

## RIGIDIZABLE EXPANDABLE AEROSPACE STRUCTURES

R. G. Spain and T. L. Graham  
Aeronautical Systems Division  
Wright-Patterson Air Force Base, Ohio

This internal research program is concerned with investigating a proposed chemical concept for rigidizing expandable aerospace structures, after inflation in space. Life (meaning the retention of designed shape) of flexible expandable aerospace structures is limited by the permeability characteristics of the materials of construction and the probability of meteoroid puncture. With loss of inflation pressure by either of these routes the flexible walled structure tends to collapse under the external pressures associated with light radiation and the gases present at high altitudes.

A review of the considerable research to attain rigidization of expandable structures has indicated that the schemes employed are of two general types. Mechanical or physically induced rigidity is exemplified by telescoping rigid members, the stressing of thin metal foils to more rigid configurations, combination rigid and flexible members in umbrella-like designs, and the vacuum induced volatilization of plasticizers from normally rigid polymeric materials. Chemical systems have utilized ultraviolet radiation induced polymerization and crosslinking of initially flexible materials, thermally induced chemical reactions yielding rigid polymeric materials, and a variety of methods for the use of foamed-in-place polymers. Several permutations are also possible by combining two or more of the above concepts.

The magnitudes of the external forces which tend to collapse an expandable structure vary with altitude. Atmospheric pressures at various altitudes are presented below. At the earth's distance from the sun, the sun's radiation pressure is given as  $3.375 \times 10^{-8}$  mm Hg (1).

Assuming the structure is not punctured by a meteoroid, retention of original shape can be assured by rigidizing the structure after it is inflated in space to a degree required to resist the prevailing external forces. Such structures could be used as solar power stations and communication satellites. Structures rigidized

### Atmospheric Pressure as a Function of Altitude

<u>Height, miles</u>	<u>Pressure, mm Hg</u>
186	$2.139 \times 10^{-5}$
500	$3.827 \times 10^{-8}$
900	$5.028 \times 10^{-10}$
1,000	$2.526 \times 10^{-10}$

to a degree capable of withstanding high internal or external forces would have a much larger potential. Highly rigidized structures could be used to induce drag on re-entry flights. Those capable of withstanding the pressure associated with a life sustaining atmosphere and the impact of micrometeoroids could serve as manned space stations.

This concept proposed for rigidizing expandable aerospace structures involves the use of viscous resin intermediates which can be polymerized into tough rigid products. The method of approach entails impregnating the flexible fabric construction with the intermediate at the time of assembly and upon expansion introducing the gaseous catalyst which effects the desired transformation.

Ideally, the resin intermediate prior to exposure to the appropriate environment which converts it into a rigid solid, should be stable indefinitely at room temperature. In the initial state, it is essential that the resin remain in place once distributed between the confining walls of the structure. It is desirable that the resin system rigidize rapidly at room or slightly higher temperature on exposure to the appropriate gaseous activator.

Three components are used in this concept of rigidizable expandable structures (see Figure 1). The exterior of the structure consists of a single ply of high

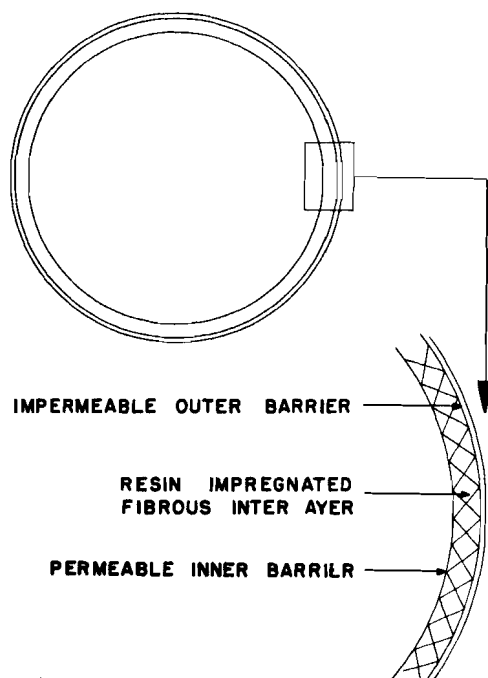


Figure 1. Cross-Section of Expandable Rigidizable Structure

strength fabric coated with a tough impermeable flexible polymer. It serves to confine the inflating gas, determines the initial bursting strength of the structure and defines its shape. The ply or plies of fabric which are impregnated with the rigidizable resin are intermediate. Finally, an inner barrier film or flexible polymer coated fabric is used which is permeable to the activating agent. The component prevents the packaged deflated structure from adhering and forces the resin impregnated substrate against the outer casing on inflation.

While there are a number of other factors to consider which effect the performance and life of the expandable aerospace structure, such as structural design and the stability of materials of construction in an outer space environment, it is not within the scope of this program to investigate these aspects.

The objectives of this program were:

1. To gather general information on the performance of available structural materials (coated and uncoated fabrics and resin-catalysts systems) in relation to their function in the structure.
2. To fabricate simple structures and thus demonstrate this rigidization concept.

These objectives were substantially achieved. The overall success of the concept seems well established. It is not limited by inputs of various types of energies but is, admittedly, dependent for rate of reaction to the thermal environment. The types of reactions envisioned are varied in type and are largely not subject to thresholds of temperature.

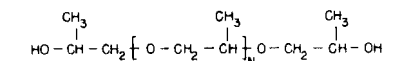
Hence, this general concept of rigidization, while directed toward space applications, also appears feasible for terrestrial uses.

#### Exploratory Vapor-Curing Studies

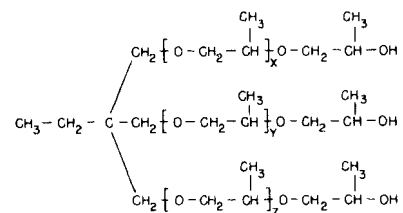
The initial effort on this program was devoted to seeking viscous resin impregnating agents which readily rigidize through the absorption of volatile curing agents. These initial experiments were qualitative. Samples of the selected resins were exposed for periods of up to several days to vapors of the chosen crosslinking agents at room temperature and periodically observed to determine the relative extent of cure.

Urethane resin intermediates were selected for evaluation as rigidizing agents for expandable aerospace structures because of their reactivity at modest temperatures with compounds containing active hydrogen. The resin intermediates obtained for evaluation were adducts of either toluene diisocyanate (TDI) or methylene bis-4,4'-phenylisocyanate (MDI) and polyoxypropylene polyol or more complex polyols prepared by coupling two or more moles of a base polyol with a diisocyanate. The base polyols used in the preparation of the prepolymers differed in molecular weight and functionality. Their chemical structures are shown in Figure 2. Reactions leading to the formation of the simple and the more complex polyfunctional isocyanate terminated prepolymers are schematically shown in Figure 3.

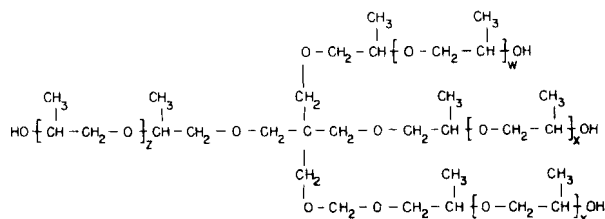
Compounds generally employed for curing these resin intermediates are: polyamines, polyols and water vapor. Figure 4 shows the reactions involving these curing agents. As shown, water and amines lead to crosslinked systems containing urea and biuret linkages. With water the formation of urea linkages is a two-stage reaction in which  $\text{CO}_2$  is released. Urethane and allophanate linkages are obtained with polyol crosslinking agents; however, since even the simplest polyols are relatively nonvolatile, the choice of curing agents was limited to water and the amines. The compounds selected for evaluation are listed in Table I.



PROPYLENE GLYCOL BASED POLYOXYPROPYLENE DIOL



TRIMETHYLOLPROPANE BASED POLYOXYPROPYLENE TRIOL



PENTAERYTHRITOL BASED POLYOXYPROPYLENE TETROL

Figure 2. Chemical Structures of the Basic Polyether Polyols

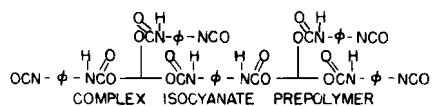
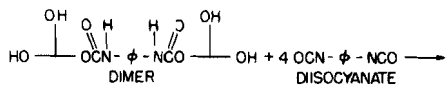
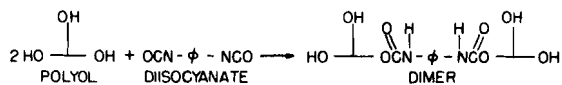
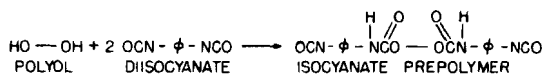


Figure 3. Isocyanate-Folyol Prepolymer Forming Reactions

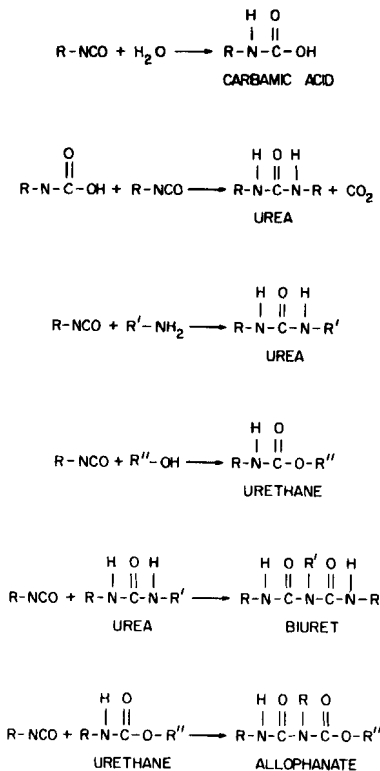


Figure 4. Isocyanate Reactions with Active Hydrogen Compounds

TABLE I. Candidate Polyurethane Curing Agents

Water  
 Ammonia  
 Ethylene diamine  
 Diethylene triamine

The most favorable results were obtained with the moisture cure. With moisture complete cures were obtained indicating that the diffusion and reaction processes occurring were generally balanced. The cured resins were foamy, however. The other candidate crosslinking agents were too reactive and in these cases curing was superficial.

Other resins evaluated which are known to be very reactive at room temperature were the epoxies. The resins evaluated were simple and higher molecular weight adducts of bisphenol-A/epichlorohydrin and a polyepoxide product prepared by the epoxidation of linseed oil. Chemical structures of these resin intermediates are given in Figure 5. Aside from the differences in molecular weight a few of the resins evaluated contained butyl or phenyl glycidyl ether as reactive diluents.

Aromatic and aliphatic polyamines, acid anhydrides, polyamides, Lewis acid and Lewis bases are common hardeners for these resin intermediates. However, as the Lewis acid, Lewis bases and aliphatic polyfunctional amines are presumably the best room temperature hardeners these types of compounds were favored. As shown in Figure 6, the curing mechanism with Lewis-type compounds is ionic whereas cures involving polyfunctional amines is an addition reaction (1). The selected candidate

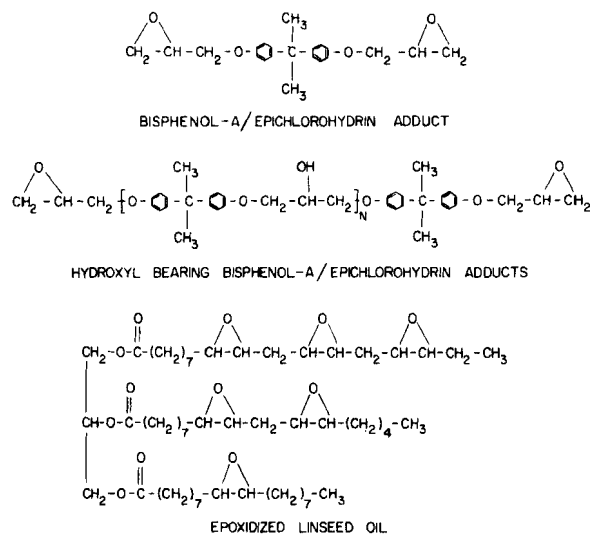


Figure 5. Chemical Structures of the Basic Epoxy Resin Intermediates

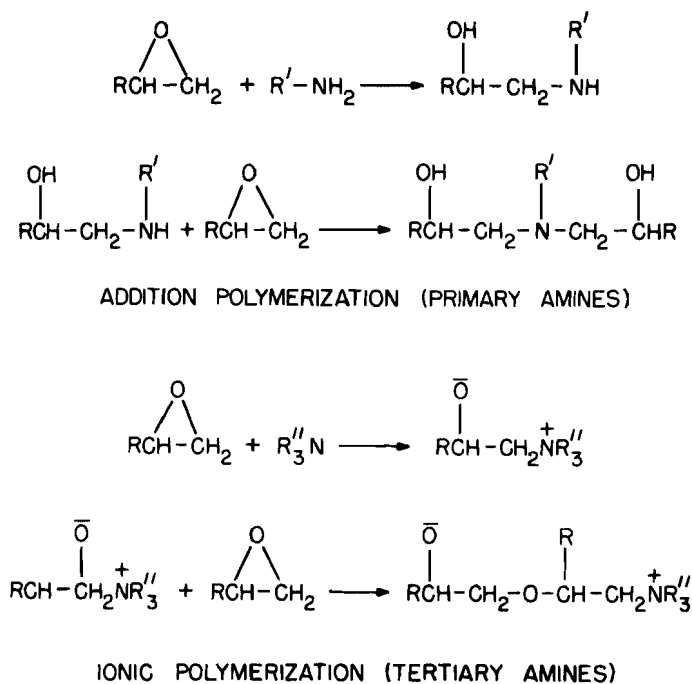


Figure 6. Ionic and Addition Epoxy-Amine Curing Reactions

curing compounds are identified in Table II.

TABLE II. Candidate Epoxy Resin Curing Agents

Trimethylamine  
 Triethylamine  
 Tripropylamine  
 Tributylamine  
 Ethylamine

TABLE II. Candidate Epoxy Resin Curing Agents (Cont'd)

Diethylamine  
 Ethylene diamine  
 Diethylene triamine  
 Ethanolamine  
 Dimethyl ethanolamine  
 Ethylene imine  
 Boron trifluoride  
 Boron trifluoride monoethylamine complex  
 Trimethyl borate

Triethylamine (TEA) proved to be an effective gaseous curing agent for low viscosity bisphenol-A/epichlorohydrin-type resin intermediates. The resin containing butyl glycidyl ether cured completely within the seven-day exposure period. Trimethylamine, a gas, surprisingly was not as active as TEA. Tripropylamine and tributylamine, due possibly to their relatively greater bulk and lower vapor pressures, did not penetrate and cure this resin to any great extent. Lewis acids and the other amine compounds were too reactive with the conventional epoxy resins and as a result, the cure was confined to the surface. The polyepoxide resin showed no significant activity toward any of the candidate curing agents evaluated.

Basic information regarding the relative reinforcing effects and curing characteristics of the promising candidate systems was obtained from the evaluation of standard laminate samples. The standard samples prepared for evaluation utilized two plies of a glass fabric as reinforcement for the candidate resin impregnates and an impermeable rubber coated fabric as a cover for the surface which was not to be exposed to the gaseous curing agent. Descriptions of these fabrics are given below:

<u>Type of Fabric</u>	<u>Coating</u>	<u>Weight, oz/sq yd</u>
Glass Cloth 181	Volan-A	8.9
Dacron	Viton-B	10.3

Polyurethane Resin Rigidized Laminates

Investigations into the structural reinforcing effects of the various systems began with the evaluation of the moisture cure. Room temperature cures were attempted on laminate samples impregnated with the urethane resin intermediates described in Table III at relative humidities of 35, 65, and 95 percent.

TABLE III. Candidate Polyurethane Resin Intermediates

<u>Resin Intermediate</u>	<u>Description</u>	<u>Equivalent Weight/NCO</u>	<u>Percent Solids</u>	<u>Solvent</u>
P101C-TDI Adduct	TDI terminated 1000 mol. wt. polyether diol	674	90	toluene
P101O-MDI Adduct	MDI terminated 1000 mol. wt. polyether diol	750	90	toluene

TABLE III. Candidate Polyurethane Resin Intermediates (Cont'd)

<u>Resin Intermediate</u>	<u>Description</u>	<u>Equivalent Weight/NCO</u>	<u>Percent Solids</u>	<u>Solvent</u>
TP440-TDI Adduct	TDI terminated 400 mol. wt. polyether triol	311	80	50/toluene
TP740-TDI Adduct	TDI terminated 700 mol. wt. polyether triol	415	90	toluene
TP740-MDI Adduct	MDI terminated 700 mol. wt. polyether triol	491	70	toluene
TP740-TDI Dimer	2 moles of 700 mol. wt. polyether triol coupled and terminated with TDI	579	70	toluene
TP740-MDI Dimer	2 moles of 700 mol. wt. polyether triol coupled and terminated with MDI	674	70	toluene
TP1540-MDI Adduct	MDI terminated 1500 mol. wt. polyether triol	750	85	toluene
PeP450-TDI Adduct	TDI terminated 400 mol. wt. polyether tetrol	233	80	50/toluene 50/MEK
NCO-1-5D	2 moles of 1500 mol. wt. polyether triol coupled and terminated with TDI	968	100	-
NCO-5-1T	3 moles of 700 mol. wt. polyether diol coupled and terminated with TDI	1,398	100	-

The results obtained with these systems were unsatisfactory. The low equivalent weight resin intermediates (TP440-TDI, TP740-TDI and PeP450-TDI) which provided relatively stiff laminates were lacking in structural integrity. Laminates impregnated with these candidate resins foamed and blistered on curing at all moisture concentrations. Laminates impregnated with NCO-1-5D and NCO-5-1T when cured at 35 percent R.H. provided non-foamed laminates but these were undesirably flexible. The polyether polyols terminated with methylene bis-4,4'-phenylisocyanates were more prone to foaming than the corresponding toluene diisocyanate adducts.

The data, Table IV, show the general range in reinforcement obtainable with these systems. These results were obtained on samples selected from those cured at 35 percent R.H. in accordance with ASTM D790-58T procedure of test for flexural strength properties using a Baldwin Universal Tester.

Several urethane systems were evaluated which contained modifiers. The modifiers were introduced in an attempt to obtain structurally sound systems. High



TABLE IV. Laminates Rigidized with Polyurethane Resins

<u>Resin Intermediate</u>	<u>Exposure Time, Days</u>	<u>Flexural Strength, psi</u>	<u>Modulus of Elasticity, psi x 10<sup>-5</sup></u>	<u>Laminate Weight, oz/sq yd</u>	<u>Equivalent Weight/ NCO</u>
TP440-TDI Adduct	4	4,840	2.82	40.49	311
	7	2,190	0.36	45.60	
	10	9,190	3.99	44.91	
	14	4,600	2.22	44.51	
TP740-TDI Adduct	4	370	-	43.81	415
	7	390	-	53.69	
	10	1,010	-	51.39	
	14	3,590	1.36	50.69	
PeF450-TDI Adduct	4	10,100	4.23	42.20	233
	7	11,350	5.77	42.93	
	10	8,250	3.92	43.97	
	14	9,030	4.18	45.48	
F1010-TDI Adduct	4	-	-	-	674
	7	-	-	-	
	10	480	-	50.95	
	14	740	-	48.61	
NCC-5-1T	4	-	-	-	1,398
	7	-	-	-	
	10	1,730	-	39.81	
	14	1,680	-	41.15	
NCC-1-5D	4	-	-	-	968
	7	-	-	-	
	10	1,940	-	39.16	
	14	2,210	-	39.11	

boiling point solvents were added with the hope of increasing the systems tolerance for CO<sub>2</sub>. Filler and silicone fluids were incorporated with the intention of improving the diffusion characteristics of the system. Compositions of the modified systems are shown in Table V.

TABLE V. Modified Polyurethane Resin Impregnants

<u>Ingredients</u>	<u>Parts by Weight</u>							
TP740-TDI	100	100	100	100	100	-	100	-
TP440-TDI	-	-	-	-	-	100	-	100
HiSil 233	20	-	-	-	-	-	-	-
Silicone Liquid L520	-	1-5	-	-	-	-	-	-
Silicone Liquid DC200	-	-	1-5	-	-	-	-	-
Silicone Liquid XF1112	-	-	-	1-10	-	-	-	-
Ethylene Carbonate	-	-	-	-	10	-	-	10
Diethyl Carbonate	-	-	-	-	-	10	10	-

These modifiers did not prove to be effective anti-foaming agents for these systems. The laminate samples impregnated with the modified resins were observed to foam to about the same extent as the laminates impregnated with the unmodified products when cured at room temperature in a relative humidity of 35 percent. As the modifiers did not show promise and the results of previous work were unsatisfactory, the urethanes were not exploited further for this use.

### Epoxy Resin Rigidized Laminates

Favorable results in the exploratory curing studies encouraged investigations concerning the reinforcing effects and curing characteristics of the epoxy resin intermediates derived from bisphenol-A and epichlorohydrin. To obtain general information regarding the merits of resins of this type, standard laminate samples were prepared using the candidate resins described in Table VI. Cures were attempted on these samples in an atmosphere saturated with TEA at room temperature (a TEA vapor pressure of 70 mm Hg) for periods as long as seven days.

TABLE VI. Candidate Epoxy Resin Intermediates

<u>Composition</u>	<u>Brookfield Viscosity at 25°C, Poises</u>
Bisphenol-A/Epichlorohydrin + Butyl Glycidyl Ether	5-7
Bisphenol-A/Epichlorohydrin + a diluent	30
Bisphenol-A/Epichlorohydrin + Phenyl Glycidyl Ether	40-100
Bisphenol-A/Epichlorohydrin	64
Bisphenol-A/Epichlorohydrin	95
Bisphenol-A/Epichlorohydrin	140
Bisphenol-A/Epichlorohydrin	180
Bisphenol-A/Epichlorohydrin	Very Viscous

The low viscosity resin intermediates proved to be quite susceptible to the gaseous curing technique. Resin intermediates with viscosities not exceeding 64 poises provided cured laminates within the seven day period. These laminates, as indicated by flexural strength data (Table VII), were much stronger than the urethane resin laminates (Table IV). The lowest viscosity system was preferred because it was relatively fast curing. Laminates impregnated with resins having viscosities in excess of 64 poises did not cure significantly.

TABLE VII. Laminates Rigidized with Epoxy Resins

<u>Resin Intermediate</u>	<u>Laminate Weight, oz/sq yd</u>	<u>Viscosity at 25°C, Poises</u>	<u>Exposure Time, Days</u>	<u>Flexural Strength, psi</u>	<u>Modulus of Elasticity, psi x 10<sup>-2</sup></u>
Bisphenol-A/ Epichlorohydrin + Butyl Glycidyl Ether	46.25	5-7	2	17,530	4.02
			3	30,500	7.08
			4	20,720	6.20

TABLE VII. Laminates Rigidized with Epoxy Resins (Cont'd)

<u>Resin Intermediate</u>	<u>Laminate Weight, oz/sq yd</u>	<u>Viscosity at 25°C, Poises</u>	<u>Exposure Time, Days</u>	<u>Flexural Strength, psi</u>	<u>Modulus of Elasticity, psi x 10<sup>-5</sup></u>
Bisphenol-A/ Epichlorohydrin - a diluent	47.55	30	2	-	-
			3	13,750	3.47
			4	18,500	4.40
			7	22,470	6.13
Bisphenol-A/ Epichlorohydrin	48.65	64	2	12,700	5.18
			3	11,750	3.57
			4	19,770	8.27
			7	32,750	10.99

The effect of TEA vapor pressure on the rate of cure and ultimate strength of standard laminate samples impregnated with this resin system are shown in Figure 7. Maintenance of the curing agent vapor pressure at 70 mm Hg (the saturation point at room temperature) for 72 hours resulted in optimum cured strength. Under these conditions as the upper curve in Figure 7 shows this system becomes less tough (more brittle) when the exposure is extended beyond 72 hours. A fair degree of strength is attained in 24 hours using an amount of curing agent sufficient to exert an initial vapor pressure of 70 mm Hg. About the same degree of rigidity can be attained using half this amount of curing agent at a sacrifice in cure rate.

#### Modified Epoxy Resin Systems

Because of the advantages associated with rapid curing systems, techniques were investigated for shortening the TEA cure of the system based on the low viscosity epoxy resin containing butyl glycidyl ether.

One attempt to accelerate the cure involved the use of hydroxyl bearing compounds. The literature indicated that the presence of such materials activated the opening of the oxirane ring. The compounds evaluated as activators were ethylene glycol, glycerin and resorcinol. As resorcinol is only partially soluble in the resin a saturated solution was prepared and evaluated. The other compounds were evaluated by mixing a gram-equivalent weight of the resin with a gram-equivalent weight of the modifier. Using these systems standard laminates were prepared and exposed to cure in an environment containing an initial TEA vapor pressure of 70 mm Hg. After six hours exposure inspection of the samples had shown that curing had not been initiated, thus indicating that the modifiers had not exerted the expected activating influence.

Another approach to shortening the cure which also proved unsuccessful involved a pre-curing step. Samples of the resin were gelled with 0.25 to 2.5 percent of diethylene-triamine by weight and then subjected to post-cures in an atmosphere saturated with TEA. Examination after approximately 16 hours exposure revealed that the resulting gels had only cured on the surface.

EFFECT OF TRIETHYLAMINE VAPOR PRESSURE ON EPOXY RESIN LAMINATE PHYSICAL PROPERTIES AND CURE RATE

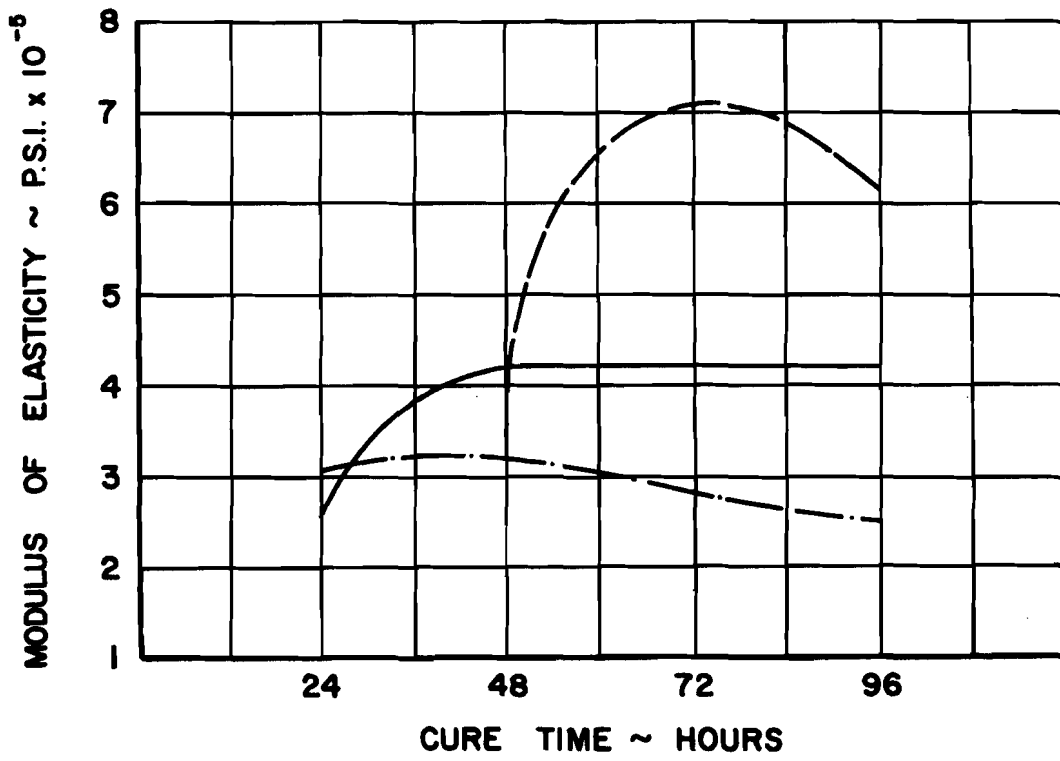
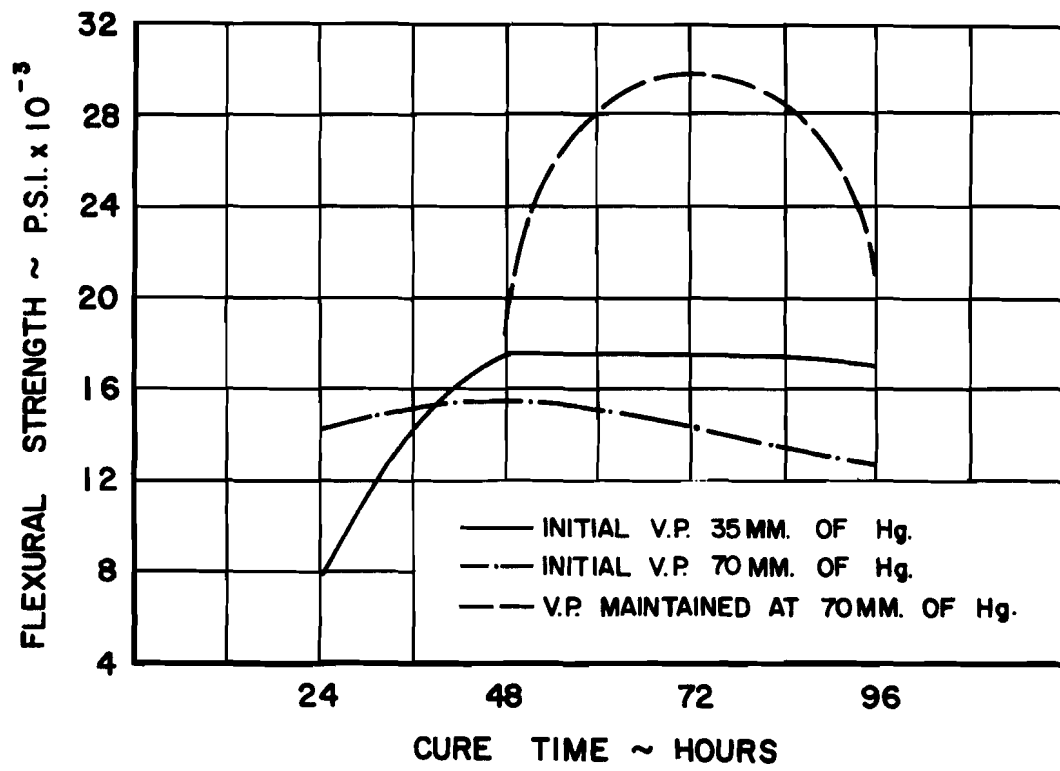


Figure 7. Effect of Triethylamine Vapor Concentration on Epoxy Resin Laminate Flexural Properties and Cure Rate

### Permeable Barrier Structural Components

As previously mentioned, the rigidizable structure requires a liner which can be permeated by the crosslinking agent. This liner prevents the structure from adhering when packaged by keeping the inner wall of the resin impregnated composite separated and forces the plies of the structural assembly together on inflation. Since both the epoxy and the urethane resin impregnates appeared promising at this point in the program, materials were sought which could be readily permeated by their respective vapor phase curing agents (water and triethylamine).

Permeability measurements (Table VIII) made at room temperature speak for themselves. Silicone rubber coated fabrics were found to be the most permeable to the epoxy resin curing agent while microporous polyethylene on fabric possessed the highest permeation rate to the urethane curing agent.

TABLE VIII. Permeability of Candidate Barrier Materials

<u>Candidate Barrier</u>	<u>Permeability*</u>	
	<u>H<sub>2</sub>O</u>	<u>TEA</u>
Microporous Polyethylene Coated Fabric	21.08	38.03
Cellophane Film	1.13	-
Cellulose Acetate Film	0.83	-
Silicone Rubber Coated Nylon	-	44.44
Silicone Rubber Coated Dacron	-	53.65
Polyethylene Film	-	3.02

\* oz/sq yd/hr/mil thickness

As emphasis on the program shifted to the epoxy resins, further analysis of the silicone rubber type of barrier was undertaken to determine its effect on cure rate and structural strength. Standard laminate samples prepared for this investigation were impregnated with the epoxy resins which showed a fair degree of activity and covered with the candidate barrier material.

The results obtained on samples cured at room temperature for two, three, and four days in an atmosphere kept saturated with TEA were very favorable. Comparison of the data obtained on these samples (Table IX) and the results of previous data

TABLE IX. Barrier-Covered Laminates Rigidized with Epoxy Resins\*

<u>Resin Intermediate</u>	<u>Laminate Weight, oz/sq yd</u>	<u>Viscosity at 25°C Poises</u>	<u>Exposure Time, Days</u>	<u>Flexural Strength, psi</u>	<u>Modulus of Elasticity, psi x 10<sup>-5</sup></u>
Bisphenol-A/Epi-chlorohydrin + Butyl Glycidyl Ether	45.38	5-7	2	29,130	7.05
			3	21,250	4.84
			4	18,330	3.19

TABLE IX. Barrier-Covered Laminates Rigidized with Epoxy Resins\* (Cont'd)

<u>Resin Intermediate</u>	<u>Laminate Weight, oz/sq yd</u>	<u>Viscosity at 25°C Poises</u>	<u>Exposure Time, Days</u>	<u>Flexural Strength, psi</u>	<u>Modulus of Elasticity, psi x 10<sup>-5</sup></u>
Bisphenol-A/Epi-chlorohydrin + a diluent	49.95	30	2	24,550	5.87
			3	12,160	3.60
			4	7,540	-
Bisphenol-A/Epi-chlorohydrin	48.26	64	2	14,150	3.68
			3	25,130	6.09
			4	1,270	-

\* Barrier material removed prior to determining physical properties.

obtained on unshielded laminates (Table VII) revealed that the barrier had not interfered but enhanced the rate of cure. The properties, in general, as can be seen, were not significantly affected and, accordingly, this type of material was used as an inner liner for an experimental expandable rigidizable structure assembled to demonstrate the proposed concept.

Demonstration of the Rigidization Concept

An expandable structure was assembled for demonstrating the overall feasibility of the rigidization concept under vacuum. The vacuum chamber used, as shown in Figure 8, was equipped with two exhaust systems (one for exhausting the air from the structure and the other for exhausting the air from the chamber and maintaining the vacuum on the structure).

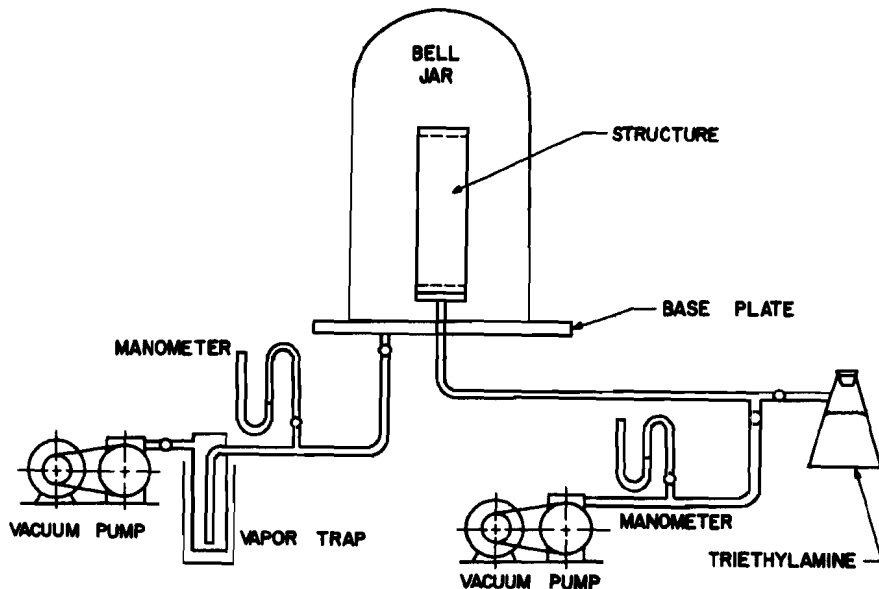


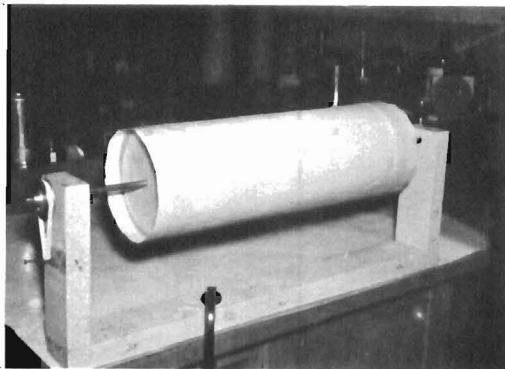
Figure 8. Experimental Vacuum Apparatus Assembly

The structure assembled utilized the Viton-B coated Dacron fabric as an outer impermeable cover. A single ply of glass fabric (style 183 with a Volan-A finish) impregnated with the low viscosity epoxy resin intermediate thickened with 7 percent by weight Cab-O-Sil to prevent resin flow served as the rigidizable structural component. A silicone rubber coated nylon fabric was employed as the permeable barrier material.

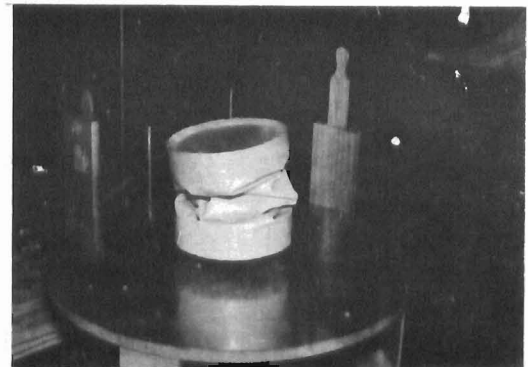
For ease of assembly the structural fabric components were cemented to epoxy resin impregnated glass tape end rings, fitted to the 11" diameter, 25" length cylindrical shaped building form (Figure 9.a.). Wooden disc inserts were used to seal both ends of the structure. These ends were cemented in place with an epoxy resin. One end was fitted with a 1/4" pipe inlet for attaching it to the vacuum chamber base (Figure 9.b.).

After installing the structure in the vacuum chamber the external and internal pressures on the structure were simultaneously reduced to, respectively, 3 and 5 mm Hg. The inlet to the evacuated structure was then connected to the source of liquid TEA and inflated at room temperature by the vapor pressure of approximately 70 mm Hg exerted by the curing agent (Figure 9.c.). Throughout the experiment the external pressure was maintained at 5 mm Hg by continuous pumping.

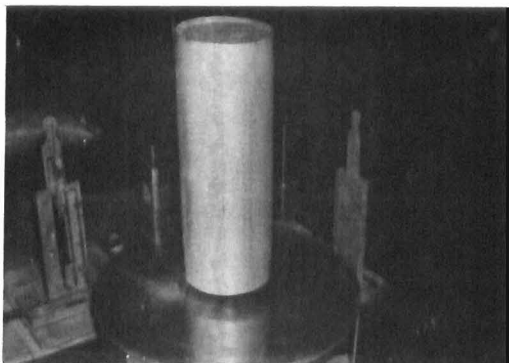
On removal after 48 hours this structure was found to be very rigid (Figure 9.d.). Based on the weight gained by the structure 1.75 ounces of TEA were absorbed per square yard of surface area which contains roughly 14 ounces of resin.



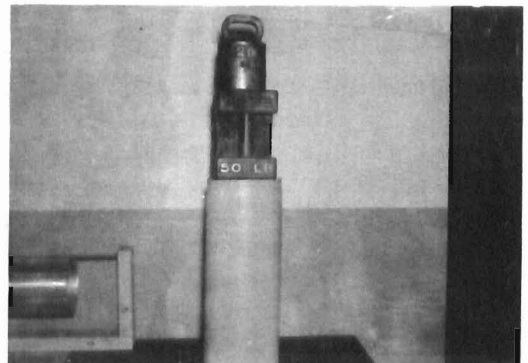
(a) FABRICATED



(b) COMPACTED



(c) EXPANDED



(d) RIGIDIZED

Figure 9. Experimental Expandable Rigidizable Structure

## Bursting Strength Before and After Rigidization

The internal pressure which an expandable rigidizable structure can withstand in a space environment can be estimated for cylindrical, spherical and torus shaped structures using the following equation (2):

$$P = \frac{2S}{R}$$

where:

P = bursting strength at zero external pressure, lbs/sq in

S = tensile strength of the effective structural component, lbs/sq in

R = radius of structure, inches

Prior to rigidization the bursting strength of a structure is determined by the outer impermeable cover and subsequent to rigidization on the properties of the rigidized segment.

The necessary data (see below) was, therefore, obtained on these components of the epoxy resin rigidizable structure to obtain an estimate of the benefits to be gained in regards to bursting strength. From these data it was calculated from the above relationship that through rigidization the bursting strength is increased in this particular case approximately 7 fold.

	<u>Breaking Load, pi</u>
Viton-B Coated Dacron	161
Resin-Rigidized Composite	1,041

### Summary

Investigations into rigidizing initially flexible impregnated fibrous composite structures after expansion required experiments with several thermal-setting resin impregnants using various crosslinking agents.

Water vapor was the most effective crosslinking agent found for curing isocyanate-terminated polyurethane resin intermediates. With water vapor, thoroughly cured systems were obtained within several hundred hours. The other candidate gaseous crosslinking agents (polyamines and  $\text{NH}_3$ ) which were evaluated only partially cured the systems. The difference in behavior appears to be related to differences in the mechanisms of the curing reactions. With moisture cures, the gelling reaction (Figure 4, Equation 2) is governed by the rate of the  $\text{H}_2\text{O}$ -NCO reaction (Figure 4, Equation 1), the latter being sufficiently slow to allow time for adequate amounts of moisture to diffuse into the system.

Gelation is direct with the other compounds; consequently, when these compounds react with the NCO groups available at the surface, a skin is formed which greatly reduces the diffusion process.

The isocyanate moisture-curing system proved to be unsatisfactory. The  $\text{CO}_2$



liberated in the reaction foamed and blistered those resin intermediates (low-equivalent weight products) which exhibited adequate reinforcing effects. The moisture-cured resin systems which did not foam (high-equivalent weight intermediates) were extremely slow to cure and gave more flexible products.

An approach involving the use of modifiers to solve the foaming problem by increasing the system's tolerance for CO<sub>2</sub> or permeability to CO<sub>2</sub> were unsuccessful. Filler, silicone fluid, and solvent-modified systems foamed practically to the same extent on curing as the unmodified systems.

The most promising results were obtained with the epoxy resin intermediates. Certain members (specifically the simple or low-molecular-weight bisphenol-A/epichlorohydrin adducts with and without reactive diluents) were found to completely rigidize on exposure to gaseous triethylamine at room temperature.

Failure of the high-molecular-weight bisphenol-A/epichlorohydrin products to completely polymerize on exposure to triethylamine was believed due to increases in reaction rates brought about by the presence of increasing numbers of pendant hydroxyl groups (which the literature (2) indicates activates the epoxy group) and decreases in diffusion rates with increase in viscosity.

Mechanical properties and curing characteristics of the simple adduct of bisphenol-A/epichlorohydrin containing butyl glycidyl ether as a reactive diluent were, in general, superior to those of other vapor-curable systems which were evaluated. With a triethylamine-saturated atmosphere, at room temperature, optimum properties were obtained with this intermediate in about 3 days. Substantial rigidity, however, was obtained in 24 to 48 hours.

The concept of rigidization involves structures which are made up of three separate components, an outer permeable coated fabric cover, a rigidizable resin-impregnated fabric interlayer, and an inner barrier material which keeps the structure from adhering in the deflated state and which is permeable to the polymerization agent. In connection with the needs for an inner barrier which could be readily permeated by triethylamine, several candidate materials were investigated.

Results of permeability measurements showed the silicone-rubber-coated fabrics to be more highly permeable to triethylamine than the other candidate materials. In a later study, involving laminates covered with a silicone-coated fabric very satisfactory results were obtained. A comparison of data obtained on laminates cured with and without this barrier indicated that it had not impeded the rigidization process.

The successful rigidization of an expandable structure under vacuum using an inflating pressure of 70 mm Hg has proved the workability of the vapor-curing concept.

### References

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