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RIGIDIZATION TECHNIQUES FOR
INTEGRALLY WOVEN COMPOSITE CONSTRUCTIONS

TECHNICAL DOCUMENTARY REPORT NO. ML-TDR-64-299

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AF Materials Laboratory
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Hughes Aircraft Company, Culver City, California;
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FOREWORD

This report was prepared by Hughes Aircraft Company under USAF Contract No. AF 33(657)-11287, Project No. 7381 Task No. 738101, "Rigidization, Techniques for Integrally Woven Composite Constructions." The work was administered under the direction of the Materials Engineering Branch, Materials Applications Division, AF Materials Laboratory, Research and Technology Division. Mr. Albert Olevitch acted as Project Engineer.

This report covers the work done from July 1963 to July 1964.

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ABSTRACT

This report describes the investigation of methods to erect and rigidize three-dimensional woven fiberglass sandwich under space conditions; and to determine the physical properties of the rigidized sandwich material. The investigation included both triangular and rectangular fluted woven sandwich in weights from 12 to 23 ounce per square yard. Impregnating or rigidizing materials and processes included gelatin rigidized by solvent loss, gas catalyzed polyurethane, ultraviolet activated polyesters, and heat activated single component polyurethane foam powder.

Methods for impregnating, storing, and automatically erecting and rigidizing the sandwich materials were developed successfully for all of the chemical rigidizing systems. Density of the space rigidized materials varied from 2 to 8 pounds per cubic foot depending on system, weight of cloth, and resin content. Strengths obtained were of the same order of magnitude as other conventional lightweight structural materials.

This technical documentary report has been reviewed and is approved.

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INTRODUCTION

Expandable space structures are usually conceived as structures having an initially small volume prior to launch, so that the ultimate in compact packaging may be achieved. After deployment in space, these structures should be easily erected to full size, and should remain in the initially erected configuration regardless of loss of internal pressure, maneuvering loads, etc. In the case of nonmetallic expandables these are usually impregnated fabric structures erected by inflation. Rigidization then takes place by either a chemical reaction or a physical change in the fabric impregnant.

In this program the fabric material utilized was a three-dimensional fabric in which core flutes and surface facings are integrally woven. Such a structure results in a sandwich laminate with exceptional core-to-facing peel strength. This very flexible fabric structure was then impregnated with a resin which remains flexible until later rigidized under simulated space conditions.

The objectives of this investigation were:

1. To develop methods for erecting the three dimensional structure from the initially compacted state.
2. To compare several different chemical rigidization techniques from the standpoint of weight, shelf life, flexibility, ease of usage, etc.
3. To determine the physical properties of the rigidized panels so that comparisons of the various systems could be made, and to provide preliminary design data.

In order to obtain a good spread of data several weights of fabrics were employed, in two different configurations; one utilizing triangular core flutes and the other with rectangular core flutes. At the same time several rigidization techniques were investigated. These included ultraviolet activated polyester resins, a water vapor catalyzed polyurethane resin, a solvent release system employing a gelatin-water solution and a heat activated, one component, polyurethane foam powder.

SUMMARY

Several different weights of woven fiberglass sandwich, with both triangular and rectangular flutes, were investigated. Two lightweight (12-14 oz/yd²) and two medium weight (21-23 oz/yd²) fabrics were selected for further evaluation. All of the fabrics were 5/8-inch thick in the expanded condition and were in the "grey goods" condition.

Erection techniques for use in a vacuum were developed based upon the expansion of thin polyethylene tubing within the flutes of the cloth. The pressurizing media, included residual air, small amounts of liquid of known vapor pressure, and volatile catalysts.

Successful erection and rigidization of the sandwich cloth under simulated space conditions was obtained with the following impregnants: aqueous gelatin solutions, various polyesters catalyzed for ultraviolet light cure, polyurethane resin specially formulated for water vapor catalysis, and a heat activated single component polyurethane foam powder. Procedures for stabilizing, impregnating, storing, handling, and deploying the impregnated cloths were developed on a laboratory scale. The control of resin content and the uniformity of impregnation within the cloth was investigated.

Panels were made with all of the rigidizing systems under simulated space conditions. Most of the work was done with the lightweight cloths. Specimens from these panels were tested for resin content, apparent density, edgewise compressive strength, flatwise compression strength, and flexural strength. The apparent density of these materials was of the order of 4-8 pounds per cubic foot, and the strengths obtained were similar to the strengths of other lightweight structural materials formed under terrestrial condition.

EXPERIMENTAL

FABRIC SELECTION

The fabrics used in the program were three dimensionally woven fabrics in which the core flutes were integrally woven into the facing sheets resulting in a material with exceptionally good resistance to peeling loads. This material was obtained from the manufacturer, The Raymond Development Company, in two configurations; rectangular core in which the flutes are vertical to the faces, and triangular core in which the flutes are approximately 60 degrees to the face sheets as shown in Figure 1. The method of obtaining the interlocking construction is illustrated in Figure 2.

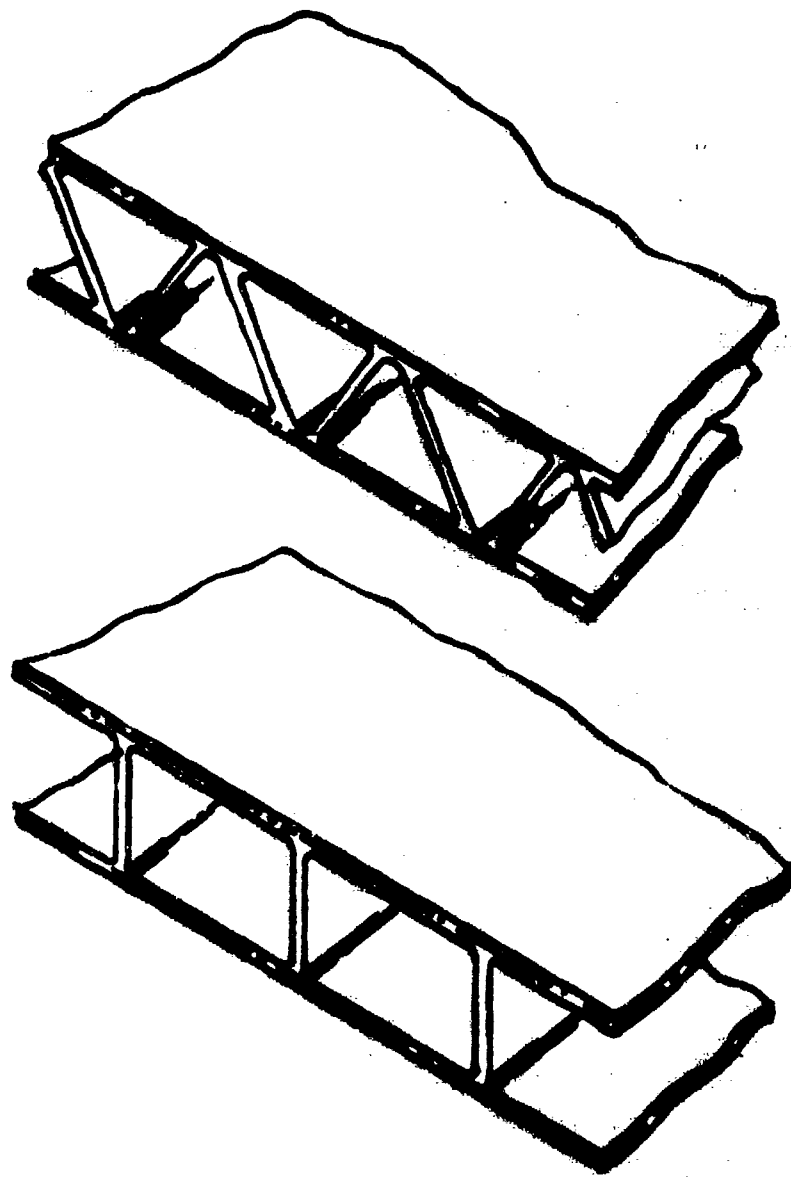


Figure 1. Triangular and rectangular construction.

RAYMOND DEVELOPMENT INDUSTRIES, INC.
INTEGRALLY-WOVEN CORE FIBERGLASS MATERIAL

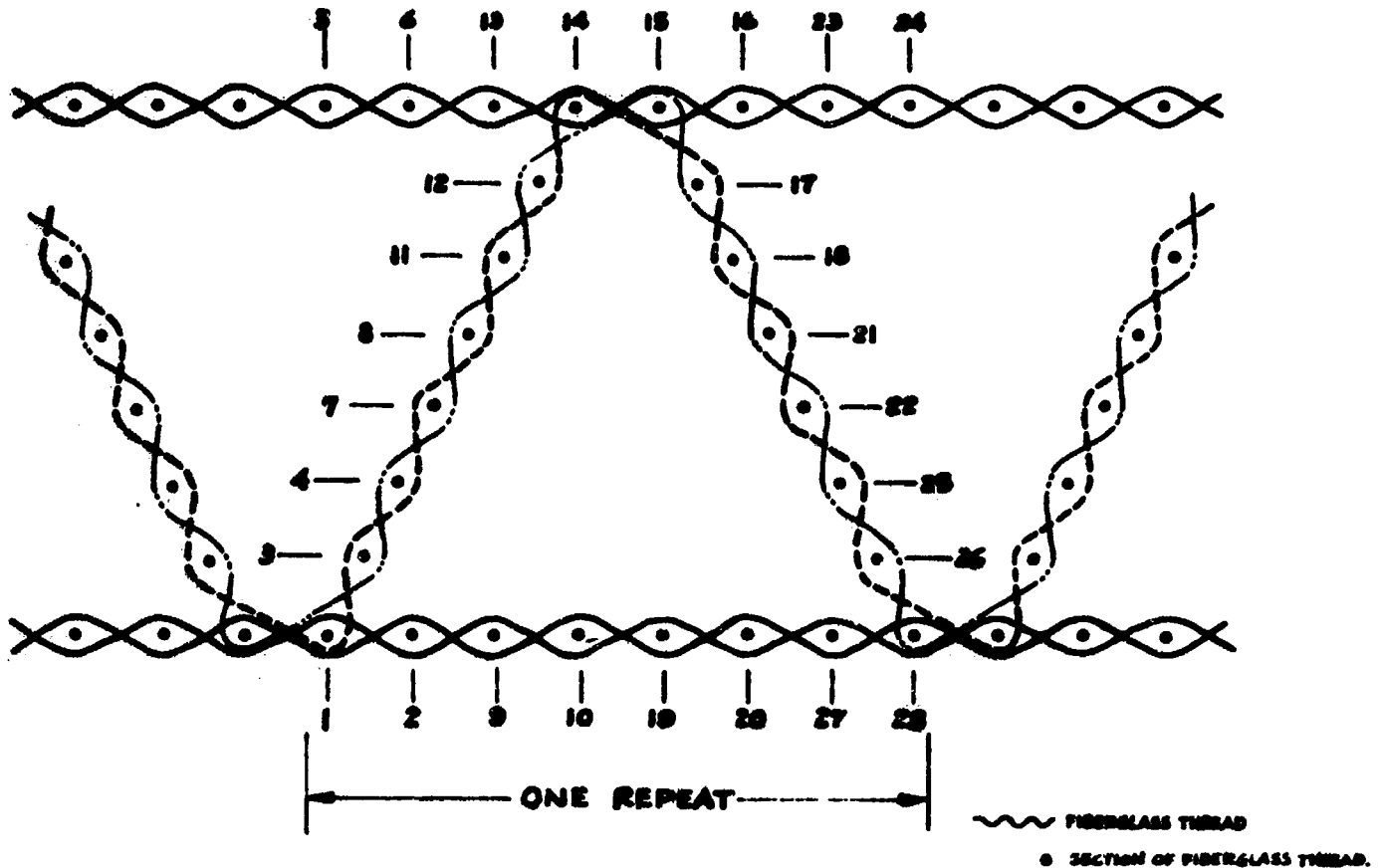


Figure 2. Illustrating the interlocking construction of the triangular material.

Since the loads which would be encountered in any spacecraft would probably be quite moderate, it was decided that very lightweight fabrics could be used. Two stock Raymond Development designs were used; a

TABLE 1. FABRIC CHARACTERISTICS

Fabric Type	Weight, oz/yd ²	Web Thickness, in. (av)	Facing Thickness in. (av)
Rectangular	14	0.0085	0.0060
Rectangular	23	0.0112	0.0075
Triangular	12	0.0038	0.0036
Triangular	21	0.0065	0.0068

triangular fluted fabric and a rectangular fluted fabric each with 5/8 inch spacing between the facing sheets. Initially the lightest weight fabrics which Raymond Development Company could make were ordered and were used to obtain the major portion of the data. Later in the program heavier fabrics were also ordered to obtain additional data. All the fabrics were procured in the "grey goods" condition and no surface treatment was used. The fabrics used, and the varying physical characteristics are shown in Table 1.

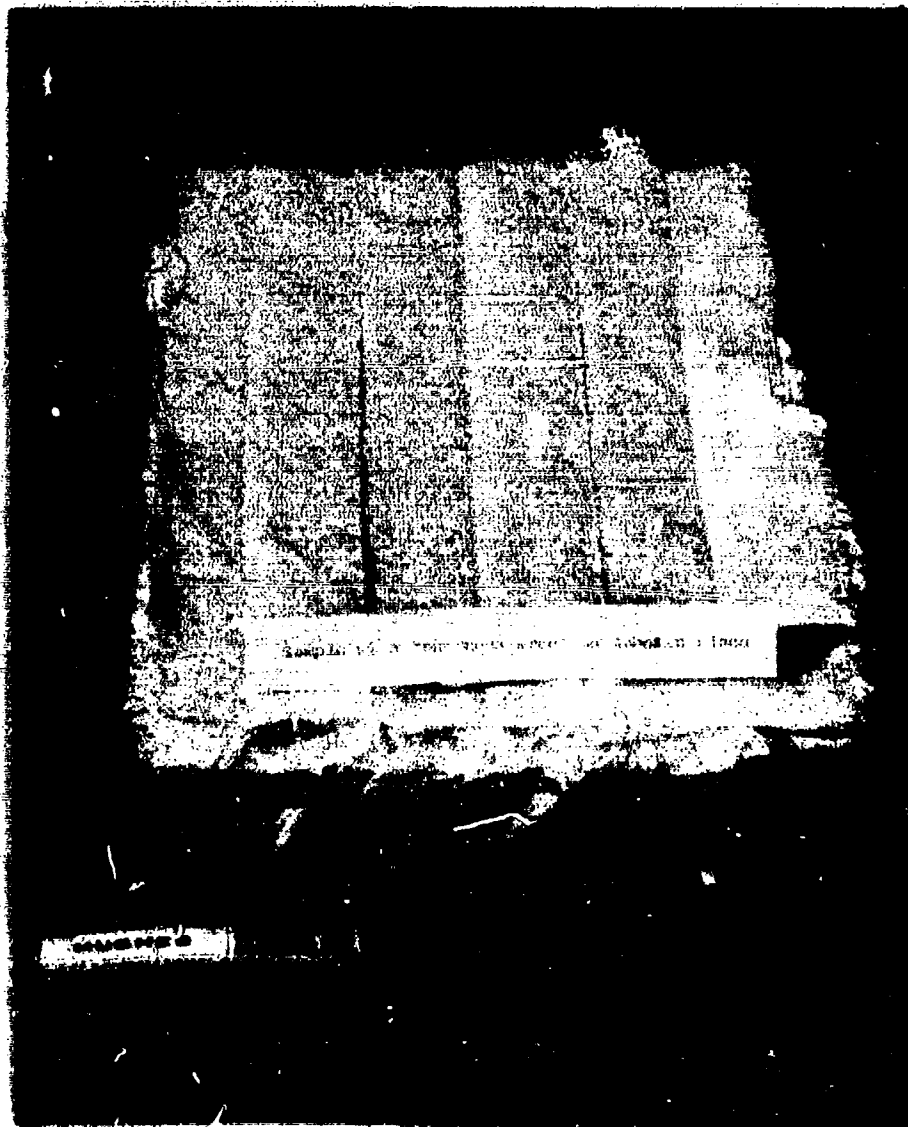
ERECTION TECHNIQUES

One of the objects of the development of the various rigidization systems was to obtain a system or systems which could be used to cause erection of the three dimensional fabric. It was desired that the erection and/or expansion system be one that preferably would work automatically, or at best require a simple activation technique.

The fundamental concept for erection of the three-dimensional structures utilized thin plastic tubes which were inserted into the flutes of the laminate. On pressurization of the tubes the structure is erected. The tube material selected for use was commercially available 2 mil extruded polyethylene, procured as 1 inch and 1-1/2 inch lay-flat tubing in continuous rolls. The 1-inch tubing weighed 0.085 ounce per yard and the 1-1/2-inch material was 0.13 ounces per yard. The 5/8 inch triangular fabric required 72 one-inch tubes per yard which weighed 6.2 ounces per square yard. The 1 x 5/8 inch rectangular fabric required 36 one and half inch tubes per yard which weighed 4.5 ounces per square yard.

Three methods of using the tubes were investigated. In one, shown in Figure 3, a continuous tube was run through all the flutes in the sample and was pressurized from one end with the other end closed. This type of construction was found efficient in operation and fairly simple to use since only one air connection was required. It, however, is not too well suited for use in fabricating a large structure, since in drawing long lengths of tubing through the flutes the chance of damage to the tube increases with the tube length. The obvious advantages of this method are the equal pressurizations of all the flutes, and the possibilities of obtaining any desired pressure in the tubes. In service, this system would then require a separate gas source together with valving and regulating equipment.

A second method of erecting the tubes, somewhat similar to the first method, utilized separate tubes in each flute. In this case all the tubes are sealed at one end, and the other end of each is connected to a manifold. This system also erected the structure as efficiently as the continuous tube method, and has the obvious advantage that if a leak develops in one tube, that tube can be replaced without affecting the other tubes. However, this system, in addition to the requirements of a gas pressurization source, a valve and regulator, would require the additional



**Figure 3. Sample with continuous erection tube in place.
(S39251)**

weight of a manifold for distribution of the gas to the tubes, plus the large number of connections required for the tubes thereby increasing the complexity of the system.

The third erection technique investigated consisted of installing completely sealed tubes in each flute. In use, when the structure was placed in a vacuum, the residual air in the tubes expanded and caused the structure to erect. The tubes were sealed by simply knotting the ends and then pretesting by being placed in a vacuum chamber to be sure of complete expansion from residual air. No special precautions were taken to insure residual air in the tubes and in almost all cases, when tested in the above manner the tubes expanded to full size. This system, therefore, appeared to be the most preferable since it was automatic, did not require special equipment, or additional weight, and the additional volume of entrapped air appeared to be negligible. Figure 4 illustrates such an "auto erection" system.

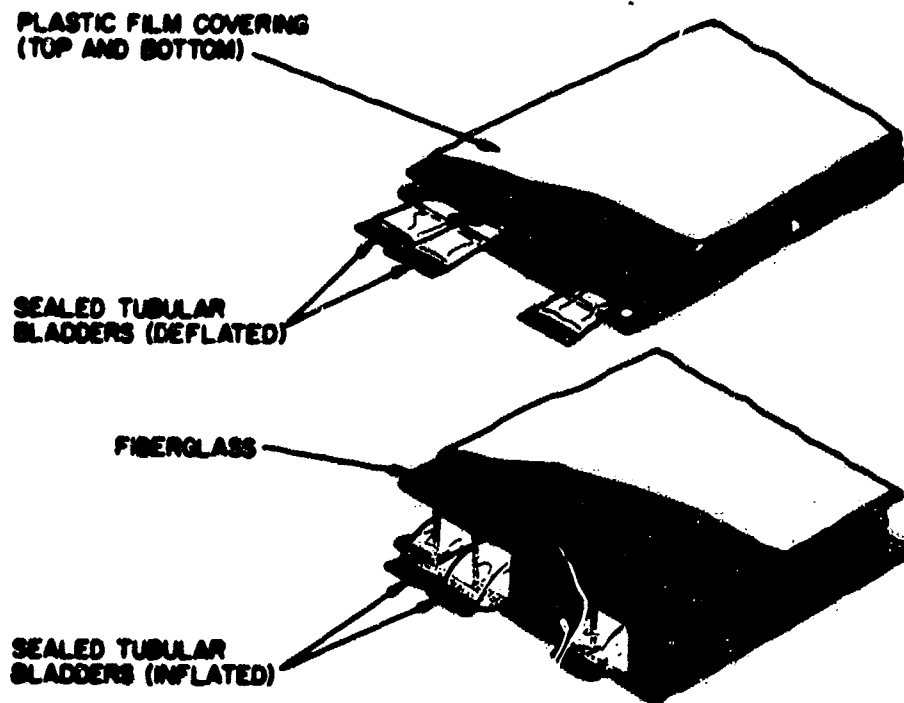


Figure 4. Illustrating the "auto-erection" system using polyethylene tubes containing small amounts of alcohol. (R95590)

The initial tests made with the sealed tubes consisted of simply cutting the tubes to length and then sealing each end. The residual air in the tube caused good inflation when the tube itself was placed in the vacuum environment. However, after a number of tubes had been tested in fabric assemblies, it was found that the amount of erection varied considerably between tubes. This indicated that the residual air in the tube was marginal and could not be relied upon to result in uniform pressurization when the fabric "load" was placed on the tubes. Tests were therefore made to develop a uniform pressure system.

The first pressure tube tests were made by inserting in each tube a small piece of urethane foam. The material used was an open celled, flexible foam of 2 lb/ft³ density. The sample was approximately 1" x 1" x 1/8" and was slipped into one end of an otherwise completely flattened 10 to 12-inch long, 1-inch diameter polyethylene tube. The air in the foam, plus the air in the space required for the foam, then caused each tube to inflate satisfactorily. However, since these foam inserted tubes were somewhat more bulky than was believed desirable, investigations were also made into the use of liquids in each tube. It was desired to use liquids whose vapor pressure at room temperature would be enough to cause good tube inflation, but not enough to cause overinflation. A number of common solvents having vapor pressures, at room temperature from 25 mm to approximately 150 mm of Hg were tried. These included water, ethyl and methyl alcohol, benzene, toluene, and carbon tetrachloride. Ethyl alcohol, with a vapor pressure of approximately 50 mm Hg at room temperature was found to be very satisfactory. In use

approximately one gram of alcohol was placed in each 10-12 inch flat tube prior to sealing. When the tube and fabric assembly was placed in the vacuum atmosphere good inflation was obtained, with sufficient pressure to firmly expand the impregnated flutes. At the same time the pressure was automatically regulated. Tests with such tubes indicated that the 2 mil wall thickness tube would not lose all the alcohol for at least 48 hours, in a vacuum, which was judged sufficient for all rigidization systems. This vapor pressure expansion technique was then used for all subsequent testing, which was conducted at chamber pressures varying from 0.1 mm to 1.0 mm Hg.

There was no attempt made to prepare samples at very high vacuums (10^{-6} to 10^{-9} mm Hg) since it is virtually impossible to duplicate the infinite vacuum capacity which would occur in rigidizing those materials in space. Experiments must be conducted in fixed volume space chambers with pumps of limited capacity. These setups cannot maintain a hard vacuum (of the order of 10^{-9} mm Hg) while the material in test is generating large volumes of gases. However, the rate of vaporization is a function of the difference in vapor pressure of the liquid and the surrounding atmosphere at any given temperature. In all cases the chamber pressure was below the vapor pressure of the liquid in the rigidizing material. Therefore it makes little difference in rate of evaporation if the surroundings are at 0.01 mm of Hg or 10^{-9} mm of Hg if the temperature is relatively high so the fluid is above its boiling temperature at that pressure. Therefore the initial rate of rigidization or drying will be primarily a function of the temperature of the surface in space, and this temperature in turn will be a function the heat input to the surface by thermal absorption and conduction from the underlying mass. In later stages of rigidizing the rate of diffusion of the water through the gelatin colloid will also be primary factor.

In the initial tests of any of the systems, the fabric samples were erected by simply pressurizing the tubes within a section of the fabric. The rigidized panels were ridged on both outer surfaces, since the tubes tended to form circular cross-sections in each cell. To counteract this, a fixture was constructed which could hold one or both surfaces of the panels under spring tension during rigidization. Samples made in such a fixture had relatively flat surfaces more closely simulating rigidization of an inflated structure. Figure 5 shows a sample of this jig with the manifold erection tubes in place. Figure 6 shows a typical rectangular sample erected and rigidized in vacuum using this fixture. Figure 7 shows a typical triangular structure.

RIGIDIZATION TECHNIQUES

The purpose of this project was to determine the differences in several existant space rigidization techniques, rather than attempt to develop any new rigidization methods. The choice of the optimum rigidization technique could then be made after preparation of similar three dimensional samples by each system. The various systems selected for test each represented a different chemical technique for rigidization and included the following:

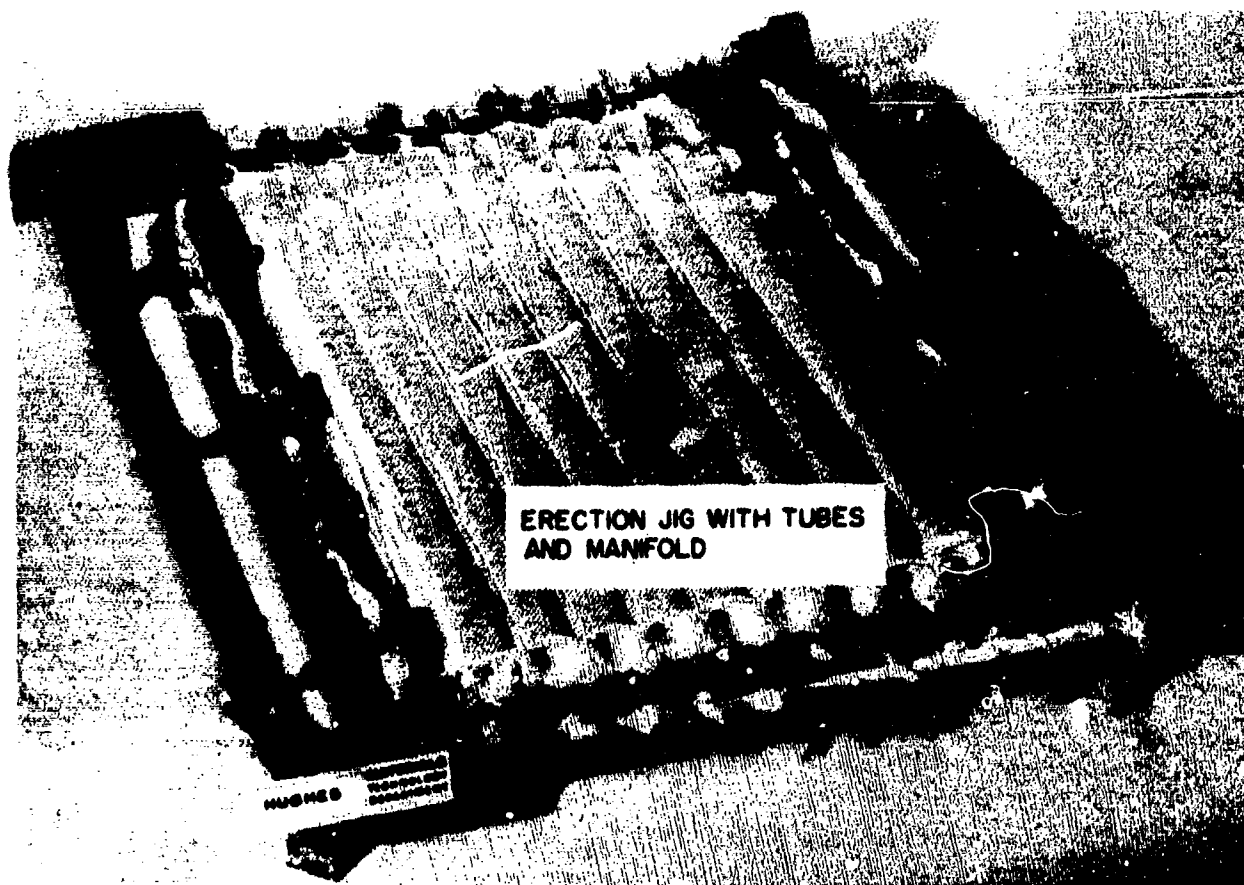


Figure 5. Continuous tension erection fixture. (R95578)

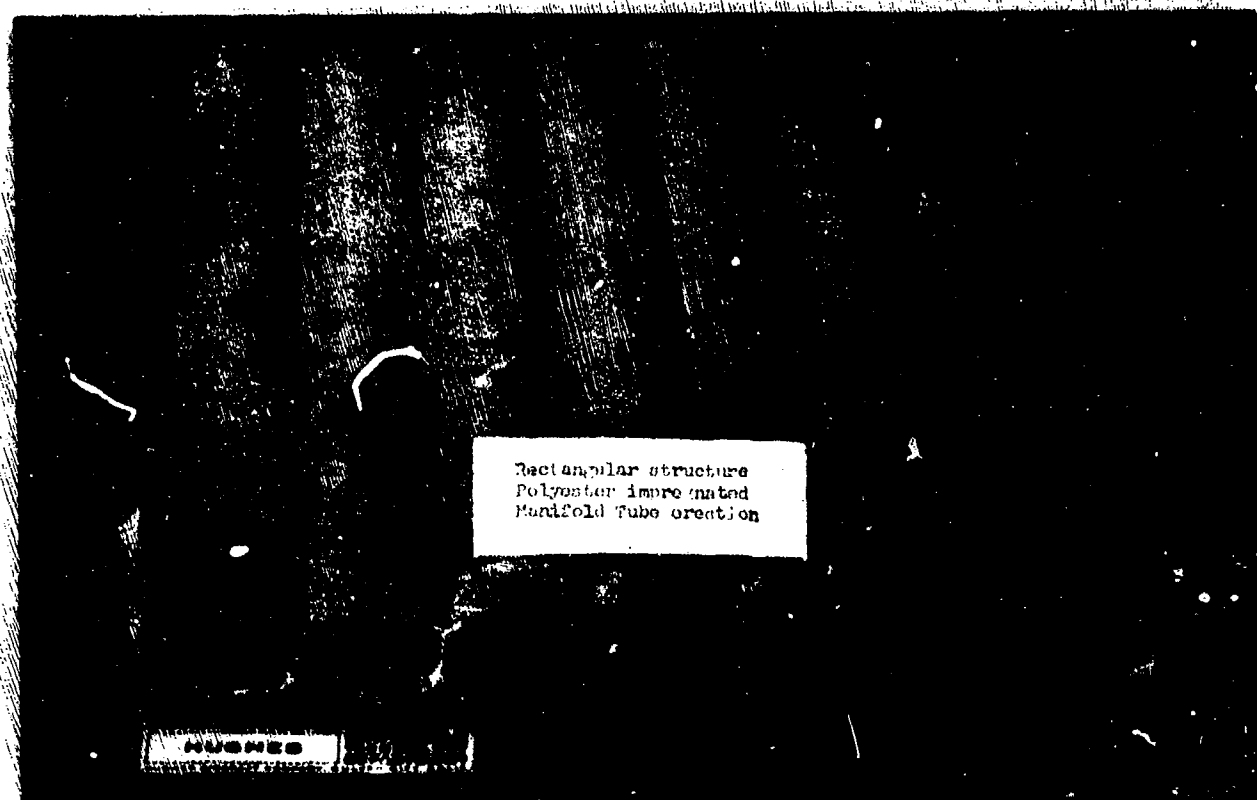


Figure 6. Typical rectangular structure erected and rigidized in the vacuum environment. (S39250)

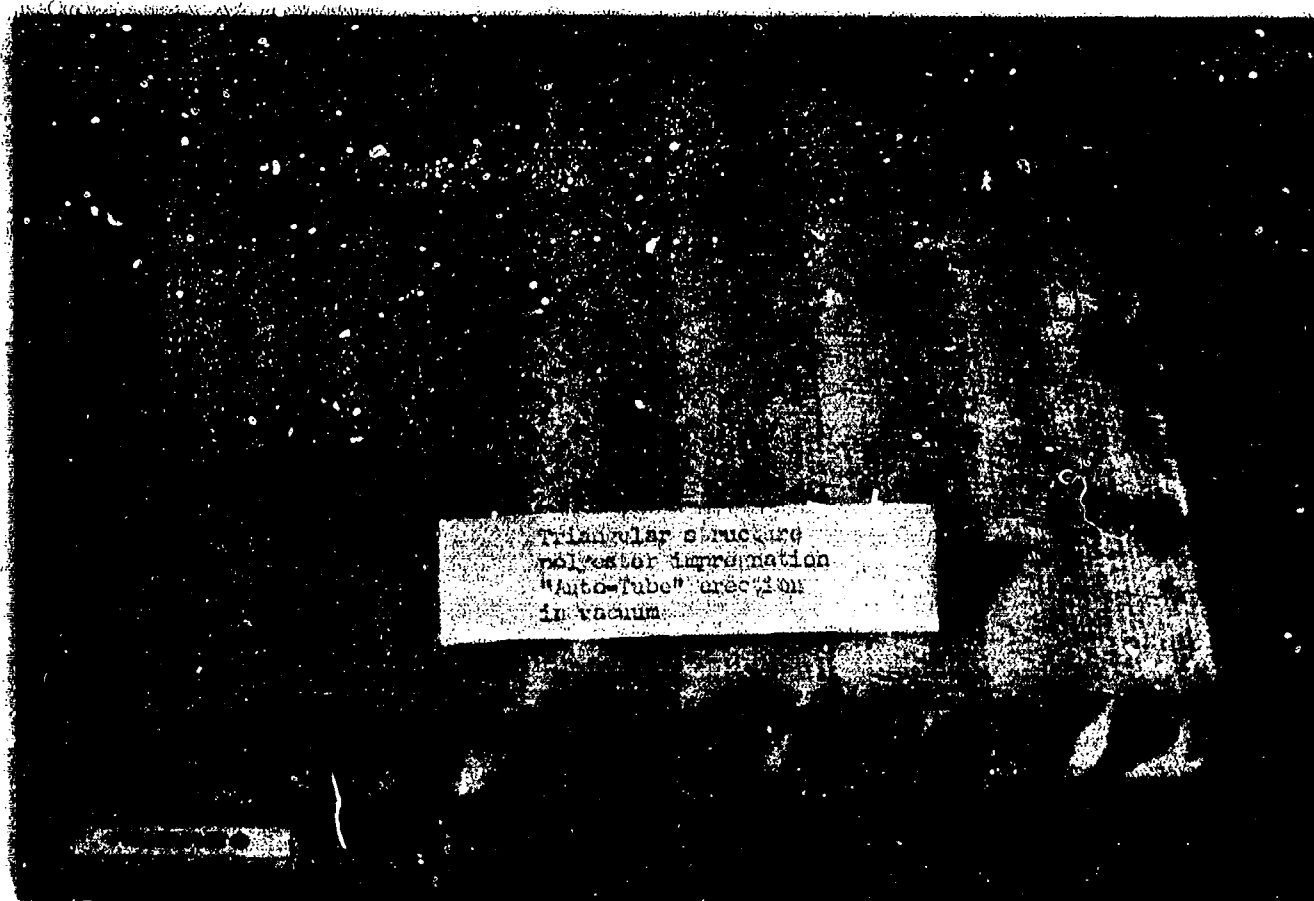


Figure 7. Typical triangular structure erected and rigidized in the vacuum. (S39249)

1. A gelatin solution rigidized by solvent release in the vacuum.
2. A polyester resin rigidized by ultraviolet activation.
3. A polyurethane resin rigidized by absorption of water vapor.
4. A polyurethane foam powder rigidized by infrared radiation.

In each case (except the foam powder) the resin solution was impregnated into the fabric, which already had the polyethylene tubes inserted into the flutes. The samples were then rigidized by whatever activation was required. (The technique used for the foam powder is described below.)

Very little or no development work was done on the rigidization materials as such. However, it was found necessary that each system be tested individually to determine the optimum technique for impregnation and rigidization of each resin.

Gelatin — Solvent Vapor Release

The use of gelatin as a rigidization media represented essentially a new method of preparing fiberglass reinforced structures. Consequently

the greatest portion of the effort was devoted to determination of techniques to use this material.

This system, which was proposed by the Materials Applications Division of the Air Force Materials Laboratory, utilizes commercial gelatin dissolved in water as the impregnating medium. In this system, it was proposed that rigidization would take place simply by loss of water when the material was exposed to the space environment. Such a system has a number of obvious advantages; low cost, reliability, ease of preparation and, in space, no possibility of moisture damage. In addition to the above, recent investigations (Reference 1) have indicated this material to be well suited for space applications because of good resistance to the deteriorating effects of ultraviolet and gamma rays, etc. in space.

In order to utilize gelatin most effectively as a fabric impregnant a number of factors had to be determined. These include methods of preparing the gelatin solution, determination of solution concentrations, optimum wetting agent concentrations, fungicide incorporation, etc. Of even greater importance than learning to make the proper solutions is the determination of optimum techniques for using the material. Factors such as proper impregnation techniques had to be learned, resin contents for optimum physicals must be determined, storage techniques must be developed, etc. The determination of all these factors then was vitally necessary before preparation of physical property samples could be commenced.

Solution Preparation

Several methods of solution preparation were tried. The first method was a slow addition of a fine gelatin powder to hot (160-180°F) water with constant slow stirring. By this method it was possible to prepare a 20 percent solution (200 gm gelatin in 800 ml water) in approximately 2 hours. However, such a mixture had the disadvantage of excessive foam build-up. Even allowing the solution to stand for several hours with no stirring did not rid the mixture of all the foam. Antifoaming agents, Dow-Corning DC-200 fluid and Antifoam Emulsion A, diluted 1:3 with water and then added in the ratio of 50 mg per 1000 ml of solution appeared to give the best results.

Initial impregnation tests with such a solution indicated that the surface tension was high enough to materially affect the wetting time of the glass samples. This would be of little consequence with laboratory samples, since sufficient time could always be used to insure complete wetting. However, if the process were to be successful on large parts, some means would have to be devised to impregnate at reasonable rates on such surfaces. Therefore, a small amount of a wetting agent was added to the solution and a distinct improvement was noted.

A series of tests using 2-inch diameter glass fabric discs, in a modified Draves-Clarkson test*, was initiated to determine the optimum wetting agent concentration. In this test the fabric sample is gently placed on top of the solution, and the time required for the sample to become thoroughly wetted and sink is then recorded. By the use of a wetting agent the impregnation time can be reduced to approximately 55 percent of the nonwetted time, and a more thorough impregnation is assured. The wetting agents tested included Rohm and Haas Triton X-100, American Cyanamide Aerosol OT (75%), FMC Corporation Kronitex AA and KP-140 and Central Scientific Co. Cenco wetting agent. A review of the literature indicated that these were leading nonionic surface active agents, specially formulated to increase aqueous wetting properties. The results of the initial tests with the 2-inch glass fabric discs indicate that approximately 0.1 - 0.2 percent of Triton X-100 gave the best results. Further tests made using the three-dimensional woven cloth confirmed these findings. Later information from Swift and Company, indicated that Rohm and Haas CF-21 wetting agent was a preferred wetting agent.

In running the first impregnation tests using the gelatin solution it was found that samples stored wet, within a few days showed signs of microbiological attack. This was not unexpected since gelatin is considered a good nutrient. No attempts were made to identify the organisms. Two fungicides, phenol and Dow Chemical Company Dowcide G were tried in concentrations of 0.5 and 1 percent each. The 0.5 percent concentration of either material was found to give excellent inhibition of growth in samples stored for several weeks in the same container with contaminated samples. Further information from Swift and Company indicated that 0.1 percent by weight of the solution using Dowcide G would be sufficient fungicide.

A considerably better method for the solution preparation was later developed, using recommendations of Swift and Company. In evolution of this method it was found that an approximately 33 percent solution of gelatin in water represented close to the maximum concentration which could be easily used. Using this concentration, then as a basis, a "standard" solution was formulated as follows: 100 gm of Swift and Company Superclear 6-20 mesh gelatin is soaked in 200 gm of cold water for 30 minutes, for initial swelling. (Note - the cold water also contains 10 drops of Rohm and Haas CF-21 wetting agent and 0.3 gm Dowcide G germicide.) The container is then placed in a $130 \pm 2^{\circ}\text{F}$ constant temperature water bath. After three to four hours, with a slight amount of intermittent stirring or an overnight immersion, a clear, foamless solution is obtained. It was found that such a solution

*Draves, C. F. and Clarkson, R. G., Am. Dyestuff Reporter, 20-109-16 (1931).

could be kept liquid, for at least several days, in the constant temperature bath with no appreciable weight loss or change provided a cover was kept on the container. Liquid samples are then available at any time, or the material can be cast into pans for solidification into "bricks" for later easy usage.

Solution Control Techniques

In order to impregnate fabric uniformly it is necessary that the impregnating solution remain at a uniform concentration. Since the gelatin solution to be usable must be kept hot, it is inevitable that concentration changes would take place through evaporative water losses. Accordingly, a series of solutions were made ranging from 10 percent to 30 percent in gelatin concentration and the specific gravities of each were determined at 150°F. Figure 8 shows the curve obtained. This curve, it is believed, will be ample to assure control of the concentration at all times.

Plasticization Tests

In order to be able to use the gelatin impregnated material on a practical basis it is necessary that some means be found to keep the impregnated stock flexible for appreciable lengths of time. In the first

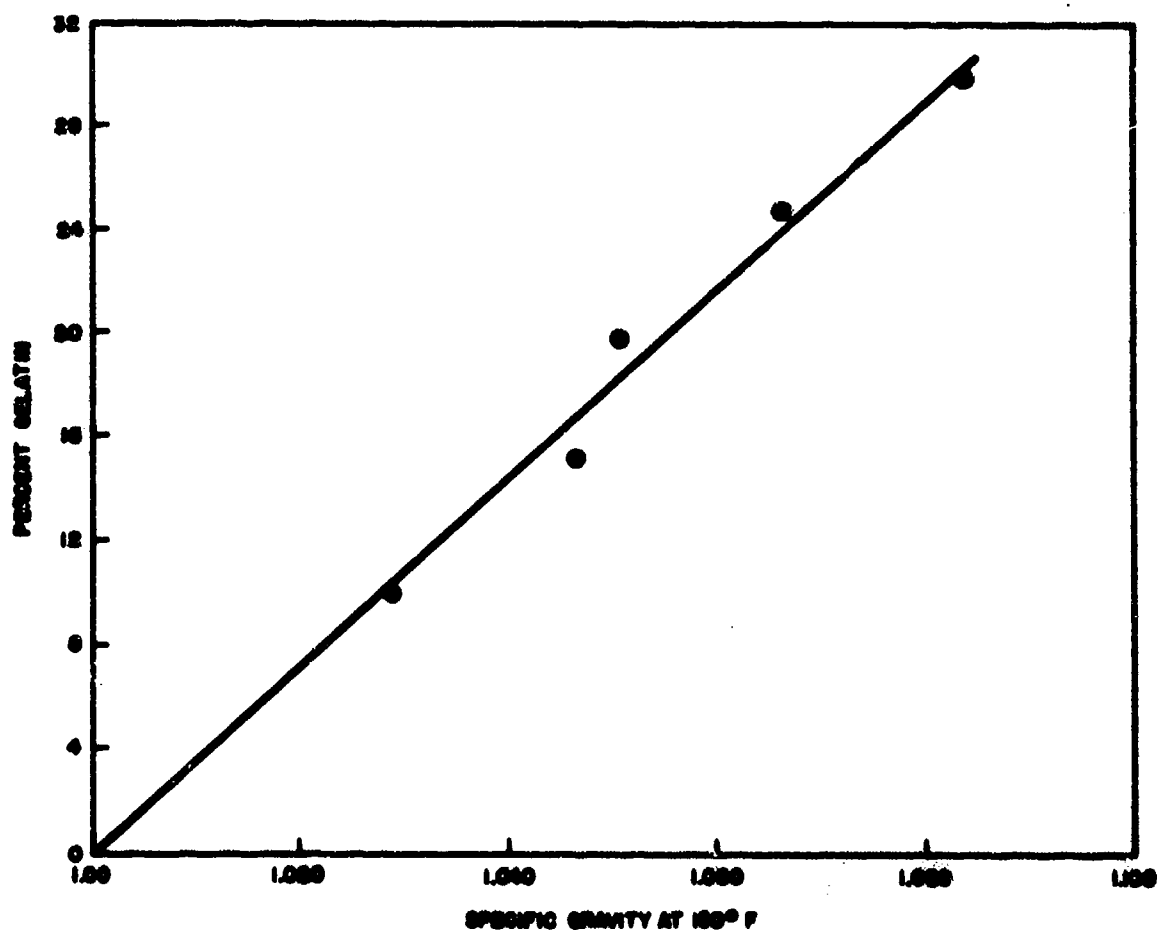


Figure 8. Gelatin solution control curve.

tests it was found that the solution had to be kept hot (approximately 120-150°F), and as soon as the impregnated part cooled and dried slightly (1 to 2 hours depending on the resin content) it became appreciably hard and difficult to handle. Some system of plasticization should then be one which could keep the impregnated structure soft for handling purposes during fabrication and also during storage. This latter requirement is considered the more necessary since the structure would undoubtedly be stored as a very compact package, and on deployment should be capable of expansion and erection with a minimum of effort.

Two systems were considered for this plasticization. The first consisted of storage of the part in a sealed container which also included a small amount of water. This would then act to maintain a water saturated atmosphere around the part. Freshly impregnated parts would first be allowed to air dry until definite stiffening commenced. On storage in the sealed, moisture saturated container, such parts were found to remain adequately flexible for at least eight weeks. On removal from the container, parts would air dry to the normally stiff condition. The sealed container used would be the same type as used for all other chemical systems.

In order to obviate the necessity for the excess water weight and the sealed container, a second system utilizing a plasticizer was investigated. It was hoped that by the use of a water soluble plasticizer having a relatively high vapor pressure, that a light weight, flexible sample could be made which could be stored in a simple container. Two plasticizers were initially investigated; ethylene glycol and glycerin. Glycerin, because of its humectant properties was considered particularly attractive, since it might extract water from the air to keep the sample flexible in storage. In both cases, however, the plasticized materials would have to rigidize in a vacuum in approximately the same time as did the water based samples.

A series of comparative samples were made up using, in each case, No. 181 fabric and a 20 percent solution of gelatin in water or in a water-ethylene glycol or a water-glycerin solution as the solvent. Samples were compared on the basis of resin pick-up, drying times and final stiffness obtained. Stiffness measurements were taken using a Taber Stiffness Tester and standard 1-1/2 x 2-3/4 inch samples cut from 6 x 12 inch impregnated stock. These same samples were also used for initial weight determinations and final resin content determinations. Initial impregnations were made using a Fisher-Payne Dip-Coater to draw the fabric strips through the solutions at a known rate. Figure 9 illustrates the impregnation equipment. The Taber Stiffness Tester is shown in Figure 10. In actual use it was found quite difficult to obtain satisfactory samples for the stiffness tester since the fully dried samples tended to curl quite badly. The curl then interfered markedly in getting good samples.

The results of the tests indicated that, in general, with this method of impregnation the initial weight pick-up with the 181 fabric ranged from 50 to 60 percent. (This weight was taken at the point where dripping stopped and the material dried slightly but was still quite flexible.) After



Figure 9. Fisher-Payne Laboratory dip coating equipment.
(S38551)

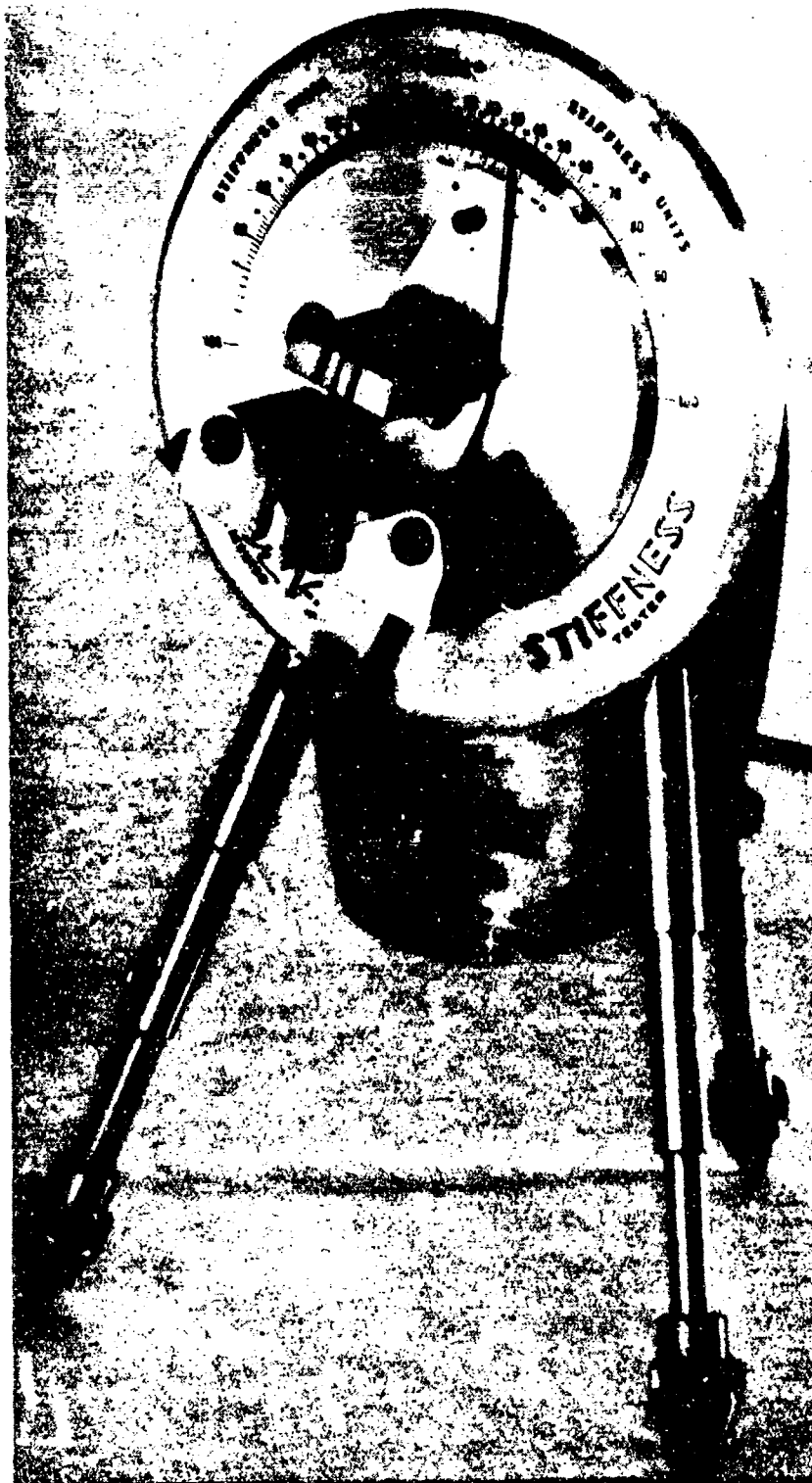


Figure 10. Taber stiffness tester.
(R94786)

1 hour of air drying the samples ranged in resin content from 33 to 45 percent and after 24 hours of air drying most of the samples ranged from 32 to 35 percent. After vacuum drying for as long as two weeks, the final resin contents were from approximately 29 to 34 percent. It was also found that, maintaining the samples for one week at 10^{-6} mm Hg, at room temperature reduced the vacuum dried weight approximately 4 percent, with no appreciable change in the Taber stiffness. Since 24 hours of vacuum drying at 1.0 to 0.1 mm Hg brought about virtually complete desiccation, this was adopted as the standard drying time.

The results of drying various samples of plasticized gelatin solutions are shown in Figure 11. In this it may be seen that extremely variable results were obtained from samples all of which had approximately the same final resin content. However, the data shown in Figure 11 did indicate some significant points. (1) Material containing 1 or 2 percent plasticizer (either glycerin or ethylene glycol) apparently hardens up as fast as does unplasticized stock. (2) Material containing from 4 to 10 percent plasticizer hardens at a much slower rate than do samples containing 1 to 2 percent plasticizer. (3) Qualitatively it was found that only the 6 to 10 percent samples appeared to be appreciably softened for long periods of time in the air. However, the fact that these materials act as plasticizers is nullified by the inordinate length of time required for hardening.

In consultation with Dr. H. H. Young of Swift and Company, a number of other plasticizing agents were suggested which, it was hoped would result in good flexibility during storage and at the same time rapid rigidization when exposed to a vacuum. The plasticizers tested then included the following: glycerin, ethylene glycol, two types of invert sugar, (a 77 percent solution, and a solution called Nulomoline, both from C and H Sugar Company), dimethylformamide, and Atlas Chemical Company "Sorbo," a 70 percent solution of sorbitol.

In each case the plasticizer performed satisfactorily, as far as initial plasticization was concerned. The amounts of the various materials varied from 20 to 40 percent by weight of gelatin and the most efficient materials were the original glycerin and ethylene glycol. However, in attempting to rigidize the impregnated samples in a vacuum, none of the materials were found to be volatile enough to allow good rigidization within a 24 hour period. Since these results were then similar to those obtained previously, it was concluded that the use of these plasticizers would offer no advantages over the use of a moisture laden atmosphere in the sealed storage container. A disadvantage to the use of any of these plasticizers was, of course, the additional weight that would have to be transported during launch. It was concluded then that plasticization of an impregnated material was not feasible, i. e., in the manner in which it was attempted.

As previously stated, in order to have a practical impregnation system, it is necessary that the impregnated material be flexible for appreciable periods after impregnation. Tests were therefore initiated

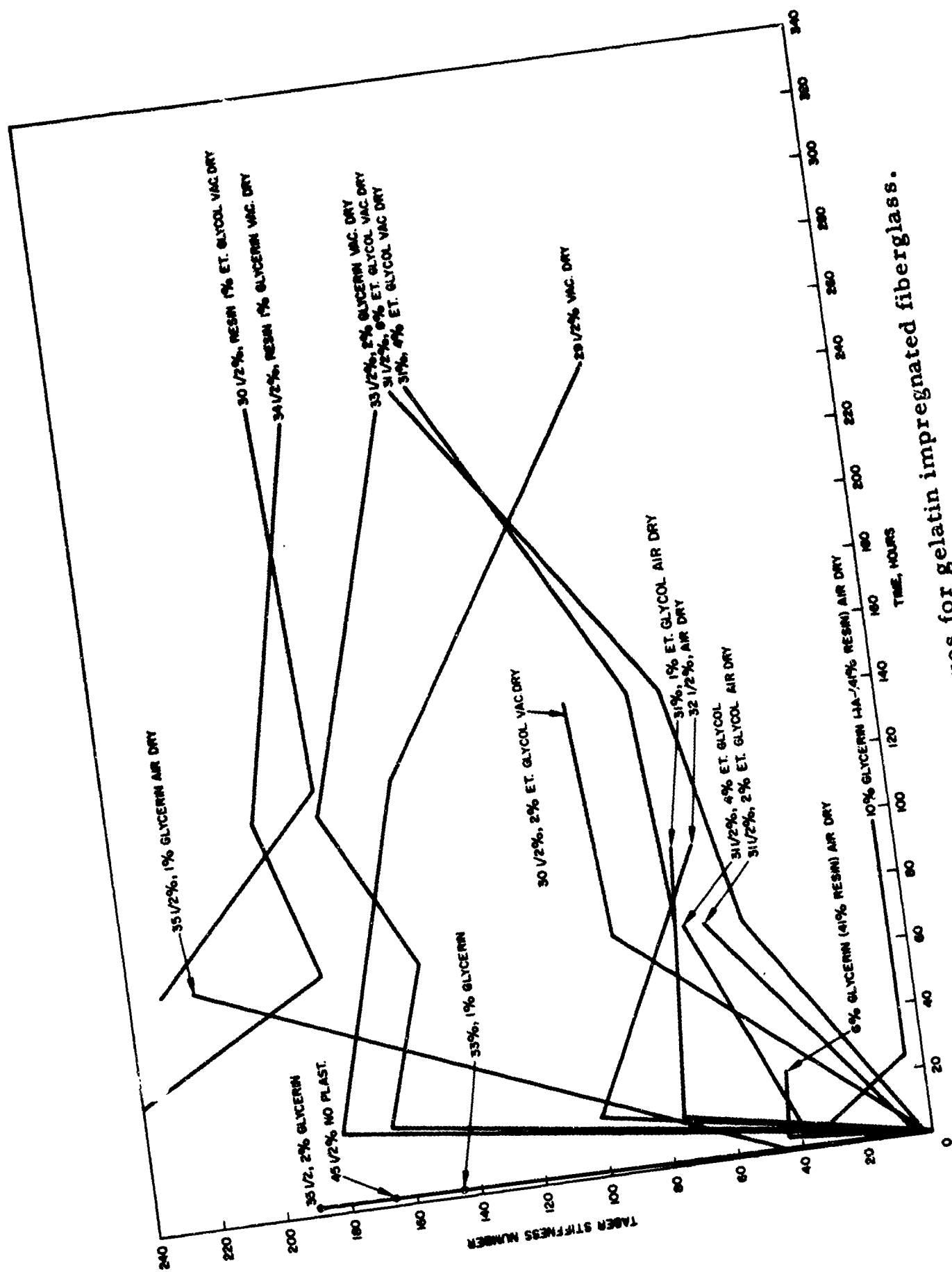


Figure 11. Drying time curves for gelatin impregnated fiberglass.

to determine techniques to improve the flexibility of gelatin impregnated materials. First samples were placed on the shelf of a desiccator which had a small amount of water contained on the bottom. It was found that samples either initially soft, or already hardened would remain soft or would readily soften when stored in such a manner. When stored for several weeks, however, these samples apparently picked up enough water to leach out the gelatin, since they became extremely soft and dripping. The same type samples were stored in a polyethylene bag with a small amount of water contained in a paper towel. These samples likewise softened up satisfactorily but did not leach out. The use of the minimum quantity of water was then seen as the key to successful storage. The use of 1 to 2 percent of Cab-O-Sil, a thixotropic agent, was also found to be helpful in preventing loss of gelatin impregnant.

Another technique utilized for flexibilizing the impregnated material consisted of simply brushing or spraying water on a hardened part, which had been allowed to air dry. It was found that very little loss of gelatin occurred as the result of adding the water, and softening would take place in 3 to 5 minutes. The amount of water required is roughly 30 percent of the weight of the dried sample. This system then could be used in fabrication of large structures simply by adding water as required to soften the structure. With this technique, and with the water vapor atmosphere storage technique, it is believed that storage flexibility can be satisfactorily obtained.

Impregnation Techniques

Most of the work described above was done using 181 fabric impregnated by dip coating. Work was also done to determine optimum techniques for impregnation of the three-dimensional fabric. The fabric which was used for these preliminary tests was a 28 ounce per square yard material obtained as samples from the Raymond Development Company pending production of the agreed upon 14 ounce per square yard stock. These first samples consequently were considerably heavier than originally planned.

The first impregnation tests were made by dipping sections of fabric and then installing the polyethylene inflation tubes in the flutes. This system was not too successful since, as the gelatin dried it became quite sticky, thus considerably impeding the insertion of the tubes. Another technique consisted of first impregnating the fabric, allowing it to dry and then inserting the tubes. This too was unsuccessful since the tubes were inevitably damaged by sharp particles of resin and/or impregnated Fiberglas.

Tests were also made using fabric samples with preinstalled tubes. The initial samples made with this technique were hand dip coated, using a dip tank and a roller, in a manner similar to impregnation of the fabric with the mechanical dip coater shown in Figure 9. Similar samples were also impregnated by brush coating the outer surface plies with the gelatin solution. In both cases, after drying the samples were

cross-sectioned and it was found that, because of the polyethylene tubes, incomplete impregnation had taken place in the vertical members. Tests were then made using a flat dip tank and total immersion of the tube installed samples for approximately five minutes. With this technique, fairly satisfactory impregnation throughout the sample was obtained. A number of rectangular and triangular samples were made in this manner. However, since the fabric in both cases was considerably heavier than originally planned, no attempts were made to make the physical test specimens. These samples were used to obtain information on drying times, resin contents, erection techniques, etc. The results of these preliminary tests indicated that resin contents from 30 to 50 percent could be obtained in the dip process, with good rigidity in the final part. Because of the rapid gelling of the solution, however, homogeneous impregnation still remained a problem.

Peptization Tests

In consultation with Dr. Young of Swift and Company, it was suggested that peptizing agents might be added to the gelatin solutions to prolong the liquefaction period. Dr. Young suggested a number of materials which could be used as peptizing agents for this purpose, and the materials tested include thiourea, calcium nitrate, zinc chloride, and ethylene chlorohydrin. It was found that all these materials will prevent the gelation, if used in sufficient quantity. However, large amounts of some of the materials appeared to weaken the gelatin structure. Tests therefore were made to determine the minimum amounts required for each peptizing agent to result in a viscous liquid solution at room temperature rather than a gel. With a room temperature gelatin solution then the fabric samples can be much more easily dip coated and then run through squeeze rolls to result in a uniform impregnation.

A number of repetitive and comparative tests were therefore made of the various peptizers. The procedure developed for these tests was as follows: 10 gm samples of the gelatin solution were placed in small aluminum cups and varying amounts of peptizing agents were added to each sample. The mixtures were warmed slightly to assure thorough solution and then the time was noted. The samples were then stirred at intervals and the time at which the material started to gel and "ball" up on stirring was then taken as the end point. Using this type of test an unpeptized gelatin solution (in the small amount tested) takes approximately 5 minutes to gel. The peptizing agents were added to the solution as weight percentages of the sample weight, i. e., 2, 4, 8, 12, 16 and 20 percent, etc. The ethylene chlorohydrin and thiourea were added directly to the solution and calcium nitrate was added as 3 part salt to 1 part water solution. Varying amounts of each peptizer were added to the gelatin samples until a solution was obtained which would remain liquid for approximately 1 hour at room temperature. The final results of these tests are shown below:

<u>Peptizing Agent</u>	<u>% by Wt</u>
Ethylene Chlorohydrin	20
Thiourea	7
Calcium Nitrate Solution*	15

Tests were also made using methyl butynol and methyl pentynol as peptizing agents. Amounts up to 30 percent by weight of each material were used but the peptizing action lasted only approximately 10 minutes for the pentynol and approximately 30 minutes for the butynol. Since the above materials were known to be more efficient no further work was done with these materials.

With the development of the 7 percent thiourea peptizing system it was considered that a satisfactory gelatin solution had been developed.

Impregnation Techniques

Coincident with the investigation of satisfactory gelatin solutions a number of tests were made to determine the best techniques for fabric impregnation. The first impregnation tests were made using nonpeptized gelatin solutions. Initial samples were prepared by simply dipping the three dimensional fabric first and then installing the polyethylene inflation tubes in the flutes. It was considered advisable to attempt to install the tubes after impregnation since it was felt the tubes might interfere with obtaining uniform impregnation. Accordingly a series of tests were made in which the fabric was impregnated without the tubes, and later tubes were installed. It was found quite difficult to install the tubes after impregnation since, as the material gelled and became adhesive, the facings would stick together and to the core flutes thus making insertion of the tubes very laborious. Tests were also run on impregnation of the cloth, allowing it to dry, and then later softening the assembly with water and then inserting the tubes. This procedure was also found to be quite laborious. Impregnation with the tubes installed then is considered the most practical from the standpoint of labor, particularly if long core sections are involved. Installation of the polyethylene tubes in the unimpregnated, dry fabric is, of course, relatively simple.

A series of tests were then run on fabric samples to determine the optimum technique to be used in impregnating the tube filled samples. One of the first techniques utilized a Fisher-Payne dip coater to draw samples through the warm gelatin solution. The flutes were oriented in the direction of draw. It was not found possible to do satisfactory impregnation, even at the lowest speed (1 inch per minute) using the

*Zinc chloride was not tested since preliminary evaluation indicated it was not as efficient as the calcium nitrate.

simple dip coat tank available. The gelatin solution, even with the added wetting agent, did not wet the material fast enough to insure even penetration. In order to do this type of impregnation it was felt that a longer tank would be necessary so that the material could remain in the solution for at least five minutes. Instead of using the dip coater then samples were dipped horizontally in a pan containing the warm gelatin solution. A five minute immersion with a small amount of "working" the sample resulted in what appeared to be an even impregnation.

Tests were made to determine if the impregnation was uniform. Several tube inserted fabric samples were dipped for five minutes at 125°F, air dried to the gel point, and then vacuum dried to a constant weight. A number of small sections (approximately 1/2 x 1 inch) were cut from each facing sheet and from adjacent areas in the core flutes. Resin content determinations, made by ignition, indicated that the average resin content was 57.3 percent with a variation of ± 6 percent. Several weighed samples were also placed in boiling water for two hours, and then the residue was dried in an attempt to determine an easier method of resin content determination. The results were not satisfactory, since the samples were too small to resist unravelling by the action of the boiling water.

The horizontal pan impregnation technique, although satisfactory for producing small test pieces was not considered practical for actual structure fabrication. Tests therefore were made to determine if satisfactory impregnation could be obtained by brushing. Brush application, if satisfactory, would be quite practical for future structural work at least until large volume production was required. After production of a number of samples by the brush technique, it was concluded it would be very difficult, if not impossible, to impregnate uniformly with the nonpeptized solutions being used. In attempting to use these solutions, it was found that the material on contacting the cold fabric would gel too rapidly to allow uniform distribution by the brush. However, with the development of a satisfactory peptizing technique it was then possible to impregnate the liquid throughout the fabric sample in an excess, and then using either a brush or roller squeeze technique the excess is removed leaving a uniformly impregnated material.

The roller assembly used was a commercial, hand driven wringer type assembly supplied with rubber rollers. This apparatus is equipped with adjustable, spring loaded pressure regulators so that samples can be put through at various pressures and at any desired speed. (See Figure 12.) The entire unit is very easily disassembled for cleaning and adjustment. This unit because of its versatility was used for all preliminary tests and for production of the physical test samples. After establishment of the proper conditions then it is anticipated that the motor driven dip coater assembly would be used in conjunction with its squeeze rollers.

With the development of satisfactory peptized gelatin solutions then it was found possible to dip the fabric samples in a room temperature



**Figure 12. Hand driven squeeze roller assembly.
(S40282)**

gelatin solution, or brush coat until thorough wetting was achieved without premature gelatin interfering with the impregnation. The sample can then be run through the wringer rolls until it appeared homogeneously impregnated. By weighing the sample before and after impregnation it was possible to determine the approximate resin pickup. In impregnating the samples with polyester resin or the polyurethanes, it was found fairly easy to procure approximately the correct amount of pickup by this procedure. However, when impregnating with the gelatin solution, because of the approximately 60 percent volatile material it was somewhat more difficult to arrive at the desired pickup, since the results are not known until after the sample is dehydrated. A series of tests were therefore made to attempt to determine quantitatively the uniformity of the resin content.

Resin Content Determinations

Since the thiourea peptized solutions were the lightest weight these solutions were selected for the tests. The first impregnated samples were found to be considerably below the desired 40 percent resin content. However, after producing a number of approximately one foot square samples, a procedure was established whereby the 40 ± 5 percent resin content was obtained. In these samples resin content was determined by obtaining three small die cut samples from the upper left corner of both facings and the web from approximately the center and from the lower right-hand corner. (See Figure 13.) It should be noted that die cut samples were used rather than normal random sized samples. It was hoped, after many samples had been tested, that it would be possible to determine the resin content by simply weighing the die cut sample, rather than by performing a complete ignition test. These data would be obtained only after a large number of samples had been analyzed, comparing original sample weight with fabric (ash) weight. In Table 2, are shown the results of the first resin content tests run on eight samples.

In Table 2, it should be noted that in the first samples made the webs were impregnated to a resin content considerably below that of the facings. In the later samples, it was found possible to increase the resin pick-up to equal or to even surpass the pickup of the facings as shown in Table 2. The low pickup was at first attributed to the differences in roller pressure in the area of the facings as compared to pressures developed where the webs and facings were between the rolls. It was later found that, by passing the sample through the rolls several times, the web pickup could be increased considerably as shown.

All of the above data relates to work done using either the 14 or 23 ounce rectangular fabric. (However, the results shown in Table 2 represent only 23 oz material). In attempting to analyze why the web pickup increased so much with the prolonged rolling it was found that the webs were actually thicker than the face sheets as shown in Table 1. This situation was not found in the case of the triangular construction, which may then necessitate additional development efforts before a system can be established for preparing test samples with a desired resin content.

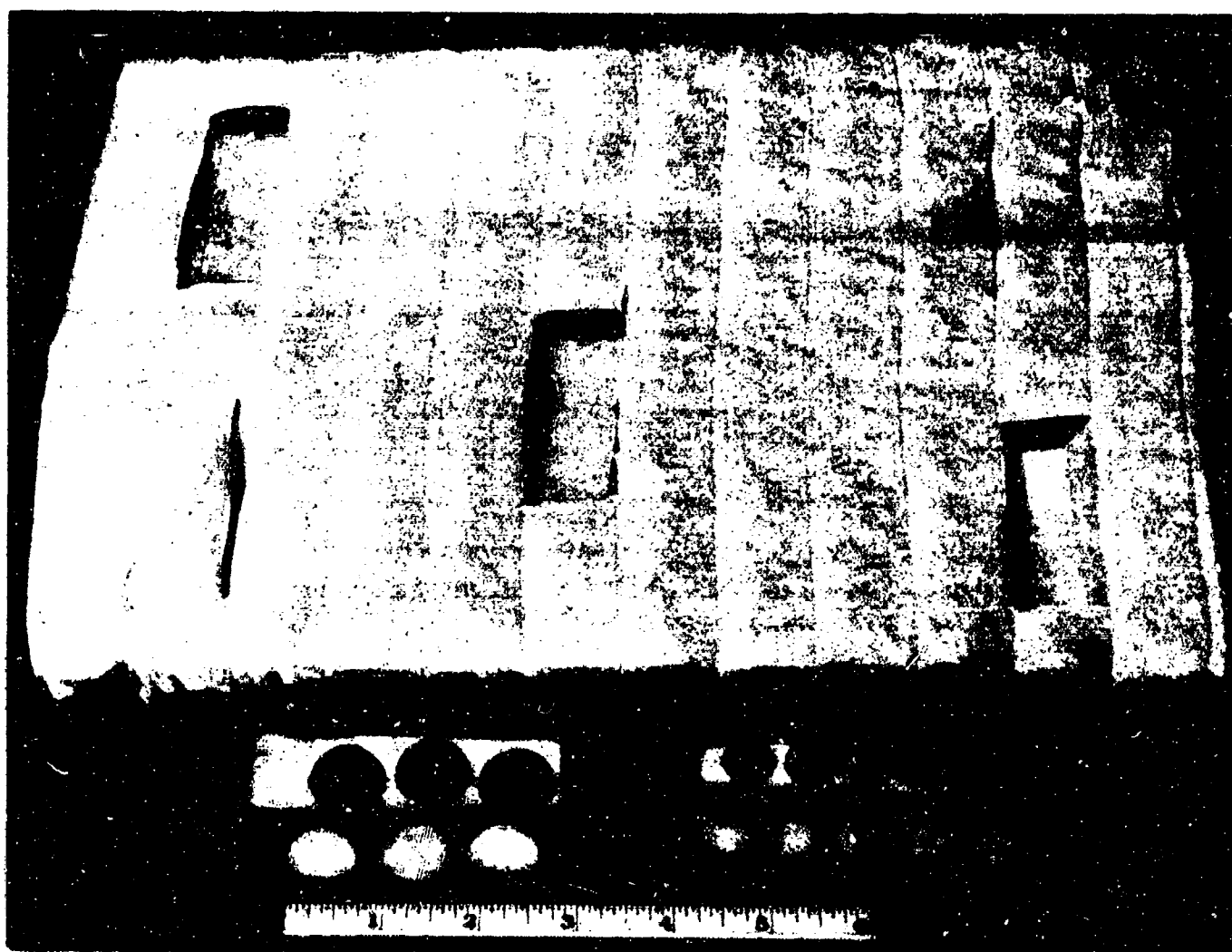


Figure 13. Resin content test samples.
(S40286)

TABLE 2. RESIN CONTENT DETERMINATIONS
(23 OZ MATERIAL)

Sample No.	13(a)	13(b)	14(a)	14(b)	14(d)	18	19	21
Top Facing, % Resin	50.4	42.6	19.7	25.0	27.1	41.5	36.8	25.9
Bottom Facing, % Resin	47.7	40.6	23.0	25.6	21.0	42.3	39.6	29.3
Web, % Resin	29.2	27.6	18.7	23.1	19.6	41.7	42.3	34.7

Polyester Resins – Ultraviolet Radiation Catalysis

The first samples prepared using polyester resins were 10 x 12 inch light weight and heavy weight three dimensional fabric utilizing American Cyanamid Laminac 4128 resin catalyzed with 1 percent benzoin dispersed in a 1:1 tricresylphosphate-benzoin mixture. Very satisfactory UV catalysis was obtained with these samples. No difficulty was encountered with UV radiation failing to penetrate the three dimensional structure, even with the polyethylene tubes present. A problem did arise concerning storage of catalyzed impregnated fabric using the Laminac 4128 resin. With this material approximately one week, at room temperature, was the maximum storage time without onset of gelation. This was considered too short a period to be practicable.

Development of new or more stable resins was considered outside of the scope of this investigation, therefore a number of other types of commercially available polyester resins were procured for test. Emphasis was placed on securing nonaccelerated resins (Laminac 4128 contains 0.06 percent of accelerator as supplied by the manufacturer). The additional resins obtained for test are shown in Table 3.

TABLE 3. POLYESTER RESINS USED FOR STORAGE

Resin	Source	Modifying Monomer
Laminac 4123	American Cyanamid Co.	Styrene
PDL 7-820-25-25	American Cyanamid Co.	Styrene
Laminac 4202	American Cyanamid Co.	Diallyl phthalate
Vibrin 158	Naugatuck Chemical Co.	Styrene
Vibrin 136A	Naugatuck Chemical Co.	Triallyl Cyanurate
Hetron 103	Hooker Chemical Co.	Diallyl phthalate

The first tests consisted of catalyzing a small amount of each resin with 2 percent benzoin, and then fabricating a small four-ply type 181 glass fabric laminate. All the samples were then cured simultaneously in bright sunlight. Only the styrene modified resins cured rapidly enough (approximately 30 minutes) to be considered. Table 4 shows the average Barcol hardness of the various resins after the 30 minute cure.

TABLE 4. BARCOL HARDNESS OF UV CURED
POLYESTER RESINS

Resin	Barcol No.
Laminac 4123	55.4
PDL 7-820-25-25	55.4
Laminac 4202	9.8
Vibrin 158	54.6
Vibrin 136A	10.5
Hetron 103	24.6

A series of tests were then initiated to determine the room temperature catalyzed storage stability of the three styrene modified resins. 100 gm. samples of each resin catalyzed with 1 and 2 percent of benzoin were inhibited with 0.2 percent of hydroquinone and similar samples were inhibited with 0.2 percent Eastman Chemical Products dihydroxybenzophenone (DHBP). Similar noninhibited samples were also prepared as controls. Within one week all the 2 percent noninhibited samples showed evidence of gelation. However, it was found that samples prepared with 0.2 percent of hydroquinone as an inhibitor stored for at least three months in sealed containers. The materials found suitable include American Cyanamide Laminac 4123, PDL-7-820-25-25 and Naugatuck Chemical Company Vibrin 158. The materials were tested by periodically withdrawing small samples, fabricating a 4-ply 181 glass fabric laminate and exposing to UV. In each case, satisfactory laminates of approximately 45 to 50 Barcol hardness were obtained after approximately four to six hours exposure to sunlight. The Laminac 4123 is judged to be the optimum material on the basis of reactivity and handling properties, therefore, the major portion of the tests were made with this resin. Samples of the other resins were prepared, however, for comparison purposes.

During the storage tests, it was also found that fabric samples impregnated with normal resin would, in storage, drain due to the action of gravity until even tightly rolled samples would result in a very low resin content. Incorporation of Cab-O-Sil, in the resin mixture, in amounts up to 2 percent by weight effectively prevented the migration. The addition of a thixotropic agent, in the amounts used, did not appear to inhibit the reactivity of the resin.

Urethane Resins — Vapor Catalysis

Tests were initiated with water vapor cured urethane resins developed by Wyandotte Chemicals Corp. Two resins were tested, PR-1550-A3 and PR-1502-E6. Each material was a medium viscosity resin well suited for brushing or dip coating with the addition of toluene as a thinner. Each material was catalyzed with 0.5 percent of 1, 2, 4 trimethyl piperazine just prior to impregnation.

In the first tests that were made, the desired quantity of resin was removed from the container, working in a dry box which was continually flushed with dry nitrogen. This was done to inhibit the possible effect of moisture laden air from causing premature gelation. As a result of these preliminary tests, it was found that even after catalysis the material could be handled in an ordinary laboratory (40 to 50 percent RH) atmosphere. Consequently all the work done with these resins was done under normal laboratory conditions which considerably facilitated the work.

Preliminary tests using impregnated fabric taped over a tin can containing water indicated that 2 mil cellophane or 1 mil solid or spark perforated polyethylene would adequately transmit water vapor to cause a cure, in a vacuum, in approximately 6-8 hours. Further tests, using the polyethylene "auto-tubes" containing a small amount of water, indicated that these 2 mil wall thickness tubes would very satisfactorily erect the integrally woven structures and at the same time allow enough water through, under vacuum conditions, to cure the resin in 6 to 16 hours.

As a result of preliminary tests it was decided that the PR-1502-E6 resin had the better physical properties therefore it was used in the great majority of the tests. This information was also confirmed in Reference 2. The physical test samples were made by uniformly brushing a desired quantity of toluene thinned resin into the fabric, then inserting the polyethylene tubes into the fabric flutes. It was also found considerably more difficult to insert the polyethylene tubes in the polyurethane impregnated flutes as compared to polyester impregnated material. Wetting the tubes with toluene, however, facilitated insertion considerably.

All the work done with the polyurethane resins during this phase was done in a normal, 50-percent RH laboratory. No particular difficulty was encountered with premature gelation or cure in the approximately one hour it took to prepare a sample. Another minor difference in the preparation of the polyurethanes as compared to polyesters is that water was used in the erection tubes instead of alcohol. The slight difference in the vapor pressures appeared negligible in the finished samples.

Storage tests were also run on samples impregnated with the Wyandotte PR-1502-E6 resins. The resin used in these tests was also

filled with 2 percent Cab-O-Sil to inhibit resin drainage. Samples appeared to remain flexible for approximately two months, then set up within a two-week period. In this case, the samples were kept in a sealed can and opened every two weeks. Before closing the can each time it was flushed with dry nitrogen. However, it is presumed that a small amount of moisture was absorbed each time, which may have contributed to the final rapid curing.

In preparing the storage samples the fabric had polyethylene tubes inserted, however, the tubes did not contain water since it was felt that the water vapors would be transmitted through the polyethylene tube with consequent curing of the resin while in storage. In actual service the water would then have to be contained in small glass ampoules or similar containers which would then be opened just prior to use.

In the tests which were made with the polyurethane resins, a second batch of the PR-1502-E6 resin was obtained somewhat coincidentally shortly after a second roll of polyethylene erection tubing had also been procured. When the first rigidization tests were run, it was found that approximately 40 hours were required to rigidize the new material as compared to approximately 16 hours for the first batch. A check of the tubing also indicated that the new material was heavier and had a much lower moisture loss rate than did the original material. In contacting the vendor, it was found that the raw materials were not purchased to any rigid specifications and strict quality control was not exercised because it was purely a low priced consumer item.

In an effort to get faster water release, a number of erection tubes were made of Union Carbide's Radel (a polyethylene oxide film two mils thick). These tubes, when placed in a vacuum with water inside, would disintegrate in a few hours, mainly at one point. While this material could, therefore, be used for this application, it was decided to continue using the polyethylene and to cure the samples for approximately 40 hours instead of 16. However, should it be necessary, it is felt very feasible to procure polyethylene tubes which would allow 16 to 24-hour cures.

Polyurethane Foam Powders — Heat Activated

The fourth rigidization system investigated concerned the use of a one component, heat activated polyurethane foam powder previously developed by Hughes Aircraft Company, Reference 3.

This material, since it was in the form of a powder, was naturally not adoptable for impregnation into a fabric. An investigation was thus made to determine if a suitable solvent or suspensoid could be found for the resin. Then this resin solution could be used to impregnate the fabric. A large number of such solvents were tested to determine which could be used to make such a solution or suspension. The materials tested are shown in Table 5.

TABLE 5. FOAM POWDER SOLVENTS TESTED

Acetone	Ether
Benzene	Ethyl Alcohol
Butanone	Ethylene Dichloride
Carbon Disulfide	Mexane
Carbon Tetrachloride	Isopropyl Alcohol
Cyclohexane	Methyl Methacrylate
Cyclohexene	Methylene Chloride
Dimethyl Formamide	Petroleum Ether
Dimethyl Sulfoxide	Pyridine
Dioxane	Tetrahydrofuran
O-Dichlorobenzene	Toluene
Ethyl Acetate	Xylene

Of all the solvents tested, acetone appeared to be the most suitable from the standpoint of high solvency, low reactivity, high volatility, ease of working, etc. It was found that, with almost all the solvents, the foamant solution, if kept for more than a few hours, showed evidence of reaction. This was shown either by presence of foam in the solution or definite hardening. A method was finally developed in which an acetone solution was made up and immediately spread on the fabric. The solvent then quickly evaporated leaving the foamant in the fabric. Such samples foamed fairly well when heated in a vacuum. Figure 14 shows the appearance of fabric coated in this manner. The use of this technique, however, was found to have two drawbacks: (1) in long term storage it is believed the samples would become very dry and stiff and would thus interfere with easy erection and (2) it was not possible to saturate the vertical cell walls with sufficient foamant to cause good foam in flute interiors. However, uniform impregnation of the flutes can undoubtedly be achieved with further work; long term storage can probably be achieved by use of anhydrous acetone and storage in an anhydrous acetone atmosphere.

In another series of tests it was found possible to spread the powder on fabric and using a heat sealing iron and cellophane the material could be melted into the fabric. By allowing the heat to remain on the powder just long enough to melt the foamant and then quickly cooling no reaction occurred. Such a system could be used to produce a "foaming film," however, this technique also had the same two drawbacks as given above.

Small quantities of foam were also sealed in polyethylene tubes which were then inserted into the flutes of the fabric. The initial tests were made using 2 mil polyethylene tubes. Poor foaming resulted.

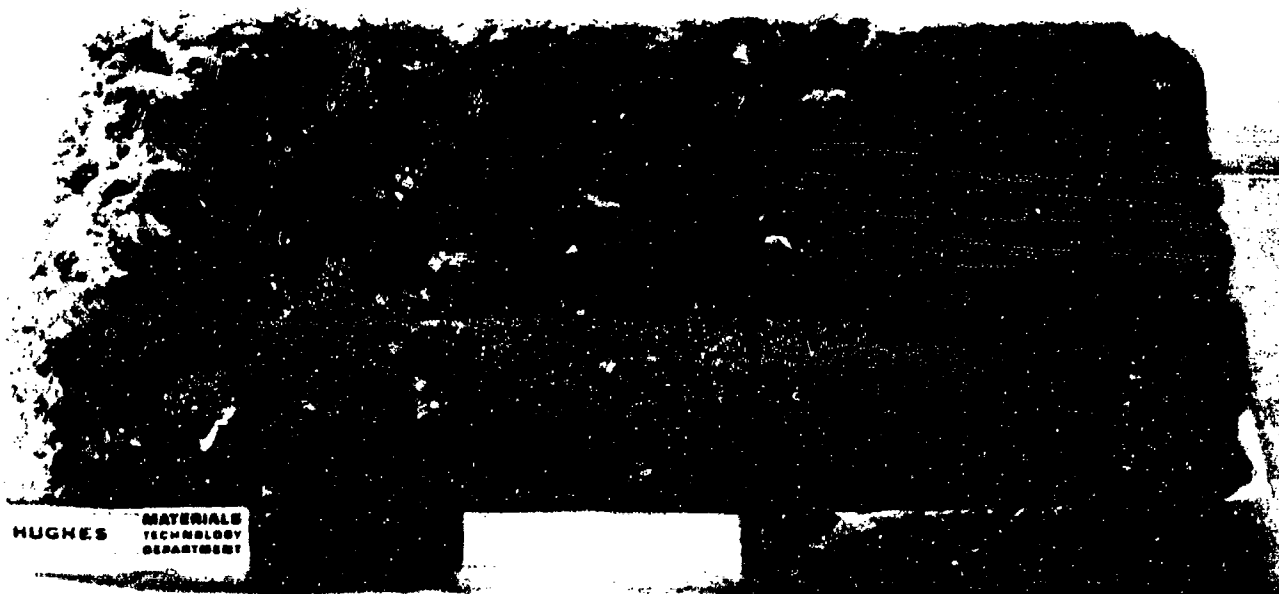


Figure 14. Foamant coated glass fabric. Foamed in vacuum.
(S39245)

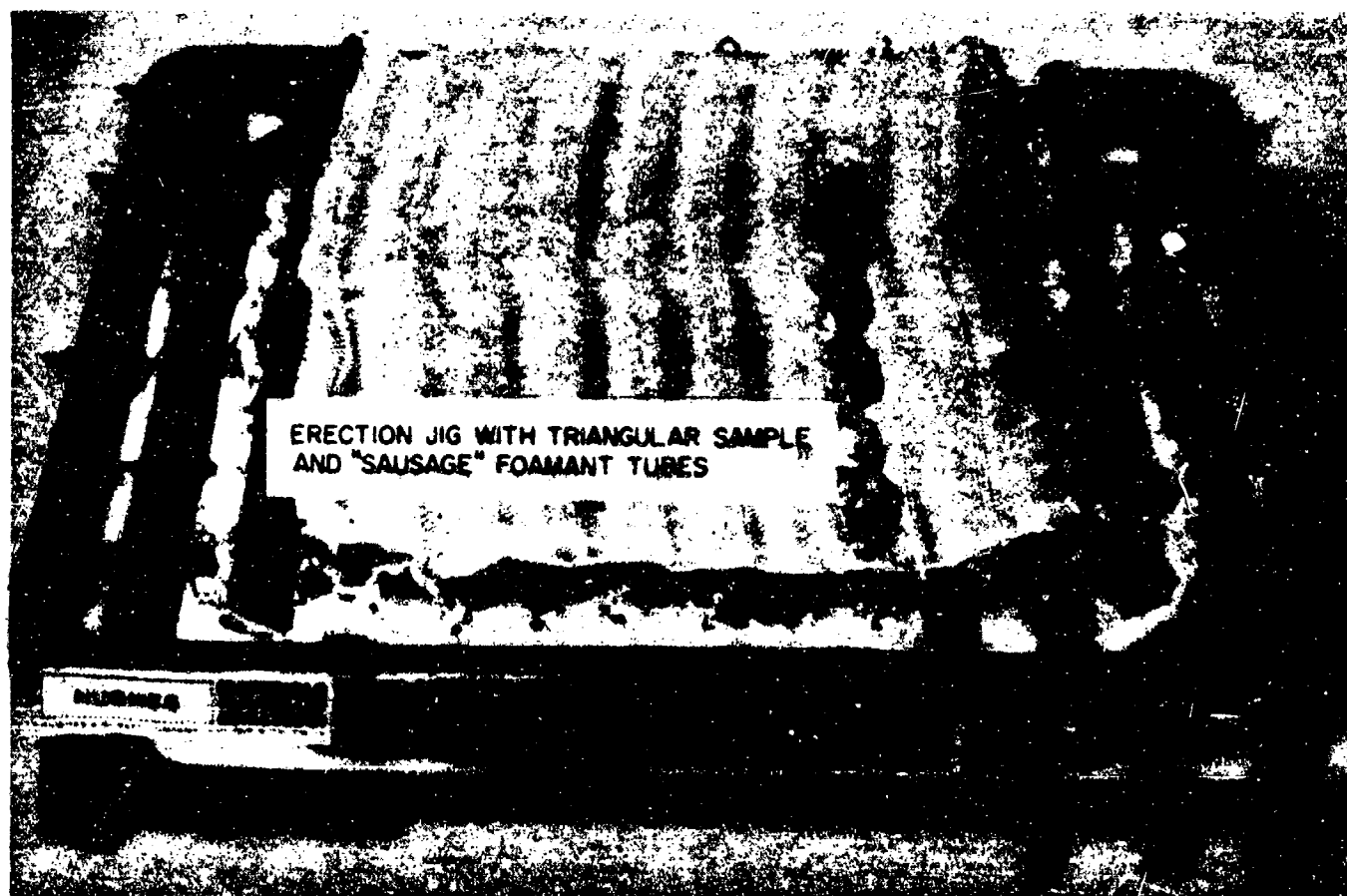


Figure 15. "Sausage" foamant tubes.
(R95581)

Changing to 1 mil tubing and placing the powder into small "sausage" containers gave much better results. Figure 15 shows the appearance of the foam powder "sausage" and how they are installed in an assembly. Figures 16 and 17 illustrate the appearance of rectangular and triangular fabric samples made by this technique.

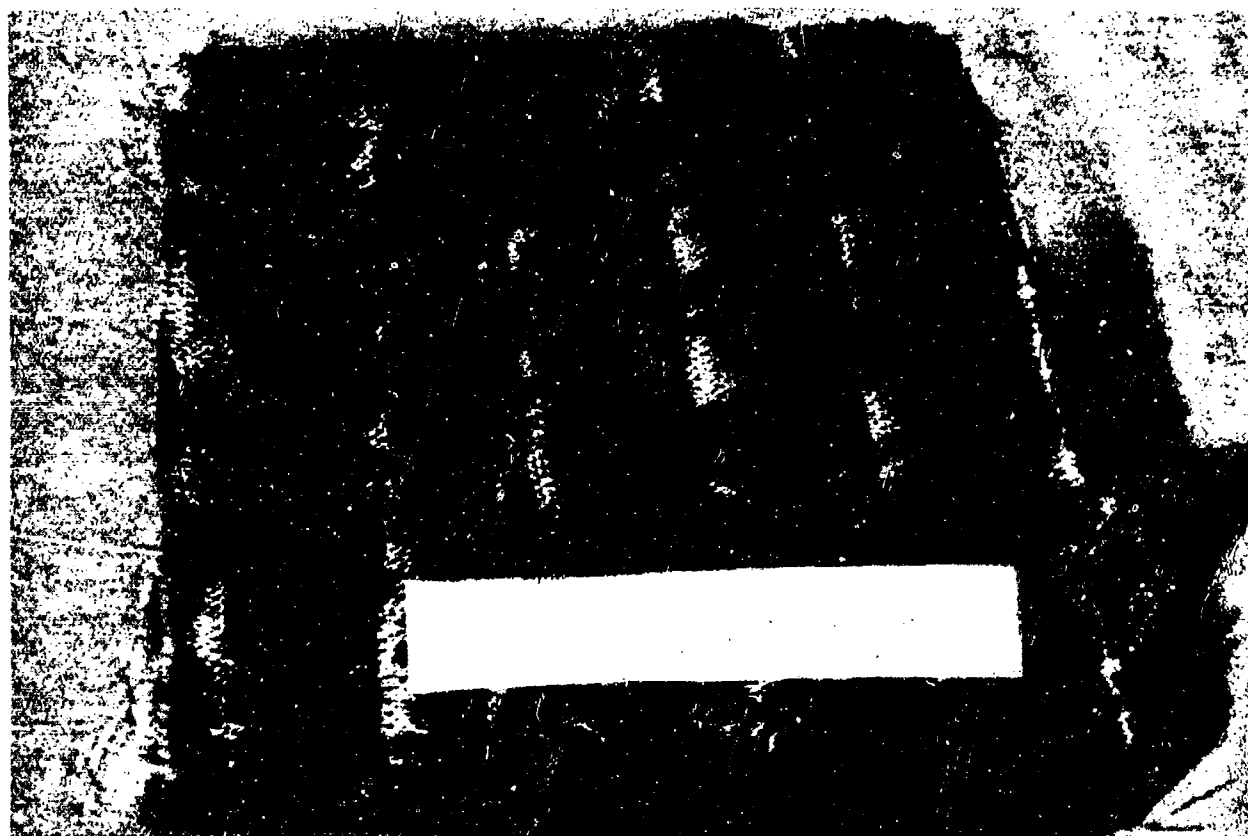


Figure 16. Foam filled rectangular structure.
(S39246)

The major disadvantage to the use of the Hughes solid polyurethane foamant is the fact that this material is not exothermic. Consequently, a good deal of heat is required to cause foaming and complete curing of the material. The heat necessary to form the samples shown in Figures 16 and 17 was applied for approximately 20 minutes and temperatures of 250 and 300°F were required.

A formula for an exothermic solid foamant was supplied by the Materials Application Division of the Air Force Materials Laboratory and tests were made to determine its properties. The formula given is shown below:

	<u>Eq.</u>	<u>gm.</u>
Adipic Acid	0.3	22.0
Trimethylolpropane	0.6	27.0
Methylene bis (4-phenyl isocyanate)	0.4	50.0

	<u>Eq.</u>	<u>gm.</u>
Catalyst (diethylenetriamine)		1.0
Aluminum Chloride		2.0
Tween 21 wetting agent		3.0

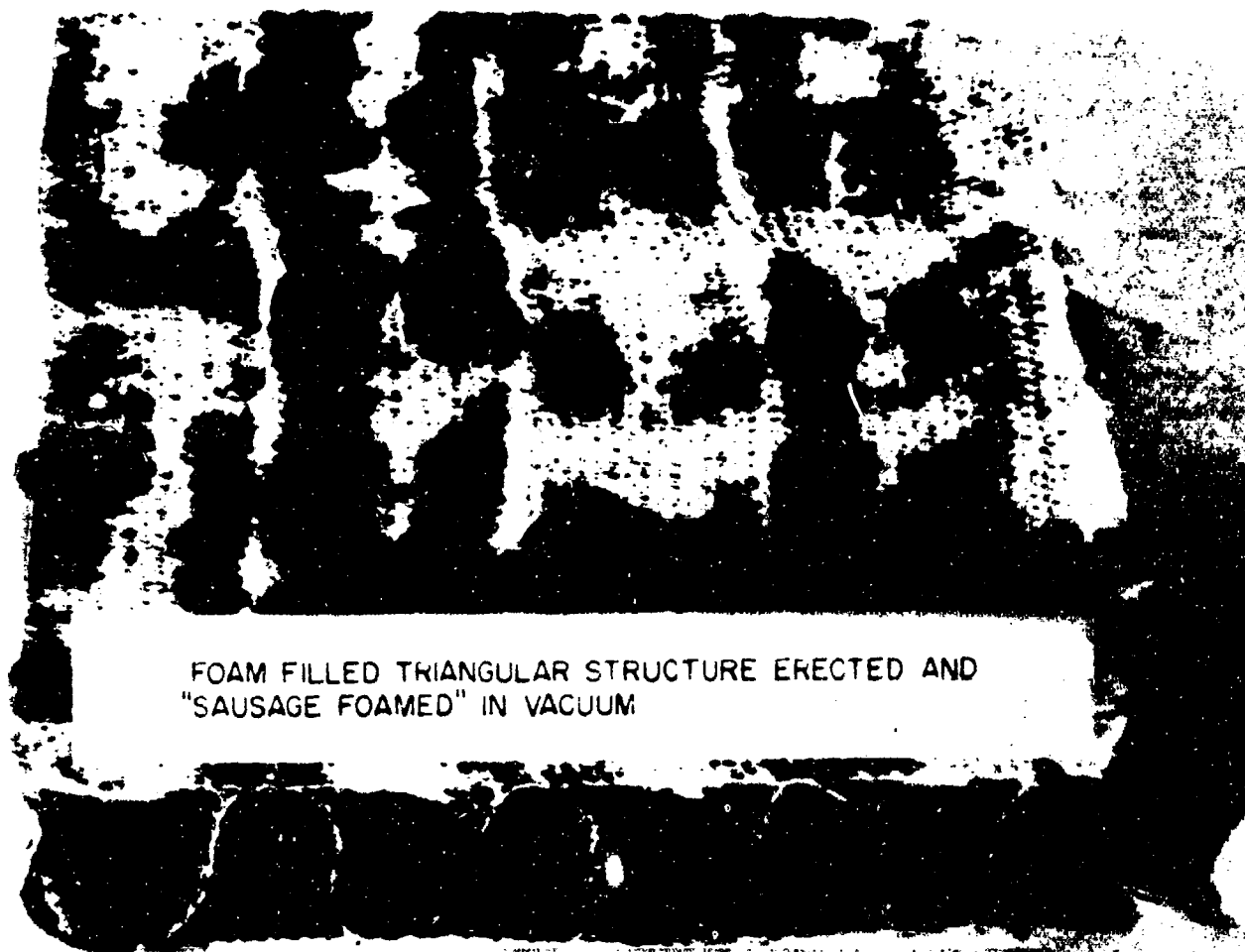


Figure 17. Foam filled triangular structure.
(R95580)

In an initial test of the above formulation, it was found that after one minute of heating in a vacuum the reaction did exotherm and continued to exotherm and foam for approximately 20 minutes. Samples made up as shown in the above formula appeared to foam approximately four to six times their original size or to a foam of approximately 20 pound per cubic foot density. On changing the surfactant from Tween 21 to Dow-Corning DC-113 a marked increase was obtained in the amount of foam produced. Such foam appeared to be approximately 2-3 pounds per cubic foot density.

Storage tests were made on the foamant mixture and it was found that a shelf life of approximately 24 hours at room temperature was the maximum obtainable without onset of inhibition of the reaction. Storage under refrigeration increased this time to 3-4 days.

The foam produced with the Tween 21 surfactant was a hard, tough material somewhat comparable to similar high density foams but not quite as strong. The lighter material produced by use of the DC-113 surfactant was also not as strong and tough as comparable density conventional foams. Because of the short storage stability of this mixture stannous chloride (1.0 gm) was used in place of the aluminum chloride. The shelf life increased to approximately one week without noticeable inhibition of the reaction. As a result of this work, it may be concluded that while the formulation is not stable enough for use in the fabrics at this time the decided advantage of the exotherm reaction definitely warrants additional efforts.

"B" Staging Tests

One of the disadvantages in the use of the liquid impregnants in the fabric structure is the high degree of tackiness exhibited by each system. Therefore, it is necessary to use a parting film on each side of the fabric so that any of the materials may be rolled or folded up. This parting film, usually 1 mil polyethylene, Mylar, etc. adds weight and bulk to the structure and, therefore, is undesirable. Any technique where the surface could be made nontacky would be of considerable benefit.

Polyurethanes. The polyurethane materials were tested by allowing impregnated fabric to remain exposed to the 50 percent RH laboratory atmosphere until evidence of surface curing was observed. This took approximately five hours. The surface was then talc coated after which it could be handled and folded on itself without evidence of sticking. One such sample was then placed in a humidity cabinet and after an overnight exposure to approximately 90 percent humidity the material appeared to harden normally. Another simple sample, to be used as a control, was placed in a jar which was then flushed with dry nitrogen. Unfortunately, this sample also appeared to harden completely in one day. This then indicated that the initial cure apparently was not just a surface phenomena, but appeared first on the surface and then continued through the material depth.

Gelatin. Tests were initiated on "B" staging of gelatin impregnated samples using techniques suggested by Dr. Young of Swift and Company. In these tests fabric samples were impregnated first with thiourea peptized solutions and then dipped in a 10 percent formaldehyde solution followed by a kerosene dip. The result of this treatment is a sample considerably reduced in surface tackiness as compared to an untreated sample. Such samples can be touched, folded on themselves, and handled with a great deal less trouble than similar untreated samples. Storage tests with such samples when kept in a loosely rolled condition appear to be satisfactory for two weeks. However, the same type samples folded to themselves and clamped tightly together with no parting films are stuck together very tightly in a few days and probably could not be erected uniformly by the normal tube pressure.

It may be concluded that in the preliminary tests, this process has considerable promise. There are, however, a number of details to be worked out in order to develop the optimum system. These include a determination of the preferred time interval between impregnation and formaldehyde dipping, i. e., if the sample is dipped immediately after impregnation there is danger of gelatin being leached out, whereas if a long period ensues the material will gel. Other factors to be determined are the optimum formaldehyde concentration, dip time, and the time interval between formaldehyde dipping and kerosene dipping, and the possibility of using talc or other powder to inhibit blocking. Until the above details are satisfactorily developed, the best system for B staging consists of allowing impregnated material to dry and then reflexibilizing with water vapor in a sealed container. (Note: see page 16 also).

Polyesters. Only a small amount of experimental work was done on the problem of "B" staging the polyesters. Limited literature research and inquiries indicated that the low temperature catalyst system used (benzoin and benzoyl peroxide) would not be amenable to starting a surface reaction and then stopping the reaction, other than by cryogenic cooling.

Part of the reason for the tackiness and difficulty in "B" staging the polyester resins used lies in the type and amounts of monomer and resins used in the current materials. The monomer, styrene, is a very reactive monomer and is used to the extent of approximately 30 percent which contributes to relatively low viscosity. The resinous constituents, isophthalic and maleic and fumaric anhydrides, are also quite reactive and do not lend themselves too well to "B" staging by the normal techniques.

Inquiries were made to the American Cyanamide Company with a view to obtaining a resin with better possibilities of "B" staging. Such a resin with a 20 percent styrene content was a good deal "drier" and had a higher viscosity. By "loading" this resin with a thixotropic agent and using talc on the surface a pseudo "B" staged material was made. After several days storage, however, the resin completely wet the surface again and became quite tacky.

Tests were also made with resins containing diallyl phthalate monomer instead of the styrene monomer. These resins were appreciably higher in viscosity than the styrene based materials and were also much slower in reactivity. It was found that these materials could be surface cured when a laminate of approximately 1/32 of an inch or greater was used. With the thin facings and webs used in the three dimensional fabric, any cure was throughout the entire thickness and was not just on the surface. Even though the material could be cured partially to result in very low surface tackiness, it was still found to be quite unsatisfactory from the standpoint of usability. It was found that if a partially cured sample was folded or rolled there inevitably resulted some break-away of the resin from the fabric visible as a "starved" spot. On subsequent final cure of this fabric, since the material would not soften and

run together, the starved spot would remain as a defect, thus this technique was not considered practical for this system.

The initial tests done on "B" staging of any of the resin systems showed very little success. In all of these tests the approach taken was that of partially curing the resin and then attempting to arrest the cure. Since this approach apparently did not work satisfactorily with any of the resins it was decided to try a completely different approach.

The technique then investigated consisted of applying a fast drying coating to the various impregnated fabrics. In each case a coating was applied which, it was hoped, would have no reaction with the underlying wet resin impregnate.

The polyester impregnated fabrics utilized the low styrene content resins. After impregnation the samples were surface coated with a small amount of talc to give an initially dry-to-touch surface. A brush coating of a water-alcohol solution of polyvinyl alcohol was then immediately applied to the talc coated surface. This type of material is commonly used as a parting film in preparation of polyester laminates and should show good resistance to the solvent action of the styrene. Unfortunately it was found that in this case the film resisted the wet resin for only a few days, after which surface tackiness set in.

Gelatin impregnated samples, which were formaldehyde and kerosene soaked, were similarly treated with a polyvinyl chloride solution. This coating, which is also commonly used as a parting agent, should have had good resistance to the action of the water in the gelatin. These samples, however, also failed in a few days in hermetic storage.

Examination of both types of samples indicated that the primary reason for failure in each case was the inability to form a perfect film over a wet surface. In each case the film appeared perfect when initially applied, however, after a short drying period and during storage both types of film broke down rapidly. Possibly the application of many coats might have been successful, however, this approach was not considered practical.

Compound Curvature Samples

A sample of rectangular fabric impregnated with polyester resin, using "auto"-erection tubes was laid up over a small form made to a 3-1/2 foot spherical radius. The sample was forced to conform to the radius by means of a retaining ring which was fitted around the form. After cure was complete the sample was found to have assumed the compound curvature very well. (See Figure 18.) This then indicated that the three dimensional material was well adapted for fabrication of compound curvature structures with radii at least as small as 3-1/2 feet.

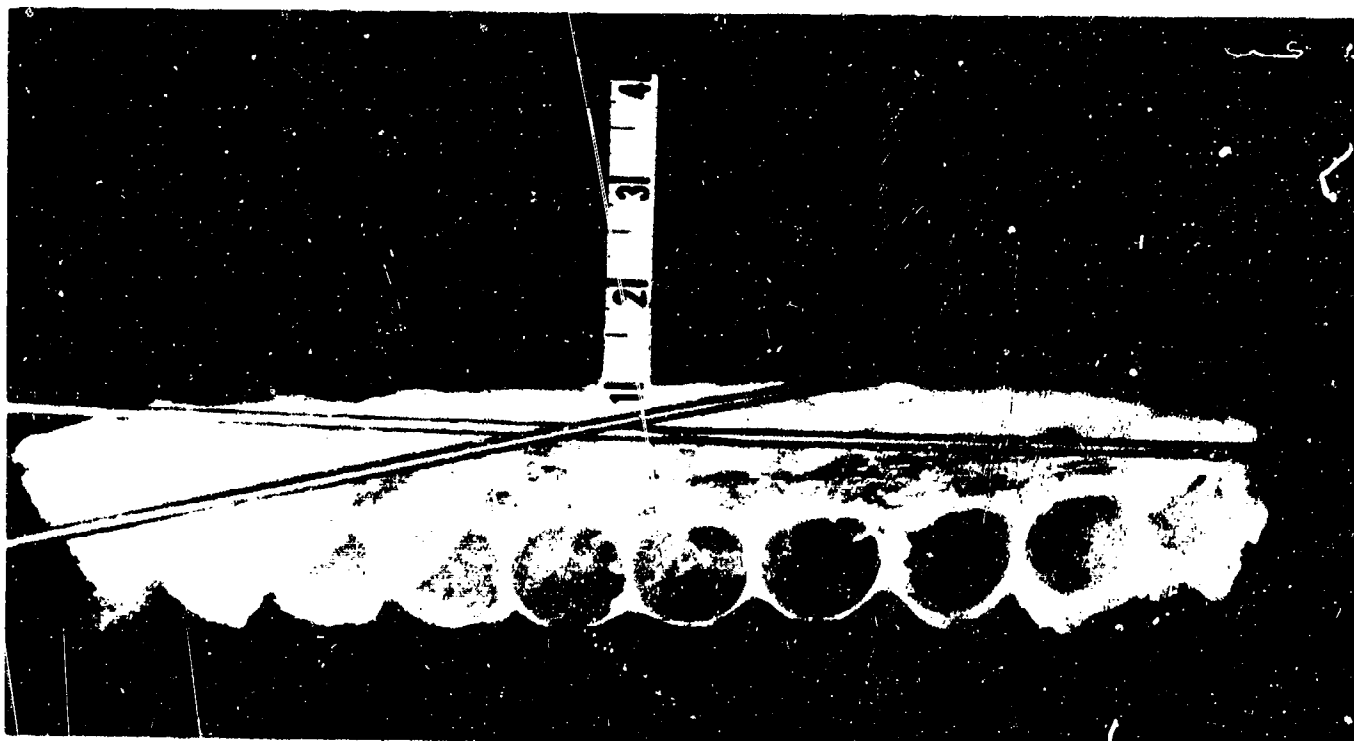


Figure 18. Three dimensionally curved sample. (R100568)

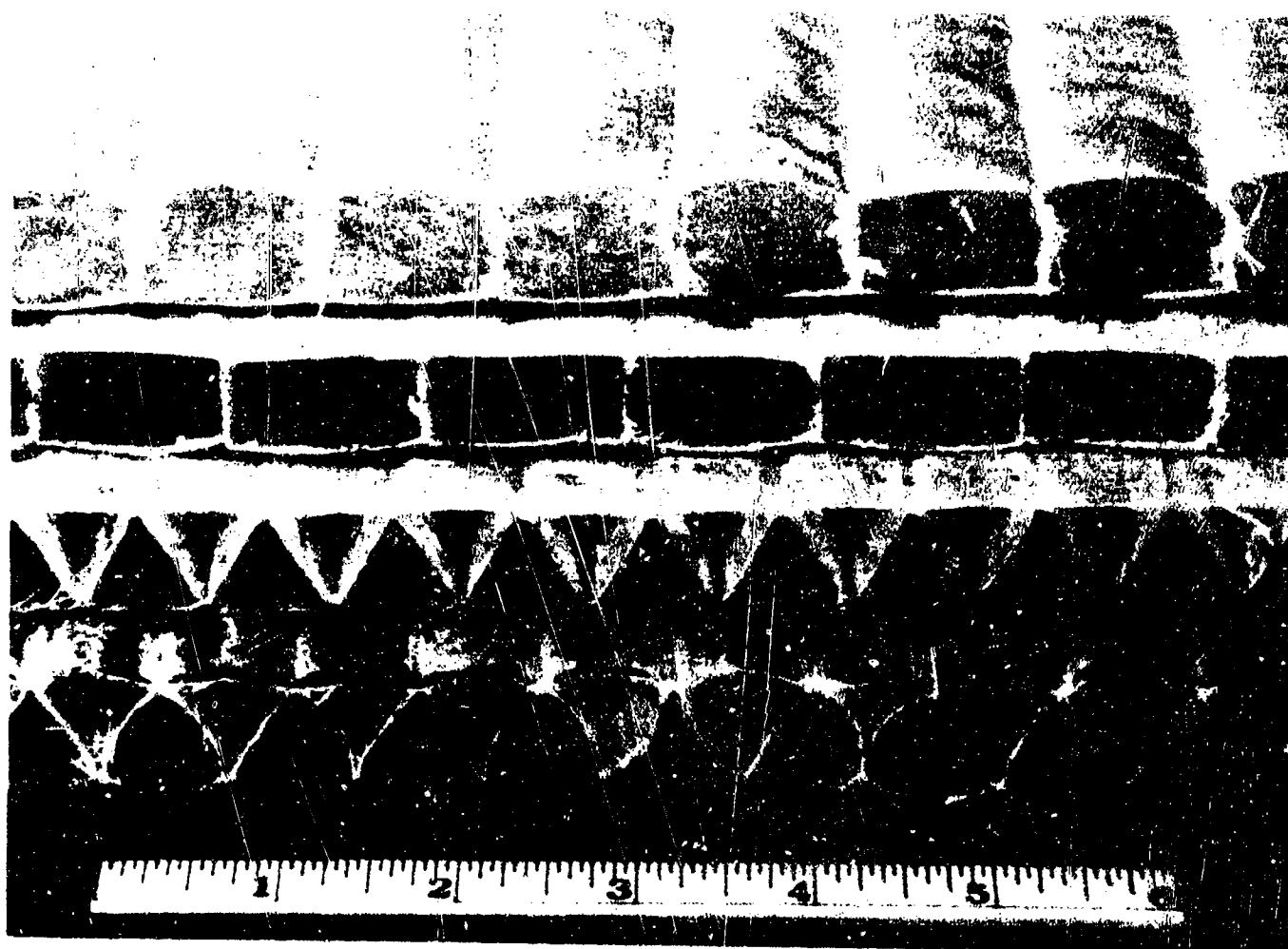


Figure 19. "Ideal" sample versus "regular" samples. (S40285)

PHYSICAL PROPERTIES DETERMINATIONS

After development of standardized processing techniques for each system a number of test specimens were prepared of each type. Initially the test pieces were prepared as 10" x 12" specimens, using the holding fixtures previously described. Each large sample was then used to obtain one 4 x 10 inch flexural test sample, one 5 x 5 inch flatwise compression sample and one 4 x 4 inch edgewise compression sample. An attempt was made to produce all the test samples of similar resin content and curing conditions in groups of three. However, since resin contents could only be estimated in the "wet" condition, after the ignition tests were run it was found that not all types of samples were produced in all the desired resin contents and fabric types. The majority of the samples were made with a resin content of 40 ± 5 percent. However, a number of samples were also made with 50 ± 5 and 60 ± 5 percent resin for comparison purposes.

Two types of samples were prepared. One type, designated "regular" were those cured under vacuum conditions using inflated polyethylene tubes for expansion. The second type, designated "Ideal", used the same resins and methods of impregnation as the "Regular" samples. However, solid polypropylene mandrels were used in the fabric flutes and the samples were cured between press platens under moderate pressure. These latter samples were then intended to represent the maximum in physical properties which might be secured from each system. Figure 19 shows the appearance of the two types of samples.

Test methods used were essentially those given in MIL Specification MIL STD 401A, "Sandwich Constructions and Core Materials; General Test Methods," except with regard to the sample sizes and configurations which were changed slightly in order to obtain better specimens. The flexural specimens met MIL STD 401A requirements in being 4 x 10 x 5/8 inches. The flatwise compression samples were made 5 x 5 inches (instead of 2 x 2) in order to ensure an adequate number of webs per sample. The edgewise compression samples met the size requirements of MIL STD 401A, but because the first samples showed edge failures, latter samples had the bearing edges encapsulated in either epoxy resin or Hydrocal plaster.

Flexural strength tests were run at a head speed of 0.05 inch per minute, using an eight inch span between two load points as shown in Figure 20. The following formulas were used, with the exception of the foam samples, for calculations for loads 90 degrees to the flute axes:

$$\text{flexural strength} = \frac{PS}{8Z\bar{w}a}$$

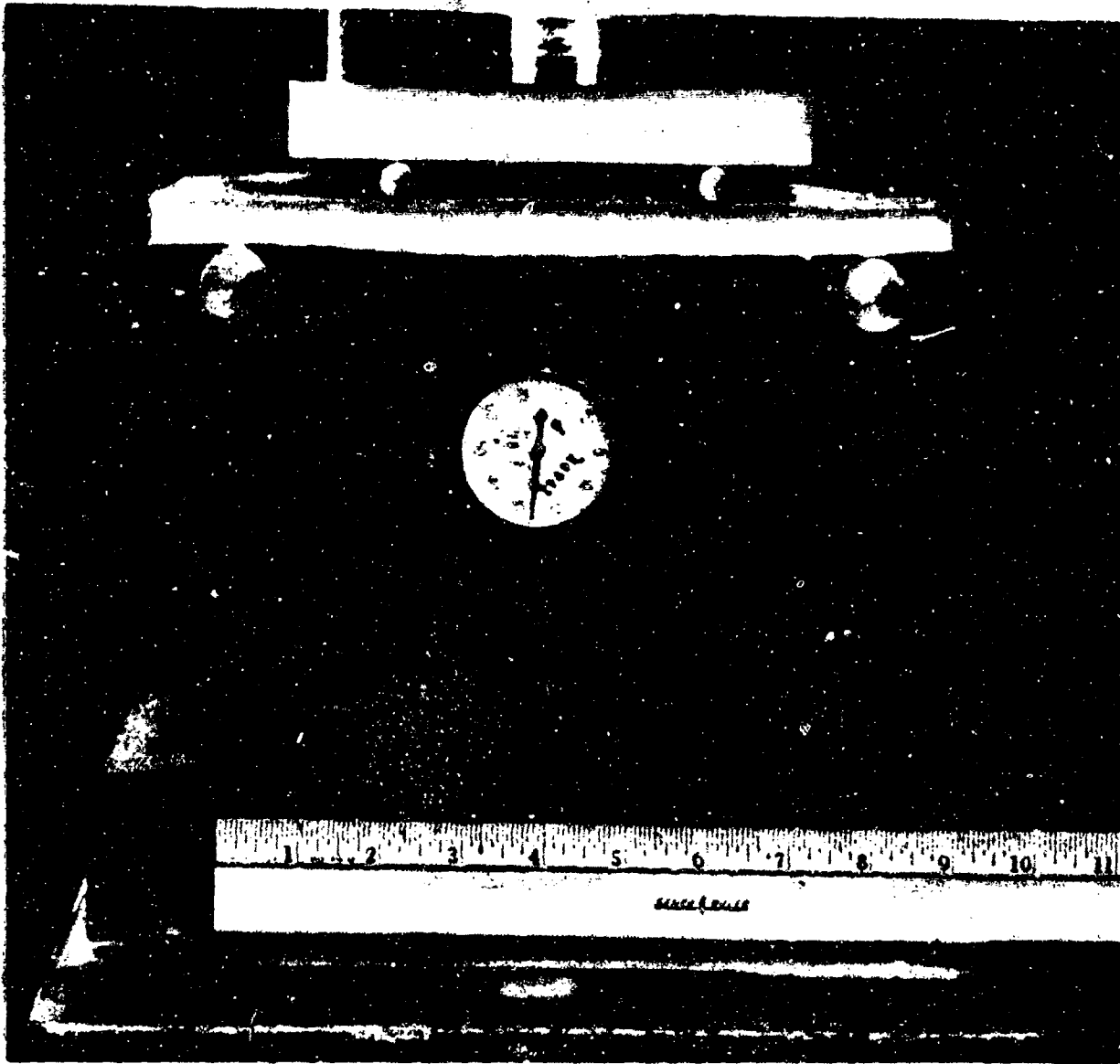


Figure 20. Flexural strength test set-up. (R95677)

where P = load
 S = span = 8 inches
 a = specimen width
 $Z\bar{W}$ = section modulus

The section modulus formulas were:

$$Z\bar{W}(\text{rectangular}) = \frac{t_f H^3 (\text{no webs/in}) + t_f (H^2 + 2Ht_f + t_f^2)}{H + t_f}$$

$$Z\bar{W}(\text{triangular}) = \frac{4t_f^3 + 2Ht_f^2 + H^2t_f + \frac{tw H^3}{6(t_w + H \sin \theta)}}{H + 2t_f}$$

where t_f = thickness of the facing
 H = specimen thickness less facings
 T_w = web thickness
 θ = web thickness

$$\text{flexural modulus} = \frac{PS^3}{128 (\delta_2 - \delta_1) Z\bar{W} Ta}$$

where δ = deflections at midpoints and load points
 T = specimen thickness

It should be pointed out that even though the formulas were used as given, the results were only approximate since the specimen thickness varied throughout the specimen, the angles were not constant and the facing and web thicknesses varied.

Flatwise compression tests were run at a head speed of 0.05 inch per minute. The ultimate flatwise compressive strength was calculated simply as:

$$f_c = \frac{P}{A}$$

where

P = maximum load

A = specimen area

The modulus was calculated as

$$E_c = \frac{PT}{A\delta}$$

where

δ = deflection

Edgewise compression tests were also run at a head speed of 0.05 inch per minute. The formula used to calculate the stress was as follows.

$$f_c = \frac{P}{A}$$

where

A = total area enclosed by the periphery of the specimen

The modulus was determined by the formula

$$E_c = \frac{Pb}{A\delta}$$

where

b = column length

δ = deflection

In determining the edgewise compression stress the actual stresses could have been determined by using only the areas of the facings and webs, in which case strengths would have appeared considerably higher. However, since it was desirable that comparisons be made between the various differing samples, it was felt using the same area in each case would be valid. These values then are in actuality load bearing values for each specimen, in terms of pounds per square inch. The appearance of these encapsulated samples is shown in Figure 21.

In all cases the tests were continued to the point of diminishing load, and an autographic record of deflections was made for each test so that modulus values might be obtained.

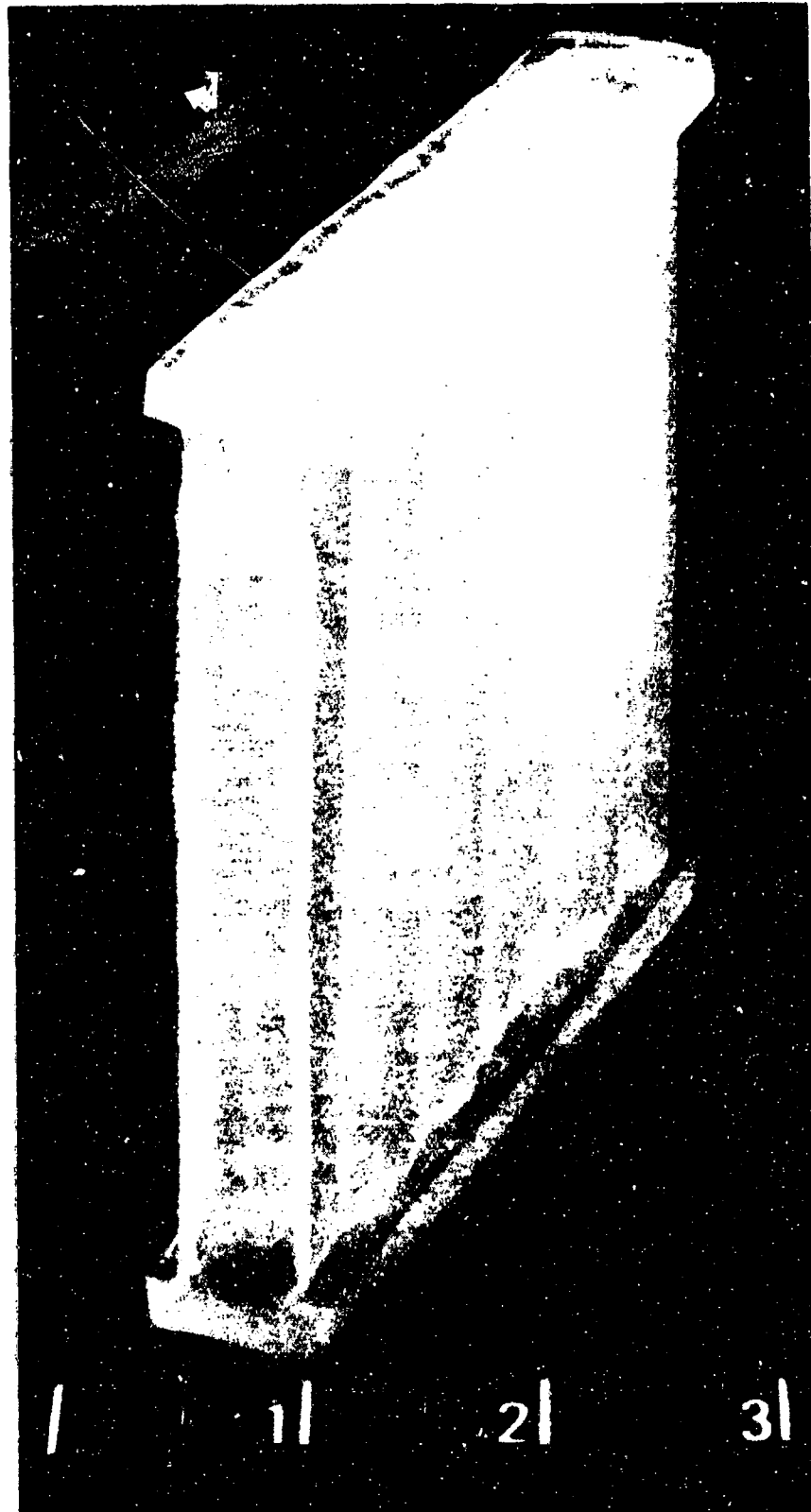


Figure 21. Encapsulated edgewise compression samples. (S40283)

One of the most important factors found to determine the final physical properties of the sample is the resin content.

A good deal of effort was therefore put into the resin content determinations. The system used for resin content determination, as previously described, consisted of obtaining small die cut samples from the large rigidized sample and then determining the resin content by ignition. In the initial tests, run on gelatin samples, three test pieces were used from each sample. In the final tests using all types of samples, one set of ignition test pieces was taken from each flexural, flatwise and edgewise sample, which thus gave three samples from each large rigidized specimen. Resin contents of the webs, and the top and bottom facings were all determined separately, so that an indication might be obtained if the fabric was being homogeneously impregnated.

In addition to determination of the resin content by ignition tests a correlation was obtained between resin contents obtained by ignition and those obtained by weights of the small die cut samples and of the entire rigidized sample. All the large samples were weighed before and after impregnation, and the individual samples were all weighed after being cut from the large rigidized sample. These weights were then compared with the values obtained in the ignition tests.

RESULTS

The results secured in the physical properties tests and the resin content determinations are shown in the following tables and charts.

Table 6 is a summary of the flexural strength and flexural modulus values obtained for the "Regular" samples.

The values for flatwise and edgewise compressive strengths are shown in Tables 7 and 8.

The data shown in Tables 6, 7 and 8 give information that may be used for preliminary design data. However, in order to show more graphically comparisons of a number of different factors, this data is also shown in the following charts. The charts show comparison in strengths between the various rigidization systems, the effects of varying resin contents and the differences produced by varying fabric configurations and weights. In preparing the charts, all the strength values obtained are shown as ordinates. Then, in order to show valid comparisons between the various systems, fabrics and resin contents, the abscissas are all shown as weight per unit of surface area, i.e., sample weights in terms of grams per square inch, since all the samples had a nominal thickness of 5/8 inch.

In the charts shown in Figures 22, 23 and 24, flexural strengths are given individually for each resin at varying resin contents. In addition each chart shows the spread of values obtained for both the "ideal" and the regular samples, and on the same charts, a comparison is shown between two, and in some cases, three different fabrics.

The charts shown in Figures 22 to 36, inclusive, graphically illustrate the results secured in the physical property tests. It should be noted that data shown are not presented as design data, but simply as information to indicate trends, the spread of results, and various types of comparisons.

The results of each type of test, e.g., flexural strength, are shown in two manners:

1. A group of three charts shows, for each resin, the comparisons between sample weight (resin content) and the ultimate flexural strength for each type of fabric.
2. A group of two charts allows comparisons between the resin systems, using the same fabrics, at varying resin contents.

Thus in Figure 22 the resin system being tested was the polyester. The chart then shows the relationship between ultimate flexural strengths and sample weights for the three different fabrics. The chart also allows a comparison between the various fabrics, when impregnated to

approximately equal weights. Further comparisons are shown between "ideal" vs "regular" samples. In a similar manner Figure 23 shows the results of the same type tests on polyurethane impregnated samples, and Figure 24 shows the results of gelatin impregnated samples.

The next group of two charts, Figures 25 and 26, were made using the same data as used in preparing the charts in Figures 22, 23 and 24. However, the data was arranged differently. In these two charts the three resin systems are compared, using the same fabrics. Thus these charts allow comparison between resin systems, using samples of the same fabric impregnated to approximately the same resin contents.

The second group of five charts deals with edgewise compressive strengths and is shown in Figures 27 to 31 inclusive. In this group Figures 27, 28 and 29 show the comparison between various fabrics and given resins and Figures 30 and 31 then allow comparison between various resin systems for given fabrics.

The third group of charts shown in Figures 32 to 36, inclusive, gives the same type of information for flatwise compressive strengths.

Persual of the charts then indicates the following:

1. For all the samples there appeared to be a fairly straight line relationship between ultimate strength and sample weight, up to the maximum prepared.
2. Samples with resin contents ranging from 60 to 70 percent show higher strengths, in all cases, than samples with 30 to 40 percent resin content. This is contrary to experience with conventional fiberglass samples made as "normal" i. e., pressurized laminates. Control of resin content is evidently a crucial factor, particularly with gelatin.
3. In most cases the gelatin samples show a greater spread in values than do corresponding polyester or polyurethane samples. This then indicates that more refinement is required in impregnation and the control of resin content. It is to be expected that polyester and polyurethane, being conventional resin systems, would yield more readily to control than gelatin at this stage in the development. However, it should be noted that the upper ranges of strength and stiffness for gelatin are often considerably greater than the other systems, indicating that gelatin may be superior when its processing is fully developed.
4. Examination of charts showing fabric comparisons indicates that in most cases the rectangular fabric samples appear slightly superior to the same weight of triangular fabric sample.
5. There appears to be no definite, overall physical properties superiority for any one resin system, in any of the tests, with the exception that gelatin shows a greater spread and many higher upper limits than the other systems.

TABLE 6. FLEXURAL STRENGTH - 12 OZ TRIANGULAR FABRIC

Resin	Sample Type	Resin Content, Percent											
		30 ±5				40 ±5				50 ±5			
		Flexural Str. psi	Flexural Mod. psi	Apparent Density lb/ft ³	Specific Strength*	Flexural Str. psi	Flexural Mod. psi	Apparent Density lb/ft ³	Specific Strength	Flexural Str. psi	Flexural Mod. psi	Apparent Density lb/ft ³	Specific Strength
Polyester	Regular	-	-	-	-	720	3.93×10^4	2.7	452,700	780	1.6×10^5	4.2	321,400
Polyester	Ideal	188	4.9×10^4	2.2	152,200	-	-	-	-	526	4.8×10^5	3.2	285,100
Polyurethane	Regular	-	-	-	-	354	3.3×10^4	2.9	214,200	753	2.9×10^4	3.5	371,500
Polyurethane	Ideal	707	9.5×10^4	2.4	509,760	657	3.9×10^4	3.0	378,400	-	-	-	-
Gelatin	Regular	299	1.5×10^4	2.3	224,640	312	1.8×10^4	2.8	191,800	978	6.1×10^5	3.0	561,600
Gelatin	Ideal	-	-	-	-	-	-	-	-	950	2.8×10^5	3.4	482,100
14 OZ Rectangular Fabric													
Polyester	Regular	214	6.98×10^5	2.7	136,700	570	1.8×10^5	3.0	328,300	674	3.1×10^4	3.8	305,800
Polyester	Ideal	356	1.3×10^5	2.6	236,700	-	-	-	-	-	-	-	-
Polyurethane	Regular	258	4.8×10^4	2.7	165,000	712	2.4×10^4	3.1	397,400	966	2.1×10^4	4.0	416,400
Polyurethane	Ideal	656	8.2×10^4	2.7	419,900	878	8.8×10^4	3.1	489,000	-	-	-	-
Gelatin	Regular	463	2.6×10^4	2.5	319,700	-	-	-	-	1731	2.3×10^4	3.7	808,700
Gelatin	Ideal	-	-	-	-	-	-	-	-	704	1.7×10^5	3.7	328,300
*Specific strength calculated as the ratio of ultimate strength in lb. per in. ² to specific gravity in lb. per in. ³													

TABLE 7. FLATWISE COMPRESSION — 12 OZ TRIANGULAR FABRIC

Resin	Sample Type	Resin Content, Percent											
		30 ±5				40 ±5				50 ±5			
		Com- pressive Str. psi	Com- pressive Mod. psi	Apparent Density lb./ft. ³	Specific Strength ^a	Com- pressive Str. psi	Com- pressive Mod. psi	Apparent Density lb./ft. ³	Specific Strength	Com- pressive Str. psi	Com- pressive Mod. psi	Apparent Density lb./ft. ³	Specific Strength
Polyester	Regular	—	—	—	—	0.72	5.6	2.9	432	—	—	—	—
Polyester	Ideal	0.68	2.2	2.3	518	0.64	1.0	2.6	432	—	—	—	—
Polyurethane	Regular	—	—	—	—	2.00	4.7	2.8	1226	—	—	—	—
Polyurethane	Ideal	0.96	9.3	2.3	726	1.1	11.0	2.6	725	1.2	10.1	3.2	657
Gelatin	Regular	0.62	4.1	2.4	449	2.5	6.6	2.9	1486	1.7	26.1	3.2	915
Gelatin	Ideal	—	—	—	—	—	—	—	—	—	—	—	—
14 OZ Rectangular Fabric													
Polyester	Regular	0.68	2.6	2.7	432	1.2	3.5	3.3	622	3.8	13.1	3.6	1814
Polyester	Ideal	2.0	3.8	2.8	1244	—	—	—	—	9.3	18.3	3.9	5529
Polyurethane	Regular	0.9	3.2	2.7	570	2.9	6.4	3.0	1676	8.5	22.2	3.9	3600
Polyurethane	Ideal	—	—	—	—	2.8	37.2	2.8	1728	—	—	—	—
Gelatin	Regular	0.26	2.1	2.7	167	—	—	—	—	6.0	48.5	4.0	2592
Gelatin	Ideal	—	—	—	—	—	—	—	—	1.1	5.1	3.6	518
HAC Foam													
										542.0 [±]	—	7.0	134,000

^aSpecific strength --- (see footnote on table 6).

[±]Foam samples had a resin content of 72 percent.

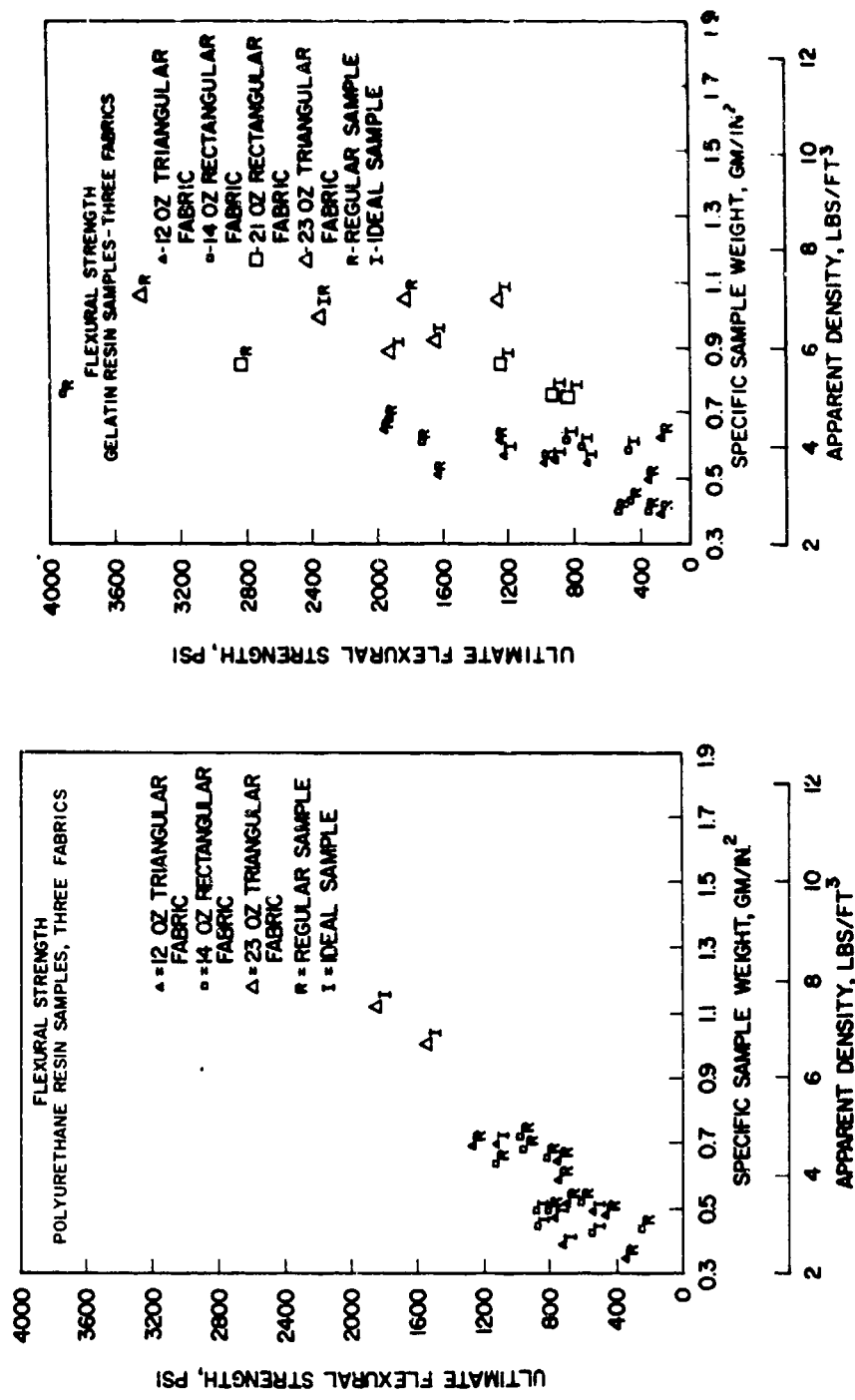


Figure 22. Flexural strengths versus specific sample weight—polyester resin.

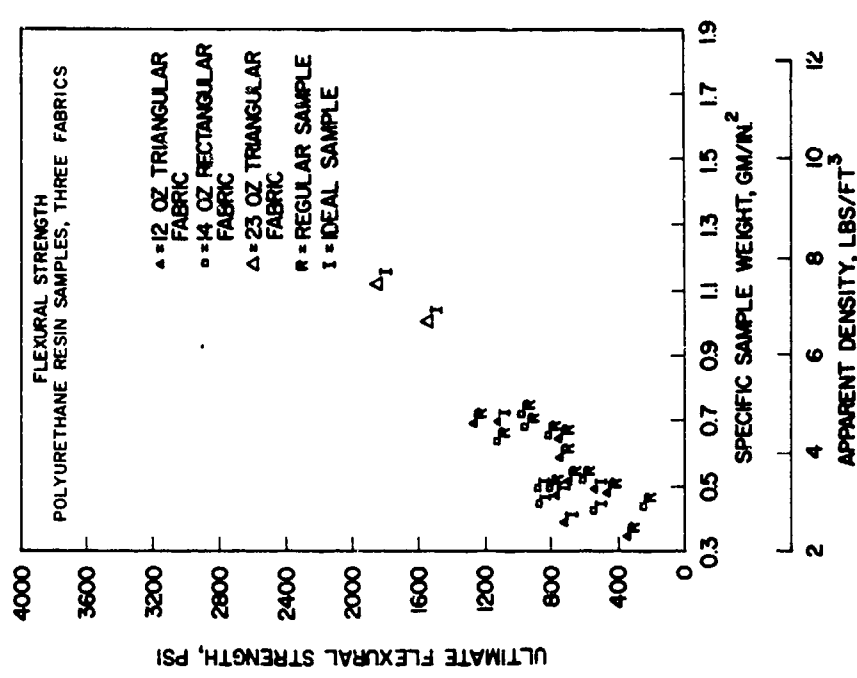


Figure 23. Flexural strengths versus specific sample weight—polyurethane resin.

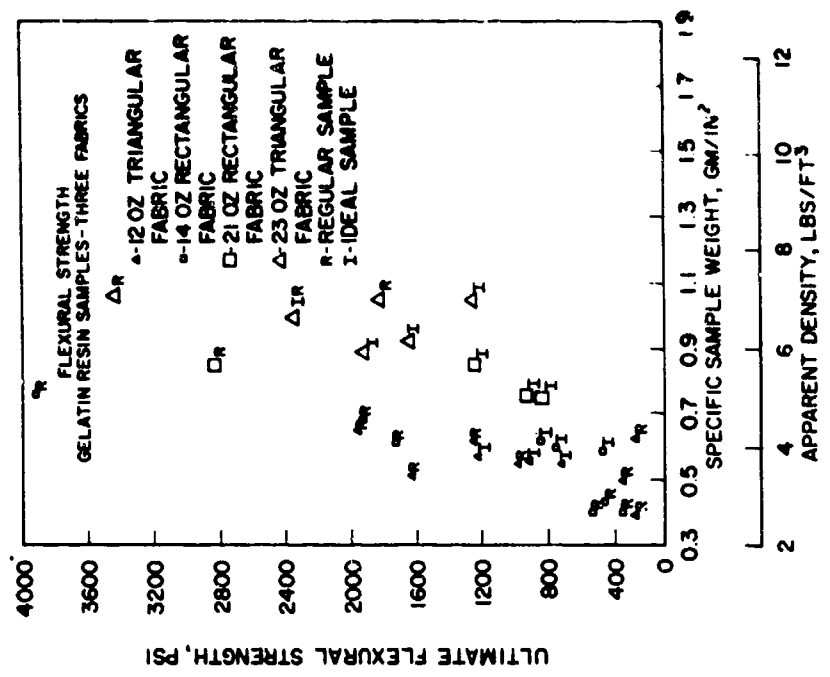


Figure 24. Flexural strengths versus specific sample weight—gelatin resin.

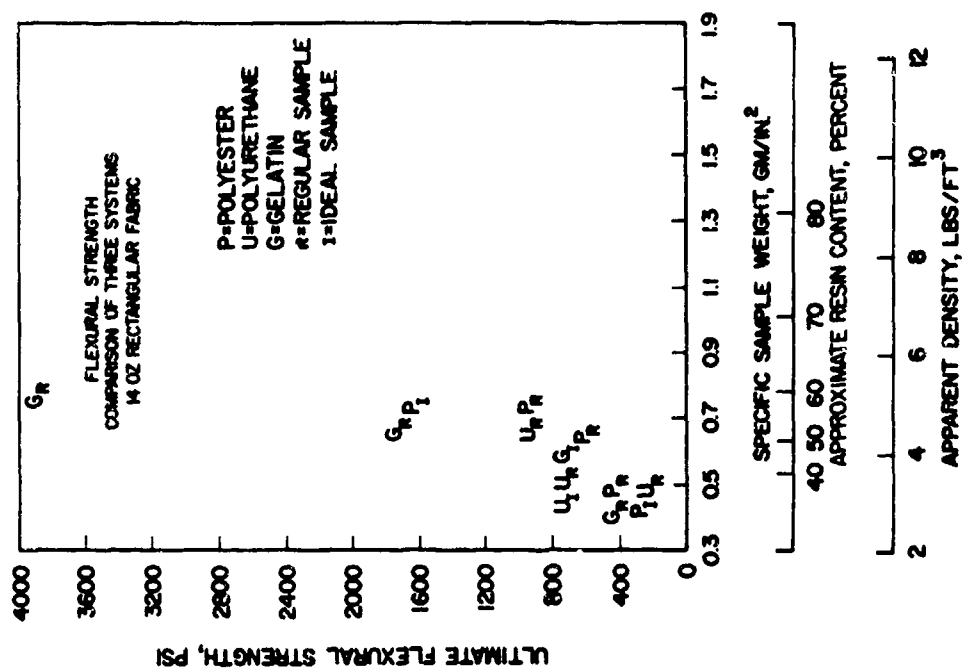


Figure 26. Comparison of flexural strengths—14 ounce rectangular fabric.

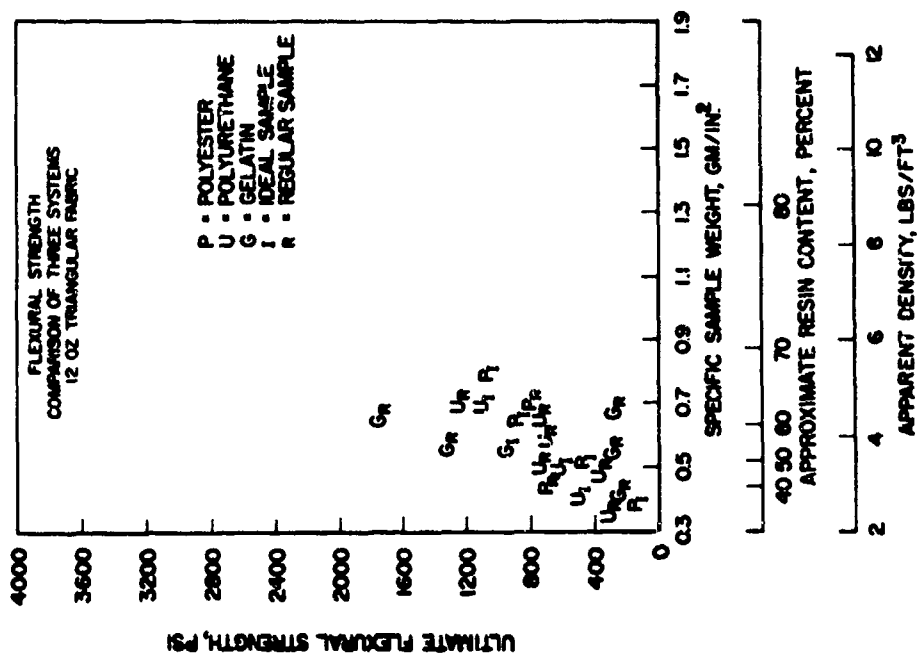


Figure 25. Comparison of flexural strengths—12 ounce triangular fabric.

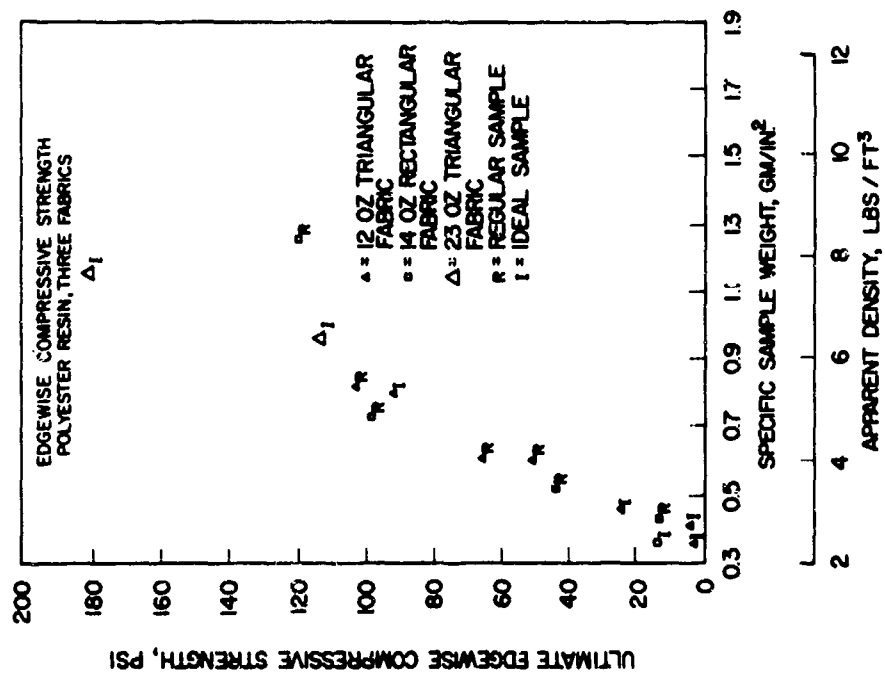


Figure 27. Edgewise compressive strengths—polyester resin.

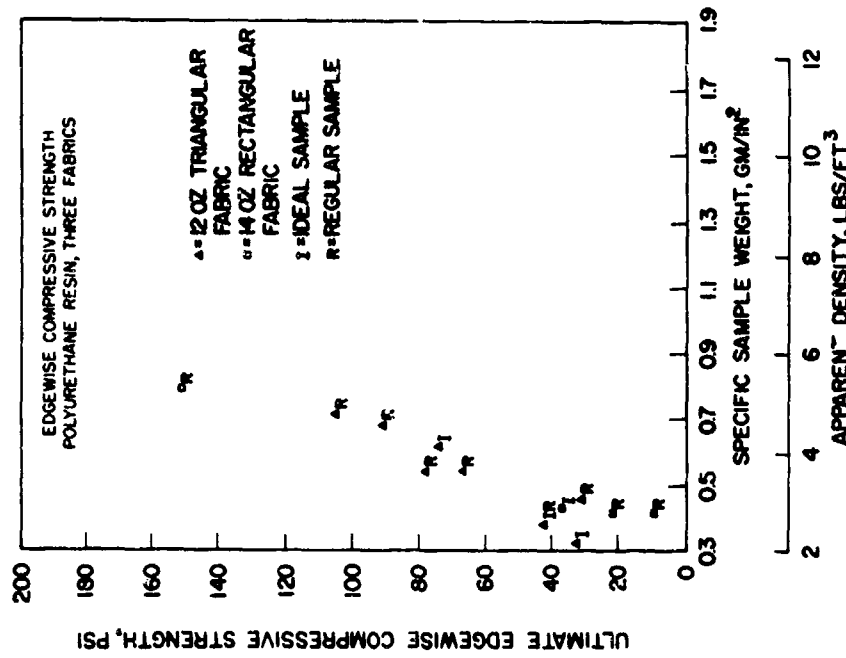


Figure 28. Edgewise compressive strengths—polyurethane resin.

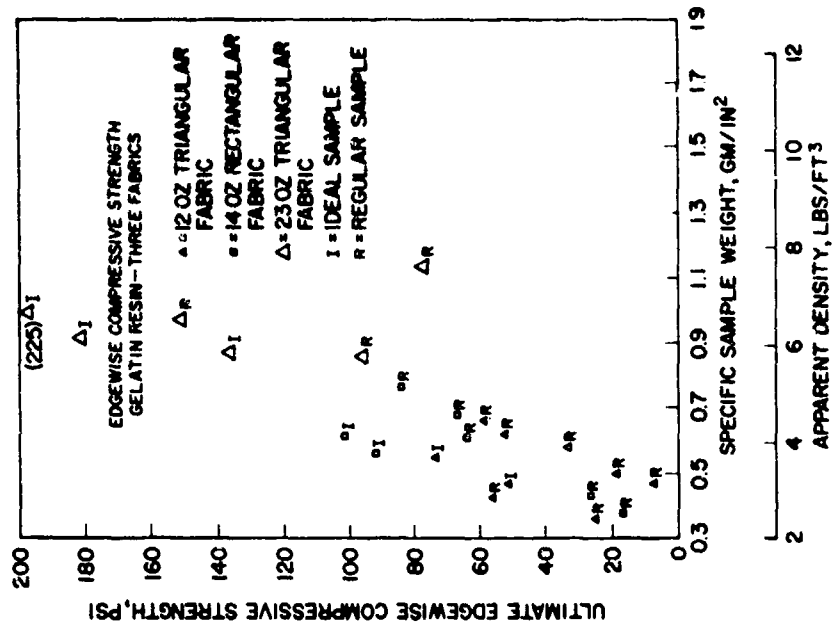


Figure 29. Edgewise compressive strengths—gelatin resin.

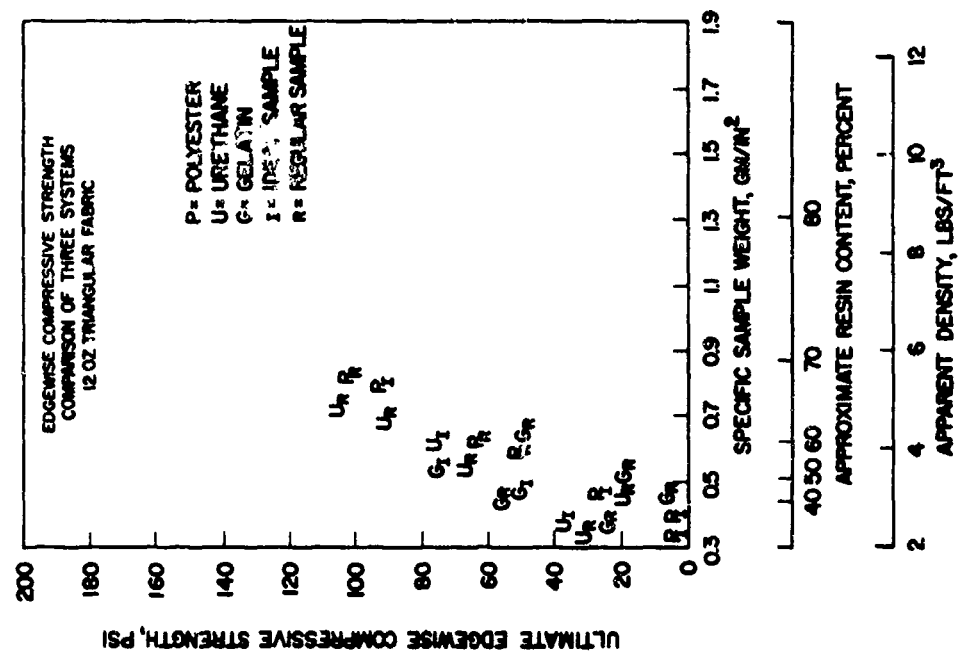


Figure 30. Comparison of edge-wise compressive strengths -- 12 ounce triangular fabric.

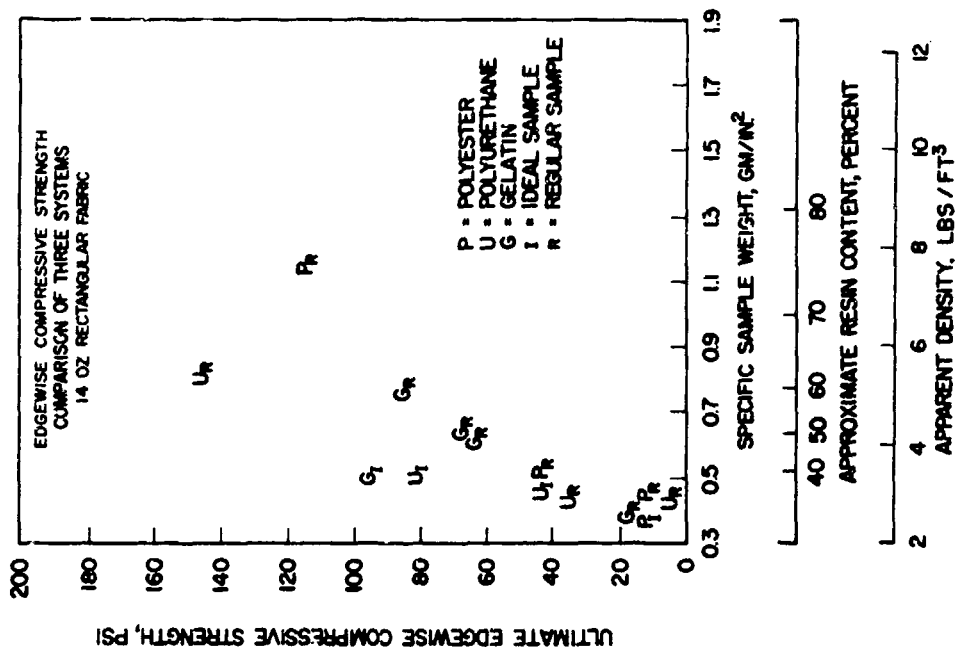


Figure 31. Comparison of edge-wise compressive strengths -- 14 ounce rectangular fabric.

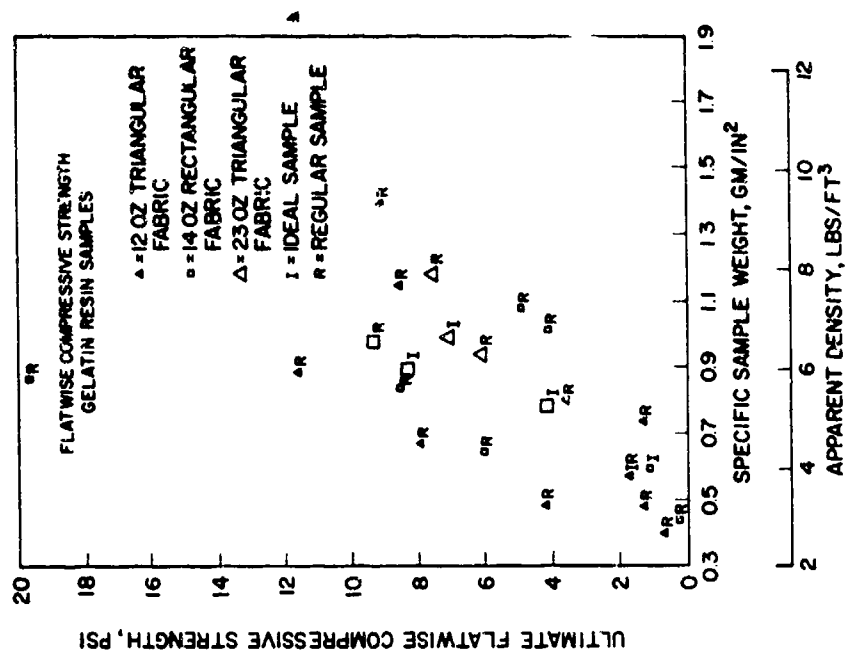
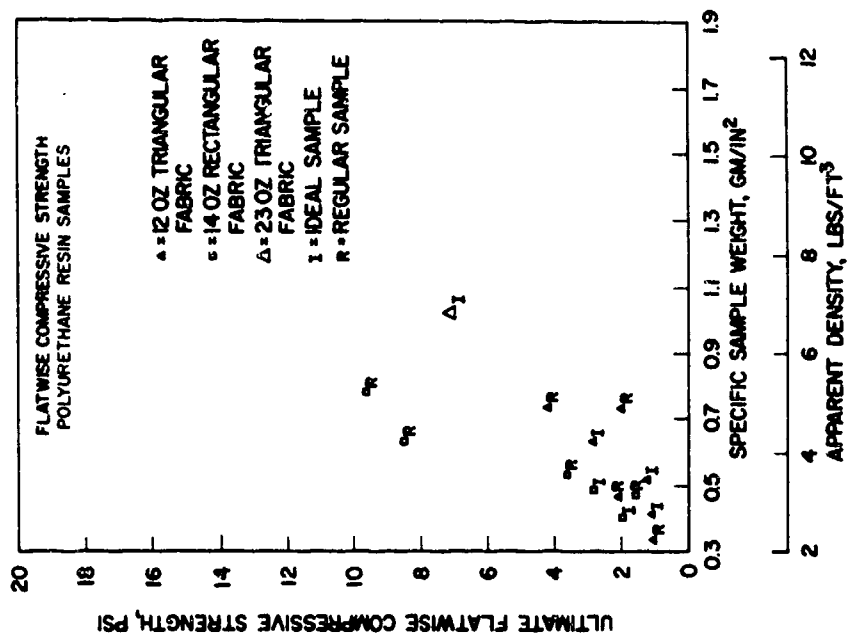
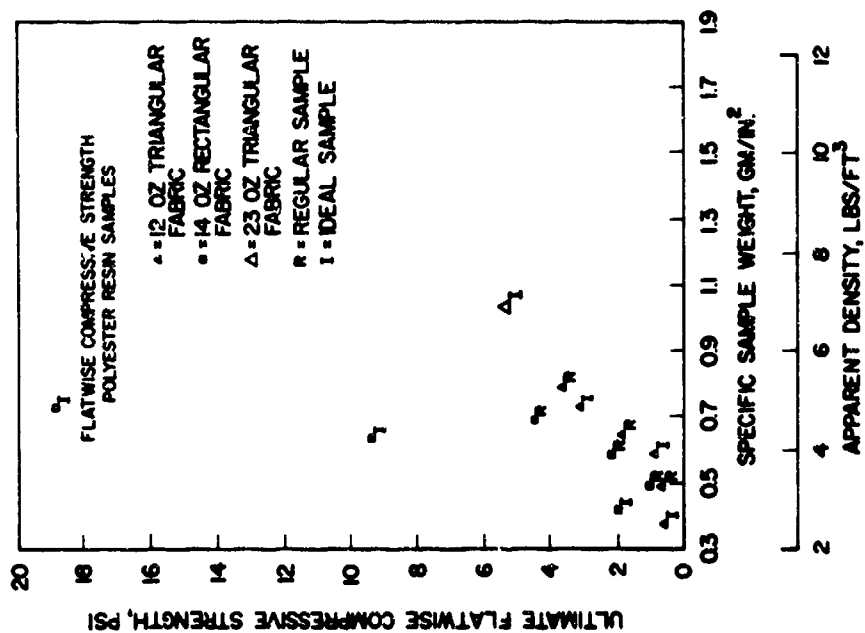


Figure 32. Flatwise compressive strengths—polyester resin.

Figure 33. Flatwise compressive strengths—polyurethane resin.

Figure 34. Flatwise compressive strengths—gelatin resin.

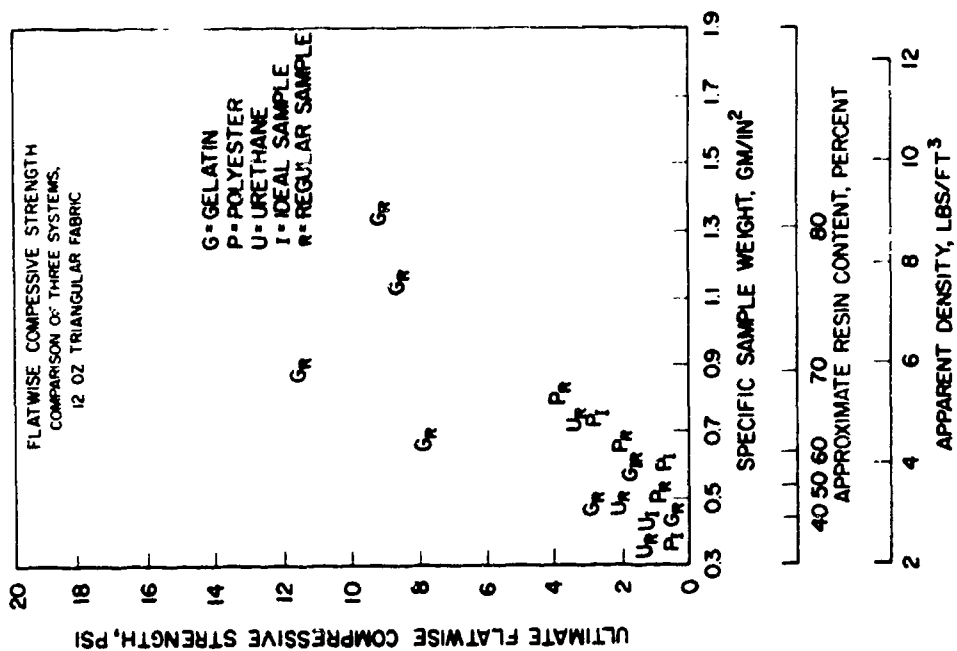


Figure 35. Comparison of flatwise compressive strengths -- 12 ounce rectangular fabric.

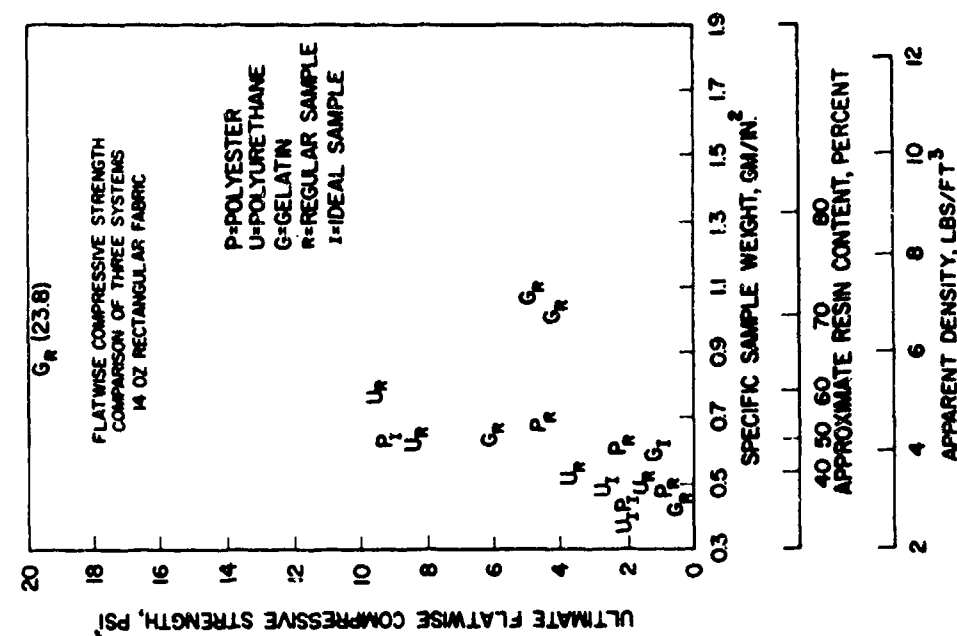


Figure 36. Comparison of flatwise compressive strengths -- 14 ounce rectangular fabric.

RESIN CONTENT DETERMINATIONS

The results of the resin content determinations are shown in Tables 9 and 10. In Table 9, resin content determinations are shown for 45 samples, representing all different types of samples. It should be noted that for each sample, nine determinations were made, three each from the edgewise compression, flatwise compression and flexural strength samples. In each case one sample was taken from the top facing, the bottom facing and the webs. The entire data for this group of samples is shown in order that the spread may be noted.

In compiling the data for Table 9 a considerable amount of time was required for the initial sample weighing, ignition, final sample weighing and the calculations to determine the resin content. Early in the determinations it was decided to use uniform samples, which would be die stamped from the cured, three dimension samples. The type of samples thus secured are shown in Figure 13. In each case the weight of three uniform discs was used for each ignition test. These weights are then charted against the corresponding resin contents as shown in Figures 37 and 38. A definite correlation was found. This then indicates that this technique could be used for resin content determinations with a considerable saving in time and labor.

The average resin content of each sample as shown by ignition tests was also compared to the resin content calculated from the total weights of the three physical property test samples. This comparison is shown in Table 10. Like the previous comparisons, a definite correlation was believed found, well within experimental limits and within the limits of weight variation as shown by the fabric. This data also indicates that a weighing technique could be used to determine resin contents.

TABLE 9. RESIN CONTENT DETERMINATIONS BY IGNITION

Samples		Top Facing Samples				Bottom Facing Samples				Web Samples			
Nos.	Types	Edge. Comp.	Flat. Comp.	Flex. Str.	Aver.	Edge. Comp.	Flat. Comp.	Flex. Str.	Aver.	Edge. Comp.	Flat. Comp.	Flex. Str.	Aver.
183	P12ΔR	57.5	63.2	61.6	61.0	67.1	63.4	63.6	65.0	58.2	57.6	62.8	59.8
184	P12ΔR	63.3	65.0	65.7	66.3	65.9	63.0	53.8	61.2	58.3	35.0	66.2	53.4
					63.6				63.1				56.6
185	P12ΔI	51.0	54.9	54.7	53.8	58.7	53.4	48.0	53.6	47.4	52.4	51.0	50.0
188	P12ΔI		31.3	41.3	36.5		23.7	36.6	30.2		30.6	26.3	28.6
190	P12ΔI	42.3	42.6	39.1	41.6	44.3	44.2	48.3	45.9	32.9	30.0	32.9	32.2
					43.9				43.2				37.1
187	P14□R	47.9	47.5	45.6	47.3	51.9	47.0	51.9	50.5	44.0	42.0	45.5	44.1
182	P14□R	49.3	46.6	43.1	46.6	49.2	45.8	47.6	47.8	45.1	37.8	39.9	41.2
196	P14□R	46.7	39.2	36.4	41.0	45.8	48.2	46.2	47.0	37.9	36.3	32.9	36.0
					44.9				48.4				40.4
193	P14□I	38.0	41.5	40.9	38.2	42.0	39.5	40.2	33.1	31.7	31.7	36.2	33.9
198	U12ΔR	42.1	58.5	47.8	49.7	42.7	45.4	45.4	44.8	41.6	43.8	32.2	39.5
224	U12ΔR	60.5	63.0	63.2	62.5	54.3	59.7	67.7	69.1	44.7	50.0	43.6	46.1
225	U12ΔR	76.0	68.1	73.1	72.5	61.7	72.2	63.9	66.2	52.0	58.8	47.1	52.6
226	U12ΔR	66.4	60.5	58.6	62.1	70.5	75.5	67.8	71.5	43.1	46.0	34.4	41.2
					61.7				61.7				44.9
199	U12ΔI	42.9	44.9	41.6	43.4	44.2	43.9	41.9	43.6	37.6	37.8	37.9	37.8
195	U12ΔI	49.7	52.0	47.9	50.1	51.3	57.7	57.1	57.6	40.6	45.6	46.4	44.5
					46.8				50.6				41.2
215	U14□R	62.5	67.2	58.7	63.1	59.4	57.0	62.3	59.8	63.9	57.3	57.4	59.5
216	U14□R	55.1	58.8	60.9	58.5	63.1	59.0	69.0	64.0	60.9	62.5	51.8	58.4
217	U14□R	59.1	59.8	59.9	59.9	61.2	62.3	59.2	61.2	61.7	65.5	61.1	62.8
					60.5				61.7				60.2
219	G12ΔR	54.3	58.8	69.7	59.5	61.7	63.4	62.6	62.8	50.4	65.9	48.0	54.8
220	G12ΔR	56.9	60.1	56.0	57.9	59.5	61.4	57.9	59.9	47.9	68.6	49.0	55.2
221	G12ΔR	69.6	69.7	52.2	62.4	54.2	49.9	58.0	54.3	54.0	46.1	55.6	51.9
222	G12ΔR	52.0	55.7	50.3	52.9	67.0	66.8	63.5	66.0	57.2	49.8	50.2	52.4
223	G12ΔR	52.4	54.5	44.8	50.8	38.5	67.9	58.3	55.2	39.1	56.6	46.8	47.5
					56.7				59.6				52.4
233	G12ΔI	64.6	58.5	67.5	63.5	59.5	61.7	48.3	56.5	48.1	48.5	65.5	54.3
234	G12ΔI	54.3	64.5	60.6	59.8	67.1	67.2	68.2	67.5	48.9	54.3	54.6	52.6
235	G12ΔI	56.2	62.1	58.8	59.0	56.5	64.5	62.5	61.2	46.9	52.1	48.2	49.1
					60.8				61.7				51.9
212	G14□R	59.4	59.9	53.9	58.0	58.9	57.3	48.3	55.1	64.2	66.3	59.5	63.3
213	G14□R	55.5	58.3	54.5	56.4	51.4	58.2	42.1	50.8	52.3	71.6	63.8	62.6
214	G14□R	59.8	44.7	56.7	54.0	62.3	53.5	57.7	58.1	62.2	71.1	67.4	66.9
					56.1				65.7				63.9
236	G14□I	56.0	58.8	60.1	58.3	57.7	57.7	56.5	57.3	43.6	46.4	45.1	45.0
237	G14□I	55.5	59.8	60.2	58.5	59.4	57.8	63.0	60.1	45.5	44.8	47.6	46.0
238	G14□I	59.7	61.5	62.9	61.4	59.7	57.5	60.6	59.3	43.2	46.1	47.5	45.8
					59.4				58.9				45.6
206	G23ΔR	51.7	61.2	55.9	56.5	49.6	47.2	46.7	48.1	41.6	41.6	41.5	41.6
208	G23ΔR	49.8	58.7	43.1	50.8	40.3	60.0	56.0	52.4	37.9	49.9	49.2	45.7
218	G23ΔR	57.4	54.7	48.4	53.8	64.5	55.7	55.0	58.7	57.3	40.7	46.4	48.1
					53.7				53.1				45.1
227	G23ΔI	49.4	49.7	48.0	49.3	47.9	40.5	47.6	45.6	36.1	34.6	36.3	35.6
228	G23ΔI	57.4	45.3	52.1	51.9	47.7	47.3	48.5	48.1	41.7	57.5	36.8	45.3
229	G23ΔI	52.5	55.6	54.1	54.3	50.0	50.2	52.3	51.1	32.9	49.7	41.8	41.5
					51.8				48.3				40.8
203	G21□R	39.3	31.4	45.1	38.9	38.9	45.0	37.8	41.2	51.0	47.3	42.8	47.0
205	G21□R	36.3	46.7	49.5	44.4	67.7	49.8	49.8	56.0	50.8	46.9	39.9	45.9
207	G21□R	60.7	57.4	56.0	58.3	52.5	49.4	37.8	46.8	51.6	56.1	57.5	55.1
					47.2				48.0				49.0
230	G21□I	48.2	47.2	47.5	47.6	50.1	49.3	51.9	50.4	41.4	41.4	37.3	40.0
231	G21□I	48.0	48.8	47.5	48.1	48.0	48.2	48.5	48.2	37.0	37.1	38.4	37.5
232	G21□I	53.3	52.5	53.7	53.2	55.5	51.5	54.0	53.7	42.2	57.3	44.4	48.0
					49.6				50.8				41.8

TABLE 10. COMPARISON OF RESIN CONTENTS BY IGNITION
VERSUS SAMPLE WEIGHTS

Sample No.	Type	Resin Content, Percent		Difference. Percent
		Ignition	Sample Wt.	
183	P12 Δ R	62.9	63.3	+ 0.4
184	P12 Δ R	64.6	60.0	- 4.6
185	P12 Δ I	52.6	51.4	- 1.2
188	P12 Δ I	31.8	28.0	- 3.8
190	P12 Δ I	39.9	32.0	- 7.9
182	P14 □ R	45.2	40.0	- 5.2
187	P14 □ R	47.3	42.0	- 5.3
196	P14 □ R	41.3	36.5	- 4.8
191	P14 □ I	38.2	31.5	- 6.7
198	U12 Δ R	44.7	45.5	+ 0.8
224	U12 Δ R	57.6	59.5	+ 1.9
225	U12 Δ R	63.8	63.4	- 0.4
226	U12 Δ R	58.3	61.3	+ 3.0
199	U12 Δ I	41.6	33.8	- 7.8
195	U12 Δ I	50.7	42.0	- 8.7
215	U14 □ R	60.8	59.5	- 1.3
216	U14 □ R	60.3	59.0	- 1.3
217	U14 □ R	60.7	59.0	- 1.7
219	G12 Δ R	59.0	60.2	+ 1.2
220	G12 Δ R	57.7	61.5	+ 3.8
221	G12 Δ R	56.2	49.5	- 6.7
222	G12 Δ R	57.1	58.2	+ 1.1
223	G12 Δ R	51.2	51.0	- 0.2
233	G12 Δ R	56.1	52.0	- 6.1
234	G12 Δ R	60.0	54.8	- 7.2
235	G12 Δ R	56.4	52.0	- 4.2
212	G14 □ R	58.8	52.8	- 6.0
213	G14 □ R	56.6	---	---
214	G14 □ R	59.7	64.2	+ 4.5
236	G14 □ I	53.5	48.3	- 5.2
237	G14 □ I	54.9	49.8	- 5.1
238	G14 □ I	55.5	51.0	- 4.5
206	G23 Δ R	48.7	49.1	+ 0.4
208	G23 Δ R	49.6	55.6	+ 6.0
218	G23 Δ R	53.5	54.0	+ 0.5
227	G23 Δ I	43.5	44.8	+ 1.3
228	G23 Δ I	48.4	50.0	+ 1.6
229	G23 Δ I	49.0	46.8	- 2.2
203	G21 □ R	42.4	---	---
204	G21 □ R	48.7	56.1	+ 7.4
207	G21 □ R	53.4	52.5	- 0.9
230	G21 □ I	46.0	41.0	- 5.0
231	G21 □ I	44.6	44.2	- 0.4
232	G21 □ I	51.6	44.2	- 7.2

Figure 37. Resin content versus facing disc sample weight.

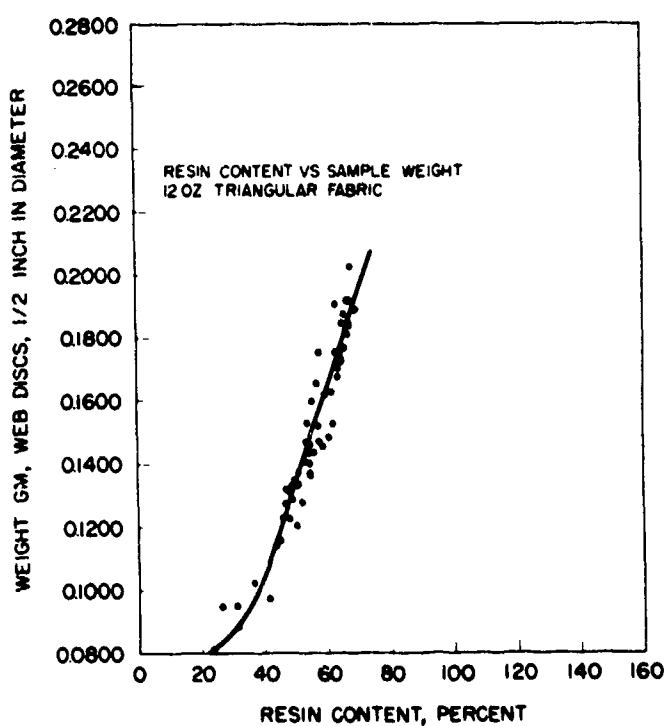
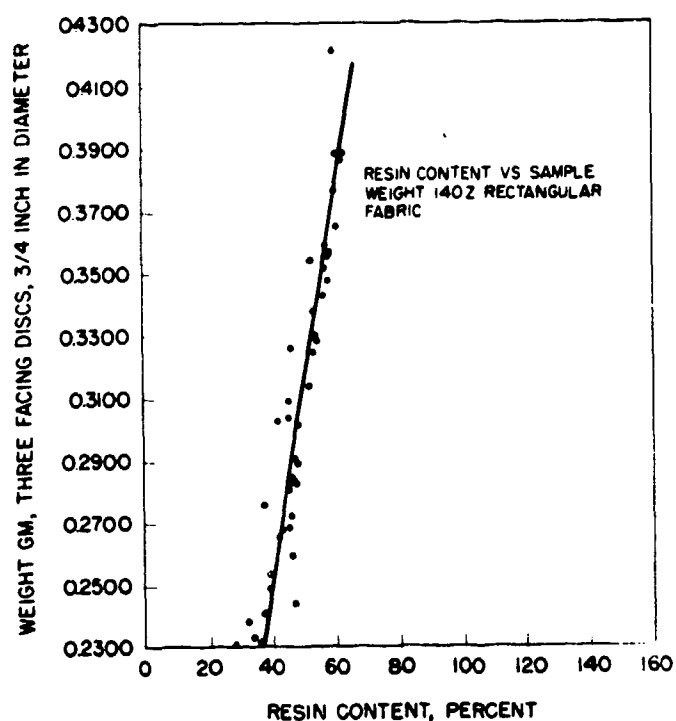


Figure 38. Resin content versus web disc sample weight.

DISCUSSION

Although the strength figures obtained for both the "regular" and "ideal" samples appear low when compared to conventional high density engineering materials, the strength levels obtained are quite high when compared on a density or specific strength basis. For instance, all the materials tested fall into the range 2.0 to 10 lbs/ft³ overall density. The only similar structural materials of similar density are rigid plastic foams, balsa wood, and very lightweight honeycomb sandwiches. An informal comparison of the strengths of other light weight materials to typical values taken from the test results from this project is shown in Table 11. Although direct comparison of these figures must be done with caution because of differences in test method and calculation of results, the data shows that the space erected and cured sandwiches are structurally similar to other lightweight materials produced under far more ideal conditions.

In assessing the values obtained in the physical property tests of the "Regular" samples two factors should be borne in mind; (1) that the samples were not pressurized during cure and (2) that the samples were erected by relatively low pressure inflation tubes. The fact that the samples were not pressurized was partly responsible for the relatively wide spread in values obtained, since laminates made under higher pressures are inherently more homogeneous than those made under low pressures. An even more serious effect of the lack of pressure is that the reinforcement fibers were not closely held together by the resin, as in the case of "normal" reinforced or laminated parts. Close examination of the samples indicated, in fact, many bubbles and discontinuities in all the resin binders, as a result of the vaporization of the volatile constituents in each material. Under such circumstances, then it was to be expected that the physical property values would be somewhat lower than might be expected from "normal" structures.

The fact that the samples were three dimensional structures, erected by inflation tubes, also contributed to a good deal of the non-homogeneity. In this case it was found, even though all the inflation tubes theoretically had the same pressures that probably because of nonhomogeneous impregnation and irregularities in the fabric, the structure was not erected in the optimum shape; i. e., with all the webs and facings pulled tight. Because of these shape irregularities then the physical properties were again somewhat lower than was found with the "Ideal" samples. The surprising result was not how low the values were, but rather how near to the "Ideal" samples the results were:

All of the fabrics were used in the as received "grey goods" condition; i. e., with the weaving lubricants and starch still on the yarns. The material was furnished in this condition by error, and was not so specified. The use of special finishes on the fabric should possibly raise the values by 30-40%.

TABLE 11. COMPARISON OF PHYSICAL PROPERTIES OF LIGHT-WEIGHT STRUCTURAL MATERIALS

Material Description	Apparent Density lbs/ft ³	Compressive Strength lbs/in ²	*Specific Compressive Strength	Flexural Strength lbs/in ²	*Specific Flexural Strength
Rigid Polyurethane Foam ⁽¹⁾	4	100	4,000	180	78,000
Rigid Polyurethane Foam ⁽¹⁾	8	250	54,000	350	76,000
Balsa Wood ⁽²⁾	8.8	1000	197,000	2200	32,000
Honeycomb Core Aluminum Hexcel AL 1/8-3003-0007 P	3.1	210 Bare Flatwise	117,000	125 (Avg) ⁽³⁾	70,000
Honeycomb Core Aluminum Hexcel AL 3/16-5052-003 P	8.1	1160 Bare Flatwise	247,000	1200 (Avg) ⁽³⁾	250,000
Honeycomb Sandwich Fiberglas Hexcel NP 3/16-112-4.5	4.5	440 Bare Flatwise	170,000	155 (Avg) ⁽³⁾	60,000
Honeycomb Sandwich Fiberglas Hexcel NP 1/4-21-8.0	8.0	1300 Bare Flatwise	281,000	475 (Avg) ⁽³⁾	105,000
Fabricated Honeycomb Sandwich ⁽⁴⁾ 1/4 thick, .003 MIL Epoxy Fiberglas Faces AL 3/16-5052-0007 P Core	7.0	240	59,000	100	25,000
Typical Values					
Space { 12 oz Δ	4.0	65	28,000	1000	435,000
Rigidized { 14 oz □	4.0	80	35,000	1100	470,000
Materials (5) { 23 oz Δ	6.5	120	31,000	2000	510,000
(1) Ferrigno, T. H., "Rigid Plastic Foams," Reinhold, 1963. (2) Materials Selector, 1963 issue. (3) Measured with bonded on faces. *The ratio of ultimate strength in lbs/in² to density in lbs/in³. (4) Hughes Aircraft Company data. (5) Hughes Aircraft Company data. 1. 12 oz Δ - 12 oz per yd² triangular fabric 2. 14 oz □ - 14 oz per yd² rectangular fabric 3. 23 oz Δ - 23 oz per yd² triangular fabric Composite of values for various systems					

The modulus values shown in Tables 6, 7 and 8 were all calculated from load and deflection values in the normal manner. However, the measured deflections, in all cases, were almost entirely structure distortions and column buckling, and were not due to material elongations or compressions. Therefore it is considered that the modulus values as given should be considered more as indications of deflections under load, rather than actual modulus values. Buckling accounted particularly for the low strength and modulus values in flatwise compression. The tendency to buckle was accentuated in the 14 oz rectangular weave by a stress concentration point in the center of the flute as shown in Figure 39.

A comparison of the strength of the foam filled sandwich and the hollow core structures on the basis of stress levels developed during testing is misleading. The flexural strength value for the foam filled material of 100 psi appears very unfavorable compared to the considerably higher values reported for the hollow core samples. A comparison on the basis of load bearing capability is much more significant. Here the 60.5 pound test value for the foam filled material is higher than most values for the hollow core materials. Therefore, the strength of the former material compares favorably with the hollow core materials.



Figure 39. Web configuration of 14 ounce rectangular cloth.
(R99754)

The reason for this apparent discrepancy in the stress values is founded in the method of calculation. The general formula for the flexural stress in a simple beam is

$$S = \frac{M}{Z}$$

where M is the applied moment dependent on the load and Z is the section modulus dependent only on the cross section geometry of the structure. In the preceding analysis the section modulus of hollow core samples was calculated on the cross sectional area of the facing sheets and webs of the sandwich. This results in a low Z value and consequently a high calculated stress. The section modulus of the foam filled samples was calculated on the cross section of a solid, homogeneous area of the foam core. This results in a high Z value and consequently a low calculated stress. (The assumptions for the value of Z for the foam filled samples were based on the condition of the samples. Since the fiberglass facing sheets were relatively unimpregnated they were considered not to contribute to the load bearing capability in flexure.) In other words the hollow core sandwich may be compared to the foam filled materials as an I-beam compares to a solid beam of the same overall depth and width of cross section. Both may carry the same load, but a higher stress level is developed in the I-beam which is lighter in weight and, therefore, a more efficient structure.

Several conclusions can be drawn from the data in this report concerning the course of future work. It is obvious from the data, that there is a strong trend to higher ultimate strengths and higher specific strengths as resin content increases. Therefore work should be done at higher resin contents until evidence of diminishing returns is noted. It would also be desirable to determine if this relationship of high resin content to strength is peculiar to the weave of Raypan material. This could be accomplished by working with flat laminates made from standard cloths per MIL-F-9084 with various resin contents for each system. Since the relationship of strength to resin content is well known for standard fiberglass cloth it would be easy to determine if a high optimum resin content is a function of the Raypan weave or the resin system. The optimum glass finish for each resin system should also be determined.

Also it would be highly desirable to compare newly available sewn sandwich material to the integrally woven sandwich. The sewn three dimensional fabrics are made from standard fiberglass cloth.

Another trend in the present data is the higher specific strength and more useful load bearing range of the high weight fabrics. Future work should concentrate on fabrics of 23 oz/yd and above. Also, it would be advantageous to check the properties of specimens which have additional plies of cloth sewn to the sandwich faces. Such construction should provide more efficient section moduli for flexural and tensile loading.

Additional work is indicated in optimizing fabric impregnation techniques for all systems. Techniques utilizing dipping in conjunction with relatively soft rollers are suggested for the first effort.

Gelatin, despite the scatter of the present data, is the most promising material from the standpoints of strength, stiffness, low cost, simplicity, and efficacy under all space conditions. However, it is evident that the technology for using gelatin as a plastics resin in reinforced structures is in its infancy compared to conventional resins such as polyesters, and polyurethanes. The techniques of plasticization, impregnation, and stabilization of gelatin resin solutions need considerable refinement. Some means is also needed to set the gelatin after impregnation to reduce blocking and eliminate migration.

Current plans are for a continuation of this investigation. It is planned to emphasize development of the gelatin and polyurethane processes. Specifically efforts will be made to optimize the gelatin process so that fabrication of large panels can be done more uniformly and with less effort. Tests will also be made to determine techniques of pre-setting the gelatin impregnants, and to develop impregnants with lower volatile contents.

In addition to optimization of processing techniques it is also planned to extend the investigation of fabrication techniques to more complex elements than the small flat or carved panels produced in the initial project. The fabrication of columns, beams, large panels, and hemispheres will constitute a scaling up of the present techniques and should illuminate the problems which must ultimately be encountered in the deployment of actual space hardware.

As a result of the fabrication tests of larger elements it is anticipated that it will then be possible to make comparisons of the different systems, for specific sizes and structures. Complete data on the non-rigid, "launch" weights, which would include all factors, and the final rigidized weights will then be obtainable. All of the data from the continuation effort then will be combined in a supplementary report, which will, in combination with the present report, provide a complete design and process handbook for composite, three dimensional structures.

SYSTEMS COMPARISONS

The results of the physical properties tests indicated that, for equal resin contents, the strengths are approximately the same for each of the resin systems, although there are indications of the eventual superiority of gelatin. It was then considered that another measure of the resin systems could be a comparison of the initial impregnated weights vs the final rigidized weights. The ratio of the two weights could then be considered an indication of the system efficiency.

In considering the factors which made up the initial or "launch" weights it was found that the systems were all equal with regard to the

fabric used, the requirements for parting films and the polyethylene tubes used for expansion of the structure. The major differences in the launch weights of the polyester or gelatin impregnants then were due to amount of volatiles incorporated in either the polyester resins, or the gelatin, and these could easily be determined by test. However, the polyurethane system in addition to incorporated volatiles in the prepolymer, also required an external supply of water vapor. The required amount of water vapor is very difficult to determine since some is used in the reaction, but the major portion is lost by diffusion through the fabric into the vacuum. In addition to the water vapor a calculation of the "launch" weight of the polyurethane system would have to include the vapor distribution system, the sealed (until required) storage containers and the activation techniques. The percentage of weight contributed by these last ancillary items would vary considerably with the structure size and shape. It is therefore concluded that a comparison of "launch" weights, without a consideration of the particular size and structure configuration would, at this time be inconclusive.

A more meaningful, but less quantitative, comparison of the different systems can then be made by a consideration of all the factors entering into the utilization of each system. Such factors, then which distinguish one system from another include differences in processability, cure times and activation methods, reliability, etc. Table 12 then shows a comparison of the systems in which consideration is given to the various factors.

Analysis of the information shown in Table 12 indicates that currently no one system could be considered the "best" system. The processing simplicity and low volatile weight loss shown by the polyester system is offset by the serious limitation that such structures must be exposed to the sun's rays. Therefore these structures could not be launched in the earth's shadow or in deep space. The structure could not have a metallized surface or a foam meteorite bumper surface.

The polyurethane system theoretically requires only approximately 3 percent of water for activation. However, because a good deal of water is lost by diffusion through the material rather than by reacting, an excess must be used. Likewise provisions must be included for a sealed water source during storage. In space a water releasing mechanism and a distribution system of tubes, manifolds, etc., would also have to be provided. Since this ancillary equipment would vary in weight depending on the size and shape of the structure, no definite figures can be given at this time for the "specific launch" weights of such a system. The effect of very low temperatures or the reaction has also not been determined.

The gelatin system is the newest of the impregnating systems and as such appears to offer the most promise from the standpoint of ultimate reliability and simplicity. Current processing techniques result in unrigidized materials whose weight approaches the polyurethane system, and somewhat exceeds the polyesters. However, it is anticipated

TABLE 12. COMPARISON OF SPACE RIGIDIZATION SYSTEMS FOR INFLATABLE STRUCTURES

System	Impregnated, Unrigidized Material				Rigidized Material					Processibility (Impregnation conditions)	Remarks	
	Catalyzed Storage Life		Packaging Required	Effect of Thermal Cycling	Weight* oz/yd ²	Activation Means	Cure Time Hour at 72°F	Weight** oz/yd ²	Effects of U.V.			Thermal Effects
	Room Temperature	-20 to +160°F										
Polyester	1-3 Mo.	-20 indefinite +60 not over 30 days	Sealed container	High temperature reduces storage life	29.4	Ultraviolet radiation	1-4 hr	28.0	Fair to good	Satisfactory up to 250°F	Serious limitation in that part must be sun oriented for rigidization. Surface must allow entrance of U. V. radiation.	
Polyurethane	1-1 1/2 Mo.	-20 indefinite +60 not over 30 days	Nitrogen flushed sealed container	High temperature reduces storage life	29.7	Water vapor in special containers	12-24 hr	28.0	Good	Satisfactory	Special requirements for water vapor distribution, i.e., valves, tubes, etc., could add considerably to launch weight.	
Gelatin	Indefinite	-20 indefinite +60 indefinite	Moisture filled sealed container	No effect	Approx. 41.0	Exposure to vacuum	12-24 hr	28.0	Excellent	Satisfactory up to 250°F	Most reliable system. No special requirements for rigidization.	
One component urethane foam powder	Indefinite	No effect	Closed package	No effect	20-40	Infrared radiation to 250-300°F	1/2-1 hr	16-38	Good resistance	Satisfactory to 250°F	Currently no easy means of applying heat and then reducing surface temperature in space.	

*The unrigidized material weight refers to the weight of an impregnated sample of 14 oz rectangular fabric, with 50% final resin content.
**Weight of same material after rigidization has taken place.

*The unrigidized material weight refers to the weight of an impregnated sample of 14 oz rectangular fabric, with 50% final resin content.
 **Weight of same material after rigidization has taken place.

that the new techniques being developed will result in easier processing and lighter weight initial impregnants. With the development of such materials this process should be competitive in weight with the polyesters, and should have considerable advantages over the polyesters and none of the limitations. The gelatin may be considered as currently the only universal space rigidization system in that it will rigidize without any activation means other than the ever present vacuum of space. The rate of rigidization will be primarily a function of the temperature (heat input) and thickness of material to be rigidized.

CONCLUSIONS

As a result of the processing tests made and the physical properties tests run the following conclusions are made:

1. The rigidization under space conditions of three dimensional woven sandwich material, preimpregnated with several resin systems has been repeatedly demonstrated. Both flat and compound curvature structures have been produced.
2. Three dimensional fabrics weighing from 12 to 23 ounces per yard can be rigidized under space conditions to produce structural sandwiches having overall densities ranging from 2 to 8 pounds per cubic foot depending on fabric and resin content. These sandwiches have strengths of the same magnitude as other conventional structural materials of similar density.
3. Room temperature storage periods for impregnated structures, at the current state of the art, appear to be at least three months for polyester impregnated materials, approximately one and one-half months for polyurethane and an indefinite storage for gelatin impregnants. All systems require hermetic storage.
4. There appears to be no overall marked physical property superiority for any one of the "wet" rigidization systems. (The gelatin system does appear to be superior, based on the limited test data available.) There are, however, marked processing and usage differences which distinguish each system. These differences may be summarized as follows:
 - a. The simplest system to use from the standpoint of processing is the polyester system. Materials are readily available commercially and are well adaptable for large structure fabrication. This system results in the fastest cure cycle. The major limitation of the polyester system is the requirement that rigidization must take place in an environment illuminated by ultraviolet radiation.
 - b. The gelatin system offers high strength, high stiffness and unlimited storage life coupled with no limitation on space usage. The major problems with this system concern difficulty in obtaining uniform impregnation of large structures.
 - c. The polyurethane material is almost as easy to impregnate in fabric as the polyester. However, impregnation of large structures would require development of a moisture-free environment processing technique. The

major limitations to the use of this system are the current relatively short storage life, (probably because of moisture absorbed from the air), and the requirement that the part be some type of inflatable or double walled structure in order that the water vapor catalyst can be passed to all areas.

5. Test samples made with rectangular fabrics appear to show slightly better properties than the same weight samples made with triangular fabric. Rectangular samples are also considerably easier to fabricate and are more reliable since they require a lower number of inflation tubes for a given area.
- (6. Based on the cloth weights investigated in all cases the physical properties increase with increasing resin content, up to at least a maximum of 60 to 70 percent resin.
7. Additional work is required to optimize the fabric impregnation techniques, since the resin content data indicated that in almost all cases the webs showed a lower resin content than either of the facings. (Note - This held true also for the "Ideal" gelatin samples which were made by Monsanto Research Laboratories of Dayton, Ohio.) In most cases both facing sheets were impregnated approximately the same amount. The lower resin content of the webs is then attributed to higher pressure in the web area as the fabric was drawn through the squeeze rolls.
8. The data collected indicates that resin content determinations can be made by use of simple weighing techniques. The use of this method offers the advantages of time and labor savings as well as resulting in better "average" values.

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