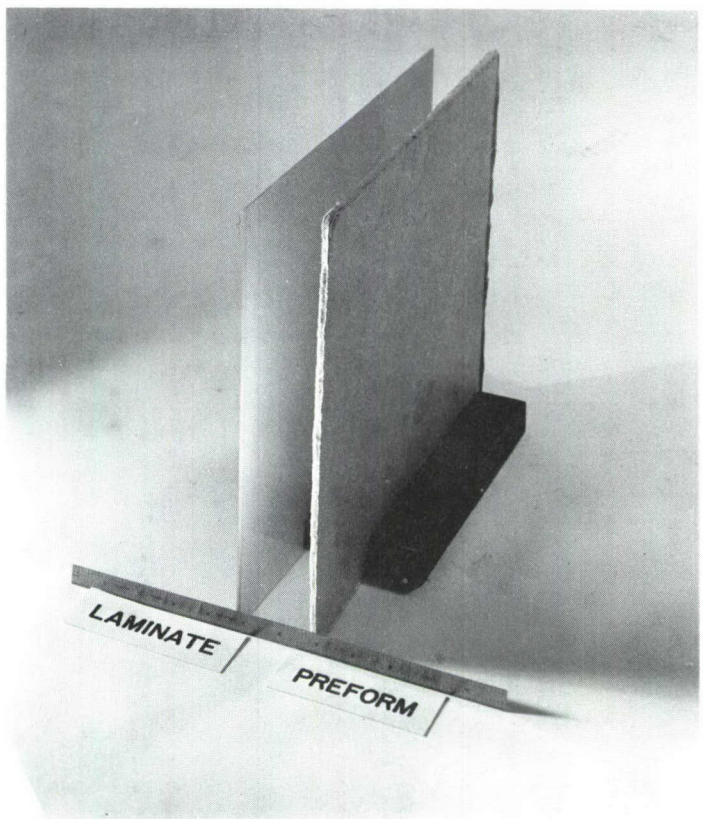


Figure 20 - A Comparison Between An X270 Preformed Biscuit And An X270 Laminate Made From A Similar Preform. Note That The Bulk Factor Has Been Reduced From The 15 Or 20 To 1 Inherent To Loose Material To Approximately 3 To 1

could not be produced using the same materials and the same processing parameters which had produced satisfactory 6" laminates.

From the appearance of the initial moldings made in the 12" mold, (the moldings were translucent in the center and were white and crumbly around the edges), it was assumed that the problem was caused by an insufficient amount of flow during the molding cycle. Changes in processing which normally increase flow are:

1. Shorter contact time or preheating time
2. Increased pressure
3. Higher resin contents
4. Modified molds

The first three changes were the most convenient and the first to be tried. The shortened contact time and increased pressure improved the appearance of the laminates but did not entirely solve the problem. Higher resin contents yielded good laminates, but this solution was rejected because of the lower strengths associated with higher resin contents. The remaining possibility, the use of a modified mold, was therefore investigated. A temporary 3/4" thick aluminum plate, which was 1/16" per side smaller than the mold cavity, was fitted to the mold plunger to determine whether or not this modification would produce satisfactory laminates.

Laminates made in the modified mold were of very good quality and it appeared that the problem had been solved. A permanent modification of the mold was made by machining a step, 1/16" deep and 3/4" wide, around all four sides of the mold plunger.

2.3 Mechanical Preparation of Test

Specimens were cut from the 12" and 6" laminates with a water-cooled diamond saw. When required, because of the specimen configuration, (e.g. the tensile and fatigue specimens), conventional grinding equipment was used to finish the edges of the specimens.

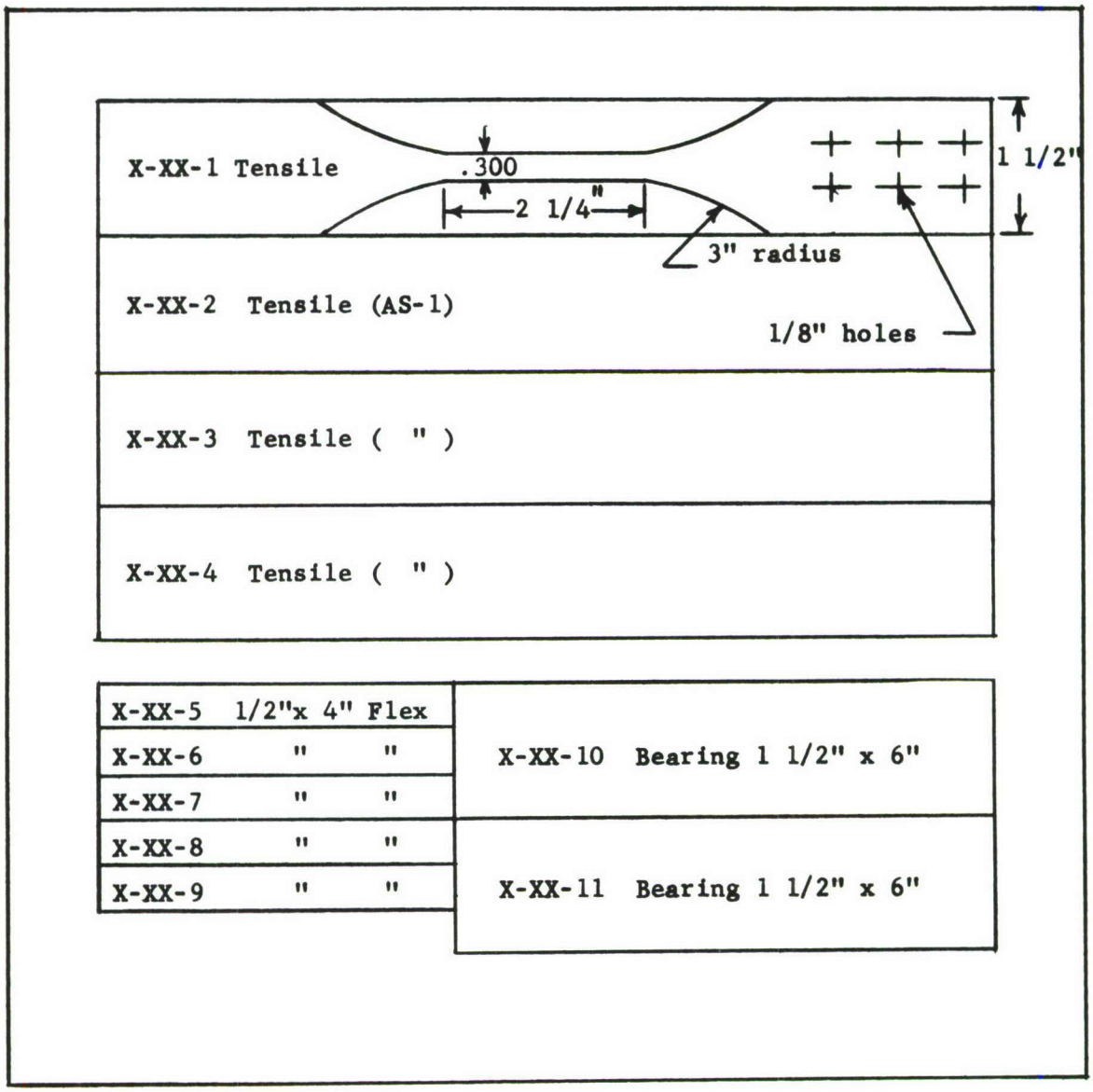
Drawings of the layout used to cut specimens from the laminates are shown in Figures 21 through 24 inclusive. Photographs of finished specimens, before and after testing, appear in Figures 25 and 26.

2.4 Test Methods

2.4.1 Physical Tests

2.4.1.1 Specific Gravity

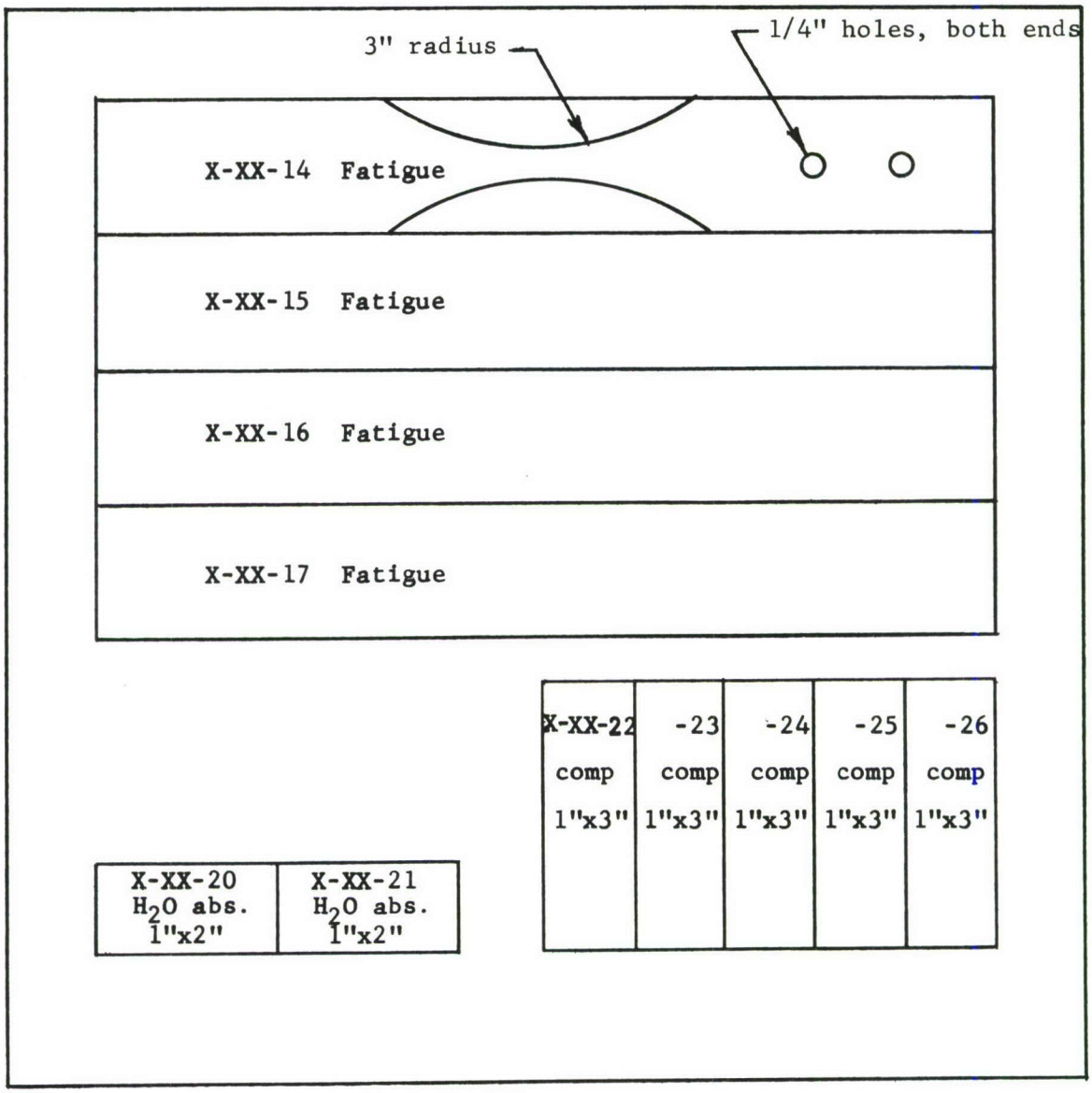
a. Scope: This method is in accordance with L-P-406b, method 5011. It is designed for use in determining the specific gravity of solid plastics which are not affected by water.



Note: This sketch used for cutting laminates: 1/2 scale

F-13, -16 & -19
G-13, -16 & -19
H-13, -16 & -19
J-13, -16 & -19

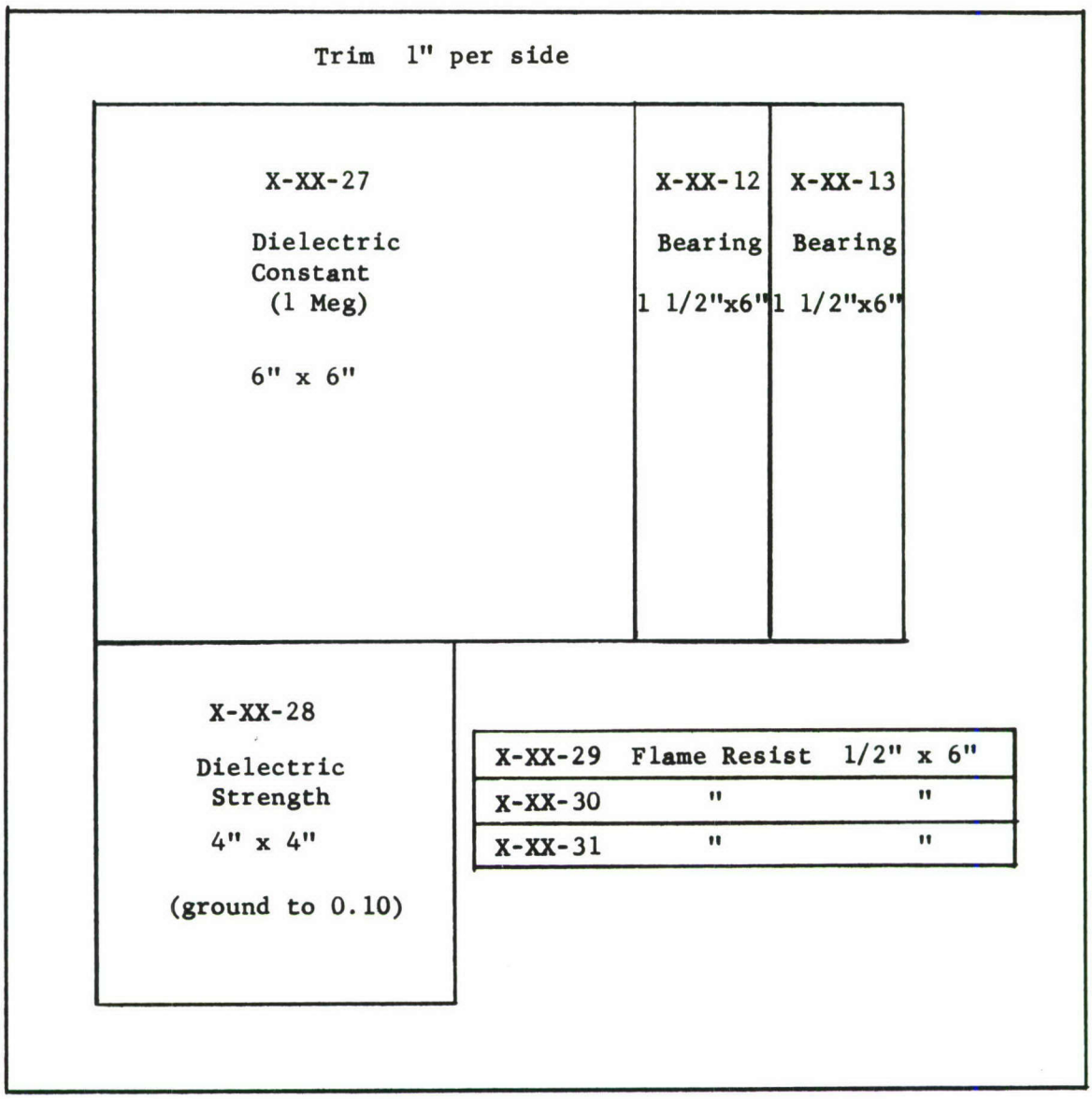
Figure 21 - Layout Of Test Specimens On Laminates



Note: This sketch used for cutting laminates: 1/2 scale

F-14, -17 & -20
G-14, -17 & -20
H-14, -17 & -20
J-14, -17 & -20

Figure 22 - Layout of Test Specimens on Laminates

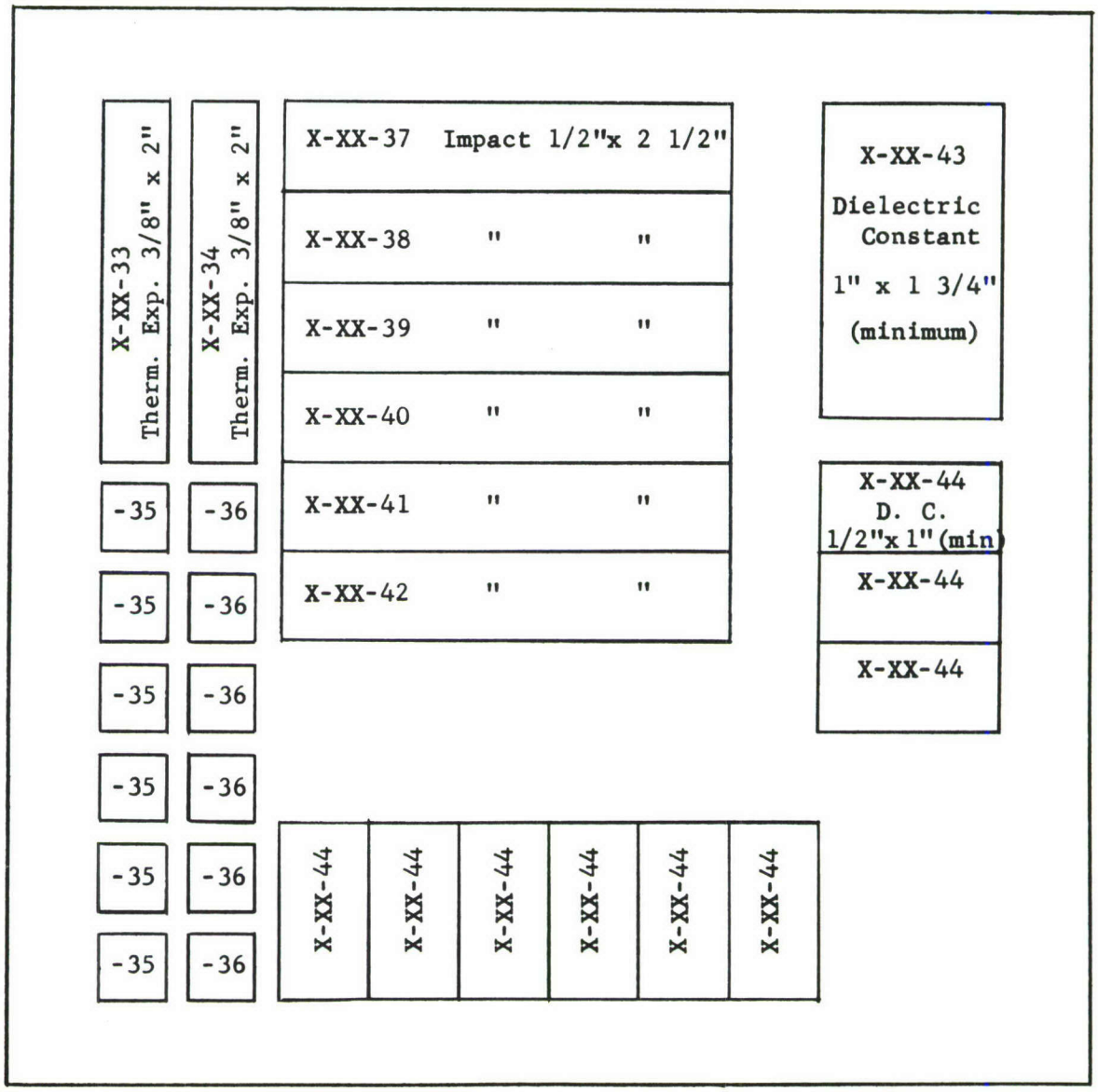


Note: This sketch used for cutting laminates:

F-15, -18 & -21
G-15, -18 & -21
H-15, -18 & -21
J-15, -18 & -21

1/2 scale

Figure 23 - Layout Of Test Specimens On Laminates



Note: This sketch used for cutting laminates:

F-22, -23 & -24
G-22, -23 & -24
H-22, -23 & -24
J-22, -23 & -24

Full size

Figure 24 - Layout Of Test Specimens On Laminates

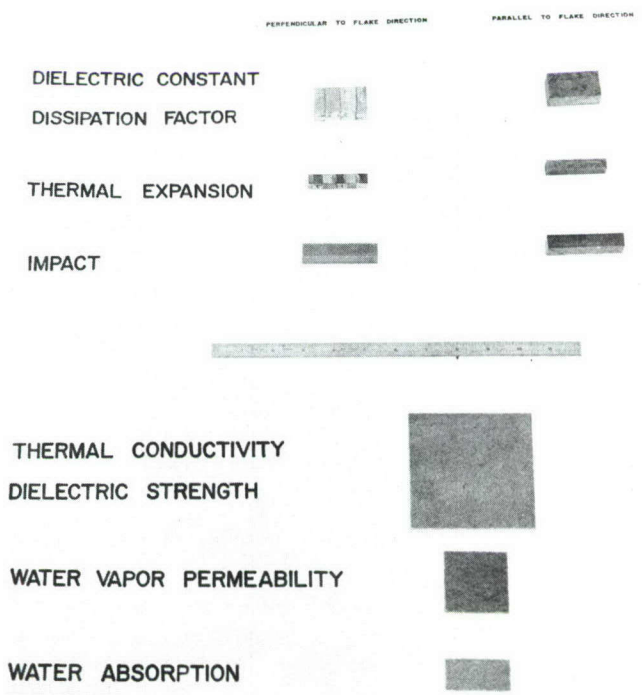


Figure 25 - Test Specimens For The Control Test Series

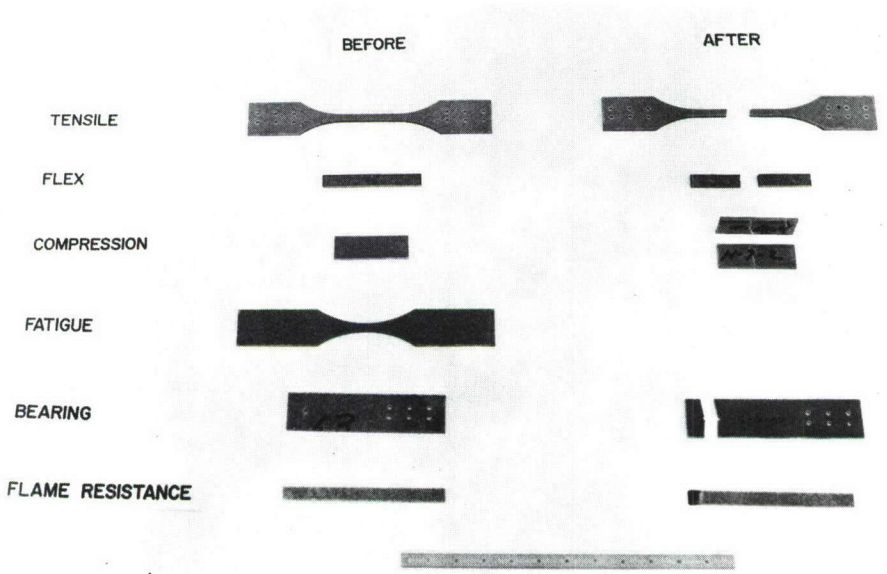


Figure 26 - Test Specimens For The Control Test Series

b. Test Specimen: The test specimen was a solid piece, with a total volume of approximately 0.2 cubic inches. This specimen was cut from the undamaged portion of a specimen used for mechanical tests.

c. Apparatus: An analytical balance and a specific gravity bridge were used.

d. Procedure: The test specimen was weighed in air and in distilled water at 23° ± 2°C. The specific gravity was calculated from the following equation:

$$\text{Specific Gravity} = \frac{\text{Weight in Air}}{\text{Weight in Air - Weight in Water}}$$

2.4.1.2 Water Absorption

a. Scope: This method is in accordance with L-P-406b method 7031. It is designed to determine the rate of water absorption of immersed plastics.

b. Test Specimen: Test specimens were cut one inch by three inches by the thickness of the material, (approximately 0.100 inch). The cut edges were smoothed with number 000 sandpaper.

c. Apparatus: A circulating air oven, desiccator, analytical balance, and micrometer gage were used.

d. Procedure: The specimens were conditioned in an oven at 122° ± 4°F for 24 hours. After conditioning, the specimens were cooled in a desiccator and weighed. Dimensions were measured to the nearest 0.0001 inch with a micrometer gage. The specimens were then immersed in distilled water maintained at a temperature of 73.5° ± 4°F. At the end of 24 hours of immersion, each specimen was removed, the surface moisture quickly absorbed by a dry cloth and the specimen reweighed. The dimensions also were remeasured.

The percentage increase in weight during immersion, calculated to nearest 0.01 percent, was derived from the following equation:

$$\text{Increase In Weight, Percent} = \frac{\text{Wet weight - Conditioned weight}}{\text{Conditioned weight}} \times 100$$

No dimension changes were observed.

2.4.1.3 Thermal Conductivity

a. Scope: This method is designed to determine the thermal conductivity of poorly conducting materials.

b. Test Specimens: Specimens are described in Figures 23 and 25. These same specimens were used for dielectric strength measurement.

c. Apparatus: A Cenco-Fitch conductivity apparatus and a Leeds Northrup K-3 Universal potentiometer were used.

d. Procedure: To measure thermal conductivity of the thin piece of material, it was placed between a lower vessel, kept at constant temperature by boiling water, and an upper insulated block of copper of known thermal capacity. The heat conducted through the material raised the temperature of the copper block by a measured amount. Thermocouples and a Leeds Northrup potentiometer were used to indicate temperature differences. Thermal conductivity was calculated from the rate at which heat was conducted through the material, from the area and thickness, and from the temperature difference between the faces of the specimen. The formula used was

$$K = \frac{C \times \ell \times \Delta T}{Ti}$$

where

C = A constant for the system which was established empirically from measurements on a material with a known thermal conductivity

ℓ = Thickness of sample in inches

ΔT = Temperature range of measurements

Ti = Time in minutes

2.4.1.4 Thermal Expansion

a. Scope: Linear thermal expansion of each of the molding materials under consideration was determined in accordance with Federal Specification L-P-406b, Method 2031. Expansion was determined both in the plane of the flake and in a direction perpendicular to that plane.

b. Test Sample Preparation: Specimens were cut from laminates No. 22, 23 and 24, which were described above. The specimens, identified as -33 and -34, were used to determine expansion in the plane of the flake reinforcement. Specimens for the determination in the direction perpendicular to the plane of the flake, were prepared by laminating the portions of these laminates identified as -35 and -36. The parent resin for each molding compound was used as the laminating adhesive, except in the case of the X274 silicone specimens. Specimens were laminated with Narmco 3135 epoxy polyamide adhesive due to the poor adhesive qualities of the silicone resin.

c. Test Procedure: Thermal expansion was determined in accordance with procedures specified in Federal Specification L-P-406b, Method 2031. A quartz tube dilatometer was used for the measurement. Elevated temperatures were maintained by immersing the dilatometer into a heated oil bath. Reduced temperatures were attained with a solid CO_2-acetone bath.

2.4.1.5 Flammability of Plastics

a. Scope: This method is in accordance with L-P-406b method 2021.1. It is designed for use in determining the flammability of plastics in the form of sheets or plates over 0.050 inches in thickness.

b. Test Specimen: Specimens were 0.5 inches in width, 6 inches in length, and of tested material thickness, (approximately 0.100 inch). Two lines were scribed on the specimen, one inch and five inches from one end.

c. Apparatus: A box enclosure, protected from air currents, with a means of venting the fumes from burning specimens, was used. Figure 27 shows a sketch of the test setup.

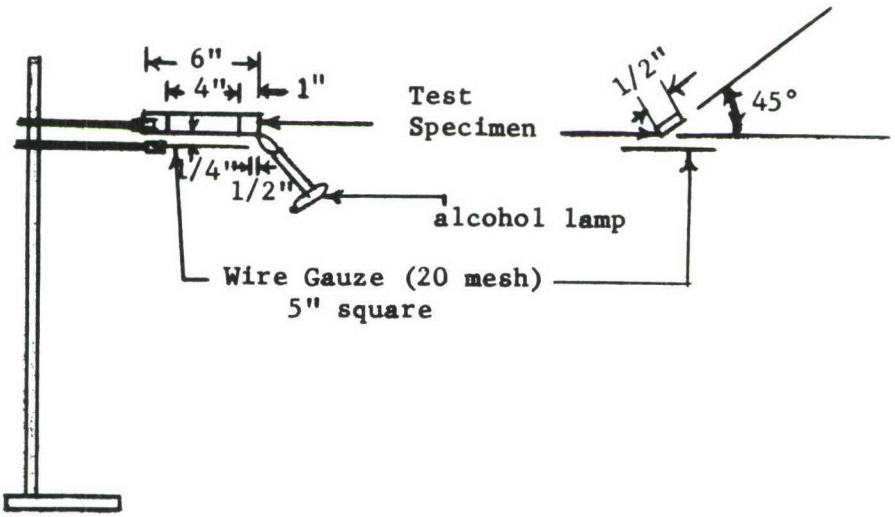


Figure 27 - Apparatus for Flammability Test

d. Procedure: After the setup was made, (Figure 27), an alcohol lamp, with a flame 1/2 to 3/4 inch in height, was placed under the free end of the test specimen. The flame tip was barely in contact with the specimen. After 30 seconds with the flame in contact with specimen, the flame was removed and the specimen allowed to burn. When the specimen did not continue to burn after the first attempt, the burner was placed under the free end for another 30 seconds. The purpose of the test was to determine the time required to burn from the 1 inch line to the 5 inch line. Materials which did not continue to burn were reported as being self-extinguishing.

2.4.1.6 Water Vapor Permeability

a. Scope: The test was designed to determine the water vapor permeability of the four flake composites under study.

b. Test Specimens: Specimens were cut from those used for dielectric constant measurement at 1 megacycles, as described in Figure 23.

c. Apparatus: The test dish was modeled after L-P-406b. Its size was such that a sample with an area of 8 square inches could be utilized. This dish is shown in Figure 28.

Two types of wax were used to seal the specimens to the test dishes. The first, a commercial grade of sealing wax, did not provide an adequate seal. The second, a formulation of bees wax, rosin, and commercial sealing wax, provided a satisfactory seal.

A constant temperature humidity chamber was built to maintain standard conditions throughout the test period. A fan in the system circulated a current of air over the surface of the specimens.

d. Procedure: Tests were conducted according to L-P-406b, method 7032. Two systems were used in the study of the glass flake composites. One approach was the use of a desiccant; anhydrous magnesium perchlorate, sealed within the test dish. A poor sealing wax made this test inconclusive. In the second approach, water was sealed within the test dish. A complete seal was achieved by using a better wax mixture. After the specimens were stabilized in the test chamber, their weights were recorded. Weight measurements were taken every 24 hours thereafter until a total of 100 hours of exposure had been accumulated. Pressure buildup within the test dishes caused the lids to pop-off, halting the tests. Since it was quite apparent that all four resin systems were impervious to water vapor, the desiccant method was not repeated. For the same reason, tests were conducted with only one side of the specimens exposes to the chamber atmosphere.

2.4.2 Mechanical Properties

2.4.2.1 Compressive Properties of Plastics

a. Scope: This method is in accordance with Aircraft Industries ARTC-11, similar to L-P-406b, method 1021.1, covering procedures for determining the edgewise compressive strength and the stress-strain relationship of structural plastic laminates.

b. Test Specimens: For edgewise compression tests, specimens were 3" long, 1" wide and the thickness of material to be tested, (Figures 22 and 26). Ends of test specimens were ground smooth and parallel to each other within .0005" and perpendicular to the long axis of the specimen.

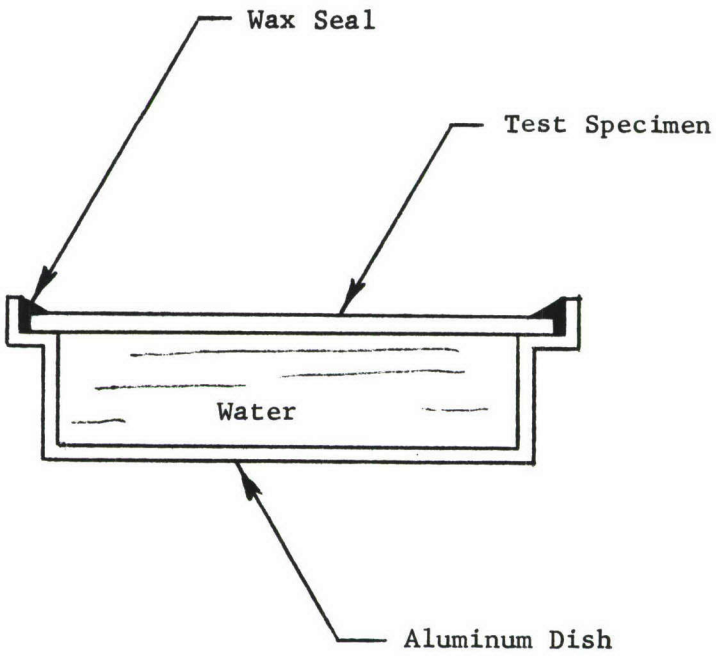


Figure 28 - Specimens for Water Permeability Tests

c. Apparatus: The specimens were tested in a 20,000 pounds capacity Tinius Olsen Electomatic testing machine.

The extensometer used to measure compressive deformation was capable of averaging the deformation from the two edges of the specimen, and had a sensitivity of .0155 inches per inch on a 2 inch gage length. An extensometer is shown with the compression fixture in Figure 29.

The compression fixture (Figure 30) was designed so the load was transmitted to the specimen ends by means of a self-aligning compression jig which reduced the effects of unequal loading. The jig was designed to test specimens up to 0.3" in thickness. Thicker specimens were tested without the jig. Modulus valves were not obtained for specimens thicker than 0.3". Stabilization was accomplished with side supporting steel fingers to prevent buckling of specimens.

d. Procedure: The rate of head travel of the testing machine under load was 0.05" per minute during the entire test. All specimens were tested in the direction perpendicular to molding pressure and parallel to flake direction. Load-deformation curves were obtained for each specimen up to 0.3" inch thickness. Modulus and ultimate strength values were calculated and recorded.

2.4.2.2 Tensile Properties

a. Scope: This method was designed to determine the ultimate tensile strength and tensile modulus of reinforced plastics. It is essentially in accordance with method 1011 of Federal specification L-P-406b.

b. Test Specimens: Specimens were cut from laminates identified as -13, -16, and -19 in Figure 21. These specimens were not as prescribed in L-P-406b, because previous experience had shown that the wider grip areas and narrower necked down sections were required to insure that a high percentage of the failures were within the gage area. Specimens were bolted between 1/8" thick flat steel plates through the holes shown in Figure 21. The plates were in turn mounted to a universal joint on the test machine to minimize bending during loading.

c. Apparatus: The specimens were tested in a 20,000 pounds capacity Tinius Olsen Electomatic testing machine. A Tinius Olsen tension extensometer S-1 was used. Sensitivity of .004 inches/inch was measured over a gage length of two inches.

d. Procedure: The rate of head travel of the testing machine was 0.05 inches per minute during the entire test. All of the specimens were tested in the direction perpendicular to molding pressure and parallel to the flake direction. Load extension curves were

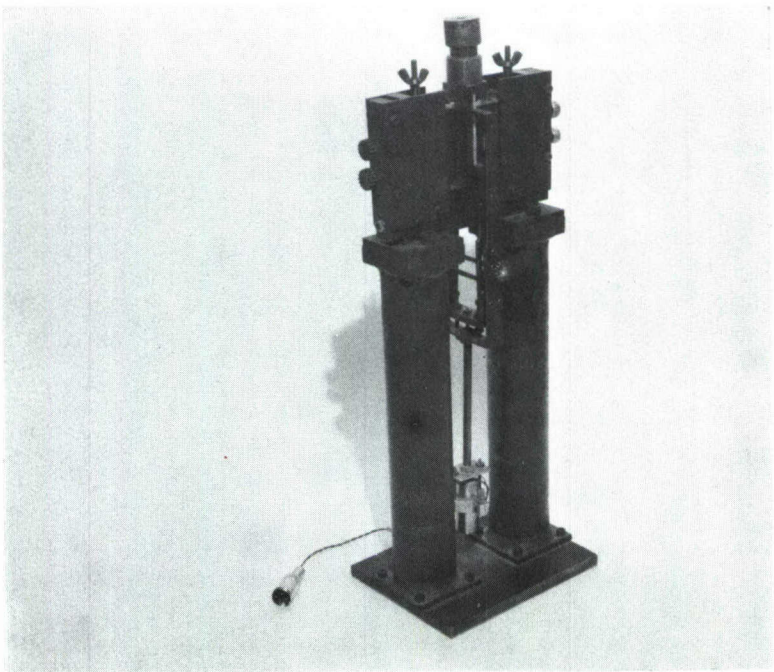


Figure 29 - Apparatus For Compression Tests
Showing Extensometer

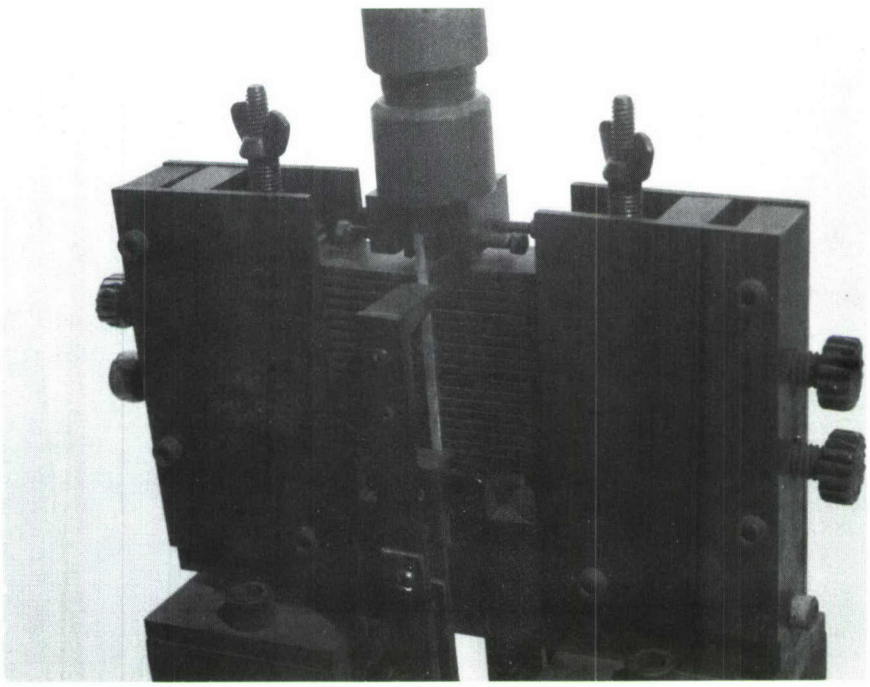


Figure 30 - Compression Testing Apparatus Showing Steel Fingers Supporting The Test Specimen

obtained for each specimen. Modulus and ultimate tensile strength values were calculated for each.

2.4.2.3 Flexural Properties

a. Scope: This method was in accordance with L-P-406b, method 1031. It was designed for use in determining the flexural properties of rigid plastic materials and laminated plastics.

b. Test Specimens: Specimens 4" x 1/2" x the material thickness were used. Thicknesses of the materials were sufficiently near 0.100" so that a 17 ± 1 span to depth ratio could be maintained with the 4" specimen.

c. Apparatus: The specimens were tested in a 20,000 pound capacity Tinius Olsen Electomatic testing machine. The extensometer used to measure deformation had a sensitivity of .0153 inches per inch. The contact edges of the supports and the pressure piece were rounded to a radius of 1/8 inch.

d. Procedure: The rate of head travel of the testing machine during the test was 0.05 inches per minute. A deflection was measured on a Tinius Olsen type of extensometer. All specimens were tested in the direction parallel to molding pressure and perpendicular to flake direction. Load deflection curves were obtained for each specimen. Modulus and ultimate flexural strength values were calculated.

2.4.2.4 Bearing Strength

a. Scope: This method was in accordance with L-P-406b, method 1051, designed for use in determining the ultimate bearing strength of rigid plastics. It was intended to apply where rivets, bolts or similar fasteners were to be used in joining sections.

b. Test Specimen: Bearing specimens were nominally 0.100 inch thickness with a 0.125 inch diameter bearing hole and an edge distance of five times the diameter of the hole. To minimize tensile failures, the specimen width was increased from 7/8 inch, specified in L-P-406b to 1.5 inches. The specimen is shown in Figures 21 and 26.

c. Apparatus: The specimens were tested in a 20,000 pounds capacity Tinius Olsen Electomatic testing machine. A three plate jig, such as shown in Figure 31, was used.

d. Procedure: The rate of head travel was 0.05 inches per minute. Bearing stress versus the deformation of the hole was not determined. Instead, the ultimate bearing strength was calculated from the maximum load that was supported by the specimens.

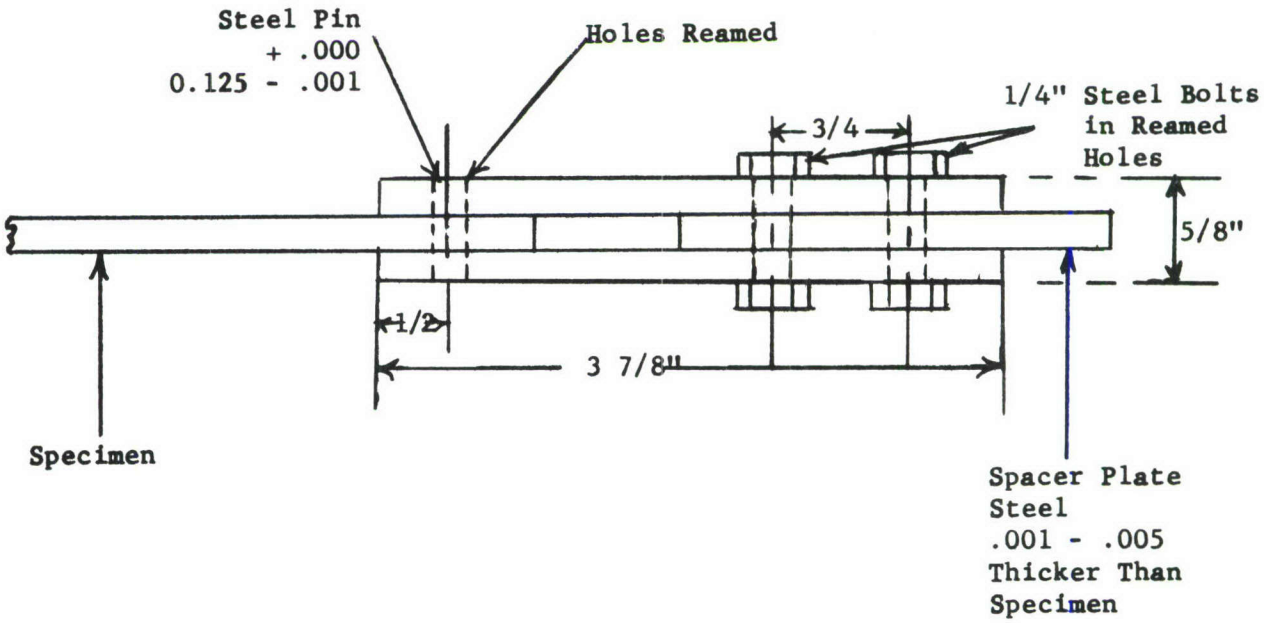


Figure 31 - Bearing Test Jig

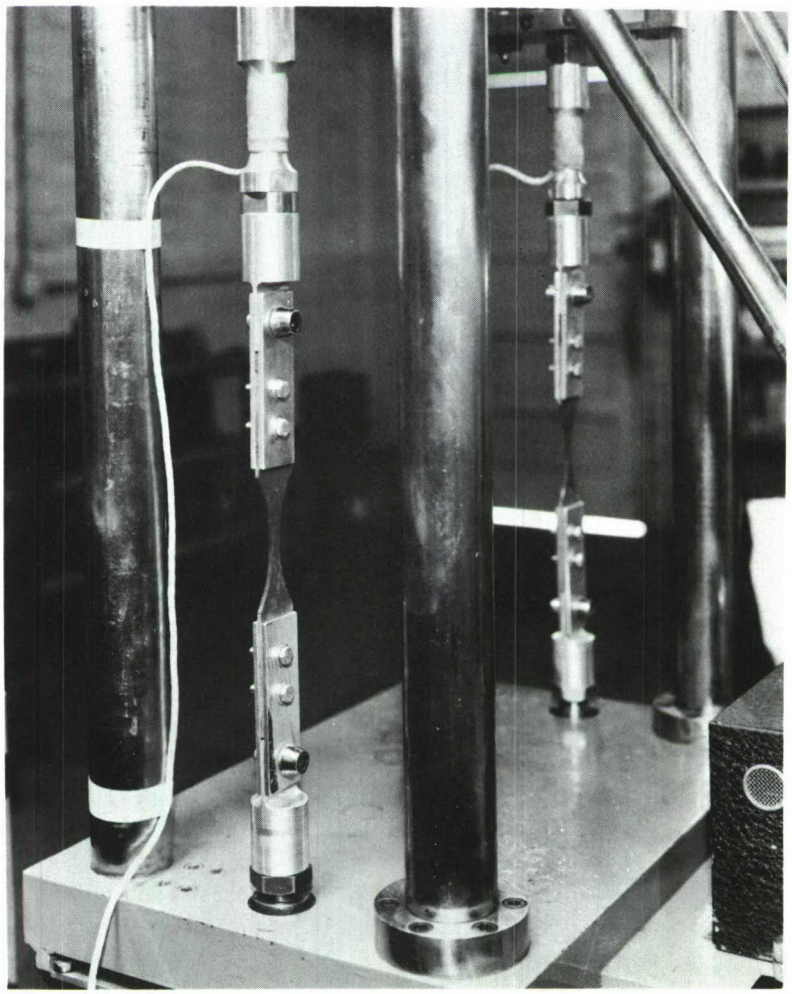


Figure 31A - Fatigue Specimens Mounted in the Krouse Fatigue Machine. Load cells are shown.

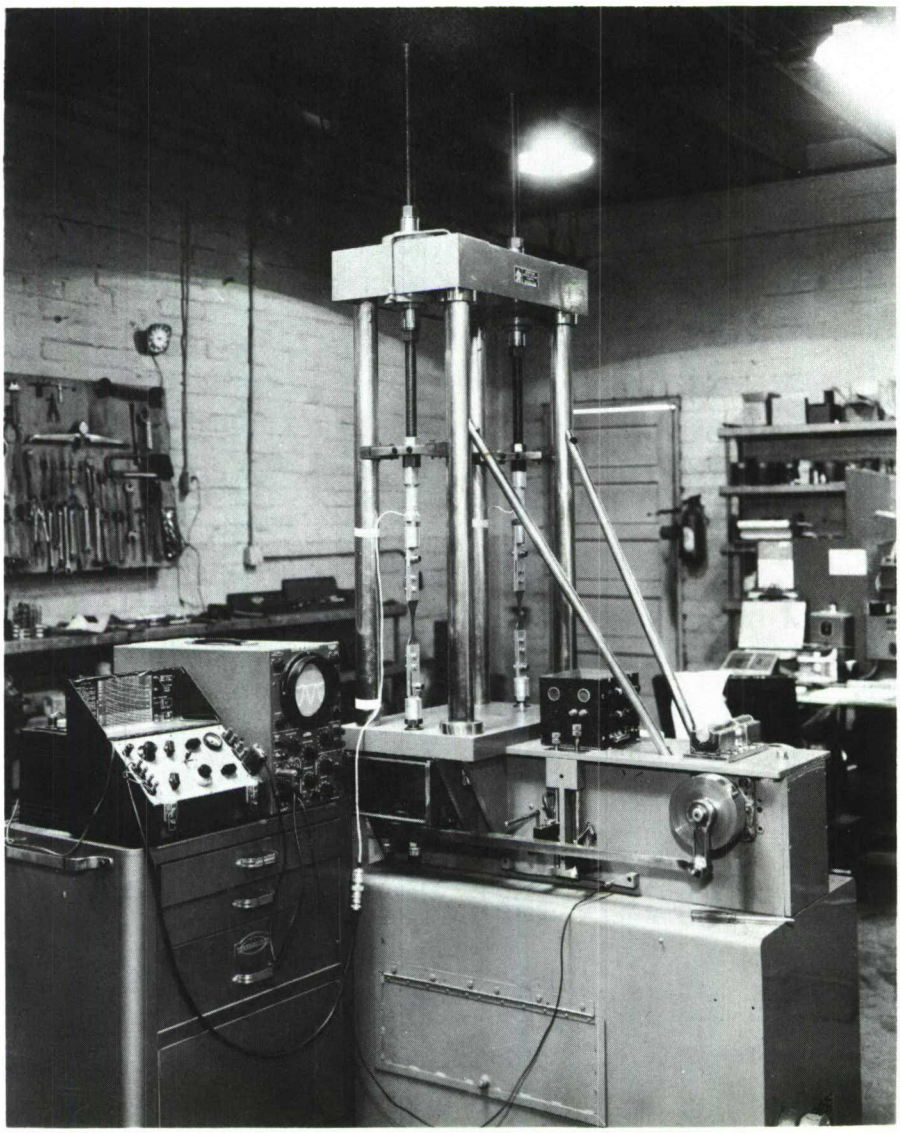


Figure 31B - The Krouse Fatigue Test Machine and Associated Dynamic Load Measuring Equipment

2.4.2.5 Impact Tests

a. Scope: The Izod impact strength test, performed in accordance with Federal Specification L-P-406b, method 1071, was used to determine the relative susceptibility of flake reinforced composites to fracture by shock. A standard pendulum-type impact test apparatus was used to determine the impact strength. Specimens were notched either on one face or on one edge.

b. Test Specimens: Three 1/2" x 6" x 6" laminates were molded from each of the four materials under investigation in the manner described above. Six 1/2" x 1/2" x 2-1/2" specimens were cut from each of these laminates. These specimens were then notched, as shown in Figure 32, by means of a special milling cutter. The notch was cut into the face or molded surface of three specimens from each laminate and into the edges of the remaining specimens.

c. Test Procedure: Since Narmco Industries, Inc. was not equipped with an Izod impact test machine, these tests were sub-contracted to Metal Control Laboratories, Inc., and independent testing laboratory. This laboratory performed the actual testing on a cantilever-beam, pendulum-type Izod impact testing machine.

2.4.2.6 Fatigue Tests

a. Scope: Approximate S-N diagrams for each of the 4 compositions were prepared. Specimens were loaded axially in tension.

b. Sample Preparation: A description of the samples is presented in Table 14 and in Figure 22. The necked-down section was surface ground to a uniform radius of 3". The ground area was polished with emery cloth following grinding.

c. Test Procedures: Narmco Inc. was not equipped to perform fatigue tests at that time; therefore, Metal Control Laboratories, Inc., of Huntington Park, Calif. was selected to perform these tests. The fatigue tests were performed on a Krouse 5000 lbs capacity fatigue machine modified so that the actual capacity during this series of tests was 1500 lbs. The machine has two loading stations which enabled us to run two specimens simultaneously. A 1000 lb capacity load cell was installed in series with each specimen. The specimens and load cell are shown in Figure 31A. Dynamic load cell readout equipment consisting of an Ellis BA-12 bridge and amplifier, and a Dumont oscilloscope was used to monitor the tests so that it was not necessary to inerrupt the fatigue tests to determine whether the loads were proper. This equipment is shown in Figure 31B. Each specimen was measured at the narrowest portion of the reduced section. Actual test loads were then calculated for each specimen based on the cross sectional area of the specimen, and the stress level desired.

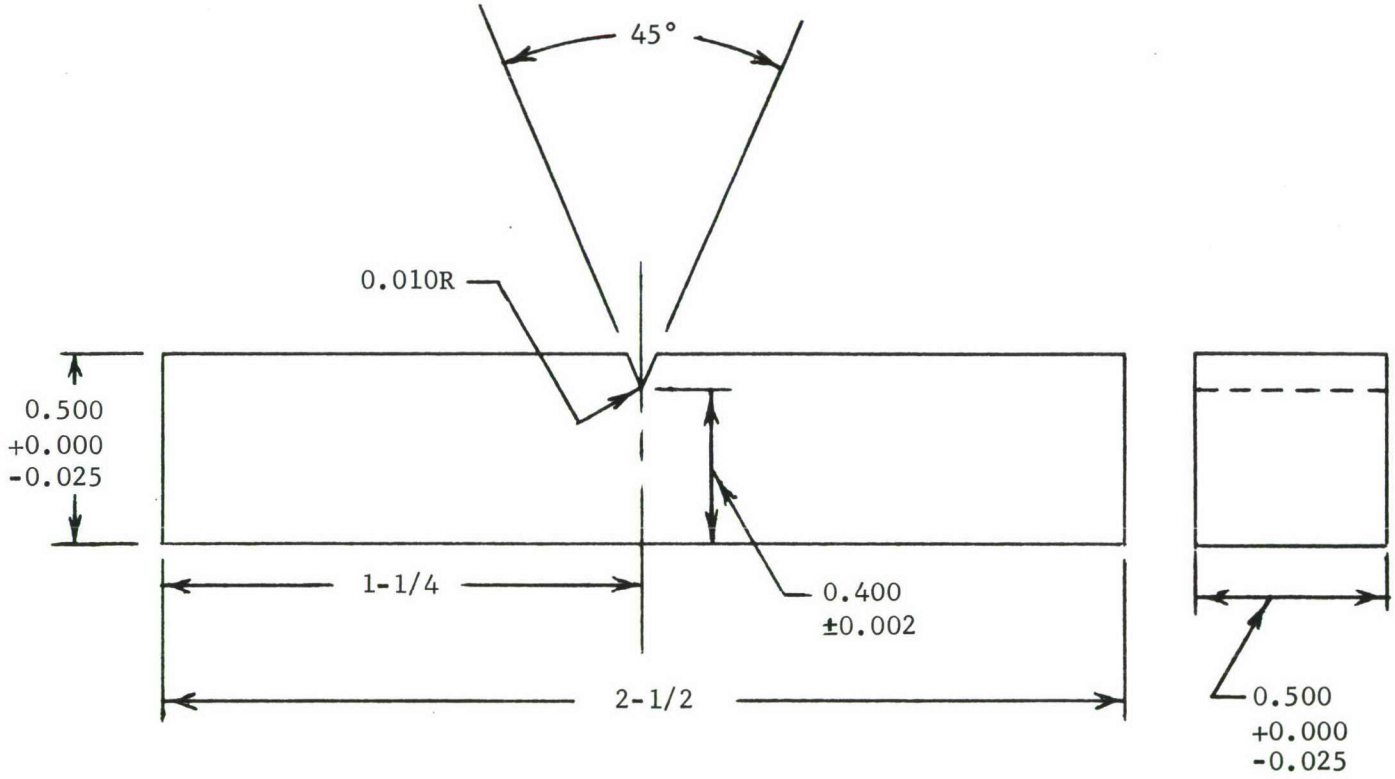


Figure 32 - Izod Impact Specimen

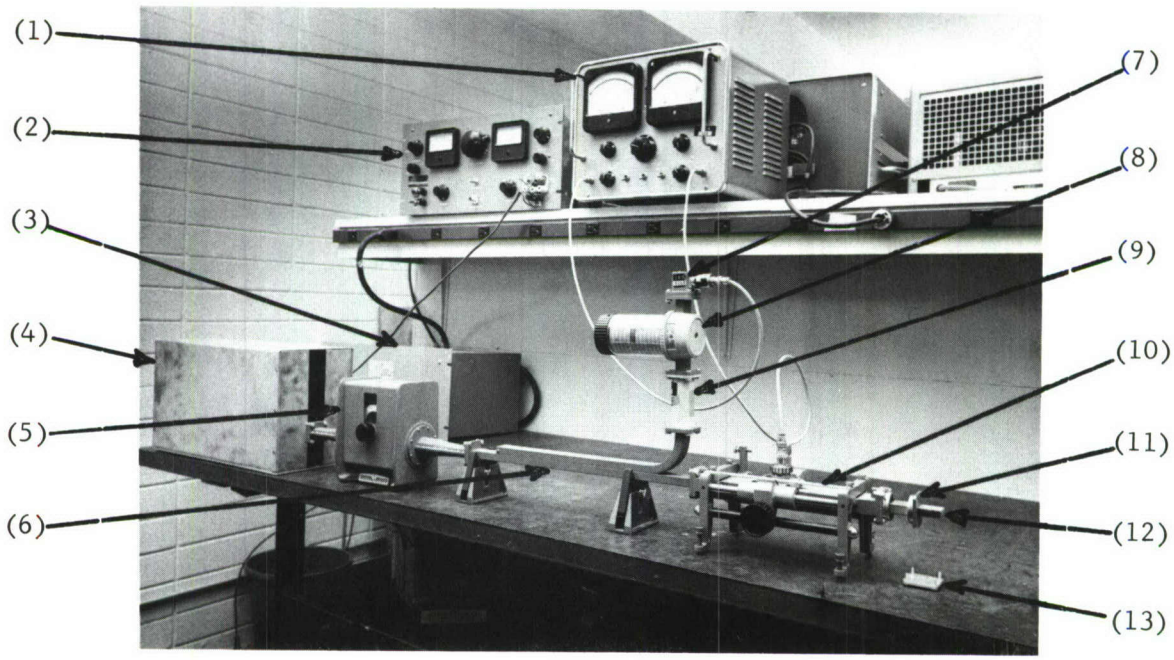
Loads were adjusted so that the minimum tensile load that was applied to each specimen was equal to 10 percent of the maximum tensile load.

2.4.3 Electrical Properties

a. Scope: The electrical properties which would be pertinent to the designer of radomes or similar structures were determined.

b. Test sample Preparation: Prepration of specimens was described above and specimens are shown in Figures 23-26. Specimens for dissipation factor and dielectric strength at 9375 megacycles in the direction perpendicular to the plane of the flakes were prepared by laminating the blocks, identified as -44 in Figure 24, with the parent resin for the respective molding compound. Specimens for dielectric strength measurements were surface ground to a thickness of 0.010 inches.

c. Test Procedure: The dielectric constant and loss tangent of the materials were evaluated at 1 mc by the resonant circuit susceptance variation method. The method was similar in important details to method 4021 of L-P-406b. The apparatus used for those tests is shown in Figure 36. During the tests the room temperature was 72°, relative humidity 40%. The active area of the sample holder (capacitive plates) is a two inch circle. The sample thickness was 0.125 inches. It was noted in connection with the measurements taken after immersion in boiling water that a delay of the order of two minutes was necessary between drying the surface of the sample and performing the measurements. A very rapid decay during this two minute interval was noted in the loss tangent. Since this time period was much too short to allow any significant change in internal water content, the short delay in measurement after the water conditioning was adopted for these data. The measurements of dielectric constant and loss tangent at 9375 mc were performed by the shorted waveguide technique essentially as described in method 4021 of L-P-406b. The sample was sanded to fit precisely into a sample holder which was fabricated from 1" x 1/2" waveguide. Testing equipment is shown in Figure 33. The laboratory temperature and relative humidity were as before (71°F and 42% respectively) during these tests. The measurements were made on the same samples before and after the immersion for two hours in boiling distilled water. It was noted that the physical dimensions of the samples increased as a result of the water immersion. The amount of increase varied from sample to sample. The procedure used in preparing the samples for re-measurement after the water immersion was 1) dry the surface of the sample by a short airblast, 2) allow the specimen to cool for 5 minutes, then retrim the sample to the proper size to fit the internal dimensions of the sample holder. This uniform procedure was used for all tests. There is an apparent difference between the effects of water



(1) Tuned Amplifier

(2) AC Regulator

(3) Power Supply

(4) X-Band Source

(5) Precision Attenuator

(6) Directional Coupler

(7) Detector

(8) Wave Meter

(9) Isolator

(10) Slotted Line

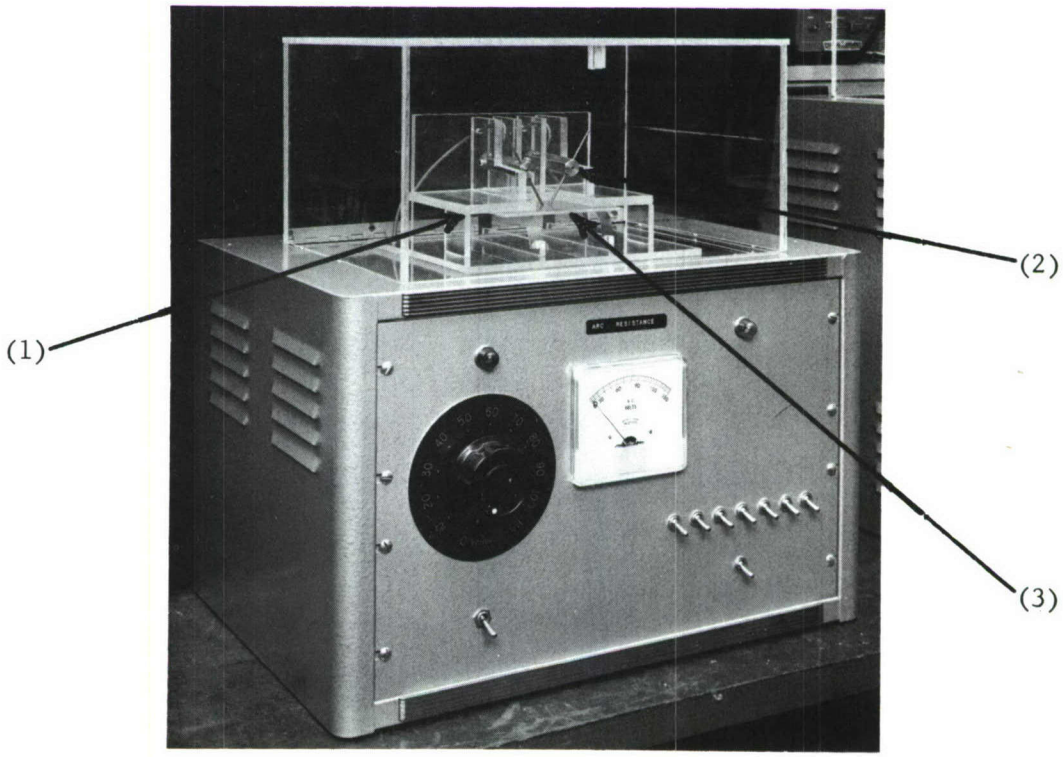
(11) Specimen Holder

(12) Specimen

(13) End Plate

Figure 33 - 9375 MC Dielectric Constant Tester
(Smyth Research Associates)

immersion in this case and the effects described in Para. 2.4.1.2 of this report. In the tests described in Para. 2.4.1.2, the specimens were exposed to a 24 hour immersion in water at room temperature and no measurable dimensional changes were observed. The exposire described above for the dielectric constant and loss tangent measurements was an immersion in boiling water for 2 hours. This exposure resulted in a change in the size of the samples that was sufficiently large to prevent reinsertion of the specimens in the sample holder. However, as the samples had originally been precisely fitted to the sample holder, only very small dimensional change would cause this interference. These changes were very small, in fact, all were less than .002 inch or 0.2% along the shorter dimension. The dielectric strength measurements were made using the "test to breakdown, short time test" procedures at power line frequency. In accordance with method 4031 or L-P-406b the rate of voltage rise used was 1.0 kilovolt per second until rupture of the material occurred. Two inch diameter flat metal electrodes were placed on either side of the samples during the tests. All samples were supplied machined to 0.010 inches thickness. Figure 34 shows the test equipment. An apparent increase in dielectric strength indicated by some of the measurements after water soak. These were caused by an unavoidable test area selection which was obviously different for the "as received" and water soaked conditions. From the results of these measurements, trends can be established, but more conclusive is the indication that other sample preparation means would be necessary to produce uniform electrical test specimens of this thickness from these materials. Arc resistance tests were made to evaluate the effects of a high voltage, low current arc on the insulation resistance of the dielectric material. Apparatus which was used for these tests is shown in Figure 35. Considerable variability was noted in the characteristics measured for some of the samples tested. In the cases where arc resistance time was short, (less than 50 seconds) the arc was formed almost immediately in the surface of the material. The surface material burned with a slight red glow during the arc duration. A dark charred residue formed which could be flaked off leaving a thin craze in the surface. The tests performed on the material which withstood the arc for longer periods of time burned on the surface only after long exposure to the arc. The resulting dark charred residue again could be easily chipped off and the burned area seen. The width of this burned zone was about 1/10 inch in width over the entire 1/4 inch arc path. All specimens measured 0.125 inches in thickness. As Narmco Inc.'s laboratories were not equipped for these electrical measurements, testing was performed by Smyth Research Associates, an independent laboratory.

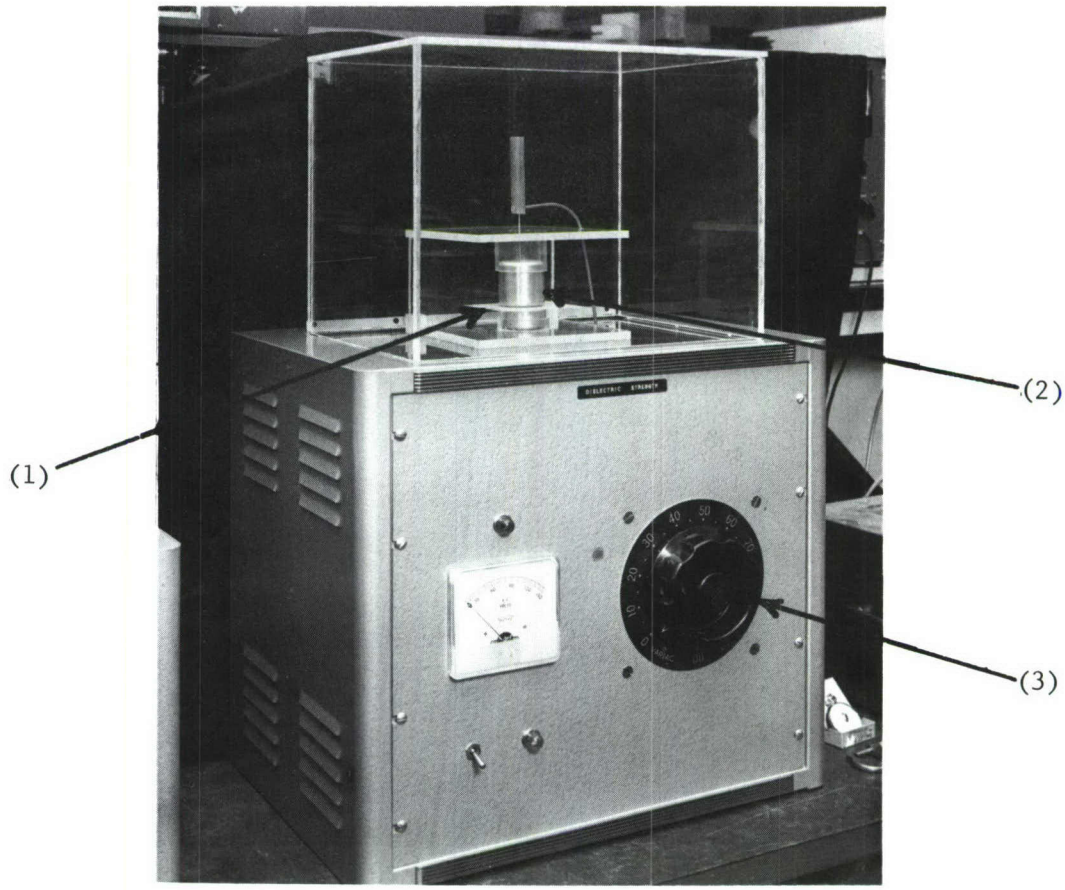


(1) Specimen

(2) Electrodes

(3) Hy-pot

Figure 34 - ARC Resistance Tester
(Smyth Research Associates)

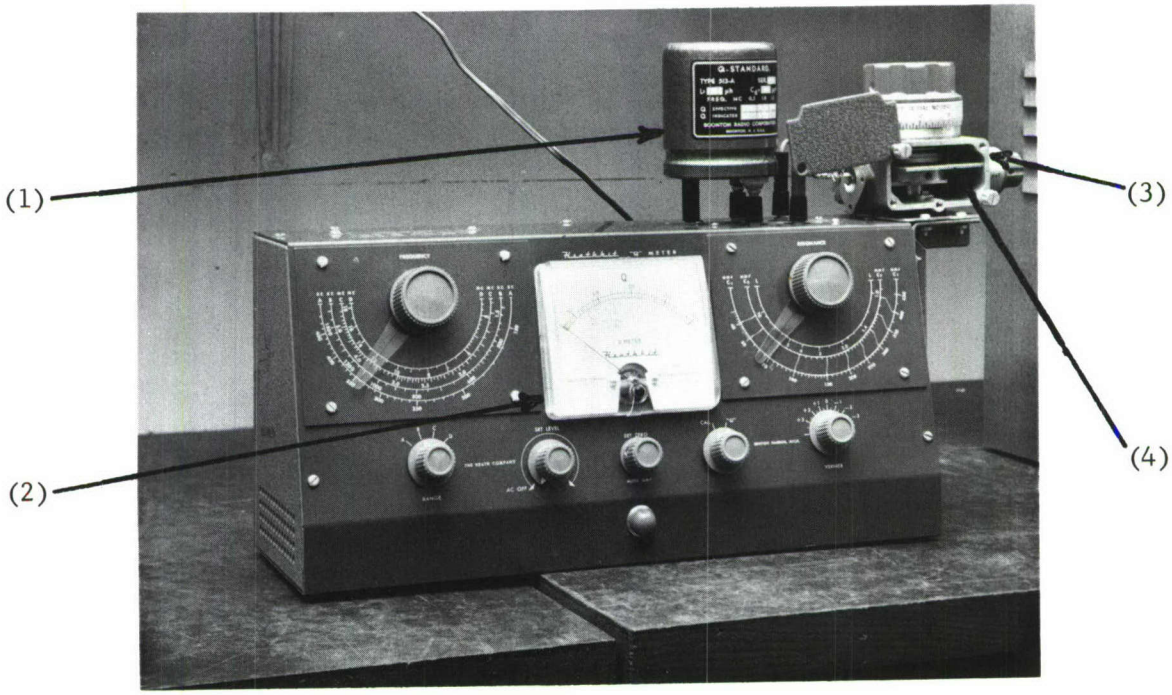


(1) Specimen Holder

(2) Electrodes

(3) Specimen

Figure 35 - Dielectric Strength Tester
(Smyth Research Associates)



(1) Q Standard

(2) Q Meter

(3) Specimen Holder

(4) Specimen

Figure 36 - 1 MC Dielectric Constant Tester
(Smyth Research Associates)

3.0 TEST RESULTS

3.1 Physical Tests

The results of the tests to determine physical properties are summarized in Table 15 and in Figures 37-40.

TABLE 15 A SUMMARY OF THE RESULTS OF THE PHYSICAL PROPERTY DETERMINATIONS

TEST	TEST METHOD	TEST RESULTS			
		X 270	X 271	X 273	X 274
Specific Gravity	L-P-406b Method 5011	1.98 (12)	1.93 (12)	1.94 (12)	1.94 (12)
Water Absorption (a) % Chg. in weight	L-P-406b Method 7013	.07 ± .02 (6)	.06 ± .01 (6)	.09 ± .03 (6)	.06 ± .03 (6)
(b) Dimensional Change		NIL	NIL	NIL	NIL
Thermal Conductivity	Cenco-Fitch Conductivity Method	1.8 ± .02 (3) BTU-in/°F-hr/ft^2	2.1 ± .18 (3) BTU-in/°F-hr/ft^2	1.8 ± .17 (3) BTU-in/°F-hr/ft^2	1.5 ± .07 (3) BTU-in/°F-hr/ft^2
Thermal Expansion	L-P-406b Method 2031	See Figure 37	See Figure 38	See Figure 39	See Figure 40
Flame Resistance	L-P-406b Method 2021.1	Self Extinguishing	Self Extinguishing	Self Extinguishing	Self Extinguishing
Water Vapor Permeability	L-P-406b Method 7031	less than 5x 10^{-3} gram/meter/day	less than 5x 10^{-3} gram/meter/day	less than 5x 10^{-3} gram/meter/day	less than 5x 10^{-3} gram/meter/day

NOTES: 1. Figures in parentheses are the number of samples used.

2. Figures following plus or minus signs are standard deviations (estimated) from the formula

$$\sigma^2_{est} = \frac{\sum_{i=1}^{N} (X_i)^2 - \frac{\left(\sum_{i=1}^{N} X_i\right)^2}{N}}{N - 1}$$

σ = standard deviation
N = number of samples

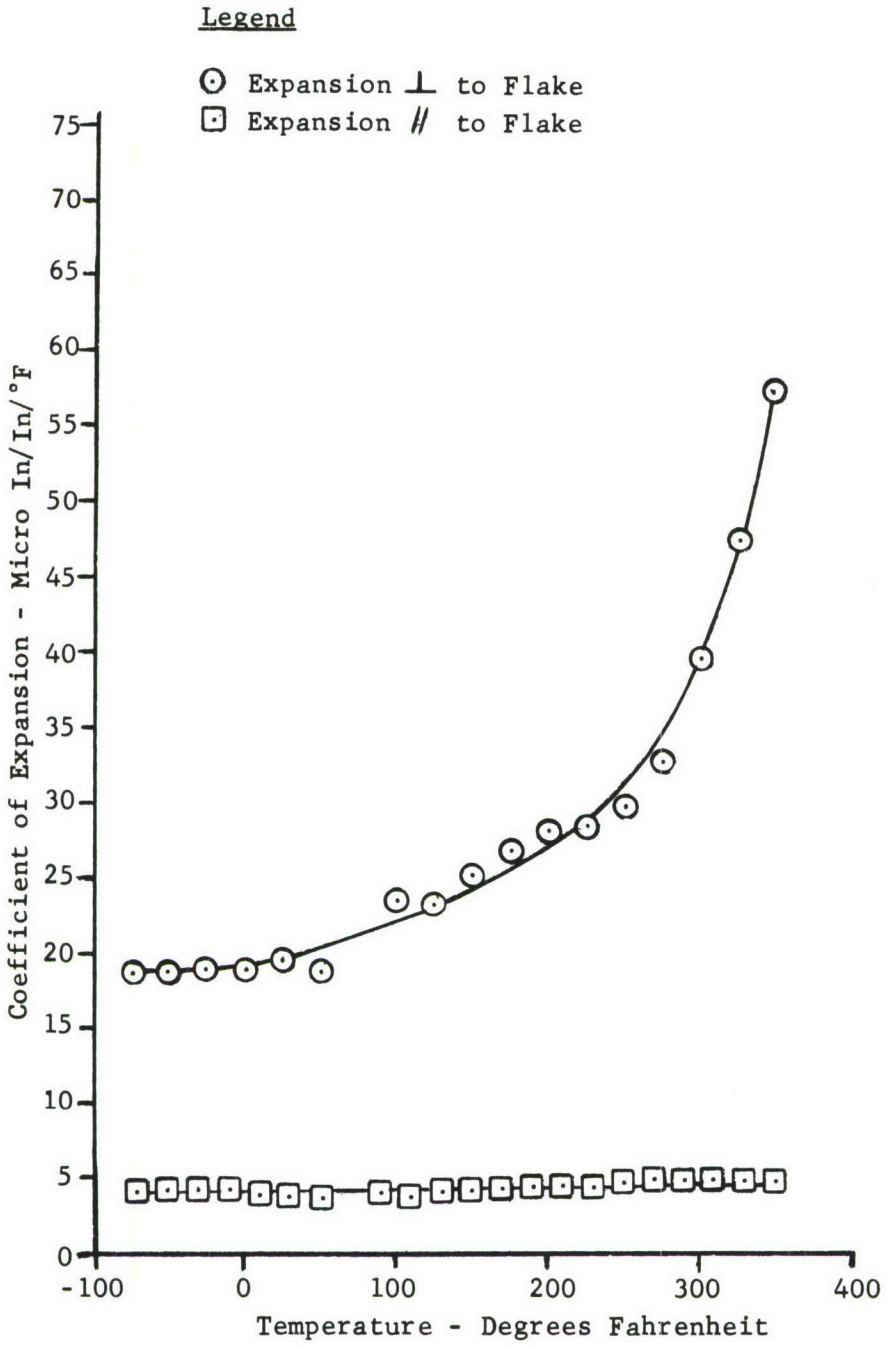


Figure 37 - Thermal Expansion for X270 Molding Material

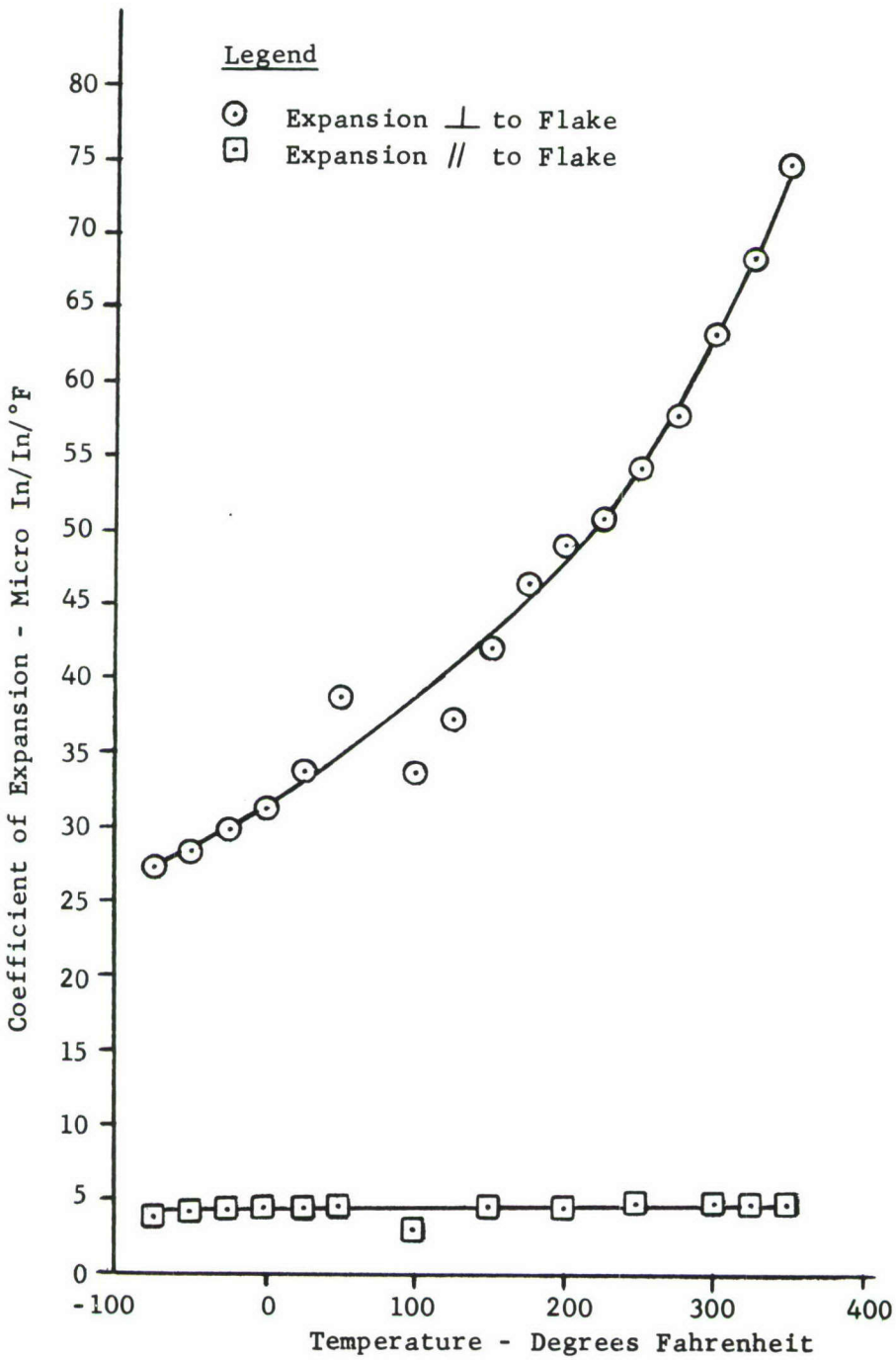


Figure 38 - Thermal Expansion for X271 Molding Material

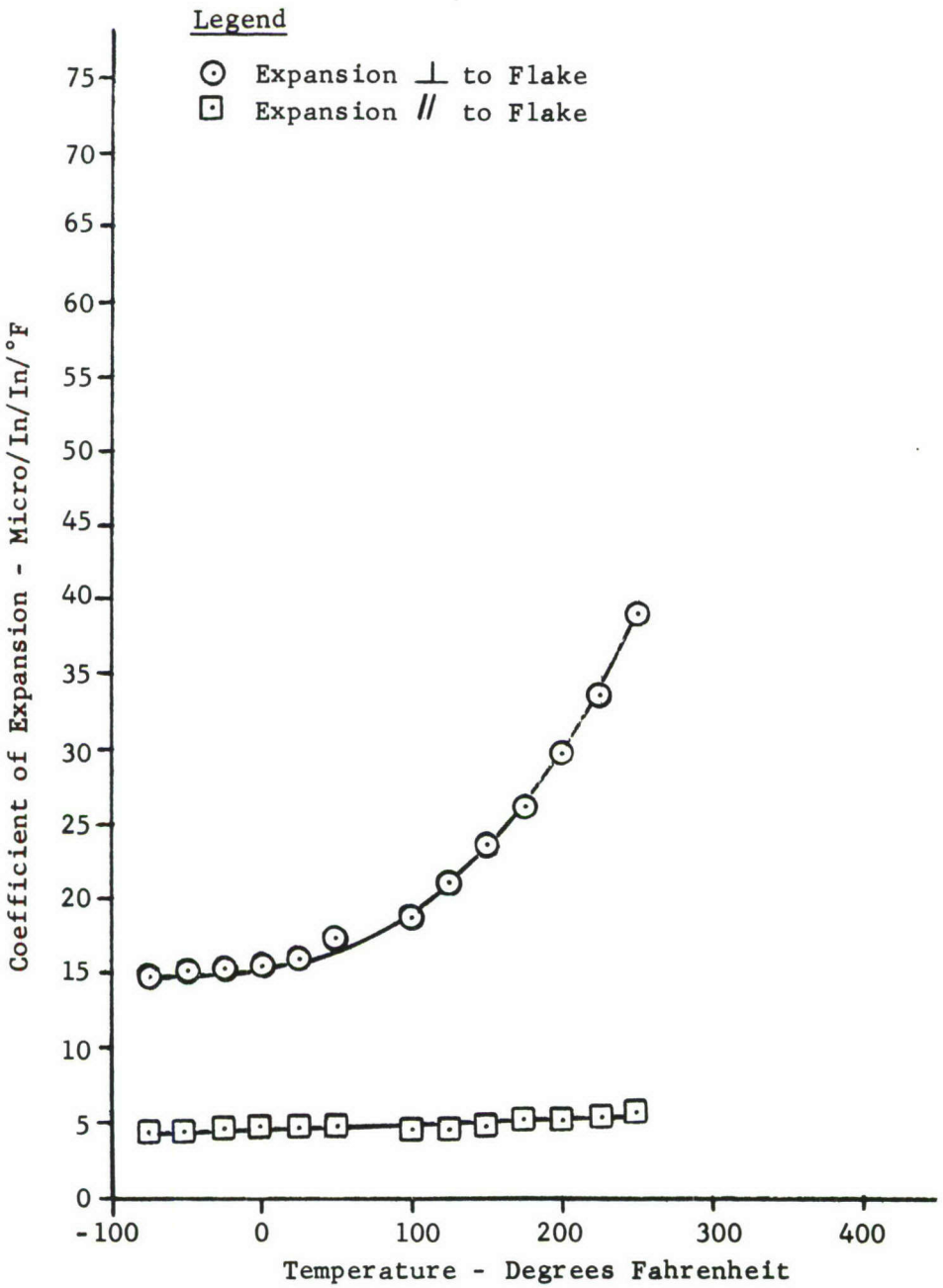


Figure 39 - Thermal Expansion for X273 Molding Material

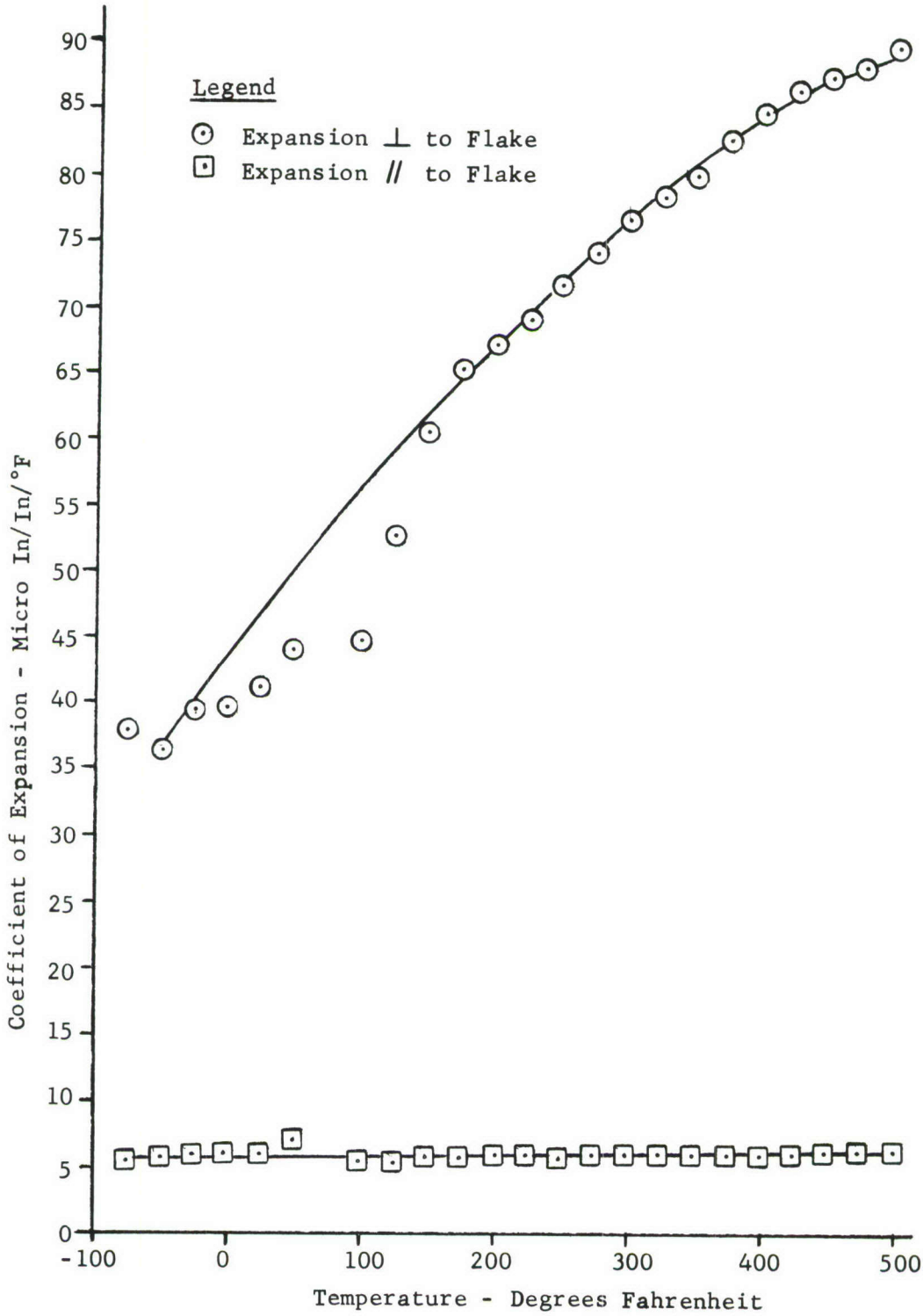


Figure 40 - Thermal Expansion for X274 Molding Material

3.2 Mechanical Tests

TABLE 16 A SUMMARY OF MECHANICAL PROPERTY DETERMINATIONS

Test	Test Method	Test Results			
		X270	X271	X273	X274
Ultimate Compressive Strength(psi)	Aircraft Industries ARTC-11	48200±3800 psi (13)	46500±2800 psi (15)	31900±6100 (15)	17200±1500 (15)
Compressive Modulus (psi x 10^{-6})	"	4.87 ± .17 (13)	4.44 ± .36 (15)	4.90 ± .30 (15)	4.55 ± .34 (15)
Ultimate Tensile Strength(psi)	L-P-406b Method 1011	21100 ± 1600 (12)	21600 ± 2700 (12)	12000±3200 (12)	9800+1300 (12)
Tensile Modulus (psi x 10^{-6})	L-P-406b Method 1011	5.19 ± .35 (12)	4.89 ± .19 (12)	5.02 ± .23 (12)	4.66 ± .46 (12)
Ultimate Flexural Strength(psi)	L-P-406b Method 1031	38400 ± 2400 (15)	34400 ± 3900 (15)	25300±3700 (15)	20800±2300 (15)
Flexural Modulus (psi x 10^{-6})	L-P-406b Method 1031	5.42 ± .20 (15)	4.99 ± .14 (15)	5.28 ± .12 (15)	4.38 ± .20 (15)
Bearing (a) Ult. Stress(psi)	L-P-406b Method 1051	51800±2100 (12)	47500±2500 (12)	41200±3000 (12)	32700±6100
(b) Type of failure		100% Tensile	100% Tensile	84% Tensile	100% Bearing
Impact Strength	L-P-406b Method 1071		See Table 17		
Fatigue Tests	Axial Loading	see Figure 41	see Figure 42	see Figure 43	see Figure 44

NOTES: 1. Figures in parenthesis are the number of samples used.

2. Figures following the plus or minus sign are standard deviations (estimated) from the formula

$$\sigma_{est} = \frac{\Sigma x^2 - \frac{(\Sigma x)^2}{n}}{n - 1}$$

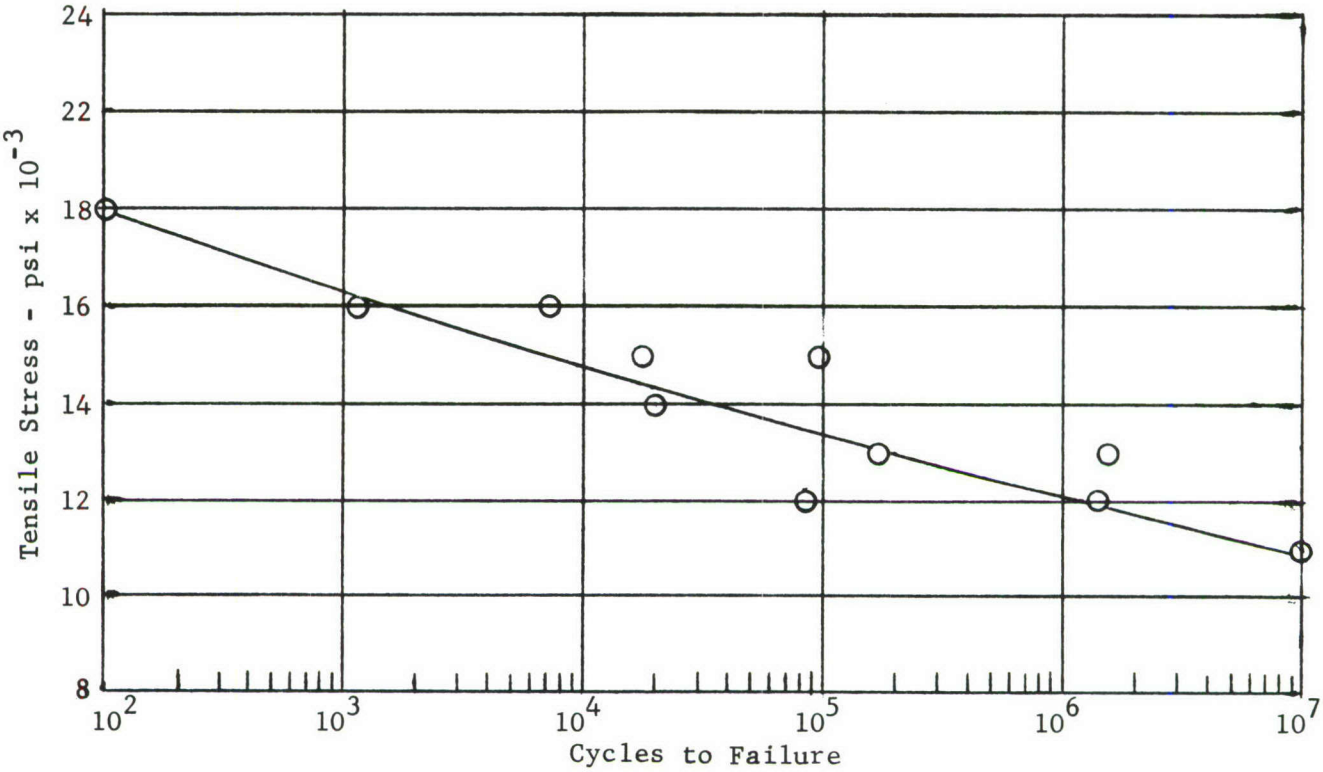


Figure 41 - S/N Diagram for X270 Molding Material

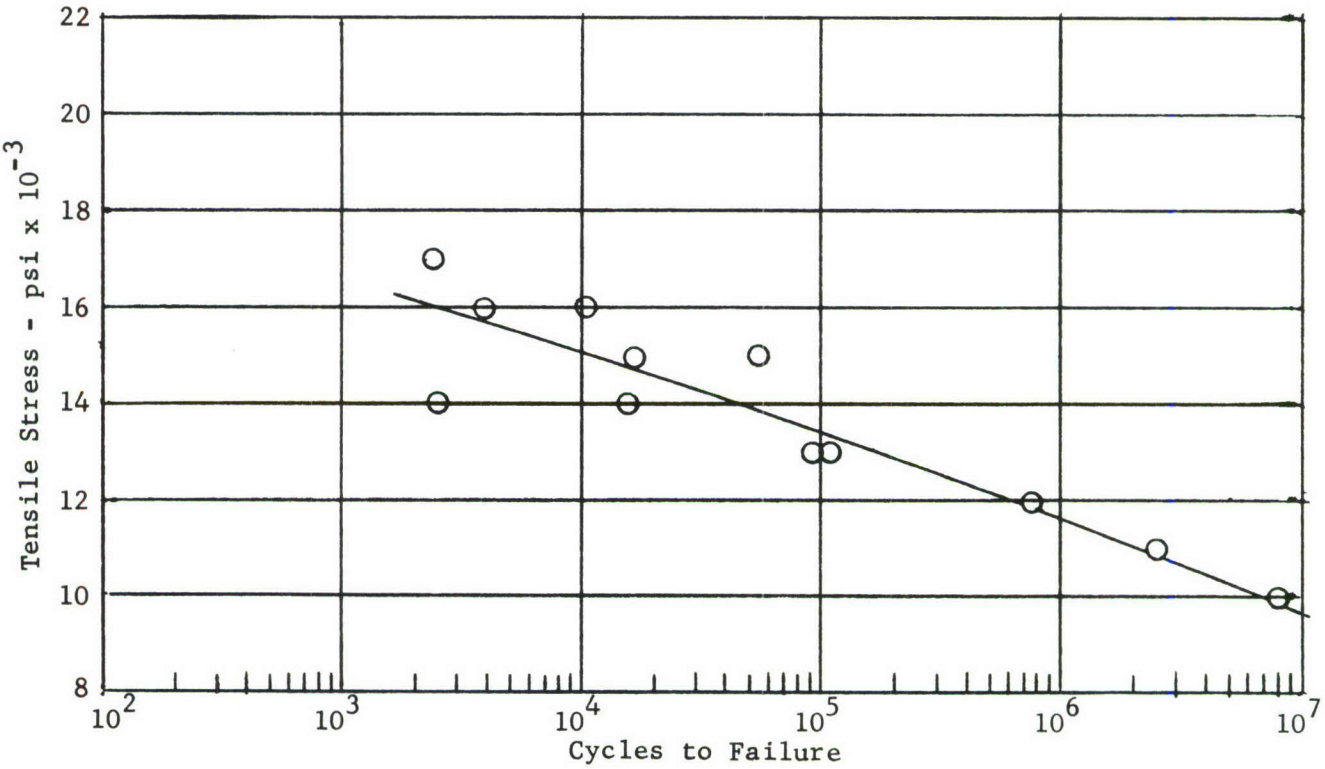


Figure 42 - S/N Diagram for X271 Molding Material

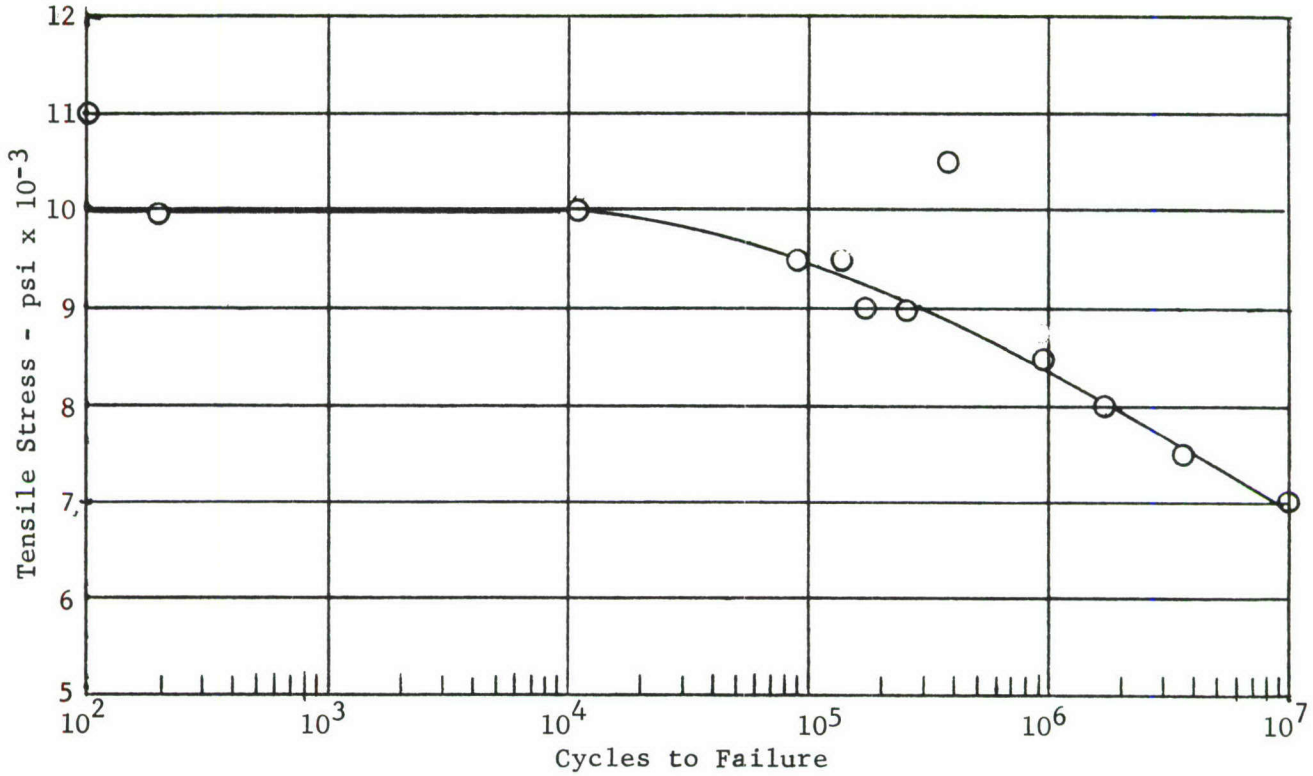


Figure 43 - S/N Diagram for X273 Molding Material

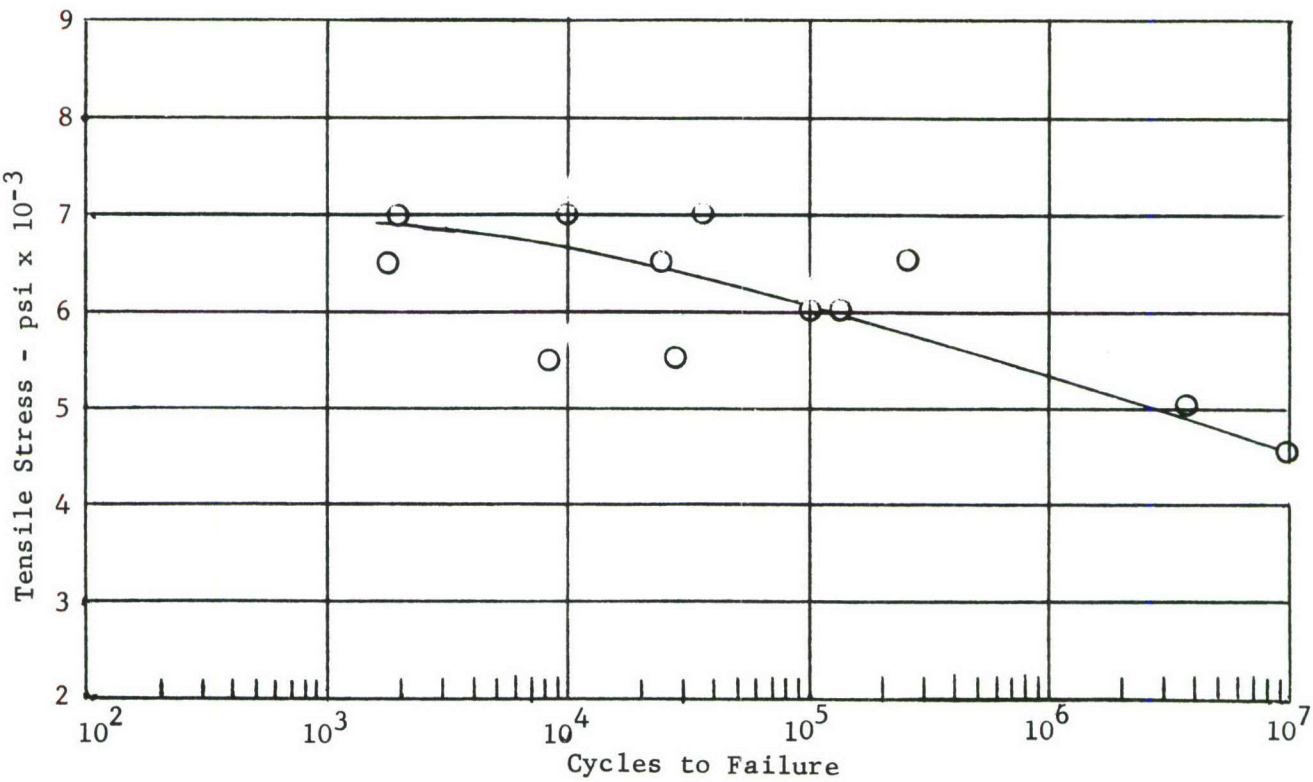


Figure 44 - S/N Diagram for X274 Molding Material

TABLE 17 IZOD IMPACT TEST RESULTS

Molding Composition	Notch Located in Face of the Original Material		Notch Located in the Edge of the Original Material	
	Impact Strength (ft-lb/in of Notch)	Standard Deviation (est)	Impact Strength ft-lb/in of Notch	Standard Deviation (est)
X 270 Epoxy	1.72	0.50	0.88	0.24
X 271 Epoxy	2.18	0.69	1.01	0.01
X 273 Phenolic	1.95	0.51	0.79	0.18
X 274 Silicone	1.44	0.33	0.89	0.33

3.3 Electrical Tests

The results of the test to determine the electrical properties of the molding materials are summarized in Tables 18-21.

TABLE 18 DIELECTRIC CONSTANT AND LOSS TANGENT

(1 Megacycle Tests)

Samples	Standard Condition		After 2 hours Immersion in Boiling Water	
	Average Dielectric Constant	Average Loss Tangent	Average Dielectric Constant	Average Loss Tangent
X-270	4.48 ± (.12)	.012 ± (.001)	4.38 ± (.17)	.013 ± (.004)
X-271	5.02 ± (.13)	.016 ± (.001)	5.02 ± (.08)	.016 ± (.001)
X-273	5.32 ± (.26)	.020 ± (.005)	5.44 ± (.25)	.025 ± (.008)
X-274	3.96 ± (.08)	.003 ± (.001)	3.94 ± (.03)	.003 ± (.001)

Note: Numbers in parentheses are estimate standard deviations

TABLE 19 DIELECTRIC CONSTANT AND LOSS TANGENT

(9375 Megacycle Tests)

Samples	Standard Condition		After 2 hours Immersion in Boiling Water	
	Average Dielectric Constant	Average Loss Tangent	Average Dielectric Constant	Average Loss Tangent
X-270	4.65 ± (.21)	.033 ± (.003)	4.64 ± (.18)	0.36 ± (.004)
X-271	4.75 ± (.21)	.034 ± (.005)	4.76 ± (.23)	.035 ± (.004)
X-273	5.02 ± (.12)	.031 ± (.002)	5.03 ± (.14)	.034 ± (.003)
X-274	4.35 ± (.23)	.008 ± (.001)	4.40 ± (.24)	.012 ± (.003)

Note: Numbers in parentheses are estimated standard deviations

TABLE 20 DIELECTRIC STRENGTH

(Breakdown Voltage)

Samples	Standard Conditions Average Dielectric Strength Volts/mil	After 2 hours Immersion in Boiling Water Average Dielectric Strength Volts/mil
X-270	995 (580-1150)	775 (282-1115)
X-271	851 (470-1210)	1079 (865-1230)
X-273	396 (110-930)	294 (221-386)
X-274	499 (210-805)	396 (171-820)

Note: Numbers in parentheses are ranges

TABLE 21 ARC RESISTANCE VOLTAGE BREAKDOWN

Samples	Standard Conditions				After 2 hours Immersion in Boiling Water			
	Average time (second)	V_1	V_2	V_2/V_1	Average time (second)	V_1	V_2	V_2/V_1
X-270	199 (192-210) [9]	5925	29	.0049	202 (193-211) [9]	5700	20	.0035
X-271	227 (225-229) [5]	5850	29	.0050	234 (217-242) [5]	5775	77	.0133
	15 (13-17) [4]				10 (7-14) [4]			
X-273	184 (182-185) [3]	5700	15	.0026	153 (103-185) [8]	4867	15	.0031
	36 (22-50) [7]				11 (11) [1]			
X-274	192 (183-207) [9]	5700	76	.0133	244 (242-245 [9]	5425	57	.0105

Note: (1) Numbers in parentheses are ranges

(2) Numbers in brackets are number of tests

4.0 DISCUSSION OF THE TEST RESULTS

4.1 Physical Property Determinations

In general, the physical properties of the flake reinforced composites were essentially as would be predicted from the arrangement of the flake reinforcement within these moldings. For example, because of the laminar nature of the moldings, very low water vapor permeability and water absorption would be anticipated for these composites. Our determinations showed that this was true. One anomaly was noted, in that the thermal expansion of the flake reinforced composites is anisotropic. In the plane of the flake, the thermal expansion of the composite approaches that of the E glass flakes (2.8×10^{-6} in/in/°F), whereas, in a direction perpendicular to the flake, these values are of the same order as the thermal expansion of the resin systems, (approximately 30×10^{-6} in/in/°F).

4.2 A Discussion of The Mechanical Property Determinations

In general, the mechanical properties were much as expected from earlier work with these materials. Several exceptions were noted; for example, nearly all bearing tests resulted in tensile, rather than bearing, failures of the specimens. As a measure of the notch sensitivity of these materials, the Table below was prepared to compare the average tensile strength from conventional dog bone specimens to the tensile strength observed as the bearing specimens failed.

TABLE 22 NOTCH SENSITIVITY OF FLAKE COMPOSITES

Molding Material	Tensile strength of Dog-bone specimens (L-P-406b)	Tensile Strength at Failure of the Bearing Specimens
X270	21,100 psi	4380 psi
X271	21,600 psi	4300 psi
X273	12,000 psi	3780 psi
X274	9,800 psi	All bearing failures

An additional indication of this notch effect was noticed earlier during the tensile testing. At that time it was found that the width of the shank portion of the dog bone tensile specimens had to be increased from 0.75" to 1.50" before it was reasonably certain that the failures would occur within the desired area.

It appears that this notch sensitivity is a definite shortcoming of the molded flake composites. Although it is not within the scope of this program,

a further investigation of this phenomena would be required before a bolted or riveted structure could be designed from these materials.

It was also noted that the values for impact strength for all of the composites were only about 1/10 of the values normally associated with parallel laminated fiberglass laminates. Although these low values could quite probably be improved if a resin system were specifically selected for impact resistance, or if resin content were increased, it is not likely that sufficient improvement could be achieved to reach the same impact resistance normally found in fiber reinforced laminates.

4.3 A Discussion of Electrical Properties Determinations

Among the electrical properties determined, only the dielectric strength was lower than had been anticipated. In this case, it was predicted that flake reinforced composites would have very high dielectric strengths, because the laminar nature of the flake in the composites would cause any electrical currents to follow tortuous paths through the laminate. However, as is shown in Table 20, the dielectric strength of the composites is not particularly outstanding and the results of these determinations were badly scattered. It should be noted that these low values could be attributed to a poorly selected sample configuration, rather than to an inherent weakness in the molded composites. As was specified in the section at this report that deals with sample preparation, the dielectric specimens were prepared by grinding .125 inch thick specimens to a thickness at .010 inch. The thin specimen was required because dielectric strengths at over 1000 volts/mil had been observed for flake reinforced composites and the maximum potential difference that available test equipment could supply was limited to 100,000 volts. Grinding of thicker specimens was selected as a method for sample preparation rather than the direct molding of thinner specimens because all of our efforts to mold flake reinforced composites less than 0.020 inch thick were unsuccessful. The problem observed when attempts were made to mold very thin specimens was one of poor flow of the resin in the composites. Even when pressures up to 4000 psi were applied to 6" square moldings, the area around the periphery of the laminates was white and crumbly, as though the flakes had not been wet by the resin. These laminates resembled the 12" square laminates that were described in paragraph 2.2 of this section.

Ground specimens were thought to be a poor selection, because some slight wrinkling occured even in the apparently good quality laminates. This wrinkling was not serious enough to adversely affect the mechanical properties of the 1/8" laminates; however, when the laminates were ground to 0.010", the wrinkled areas provide relatively short paths for the electric current.

SECTION IV TESTS AT ELEVATED TEMPERATURES

1.0 SCOPE

This series of tests was designed to determine the effect of exposure to elevated temperatures on the mechanical properties of flake reinforced composites. The elevated temperature resistance of resins used in the glass flake reinforced composites is normally improved by exposing these resins to a post cure. Therefore, it was necessary to make a short study of the effect of a post cure cycle on the glass flake reinforced composites before attempting to test the elevated temperature resistance of these composites. In addition, such a study was necessary because of the "blowing" or delamination of glass flake composites during the screening tests. This was noted when attempts were made to remove composites from the mold without cooling to well below the curing temperature. Because of the blowing problem, this study was designed to find a post cure cycle which would allow each system to be exposed to elevated temperatures without a deleterious effect on the room temperature mechanical properties of the composites.

2.0 PREPARATION OF SPECIMENS

To determine if a post cure cycle had a deleterious effect on the room temperature mechanical properties of the composites, it was desirable to compare the two halves of a laminate after one of these portions had been subjected to a post cure.

Specimens were supplied for these tests from three 12" square, 1/8" thick laminates molded with each of the four compositions selected during the screening tests. Processing details are presented in Table 23. After molding, each laminate was cut into two 6" x 12" pieces and one-half of each laminate was tested in tension, flexure and compression at room temperature in the same manner as the laminates described in Section II of this report. The remaining half of each laminate was subjected to one of the post cure cycles described below:

2.1 Post Cure Number 1

This post cure was the first exposure to which the moldings were subjected and was intended to demonstrate the maximum temperature which each system could withstand. This cycle was performed by placing the moldings into an air circulating oven at 200°F. After this temperature had been maintained for 24 hours, the temperature was increased to 250°F and again the temperature was maintained for 24 hours. This process was repeated, using 50°F increases in temperature and 24 hour soak periods, until the laminates had been exposed to 500°F for 24 hours. The laminates were examined visually after 12 hour intervals during the post curing cycle.

2.2 Post Cures Number 2 and Number 3

Both these post cures were based on cycle number 1 and were intended to expose the systems to the maximum temperature which they could withstand without

TABLE 23 A DESCRIPTION OF PROCESSING PARAMETERS FOR 12" SQUARE LAMINATES USED FOR THE POST CURE STUDIES

Molding System	Normal Resin Content (% BYWT)	Molding Parameters		Cure Cycles			Comments
		Contact Time (min)	Preform	Temp.[1] (°F)	Time	Pressure (psi)	
X270 Epoxy	30	2	Yes	350	2 hr	1000	
X271 Epoxy	30	0 to 2 [2]	"	350	2 hr	800	Cooled to 300°F under Pressure
X273 Phenolic	30	none	"	250° 300 400	1 hr 1 hr 1 hr	1000	Cooled under Pressure to Room Temp.
X274 Silicone	30	10	"	500	12 hr	1000	Cooled under Pressure to Room Temp.

1) Mold was heated to temperature of the first step of the cure before charging.

2) Contact time based on gel time at 350°F for base resin.

delamination. Different post cures were naturally required for each system, as the highest temperature at which blowing or delamination had not occurred varied for each. Details of the various cycles used are presented in Table 24. Laminates which were not obviously delaminated during the molding cycle were tested at room temperature in the same manner as the original halves of these laminates. A comparison between these two sets of data and a satisfactory post cure cycle was selected. This comparison appears in Tables 25 through 28.

Two 12" x 12" x 1/8" laminates were prepared, in the manner described in Table 38, from each of the four composites under investigation. One laminate from each of the composites was cut into fourteen 4" x 1/2" x 1/8" flexural test specimens. The other laminate from each composite was cut into sixteen tensile specimens, with the same configuration as those used for post cure study. Before these laminates were cut for testing, each was post cured in accordance with the appropriate cycles shown in Tables 25 through 28.

TABLE 24 THE EFFECT OF POST CURING ON THE APPEARENCE OF GLASS FLAKE REINFORCED COMPRESSION MOLDED COMPOSITES

Molding Composition	After Post Cure No. 1	After Post Cure No. 2	After Post Cure No. 3
X270 Epoxy	Delaminated during 400°F step	No change	No change
X271 Epoxy	Delaminated after 12-15 hrs at 500°F	Delaminated during 400°F step	No change
X273 Phenolic	Delaminated at 350°F	No change	No change
X274 Silicone	No change	No change	X274 system was not exposed as P.C. No. 1 was satisfactory

Post Cure No. 1

24 hrs ea at 200°F, 250°F, 350°F, 400°F, 450°F and 500°F for all systems.

Post Cure No. 2

a. For X270 system
   2 hrs ea at 200°, 300° and 350°F
b. For X271 system
   2 hrs ea at 200°, 300°and 400°
c. For X273 system
   2 hrs ea at 200° and 250°F
d. For X274 system
   24 hrs at 200°F; 2 hrs ea at 250°, 300°, 350°, 400° and 450°F; then 24 hrs at 500°F

Post Cure No. 3

a. For X270 system
   Same as post cure No. 1 except post cure was stopped after 350°F step
b. For X271 system
   Same as post cure No. 1 except post cure was stopped after 400°F step
c. For X273 system
   Same as post cure No. 1 except post cure was stopped after 250°F step.

TABLE 25

THE EFFECT OF POST CURING ON THE ROOM TEMPERATURE MECHANICAL PROPERTIES OF FLAKE REINFORCED X 270 MOLDINGS

Molding Compound	Post Cure Cycle	Laminate Number	Property Measured	Before Postcure	After Postcure	% Change in Strength
X 270 Epoxy	2	F-1	Tensile	16,900	22,700	+ 34.4 %
			Compression	53,300	45,400	- 14.8 %
			Compressive Modulus	5.09	5.22	+ 2.6 %
			Flexure	36,600	35,900	- 1.9 %
			Flexural Modulus	5.04	5.23	+ 3.4 %
	3	F-2 (1)	Tensile	22,000	23,300	+ 5.9 %
			Compression	52,200	50,300	- 3.6 %
			Compressive Modulus	5.18	5.14	- 0.8 %
			Flexure	35,900	36,200	+ 0.8 %
			Flexural Modulus	5.15	4.85	- 5.8 %

Post Cures

No. 1	No. 2
2 hrs at 200°F	24 hrs at 200°F
2 " " 300°F	24 " " 250°F
2 " " 350°F	24 " " 300°F
	24 " " 350°F

Notes:

(1) This post cure was selected to condition X 270 laminates prior to tests at elevated temperature.

TABLE 26

THE EFFECT OF POST CURING ON THE ROOM TEMPERATURE MECHANICAL PROPERTIES OF FLAKE REINFORCED X 271 MOLDINGS

Molding Compound	Post Cure Cycle	Laminate Number	Property Measured	Before Postcure	After Postcure	% Change in Strength
X 271 Epoxy	2	G-1	Tensile	22,600	Delaminated	0
			Compression	44,200	"	0
			Compressive Modulus	4.92	"	0
			Flexure	33,900	"	0
			Flexural Modulus	4.81	"	0
	3	G-2 (1)	Tensile	22,000	22,200	+ 0.9
			Compression	48,400	36,000	-25.6
			Compressive Modulus	4.84	4.71	- 2.6
			Flexure	33,400	31,400	- 6.0
			Flexural Modulus	5.62	4.71	-16.2

Post Cures

No. 2	No. 3
2 hrs at 200°F	24 hrs at 200°F
" " 300°F	" " 250°F
" " 400°F	" " 300°F
	" " 400°F

Notes:

(1) This post cure was selected to condition X 271 laminates prior to tests at elevated temperature.

TABLE 27

THE EFFECT OF POST CURING ON THE ROOM TEMPERATURE MECHANICAL PROPERTIES OF FLAKE REINFORCED X 273 MOLDINGS

Molding Compound	Post Cure Cycle	Laminate Number	Property Measured	Before Postcure	After Postcure	% Change in Strength
X 273 Phenolic	2	H-1 (1)	Tensile	10,000	Delaminated	0.0
			Compression	35,500	"	"
			Compressive Modulus	4.64	"	"
			Flexure	21,600	17,900	- 17.1 %
			Flexural Modulus	4.86	3.73	- 23.2 %
	3	H-2 (1)	Tensile	9,100	Delaminated	0
			Compression	22,500	19,400	- 13.8 %
			Compressive Modulus	5.03	4.12	- 0.6 %
			Flexure	17,100	20,000	+ 17.0 %
			Flexural Modulus	3.89	4.23	+ 8.7 %

Post Cures

No. 2	No. 3
2 hrs at 200°F	24 hrs at 200°F
2 " " 250°F	24 " " 250°F

Notes:

(1) Neither post cure was selected to condition X 273 laminates prior to tests at elevated temperature. These laminates did not receive a post cure.

TABLE 28

THE EFFECT OF POST CURING ON THE ROOM TEMPERATURE MECHANICAL PROPERTIES OF FLAKE REINFORCED X 274 MOLDINGS

Molding Compound	Post Cure Cycle	Laminate Number	Property Measured	Before Postcure	After Postcure	% Change in Strength
X 274 Silicone	1	J-1 (1)	Tensile	8,000	9,200	+ 15
			Compression	16,200	10,200	- 37
			Compressive Modulus	4.10	3.47	- 8.5
			Flexure	17,700	16,200	- 8.5
			Flexural Modulus	3.67	3.82	+ 4.1
	2	J-2	Tensile	10,800	8,600	--
			Compression	Delaminated	13,600	--
			Compressive Modulus	"	3.61	--
			Flexure	11,700	16,000	--
			Flexural Modulus	2.84	3.53	--

Post Cures

No. 1	No. 2
24 hrs at 200°F	24 hrs at 200°F
" " 250°F	2 " " 250
" " 300	2 " " 300
" " 350	2 " " 350
" " 400	2 " " 400
" " 450	2 " " 450
" " 500	24 " " 500

Notes:

(1) This post cure was selected to condition X 274 laminates prior to test at elevated temperatures.

TABLE 29 A DESCRIPTION OF LAMINATES USED FOR TESTS AT ELEVATED TEMPERATURE

Molding Composition	Laminate Number	Resin Content	Sp. Gravity	Molding Conditions		
				Temp	Pressure	Time
X 270 Epoxy	F-27	30.6%	2.00	350°F	contact 1000 psi	1.5 min 120 min
	F-28	30.3%	1.99	350°F 350°F	contact 1000 psi	1.5 min 120 min
X 271 Epoxy	G-27	30.5%	1.96	350°F	800 psi	120 min
	G-28	29.4%	1.95	350°F	800 psi	120 min
X 273 Phenolic	H-27	30.4%	1.96	250°F 300°F 400°F	1000 psi 1000 psi 1000 psi	60 min 60 min 60 min
	H-28	30.6%	1.97	250°F 300°F 400°F	1000 psi 1000 psi 1000 psi	60 min 60 min 60 min
X 274 Silicone	J-27	30% *	N.D.	350°F 350°F 500°F	contact 1000 psi 1000 psi	60 min 60 min 8 hrs
	J-28	30% *	N.D.	350°F 350°F 500°F	contact 1000 psi 1000 psi	60 min 60 min 8 hrs

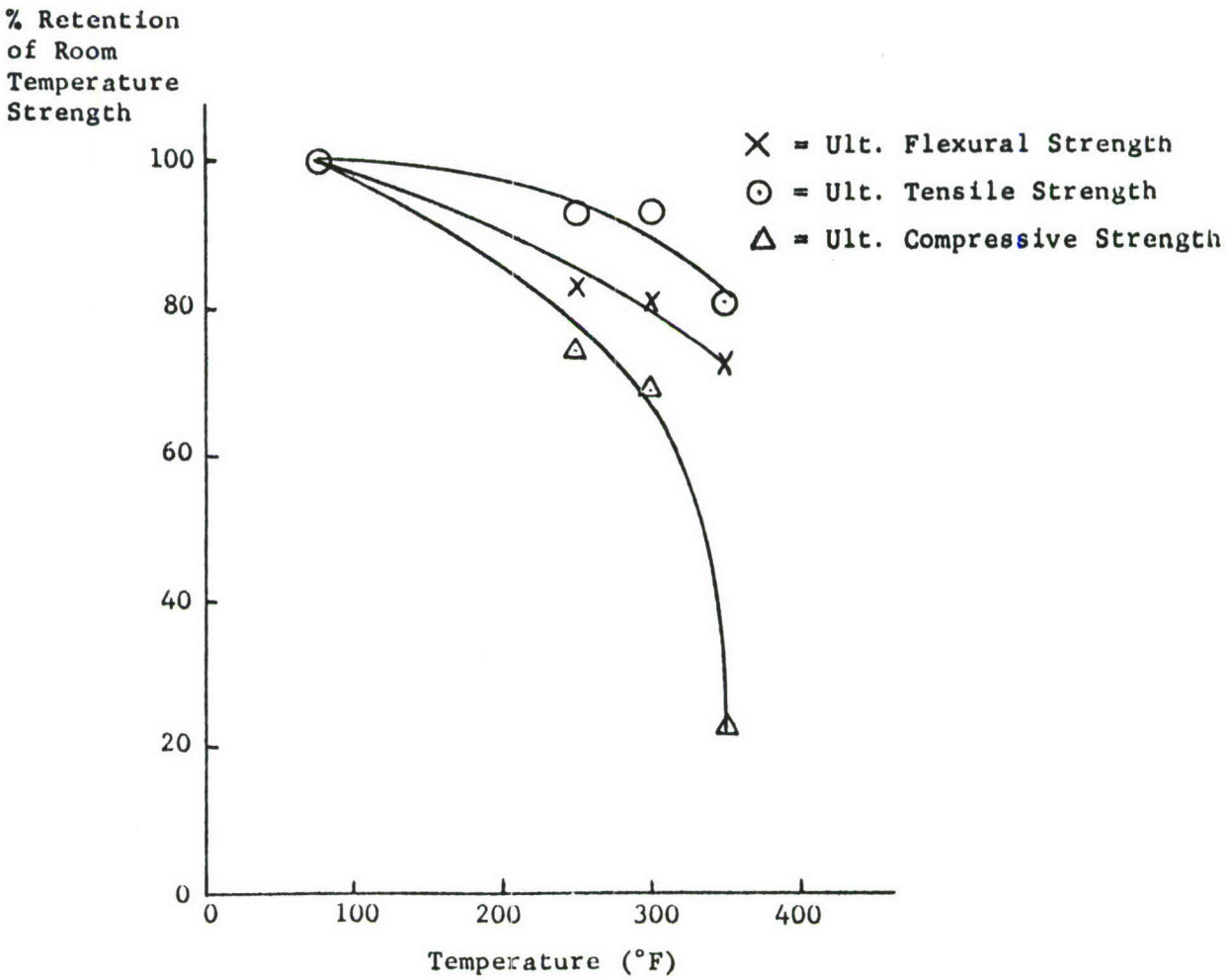
* Nominal composition of the flake-resin mixture.

3.0 TEST PROCEDURES

Mechanical properties were determined in accordance with the procedure described in section 2 of this report, except that the specimens were enclosed in an air circulating oven which was maintained at the desired temperature. The specimens were exposed to these temperatures for ten minutes before they were tested. A minimum of two specimens from each composite was tested at each temperature.

4.0 TEST RESULTS

The results of the tests at elevated temperature appear in Figures 45 through 48.



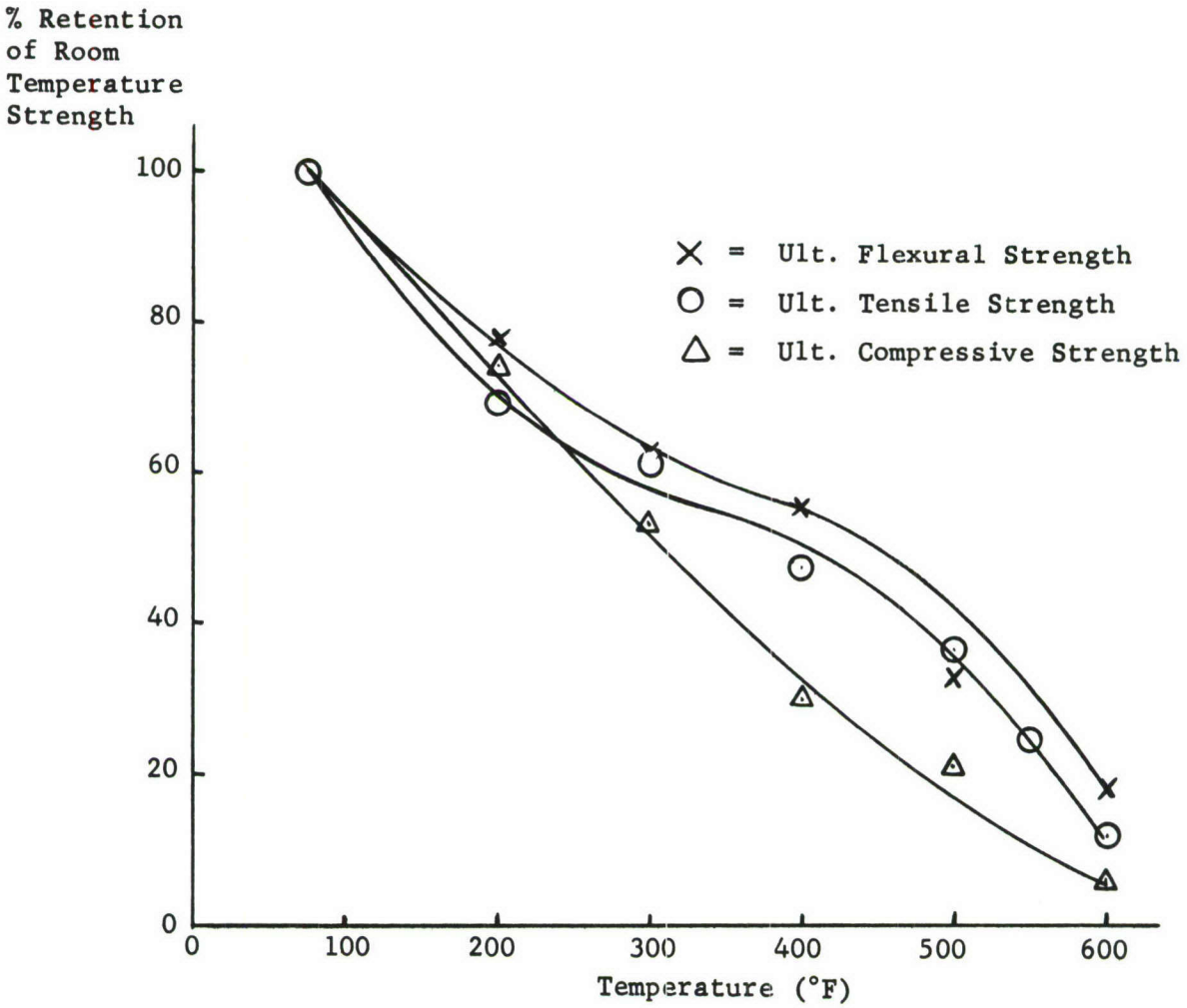
NOTES:

1. Specimens were tested at the indicated temperatures after a ten minute soak at these temperatures

2. Room temperature mechanical properties were as follows:

Ult. Flexural Strength	=	36,100 psi
Ult. Tensile "	=	22,900 "
Ult. Compressive "	=	54,000 "

Figure 45 - The Effect Of Elevated Temperature Exposure On The Mechanical Properties Of X270 Molding Material

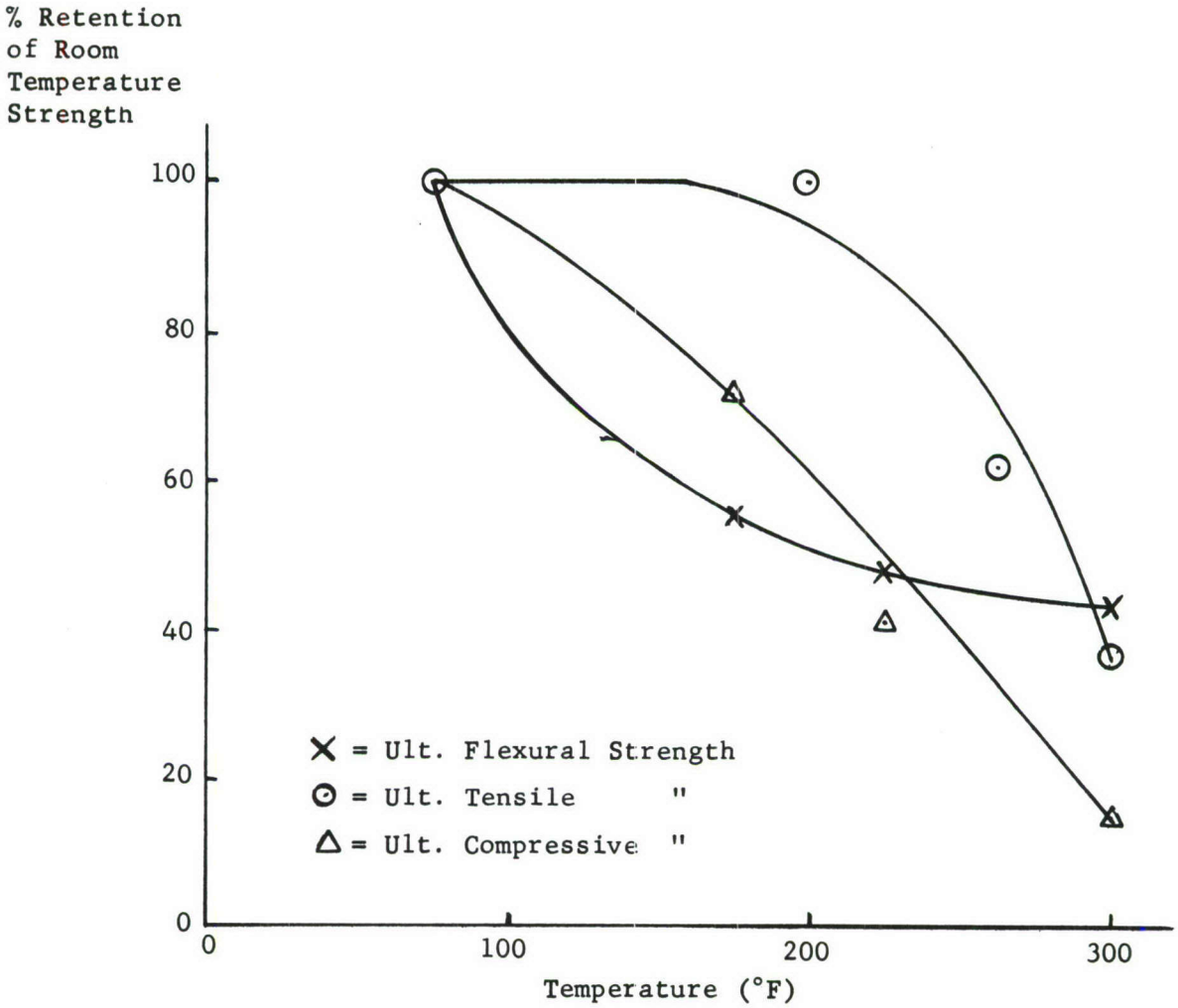


NOTES:

1. Specimens were tested at the indicated temperatures after a ten minute soak at these temperatures
2. Room temperature mechanical properties were as follows:

Ult.	Flexural Strength	=	38,700 psi
"	Tensile "	=	18,800 "
"	Compressive "	=	33,700 "

Figure 46 - The Effect Of Elevated Temperature Exposure On The Mechanical Properties Of X271 Molded Material



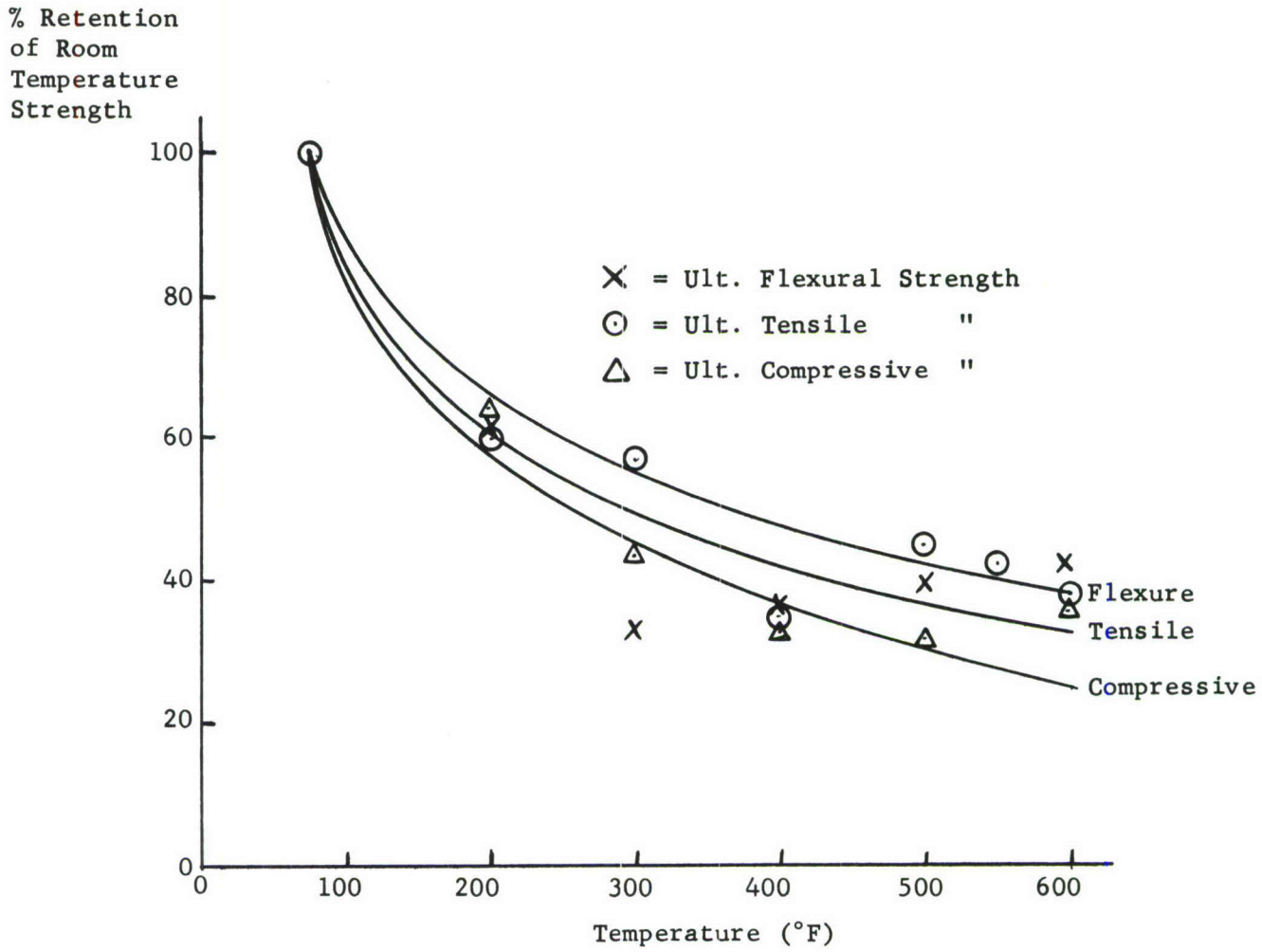
NOTES:

1. Specimens were tested at the indicated temperature after a ten minute soak at these temperatures

2. Room temperature mechanical properties were as follows:

Ult.	Flexural Strength	=	24,900 psi
"	Tensile "	=	12,700 "
"	Compressive "	=	31,400 "

Figure 47 - The Effect Of Elevated Temperature Exposure On The Mechanical Properties Of X273 Molding Material



NOTES:

1. Specimens were tested at the indicated temperatures after a ten minute soak at these temperatures

2. Room temperature mechanical properties were as follows:

Ult.	Flexural Strength	=	17,500 psi
"	Tensile "	=	9,700 "
"	Compressive "	=	15,000 "

Figure 48 - The Effect Of Elevated Temperature Exposure On The Mechanical Properties Of X274 Molding Material

5.0 DISCUSSION OF THE TEST RESULTS

With the exception of the X 274 silicone system, it appears that definite limitations on the maximum usable temperature of the flake composites exist. These limits are relatively independent of whether or not these systems are exposed to a post cure before being exposed to these temperatures. The results, in general, are quite surprising in that both epoxy systems tended to delaminate or blow at 500°F or below, even though both systems are based on resins with excellent elevated temperature resistance and should remain usable at 500°F for moderately long times. In addition, while both epoxy resins are normally not considered to evolve volatile materials during the curing cycle, and while the silicone resin in the X 274 system is known to liberate water as a by-product of the polymerization reaction, the blowing or delamination occurred in the epoxys and not in the silicone. Normally, one would expect this silicone system to behave in the same manner as did the other condensation polymer system, the phenolic or X 272. However, the results of this test series are directly the opposite of the results which would be predicted from an examination of the resin systems in the composites.

SECTION V THE MOLDING OF VARIOUS GEOMETRIC SHAPES

1.0 SCOPE

Several geometric shapes were selected to compare the properties of these moldings to the properties associated with flat laminates. The results of tests performed on segments cut from some of these moldings, together with our observations on the molding processes and the moldings, are presented in this section of the report.

2.0 THE EFFECT OF LAMINATE THICKNESS ON THE MECHANICAL PROPERTIES OF GLASS FLAKE REINFORCED COMPRESSION MOLDED COMPOSITES

2.1 Scope

This section of the report deals with the results of an investigation of the effect of laminate thicknesses ranging from 1/8" to 3/4" on the room temperature tensile, compressive and flexural strengths of these laminates. Each of the 4 resin systems selected for this program were studied.

2.2 Preparation of Test Specimens

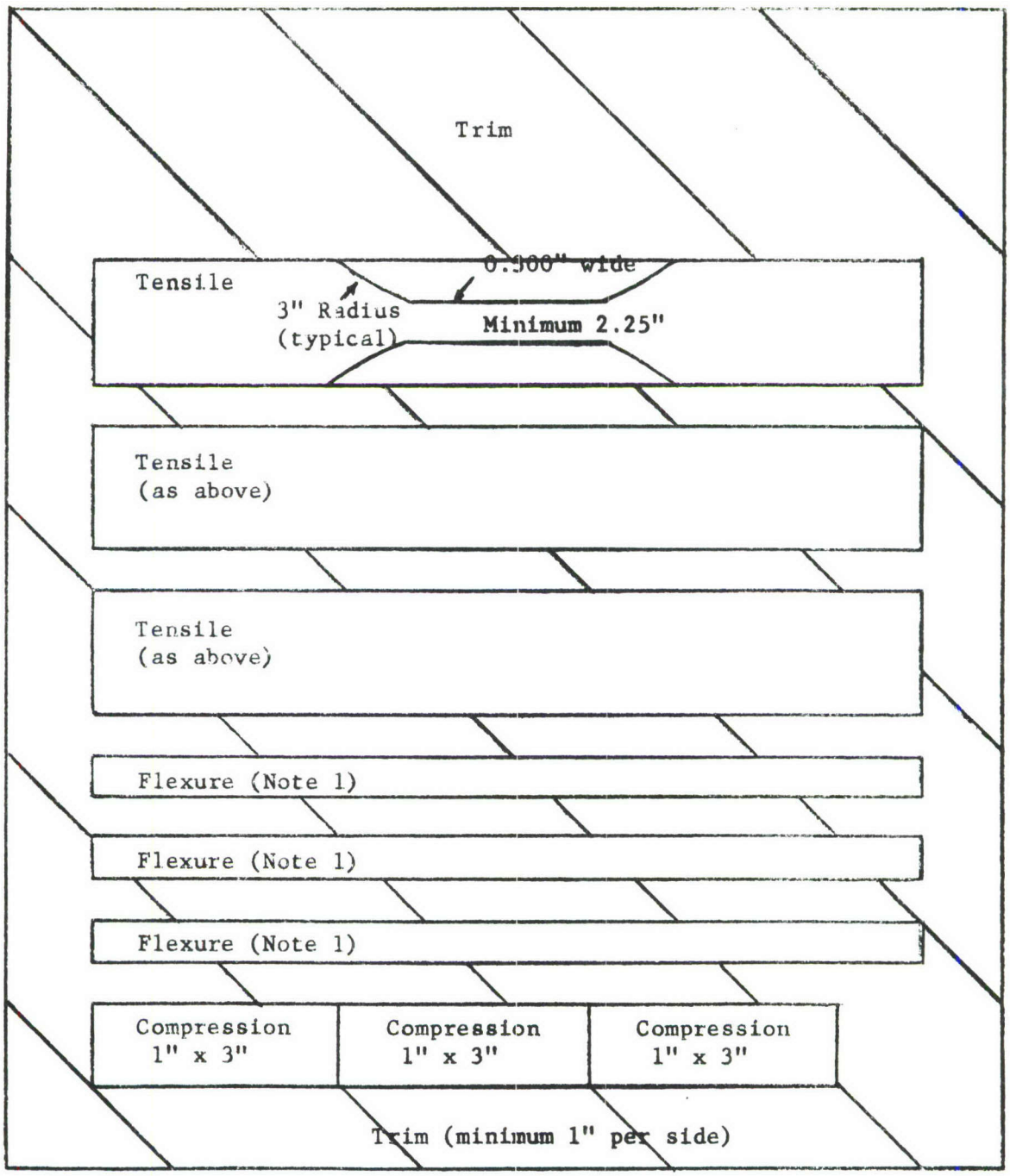
The same molding techniques which were employed to mold the 1/8" thick laminates used in the post cure study described in the preceding section of this report (Table 23), were used to mold the laminates for this test series. After molding, each laminate was cut into test specimens in accordance with Figure 49.

Flexural and tensile tests were performed in accordance with the procedures specified in Federal Specification L-P-406b, except, that because of the limited length of the flexural specimens, in the case of the 3/4" thick laminates, it was impossible to maintain the span to depth of 16 : 1 to 18 : 1 specified in this procedure. Instead, an 8" span (or span to depth ratio of approximately 10 or 12 to 1) was used.

The same compression tests which were described in Section III of this report, were used for specimens up to 3/8" thick. However, as this was the largest specimen size that the existing compression testing fixture would accommodate, a simple unsupported compression test of a 1" x 3" specimen was employed for the thicker samples. Modulus values in compression were not determined for specimens over 3/8" thick.

2.3 Test Results

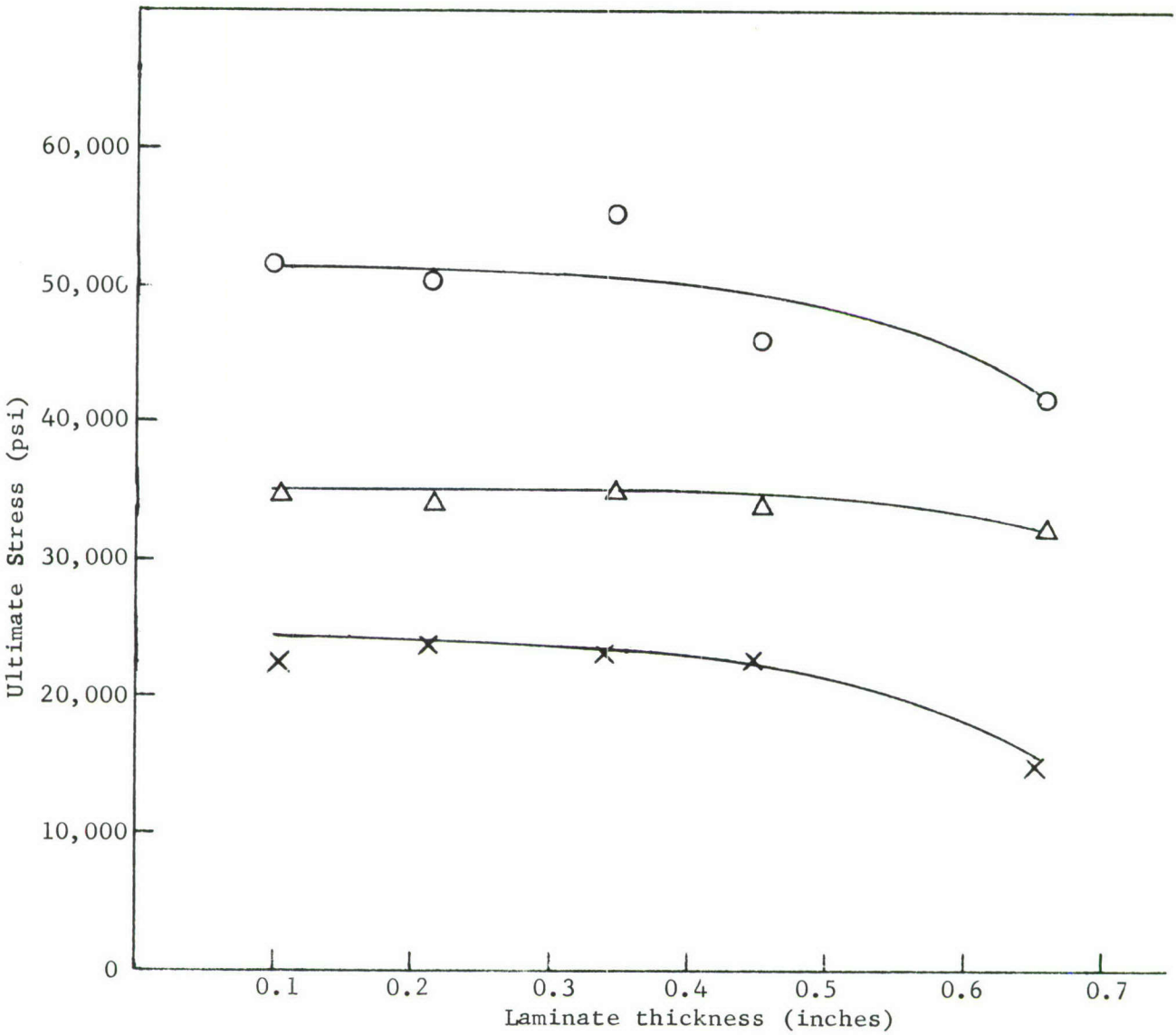
The results of these tests are presented in Figures 50 through 52 and in Table 30.



NOTE 1

Flexural specimens varied in length to conform as nearly as **possible to the 16:1 to 18:1 span to depth ratio specified in Federal Specification L-P-406b.**

Figure 49 - A Diagram Of Specimens Used For Thickness Study

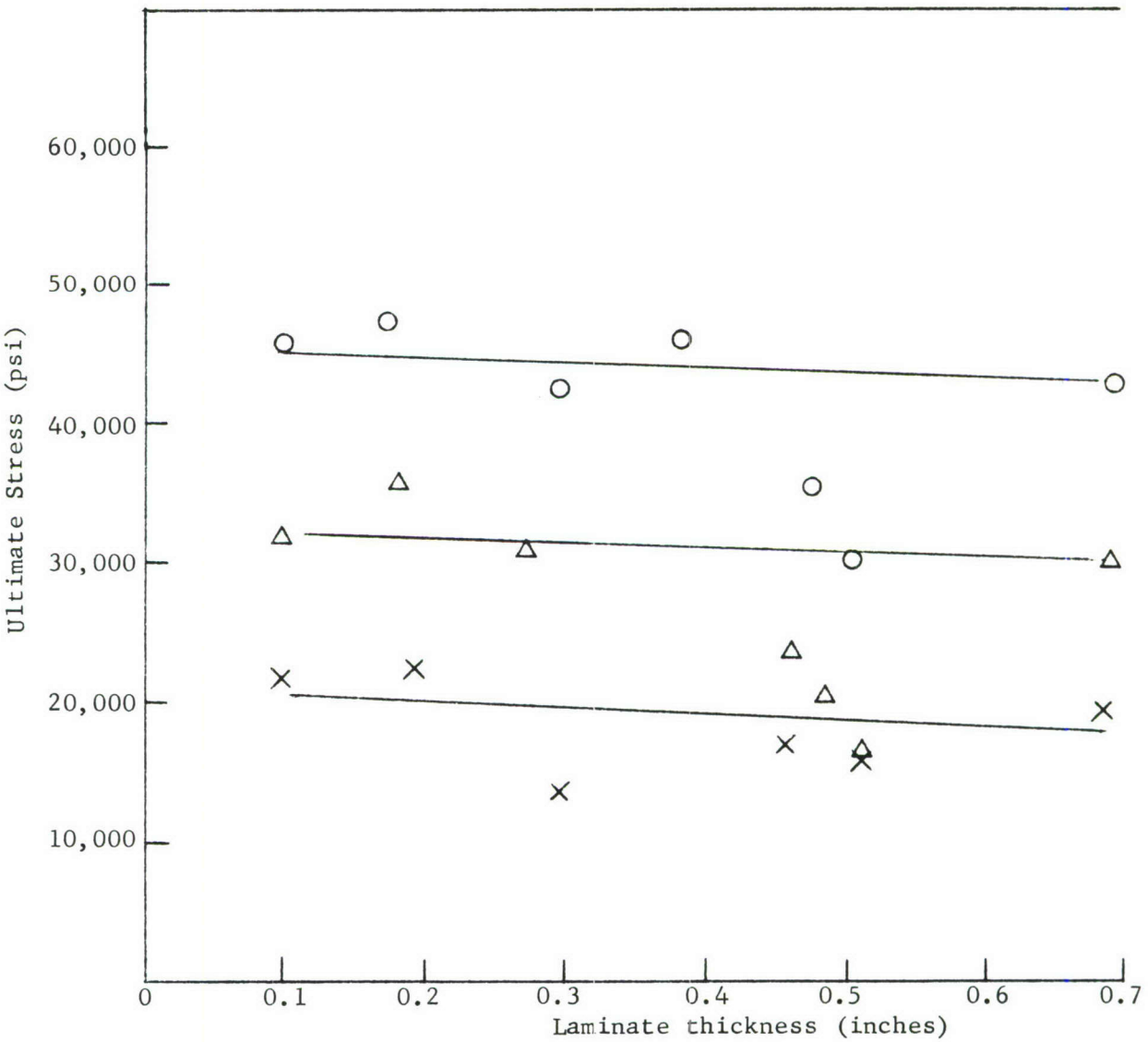


○ - Ultimate Compressive Strength

× - Ultimate Tensile Strength

△ - Ultimate Flexural Strength

Figure 50 - The Effect Of Laminate Thickness On The Mechanical Properties of X270 Moldings



LEGEND

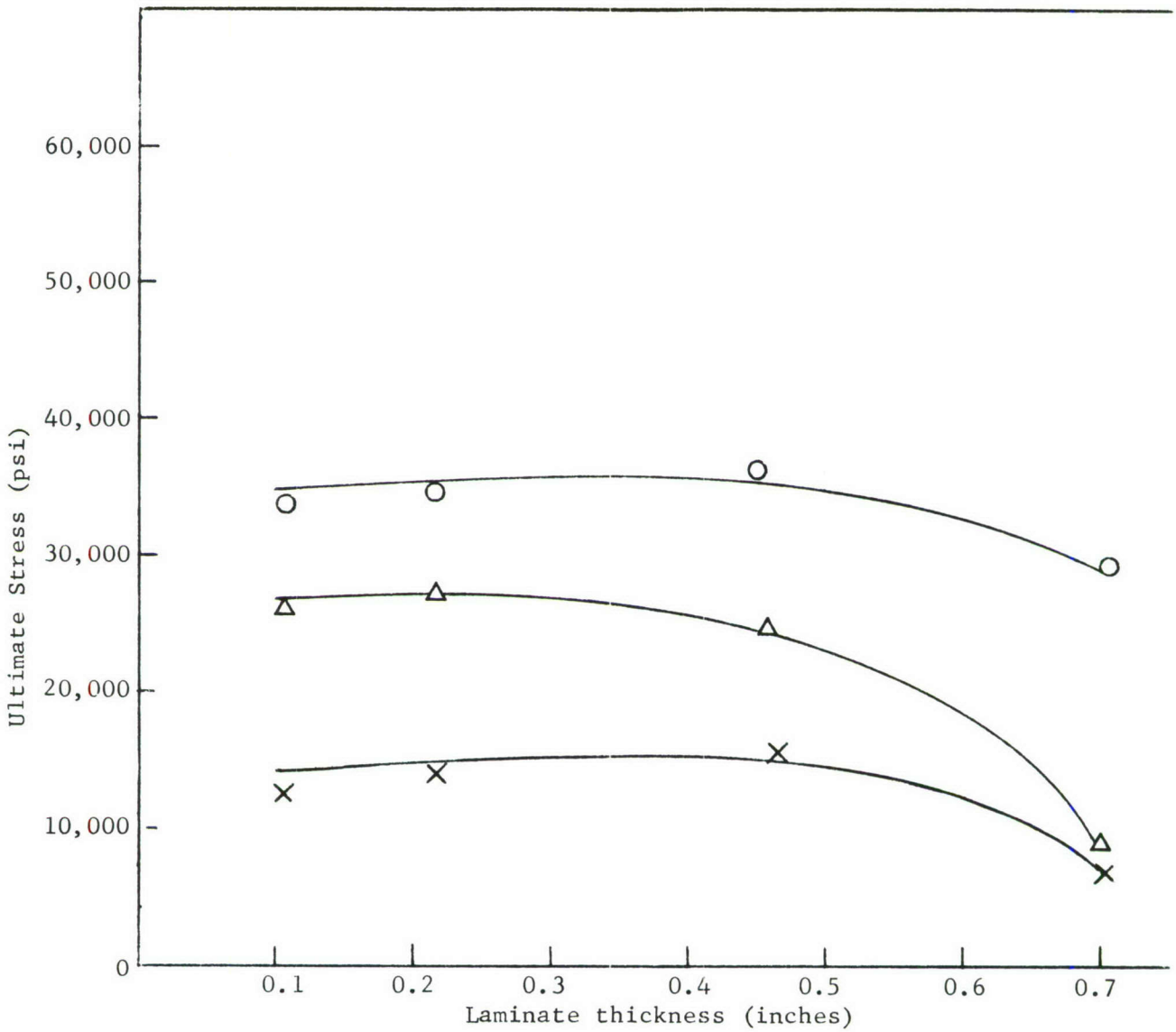
○ - Ultimate Compressive Strength

△ - Ultimate Flexural Strength

× - Ultimate Tensile Strength

NOTE: An obvious flaw was noted in the laminate used for the 0.500" thickness data. All specimens failed at this flaw.

Figure 51 - The Effect Of Laminate Thickness On The Mechanical Properties of X271 Moldings



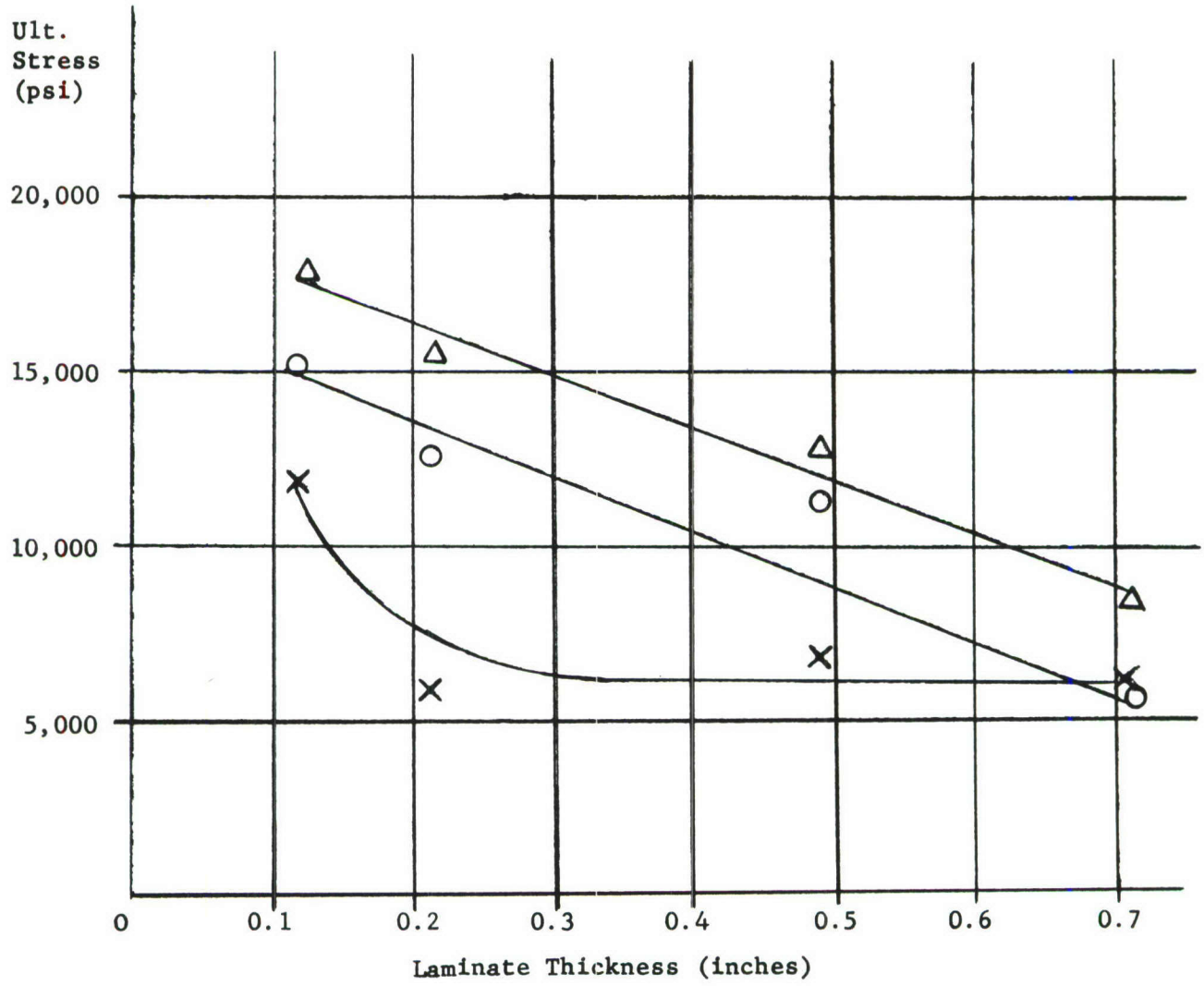
LEGEND

○ - Ultimate Compressive Strength

△ - Ultimate Flexural Strength

× - Ultimate Tensile Strength

Figure 52 - The Effect of Laminate Thickness On The Mechanical Properties of X273 Moldings



Legend

△ = ult. flex. strength

○ = " comp. "

× = " tensile "

Figure 53 - The Effect of Laminate Thickness on The Mechanical Properties of X274 Moldings

TABLE 30 MECHANICAL PROPERTIES OF LAMINATES USED TO DETERMINE THE EFFECT OF LAMINATE THICKNESS

Molding System	Laminate No.	Average Thickness (inches)	Compression (psi)				Tension (psi)				Flexure (psi)			
			Ultimate Stress	Std. Dev. of ult. stress (EST)	Modulus	Std. Dev. of Modulus (EST)	Ult. Stress	Std. Dev. of Ult. Stress (EST)	Modulus	Std. Dev. of Modulus (EST)	Ultimate Stress	Std. Dev. of Ult. Stress (EST)	Modulus	Std. Dev. of Modulus (EST)
X270 Epoxy	F-3	0.100	51000	3100	5.3	.2	22500	2800	5.6	.1	34700	1600	4.8	.3
	F-4	0.212	50100	3500	5.1	.3	23800	2200	4.4	.4	34000	2600	4.9	.5
	F-5	0.452	46000	5600	-	-	22500	120	4.6	.5	33100	1200	5.4	.1
	F-5A	0.344	55300	1400	4.9	.06	23100	1800	4.6	.3	35000	1300	5.0	.2
	F-6	0.657	41700	900	-	-	15100	3700	5.0	.5	32100	3700	4.9	.4
X271 Epoxy	G-3	0.101	45800	1400	4.9	.1	21600	3600	5.4	1.0	31100	2600	4.6	.3
	G-4	0.270	42500	7000	4.6	.6	13200	5000	4.6	.3	31000	5000	5.1	.8
	G-5	0.513	30100	11000	-	-	15500	5200	4.7	.8	15700	4300	4.5	.7
	G-6	0.691	42800	2900	-	-	19200	300	5.0	1.0	30400	120	4.8	.1
X273 Phenolic	H-3	0.106	34000	3900	5.0	.2	12600	2600	5.2	.2	26300	3200	4.7	.4
	H-4	0.216	34800	3200	4.7	.4	14200	2900	4.9	1.5	22500	4500	4.9	.7
	H-5	0.456	36600	1800	-	-	15700	1100	4.6	.2	25100	900	5.2	.1
	H-6	0.702	29500	2400	-	-	7200	2300	4.7	.5	9300	3000	4.7	.1

2.4 Discussion Of The Test Results

Both the X270 epoxy and the X273 phenolic systems showed results which would indicate that there is little or no change in mechanical properties for laminates up to 1/2" thick, and that there is a definite reduction in properties if thicker laminates are molded. As this reduction was much more pronounced with the phenolic system, and as experience has shown that the phenolic is a more difficult system to mold than the epoxy, the possibility should be considered that these tests have not indicated an inherent property of the molded composites, but rather have actually been an indication of unsatisfactory molding procedures for the thicker specimens.

In the case of the X274 silicone, there appears to be a definite correlation between the thickness of a molded composite and the strength of that composite.

3.0 MOLDING AND TESTING OF THE CYLINDRICAL SHAPE

3.1 Scope

A 10 inch section of one-half of a right circular cylinder, with a diameter of approximately 9 inches and a wall thickness of 0.100 inches, was selected as an example of a simple curvature.

The cylindrical moldings were evaluated by tests in tension, flexure, compression, bending, and by visual examination. The tests in tension, flexure, and compression were performed on specimens cut with their longer axis parallel to the axis of the cylinder, so that the effect of the curvature of the specimens was minimized. The bending tests were performed on specimens cut with their longer axis perpendicular to the axis of the cylinder.

3.2 Molding Procedures

To facilitate the fabrication of a mold for this shape, closed ends with a draft angle of 1°30' and a wall thickness of 0.100 inches were planned on the cylindrical section, even though plans were not made for testing these end pieces. (Figures 54 and 55 show the cylindrical mold).

Our initial attempts to mold flake composites in this mold were unsuccessful because of these ends. The amount of flow of the flake-resin mixture could not adequately be controlled during the molding process. If molding pressure and contact time were adjusted so that there was sufficient flow of the mold compound to fill the end portions of the mold, then there was an excessive amount of flow in the curved section of the mold. As a result, extremely poor alignment of the flake, as characterized by wrinkles, occurred in the moldings. It was noted that these wrinkles appeared with their long axis aligned perpendicularly to the direction in which the flow had occurred. Figure 56 shows a typical composite which was molded in the cylindrical mold. This photograph shows the wrinkles which are associated with the poor flake alignment.

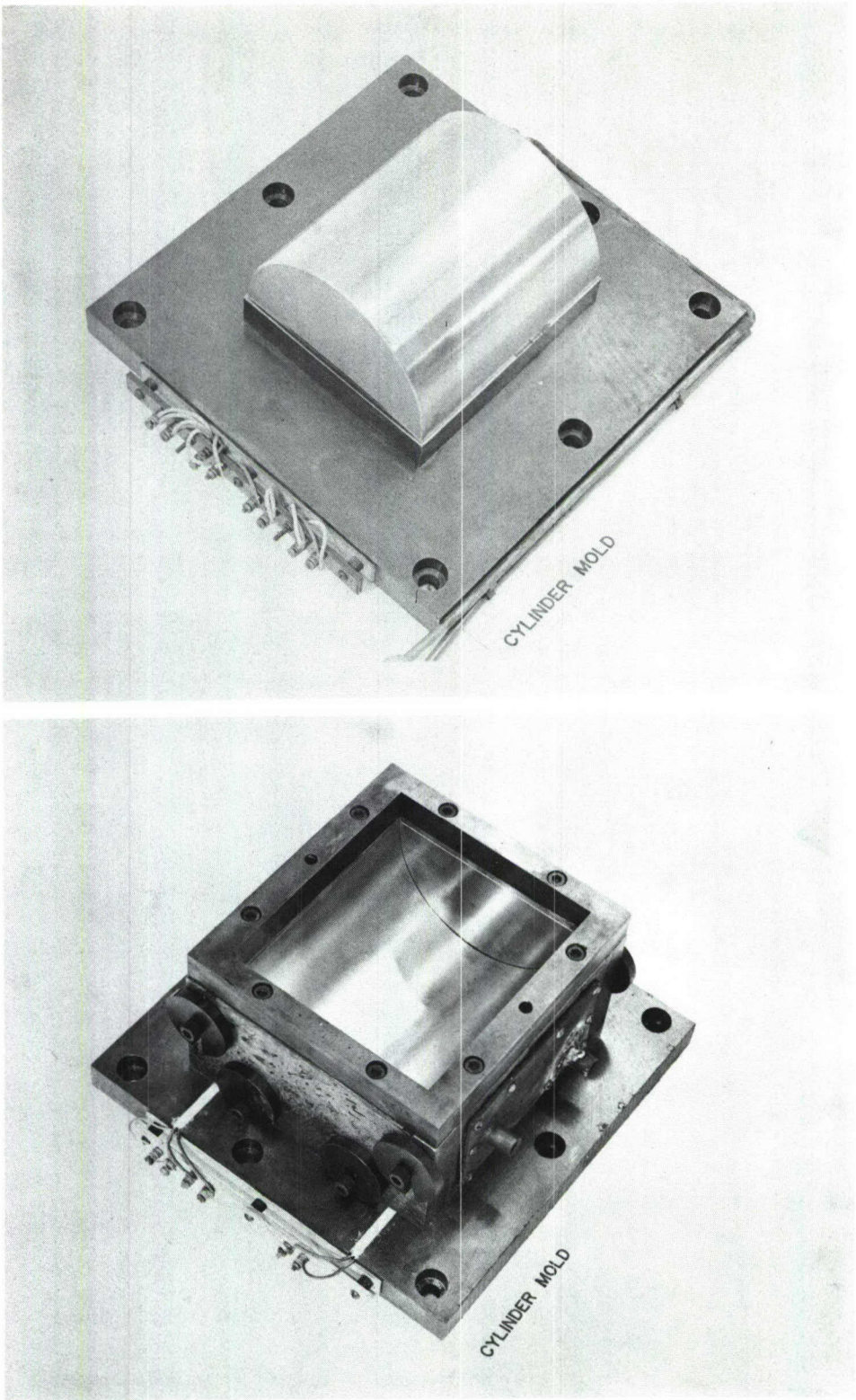


Figure 54 - The RE5204 Mold Which Was Used To Mold The Cylindrical Sections

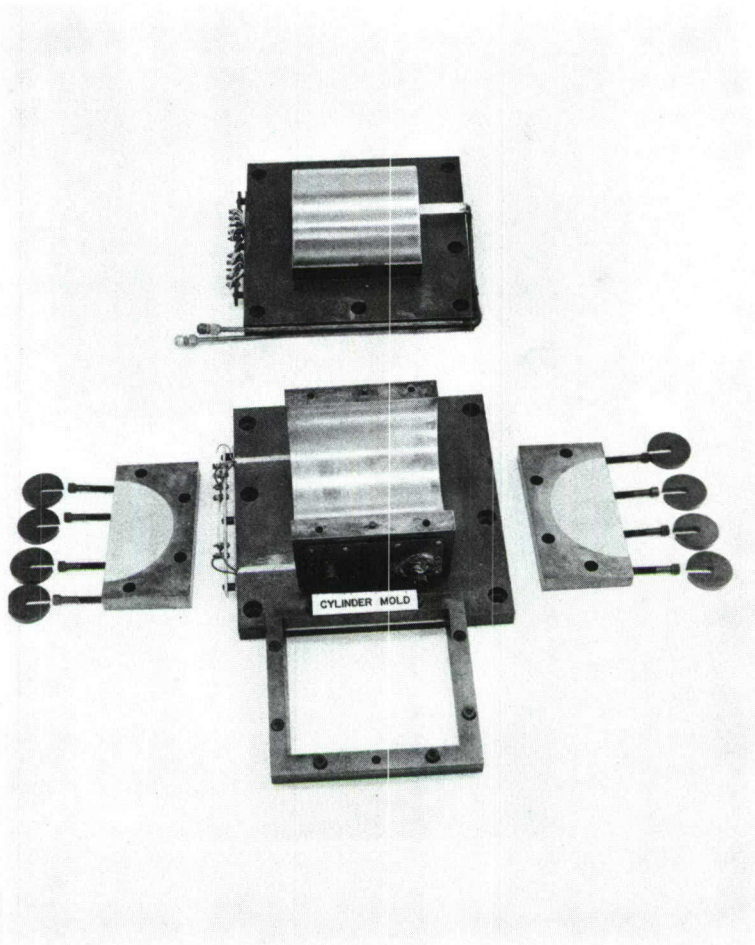


Figure 55 - The RE5204 Cylindrical Section Mold Disassembled to Show Its Component Parts.

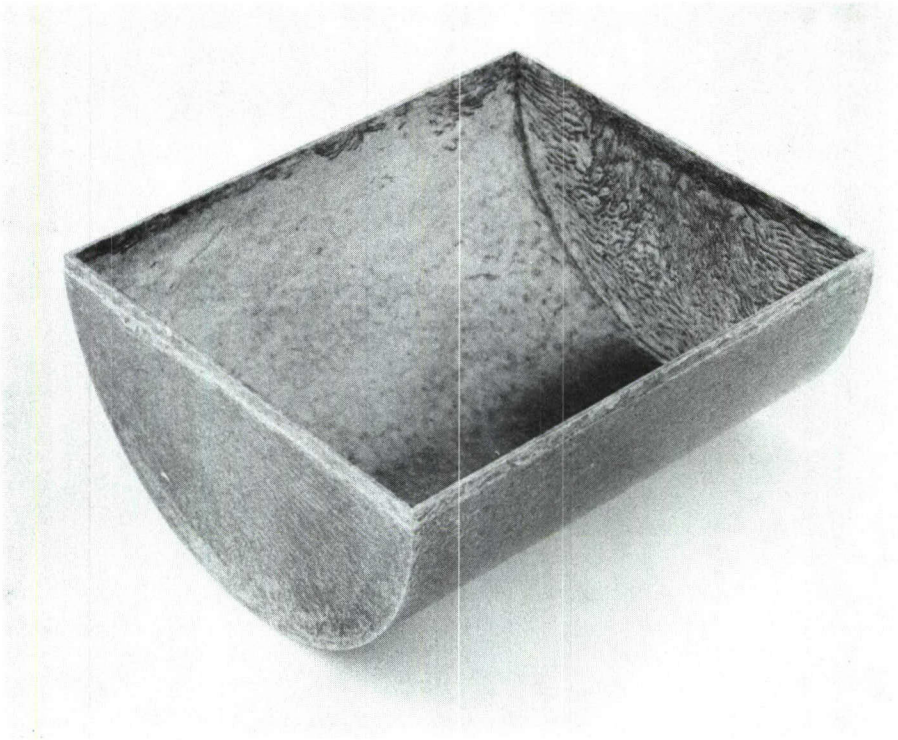


Figure 56 - An X270 Epoxy Cylinder Section Showing Wrinkle End Closures

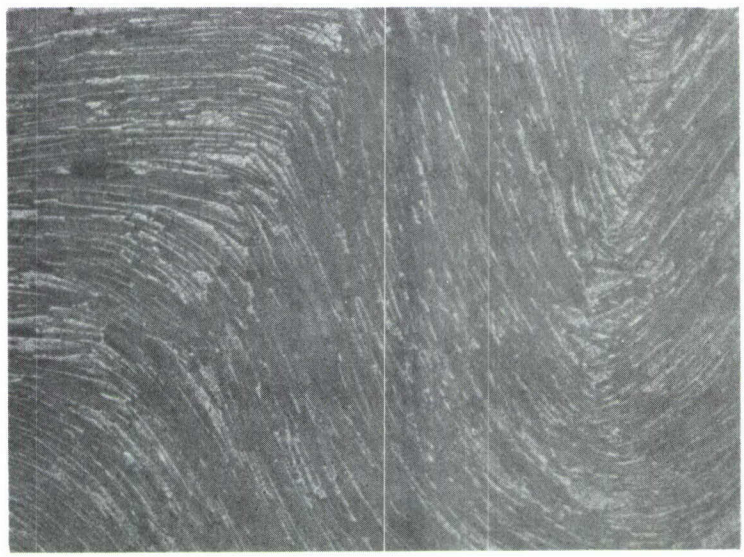


Figure 57 - A Photomicrograph Showing Alignment of Flakes Within A Wrinkled Area Of An X270 Epoxy Molding

A photomicrograph was taken of a cross section cut from a similar wrinkle in a flat laminate and appears in Figure 57. This micrograph shows the extent of disruption which took place in the alignment of the flakes when wrinkling occurred. The assumption was made at that time that such wrinkles would obviously have a deleterious effect on the mechanical properties of the molding. However, the extremely poor quality of the end portions of these moldings made measurements of these properties impractical. Consequently, these measurements were deferred until segments cut from the walls of the box molding could be tested. These walls were subjected to the same conditions as the ends of the cylinder, and were more readily tested.

In view of the unsatisfactory results that were obtained when the cylindrical section with ends was molded, it was decided that this mold would be modified to omit the ends. This modification was made by silver brazing 0.90 inches thick flat steel plate to the ends of the mold cavity. The modification changed the direction of flow within the molding by prohibiting flow into the ends and requiring that the material move along the curved portion of the mold. As demonstrated in Figure 58, this modification did not prevent wrinkles, but instead, shifted the positions of these wrinkles. It was again noted that the wrinkles tended to align with their long axis perpendicular to the direction of flow.

In view of these results, it was decided that any further attempt to modify the mold to correct this problem would be futile and the mold was used in this configuration to mold the balance of the samples.

Cure cycles and processing parameters were the same as those used to fabricate the samples for the control tests. These conditions are described in Table 14 of this report. To reduce flow and correct the wrinkled condition, an attempt was made to charge the mold with preformed biscuits, as those used for the flat laminates. This technique was not useable, as the molding compounds still had a bulk factor of 3 or 4 to 1 after preforming. This bulk would not allow the mold to close without damage to the preforms and satisfactory laminates could not be obtained.

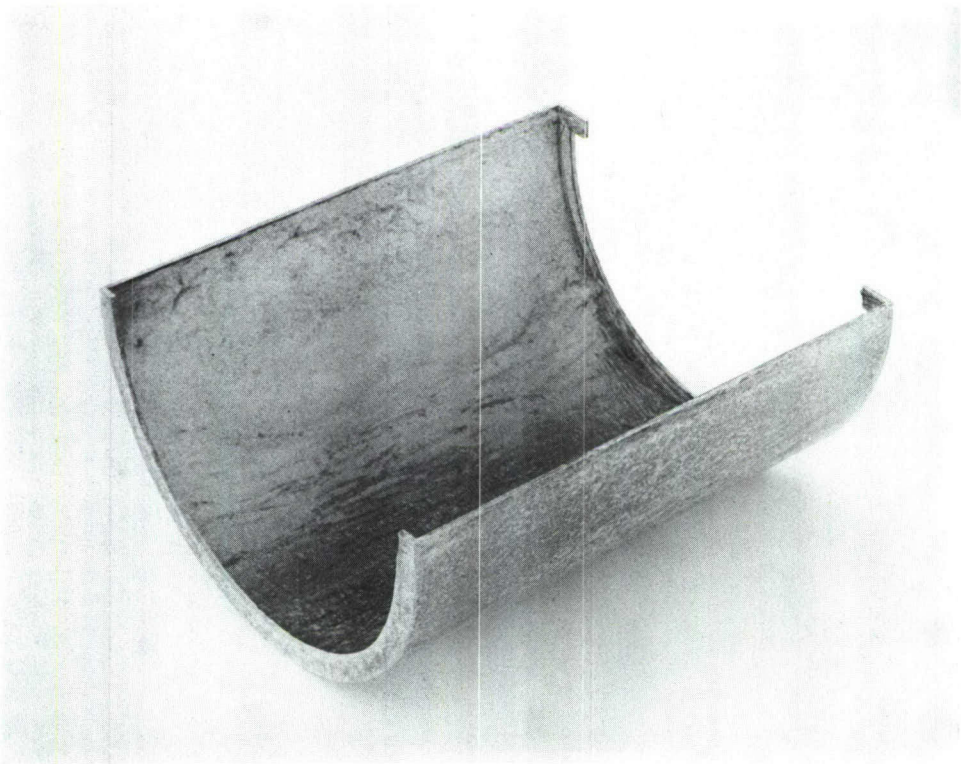


Figure 58 - An X270 Cylindrical Section Without Ends
Showing Wrinkled Areas

3.3 Testing The Cylindrical Moldings

3.3.1 Tension, Flexural and Compressive Tests

a. Scope: The methods for testing sections of the cylinder were in accordance with the tensile, flexural and compressive tests described in Section III of this report. All of these tests specimens were cut with their long axis parallel to the axis of the cylinder and are, henceforth, noted as being cut longitudinally.

b. Test Specimens: A drawing of the layout used to cut specimens from the cylinder is shown in Figure 59. Ths specimens were cut and finished to meet the requirements of their respective tests, as described in Section III of this report and as shown in (A), (B), and (C) of Figure 60.

c. Apparatus: The jigs and fixtures, used to test the tensile, flexural, and compressive specimens of the flat laminates, were modified to fit the curvature of the cylindrical specimens.

d. Procedure: Techniques as described in the approporiate section of Federal Specification, L-P-406b, with the exception noted above, were employed.

e. Test Result: The results of tests on the simply curved specimens appear in Table 31.

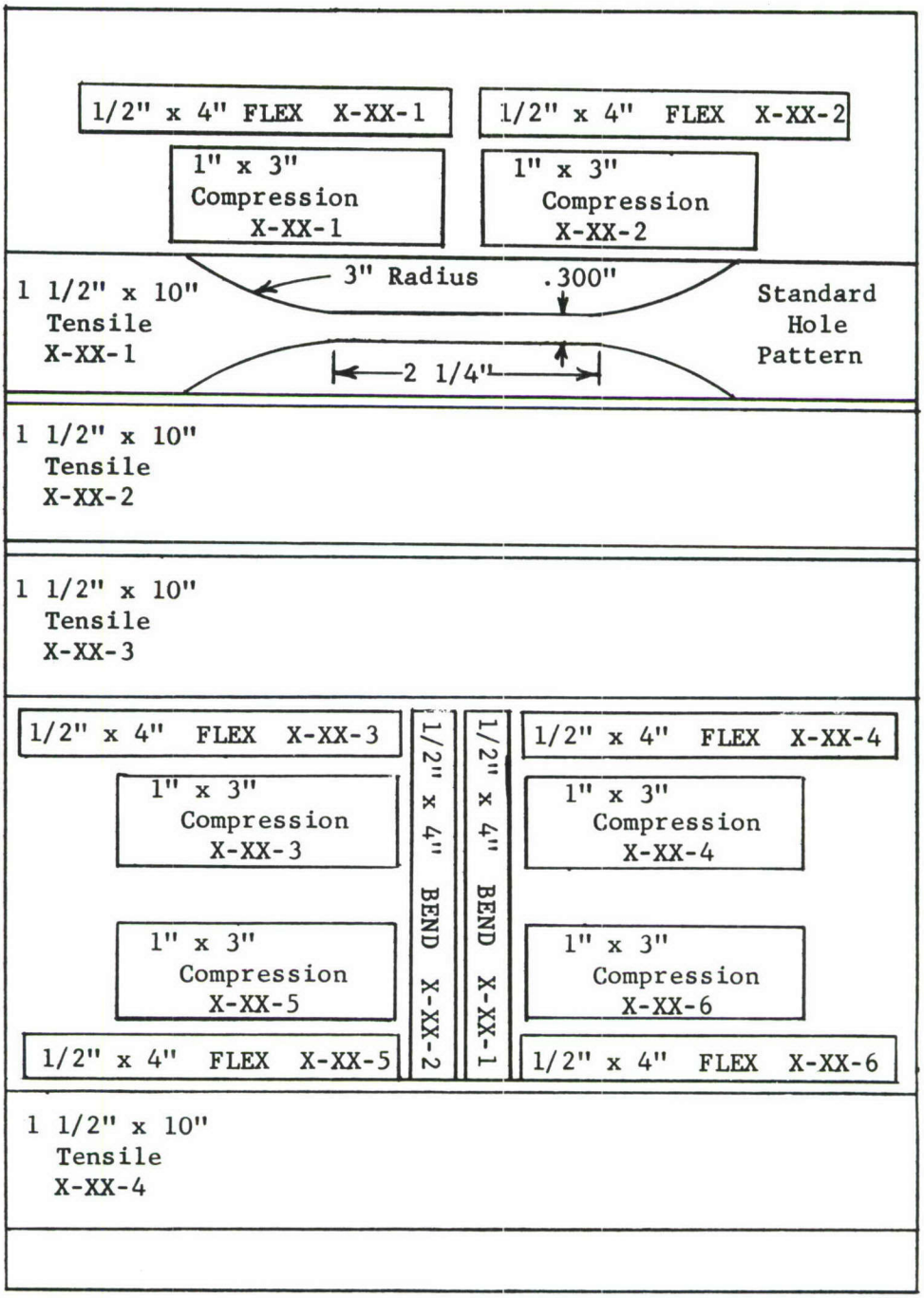
3.3.2 Bending Tests of Specimens From The Cylindrical Shape

a. Scope: The bending test was selected as the type of test that could be used conveniently on test specimens cut circumferentially from the cylinder. This method was selected over the three point flexural tests, by means of a set of comparison tests with resistance type strain gages mounted on the samples. Better agreement of flat laminate data was obtained when the bending test was used to measure the tensile properties of the materials.

b. Test Specimen: A drawing of the layout used to cut specimens from the cylinder is shown in Figure 59. The specimens were cut and finished as shown in (D) of Figure 60.

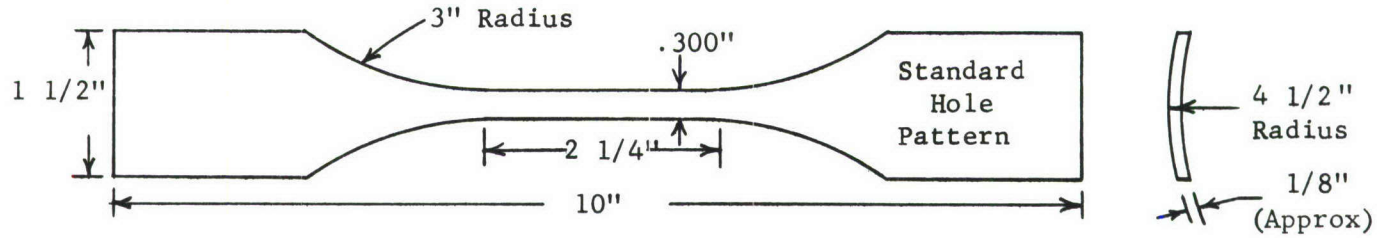
c. Apparatus: A 200 pound capacity Instron testing machine was employed to supply the load needed to test the specimens. The specimens were unsupported.

d. Test Procedures and Calculations: The following formulas, used to calculate the strength of material in the curved specimens, were taken from page 366 in the book, "Resistance of Materials," by F. B. Seely, Professor of Theoretical and Applied Mechanics, University of Illinois.

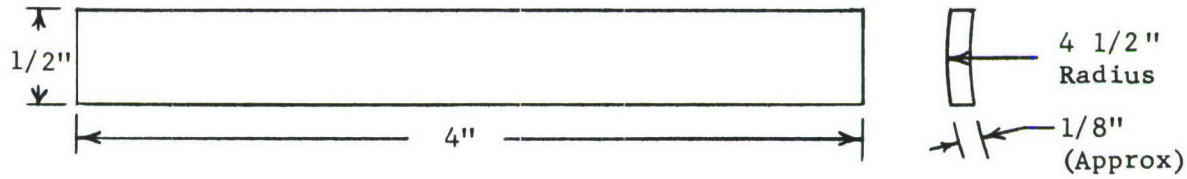


(1/2 scale)

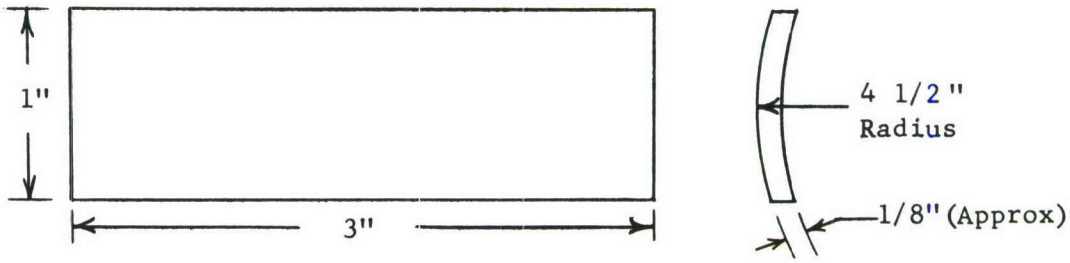
Figure 59 - Layout of Test Specimens on Cylinder Shape



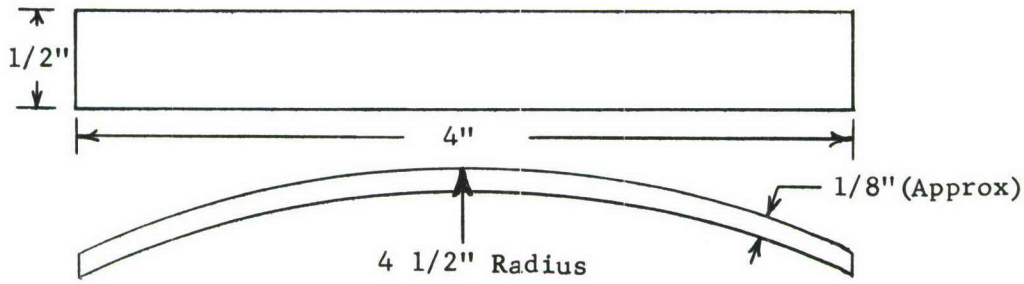
(A) Tensile Specimen



(B) Flex Specimen



(C) Compression Specimen



(D) Bend Specimen

Figure 60 - Test Specimen From The Cylindrical Molding

TABLE 31 THE RESULTS OF TENSILE, COMPRESSIVE, AND FLEXURAL TESTS ON SIMPLY CURVED SPECIMENS CUT FROM THE CYLINDRICAL MOLDINGS

TENSILE DATA

Material Composition	Standard Specimens (Flat Laminates)				Curved Cylinder Specimens (Cut Longitudinally)				% Deviation From Flat Specimens	
	Ult. Stg. (psi)		Modulus (psix10^{-6})		Ult. Stg. (psi)		Modulus (psix10^{-6})			
	ave.	σ	ave.	σ	ave.	σ	ave.	σ	Ult. Stg.	Modulus
X-270 Epoxy	21,100 (12)	± 1600 (12)	5.19 (12)	± .35 (12)	15,400 (28)	± 5400 (28)	5.94 (28)	± 1.15 (28)	- 27.0	+ 14.5
X-271 Epoxy	21,600 (12)	± 2700 (12)	4.89 (12)	± .19 (12)	12,300 (8)	± 3000 (8)	4.12 (8)	± .45 (8)	- 43.0	- 15.7
X-273 Phenolic	12,000 (12)	± 3200 (12)	5.02 (12)	± .23 (12)	12,800 (24)	± 2600 (24)	4.93 (24)	± 1.00 (24)	+ 6.7	- 1.8
X-274 Silicone	9800 (12)	± 1300 (12)	4.66 (12)	± .46 (12)	4700 (19)	± 1600 (19)	4.07 (17)	± 1.12 (17)	- 52.0	- 12.7

COMPRESSION DATA

Material Composition	Standard Specimens (Flat Laminates)				Curved Cylinder Specimens (Cut Longitudinally)				% Deviation From Flat Specimens	
	Ult. Stg. (psi)		Modulus (psix10^{-6})		Ult. Stg. (psi)		Modulus (psix10^{-6})			
	ave	σ	ave.	σ	ave.	σ	ave.	σ	Ult. Stg.	Modulus
X-270 Epoxy	48,200 (13)	± 3800 (13)	4.87 (13)	± .17 (13)	46,500 (36)	± 7300 (36)	5.32 (36)	± .37 (36)	- 3.5	+ 9.2
X-271 Epoxy	46,500 (15)	± 2800 (15)	4.44 (15)	± .36 (15)	28,700 (12)	± 3900 (12)	3.73 (11)	± .25 (11)	-38.3	-16.0
X-273 Phenolic	31,900 (15)	± 6100 (15)	4.90 (15)	± .30 (15)	38,200 (36)	± 6700 (36)	4.39 (36)	± .24 (36)	+19.7	-10.4
X-274 Silicone	17,200 (15)	± 1500 (15)	4.55 (15)	± .34 (15)	9900 (38)	± 2100 (38)	3.62 (34)	± .40 (34)	- 42.4	- 20.4

(Continued on next page)

TABLE 31 THE RESULTS OF TENSILE, COMPRESSIVE AND FLEXURAL TEST ON SIMPLY CURVED SPECIMENS (Continued)

FLEXURAL DATA

Material Composition	Standard Specimens (Flat Laminates)				Curved Cylinder Specimens (Cut Longitudinally)				% Deviation From Flat Specimens	
	Ult.Stg. (psi)		Modulus (psix10^{-6})		Ult.Stg. (psi)		Modulus (psix10^{-6})			
	ave.	σ	ave.	σ	ave.	σ	ave.	σ	Ult.Stg.	Modulus
X 270 Epoxy	38,400 (15)	± 2400 (15)	5.42 (15)	± .20 (15)	36,300 (30)	± 3300 (30)	5.51 (30)	± .49 (30)	- 5.5	+ 1.7
X 271 Epoxy	34,400 (15)	± 3900 (15)	4.99 (15)	± .14 (15)	28,000 (12)	± 6400 (12)	4.85 (12)	± .43 (12)	-18.6	- 2.8
X 273 Phenolic	25,300 (15)	± 3700 (15)	5.28 (15)	± .12 (15)	28,900 (30)	± 4000 (30)	4.92 (30)	± .44 (30)	+14.2	- 6.8
X 274 Silicone	20,800 (15)	± 2300 (15)	4.38 (15)	± .20 (15)	13,800 (36)	± 3100 (36)	3.74 (36)	± .65 (36)	-33.6	-14.6

NOTES: 1. Figures in parentheses are the number of samples used.

2. Symbol (σ) stands for standard deviations.

To determine if Formula (6) on page 128 of this report was applicable to these tests, actual strain measurements were made for two specimens taken from an X270 composite cylinder. Strain was measured with resistance type strain gages applied to each face of the specimen, as shown in Figure 61.

The strain data were plotted against the tensile stress, calculated from Formula (6), Figure 61, and is shown in Figure 62. The modulus was calculated to be 4.8×10^6 psi. This value agrees with the previous data obtained from flat laminates of this material. Therefore, Formula (6) was accepted as valid, and the balance of the specimens were tested without strain gages.

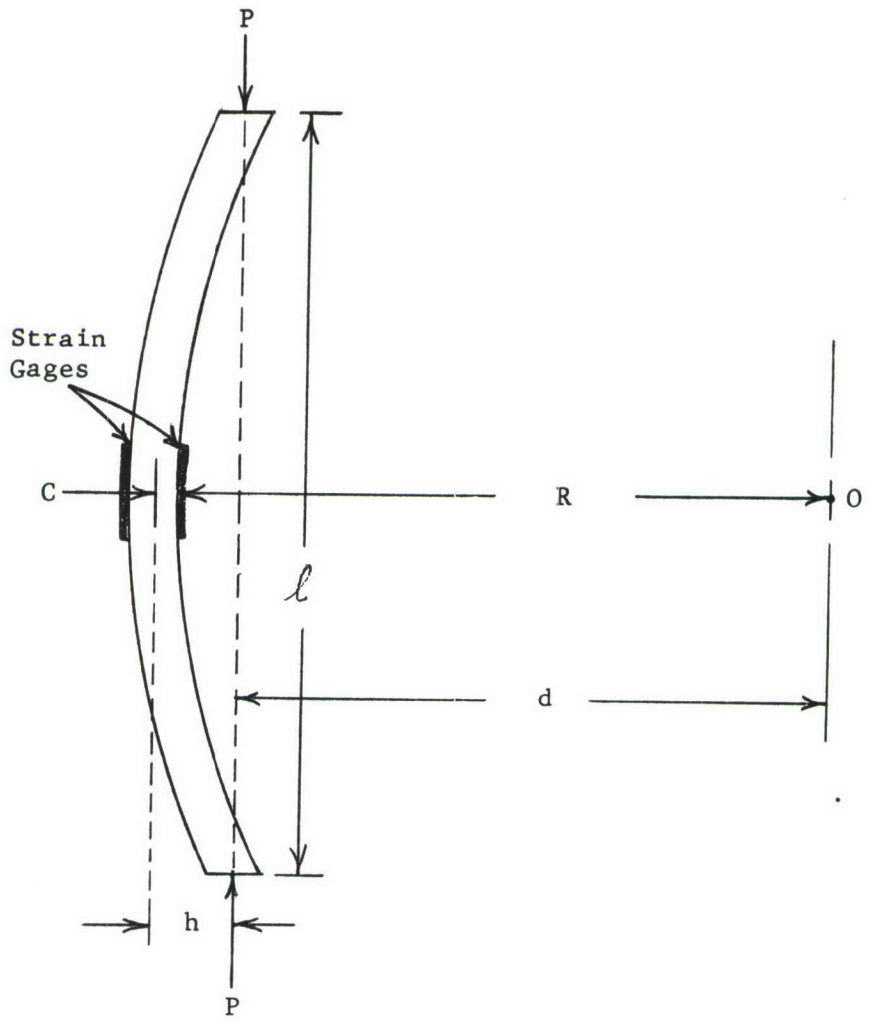
e. Test Results: The results of the bending tests appear in Table 32.

3.3.3 A Discussion of The Molding And Testing of The Cylindrical Section

Where the conventional mechanical tests were applied, a definite trend toward reduced strength, on the order of 10% to 20% below that observed for flat laminates, was noted with the cylindrical shape. We feel that these lowered strengths can be attributed to the wrinkles, "mudcracks," and other obvious evidences of poor flake alignment that were observed with the cylindrical shapes. It appears that flaws of this nature are unavoidable, and must be accepted as the penalty to be paid if the flake-resin mixture is forced to flow into geometrical shapes. Two definite exceptions to the trend showing reduced strengths were observed. These were the modulus values for the X270 epoxy and the properties of the X273 phenolic system. A satisfactory explanation for these abnormal values was not readily apparent.

When the bending test was applied to the cylindrical shape, the data presented in Table 32 were obtained, and again it was very difficult to offer precise explanations. However, in the actual testing of these materials, certain observations were made which tend to temper these results. For example, the failures of 50% of the X274 silicone specimens were observed as shear or delamination, rather than the clear tensile type failures which were noted for the other materials. This fact indicates that the nature of the silicone laminates is such that the bending test was not a good measure of tension. In the case of the X271 epoxy, examination of the failed areas showed an obvious flaw in one of the four specimens. As only three other specimens were tested, there is a definite question as to whether or not these data are worthy of consideration.

It should be noted at this time that the small number of X271 specimens resulted from the decision to discontinue testing of the material. This decision was made when the results of the temperature exposure showed that the X271 epoxy did not offer the expected advantages over the X270 epoxy.



(1) $C = 1/2$ thickness

(2) $d = 1/2 \sqrt{[2(R+C)]^2 - \ell^2}$

(3) $h = R + C - d$

(4) $M = Ph$

(5) $S_{comp} = \frac{P}{A} + K \frac{MC}{I}$

(6) $S_{ten} = \frac{P}{A} - K \frac{MC}{I}$

NOTE: K is constant for curved specimens

Figure 61 - Formulas For The Calculation Of Tensile Stress in Bending Specimens

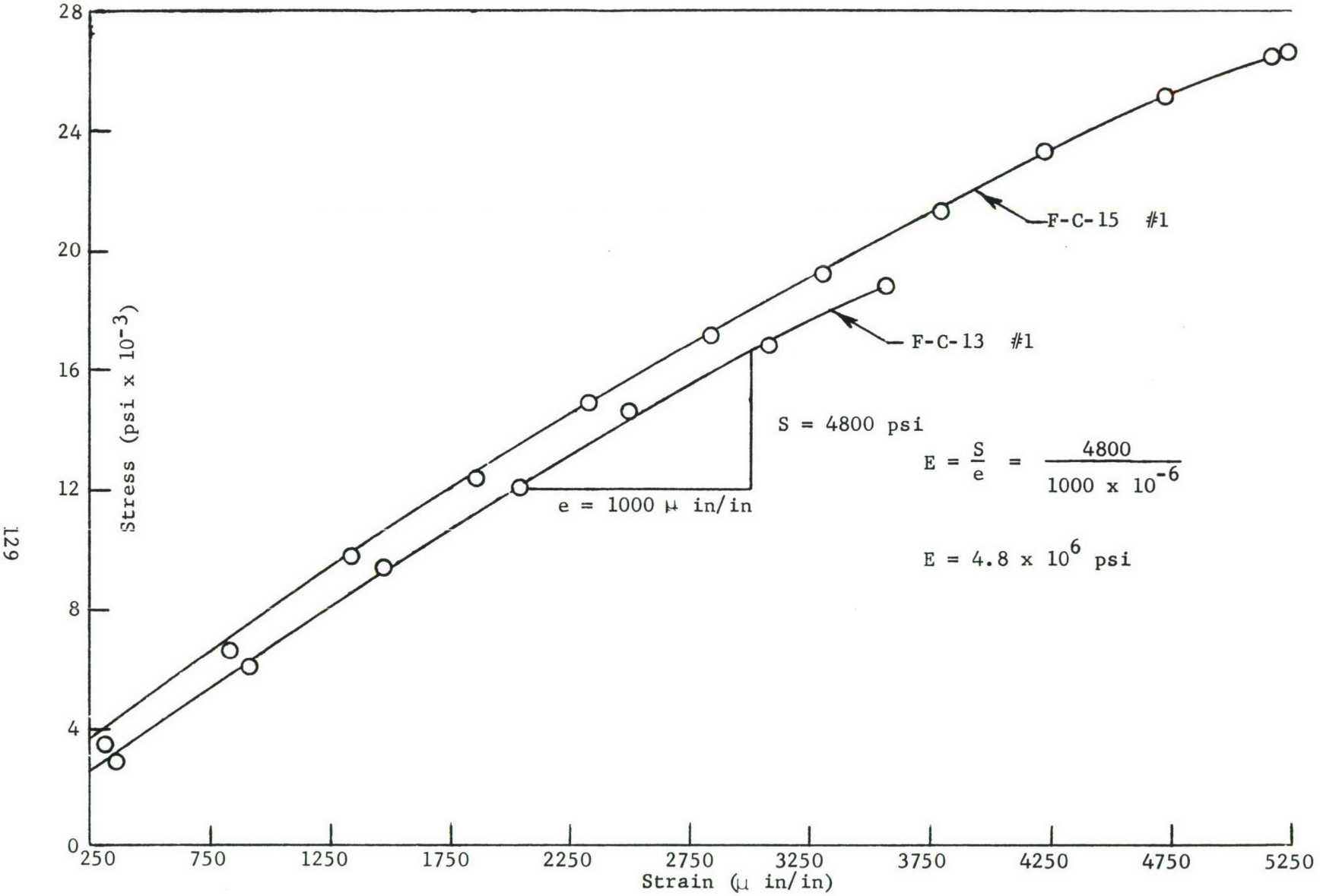


Figure 62 - Calculation of Modulus of Elasticity in Tension For Bending Specimens

TABLE 32 RESULTS OF THE BENDING TESTS ON THE CYLINDRICAL MOLDING

Material Composition	Standard Specimens (Flat Laminates)				Curved Cylinder Specimens (Cut Circumferencially)				% Deviation from flat Specimen (Ult. Stg.)
	Tensile Strength		Tensile Modulus		Tensile Strength		Tensile Modulus		
	ave.	σ	ave.	σ	ave.	σ	ave.		
X-270 Epoxy	21,100 (12)	± 1600 (12)	5.19 x 10^{-6} (12)	± .35 x 10^{-6} (12)	25,300 (7)	± 4900 (7)	4.8 x 10^{-6}		+ 19.9
X-271 Epoxy	21,600 (12)	± 2700 (12)	4.89 x 10^{-6} (12)	± .19 x 10^{-6} (12)	12,900 (4)	± 2800 (4)	Not Det.		- 40.3
X-273 Phenolic	12,000 (12)	± 3200 (12)	5.02 x 10^{-6} (12)	± .23 x 10^{-6} (12)	20,400 (10)	± 2200 (10)	Not Det.		+ 70.0
X-274 Silicone	9800 (12)	± 1300 (12)	4.66 x 10^{-6} (12)	± .46 x 10^{-6} (12)	8200 (9)	± 1700 (9)	Not Det.		- 16.3

TABLE 32 RESULTS OF THE BENDING TESTS ON THE CYLINDRICAL MOLDING

Material Composition	Standard Specimens (Flat Laminates)				Curved Cylinder Specimens (Cut Circumferencially)				% Deviation from flat Specimen (Ult. Stg.)
	Tensile Strength		Tensile Modulus		Tensile Strength		Tensile Modulus		
	ave.	σ	ave.	σ	ave.	σ	ave.		
X-270 Epoxy	21,100 (12)	± 1600 (12)	5.19 x 10^{-6} (12)	± .35 x 10^{-6} (12)	25,300 (7)	± 4900 (7)	4.8 x 10^{-6}		+ 19.9
X-271 Epoxy	21,600 (12)	± 2700 (12)	4.89 x 10^{-6} (12)	± .19 x 10^{-6} (12)	12,900 (4)	± 2800 (4)	Not Det.		- 40.3
X-273 Phenolic	12,000 (12)	± 3200 (12)	5.02 x 10^{-6} (12)	± .23 x 10^{-6} (12)	20,400 (10)	± 2200 (10)	Not Det.		+ 70.0
X-274 Silicone	9800 (12)	± 1300 (12)	4.66 x 10^{-6} (12)	± .46 x 10^{-6} (12)	8200 (9)	± 1700 (9)	Not Det.		- 16.3

The two remaining materials which were tested in bending were the X270 epoxy and the X273 phenolic. Both of these systems show a significantly higher tensile value than was observed for the flat dogbone tensile specimens. This phenomenon is frequently observed for brittle material, perhaps due to the lower cross section area under stress and hence the lower likelihood of a microscopic flaw.

3.3.4 Conclusions Regarding the Cylindrical Moldings

In our initial attempt to mold the half section of a right circular cylinder, end closures for the cylinder were included in the molding. It was observed that the material was forced to flow from the curve portions of the mold into these ends. Poor alignment of the flake or wrinkling was observed in the areas in which flow had occurred. The mold was modified to delete these ends, and it was observed that the wrinkles appeared regardless of any changes that were made in processing parameters, such as molding pressure, temperature, time, etc. This fact indicated that flaws of this nature, with their attendant reductions in mechanical properties, were an inherent property of composites in which the flake-resin mixture was forced to flow.

4.0 MOLDING AND TESTING OF THE BOX MOLDING

4.1 Scope

The second shape which was selected for evaluation was an open box, 3" deep, with a 6" by 8" base. These boxes were tested in tension, flexure, and compression. The tests were intended to provide a comparison between the properties of flake composites when molded into flat laminates, and when molded into the box configuration. Various radii were formed at the edges of the box and were tested in bending, to furnish a comparison in properties of each radius.

4.2 Test Specimen Preparation

During molding of the boxes, molding parameters were maintained as near as possible to those used for the flat laminates. The mold used for these boxes is shown in Figures 63 and 64. Problems occurred in connection with this molding. The most troublesome was a purely mechanical problem of mold release. During the molding process, it was necessary for the flake-resin mixture to flow from the bottom of the box into the wall and flange areas. This flow was accompanied by a scouring action, which completely removes the release agent from these areas and results in moldings which stuck to the mold surface. This sticking was particularly a problem on the male or punch portion of the mold. Therefore, two grooves were ground into the cavity portion of the mold so that the moldings would be pulled free of the plunger as the press was opened. When we undertook to solve the problem in this manner, we underestimated the tenacity with which the

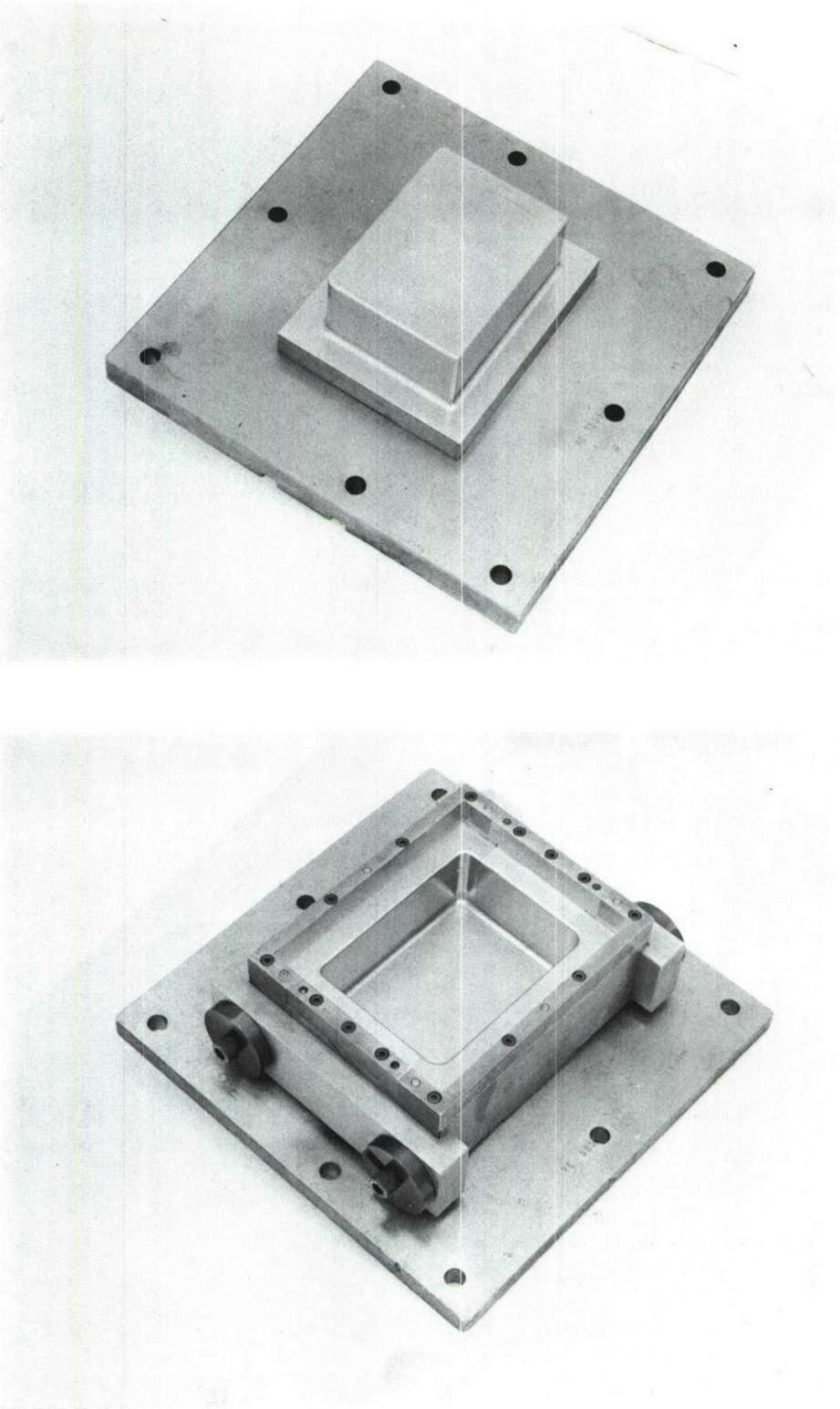


Figure 63 - The RE5206 Box Mold

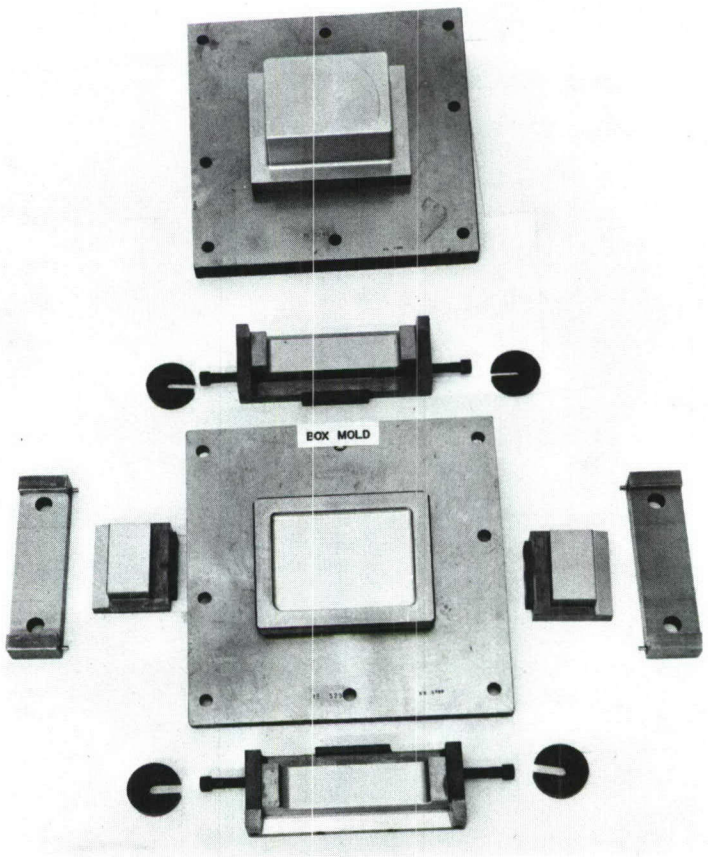


Figure 64 - The RE5206 Box Mold Disassembled
To Show Its Component Parts

moldings were bonded to the plunger. These adhesive forces were so great that the moldings were literally ripped to pieces when the press was opened.

Our second attempt to solve this problem was the grinding of about .010" draft or taper into the two longer sides of the plunger, as these were the areas where the majority of the sticking had occurred. This was not a solution, as sticking still occurred at the smaller ends of the plunger and at the radii. This was adequate in that it allowed the specimens described below to be prepared.

4.3 Tension, Flexure, and Compressive Tests of The Box

The methods for testing sections of the box were in accordance with the tensile, flexure, and compressive tests described in Section III of this report. A diagram showing the position in the molding, from which each specimen was cut, appears in Figure 65. Test results appear in Table 33.

4.4 Bending Test of Edges of The Box Molding

a. Scope: The bending test was selected as the type of test that could be used conveniently on test specimens cut from the edges. It was the intent of this test to establish the minimum radius which should be used by the designer of such a structure.

b. Test Specimen: A drawing of the layout used to cut specimens from the box is shown in Figure 65. The specimens were cut and finished to meet requirements, as described in Section III of this report.

c. Apparatus: A 200 pound capacity Instron testing machine was employed to supply the load needed to test the specimens. The specimens were unsupported.

d. Procedure: The test was conducted in a similar manner to that of the curved specimens that were tested from the cylinder. Formula No. 6 from page 128 of the report, was used to determine the tensile strength by bending, through a compression load applied to the edges. Modulus was not determined for these specimens. Figure 66 illustrates the position of the specimen in the test setup.

4.5 A Discussion of The Molding And Testing of The Box

Excellent agreement between the values obtained for the edges of the box and the segment cut from the cylinders was secured, except for the 1/8" radius. These results indicated that a minimum radius of 1/4" should be specified for all similar structures designed from glass-flake reinforced composites. These were the only satisfactory results that were obtained during the mechanical testing of segments cut from the box molding. The poor alignment of the flake, described in

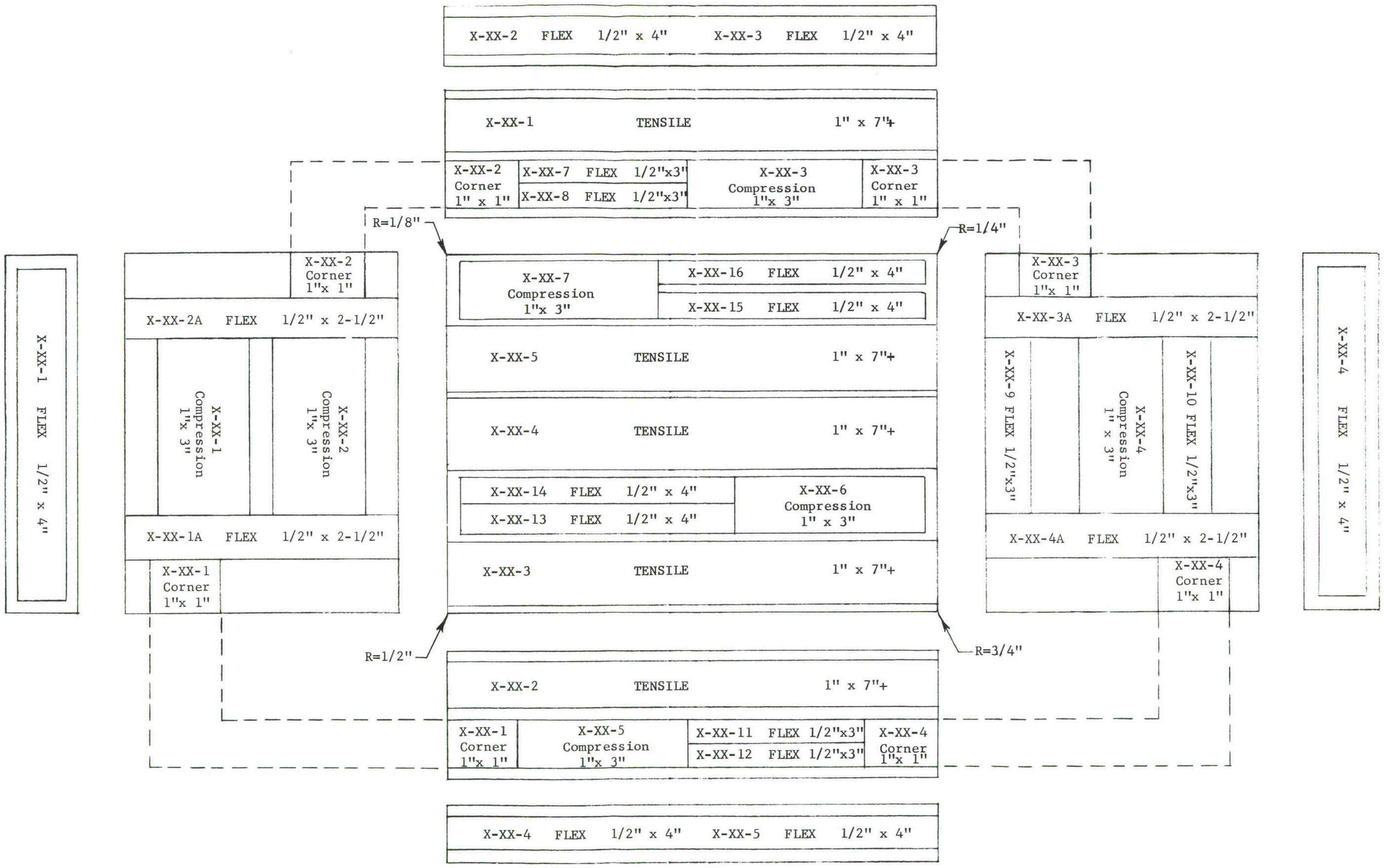


Figure 65 - Layout Of Test Specimens on Box Mold

TABLE 33 MECHANICAL PROPERTIES OF SPECIMENS CUT FROM BOXES MOLDED WITH THE X 270 EPOXY COMPOSITION

Sample Description	Ult. Tensile St. (psi)	Tensile Modulus (psi)	Ult. Flexural St. (psi)	Flexural Modulus (psi)	Ult. Compression St. (psi)	Compression Modulus (psi)
A. Bottom of Box *	16,800	5.1×10^6	32,500	5.9×10^6	38,000	4.9×10^6
B. Sides of Box						
1. Specimens parallel to wrinkle	16,400	5.0×10^6	30,300	5.6×10^6	43,400	$4,9 \times 10^6$
2. Specimens perpendicular to wrinkles	N.D.	N.D.	22,000	5.0×10^6	N.D.	N.D.
C. Flanges (specimen parallel to wrinkle)	N.D.	N.D.	34,300	6.1×10^6	N.D.	N.D.
D. Typical flat laminate values (wrinkle free)	21,100	5.2×10^6	38,400	5.4×10^6	48,200	4.9×10^6

* There were no obvious wrinkles within the bottom of the box.



Figure 66 - Testing The Edges of The Box Molding

the section of this report which deals with the cylindrical section, was noted to an even greater extent when attempts were made to mold the box. In the box, it was necessary for the material to flow from the bottom portion of the box, up the sides and into the upper flanges. As is shown in the photograph in Figure 67, the extensive flow resulted in wrinkling to a far greater extent than had been observed in the cylindrical section.

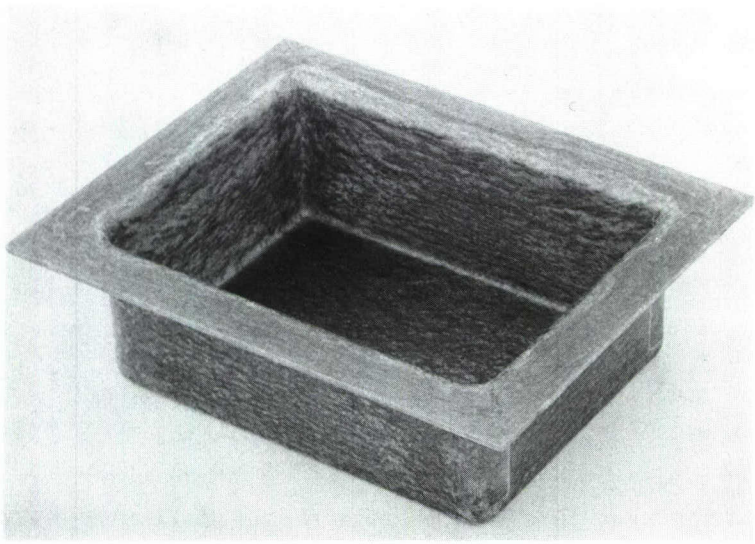


Figure 67 - A Photograph Of A Box Molded With X 270 Compound

The effect of this poor flake alignment on the mechanical properties of the specimens cut from the box is illustrated in the summary presented in Table 33. All of these values were well below those obtained with flat laminates.

It was interesting to note that the direction in which specimens were cut, with regard to the long axis of the wrinkles, definitely influenced the magnitude of the strength reduction. For example, as shown in Table 33, flexural strengths of specimens cut from the sides of the box, with their long axis parallel to the wrinkles, showed a 21% reduction in strength as compared to flat laminates; whereas, specimens cut perpendicular to the wrinkles showed 43% lower strength. Although this difference was of interest, it must be stressed that all the ultimate mechanical properties of specimens cut from the boxes were 11% to 43% lower than similar specimens cut from wrinkle-free flat laminates.

In addition to the reduced mechanical strengths, it was necessary also to consider the general over-all appearance of the molded boxes. The appearance was even more discouraging than the actual strength reductions observed. As shown in Figure 57, the glass flakes in a wrinkled area were not aligned parallel to the surface of the structure, but were tipped until some flakes were more nearly perpendicular to this surface.

Thermal expansion data was presented, in an earlier section of this report, showing that the expansion perpendicular to the plane of the flake is several times greater than the expansion in the plane of the flake. Hence, at a wrinkle, differential shrinkage occurred when the specimen was cooled from molding temperature. This resulted in a corrugation of the surface and in a buildup of stress within the wrinkles. This stress was evidenced by actual cracking and by optical inspection with polarized light. Such stressed areas would undoubtedly have an adverse effect on the fatigue life of molded structures.

4.6 Conclusion Regarding The Box Molding

Thus, it can be concluded that there is a definite wrinkling, or poor flake alignment, which occurs in the vertical walls and flange areas of the mold because of the flow during the molding process. It has become increasingly apparent that problems of this nature are an inherent property of the flake-reinforced composites and cannot be avoided. This shortcoming of the flake materials will quite likely prove to be the one single factor which prohibits their widespread use. If flow must be limited during the molding process to produce satisfactory moldings, then there is actually little or no advantage to flake composites.

5.0 MOLDING AND TESTING OF THE TAPERED PLATE

A tapered plate was selected as a shape which would allow a convenient evaluation of the moldability of flake reinforced molding mixtures. Furthermore, this shape could be tested as a cantilevered beam in the "as molded" condition

without specimen preparation. Thus, the effect of flaws within the structure could readily be determined.

5.1 Molding of The RE 5207 Plate

In all cases, the standard mixture of 70% glass and 30% resin was employed. Flakes were untreated, 2 micron, "E" glass which was sorted to U. S. Standard mesh sizes between 10 and 40. An attempt was made to control the processing of the tapered plates, so that all of the molding conditions were essentially the same as those which were used for the flat laminates. A detailed description of the processing conditions used for each tapered plate appears in Table 34.

Since this particular shape required considerable movement of the material, and since the direction of this flow could be readily predicted, the tapered plate provided an opportunity to observe the effect of flow in the mold on the alignment of flake within a composite. Photographs of the results of this study appear in Figures 68 through 72. The thicker end of the moldings is at the bottom of all photographs. The results of a molding obtained by directly charging the mold with the lose resin-flake mixture are shown in Figure 68. In this case, the direction of flow was from the thinner end of the taper into the thicker section. On the basis of observation made on the moldings of other geometric shapes, it was predicted that wrinkles would occur parallel to the thinner end and concentrated in the thicker areas of the molding. The resulting laminate was wrinkled in exactly this manner.

TABLE 34 PROCESSING PARAMETER FOR THE RE 5207 TAPERED PLATES

S/N	Initial Wt. (gm)	Loading Cond.	Contact Time	Pressure (psi)	Cure Temp °F	Cure Time	Comments
Epoxy X 270 1	1230	Preform	2 Min.	800	350	2 Hrs.	Loaded in thickside only with a 6" x 12" preform
2	1300	Preform	2 Min.	800	350	2 Hrs.	Loaded on thickside only with a 6" x 12" preform
3	1300	Loose	2 Min.	800	350	2 Hrs.	Loose material was charged directly into preheated mold
4	1400	Loose 2	2 Min.	800	350	2 Hrs.	Loose material was charged directly into preheated mold
5	1400	Preform	2 Min.	800	350	2 Hrs.	Material was preformed in the mold @ 200°F in (3) 466 GM sections
6	1400	Preform	2 Min.	800	350	2 Hrs.	Loaded on thin side only with a 6" x 12" preform
7	1400	Preform	2 Min.	800	350	2 Hrs.	Loaded on thick side only with a 6" x 12" preform
8	1400	Preform	2 Min.	800	350	2 Hrs.	Six 1/8" thick preforms stepped @ 2" intervals were charged into the mold
9	1400	Preform	2 Min.	800	350	2 Hrs.	Four 3/32" thick preforms stepped @ 1" intervals and one 12" x 12" on top, bottom and middle were charged into the mold
10	1400	Preform	2 Min.	800	350	2 Hrs.	Eleven 3/32" preforms stepped @ 1" intervals were charged into the mold

NOTE: All specimens consisted of 2 micron, untreated, composition E, glass flake used with a glass resin ratio of 70 to 30 by weight.

(Continued on next page)

TABLE 34 PROCESSING PARAMETER FOR THE RE 5207 TAPERED PLATES (Continued)

	S/N	Initial Wt. (gm)	Loading Cond.	Contact Time	Pressure (psi)	Cure Temp °F	Cure Time	Comments
Phenolic X 273	1	1400	Loose	0	800	250, 300, 400	1 Hr. @ each temp	Preheated mold cavity loaded with loose material
	2	1400	Loose	0	800	250, 300, 400	1 Hr. @ each temp	Preheated mold cavity loaded with loose material
	3	1400	Loose	0	800	250, 300, 400	1 Hr. @ each temp	Preheated mold cavity loaded with loose material
Silicone X 274	1	1400	Loose	1 Hr.	1000	350 500	1 Hr. 8 Hrs.	Loose material was directly loaded into preheated mold cavity
	2	1400	Loose	1 Hr.	1000	350	1 Hr.	Loose material was directly loaded into preheated mold cavity
	3	1400	Loose	1 Hr.	1000	350 500	1 Hr. 8 Hrs.	Loose material was directly loaded into preheated mold cavity

NOTE: All specimens consisted of 2 micron, untreated, composition E, glass flake used with a glass resin ratio of 70 to 30 by weight.

Figure 69 shows a laminate which was molded in a manner similar to the molding shown in Figure 68, except that the material was charged into the mold in three steps. Each charge was compacted at 200°F and approximately 100 psi before the next portion was added. In this manner, the amount of flow was reduced, but the direction of flow remained the same as in the molding shown in Figure 68. Thus, the amount of wrinkling should have been reduced, whereas the pattern of wrinkles should have been substantially the same as previously noted. Examination of the molding shown in Figure 69 shows this to be true.

Next, to confirm that this wrinkling was a function of the direction and degree of flow, a molding was prepared from a series of pre-formed biscuits which were carefully tailored to minimize the amount of flow within the mold. These preforms were made in flat 12" x 12" slabs by compacting the resin-flake mixture at 200°F and 100 psi. The preforms were trimmed and cut to the appropriate size, so that they could be assembled into a wedge with increases in thickness at one inch intervals. This assembly was molded in the usual manner and the laminate, which is shown in Figure 70, resulted. Careful examination of this molding showed that although flow had been minimized, some movement of the material took place along each of the 1" steps. Hence, some slight wrinkling occurred in these areas and each step was clearly defined.

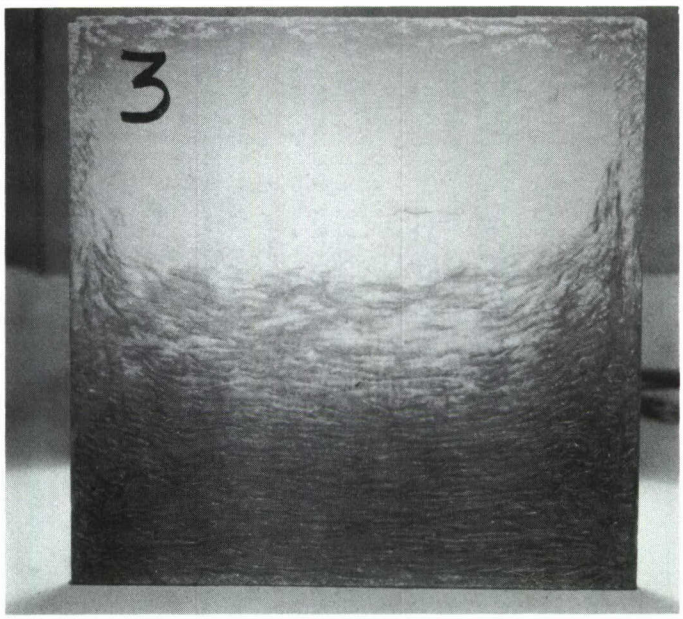


Figure 68 - Tapered Plate Molded From X 270 Epoxy. A Loose Charge of The Resin Flake Mixture Was Molded With No Attempt to Control The Flow of The Molding Compound

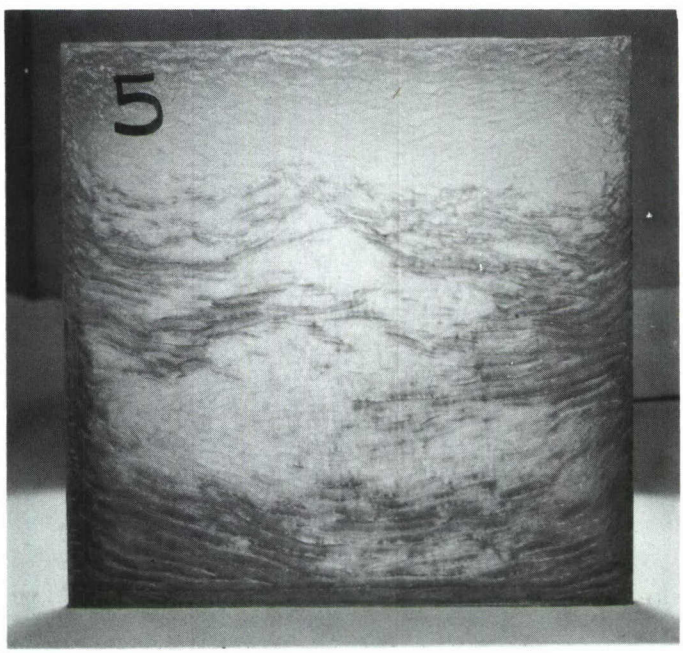


Figure 69 - A Molding Similar to The One Shown in Figure 68 Except Flow Was Reduced By Partially Pre-compacting The Molding Compound Prior To Molding

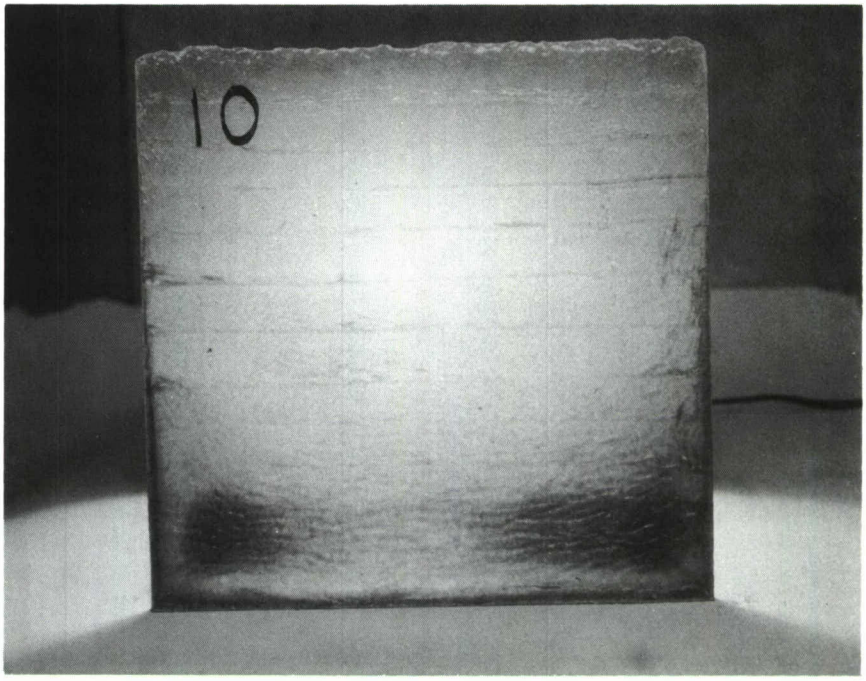


Figure 70 - A Molding Similar To The One Shown in Figure 68 Except Flow Was Minimized By Charging The Mold With A Carefully Tailored Precompacted Biscuit Of X270 Epoxy

Finally, the laminates shown in Figures 71 and 72 were prepared from 6" x 12" preformed biscuits of the required thickness. The laminate shown in Figure 71 was molded by placing the preformed biscuit in the thick end of the laminate and hence causing the material to flow into the tapered section of the mold. The laminate shown in Figure 72 was made from a preform charged into the thinner end of the mold cavity.

5.2 Testing of The Tapered Plates

5.2.1 Scope

Each of the tapered plates was tested as a cantilevered beam. Stress distribution within the tapered section was analyzed, and the stress at the point of failure was compared to the maximum stress within the panel. In this manner, the magnitude of the effect of the flaws in the laminates could be estimated.

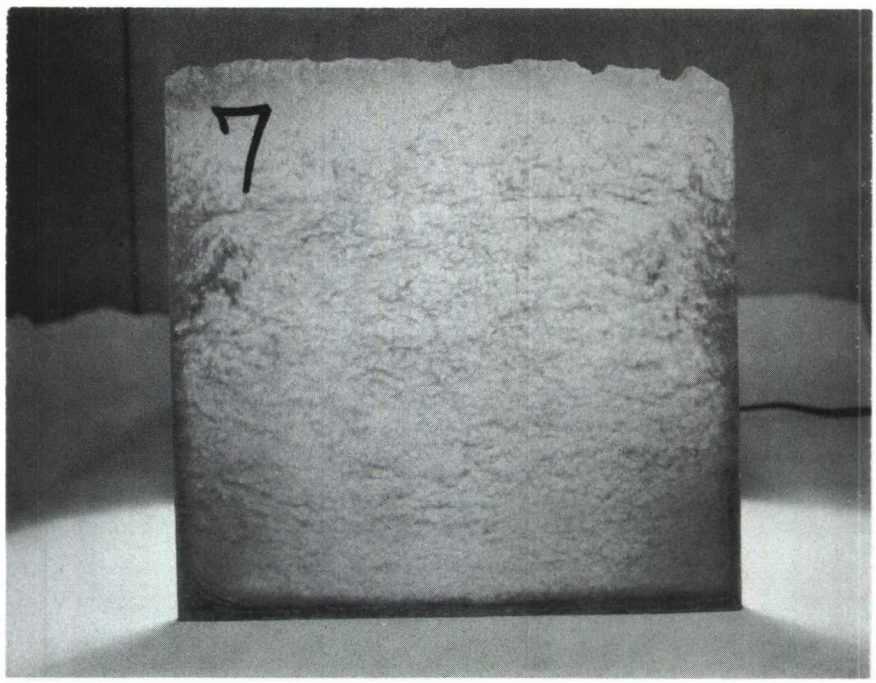


Figure 71 - A Tapered Plate Molded From a 6" x 12" Preformed Biscuit Placed In The Thicker End Of The Mold Cavity

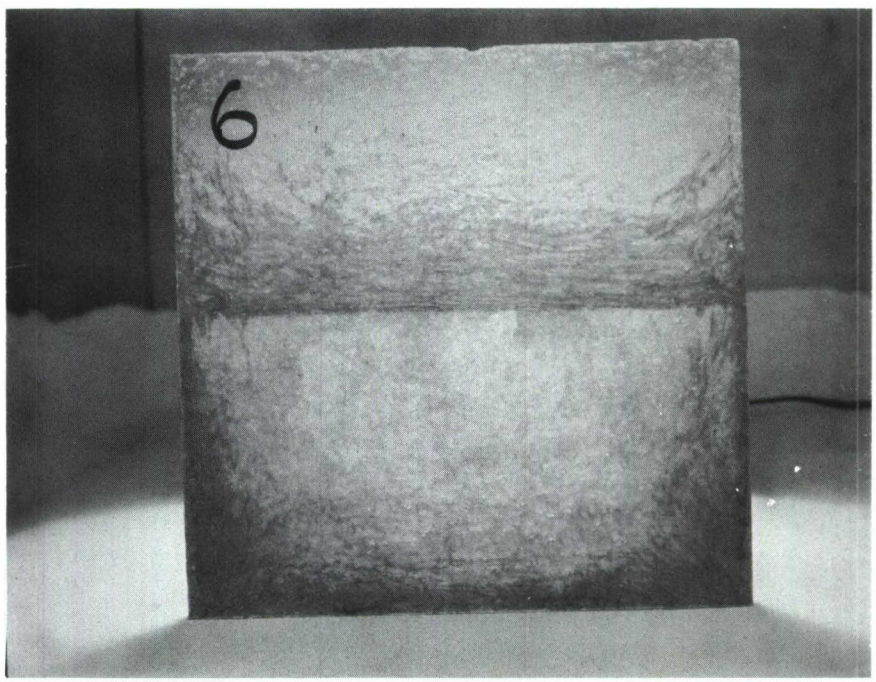


Figure 72 - A Tapered Plate Molded From A 6" x 12" Preformed Biscuit Placed In The Thinner End Of The Mold Cavity

5.2.2 Test Procedure

The 2" wide sections of uniform thickness at the thicker end of the moldings were clamped into the test fixture shown in Figure 73. Load was applied through the hydraulic mechanism until failure occurred. The ultimate load, the position of the failure, and the type of failure was recorded.

5.2.3 Test Results

The results of the bending tests of the cantilevered tapered plates appear in Table 35 and Figure 74.

5.2.4 A Discussion of The Results of The Bending Test of The Tapered Plates

Our analysis of the stress within the tapered plate, when loaded as a cantilevered beam, appears in Figure 75. This analysis shows that the stress within the tapered plate is not uniform, but rather that there is a peak stress that depends upon the position from which the load is applied. In the case of the specimens loaded at the 9" line, the peak stress occurs at the 8" line, whereas, the specimens loaded at the 6" line have a peak at approximately the 2" line. See Figure 74 for a definition of terms. An examination of the results shown in Table 35 reveals that the failure of nearly all the test specimens did not occur at the point of peak stress, but rather occurred at one of the obvious flaws. These failures all took place at stresses ranging between 59% and 90% of the peak stress within the panel.

It is further interesting to note, that even in the cases of the few samples which failed at the point of peak stress, all the specimens failed at values that were only 28% to 98% of the tensile strength values observed for typical flat laminates.

Thus, we have a further indication that the properties which were determined for flat laminates, without microscopic flaw, cannot be applied directly to molding with relatively simple shapes.

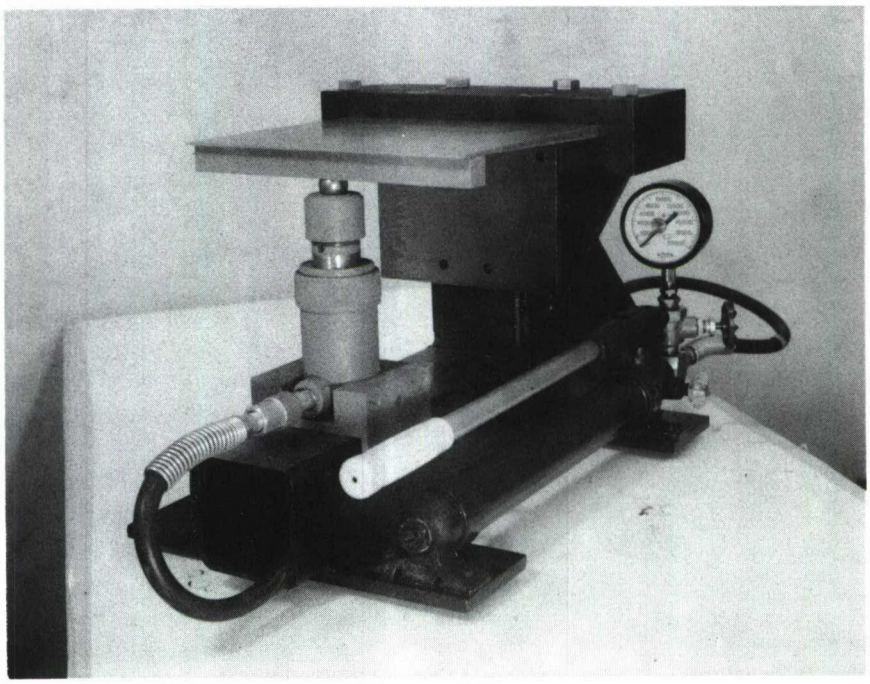


Figure 73 - A Photograph of The Fixture to Test The
RE 5207 Tapered Plates as Cantilevered Beams

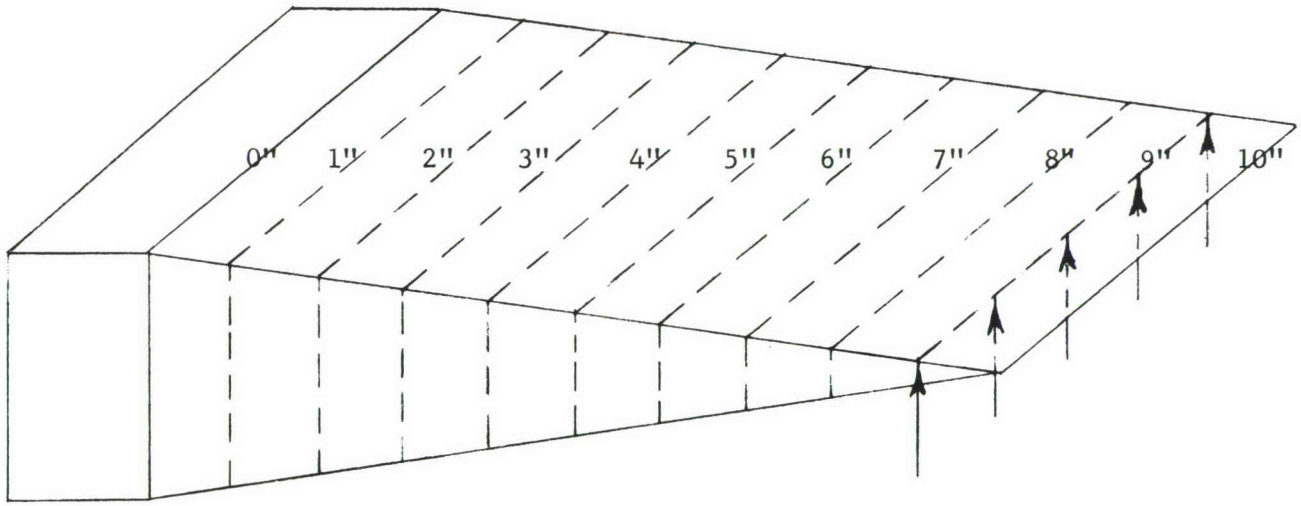


Figure 74 - A Sketch Of A Tapered Plate Showing Location Of Lines Which Were Used For Reference

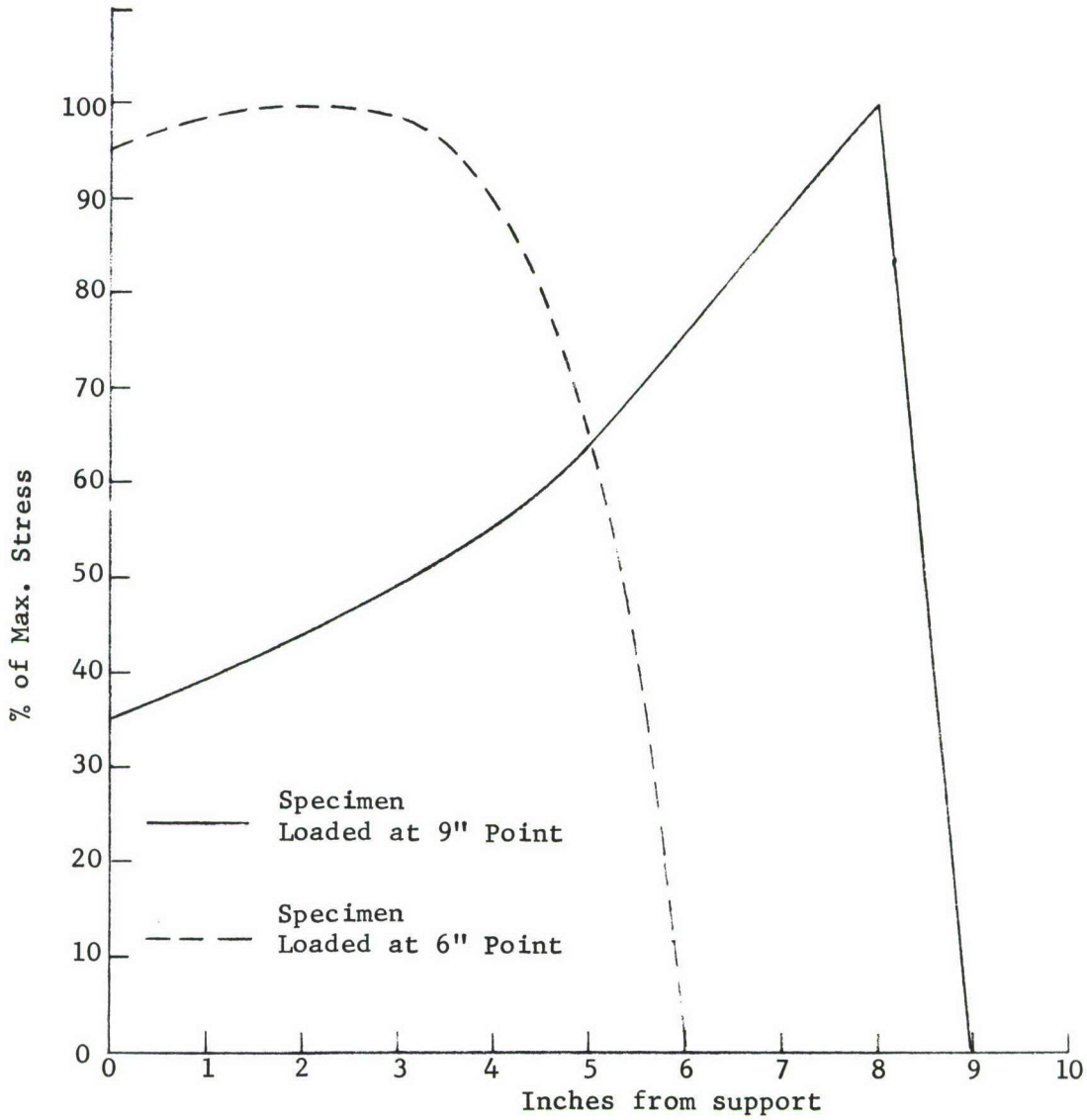


Figure 75 - Typical Stress Distribution in RE 5207 Tapered Plate At Failure During The Bending Test

TABLE 35

THE RESULTS OF TESTS IN BENDING OF THE RE 5207 TAPERED PLATES
X-270
EPOXY TAPER PLATES

Specimen Number	Stress in Taper Plate at Point of Failure psi	Maximum Stress in Taper Plate psi	% of Maximum Stress in Taper Plate at Point of Failure	Tensile Strength of Good Quality Flat Laminates psi	% of Tensile Strength of Flat Laminates at Point of Failure in The Taper Plate	Comments
3	10,500	17,900	58.6%	21,100 ± 1600	49.8%	Failed at wrinkle at 1" line
4	7,600	11,025	68.9%	21,100 ± 1600	36.0%	Failed at wrinkle at 0.5" line
5	11,400	13,500	84.4%	21,100 ± 1600	54.0%	Failed at wrinkle at 4" line
7a	11,625	12,475	93.2%	21,100 ± 1600	55.1%	Failed at wrinkle at 7" line
7b	13,825	15,750	87.8%	21,100 ± 1600	65.5%	Failed at wrinkle at 2" line
9b	10,325	10,375	100.0%	21,100 ± 1600	48.9%	Failed at wrinkle at 1.5" line
10b	18,725	18,725	100.0%	21,100 ± 1600	88.7%	Failed at wrinkle at 1.5" line
11	6,200	10,575	58.6%	21,100 ± 1600	29.4%	Failed at wrinkle at 0" line
12	15,875	23,800	66.7%	21,100 ± 1600	75.2%	Failed at wrinkle at 1" line
Average	11,775	14,900	79.0%	21,100	55.8%	
6	5,850	7,275	80.4%	21,100 ± 1600	27.7%	Failed at edge of preform at 4.5" line
8a	9,475	10,600	89.4%	21,100 ± 1600	44.9%	Failed at edge of preform at 6" line
8b	16,400	19,400	84.5%	21,100 ± 1600	77.7%	Failed at edge of preform at 1" line
9a	13,500	13,500	100.0%	21,100 ± 1600	64.0%	Failed at edge of preform at 7" line
10a	20,600	20,600	100.0%	21,100 ± 1600	97.6%	Failed at edge of preform at 8" line
Average	13,175	14,275	92.3%	21,100	62.4%	

Note: Specimens 7b, 8b, 9b & 10b, loaded uniformly at 6" line
All other specimens loaded uniformly at 9" line

TABLE 35 (Continued)

X-273

PHENOLIC TAPER PLATES

Specimen Number	Stress in Taper Plate at Point of Failure psi	Maximum Stress in Taper Plate psi	% of Maximum Stress in Taper Plate at Point of Failure	Tensile Strength of Good Quality Flat Laminates psi	% of Tensile Strength of Flat Laminates at Point of Failure in the Taper Plate	Comments
1	7550	9,250	81.6%	12,000 ± 3200	62.9%	Failed at wrinkle at 0" line
2	9750	12,400	78.6%	12,000 ± 3200	81.2%	Failed at wrinkle at 0" line
3	8000	9,700	82.5%	12,000 ± 3200	66.7%	Failed at wrinkle at 0" line
Average	8433	10,450	80.7%	12,000	70.3%	

Note: All specimens loaded uniformly at 9" line

X-274

SILICONE TAPER PLATES

Specimen Number	Stress in Taper Plate at Point of Failure psi	Maximum Stress in Taper Plate psi	% of Maximum Stress in Taper Plate at Point of Failure	Tensile Strength of Good Quality Flat Laminates psi	% of Tensile Strength of Flat Laminates at Point of Failure in the Taper Plate	Comments
1	4000	6400	62.5%	9800 ± 1300	40.8%	Failed at wrinkle at 0.5" line
2	3500	5300	66.0%	9800 ± 1300	35.7%	Failed at wrinkle at 0" line
3	3750	6325	59.3%	9800 ± 1300	38.3%	Failed at wrinkle at 0" line
Average	3750	6000	62.5%	9800	38.3%	

Note: All specimens loaded uniformly at 9" line

6.0 MOLDING AND TESTING OF THE HEMISPHERE

A nine-inch diameter hemisphere with a wall thickness of approximately 0.10 inch was selected as an example which would demonstrate the molding of parts with a compound contour. Conventional mechanical tests were not practical because of the compound contour, so this shape was tested hydrostatically in tension.

6.1 Molding of Test Specimens

The mold shown in Figures 76 and 77 was used to mold the hemispherical parts. A problem with the temperature controller on this mold caused the first four moldings to be discarded. After this difficulty was corrected, molding proceeded smoothly. The hemispherical shape seemed to be nearly ideally suited to the flake molding compounds. This shape was more successfully molded than any of the others investigated. Some wrinkling of the flake in the composite occurred, but this wrinkling was limited to a strip approximately one inch wide around the lip of the specimen. Processing parameters were changed as noted in Table 36, but these changes did not correct the wrinkling problem. A photograph of one of the molded hemispheres is shown in Figure 78.

6.2 Test Procedures

The compound contour of the hemispherical molding made conventional mechanical testing impractical. For this reason, the moldings were tested hydrostatically in tension. The test was accomplished by bonding the hemispheres between two stainless steel rings, which were in turn sealed into the pressure chamber shown in Figures 79 and 80. Since the hemisphere was restrained at the attachement to the stainless steel rings, an unknown binding stress was introduced during the pressure test and conventional formulas for hoop tension could not be applied. This problem was avoided by directly measuring the strain during the burst test with electronic resistance strain gages, attached as shown in Figure 81. Since the modulus of elasticity was known from previous work, tensile stress in the hemisphere could be calculated and related to the hydraulic pressure. It should be noted that all of the hemispheres failed essentially in the strain gaged area. This was the same area in which wrinkling of the reinforcement was noted.

6.3 Test Results

The results of the hydrostatic pressure test appear in Table 37.

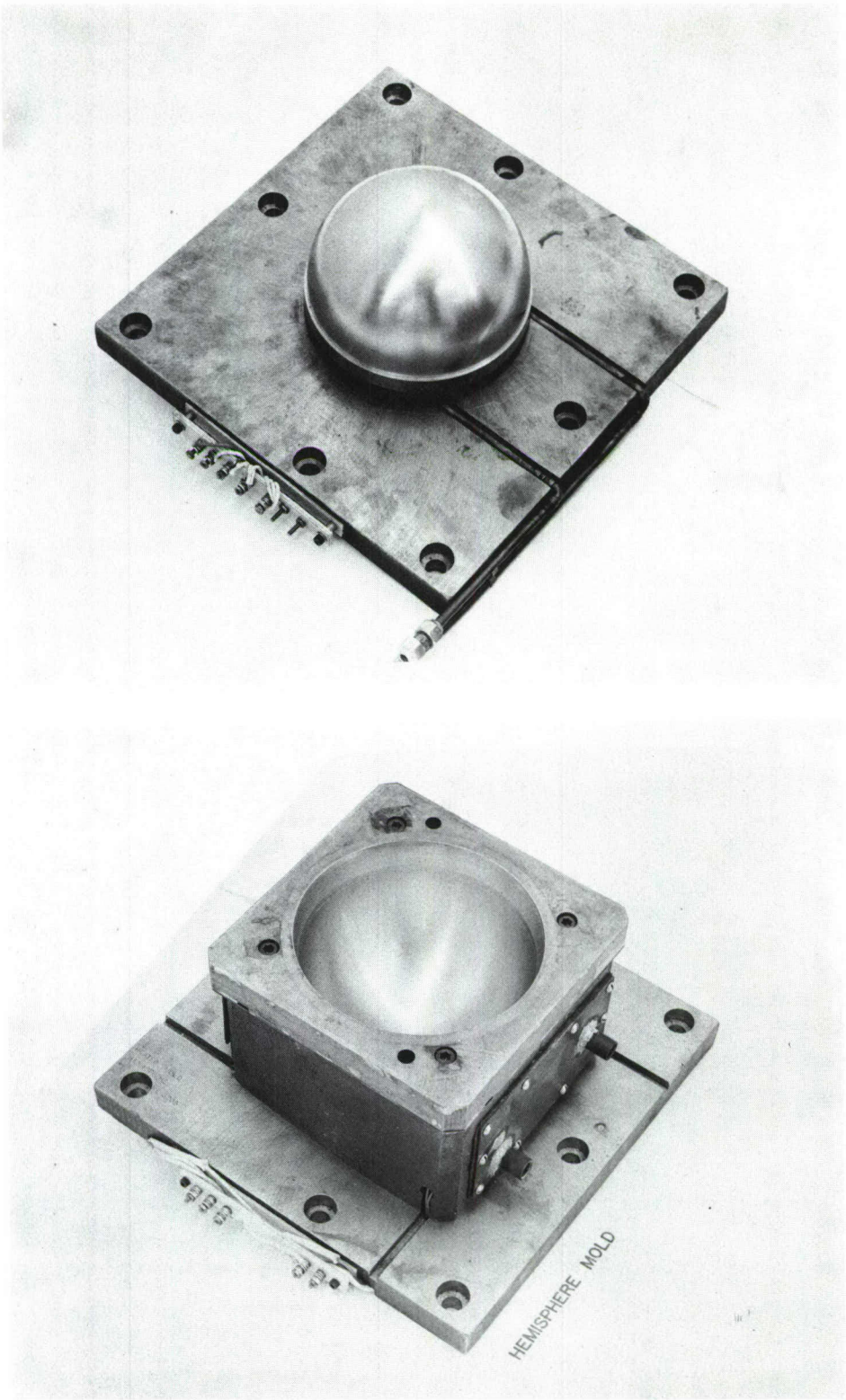


Figure 76 - The Hemisphere Mold

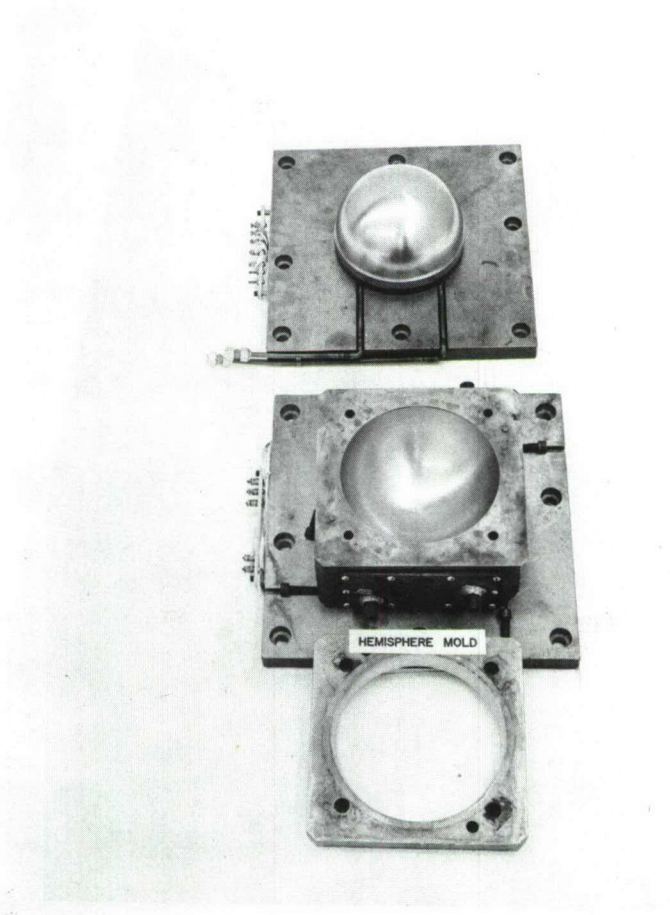


Figure 77 - The Hemisphere Mold Disassembled to Show its Component Parts

TABLE 36 MOLDING OF THE HEMISPHERE

MOLDING CONDITIONS

Molding Composition	Sample No.	Mold Temp.	Loading Condition	Contact Time	Cure Temp.	Pressure (psi)	Cure Time	Remarks
X270	F-H-1	350°F	Loose	2 min	350°F	800	2 hrs	Very dry on one side
X270	F-H-2	350°F	Loose	None	350°F	1000	2 hrs	Wrinkles and mud creases
"	F-H-3	350°F	Loose	2 min	350°F	1000	2 hrs	Wrinkles and dry surface
"	F-H-4	350°F	Loose	2 min	350°F	1000	2 hrs	Several Wrinkles
"	F-H-5	350°F	Loose	2 min	350°F	1000	2 hrs	Mud cracks and blown areas
"	F-H-6	350°F	Loose	2 min	350°F	1000	2 hrs	Mud cracks and wrinkles
"	F-H-7	350°F	Loose	3 min	350°F	1200	2 hrs	Slightly glazed surface (one side)
"	F-H-8	350°F	Loose	2.5 min	350°F	1600	2 hrs	Small dry spots
"	F-H-9	350°F	Loose	2.5 min	350°F	3000	2 hrs	Wrinkles and mud cracks
"	F-H-10	350°F	Loose	2.5 min	350°F	1600	2 hrs	Wrinkles and mud cracks
"	F-H-11	350°F	Loose	2.5 min	350°F	1600	2 hrs	Several wrinkles
"	F-H-12	350°F	Loose	2.5 min	350°F	1600	2 hrs	Several wrinkles
"	F-H-13	350°F	Loose	2.5 min	350°F	1600	2 hrs	Wrinkles
"	F-H-14	350°F	Loose	2.5 min	350°F	1600	2 hrs	Wrinkles
X270	F-H-15	350°F	Loose	2.5 min	350°F	1600	2 hrs	Wrinkles

(Continued on next page)

TABLE 36 continued

MOLDING CONDITIONS

Molding Composition	Sample No.	Mold Temp.	Loading Condition	Contact Time	Cure Temp.	Pressure (psi)	Cure Time	Remarks
X273	H-H-1	200°F	Loose	None	200°F 300°F	800	1 hr	UPPER LIP DRY Very poor appearance
X273	H-H-2	200°F	Loose	None	200°F 300°F	1200	1 hr	Very poor appearance
X273	H-H-3	200°F	Loose	None	200°F 300°F	1600	1 hr	Very poor appearance
X274	J-H-1	350°F	Loose	1 hr	350°F 500°F	1600	2 hrs 10 hrs	DRY SPOT, WRINKLES Very poor appearance
X274	J-H-2	350°F	Loose	1 hr	350°F 500°F	1000	2 hrs 10 hrs	Very poor appearance
X274	J-H-3	350°F	Loose	1 hr	350°F 500°F	800	1 hr 10 hrs	Very poor appearance

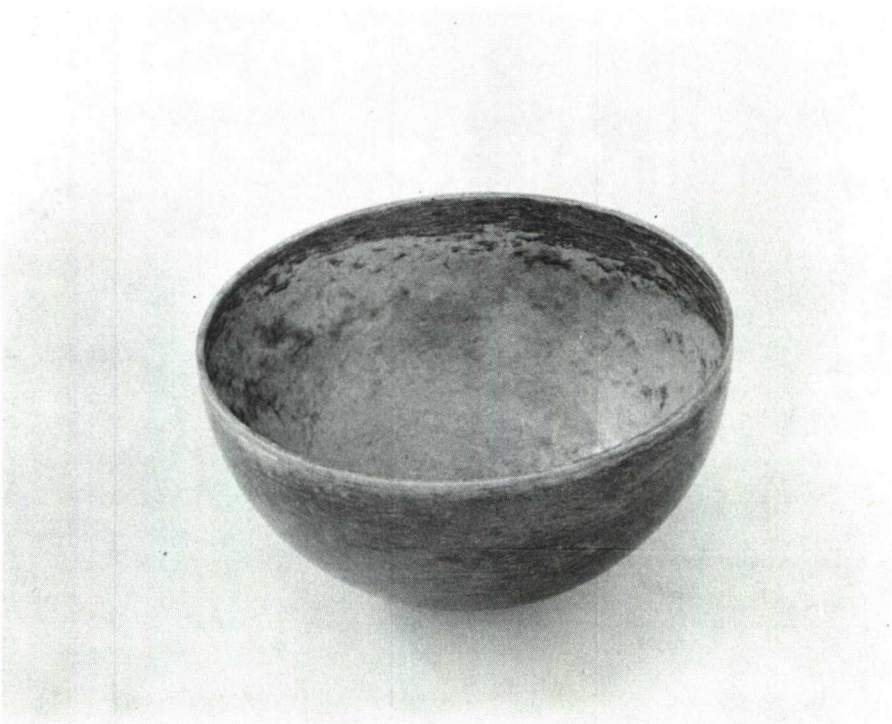


Figure 78 - An X270 Epoxy Hemisphere

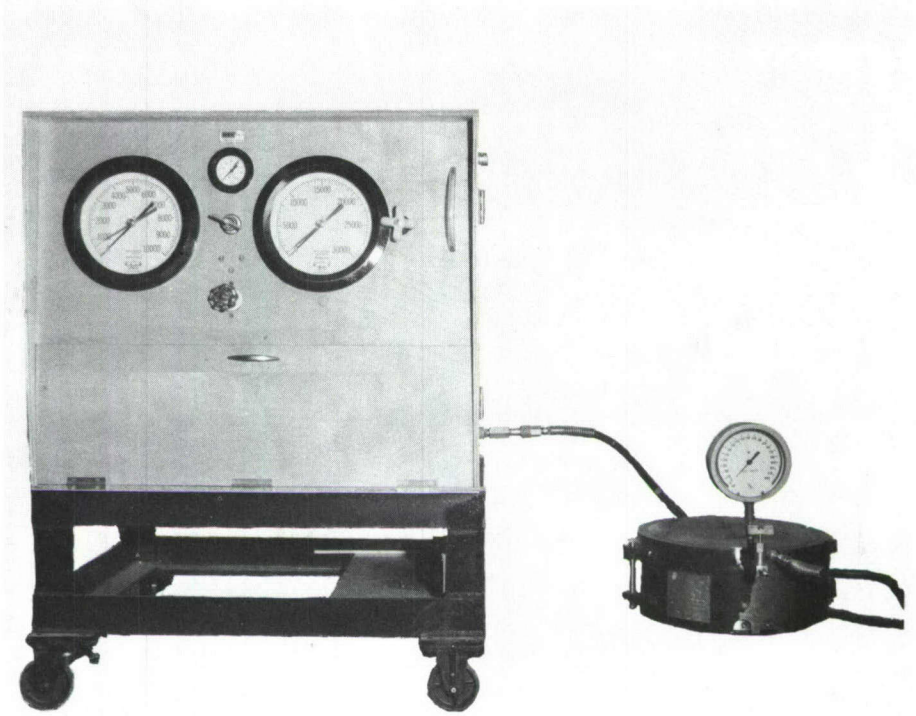


Figure 79 - The Pressure Chamber And Pump Mechanism Used To Test Hemisphere



Figure 80 - The Pressure Chamber Showing A Hemisphere In Place For Test

TABLE 37 THE RESULTS OF THE HYDROSTATIC PRESSURE TESTS OF THE HEMISPHERES

Molding Compound	Sample No.	Thickness at Failure	Ultimate Tensile Stress at Failure(psi)
	F-H-5	.131 inch	11,200
	F-H-8	.130 inch	15,800
	F-H-10	.130 inch	17,400
X270	F-H-11	.128 inch	10,900
Epoxy	F-H-12	.133 inch	20,200
	F-H-13	.128 inch	10,400
	F-H-14	.112 inch	12,500
	F-H-15	.125 inch	15,600
	Average	---	14,500
X273 Phenolic	1		7,650

NOTE: None of the X274 silicone molding was good enough to warrant testing.

6.4 A Discussion of The Molded Hemispheres

As was noted above, the hemisphere was the most successful of the shapes that were molded, yet poor flake alignment was a problem even with this shape. The poor alignment of the flake was characterized as usual by wrinkles. It was observed during the pressure tests that all failure occurred in the wrinkled areas and that lower strength was associated with the more badly wrinkled parts. It was also observed that the wrinkling could not be corrected by changing processing parameters. Thus, the hemispherical shape providesfurther evidence that flake-reinforced moldings cannot be prepared by sample compression molding techniques.

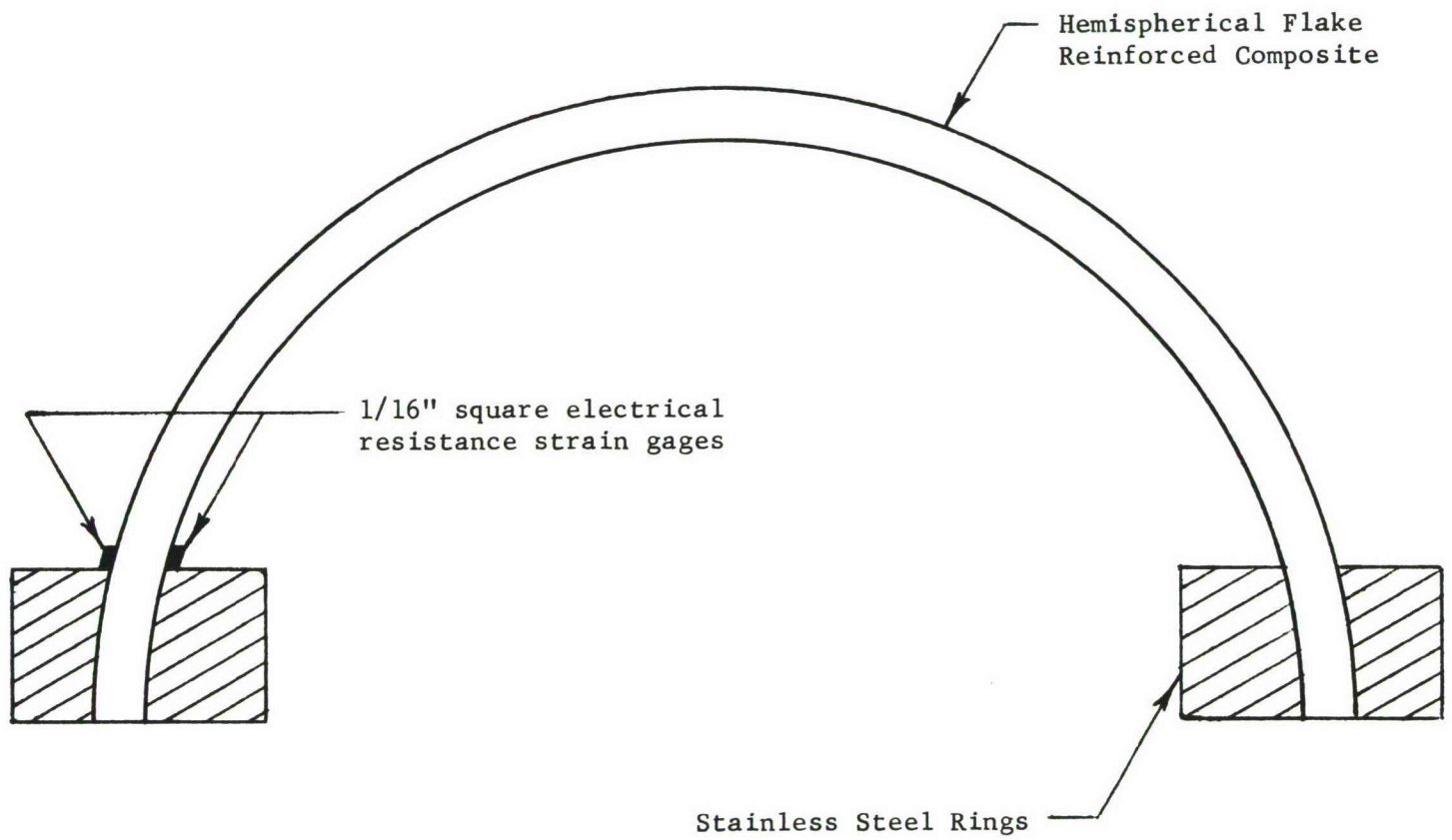


Figure 81 - A Diagram Showing The Stainless Steel Ring And Strain Gages Used During The Pressure Test Of The Hemispheres

7.0 MOLDING AND TESTING OF A FLAT LAMINATE WITH ABRUPT THICKNESS CHANGES

7.1 Scope

A 12" square, flat laminate, with thicknesses ranging from 1/16" to 1/2", was selected to evaluate the effect of abrupt thickness changes on the properties of flake reinforced molding. These laminates were evaluated by visual examination and by conventional mechanical tests.

7.2 Molding of Specimens

The molding with abrupt thickness changes had the configuration shown in Figure 82. Initial attempt to mold this shape was made by charging the mold with material that had not been precompacted. Results similar to those observed for the tapered plate were obtained; that is, extremely poor flake alignment and dry areas. In an attempt to correct this problem, carefully tailored preforms were used to change the mold. These preforms reduced the amount of flow during the molding process. It was assumed that this would produce more satisfactory moldings, as it did in the case of the tapered plate. In the case of molded laminates with abrupt thickness change, this technique did not reduce the amount of flow sufficiently and laminates similar to the one shown in Figure 83 resulted. Details of the processing of these moldings are presented in Table 38.

7.3 Test Procedures and Results

Flexures and compressive specimens were cut from the moldings with abrupt thickness changes as shown in Figure 84. These specimens were tested in the usual manner and the results of these tests are tabulated in Table 39.

7.4 A Discussion of The Molding of The Panel With Abrupt Thickness Changes

The plates with abrupt thickness changes could not be satisfactorily compression molded. The problem with this plate, as with the other shapes, was the wrinkles that characterized poor flake alignment. Processing changes did not correct this condition, which led to substantially reduced mechanical properties.

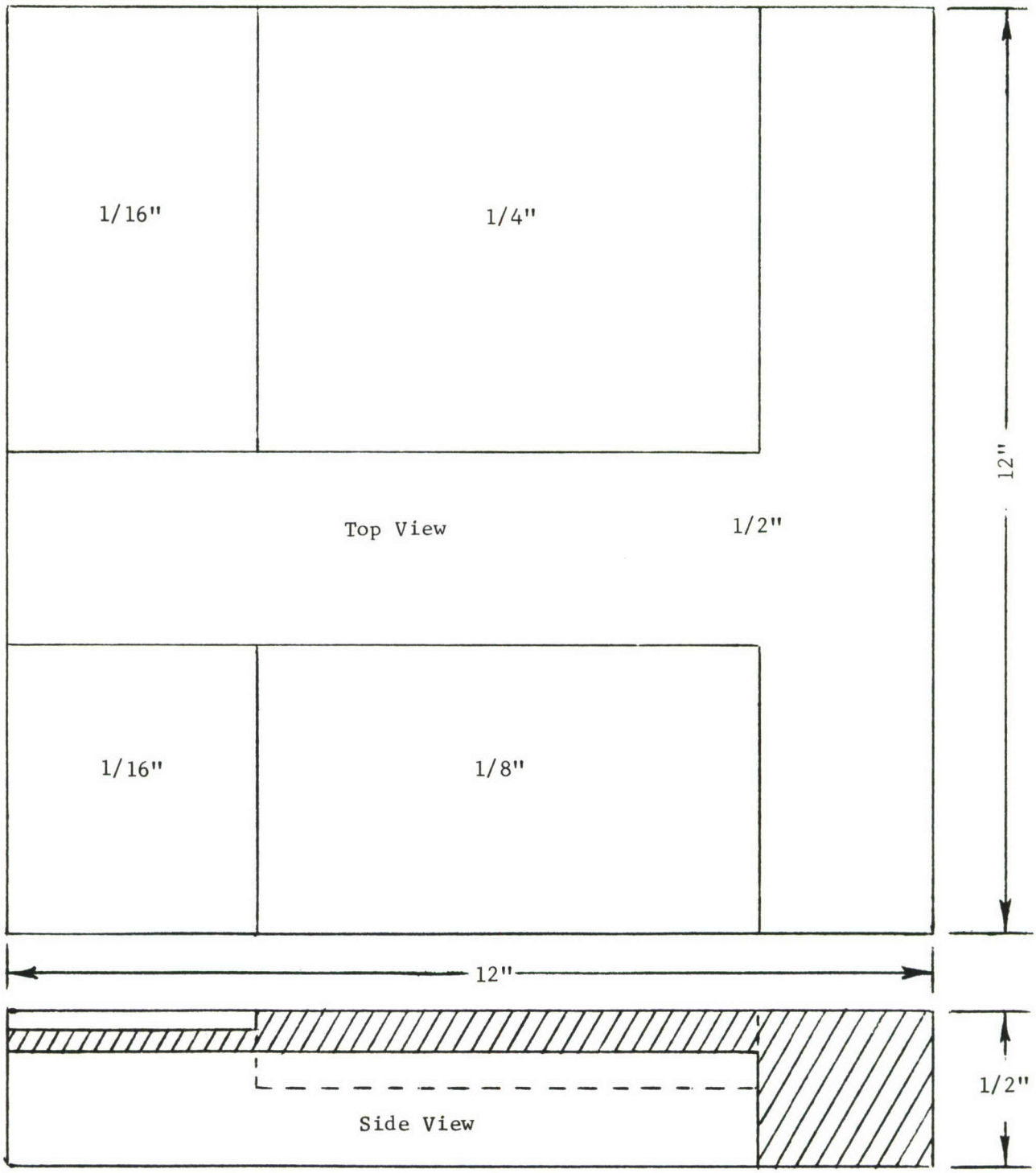


Figure 82 - A Molding With The Abrupt Thickness Changes

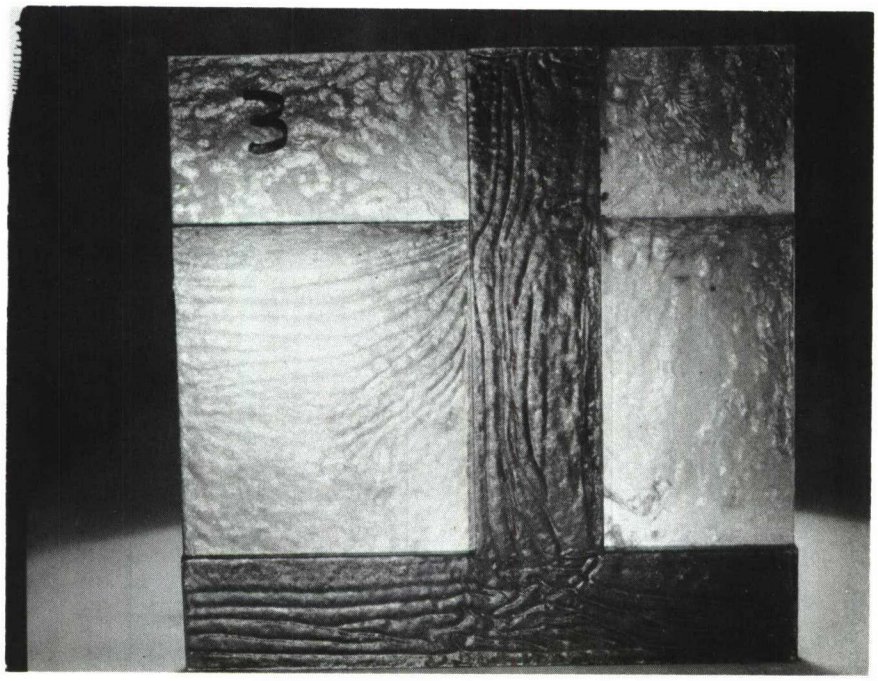


Figure 83 - Photograph Of A Plate With Abrupt Thickness Changes

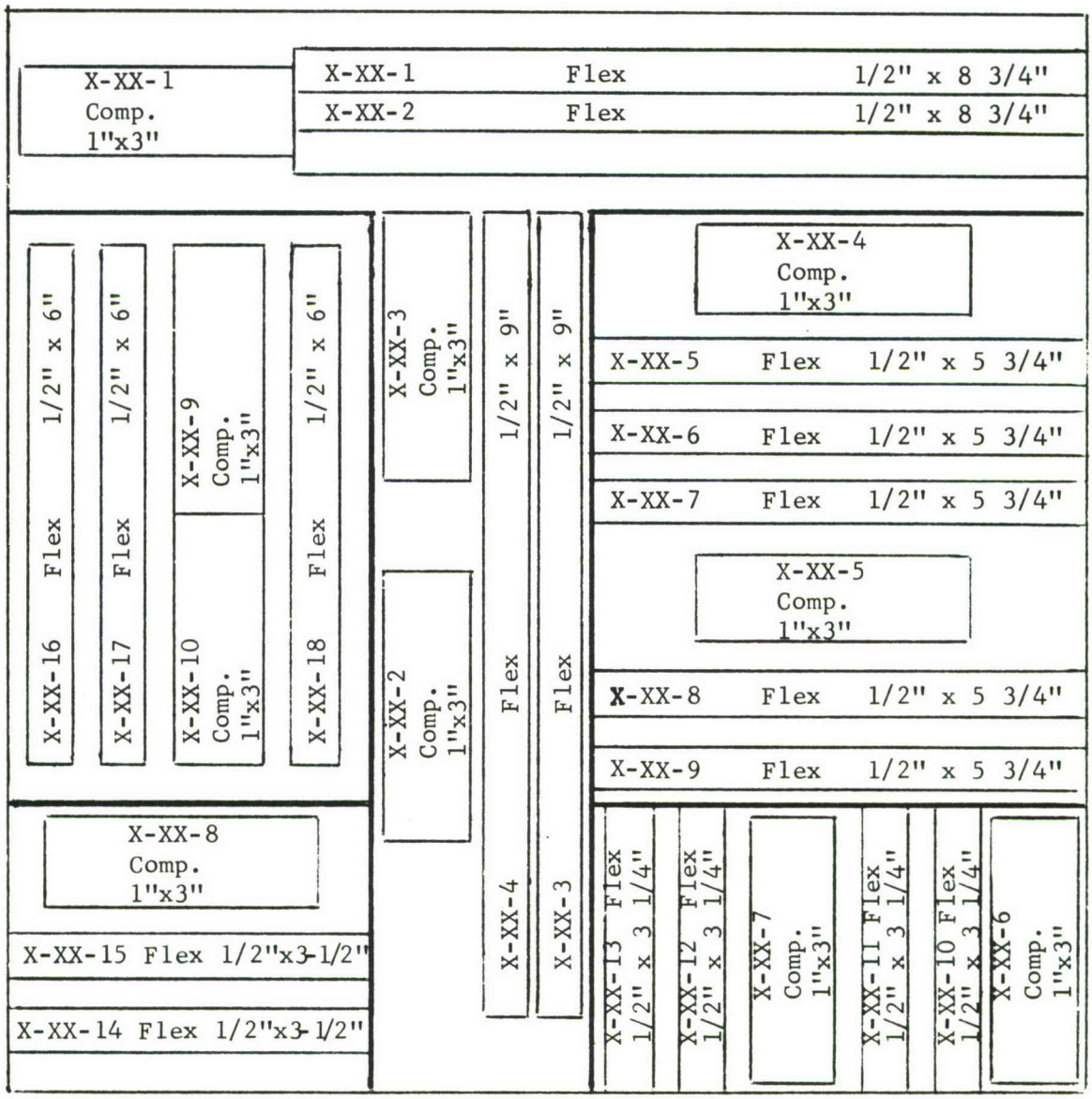


Figure 84 - Layout Of Test Specimens On The Molding
With Abrupt Thickness Changes

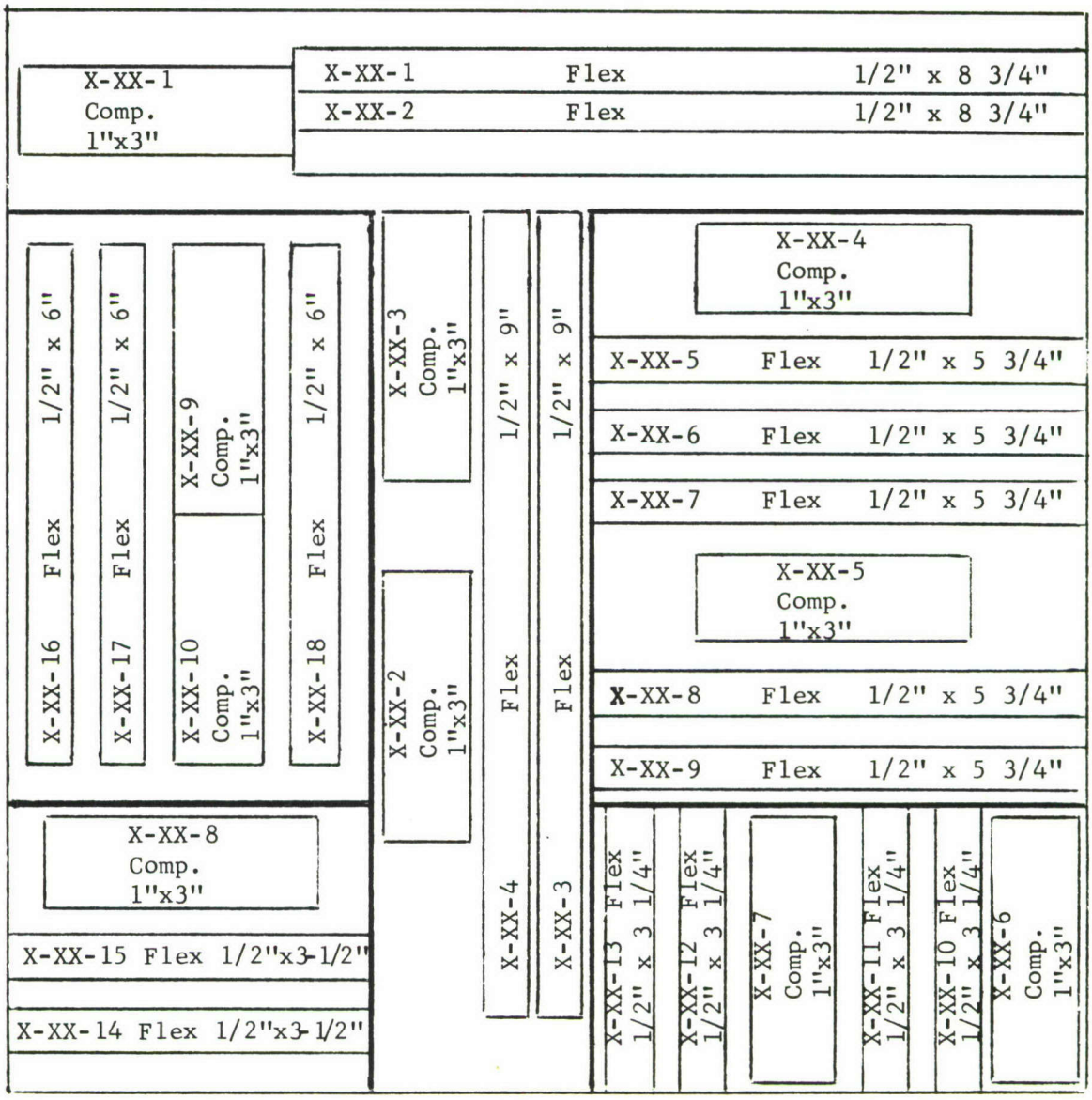


Figure 84 - Layout Of Test Specimens On The Molding
With Abrupt Thickness Changes

TABLE 38 MOLDING CONDITIONS FOR LAMINATES WITH ABRUPT THICKNESS CHANGES

MOLDING CONDITIONS

Molding Composition	Sample No.	Mold Temp.	Loading Condition	Contact Time	Cure Temp.	Pressure (psi)	Cure Time	Comments
X270	F-A-1	350°F	Loose	2 min	350°F	800	2 hrs	Numerous wrinkles Several dry spots
X270	F-A-2	350°F	Loose	2 min	350°F	800	2 hrs	Numerous wrinkles Several dry areas
X270	F-A-3	350°F	Preforms	2 min	350°F	800	2 hrs	Numerous wrinkles Several dry areas
X270	F-A-4	350°F	Preforms	2 min	350°F	800	2 hrs	Numerous wrinkles Several dry areas
X273	H-A-1	250°F	Loose	None	250°F 300°F 400°F	800	1 hr 1 hr 1 hr	Numerous wrinkles Several blown areas
X273	H-A-2	250°F	Preforms	None	250°F 300°F 400°F	800	1 hr 1 hr 1 hr	Numerous wrinkles at all thickness changes
X273	H-A-3	250°F	Preforms	None	250°F 300°F 400°F	800	1 hr 1 hr 1 hr	Numerous wrinkles at thickness changes
X274	J-A-1	350°F	Loose	1 hr	350°F 500°F	1000	1 hr 8 hrs	Numerous wrinkles at thickness changes cloudy appearance
X274	J-A-2	350°F	Loose	1 hr	350°F 500°F	1000	1 hr 8 hrs	Numerous wrinkles at thickness changes cloudy appearanced

TABLE 39 MECHANICAL PROPERTIES OF SPECIMENS CUT FROM VARIOUS PORTIONS OF THE MOLDINGS WITH ABRUPT THICKNESS CHANGES

Molding Composition	Nominal Thickness of Laminate Areas From Which Specimens Were Cut (Inches)	Ult. Flexural Strength (psi)	Flexural Modulus (psi x 10^{-6})	Ultimate Compressive Strength (psi)	Compression Modulus (psi x 10^{-6})
X270	1/16	19,800	5.11	19,500	4.76
	1/8	27,000	4.78	22,600	4.70
	1/4	25,200	5.43	35,700	4.94
	1/2	20,300	5.08	32,000	5.15
	Average	23,075	5.10	27,450	4.89
X273	1/16	21,400	4.65	23,100	4.16
	1/8	19,300	4.17	25,000	3.80
	1/4	19,200	4.72	28,100	4.75
	1/2	12,400	3.92	24,400	3.62
	Average	18,075	4.37	25,150	4.08
X274	1/16	7,100	4.00	6,600	3.76
	1/8	16,500	3.33	6,600	3.07
	1/4	15,400	4.69	14,000	3.72
	1/2	7,900	3.58	8,000	2.76
	Average	11,725	3.90	8,800	3.33

SECTION VI CONCLUSIONS

The goals of this program were:

1. To determine the properties of compression molded, glass flake reinforced resin composites in the form of flat laminates from which conventional test specimens could be prepared.

2. To determine the effect of processing and composition changes on these properties and to select an optimized set of condition for molding these composites.

3. To mold simple geometric shapes using these conditions and to compare the properties of these moldings to the properties obtained for the flat laminates.

The first two goals were satisfactorily met; however, the molding of geometric shapes was not entirely successful. This goal was not met because of a problem of poor alignment of the glass flake reinforcement in the moldings. The problem was apparently caused by flow or movement of the reinforcement during the molding process, and resulted in greatly reduced mechanical properties. Changes in processing techniques did not correct this problem and it appears that this phenomena is inherently related to the amount of the flow necessary to fill a shaped mold. Thus, the use of glass flakes as reinforcement in compression molded articles will be limited to very simple shapes, such as flat panels. Additional work is required to develop a different molding concept before the properties of flake reinforced composites can be exploited.