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CR-114837

BIS (EPOXY ALKYL) CARBORANE ADHESIVES

Contract NAS9-9616

Final Report

By

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Submitted to:

National Aeronautics and Space Administration
Manned Spacecraft Center
Houston, Texas

30 October 1970

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LITTLE FALLS, NEW JERSEY

ABSTRACT

Contract NAS 9-9616

The steel-on-steel (17/7 PH) adhesive bonding technology of a new class of high temperature stable epoxy adhesive based on the monomer bis(epoxybutyl)carborane has been developed. This monomer can be cured with conventional curing agents and reactive hardeners without the evolution of volatile by-products. The following lap-shear strengths have been obtained in air environments (10 minute hold at temperature).

- 320°F	4040 psi
- 65	3480
75	3060
350	3100
450	2960
500	2620

Lap-shear strengths as high as 2180 psi have been obtained at 500°F after aging at 500°F for eight hours in an air environment. The honeycomb bonding technology (stainless steel core and face sheets) has also been developed. In beam flexure tests, ultimate loads of 1580 pounds and 805 pounds were obtained at 75°F and 500°F respectively. In sandwich peel tests, peel torque values of 6.5 and 3.1 inch pounds/inch width of specimen were obtained at 75°F and 500°F respectively. These values were obtained on a formulation which had not been modified with an anti-oxidant. Further improvement in these values can be expected with additional optimization of the formulation and cure-assembly procedures.

The adhesive system based on the bis(epoxybutyl)carborane monomer can be processed by conventional epoxy handling techniques. The B-staged adhesive can be applied to substrates by hot-melt techniques or in the form of an impregnated glass cloth carrier tape.

The lap-shear and honeycomb adhesive values reported above were obtained using a cure-assembly cycle of 307-351°F, 60 psi, 2½ hours and a post-cure of one hour at 435-465°F. Specimens can be bonded using press or vacuum bag techniques.

ADMINISTRATIVE INFORMATION

This 16 month program was sponsored by the NASA Manned Spacecraft Center, Houston Texas under contract NAS9-9616. The NASA Technical Representative was Mr. Ivan K. Spiker/ES8. The Principal Investigator at the Research Center of Kearfott Division, Singer-General Precision Inc. was Dr. Robert Barnes. Contributors to the program were Mr. William Benko, Mr. William Block, Dr. Edward Hughes and Mr. Charles Maccia. Other contributors included Mr. E. Kosloski, Mr. T. Magnini and Mr. A. Skurna. Adhesive test specimens were machined at the Research Center Model Shop.

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1. INTRODUCTION

The objective of this program was to develop the steel-on-steel adhesive bonding technology of a new class of high-temperature-stable epoxy adhesive based on the newest class, bis (epoxyalkyl) carboranes. A need exists for an adhesive that cures without the evolution of volatile by-products and which is useful for bonding applications under long-time exposure at 500°F and above in air environments. Steel-on-steel lap-shear strengths of greater than 1700 psi had previously been obtained at 500° F with bis (epoxyalkyl)carboranes cured with certain agents.(1,2) These results demonstrated that this system was a potentially useful adhesive for high temperature bonding applications.

1.1 ADHESIVE REQUIREMENTS

High temperature stable adhesives are required for bonding of primary load-carrying structures in spacecraft and for new high speed aircraft such as the SST. The exterior structures of high speed aircraft are heated by air friction and will have to withstand temperatures of 450-500°F for hours at a time during flight and for thousands of hours during the service life of the airplane. Honeycomb sandwich materials are to be extensively used in construction because of the strength and rigidity of these structures for minimal weight of material used. Presently, polyimide is the material used for bonding the honeycomb structure because it is stable at 500°F and is commercially available.

Although polyimides do have attractive strength-temperature relationships, a serious limitation exists in their processability. The mechanism by which the polymer cures to a crosslinked structure results in the evolution of a volatile by-product, water (i.e. steam). The evolution of volatiles during the cure-assembly cycle presents serious problems to the processor in certain applications. Some marginal improvement in processing can be made by going to higher bonding pressures. However, this bonding pressure requires

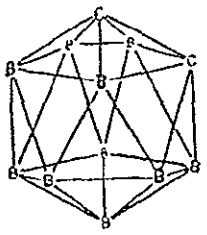
extensive modifications in manufacturing equipment and special jigs.

Epoxies are more attractive candidates for adhesive systems than are polyimides. Their advantages are that no volatiles are evolved during the cure cycle and lower temperatures and pressures are normally required for cure (as compared to polyimides). However, epoxies which are presently available do not satisfy the high temperature requirements of new aerospace applications. Standard bisphenol-A-based epoxy adhesives generally are not useful above 350°F. Some epoxy phenolic can be used for short time periods at 500°F but volatiles are evolved during the cure of these materials.

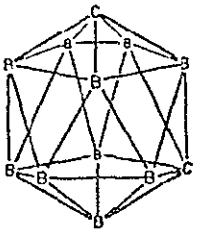
1.2 CARBORANE CHEMISTRY

Because of the excellent handling characteristics of epoxies and an improved order of thermal stability induced by the carborane group to polymer systems, we have undertaken the synthesis and evaluation of a new class of epoxy, the bis (epoxyalkyl) - carboranes. Carboranes are compounds which contain boron, carbon and hydrogen and have recently been reviewed by Williams.[3] Investigations on these materials have led to important advances in bonding theories and in structure-property relationships. Of the many compounds which have been reported, those of the series $C_2B_nH_{n+2}$ have received the most attention. These have been termed "closo" carboranes because of their closed polyhedral structure. There appears to be some degree of resonance stabilization in the carboranes. However, Muetterties and Knoth point out that there are no quantitative figures for this resonance stabilization because of a lack of thermodynamic data on proper reference compounds.[4] The species that has been most readily available for study is 1,2-$C_2B_{10}H_{12}$(closo-1,2-dicarbadodecaborane). This is an icosahedral structure with the ten boron and two carbon atoms occupying the twelve vertices. A hydrogen atom is attached to each carbon and boron atom. In this structure, the two carbon atoms are in the adjacent (ortho) configuration. This carborane is prepared by the reaction of decaborane with acetylene in the presence of a Lewis base. Two other isomers exist in which the carbon atoms are in the 1,7 and 1,12

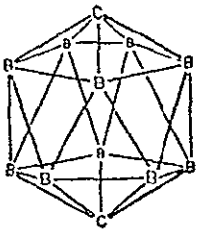
positions. These structures are illustrated below.



1, 2 - $C_2B_{10}H_{12}$



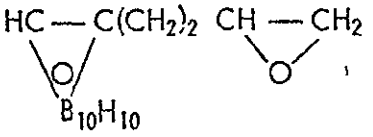
1, 7 - $C_2B_{10}H_{12}$



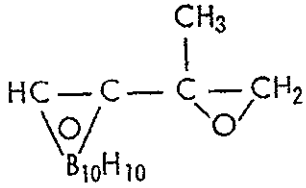
1, 12 - $C_2B_{10}H_{12}$

For clarity purposes, the hydrogen atoms have been omitted from the above structures. The 1,7 (meta) and 1,12 (para) structures can be prepared in quantity by the high temperature isomerization of the 1,2 species.[5,6]

Techniques have been developed for the replacement of the carbon-bonded hydrogen atoms with organic functional groups. The carborane polyhedron is remarkedly stable to oxidizing and reducing agents. This property permits selective oxidation and reduction reactions to be performed on the organic substituents. The carborane cage can be incorporated into the backbone of polymer structures by polymerization of functional groups bonded to the carborane carbon atoms. A strong electron withdrawing character, attributed to the polyhedron appears to affect the chemistry and properties of various groups bonded to the icosahedral carbon atoms. For example, the epoxy linkage in epoxybutylcarborane



is readily cleaved by acids whereas, under identical conditions, the epoxy linkage in epoxyisopropylcarborane



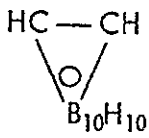
is stable to attack by acids[7]*.

The magnitude of this electron-withdrawing character varies from isomer to isomer. For example, the C- carboxyacids are fairly strong acids with the 1,2 series forming slightly stronger acids than the 1,7 series.[8] Hawthorne and co-workers have conducted quantitative studies of the electronegativities of the 1,2 and 1,7 carboranyl groups and the results are reproduced below.[9]

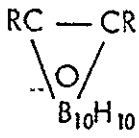
	1,2-Carboranyl	1,7-carboranyl
Hammett σ-constant	~0.47(m),~0.49(p)	——
σ_I	+0.375 ± 0.010	+0.194 ± 0.10
σ_r	+0.003 ± 0.005	-0.039 ± 0.005

These values could be quite useful in helping to explain the effect of the carborane structure on properties of polymers in which it is incorporated.

* The following symbols represent the ortho carborane structure.



unsubstituted carborane



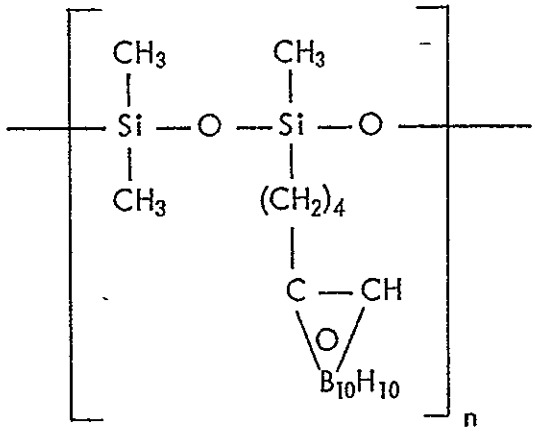
substituted carborane

1.3 THERMAL STABILITY OF CARBORANE CONTAINING POLYMERS

Of the many reported carborane containing polymer systems, thermal analyses in detail have been reported on only three. In one system, the carborane group is pendant to the polymer backbone. In the other two systems, the carborane group is a portion of the backbone.

1.3.1 POLYMERS CONTAINING PENDANT CARBORANE GROUPS

The copolymer 4-(o-carboranyl)-1-butylmethyl siloxane - dimethyl siloxane contains the carborane group pendant to the chain and separated from it by four methylene (CH_2) groups. The polymer has the following repeating structure

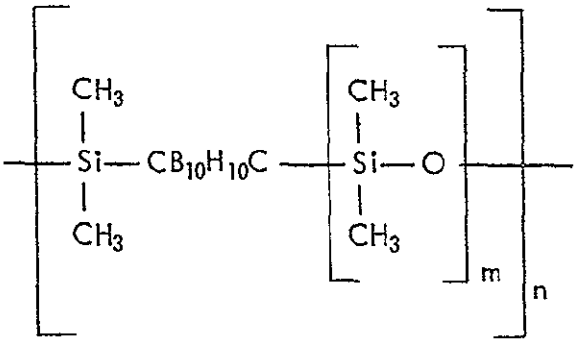


The structure can be considered as a modified alkyl polysiloxane since the carborane nucleus is attached through a 1,4, butylene group to a silicon atom. Delman and co-workers found that this polymer was more resistant to thermooxidative degradation than the conventional dimethylpolysiloxane.[10] These workers concluded that the carborane group had some influence on the stability of the CH_2 group as well as on the pendant methyl groups bonded to silicon. Such stabilizing effects were greater on the methyl

group attached to the same silicon atom as the carboranylbutyl group. They concluded, based on the work of Hinshelwood,[11] that if polar effects of the carborane cage were predominant, the polymer would be less resistant to thermooxidation than polydimethylsiloxanes. This contradicted their findings. They suggested that the bulky carborane cage stabilized the molecule by sterically hindering the acess of oxygen to the methylene groups of the butyl structure.

1.3.2 POLYMERS CONTAINING CARBORANE GROUPS IN THE BACKBONE

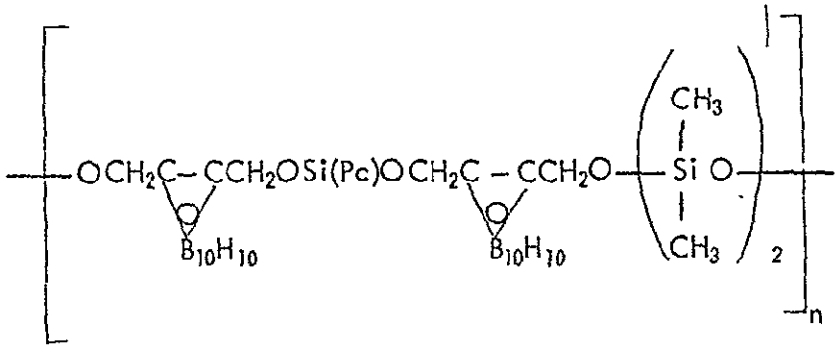
Two series of polymers have been studied. In the first, meta carborane is bonded to dimethylsiloxane groups in a repeating unit with the following structure.



Thermal stability studies conducted by Delman and co-workers indicated the high stability of the meta carborane group; thermooxidative degradation of this part of the structure did not set in until 600°C.[12] The compounds exhibited much lower volatility losses than conventional polydimethylsiloxanes. These authors attributed this stabilization to the influence of the m-carborane group on the thermooxidative decomposition of methyl groups. The group which were adjacent to the carborane cage were more protected than those which were further away.

Finally, Delman and co-workers also studied the thermooxidative stability of siloxane polymers containing both carborane and phthalocyanine groups.[13] These polymers

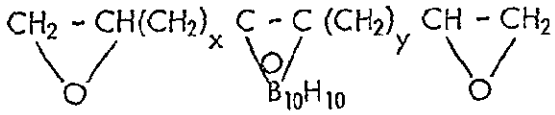
had the following structure where the notation (Pc) represents the phthalocyanine ring.



These authors concluded that the carborane nucleus exerts a stabilizing influence on the oxidative decomposition of adjacent methylene groups.

1.4 PRIOR RESULTS ON BIS (EPOXYALKYL) CARBORANE ADHESIVES

We had previously synthesized and characterized a series of bis (epoxyalkyl) carborane monomers having the following general structure.[1,2]



where
- x = y = 1, Bis (epoxypropyl)carborane
- x = y = 2, Bis (epoxybutyl) carborane
- x = y = 3, Bis (epoxypentyl) carborane
- x = y = 4, Bis (epoxyhexyl)carborane
- x = 2, y = 3, (epoxybutyl) (epoxypentyl) carborane
- x = 2, y = 4, (epoxybutyl) (epoxyhexyl) carborane

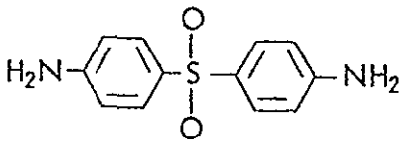
Bis (epoxybutyl)-meta-carborane was also prepared but was not extensively investigated as an adhesive.

These monomers could be cured with conventional catalysts and reactive hardeners to give adhesives with excellent room temperature steel-on-steel bonding characteristics. For example, in the BF_3:$EtNH_2$ catalyzed system, the following bond strengths were obtained.

	Lap-shear Strength (psi)
Bis (epoxybutyl) carborane	2300
Bis (epoxypentyl) carborane	2810
Bis (epoxyhexyl) carborane	3600

However, lap-shear strength dropped off quite rapidly with increasing test temperature. In the bis (epoxybutyl) carborane system, the cured adhesive experiences a drastic loss in strength between 350 and 375°F. At 400°F, only 16 percent of the room temperature strength is retained. The drop-off in lap-shear strength in the bis (epoxyhexyl) carborane system occurs at a lower temperature. Less than 10% of the room temperature strength is retained at 225°F.

Several reactive hardeners were considered as the curing agent. Of these, a poly-functional aromatic amine (p,p' - sulfonyl dianiline)



appeared to give the most encouraging 500°F bond strengths. Others have utilized this hardener for the development of epoxy novalac systems to be used at high temperatures.[14] Because of sluggish reactivity arising from its low basicity, the epoxy-hardener mixture

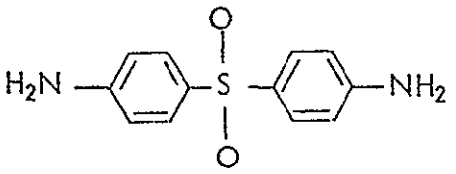
must be heated to effect cure. Grafstein and Dvorak have reported the degradation of the carborane cage by attack of strong bases.[8] However, no such attack of the sulfone hardener (a weak base) is expected on the carborane cage. The well-known thermooxidative stability of the polyarylsulfones has been attributed by Vogel to the electron-withdrawing character of the sulfone group.[15] The sulfone group acts as an electron sink, withdrawing electrons from the aromatic rings and linkages in the polymer backbone to make them more resistant to oxidation. Therefore, this aromatic amine hardener was an attractive choice as a curing agent.

Preliminary tests using bis (epoxybutyl) carborane monomer cured with this aromatic amine gave steel-on-steel lap-shear strengths of 1700 psi at 500°F in air. This system was not modified with fillers or anti-oxidants. The value of 1700 psi represents almost an 85% retention of room temperarure bond strength. Specimens soaked at 500°F in air for 24 hours gave lap-shear strengths of almost 1500 psi at 500°F. Preliminary tests with other carborane derivatives (ex. bis (epoxypropyl) carborane and bis (epoxyhexyl)-carborane) did not give as good high temperature bond strengths.

Because of the excellent handling characteristics of the epoxyalkyl carborane based adhesives, their ready polymerization under a variety of conditions, promising 500°F bond strengths, and the absence of volatile products, it was believed that this system offered an excellent system for practical high temperature bonding requirements. The objective of this program was to improve the steel-on-steel adhesive bonding characteristics of this system and to develop a honeycomb bonding technology.

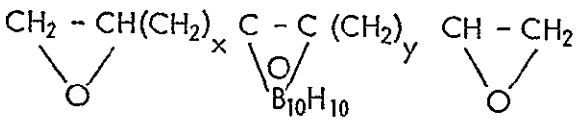
2. DISCUSSION

Based on results of a prior study[2] the hardener which was selected for this evaluation was p, p' - sulfonyl dianiline.



This hardener is termed G-50 for the remainder of this report.

Selection of a monomer for extensive study was based on a limited number of lap-shear test results on several cured epoxyalkylcarborane monomers. As a result of these studies, the monomer which was selected for study was bis(epoxybutyl)carborane, the member of the series where $x = y = 2$.



First, suitable procedures were developed for the synthesis of quantities sufficient for honeycomb testing. Then, the effects of fillers such as aluminum to match thermal expansion coefficients of cured epoxy and steel substrates and asbestos to control flow properties were investigated. Finally, honeycomb specimens, steel core and face sheets, were assembled with the adhesive and evaluated by beam flexure and sandwich peel tests.

2.1 LAP-SHEAR EVALUATION

Substrates were 17/7 PH stainless steel, condition TH 1050.

2.1.1 BIS(EPOXYBUTYL)CARBORANE, HARDENER G-50

A series of tests was initially undertaken to determine a satisfactory cure-assembly schedule for the system consisting only of epoxy monomer and hardener. Investigated were level of hardener, B-staging conditions, cure temperature, cure time, and cure pressure on the bonding area. A low cure temperature was selected so that a subsequent post-cure at a higher temperature could be used to develop a step-wise cure, post-cure cycle. The following results were obtained on this initial work.

- A satisfactory B-stage (from an application standpoint) could be achieved at 300°F over a one to two hour period.
- A cure schedule of 2 ½ hours, 60 psi at a temperature of 300-350°F gave promising room temperature bond strengths.
- Post-cure of 64 hours at 400°F resulted in an increase in 500°F strength. Small changes in 75°F strength were observed at the 25 and 33 phr hardener level whereas a significant drop in 75°F strength occurred at the 43 phr level. The post-cure schedule was modified (toward the end of the program) to improve results and processibility.
- Of the hardener levels which were investigated (25, 33 and 43 phr - stoichiometric amount required for complete reaction of N-H groups with epoxy linkages) the best 500°F results were obtained at the 33 and 43 phr level. Strengths at 500°F at 25 phr were lower.

A summary of these results is given below.

Lap-Shear Strengths (psi)

	75°(No P.C.)	75°(P.C.)	500°F (No P.C.)	500°F (P.C.)
	2740	2640	--	920
	2360	2560	1210	1620
	3220	2520	--	1480

These values are given only to indicate trends which were observed in the initial portion of the program. Higher values are to be expected if improvements in processing and cure-assembly conditions developed during the last portion of the program are incorporated into these systems.

1,2 BIS(EPOXYBUTYL)CARBORANE, HARDENER G-50, ALUMINUM

The use of aluminum filler was investigated with the objective of reducing stresses resulting from differences of the coefficients of thermal expansion of the cured epoxy and steel substrates.

A. Vary Aluminum Level - A series of tests was conducted in which the concentration of hardener was held at 33 phr and the level of aluminum filler was varied. Cure assembly for all specimens was about 305-345°F, 60 psi for 2 ½ hrs. Lap-shear test results are listed below. These results indicated that an aluminum filler loading of 50 phr would give the best lap-shear strengths.

i. 75°F Values Without Post-Cures - These values indicated that the use of aluminum filler would improve 75°F strength.

0 phr aluminum - 2540 psi (Series 44-37)

50 phr aluminum - 3320 psi (Series 44-40)

100 phr aluminum - 3260 psi (Series 44-39)

ii. 75°F Values With Post-Cure (As Shown) - Of the aluminum filled systems, slightly higher results were obtained at 50 phr. The highest value was obtained at 0 phr. However, these values may all be within normal scatter.

	4 hours at 490 ± 10°F	64 hours at 400 ± 10°F
0 phr aluminum	--	2560 (Series 44-33)
50 phr aluminum	2000 psi (Series 44-46)	2360 (Series 44-40)
100 phr aluminum	1780 psi (Series 44-43)	2220 (Series 44-39)

iii. 500°F Values With Post Cure (As Shown) - These values indicated that a 50 phr level of aluminum filler would be more beneficial than 100 phr.

	4 hours at 490 ± 10°F	64 hours at 400 ± 10°F
50 phr aluminum	2180 psi (Series 44-42)	2040 psi (Series 44-40)
100 phr aluminum	1810 psi (Series 44-43)	1860 psi (Series 44-39)

B. Optimization of Cure-Assembly Procedure - Additional efforts were undertaken to improve cure-assembly procedures. These studies included investigations of the effect of the following on lap-shear strength at various hardener levels: glass cloth finish, finger-type panels versus individual specimens, nature of etchant for specimen substrates, substrate degrease treatments, and nature of post-cure conditions. On the basis of the test results given below, a level of 43 phr G-50 hardener was chosen for accumulation of additional lap-shear data (temperature variation and aging) and for assembly of honeycomb specimens. However, it is expected that continued

optimization of bonding conditions would result in an improvement in values listed below.

i. 33 phr G-50 Hardener, 50 phr Aluminum - As a result of these studies a considerable improvement was made in lap-shear strength of this formulation. Highest values obtained were·

75°F (no post-cure) - 3380 psi (Series 44-55)
75°F (post cured 4 hours at 460-470°F) - 2460 psi (Series 44-60)
500°F - 10 minute hold (post cured 4 hours at 460-470°F) - 2440 psi (Series 44-62)

ii. 42 phr G-50 Hardener, 50 phr Aluminum - The best overall values were obtained at this hardener level which is the stoichiometric amount required for complete reaction with the epoxy ring.

75°F (no post-cure) - 3120 psi (Series 44-77)
75°F (post-cured 4 hours at 435-493°F) - 2700 psi (Series 44-77)
500°F - 10 minute hold (post-cured 4 hours at 435-493°F) - 2500 psi (Series 44-77)
500°F - 30 minute hold (post-cured 4 hours at 462-468°F) - 2630 psi (Series 44-74)

iii. 50 phs G-50 Hardener, 50 phr Aluminum - Values fell off quite sharply at 500°F.

75°F (no post-cure) - 3200 psi
75°F (post-cured at 4 hours) - 2160 psi
500°F - 10 minute hold (post-cured 4 hours at 482°F) - 1200 psi

C. Comparison of Assembly Techniques - Vacuum bag techniques for assembly using this system were also evaluated. Lower values were obtained on post-cured samples as compared to values obtained in Carver press assembly. However, this may be due to an unoptimized vacuum bag assembly procedure. Test results are compared below on samples that had been post-cured for 4 hours at 435-493°F.

	Vacuum Bag	Carver Press
75°F	2880 psi (Series 44-80)	2700 psi (Series 44-77)
500°F (10' hold)	1880 psi (Series 44-80)	2500 psi (Series 44-77)
500°F (30' hold)	2130 psi (Series 44-81)	2630 psi (Series 44-74)

Carver Press techniques were chosen for most assembly since best results were obtained using this procedure.

2.1.3 BIS(EPOXYBUTYL)CARBORANE, HARDENER G-50, ALUMINUM , ASBESTOS

Excessive adhesive flow in the cure-assembly cycle of the aluminum filled system indicated that a flow control agent would have to be added to the formulation before honeycomb assembly could be attempted. Asbestos at a level of 29 phr was chosen to reduce the amount of flow.

A. Selection of Bonding Conditions - Several series of specimens were assembled in order to accumulate data on lap-shear strength and to develop B-stage, cure-assembly-post cure procedures which would be ammenable to standard epoxy adhesive processing techniques. All specimens were assembled using impregnated glass cloth carrier. The following procedure was developed.

- Blend epoxy, aluminum and asbestos for 10 minutes at 291-311°F under vacuum to give a heterogeneous slurry.
- Add hardener, heat at 291-311°F under vacuum for 55 minutes.
- Impregnate glass carrier cloth.
- After specimen lay-up, cure at 307-351°F at 60 psi for 2 1/2 hours.
- Post-cure at about 435-465°F for one hour.

The following lap-shear values on post-cured specimens were obtained in Series 44-87 using Carver Presss assembly techniques.

75°F - 3210 psi
500°F (10 minute hold) - 2620 psi
500°F (30 minute hold) - 2300 psi

Specimens were also assembled using vacuum bag techniques. Lap-shear values in Series 44-85 (where post-cure was four hours at 435 - 495°F) are given below.

500°F (10 minute hold) - 2360 psi
500°F (30 minute hold) - 2130 psi
500°F (60 minute hold) - 2360 psi

B. Lap Shear Strength Versus Test Temperature - It was of interest to determine lap-shear strength as a function of test temperature. Sixty specimens were assembled in Series 44-92. Twenty-five of these specimens were tested without post-cure and thirty-five were post-cured at 426 - 470°F for one hour. Highest values from each test condition are listed below.

	Lap-Shear Strength (psi)	
Test Temperature (°F) 10 minute hold	Post-Cured	Non Post-Cured
-320	4040	2940
-65	3480	2840
+75	3060	2960
+350	3100	2460
+450	2960	860
+500	2230	Not tested

A comparison of the highest values shows that in all cases, the post-cured values are significantly higher than the non-postcured except at room temperature where there was only a slight difference. As expected, values fell off quite sharply for the non post-cured above 350°F, probably reflecting the state of incomplete cure. Failure mode changes from predominantely adhesive at -320°F to predominantely cohesive at +450°F. At 500°F, mode of failure ranges from 50 to 80% cohesive.

A plot of high-low values for the post-cured samples appears in Figure 1. Scatter at 75 and 350°F is about the same (320 psi versus 300 psi). The scatter increases at higher and lower temperatures. This Figure shows a reasonably stable lap-shear strength over the entire range with a fall-off in the 500°F area. This data indicates that there may be a "knee" in the lap-shear strength versus temperature curve in the 500°F region.

Additional values obtained at test temperatures above 500°F support the conclusion that there may be a sharp fall-off in strength. These values and the Series in which they were obtained are listed below.

Series	Test Temperature	Lap-Shear Strength
44-84, 85	550°F	970 psi
44-84	584°F	270 psi

In the tests at 550°F and 584°F, the loading curve indicated a yield before break. This behavior indicates that a Tg might have been exceeded.

LAP-SHEAR STRENGTH VERSUS TEST TEMPERATURE

SERIES 44 - 92

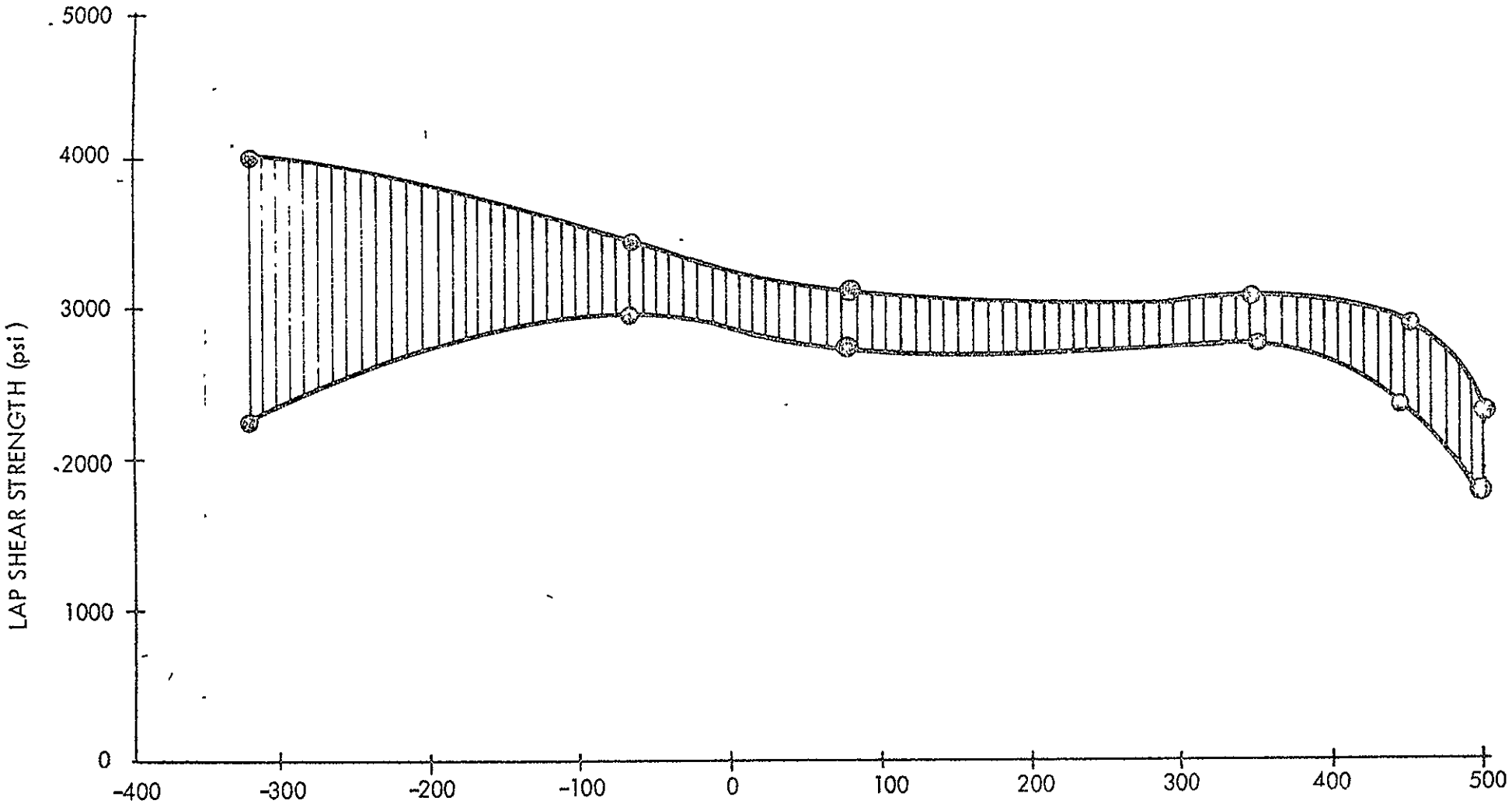


FIGURE 1

C. Lap Shear Strength Versus Aging Time - Twenty specimens were assembled to test the effect of aging at 500° F on lap-shear strength. (Series 44-88,89) All specimens were post-cured for one hour at 435-493° F. Six of the 20 specimens were tested at 500°F with varying holds at temperature.

10' hold	30' hold	60' hold	120' hold
1810 psi	1820 psi	2140 psi	2230 psi
1780			
1660			

The lap-shear values at ten minute hold were unexpectedly low, indicating a problem with the cure-assembly. The increase in values with increasing hold time at the test temperature indicates that additional curing might be taking place in the Instron oven. A DTA analysis was conducted on a chip from one of the 10-minute-hold specimens The thermogram did not show a curing exotherm in the 200-300° C region. Such an exotherm would indicate the presence of unreacted epoxy groups. However, the amount of additional curing which could be taking place might be too small to be picked up by a DTA analysis.

The remaining 14 specimens were aged for various times at 500° F in an air-circulating oven. All specimens were cooled to room temperature and then tested at 500° F (10' hold at temperature). Lap-shear test results are listed below.

Lap-Shear Values (psi)	Aging Time (Hours) at 500° F
2140,2080	2
2420,2020	4
2180,2060	8
1420,1180	16
900,860	40
830,800	66
740,740	120

The specimens that were aged for two hours saw a temperature of 470-500°F. The specimens that were aged for four and eight hours also saw this temperature during the first two hours and then a temperature of 500° F for the remainder of the aging time. The specimens aged for 16, 40, 66 and 120 hours saw 500°F during the entire period of time.

Lap-shear values held-up satisfactorily through the eight hour aging period. Mode of failure was primarily cohesive. However, values fell sharply at the 16 hour and subsequent periods. With increasing aging time and lower lap-shear values, there was a corresponding increase in the degree of adhesive failure. At the 120 hour period, failure was almost completely adhesive.

In Series 44-87, three specimens were post-cured for 160 hours at 500° F in an air-circulating oven. Lap-shear strengths of 772, 570 and 550 psi were obtained and failure was primarily adhesive.

The fall off in lap-shear strength with aging time may be explained in several different ways.

Thermooxidative Degradation - Although the adhesive darkens outside the joint during the aging, there is no such evidence for degradation in the joint area itself. However, others have also reported degradation of lap-shear strength using stainless steel substrates. For example, Krieger and Politi report that HT 424 (an epoxyphenolic supplied by American Cyanamid) shows significant retention of 500°F lap-shear strength after 200 hours aging at 500°F in air on aluminum substrates.[16] On 17/7 PH stainless steel substrates, it shows a negligible loss in strength after 500 hours at 500°F in nitrogen. However, in an air atmosphere, on stainless steel, 500°F band strength is completely degraded after 100 hours exposure at 500°F.

Eby and Brown[17] have recently reviewed bond failure caused by oxidation of the adhesive. Adhesives which give high lap-shear strengths in aluminum-to-aluminum joints in air at 550°F degrade rapidly when used between

stainless steel. These authors suggest that oxides of iron, nickel or chromium are present on the metal surface and that these specimens catalyze the decomposition. In addition, Eby and Brown suggest that the chemical structure of the adhesive influences the intrinsic thermooxidative stability of the adhesive as well as its stability with respect to degradation caused by the substrate. For example, as the phenolic content of an adhesive is increased, the thermo-oxidative stability on stainless steel decreases. Thermo-oxidative stability in adhesive systems to be used on stainless steel substrates is often improved by the addition of anti-oxidants. Such improvements might also be realized in the bis(epoxybutyl) carborane system.

Brittle Character - The aging treatment at 500°F may introduce a degree of strain into the adhesive (perhaps by additional cross-linking) which results in the adhesive being pulled away from the steel substrate. Audible clinking sounds were noted just after the specimens were removed from the aging oven and placed on the bench top. This suggests that a pull-away from the surface occurs during the temperature cycle and not at temperature. A test of this hypothesis would be to eliminate the temperature cycle to give a continuous aging-test sequence.

Non-Optimized Cure Cycle - The data indicates there is a possibility that a change in the post-cure cycle might result in an improvement in lap-shear values. The specimens that had been aged up to 80 hours at 500°F actually experienced an initial two hours at 470 - 500°F. Values of six specimens

were above 2000 psi. The specimens that were aged for 16 hours and longer did not experience this lower temperature treatment and lap-shear values fell sharply.

An extensive series of tests would have to be conducted to determine which of the above three possible causes of bond-failure is operating in the aging studies on the bis(epoxybutyl)-carborane system.

2.2 HONEYCOMB EVALUATION

The adhesive system bis(epoxybutyl)carborane - 43 phr G-50 hardener - 50 phr aluminum - 29 phr asbestos) was evaluated in stainless steel honeycomb panels. Face sheets were 17/7 PH stainless steel, condition TH1050. Core was also of the above type of steel, 0.5 in thick, 0.25 in cell size,* 0.002 in foil size, density 8.7 lbs/ft^3 and non-perforated. Specimens were evaluated by beam flexure and sandwich peel tests. Initially, specimens were assembled and tested to develop lay-up and assembly techniques (both Carver press and vacuum bag), substrate cleaning procedures and post-cure conditions. Carrier cloth impregnated with B-staged adhesive was used for this work. The same cure-assembly-post cure cycle was used for honeycomb assembly as was used for lap-shear specimen assembly.

Because of a lack of comparitive data on high performance epoxy adhesives, an available quantity of Metlbond 329 was also evaluated in honeycomb panels using beam flexure tests and sandwich peel tests. This data was used for comparitive purposes only and is not intended to represent optimized values for Metlbond 329 which might be achieved under more idealized cure-assembly conditions.

* Square cell was used in this work

2.2.1 BEAM FLEXURE TESTS

In the bis(epoxybutyl)carborane system, ultimate loads were obtained of 1580 pounds at 75° F and 805 pounds at 500° F (10 minute hold at temperature). There was excessive flow of the adhesive during the cure assembly process and this resulted in a starved glue line. There was some evidence of core failure in the specimen which gave the value of 1580 pounds at 75° F. However, in all other specimens tested at 75° F, failure was in the adhesive at the face sheet-core interface. In all tests conducted at 500° F, failure was also in the adhesive.

Beam flexure tests with Metlbond 329 gave ultimate loads of 1690 pounds at 75° F and 320 pounds at 500° F.

This comparison indicates that the bis(epoxybutyl) carborane system is slightly poorer at 75°F than Metlbond 329 but may be significantly better at 500° F. However, any further tests and comparisons will have to await the development of a system with better flow control properties.

2.2.2 SANDWICH PEEL TESTS

In the bis(epoxybutyl)carborane system, peel torques up to 6.5 inch pounds/inch width of specimen were obtained at 75° F. At 500° F (10 minute hold at temperature) peel torques up to 3.1 inch pounds/inch of specimen were obtained. These values compare with values obtained with Metlbond 329 of 13.5 and 7.1 inch pounds/inch width of specimen at 75 and 500° F respectively. Again any further tests and comparisons will have to await the development of a system with better flow control properties.

2.3 DEVELOPMENT OF CARRIER CLOTH TECHNOLOGY

The carrier cloth technology of the B-staged bis(epoxybutyl) carborane system was developed. This enabled the loading of adhesive in the glue line to be more accurately controlled, and the B-stage more easily handled. Calendering techniques were not investigated since this type of procedure would have required more material than was available for this program. The impregnated cloth had to have tack so that it would adhere to substrates, particularly during honeycomb assembly. In addition, flow during cure had to be controlled particularly during honeycomb assembly so that good fillets could be obtained while an adequate amount of adhesive was retained in the glue line area.

Early efforts indicated that, in order to obtain impregnated cloth with satisfactory tack, the B-stage period had to be shortened to about 55 minutes. However, the B-stage adhesive had an excessive amount of flow when heated at the temperature used for cure-assembly of lap-shear specimens. Asbestos was therefore added to the formulation at level of 29 phr. Use of asbestos resulted in a reduction in flow. Thickness of impregnated glass cloth was 0.011 inches and loading of B-staged adhesive was 0.07 pounds per square foot of glass cloth. Tedlar backing could be applied to both sides of the impregnated cloth. The tape was soft and flexible and could be creased without cracking. Tape stored at $-10^{\circ}F$ for periods up to 12 days did not experience a loss in degree of tack nor was there any apparent crystallization. Tape stored at room temperature for periods up to four days experienced a noticeable decrease in degree of tack with the onset of

crystallization in the film. However, attempts to prepare honeycomb specimens using this procedure still resulted in excessive flow. Additional effort will have to be undertaken, perhaps with other types of additives, in order to better control the flow properties of this system.

2.4 SYNTHESIS OF INTERMEDIATES AND MONOMERS

One objective of this work was to develop suitable high yield preparatives routes for intermediates and monomers. Of particular concern was the preparation of carborane from decaborane. On previous efforts,[1,2] we had prepared carborane by a step-wise process which was concluded by a permanganate oxidation step. Epoxy monomers were prepared from this carborane intermediate in high yield and could be cured with conventional curing agents to give adhesives with excellent bond strengths. For the present program, we switched to a route which involved the reaction of decaborane with diethyl sulfide and acetylene. This route is known to give a yield of carborane which is higher than the overall yield of the previous route. However, others have reported that carborane prepared by this method contains sulfur impurities which are difficult to remove.[7] It was conceivable that these impurities could hinder curing reactions and affect lap-shear strength. However, this was not the case. Carborane prepared by the diethylsufide-acetylene process was converted to the desired epoxy derivative using the following reaction sequence

carborane ⟶ bis(alkenyl)carborane ⟶ bis(epoxyalkyl)carborane

High purity epoxy monomer was obtained which, when cured, had excellent steel-on-steel adhesive bonding characteristics.

2.4.1 PURIFICATION OF DECABORANE

During the first part of the report period, we found that high purity decaborane was required for a high yield synthesis of carborane. The stock decaborane is yellow and probably contains quantities of higher boron hydrides (arising from the pyrolysis reaction normally used

[illegible] decaborane). Several vacuum sublimation systems were evaluated for the pur- [illegible]. A total of 14.40 pounds of crude material was purified to give 12.26 pounds [illegible] decaborane. Decaborane used was government-furnished.

[illegible] PREPARATION OF CARBORANE

[illegible] was prepared according to the following reaction sequence.[18]

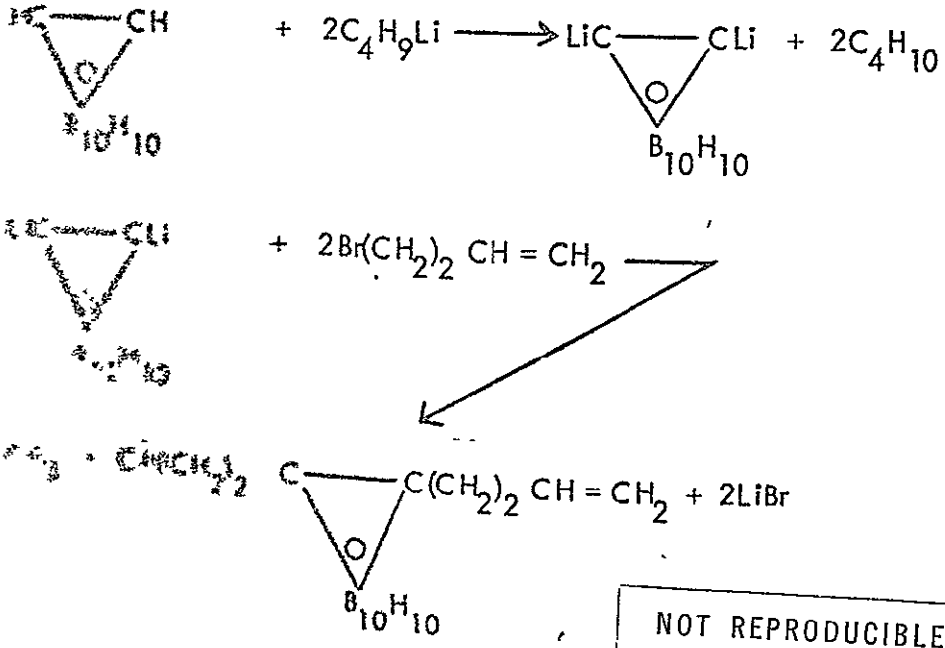
$$B_{10}H_{14} + 2(C_2H_5)_2S \xrightarrow{\text{n-propylether}} B_{10}H_{12}\left[(C_2H_5)_2S\right]_2$$

$$B_{10}H_{12}\left[(C_2H_5)_2S\right]_2 + C_2H_2 \xrightarrow{\text{n-propyl ether}} C_2B_{10}H_{12} + H_2 + 2(C_2H_5)_2S$$

[illegible], the charge of decaborane was increased from 100 g. to 225 g. A [illegible] conversion of charged decaborane to carborane was 70 percent. Thirty-four [illegible] were performed and 9.33 pounds of carborane obtained.

[illegible] PREPARATION OF BIS(BUTENYL)CARBORANE

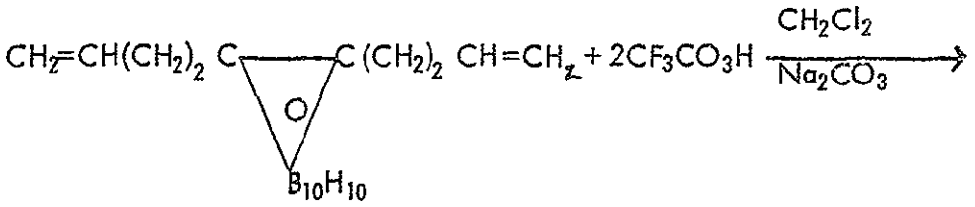
[illegible] was prepared by the following reaction sequence:

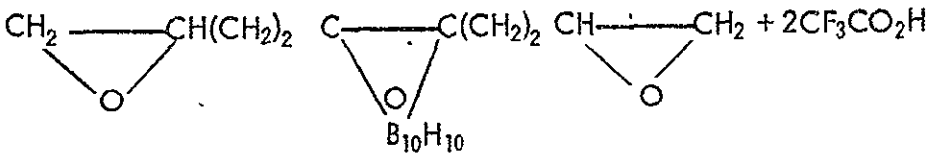


During this work, the charge of carborane per reaction was increased from 21.6 g to 225 g in a step-wise fashion. A typical conversion of charged carborane to bis(butenyl)-carborane was 81 percent. Twenty-one reactions were carried out and 12.81 pounds of product obtained.

2.4.4 PREPARATION OF BIS(EPOXYBUTYL)CARBORANE

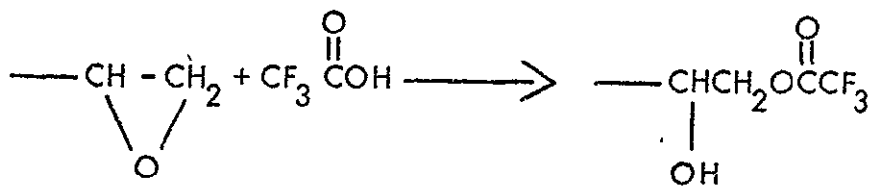
Bis(epoxybutyl)carborane monomer was prepared by the following reaction:





During this work, the charge of bis(butenyl)carborane per reaction was increased from 24.5 g to 225 g. Typical conversion of butenyl to epoxybutyl of 95 percent were realized. Thirty-five reactions were performed and 9.23 pounds of pure product were obtained.

In several reactions (not included in the summary above) semi-solid products were obtained which were difficult to distill. Primary evidence for the impurity was the presence of a band at 1780 cm^{-1} in the infrared spectrum, characteristic of a carbonyl stretch when that group is bonded to R_f, a broad band at 3500 cm^{-1} (hydrogen bonded OH) and a sharp band at 3620 cm^{-1} for free OH[19]. This impurity has been identified as the trifluoroacetate adduct obtained by a ring-opening reaction. Expoy ring-opening can take place by attack of trifluoroacetic acid, a by-product of the reaction, as illustrated below.



The experimental procedure calls for the addition of sodium carbonate to the reaction. The purpose of the sodium carbonate is to remove trifluoroacetic acid, reacting with it to form the sodium salt. This reaction reportedly is faster than the reaction of the acid with the epoxy ring to give ring-opening and the trifluoroacetate adduct.[20] Since the reaction of trifluoroacetic acid with sodium carbonate is heterogeneous, a very fine powdery grade is a requisite to prevent the ring-opening reaction from taking place. In those reactions where impure product was obtained, a granular grade of sodium carbonate had been used.

3. EXPERIMENTAL

3.1 TECHNIQUES

3.1.1 SUBSTRATE PREPARATION FOR LAP-SHEAR SPECIMENS

Only enough specimens were cleaned on any occassion to assemble the desired number of specimens. When necessary, prepared substrates were stored in a closed container for several hours until assembly was initiated. In this manner, contamination from the lab atmosphere was minimized. An extra specimen was processed along with the specimens to be used for assembly. A water-break test was conducted on this specimen after the final drying procedure (described in C below). In this manner the wetting properties of the substrate surface after the final cleaning step could be evaluated. Details for substrate preparation are given below.

A. Degreasing — Prior to Series 44-68 all substrates were cleaned by rinsing with methylethylketone. Beginning with a new supply of steel (0.063 inch) used on Series 44-63 to 44-82, it was found that this procedure did not remove all organic impurities. Water break tests were not always completely satisfactory. The steel had been supplied with an adhesive backing which was not removed with MEK. Beginning with Series 44-68 until Series 44-82 substrates were cleaned first with benzene and then with methylethylketone. Water break tests were almost always satisfactory.

B. Acid Treatment — Subsequent to degreasing, all substrates were cleaned with one of the three procedures described below.

i. Procedure I — Two acid solutions were used for this procedure. The first solution had the following composition by volume

Nitric Acid (70%)	10%
Hydrofluoric Acid (50%)	2%
Water	88%

Substrates were immersed in this solution for 10 minutes. They were then rinsed with tap water. Then, the substrates were immersed in a solution having the following composition for 10 minutes at 160°F.

Sodium Dichromate	28.5g
Sulfuric Acid	285g
Water	to make one liter

After this treatment, the substrates were thoroughly rinsed with cold running tap water and distilled water.

ii. Procedure II — The solution has the following composition:

500 ml of concentrated hydrochloric acid
20 ml of 30% hydrogen peroxide
100 ml of formalin
450 ml of distilled water

The substrates were immersed in this solution at 60-65°C for 10 minutes. They were removed and washed for one minute each in three successive baths of distilled water at room temperature.

iii. Procedure III — The solution was prepared with the following composition:

33 ml of distilled water
33 g of sodium dichromate
1000 ml of concentrated sulfuric acid (S. G. 1.87)

The strips were immersed in this solution at 57-85°C for fifteen minutes. They were then rinsed by being placed successively in three baths of distilled water for one minute each.

C. Final Specimen Dry — Subsequent to the acid treatment, the substrates were transfered to a perforated stainless steel container and dried for one hour at 70°C in a

small gravity convection oven. Beginning with Series 44-86, a special stainless steel rack was used to maintain spaces between specimens during the drying cycle. In addition, drying time was reduced to 30 minutes.

3.1.2 B-STAGING PROCEDURES

Procedures for B-staging the adhesive for a particular assembly are described in the tables which accompany this report.

3.1.3 LAP-SHEAR SPECIMEN ASSEMBLY

Both individual and panel specimens were assembled in this work. Individual lap-shear specimens were assembled in a special mounting fixture which could take a total of five specimens. The size of the strip used for individual specimen assembly was 5.094 in x 1.000 in.x 0.050 in. In some cases, 0.063 in. thick steel was used. This is indicated on the tables at the end of the report. Where no notation is made, 0.050 in. thick steel was used. Dimensions of the finger panels are shown in Figure 2. Prior to Series 44-82, B-staged adhesive was applied to the substrates by a hot melt technique. Subsequent assemblies were carried out with impregnated carrier cloth. Overlap area for all specimens was 1.0 x 0.5 in. and was fixed by alignment pins on the mounting fixture. The B-staged epoxy was warmed and carefully applied to the adherends. The latter were then warmed with an air gun and the adhesive was spread over the overlap surfaces. A strip of glass cloth was placed in the overlap area as a carrier. Prior to Series 44-85, all specimens before final assembly were outgassed in a vacuum oven to remove air which had dissolved in the adhesive during the application. Beginning with Series 44-85, this step was omitted.

The completely assembled individual specimens or panels on the mounting fixture were placed in a Carver Press or a box for vacuum bag assembly. Details on temperature, time and pressure for the cure cycle are given in the tables on each individual series. Panels

were cut into individual specimens subsequent to the post-cure.

3.1.4 HONEYCOMB PANEL ASSEMBLY

Procedures for honeycomb assembly are described in a separate section.

3.1.5 POST-CURING AND AGING STUDIES

Post-cure was conducted in a gravity convection oven. In several series, however, post-cure was conducted in an air-circulating oven. Details for each Series are given in the tables which accompany this report. Aging studies were carried out in an air-circulating oven.

3.2 LAP-SHEAR TEST EVALUATION

Substrates for this work were 17-7 PH stainless steel, condition TH 1050 tested under procedures outlined in Mil Spec MIL-A-005090E (Wep). Complete details of all B-staging, cure-assembly and post-cure procedures are given in the tables in the Appendix. Pertinent results of many of these tests are given in the following sections.

All specimens were tested in an Instron Tensile Tester in an air atmosphere. An Instron Environmental Test Chamber was used for tests over the -65 to +584°F range. The -65°F temperature was controlled with a CO_2 atmosphere. Tests at -320°F were conducted using liquid nitrogen as the coolant. Each specimen was brought to the test temperature (about 5 to 10 minutes required), equilibrated at the test temperature for the desired time period, and tested. Unless noted, all specimens were equilibrated for 10 minutes and tested.

3.2.1 BIS(EPOXYBUTYL)CARBORANE - G50 HARDENER

A. Vary Hardener Concentration — Specimens were assembled with bis(epoxybutyl)-

carborane cured with 25, 33 and 43 (stoichiometric) phr G-50 hardener. Post-cure was 64 hours at 400 $\pm$ 10°F. Data for this work (Series 44-32, 33 and 41) is summarized in Table I. The highest lap-shear value at 75°F without post-cure was obtained at 43 phr. Room temperature values with post-cure were all about the same. At the 43 phr level, post-cure resulted in a drop in room temperature strength. Lap-shear values at 500°F were significantly lower at the 25 phr level as compared to 33 phr and 43 phr where they were about the same. The slightly higher values at the 33 phr level may be normal scatter. The lower 500°F strengths at the 25 phr level probably reflect a lower degree of cross-linking with the lower concentration of hardener.

TABLE I

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane G50 Hardener

Vary Hardener Concentration

	Series 44-32 25 phr	Series 44-33 33 phr	Series 44-41 43 phr
75°F, No Post-Cure	2740 psi 2420 1930 1000	2360 psi 2120	3220 psi 3040 2100
75°F, Post-Cured	2640 2360 2240 2000	2560 2420	2520 2340 2040
500°F, Post-Cured	920 700 400	1620 1210 990	1480 1160 990 790

B. Effect of Post-Cure on 500°F Lap-Shear Strength — Several Series of specimens at a 33 phr G-50 concentration were assembled to determine the effect of a 64 hour post-cure at 400°F on 500°F lap-shear strength. Cure-assembly conditions were 302-351°F, 60 psi for 2½ hours. Post-cure at a temperature above that of the cure temperature results in an increase in 500°F strength in air. For example, test results at 500°F for Series 44-19 to 44-22 show a range of 230-1210 psi for specimens without post-cure and 1260 to 1860 psi for specimens with post-cure. Creation of higher cross link density on post-cure probably accounts for this trend.

C. Variation of Cure Temperature — The object of Series 44-27, 28 and 29 was to observe the effect on lap-shear strength of decreasing the cure temperature and pressure to 244-282°F and 30 psi from 302-351°F and 60 psi.

- In Series 44-29, cure time was 3 hours. Bond strengths without post-cure were 560 psi at 75° and 160 psi at 500°, indicating incomplete cure. Post-cure of 7 hours at 302°F followed by 72½ hours at 392°F gave strengths of 2100 and 1430 psi at 75 and 500°F respectively.

- In Series 44-28, the cure time was increased to 5 hours and all samples were post-cured at 302°F for 7 hours followed by 72½ hours at 392°F. Strength at 75°F was 1420 psi and 1680 psi at 500°F.

- In Series 44-27, the cure time was 22 hours and post-cure was 64 hours at 338°F. Strengths without post-cure were 2380 psi at 75°F and 980 psi at 500°F. After post-cure, strengths were 2430 psi and 1160 psi at 75 and 500°F respectively.

On all specimens cured at the low temperature, the epoxy was very brittle on the steel surface outside the overlap area. A step-wise cure seemed to improve the 500°F bond strength. A long cure time gave acceptable room temperature properties. However, there appeared to be no advantage to decreasing the cure temperature. Therefore, a cure

temperature of about 302-351°F was used for most work on the program.

3.2.2 BIS(EPOXYBUTYL)CARBORANE - G50 HARDENER - ALUMINUM

A. Effect of Varying Aluminum Concentration

i. 33 phr G-50

• Room Temperature Strength - No Post Cure — Lap shear strengths at room temperature are higher for filled systems than for the unfilled system; there appears to be no appreciable difference between 50 phr and 100 phr filler concentration (Table II). Lap-shear values of 3320 psi for filled systems and 2540 psi for the unfilled system were obtained.

TABLE II

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane, 33 phr G-50 Hardener

Vary Aluminum Concentration

Room Temperature, No Post-Cure

100 phr Al		50 phr Al		0 phr Al	
SERIES #	LAP SHEAR STRENGTH (psi)	SERIES #	LAP SHEAR STRENGTH (psi)	SERIES #	LAP SHEAR STRENGTH (psi)
44-39	3260 3090 2120	44-40	3320 3200 2960	44-37	2540 2140

• Effect on Room Temperature Bond Strength-With Post-Cure --- Representative values are listed in Table III. In the 400 ± 10°F - 64 hours post-cure system, slightly higher values were obtained at 0 phr Aluminum (2560 psi as compared to 2360 psi and 2220 psi for the 50 phr and 100 phr filled systems). With a 4-hour post-cure at 490 ± 10°F, higher results were obtained at 50 phr (Series 44-46, 2000 psi) as compared to the 100 phr system (Series 44-43, 1780 psi). For the comparison with a 490 ± 10°F post-cure, data at 0 phr Aluminum is not available under the same assembly-cure conditions.

TABLE III
LAP-SHEAR STRENGTH
Bis(epoxybutyl)carborane, 33 phr G-50 Hardener
Vary Aluminum Concentration
Room Temperature, Post-Cure
64 Hours Post-Cure at 400 ± 10°F

100 phr Al		50 phr Al		0 phr Al	
SERIES #	LAP-SHEAR STRENGTH (psi)	SERIES #	LAP-SHEAR STRENGTH (psi)	SERIES #	LAP-SHEAR STRENGTH (psi)
44-39	2220 1860 940	44-40	2360 2340 2300	44-33	2560 2420

4 Hours Post-Cure at 490±10°F

100 phr Al		50 phr Al		0 phr Al	
SERIES #	LAP-SHEAR STRENGTH (psi)	SERIES #	LAP-SHEAR STRENGTH (psi)	SERIES #	LAP-SHEAR STRENGTH (psi)
44-43	1780 1600 1180	44-46	2000 1930 1830	No Data	

• Effect on 500°F Bond Strength, With Post-Cure — Highest values for each post cure cycle (64 hours at 400 ± 10°F and 4 hours at 490 ± 10°F) were obtained using a 50 phr concentration of aluminum. No data were obtained with O phr aluminum. Representative values are listed in Table IV.

TABLE IV
LAP-SHEAR STRENGTH
Bis(epoxybutyl)carborane, 33 phr G-50 Hardener
Vary Aluminum Concentration
500°F With Post-Cure

64 Hours Post-Cure at 400 ± 10°F

100 phr Al		50 phr Al	
SERIES #	LAP-SHEAR STRENGTH (psi)	SERIES #	LAP-SHEAR STRENGTH (psi)
44-39	1860 1850 1730 1060	44-40	2040 1710 1270 1070

4 Hours Post-Cure at 490 ± 10°F

SERIES #	LAP-SHEAR STRENGTH (psi)	SERIES #	LAP-SHEAR STRENGTH (psi)
44-43	1810 1250 1080 1020	44-42	2180 1780 1410 1200

ii. 43 phr G-50, Stoichiometric — Two series of individual specimens (Series 44-71 and 44-74) were assembled at a hardener level of 43 phr using 50 phr aluminum filler. Post-cure was four hours at 462-472°F. Tests at 500°F were conducted at both 10 minute and 30 minute holds at temperature. Results are summarized in Table V.

TABLE V

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane, 43 phr G-50 Hardener, 50 phr Aluminum

Series 44-71 and 74 Combined

75°F (no post-cure)	75°F (with post-cure)	500°F (with post-cure) 10 minutes	500°F (with post-cure) 30 minutes
3200 psi	2460 psi	2480 psi	2630 psi
3150	2340	2320	2360
3070	2290	2290	
2880	2250	2200	
2560	2240	2120	
	2100		

As we have noted previously, 75°F bond strength decreases with post-cure. This is due to strain introduced into the glue line by additional cross-linking. With post-cure, room-temperature strength is 100% retained at 500°F at a 10 minute hold at temperature. Two specimens were tested at 500°F after a 30 minute hold at temperature. The highest lap-shear strength at 500°F (2630 psi) was obtained under these conditions. This high value on a 30 minute hold could perhaps represent scatter (only 150 psi above the best value obtained on a 10 minute hold at temperature).

One panel Series 44-93 was assembled at the end of the program to determine the effect of aging at 500°F on 500°F lap-shear strength using the 42 phr G-50, 50 phr aluminum formulation. A film type adhesive was used. All specimens were post-cured

for one hour at 435-465°F and aged for varying times at 500°F in an air circulating oven, then tested at 500°F. Values dropped from 1780 psi after two hours post-cure to 860 psi after 30 hours of post-cure. We have no previous data on a non-asbestos system which has been aged at 500°F. Data is lower than values obtained on an asbestos system under comparable aging conditions (Series 44-88, 89). Reasons for these low values are not clear.

iii. 50 phr G-50 — A series of specimens was assembled (Series 44-47) in which the hardener level was 50 phr G-50 (a 16% excess over the stoichrometric amount) and 50 phr aluminum. Post-cure was 4 hours at 482°F. Maximum lap-shear values were:

75°F (no post-cure) - 3200 psi
75°F (post-cured) - 2160 psi
500°F (post-cured) - 1200 psi

Strength at 75°F (without post-cure) is about as good as has been obtained in the bis(epoxybutyl)carborane system at a filler concentration of 50 phr aluminum. However, values at 75°F and 500°F with post-cure are lower.

B. Effect of Glass Cloth Finish on Lap-Shear Strength

i. Comparison of Volan A Versus Heat Cleaned — In the first part of this program, Style 112 glass cloth with a Volan A finish was used as the carrier in the overlap area. Since the Volan A finish may introduce chemical species into the overlap area which could weaken bond strength, the use of heat cleaned Style 112 glass cloth was initiated. A comparison was made between the use of the two types of finish in the system 33 phr G-50, 100 phr aluminum. Lap-shear values at room temperature without post-cure are listed in Table VI.

The highest room temperature strength before post-cure was obtained using heat cleaned glass cloth (Series 44-39). However, because of the spread of values obtained, this may not be due to the type of finish on the glass cloth but to subtle changes in another variable (ex. surface preparation, specimen alignment, uneven heating in the Carver Press etc). Data is not available to compare bond strengths after post-cure. Because of the slightly higher results obtained with the heat-cleaned cloth, all assemblies subsequent to Series 44-39 were with heat cleaned glass cloth.

TABLE VI

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane, 33 phr G-50 Hardener, 100 phr Aluminum

75°F, No Post-Cure

Comparison of Glass Cloth Finish

HEAT CLEANED FINISH		VOLAN A FINISH	
	LAP SHEAR STRENGTH (psi)		LAP SHEAR STRENGTH (psi)
SERIES #	75°F	SERIES #	75°F
44-39, 43	3260 3090 3040 2700 2120 1640	44-30, 31, 38, 35	3020 2920 2880 2700 2700 2680 2580 2000

ii. Pretreatment of Heat Cleaned Glass-Cloth — Completely machined panels were assembled for this work and the epoxy mixture used was bis(epoxybutyl)-carborane-33 phr G-50 - 50 phr aluminum. From Series 44-38 to 44-62 the heat cleaned 112 glass cloth was pretreated at ~338°F for periods of one to four hours prior to its application to the B-staged epoxy on the specimen substrate. In Series 44-62, the cloth was pretreated at ~750°F. The object of raising the temperature was to effect a high degree of cleanliness by pyrolysis and air oxidation of residual organic contaminants acquired during cutting and handling of the strips of fabric. Test results are listed in Table VII and are compared with those of Series 44-61 in which the cloth was pretreated at ~338°F. Post-cure was 4 hours at 460°F. The number after the lap-shear value refers to the panel (first or second) from which the specimen was taken.

Uniformly higher values were obtained in Series 44-62 in which the glass cloth was pretreated at ~750°F. Because of the small number of samples tested and scatter previously observed, it is not certain if this trend is significant. However, subsequent to Series 44-62, all cloth was pretreated at 750°F. Although excellent bond strengths were obtained in Series 44-62 at test temperatures of 75°F and at 500°F scatter at 500°F was still present, and the lowest values were obtained on the specimens from the second tray. The cause of this is not apparent. The portion of the epoxy applied to the substrate was B-staged a little longer (5-10 minutes) for the second panel assembly than the portion used for the first panel assembly. In addition, the second panel assembly on which the epoxy has been spread stood at room temperature, covered with an evaporating dish, for about 2½ hours before being placed in the outgassing oven prior to assembly in the Carver Press. The glass cloth used as the carrier was pretreated for about one hour; the cloth for the second panel assembly was pretreated for up to four hours. Also, the results might indicate that the second panel received less of a post-cure than the first panel.

Although the difference in the temperature pretreatment given the glass cloth did not appear to have an effect on bond strength, the higher temperature treatment did have a profound effect on wettability of the glass cloth by the resin. The aluminum filler was not absorbed into the fiber bundles. Both resin and filler were excluded from the interior of the fiber bundle when the fabric was pretreated at 338°F. This is probably due to non-wettability. Clear resin was observed at the fiber ends outside the joint. The absorption of resin into the fiber bundle displaces air and produces voids near the steel-resin interface. Voids were more plentiful in the higher heat treated case but not absent in the lower heated case.

TABLE VII

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane, 33 phr G-50, 50 phr Aluminum

Pretreatment of Heat Cleaned Glass Cloth

Series 44-61

Cloth Pretreated at 338°F

Lap Shear Test Results (psi)

75°F (no P.C.)	75°F (with P.C.)	500°F (with P.C.)
2820 (2)	2280 (2)	2340 (1)
2710 (2)	2240 (1)	2000 (1)
2540 (1)	2080 (1)	1860 (2)

Series 44-62

Cloth Pretreated at 750°F

Lap Shear Test Results (psi)

75°F (no P.C.)	75°F (with P.C.)	500°F (with P.C.)
3220 (2)	2300 (2)	2440 (1)
2520 (2)	2240 (1)	2350 (1)
2480 (1)	2160 (1)	2050 (2)
		1930 (2)

C. Use of Finger-Type Panels for Specimen Assembly — In the hope of reducing scatter in lap-shear values, three modifications of finger-type panels described in MIL-A-005090E were machined and evaluated. The three modifications are illustrated in Figure 2. These are completely machined (1a) partially machined with additional saw cuts (1b), and partially machined (1c). The completely machined panel was expected to give better results since less cutting is involved after assembly. The other two types were evaluated in the hope that uniform results might be obtained with a simpler panel. Each panel assembly yielded five individual specimens after being cut. Two panel assemblies, giving ten individual specimens, were evaluated for each modification. The adhesive formulation bis(epoxybutyl)carborane - 33 phr G50 hardener - 50 phr aluminum) was B-staged and cured under conditions similar to those used for an assembly of individual specimens before post-cure (4 hours at 490 $\pm$ 10°F).

Results of panel assembly are compared in Table VIII. This comparison indicates that although little difference was noted in degree of scatter, the best all-round values were obtained with completely machined-out panels (Series 44-53; 2880 psi at 75°F - no post-cure, 1820 at 75°F with post-cure, 2100 psi at 500°F with post-cure). For much of the remainder of the program, completely machined panels were used.

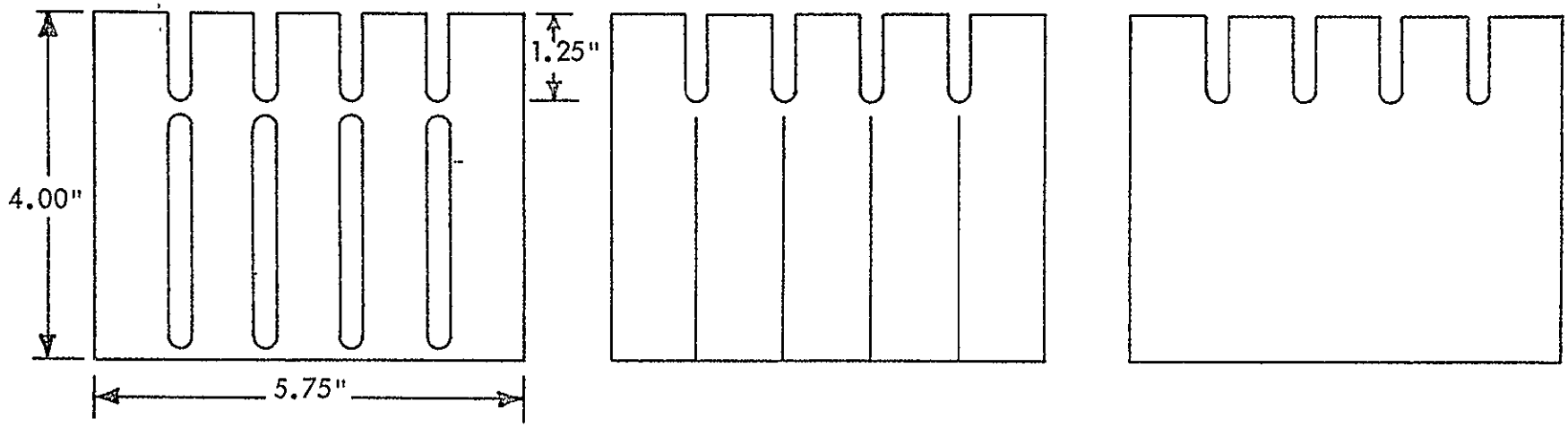
TABLE VIII

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane, 33 phr G-50, 50 phr Aluminum

Comparison of Assembly Techniques

SERIES #	COMMENT	75°F NO POST-CURE	75°F WITH POST-CURE	500°F WITH POST-CURE
44-52	Partially Machined	2850 psi	1780 psi	1420 psi
		2440	1520	1320
		1830	1400	1140
				1090
44-54	Partially Machined with Saw-cuts	2580	1510	2000
		2560	1500	1670
		2480	1490	1440
				310
44-53	Completely Machined	2880	1820	2100
		2800	1780	1840
		2640	1640	1510
				1210
44-42	Individual	2960	1600	2180
		2780	1560	1780
		2480	940	1410



DESIGN OF LAP SHEAR FINGER PANELS

FIGURE 2

D. Nature of Etchant for Specimen Substrates — The procedure used in the first part of this program for preparing the steel substrates was an $HF-HNO_3$, dichromate treatment (Procedure I). This step-wise treatment had been used in our previous efforts.[1,2] However, scatter in lap-shear values at both 75°F and 500°F was considerable. Although water-break tests following the distilled water rinse were acceptable, similar tests conducted (on spare specimens) after the drying cycle (and just prior to application of B-staged epoxy) often were not acceptable. Attempts to better control the atmosphere during drying (for example by using a flowing stream of nitrogen gas) did not have any appreciable effect on bond strength.

Two alternate cleaning procedures were investigated in the hope of obtaining better surface preparation and, at the same time, improving lap-shear strength values. Both procedures are described in detail in a previous section. Results are discussed below. All work was undertaken with the formulation 33 phr G-50, 50 phr aluminum under identical curing and post-curing conditions of 4 hours at 470°F. Special care was taken to determine if there was a correlation between lap-shear strength and the order of panel assembly.

i. $HCl - H_2O_2$ - Formalin — This treatment (Procedure II) is recommended by Lee and Neville and is a one-step process using a solution of $HCl-H_2O_2$ and formalin.[21] A waterbreak test on strips cleaned in the above manner was satisfactory. An initial comparison was made of lap-shear results using specimens cleaned with this solution (Series 44-44) with results obtained using Procedure I, (Series 44-42). There was no appreciable difference in magnitude of values or in degree of scatter. Specimens were individually assembled and this may have accounted for the failure to observe any difference.

Additional lap-shear testing was conducted using specimens cut from completely machined panel assemblies. Results of Series 44-55 in which substrates were treated with Procedure II are listed in Table IX. Excellent lap-shear strengths were obtained at both 75°F and 500°F with post-cure (2300 and 2280 psi respectively).

TABLE IX

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane, 33 phr G-50 Hardener, 50 phr Aluminum

Comparison of Substrate Preparation*

Series 44-55

Procedure II

75°F (no p.c.)	75°F (with p.c.)	500°F (with p.c.)
3380 (1) psi	2300 (1) psi	2280 (2) psi
2720 (2)	2220 (2)	2200 (1)
2600 (2)	2180 (1)	2080 (2)
		2040 (1)

Series 44-59

Procedure II

75°F (no p.c.)	75°F (with p.c.)	500°F (with p.c.)
3100 (2) psi	2400 (1) psi	2340 (1) psi
3010 (1)	2300 (2)	2320 (1)
2950 (2)	2240 (1)	1920 (2)
		1720 (2)

TABLE IX: continued

Series 44-60

Procedure II

75°F (no p.c.)	75°F (with p.c.)	500°F (with p.c.)
2770 (1) psi	2460 (2) psi	2120 (1) psi
2550 (2)	2340 (1)	2000 (1)
2520 (2)	2160 (1)	1470 (2)
		1470 (2)

Series 44-56

Procedure I

75°F (no p.c.)	75°F (with p.c.)	500°F (with p.c.)
2680 psi	1480 psi	1580 psi
2600	1260	1560
2520	1080	1500
		1480

Series 44-58

Procedure I

3280 psi	1600 psi	1900 psi
2600	1440	1720
2550	1280	1120
		990

* The number in parenthesis after the lap-shear value indicates the panel assembly from which the specimen was cut - one or two.

The important point to be noted is the low degree of scatter. Whether the specimens came from the first or second panel assembled does not seem to make any significant difference in this series although the best 75°F value - no post cure - was obtained from panel No. 1. However, this uniformity was not the case in Series 44-59 and 60. The values of 2340 psi at 500°F (Series 44-59) and 2460 psi at 75°F (Series 44-60) are very good. However, in both series, considerable scatter is found at 500°F. In each case, two high and two low values were obtained. The high values were obtained from the first panel assembled whereas the two low values were obtained from the second panel assembled. The age of the cleaning solution also appears to affect lap-shear strength. In Series 44-57, a previously used solution was used for surface cleaning. The highest 500°F strength obtained was 1890 psi.

Two series of specimens were assembled using Procedure I: Series 44-56 and 58. Completely machined panels were also used for this work. Results are listed in Table IX. Considerable scatter was obtained at 75°F and at 500°F with post-cure, and values were not as high as those previously obtained with Procedure I or with Procedure II. This surface treatment using Procedure I may be an inherent problem with this epoxy system. Consistently better results seem to be obtained when the steel surface is treated according to Procedure II.

ii. H_2SO_4 - $Na_2Cr_2O_7$ — Another treatment was briefly investigated (Procedure III). This method involves immersion of the substrates in a solution of H_2SO_4 and $Na_2Cr_2O_7$. Such a solution has been recommended by others when attempting to obtain maximum peel strength on stainless steel.[22] Bikerman has proposed that peel stress is a major component of the lap-shear test. Ten lap-shear specimens were prepared from specimens surface-treated in this manner (Series 44-45) at 33 phr G-50 Hardener with 50 phr aluminum filler. A water-break test performed on an extra specimen at assembly time was acceptable. However, lap shear values under

all conditions fell below optimum values obtained using the other cleaning procedures. Maximum values are listed below (post-cure was 4 hours at 490 $\pm$ 10°F).

75°F: (no post-cure) 2620 psi
75°F: (with post-cure) 1560 psi
500°F: (with post-cure) 1710 psi

No further work was conducted with this cleaning procedure.

E. Substrate Degrease Treatments — Initial cleaning procedures involved immersion of the substrates in a bath of methylethylketone. During the program, a new quantity of steel (0.063 in. thick) was purchased which was supplied with a paper backing bonded to the steel with a pressure sensitive adhesive. This paper was stripped from the substrate prior to the deburring operation. In Series 44-63 and 64, the specimens were given the standard solvent wash with methyl ethyl ketone (MEK) prior to treatment with the etch solution. Water break tests on these specimens were not completely satisfactory. Subsequently, we found that the pressure sensitive adhesive was not soluble in MEK. Substrates for Series 44-66 were cleaned with MEK only whereas substrates for Series 44-68 were cleaned with both MEK and benzene. Water break tests on specimens which had been given both MEK and benzene treatment were quite satisfactory whereas water break on specimens cleaned only with MEK were marginal. The surface preparation used was Procedure II. The formulation was 33 phr G-50 hardener and 50 phr aluminum. Post-cure was 4 hours at 458-462°F. The best 500°F values were obtained in Series 44-68 in which both the MEK and benzene treatments were used. Results are listed in Table X. Subsequent to this work, all 0.063 inch thick substrates were cleaned with the combination surface treatment.

TABLE X

LAP-SHEAR STRENGTH

Bis(epoxybutyl) Tetrahydrofurane, 33 phr G-50, 50 phr Aluminum

Comparison of Substrate Degrease Treatments

Series #	Organic Solvent Cleaning Treatment	Lap-Shear Strength* 75°F (no p.c.)	75°F (p.c.)	500°F (p.c.)
44-66	MEK	3640 (2)	2780 (1)	2080 (2)
		2600 (1)	2660 (2)	1710 (2)
		2540 (2)	2460 (1)	1650 (1)
				1450 (1)
44-68	MEK plus	3380 (2)	2720 (1)	2310 (1)
	benzene	2940 (1)	2640 (1)	2150 (2)
		2660 (2)	2460 (2)	2010 (2)
				1970 (1)

* The number in parenthesis after the lap-shear strength indicates the number of the tray (one or two) from which the specimen was obtained.

F. Nature of Post-Cure Conditions — Two different ovens were used for comparison of post-curing conditions. In this work, the hardener level was 33 phr with aluminum filler at 50 phr. One oven was a gravity convection type, the other was an air-circulating type. In Series 64, specimens were post-cured for four hours in the air circulating oven at a temperature which cycled from 505-520°F. Although lap-shear strengths at 75°F without post-cure ranged up to 2960 psi, strengthswith post-cure at 75°F and 500°F, were suprisingly low. The highest values obtained were 1130 psi at 75°F and 950 psi at 500°F. The drastic drop in adhesive bond strength with post-cure compared to previous values in a gravity convection oven might be due to a problem with use of the air-circulating oven, the higher temperature in the oven or the 15°F cycle range in that oven.

In the above work, a methyl ethyl ketone (MEK) cleaning treatment of the steel substrate was used prior to the acid etch. In the belief that the surface had not been adequately degreased with this solvent treatment, a series of tests was conducted in which the organic cleaning treatment consisted of a benzene rinse followed by a MEK rinse. Two series of specimens were prepared and post-cured at about the same temperature; one set in the gravity convection oven at 458-462°F (Series 44-68) and one set in the over circulating oven at 453-467°F (Series 44-67). Results are listed in Table XI.

There was no appreciable difference in color of the cured epoxy outside the specimen overlap area, indicating that air circulation versus gravity convection apparently does not make any difference in amount of oxidation that takes place. The best results with post cure were obtained on specimens cured in the gravity convection oven (Series 44-68). A possible cause of low values and scatter for specimens in Series 44-67 in which the air circulating oven was used might be the wider cycle in temperature, 14°F as compared to 4°F in the gravity convection oven. This cycle could result in stresses being set-up which are reflected in lower bond strength.

TABLE XI

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane, 33 phr G-50 Hardener, 50 phr Aluminum

Comparison of Post-Cure Conditions

Series #	Post Cure Condition	Lap-Shear Strength (psi) 75°F (no p.c.)	75°F (p.c.)	500°F (p.c.)
44-67	453-467°F Air Circulation	3660	2180	2000
		3200	1740	1680
		2730	1680	1540
				1300
44-68	458-462°F Gravity convection	3380	2720	2310
		2940	2640	2150
		2660	2460	2010
				1970

G. Vacuum Bag Assembly — Work was undertaken to develop vacuum bag assembly techniques. With this procedure, a lower and more evenly distributed pressure than could be obtained with Carver Press techniques would be applied. A fixture was constructed in which individual lap-shear specimens, lap-shear panel assemblies and 3"X8" honeycomb specimens could be assembled.

Testing was conducted with specimens assembled using the formulation 42 phr G-50 and 50 phr aluminum. Several specimens for each series were post-cured in a gravity convection oven at 435-493°F for four hours. Two trays of 5 individual specimens each were assembled per series. B-staged resin was applied to the substrate by the hot melt technique. However, contrary to the procedure used for Carver Press assembly, the specimens were not outgassed at 120°C in a vacuum oven prior to assembly. It was expected that the vacuum used for the bagging operation would pull out air dissolved in the epoxy before the epoxy cured to a highly viscous state. The first tray of each series was assembled in the fixture

and the fixture attached to the vacuum connection in the oven. The vacuum was applied (14 psi) and heating initiated. About 45 minutes was required to bring the oven to the desired temperature. The oven temperature was maintained at 340-360°F for a 2½ hour period. This temperature cycle is slightly higher than that previously reported for Carver Press assembly, 302-350°F. It was assumed that the time required to equilibrate the second tray to oven temperature was quite small. Therefore, no equilibration was used and the time period under vacuum in the oven was only 2½ hours.

Specimens obtained in Series 44-78 were wedged-shaped and not satisfactory for testing. Wedging was reduced in Series 44-79 but not completely eliminated. Lap-shear values were lower than those previously obtained using a Carver Press. In Series 44-80 and 81, improved spacers were used in the assembly process and wedging was reduced still further. Lap-shear values are listed in Table XII.

Lap-shear values at 75°F without post-cure were low as were the values at 500°F with post-cure on a 10 minute hold. Two of the three values obtained at 500°F on a 30 minute hold (2130 and 2080 psi) were higher than the values obtained on a 10 minute hold. It is possible that this differential is due to additional cure at 500°F in the hold cycle. Most encouraging were the values obtained at 75°F with post-cure. All these values were uniformly higher than those obtained without post-cure.

An inspection of the glue line showed that bubbles had formed which had not been removed by the vacuum. In several instances, there were brown channels which extended to the edge of the overlap area. Also, brown spots were present indicating that air had been entrapped. Exposure of the cured epoxy to air at elevated temperatures results in discoloration of this type due to oxidation. In addition, the majority of the joints appeared starved and the resin ran considerably more than it did in comparable Carver Press assemblies. The most likely explanation is that the resin used for the Carver Press work had a higher melt visiosity due to additional B-staging in the outgassing step subsequent to application on the metal substrates.

TABLE XII

LAP-SHEAR STRENGTH

Bis(epoxybutyl)carborane, 42 phr G-50 Hardener, 50 phr Aluminum

Vacuum Bag Assembly Techniques

75°F (no p.c.)	75°F (p.c.)	500°F (p.c.) 10 minutes	500°F (p.c.) 30 minutes
1910 psi	2880 psi	1880 psi	2130 psi
1320	2700	1840	2080
	2580	1780	830
	2520	1650	
	2450	1630	
	2400		
	2380		

3.2.3 BIS(EPOXYBUTYL)CARBORANE - G50 HARDENER - ALUMINUM - ASBESTOS

Asbestos was added to the formulation in an attempt to control the flow properties. The formulation for all the work described below is 43 phr G-50, 50 phr aluminum and 29 phr asbestos (except in one instance where the level was 36 phr). The B-staged adhesive was applied in tape form. Fabrication of the tape is described in another section. Except where noted, all assembly was with Carver Press techniques. Prior to Series 44-87, a high magnetite asbestos was used. Beginning with Series 44-87, a low magnetite content asbestos was used (ALCOA, CRL-8, 325 mesh, 0.5% magnetite content).

A. Variation in B-Stage Time — The object of Series 44-91 was to determine the effect of a slightly longer B-stage time on lap-shear values. A portion of the B-stage mixture was removed after 55 minutes and glass cloth was impregnated. The remainder of the mixture was B-staged for a total of 70 minutes and this was used for impregnation of a second batch of glass cloth. The portion of the mixture that was B-staged for 70 minutes was more viscous than the portion that was B-staged for 55 minutes. However, there was little difference in the degree of tack of the impregnated cloth. All specimens were post-cured for one hour at 435-495°F, and tested at 500°F (10 minute hold). Test results are listed below.

55 minute B-stage	70 minute B-stage
2490 psi	2320 psi
2340	2300
2180	1940
2080	1920
1980	1750

There appears to be no significant difference in lap-shear strength between the two B-stage periods.

B. Variation in Post-Cure Time

i. Series 44-86 — The usual magnetic stirrer was replaced with a mechanical stirrer in an attempt to achieve more efficient mixing and prevent lumping of the magnetic particles. The charge of epoxy used was 10.0g. During the B-staging period, the mixturewas alternately stirred with a mechanical stirrer and then outgassed without stirring. As a result, the mixture was outgassed for only abotu one-half of the 55 minute B-stage time. Cloth was impregnated using a Carver Press with 6"X6" platens at a temperature of about 75°C. Circular forms about 5" in diameter were obtained and there was some tack. The impregnated cloth which was prepared appeared quite uniform and there was no visual evidence of clumping of magnetic materials. A set of ten specimens was assembled and post-cure was varied from one to four hours at 435-495°F. Lap-shear results are listed below for specimens tested at 500°F (10 minute hold at temperature).

Post-Cure Time (hours)

one	two	four
2020 psi	1820 psi	1980 psi
1840	1700	1840
1720	1600	1770

One specimen was post-cured for one hour and tested at 500°F with a 30 minute hold. The lap-shear value was 1720 psi, within the range of the values obtained on the ten minute hold. The values indicated that a one hour post-cure was probably sufficient. The porosity of the glue line was quite high. The reason for high porosity may be the inefficient outgassing during the B-stage process used for this series. Alternatively, porosity may be due to a high degree of run-out of the adhesive. (The impregnated cloth did have a certain degree of tack).

ii. Series 44-90 — Ten specimens were assembled using individual substrates. The B-stage time was 55 minutes. A new lot of green-colored Tedlar film (1 mil thick - a Dupont product designated as 100 BG 15UG) was used in the as-received condition. All specimens were post-cured at 435-495°F for one or four hours. All tests were conducted at 500°F (10' hold at temperature). Results are summarized below.

Lap Shear Test Results (500°F)

One Hour Post-Cure	Four Hours Post-Cure
2220 psi	2340 psi
2180	2220
2100	2180
2080	2100
1910	2100

The averages of the results were 2098 psi and 2188 psi for one and four hour post-cure respectively. This slight difference is in agreement with our previous results (Series 44-86) which indicated that a one hour post-cure would be satisfactory.

C. Increased Loading of Asbestos — Ten specimens were assembled in Series 44-84. A slightly higher amount of asbestos, 36 phr as compared to 29 phr for Series 82 and 83 was used. Assembly-cure conditions were identical to those used on Series 44-82 and 83. Lap-shear test results at 500°F are listed below.

10 minutes	30 minutes
2860 psi	2420 psi
2480	2340
2240	2000
2060	1980

Specimen temperature was monitored throughout the above tests by means of a thermocouple. Temperatures of all specimens were 500°F except for the specimen which gave the highest value of 2860 psi where the temperature was 492°F. It is possible that the slight drop in temperature (8°F) could have caused a high test result. Two specimens were tested at higher temperatures; lap-shear values of 270 psi (at 584°F) and 970 psi (at 550°F) were obtained. These results suggest that there is a sharp fall-off in strength above 500°F and that a Tg might have been exceeded. The results at 500°F are similar to those reported where the level of asbestos was 29 phr. Therefore, it is not expected that the increased amount of asbestos will have any significant effect on lap-shear strength.

D. Variation in Test Temperature — The object of Series 44-92 was to compare lap-shear strength versus test temperature. A Carver Press with 8"X11½" platens was used to impregnate several pieces of glass cloth. The size of the sample obtained was approximately 8"X10". Impregnated cloth was cut to fit the overlap areas. Two sets of lap-shear panels were assembled per day over a six day period. The impregnated cloth was stored in a

refrigerator at -10°C. Twenty-five of the specimens were tested without post-cure. The remaining 35 specimens were post-cured at 426-470°F for one hour. A distribution of specimens was obtained for each test condition.

Results are discussed in depth in a previous section. The data is still plagued with a significant number of low lap-shear values at 500°F. Apparently, there is still a problem with assembly - formulation procedures which shows up in high scatter in the high and low temperature regions. One contributing factor may be possible contamination from the surface of the Tedlar. There seems to be no direct relationship between the day of assembly and the value of lap-shear strength for a particular test condition. It is conceivable that storage time (at -10°C) of the impregnated glass cloth could affect lap-shear strength. An independent series of tests would have to be conducted to investigate this possibility.

E. Variation in Aging Time

i. Series 44-87 — The objective of this work was to obtain preliminary information on the effect of aging at 500°F on lap-shear strength at 500°F. The asbestos was pretreated at 400°C for one hour prior to use. The epoxy (10.0g. charge), aluminum and asbestos were manually blended and heated at 150 ± 5°C with outgassing until the epoxy had melted and a slurry was obtained. The vacuum was broken and the hardener added. Heating was continued at 150 ± 5°C for an additional 55 minutes with outgassing and stirring. At the end of this period, there was no evidence of outgassing. The glass cloth which was impregnated with this B-stage was uniform but tackless. The average temperature during the B-stage was > 150°C. This may have caused a greater than expected degree of cure with loss of tack.

All specimens were post-cured for one hour at 435-495°F. Two specimens tested at 75°F gave lap-shear values of 3210 and 3020 psi. Five specimens were tested

at 500°F with varying time holds at temperature. Results are listed below.

10' hold	30' hold	60' hold
2620 psi	2300 psi	1500 psi
2430		
2300		

There was little or no porosity of the glue line on these specimens. This decreased porosity, as compared to Series 44-86, may have resulted in increased lap-shear values as compared to the values in that series. All failures were primarily cohesive except for the specimen giving the poor value at a 60 minute hold. Here, failure was primarily adhesive.

The remaining three specimens were aged at 500°F for 160 hours in an air-circulating oven. The specimens were then tested at 500°F (10' hold) and lap-shear values of 772, 570, and 550 psi were obtained. Failures were primarily adhesive. Although the epoxy outside the joint had darkened, the portion in the joint area was unaffected. The predominance of adhesive failure is reminiscent of data obtained in prior work where use of a gravity convection oven (Series 44-68) and an air circulating oven (Series 44-67) were compared for effect on post-cure. Use of a gravity convection oven resulted in cohesive failure whereas use of the air circulating oven apparently resulted in adhesive failure and lower 500°F bond strength.

ii. Series 44-88, 89 — The adhesive mixture was B-staged under the same conditions as those described for Series 44-87. The cloth was tacky in this instance. However, the B-staging temperature was < 150°C. The cloth was stored at room temperature for about 16 hours before Series 44-88 was assembled. It was then stored at -10°C overnight until Series 44-89 was assembled. In this way, it was hoped that the impregnated cloth would have the same history for both series. The cloth appeared to lose its tack on standing at room temperature

overnight. All specimens were post-cured for one hour at 435-493°F. Six of the 20 specimens were tested at 500°F with varying holds at temperature. The remaining 14 specimens were aged at 500°F in an air-circulating oven and tested at 500°F. Results are discussed in a previous section.

iii. Series 44-93 — The object of this work was to investigate the effect on lap-shear strength of long-term aging at 500°F of specimens whose substrates had been cleaned according to Procedure I. All specimens were post-cured for one hour at 435-465°F and were aged for various times at 500°F, then tested at 500°F. Lap-shear strength fell from 1500 psi after 2 hours aging to 560 psi after 30 hours of aging at 500°F. This surface treatment does not appear to offer any hope for improving long-term aging data.

F. Vacuum Bag Techniques

i. Series 44-82, 83 — The B-stage was 55 minutes and the impregnated tape was outgassed on the specimens before final assembly. All specimens were post-cured at 435-495°F for four hours. Results are summarized below.

75°F	500°F (10 minutes)	500°F (30 minutes)
2940 psi	1040 psi	1110 psi
2840	940	1050
2780	930	1000
2720	930	980
2630	900	
2500	900	
2480	830	
2400	830	

Although the values at 75°F were very satisfactory, 500°F values dropped drastically as compared to previously reported values from vacuum bag techniques and Carver Press techniques. Possible causes of a drop in strength are:

- outgassing oven - could have been contaminated.
- shorter B-stage - 55 minutes instead of two hours, can affect the curing characteristics.
- use of impregnated tape - variables of use, possible contamination from Tedlar backing.
- asbestos - is known to be impure, contains a metal phase which is magnetic.
- pressure - Carver Press techniques used about 60 psi whereas pressure in the vacuum bag procedure is about 14 psi.

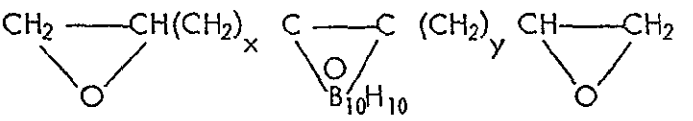
ii. Series 44-85 — Five specimens were assembled using the vacuum bag technique and a back-fill of 45 psi of nitrogen on top of the bag. The impregnated tape was not out-gassed before the cure-assembly operation was initiated. Some difficulty was encountered during the cure-assembly operation. The vacuum bag broke during the first part of the cure resulting in a pressure of less than 14 psi on the specimens and a tempature somewhat less than the oven temperature of 340-360°F (because gaseous nitrogen which had not been preheated was rushing over the specimens). The box was reassembled and cure-assembly continued successfully for an additional two hours. Since the epoxy appeared soft at the end of the cure cycle, the panels were post-cured on the jig and cut apart after the post-cure operation (4 hours at 435-495°F). The following lap-shear strengths were obtained.

500°F (10' hold): 2360, 1970 psi
500°F (30' hold): 2130 psi
500°F (60' hold): 2360 psi
550°F (10' hold): 970 psi

These results indicate that a pressure of about 60 psi on the overlap area will give acceptable 500°F lap-shear properties.

3.2.4 OTHER EPOXYALKYLCARBORANE SYSTEMS

We have previously found that, with the hardener boron trifluoride: monoethylamine, room temperature bond strength increased with an increasing value of $x = y$.[2]



However, preliminary data indicated that high temperature bond strength decreased with increasing value of $x = y$, perhaps because of decreasing thermal stability or a decreasing glass transition temperature. During the program we conducted a limited number of tests to determine if this trend also held in the G-50 hardener system.

A. Bis(epoxypentyl)carborane — Lap-shear specimens were assembled (Series 55-1) using bis(epoxypentyl)carborane cured with G-50 hardener at a concentration of 39.8 phr, the stoichiometric amount required for complete reaction of N-H groups with epoxy groups. No aluminum filler was used in this work. Post-cure was 64 hours at 400°F. Lap-shear failure at room temperature (no post-cure) was primarily cohesive. Failure was primarily adhesive on room temperature and 500°F tests with a 64-hour post-cure at 400 ± 10°F. Data is summarized in Table XIII and compared with results from a bis(epoxybutyl)-carborane series, cured and post-cured under identical conditions (Series 44-41).

Room temperature strengths in Series 55-1 were about 18% higher than those in Series 44-41. However, on post-cure, lower results were obtained in Series 55-1 than in Series 44-41. In Series 55-1 at room temperature, 53% of the original strength before post-cure was obtained. This compared to a 73% retention of strength in Series 44-41. Although there was considerable scatter in the 500°F test values, it is apparent that, under these assembly-cure conditions, higher values are obtained with bis(epoxybutyl)carborane.

TABLE XIII

LAP-SHEAR STRENGTH

Bis(epoxypentyl)carborane, 39.8 phr G-50 Hardener

	Lap-Shear (psi) Room Temperature No Post-Cure	Lap-Shear (psi) Room Temperature With Post-Cure	Lap-Shear (psi) 500°F with Post-Cure
Series 55-1	3800 3660 3700 3650 av.	2080 2020 1960 1800 av.	1360 730 705 380 av. 350
Series 44-41	3220 3040 3130 2100 av.	2520 2340 2300 2040 av.	1480 1160 1105 990 av. 790

B. (Epoxybutyl)(epoxyhexyl)carborane — The stoichiometric amount of G-50 hardener was also used with the epoxy (Series 46-1). No aluminum filler was used. Post-cure was four hours at 500°F. Highest lap-shear strengths obtained were as follows:

75°F (no post-cure)	3500 psi
75°F (with post-cure)	2100 psi
500°F (with post-cure)	350 psi

Failure at 75°F without post-cure was primarily cohesive whereas failure after post-cure (both at 75°F and 500°F) was primarily adhesive. Strength at 500°F was quite low.

C. Bis(epoxybutyl) -, Bis(epoxypentyl)carborane Blend — A limited number of tests were conducted in an attempt to improve the plasticity of the bis(epoxybutyl)carborane system by adding 10 parts bis(epoxypentyl)carborane per hundred parts epoxybutyl resin. In Series 44-72, the composition was 10 phr bis(epoxypentyl)carborane, 33phr G-50 and

50 phr aluminum filler. Post-cure was four hours at 470°F. Highest test results with post-cure were 2420 psi at 75°F and 1940 psi at 500°F. In Series 44-75, the amount of G-50 hardener was increased to 47.5 parts per hundred parts epoxybutyl resin. This value corresponds to the stoichiometric amount of hardener required for complete reaction of N-H bonds with all epoxy groups. Lap-shear strength ranged up to 2220 psi at 75°F and 1260 psi at 500°F (10 minute hold at temperature). Apparently, an increase in the amount of hardener resulted in a decrease in bond strength. Two specimens were tested at 500°F after a 30 minute hold at temperature and values of 1900 and 1700 psi were obtained. This increase in strength might be due to additional cure.

D. Bis(epoxybutyl) -, Monoepoxybutylcarborane Blend — The use of monoepoxybutyl-carborane as a reactive plasticizer in the bis(epoxybutyl)carborane - G50 hardener system was briefly investigated. (Series 44-7,8). Both 75°F and 500°F bond strengths decreased from values obtained without the monoepoxy additive.

3.3 HONEYCOMB EVALUATION

The information obtained during lap-shear testing on formulation and cure-assembly parameters was used as a basis for this work.

3.3.1 MATERIALS

The core material was 17/7 PH stainless steel (Condition TH 1050) non-perforated square honeycomb, 0.5 in. thick and having a 0.25 in. cell size and 0.002 in. foil size. Stainless steel facings were also used; 0.050 in. thick for the flexure specimens and 0.020 in. thick for the sandwich peel test specimens.

3.3.2 DEVELOPMENT OF HONEYCOMB BONDING TECHNIQUES

Vacuum bag and Carver Press techniques were used to bond honeycomb specimens. For all

specimens, the B-staged adhesive was applied as an impregnated carrier tape. Numerous specimens were assembled to develop procedures for application of the tape to substrates and for specimen assembly.

A. Vacuum Bag — Tedlar was initially used as the bagging film. However, handling difficulties were encountered and Capran was substituted. The specimens were assembled on a base plate having a vacuum connection. A sheet of the bagging film was then placed over the specimens. To effect the seal, an aluminum box, provided with an O-ring, was placed over the assembly with the bagging film projecting over the edges. When vacuum was applied, a pressure of about 14 psi was exerted by the surrounding atmosphere in the box on the bagging film and on the areas to be bonded. An inlet was provided to the box so that an external pressure could be applied in addition to the pressure exerted by the vacuum differential.

B. Carver Press — The method of stops was chosen for Carver Press assembly. Stainless steel (303) stops were used around the press edges to ensure a reasonably even glue line. The height of the stops was 0.608 ± 0.001 inches. The ideal thickness of the assembly before bonding was calculated from the following dimensions.

core:	0.500 ± 0.001 inches
skin:	0.0500± 0.0001 inches
skin:	0.0500± 0.0001 inches
impregnated cloth:	0.012
impregnated cloth:	0.012
Total	0.624 inches

Since the height of the stops was 0.608 inches, there was a certain amount of compression in the initial stages of the assembly which served to drive the core into the adhesive. Ideally, there should be little or no residual pressure applied during the cycle after the adhesive has flowed based on the following caluclation:

core:	0.500 ± 0.001 inches
skin:	0.0500±0.0001 inches
skin:	0.0500±0.0001 inches
bare cloth:	0.004 inches
bare cloth:	0.004 inches
Total	0.608 inches

C. Substrate Cleaning — All face sheets were treated according to Procedure II. Cores were treated in various ways. These are described in following sections.

D. Test Techniques

i. Beam-Flexure — Beam flexure tests were carried out using a one third span and single point loading technique. The specimen dimensions were 3.0" ± .01" x 8.0" ± .01" and the load was applied through knife edge supports. To avoid localized deformation of the face sheets, end support bars were placed between the knife edges and the specimen. A schematic view of the specimen is shown in Figure 2. In all specimens the 'L' direction of the core was parallel to the 8 in. dimension of the specimen. All tests were carried out at a cross head speed of 0.020"/min.

ii. Sandwich Peel — The sandwich peel tests were carried out in accordance with specification MIL-A-25463 (ASG). The peeling torque (T) was calculated as follows:

$$T = \frac{(F_p - F_o)\ (r_o - r_i)}{W}$$

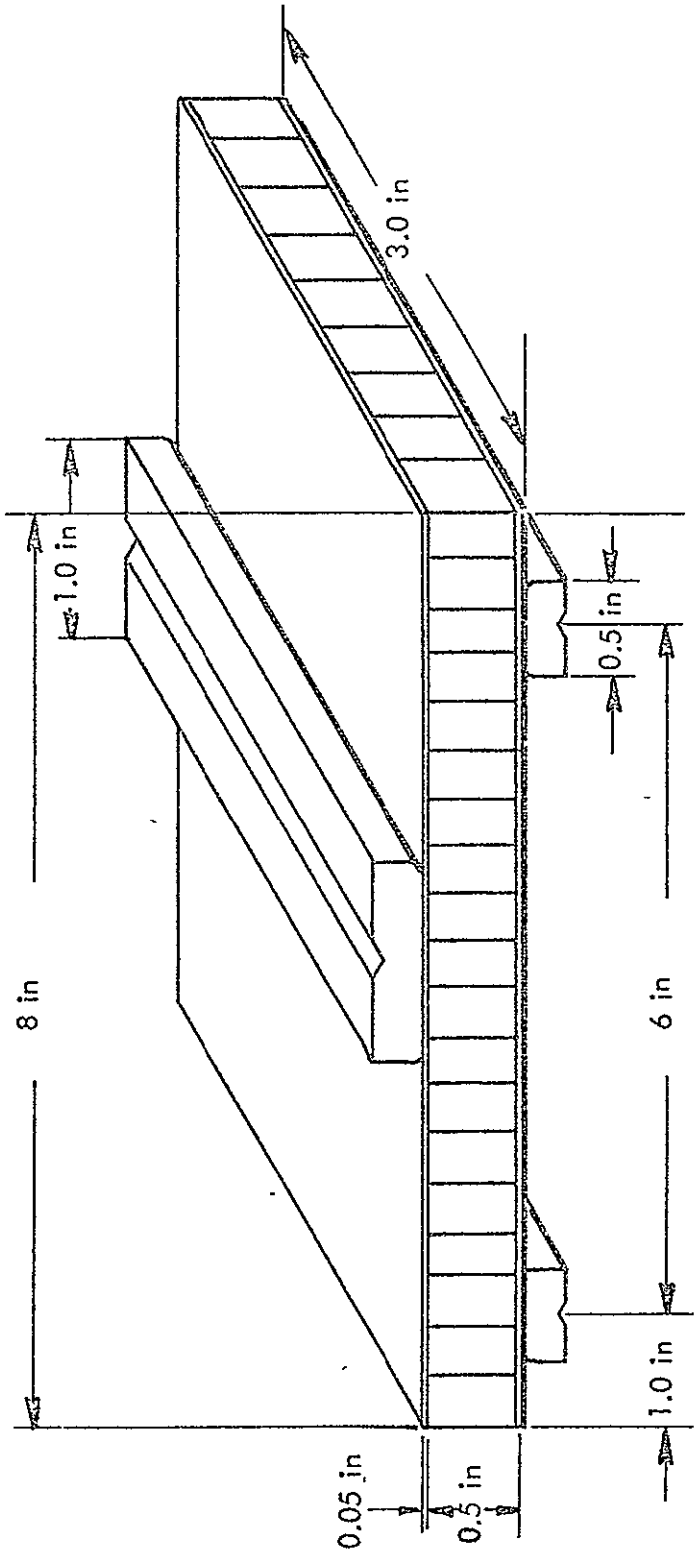


FIGURE 3
BEAM FLEXURE TEST APPARATUS

where

F_p = average peeling load for 5 inches of peeling between 1 and 6 inches

F_o = load to overcome resisting torque of drum and that required to bend the facing

$r_o - r_i$ = 0.5 inch for apparatus

W = specimen width

F_o was determined by inserting a specimen identical to the facing of the sandwich panel to be tested in place of the sandwich specimen and applying a load sufficient to roll the drum upwards around the sheet. The tests were carried out at a cross head speed of 1 inch per minute.

3.3.3 BEAM FLEXURE TESTS

A. Bis(epoxybutyl)carborane — The formulation used was 43 phr G-50 hardener, 50 phr aluminum, 29 phr asbestos.

i. Vacuum Bag Assembly — Selected test results are given in Table XIV. B-stage time for the adhesive prior to application to the glass cloth was 55 minutes. Highest strengths obtained were 1500 pounds at 75°F and 775 pounds at 500°F. Failure was in the adhesive at the thinner of the two bond lines. The thin glue line appears at the top of the specimen in the lay-up configuration and is a result of adhesive flow during cure. The effect of a four hour post-cure resulted in a slightly lower value as compared to a one hour post-cure. The low value on No. 14 suggests that a core rinse with MEK (methylethylketone) will not give satisfactory load strength.

TABLE XIV

BEAM FLEXURE TESTS

Bis(epoxybutyl)carborane, 43 phr G-50 Hardener, 50 phr Aluminum

Vacuum Bag Assembly

Specimen Number	Core Treatment	Post-Cure Time (hrs.) at 435-495°F	Ultimate Load (lbs.)	Temperature °F
7	MEK + Procedure II	one	1500	75
10	" " "	one	1425	75
12	" " "	four	1350	75
14	MEK only	four	700	75
8	MEK + Porcedure II	one	775	500

ii. Carver Press Assembly — Selected test results are given in Table XV. B-stage time for the adhesive prior to application to the glass cloth was 55 minutes except where noted. For several specimens, the core was compressed perpendicular to the L direction to increase the density (and therefore the bonding area) from 8.7 lbs/ft^3 to 14.1 lbs/ft^3. Two 8"X10" specimens (23 and 24) were assembled. In the first part of the assembly cycle for specimen No. 23, pressure was released and water vapor was evolved indicating that the core had not been adequately dried. Therefore, more precaution was taken in drying the core used for No. 24. During the assembly of No. 24, adhesive was pushed out of the glue line during the first part of the assembly cycle. This was probably due to pressure build-up of air entrapped in the cells. Pressure was momentarily released (after about 10 minutes) to allow escape of the air and then reapplied. No evolution of vapors was observed. No push-out of the adhesive was observed during the remainder of the process. In both cases, the visible fillets were good. There was some run-down of the adhesive. These panels were cut into 3"X8" specimens and flexure tested.

TABLE XV

BEAM FLEXURE TESTS

Bis(epoxybutyl)carborane, 43 phr G-50 Hardener, 50 phr Aluminum

Carver Press Assembly

Specimen Number	Core Treatment	Post-Cure Time (Hours) at 435-495°F	Core Density lbs./ft^3	Test Temperature °F	Ultimate Load (lbs.)
20*	Toluene-Acetone Water	One	8.7	75	1580
11	MEK + Procedure II	One	"	75	1450
13	" " "	Four	"	75	1330
23 (a)	Toluene-Acetone Water	One	14.1	75	1010
23 (c)	" "	"	"	75	1320
24 (a)	" "	"	"	75	1430
24 (c)	" "	"	"	75	1530
25	" "	"	"	75	1370
21*	" "	"	8.7	500	805
9	MEK + Procedure II	"	"	500	650
23 (b)	Toluene-Acetone Water	"	14.1	500	550
24 (b)	" "	"	"	500	450
26	" "	"	"	500	390
28	" "	"	"	500	455

* B-stage for the adhesive was 90 minutes

The highest value at 75°F (1580 lbs.) was obtained on specimen No. 20. In this case, there was some evidence of core failure. For all other samples tested at 75 and 500°F failure occurred in the adhesive at the thinner of the two glue lines. The highest value at 500°F (805 lbs.) was obtained on specimen No. 21. In both specimens, fillet size on the upper glue line was larger than previously observed. This improvement could be due to less flow of the adhesive since a longer B-stage time was used to prepare the adhesive for the bonding of these two specimens (90 minutes as compared to 55 minutes). The highest 75°F and 500°F values were obtained with core that had received successive treatments of toluene - acetone - water. As was found with the specimens assembled using vacuum bag techniques, a four hour post-cure (as compared to a one hour post-cure) did not have any significant effect on strength. The highest value at 75°F was obtained with a one hour post-cure. Compression of the core resulted in increased scatter but the highest value (1530 lbs. - Number 24C) approached the value obtained using core without compression (1580 lbs.).

B. Metlbond 329 Control — In order to evaluate the flexure test results obtained on the carborane system, several specimens were assembled with Metlbond 329. Samples of this adhesive were available in this laboratory. However, the properties of the material may have degraded because of long-term storage conditions. Therefore, these tests were conducted for general comparison purposes only and are not intended to represent the strengths that could be obtained from Metlbond 329 under ideal conditions.

Carver Press and vacuum bag techniques were used for the assembly of these specimens. All face sheets were treated as described in Procedure II. Cure assembly time was 335°F for 60 minutes (as recommended in NARMCO product bulletin dated 4/20/60). Results are listed in Table XVI. Fillets were small and symmetrical and there was no rundown of adhesive into the core. Failure occurred in the core at 75°F and in the adhesive at the skin-core interface at 500°F. Compression of the core resulted in an increase in the 75°F strength. However, values dropped at 500°F. This drop may be due to degradation of the adhesive during storage. Therefore, these values may not be very useful for comparison purposes.

TABLE XVI

BEAM FLEXURE TESTS

Metlbond 329 Control

Specimen Number	Core Treatment	Assembly Technique	Core Density lbs./ft^3	Test Temperature (°F)	Ultimate Load (lbs.)
16	Procedure II	Carver Press	8.7	75	1365
17	" II	Vacuum Bag	"	75	1430
18	Toluene + Acetone Water	Carver Press	"	75	1690
19	" "	" "	"	500	320
22	" "	" "	14.1	75	3350
29	" "	" "	"	75	2400
30	" "	" "	"	500	120
31	" "	" "	"	500	145

3.3.4 SANDWICH PEEL TESTS

As received core (density 8.7 lbs/ft^3) was used for all specimens. Face sheets were treated according to Procedure II. All core was treated with toluene - acetone - water.

A. Bis(epoxybutyl)carborane — The formulation used was 43 phr G-50 hardener - 50 phr aluminum - 29 phr asbestos. Three specimens tested at 75°F gave peel torque values of 6.5, 5.8 and 5.0 inch pounds/inch of specimen. Specimens tested at 500°F gave peel torque values of 3.1 and 3.0 inch pounds/inch of specimen. On all specimens, there was an excessive amount of run-down of the adhesive into the core.

B. Metlbond 329 — Two specimens tested at 75°F gave peel torque values of 10.1 and 13.5 inch pounds/inch of specimen. Two specimens tested at 500°F gave values of 7.1 and 6.5 inch pounds/inch of specimen.

3.4 DEVELOPMENT OF CARRIER CLOTH TECHNOLOGY

Several different procedures were investigated in order to determine a satisfactory means of impregnation of glass cloth. The most satisfactory technique is described in 3.4.5.

3.4.1 PROCEDURE ONE

A strip of glass cloth (heat cleaned Style 112) was placed on Tedlar film on a glass plate and the assembly placed in a warm oven at about 100°C. A portion of hot B-staged epoxy (with aluminum filler) was placed on the cloth and spread as evenly as possible with a spatula. An overlap of a second sheet of Tedlar film was placed on top of the epoxy, and pressure applied with a glass rod to press-out air bubbles. The assembly was removed from the oven and the laminate of Tedlar - impregnated glass cloth - Tedlar was trimmed. On cooling, the laminate wrinkled badly due to differences in thermal contraction characteristics of the glass cloth and the epoxy. The epoxy was very brittle and cracked off when the glass cloth was bent. The resin was poorly distributed and the bubbles had not been completely removed.

3.4.2 PROCEDURE TWO

In this work, portions of the B-staged mixture (with aluminum filler) were removed at various times during the B-stage and placed on glass cloth on the fixture described above. Six samples were removed at various time intervals and applied to the glass cloth in separate areas. These time periods were at 60 minutes (no aluminum), 75, 90, 105, 120 and 135 minutes. The fixture was at room temperature and no attempt was made to spread the epoxy at this time. The fixture was placed in an oven at about 100°C and a Tedlar overlay applied. After a 5-10 minute period to equilibrate the resin to oven temperature, the epoxy was spread by rolling with a glass rod. The fixture was removed from the oven and strips trimmed to about 1" X 3" size. The strips were better in appearance than those of No. 1 above but some wrinkles were still present. All the strips were brittle and easily damaged. The samples

at 60 and 75 minutes appeared to wet the glass cloth. At 90 minutes, the extent of wetting appeared to be marginal with even less wetting at longer times. This lack of apparent wetting might be due to the fact that not enough pressure was applied to press the resin into the glass cloth.

3.4.3 PROCEDURE THREE

The B-staged mixture with aluminum filler was quenched by pouring the mixture into an aluminum foil covered dish. The foil was peeled off the stiff B-staged material. The casting was then broken into several disc shaped pieces about 1/4" thick. Several attempts were made to impregnate 4 mil thick, heat cleaned Style 112 glass cloth with this material.

In the first attempt, 0.5 mil thick polyethylene was used as the backing. On an aluminum back-plate (1/16" thick) were placed layers of polyethylene, resin, glass cloth, resin and polyethylene. The back-plate with the lay-up was placed in a Carver Press maintained at 185°F (this is about the temperature limit since polyethylene melts at about 194°F). The lay-up was pressed for 30 seconds at 500 psi. (Brass shims, 12 mil thick, were used to fix the thickness of the lay-up). The lay-up was cooled outside the press and trimmed to give a four inch square of impregnated cloth. The specimen was about 11 mil thick (after subtracting the two thicknesses of polyethylene). It was soft and flexible and could be creased without cracking. This behavior is in sharp contrast to our previously reported work in which the impregnated cloth was brittle and easily damaged. This improvement is probably due to a shorter B-stage time, the use of a thinner backing and decreased overall lay-up thickness. The polyethylene backing could be easily peeled from the lay-up to yield a tack-free surface. The B-stage was very mobile at 302°F. When a sample of impregnated cloth was heated to this temperature, the adhesive ran considerably.

A second impregnation was carried out in a similar manner using 3 mil Tedlar and 30 mil shims for the press. Only one piece of epoxy casting was used with a press temperature of about 250°F for 30 seconds. Although the epoxy did penetrate the cloth, there appeared to

be more on one side than the other. The impregnated cloth was flexible but not as much so as that described above. It could be wrapped around a ¼ inch mandrel without cracking. However, use of a smaller mandrel did result in cracking.

In a third impregnation, the procedure described for the first was repeated except that casting was applied on only one side of the glass cloth. The epoxy did not completely penetrate the cloth.

3.4.4 PROCEDURE FOUR

In an attempt to improve flow characteristics, asbestos filler was added to the formulation. The procedure for B-staging the mixture was as follows:

- heat resin and hardener until clear, 15 minutes at 150 ± 2°C
- add aluminum, stir 5 minutes under vacuum
- add asbestos, stir with a cumulative time to 55 minutes with occasional vacuum
- quench cure by pouring mixture into aluminum foil
- cool to break into chunks

Experiments indicated that flow was reduced considerably when asbestos was added to the system. At the same time, an improvement in degree of tack was obtained. Tape stored at room temperature for periods up to four days experienced a noticeable decrease in degree of tack with the onset of crystallization in the film. Similar samples, when stored at - 10°F for 4 days, did not experience a loss in degree of tack nor was there any apparent crystallization.

3.4.5 PROCEDURE FIVE

The following procedure was developed for impregnation of glass cloth to be used for assembly of the last few series of lap-shear panels and for honeycomb panels. The B-staged

epoxy was poured onto Tedlar to obtain strips. Layers of Tedlar, resin, glass cloth, resin and Tedlar were placed on an aluminum back plate ($^1/16$" thick). The back-plate with the lay-up was placed in a Carver Press maintained at about 185°F. (Brass shims, 12 mil thick, were used to fix the thickness of the lay-up). The lay-up was cooled outside the press and trimmed to give of sample of about 8"X10".

3.5 SYNTHESIS OF INTERMEDIATES AND MONOMERS

3.5.1 PURIFICATION OF DECABORANE

Several vacuum sublimation systems were evaluated for the purification. In a typical purification, 68g of white decaborane was obtained from 80g of crude in an eight hour period (for an 85% recovery). The purification was not scaled-up any further because of potential safety problems. It was found that operation of several sublimators simultaneously would provide an adequate supply of high purity decaborane for the next reaction step. Eighty-one sublimations were conducted.

3.5.2 CARBORANE

The reaction was carried out in a one-liter, three-necked, round-bottomed flask equipped with extra coarse gas inlet tube, Allihn condenser, bubble counter, paddle stirrer, and thermometer. An acetylene-purification train was assembled consisting of three 500 ml flasks equipped with extra coarse gas inlet tubes followed by a 2"X30" column packed with an equal mixture of Drierite (indicating) and potassium hydroxide (pellets). Traps to catch back-up or overflow were inserted between the gas cylinder and acid, acid and KOH column and between the column and the reaction flask. A rotameter, inserted between the final trap and the reaction flask, permitted reproducible admission of the required volume of gas. The system was swept with prepurified nitrogen from charging of reactants to the time when acetylene was admitted.

Two hundred ml of n-propyl ether, freshly distilled from sodium benzophenone ketyl, was placed in the thoroughly dried reaction flask. The N_2 flow was started and 225g (1.82 moles) of sublimed decaborane was added to the flask. This was followed by the addition of 220 ml of diethyl sulfide which had been standing over molecular seives No. 4A for a minimum of 72 hours. The mixture was stirred for 3 hours at 40°C. The temperature was raised to 65-70°C for two hours and finally raised to 87°C and acetylene admitted. The flow rate was adjusted so that about 5 ounces of acetylene flowed into the mixture over a 42 hour period. At the end of this time the reaction mixture was cooled to 40°C before being transferred to a one liter, single neck, round bottom flask. The mixture was then placed in a flask on a rotary evaporator and the solvent mixture removed and collected in a dry ice trap. The product remaining in the flask was usually an orange solid at this stage. The solid was dissolved with 290 to 300 ml of absolute methanol, cooled to about 0°C and added dropwise over a 60 minute period to a stirred solution of 400 ml of methanol, 150 ml of acetone and 150 ml of conc. HCl. The reaction was swept with N_2 to avoid buildup of any mixtures of hydrogen and air. Stirring was carried out for 5 hours after which the mixture was allowed to stand for a minimum of twenty hours.

The reaction mixture was divided into three portions in three 500 ml dropping funnels. The contents were added dropwise to three four-liter volumes of tap water at 23°C. The crude product formed as loose curds on the surface of the water. These were suction filtered and redissolved in 500 ml of methanol. To this solution was added rapidly a cold solution of 50g of KOH in 75 ml of water. The mixture was stirred for precisely three minutes before being divided into three equal volumes and each poured into four liters of ice water. The resulting precipitate was suction filtered and vacuum dried for 16 hours. The dried material was blended with 40g of anhydrous $CaCl_2$ (12 mesh) and placed in a one liter Soxlet extraction thimble. The solid was extracted for 8 hours with 750 ml of heptane. After extraction the cooled solution was filtered and about 190g of crude carborane was obtained. The filtrate was evaporated to 50 ml and filtered again. Another 25g of product was thus

obtained. The solid was dried in a moving air stream in a fume hood for 16 hours. The product was then sublimed under vacuum at a temperature of 90-97°C to yield 184g of white crystalline material (70% conversion based on charged decaborane).

3.5.3 BIS(BUTENYL)CARBORANE

No difficulty was encountered in the scale-up of this preparation. The optimized procedure is as follows. A five liter, three neck, round-bottom flask was charged with 2770 ml of n-butyl lithium-hexane solution (1.6M, 4.42 moles of active agent). The flask was equipped with a mechanical stirrer, reflux condensor and a one liter dropping funnel. The flask was cooled with an ice water bath and stirring was initiated. A nitrogen flow was maintained over the mixture during the reaction. Carborane (225g. - 1.56 moles) was dissolved in 870 ml. of anhydrous ethyl ether. The solution was quickly transfered to the dropping funnel and added to the butyl lithium solution over a one hour period. The ice bath was removed and stirring was continued for 1½ to 2 hours. Stirring was discontinued and the solid allowed to settle. Approximately 75% of the supernatant liquid (containing unreacted butyl lithium solution) was removed with a siphon arrangement. Fresh ethyl ether (one liter) was added and the mixture was stirred for five minutes. After settling, about 75 percent of the supernatant liquid was again removed. Then 1220 ml. of fresh ethyl ether was added, stirring was initiated and the mixture heated to reflux. Bromo-butene (570g. - 4.22 moles) was added to the refluxing solution over a one hour period. Reflux was continued overnight for 16½ - 17½ hours.

The mixture was cooled to room temperature and allowed to settle. The supernatant liquid was divided into equal portions and added to two separatory funnels containing 500 ml. of distilled water each. The solids in the flask were poured slowly into a four liter beaker containing 500 ml. of distilled water. (Considerable heat is evolved in this step and caution must be exercised. The solution should be stirred with a glass rod during this step). The solution was then divided and added to the two separatory funnels. The funnels were then

shaken (with venting) and the layers allowed to separate. The water layer was removed and discarded. Each ether layer was added to a three liter beaker containing 90g. of anhydrous magnesium sulfate. The mixture was stirred occasionally for 15 minutes and filtered through a common Büchner filter. The solids on the filter were rinsed with 250 ml. of fresh ethyl ether. The filtrate was then gravity filtered and the clear solution rincoedat 70-80°C until all solvent was removed. A VPC analysis was then obtained. Normally, purity was greater than 95 percent; impurities were carborane and monobutenyl carborane.

The crude product was then initially distilled using a 30 inch Vigreaux column. Unreacted carborane and monobutenylcarborane were distilled from the main portion of the product. The pot residue was finally distilled in a short path system at a pot temperature of 190-198°C and at a vacuum of 0.02mm. In a typical reaction 316g. of product with a purity of 99% (as indicated by VPC analysis) was obtained. This quantity represents a conversion of 81% of starting material to desired product.

3.5.4 BIS(EPOXYBUTYL)CARBORANE

Reactions which were performed are summarized below. The optimized procedure is given at the end of this section.

A. SUMMARY OF REACTIONS

- Reaction # 1 — Bis(butenyl)carborane (24.5g) was treated with 16.3 ml of H_2O_2 and 84.7 ml of trifluoroacetic anhydride. The crude product (27.8g) was 96.3% pure. On distillation, 16.2g of product with a purity of 96.1% was obtained. The quantity obtained represents a conversion of 58.5% of bis(butenyl)carborane to desired epoxy.

- Reaction # 2-4 — Three reactions were carried out using 24.5g of bis(butenyl)-carborane, 16.3 ml of 90% H_2O_2 and 84.7 ml of trifluoroacetic anhydride. Gas

chromatographic analyses of the crude products showed a purity of 79.7%, 91.5% and 90.1% of the desired product. The three crude products were combined (85.6g) and treated with an additional quantity of trifluoroacetic acid (prepared from 5.5 ml of 90% H_2O_2 and 28.2 ml of trifluoroacetic anhydride). Gas chromatographic analysis of the crude product, 82.0g, showed that the purity had been increased to 94.8%. The product was distilled and 67.3g of bis(epoxybutyl)carborane with a purity of 98.33% was obtained. This quantity represents an overall conversion (for the three reactions) of 75% of bis(butenyl)carborane to desired epoxy.

• Reaction # 5-8 — Four reactions were carried out using the quantities of reagents listed above. Gas chromatographic analyses of the crude products showed a purity of 95.8%, 94.8, 94.7 and 95.4% of desired product. The four crude products were combined (115.8g) and treated with an additional quantity of trifluoroacetic anhydride. Gas chromatographic analysis of the crude product showed that the purity had been increased to 96.4%. On vacuum distillation, 90 g of product with a purity of 98.5% was obtained. This quantity represents an 82% conversion of starting material to desired product.

• Reaction # 9-10 — The charge of bis(butenyl)carborane was doubled to 49.0g and amounts of reagents were correspondingly increased. Analyses of the crude products were as follows.

	Weight Crude	Purity
Reaction # 9	54.9 g	93.9%
Reaction # 10	57.1 g	96.0%

The re-reaction step was omitted on this work. The two crude products were combined and distilled. On vacuum distillation, 90 g of product with a purity of 98.3% was obtained. This quantity represents a 79% conversion of starting material to desired product.

• Reactions # 11-19 — Product from these reactions was severely contaminated with a component which was identified as the trifluoroacetate adduct derived from the epoxy ring-opening reaction. This contamination problem is discussed more fully in an earlier section.

• Reaction # 20 — A powder grade of sodium carbonate was used for this reaction. Quantities of reactants were:

2200 ml CH_2Cl_2
65.2 ml H_2O_2
300 ml trifluoroacetic anhydride
715g. sodium carbonate
98g. bis(butenyl)carborane

A VPC analysis after a one hour reflux period showed a purity of 94.6%. No unreacted butenylcarborane was detected by infrared analysis. After 2½ hours of reflux, the reaction was worked up to give 109.7g. of crude product (purity of 96.5%). The product was distilled to give 81g. of product with a purity of 99%. The infrared spectrum was nearly identical to the previously recorded spectrum of this compound. A peak at 1780cm^{-1} was present but quite weak (as compared to spectra of impure materials obtained in reactions 11-19).

• Reaction # 21 — Quantities of reagents were changed to more nearly reflect the stoichiometric requirements of the reaction.

2200 ml CH_2Cl_2
32.6 ml (1.20 moles) H_2O_2
169 ml (1.20 moles)trifluoroacetic anhydride
318 g. (3.00 moles) sodium carbonate
100 g. (0.400 moles) bis(butenyl)carborane

At the end of the two-hour period at room temperature of Phase 2, a test with starch-iodide paper was positive indicating that an active peroxy compound was still present. After one hour of reflux, a starch iodide test was negative. An infrared analysis showed the absence of butenyl groups and a gas chromatographic analysis showed a purity of 97.1%. The mixture was worked up after 2½ hours of reflux and 113.2g. of product (purity of 97.1%) was obtained. On distillation 96g. of product with a purity of 99.1% was obtained.

In the infrared spectrum weak bands were apparent at 1790cm^{-1} and at 1170cm^{-1} indicating the possible presence of a small amount of impurity. However, elemental analysis and a molecular weight determination were in excellent agreement with calculated values.

• Reaction # 22 —The quantities of reagents were scaled-up and the amounts used were 69.3ml of hydrogen peroxide, 400 ml of trifluoroacetic anhydride, 760g. of sodium carbonate and 225g. of bis(butenyl)carborane. Trifluoroperacetic acid intermediate was added to the butenyl carborane over a 3 3/4 hour period. After 1 3/4 hours reflux, a negative test was obtained with starch iodide paper indicating that the peracid was completely destroyed. On distillation, 224g. of product was obtained. However, the infrared spectrum of the product contained a peak at 1780cm^{-1} indicating that the hydroxytrifluoroacetate adduct was present. The powder sodium carbonate used was of a larger particle size than used in reactions 20 and 21.

• Reaction # 23 — In this reaction, the quantities of reagents used were the same as in Reaction 21; 32.6ml (1.20 moles) of H_2O_2, 169 ml (1.20 moles) of trifluoroacetic anhydride, 318g. (3.00 moles) of sodium carbonate, and 100g. (0.400 moles) of bis(butenyl)carborane. The addition, reflux time was approximately the same. An infrared spectrum of the crude product (119.1g.) contained a strong band at 1780cm^{-1}.

On distillation, 102g. of product was obtained, and a medium intensity peak was present at 1780cm^{-1} in the infrared spectrum. Since the source of sodium carbonate was the same as that used in Reaction 22, this result led us to believe that a finer grade of powder was required.

• Reaction # 24 — Again the same amounts of reagents were used as in reaction 21. However, the sodium carbonate was ball-milled, sieved through a No. 325 screen and vacuum dried at 50°C. The trifluoroperacetic acid was added over a 2½ hour period at ice-water temperature. The ice was removed and the mixture allowed to warm to room temperature over a 1½ hour period. A test with starch iodide paper was positive indicating the presence of peroxy compound. A 20ml sample was removed and worked up in the usual manner (filter and Rinco). The infrared spectrum of the solid contained a strong band at 1780cm^{-1}. A gas chromatographic analysis showed that conversion of the alkenyl to epoxy was nearly complete (92.7%). After one hour of reflux, the starch-iodide test was negative. A sample was again worked up in the usual manner. The infrared spectrum contained a peak at 1780cm^{-1} but of less intensity than before reflux. A gas chromatographic analysis showed that reaction was 96.6% complete. A similar infrared analysis after two hours of reflux showed an additional decrease in the intensity of the peak at 1780cm^{-1}. After 17 hours of reflux, only a weak peak was present at 1780cm^{-1}. The product was isolated and distilled. Distillation yielded 91.1g. of product with an infrared spectrum that contained only a weak peak at 1780cm^{-1}. A gas chromotographic analysis showed a purity of 97.2%.

• Reaction # 25 — Conditions and quantities for reaction 24 were repeated with the exception that as-received powdery sodium carbonate (dried at 50°C overnight) was used and the addition period was extended. The object of this work was to determine if a 325 sieve fraction is required (only about 40% of the as-received

powder sodium carbonate will pass through this size screen). Universal indicator paper was used to monitor the acidity of the reaction. Trifluoroacetic anhydride gave a pH of 3. The pH of the trifluoroperacetic acid was 0 (the limit of the indicator). The pH was monitored at hour intervals over a six hour addition period. During this period, the pH ranged between 4 and 5. After 15 hours of reflux, the pH of the mixture was 5. The reaction was worked up, and the crude product was distilled. The infrared spectrum of the product (89g.) contained a weak absorption at 1780cm^{-1} but the absorption appeared to be somewhat more intense than the band in the product from reaction No. 24. This comparison indicates that sodium carbonate sieved through a No. 325 screen is probably required.

• Reaction # 26 — The procedure and quantities for reaction No. 24 were repeated. The sodium carbonate was ball-milled, sieved and dried. The pH was monitored over the 2½ hour addition period and ranged between 4 and 5. Samples were removed before reflux was initiated and at various intervals during the 22 hour reflux period. Infrared spectra of the filtered liquid were recorded in a 0.1mm pathlength cell.

The intensity of the 1780cm^{-1} band falls quite rapidly during the first part of the reflux period and appears to be constant after 21 hours. The mixture was worked up, and the crude product distilled to give 92.2g. of product. No peak at 1780cm^{-1} was detected in the infrared spectrum. A gas chromatographic analysis indicated that the product was greater than 99 percent pure.

In summary, a finely divided grade of powdery sodium carbonate appears to be a requisite for obtaining high purity product. This is probably because the reaction of trifluoroacetic acid with sodium carbonate is heterogeneous. An extended reflux period is required to remove the $R_fC(=O)-$ species from solution. The species could be the free acid $R_fC(=O)OH$ or an adduct of the olefin which is gradually converted to

epoxy during the reaction. Further investigations are required to determine the mechanism of epoxy formation. The product obtained from reactions 24-26 appears to be of high enough purity for adhesive bond evaluation.

• Reaction # 27 — In reaction 27 and 28, the sodium carbonate was sieved through at 325 screen and dried at 50°C overnight. The charge of bis(butenyl)-carborane was increased to 150g. The quantities of reagents were 48.9 ml of H_2O_2, 253.5 ml. of trifluoroacetic anhydride and 477g. of sodium carbonate. Samples were removed before reflux was initiated and at various time intervals during the 39½ hour reflux period. Infrared spectra of the filtered liquid were recorded in a 0.1mm pathlength cell. The intensity of the band at 1780cm^{-1} was nearly constant after 19 hours. Only 114g. of distilled product was obtained, representing a 67% conversion of starting material to product. This conversion is substantially lower than that obtained in Reaction 26 where the conversion was 82% on a 100g. charge.

• Reaction # 28 — A 39 hour reflux period was used. Conversion again was low, 117g. for 69 percent.

• Reaction # 29 — A new supply of sodium carbonate which was guaranteed to pass through a 325 screen (98%) was used for this and the remainder of the work. For this reaction the sodium carbonate was vacuum dried for several hours. Conversion again was low, 65% for 110g. of product.

• Reactions # 30 and 31 — The charge of these reactions was reduced to 100g. and the reaction conditions were similar to those used for reaction 26. Sodium carbonate was dried for reaction 30 but not for reaction 31. Since the conversions were about the same (74 and 76 percent representing 84 and 86g of product), the sodium carbonate was not dried for the remainder of the reactions.

• Reaction # 32 — This reaction was undertaken using a 150g. charge of bis(butenyl)carborane. The reflux period was reduced to 22 hours since the stepwise analysis conducted in reaction 27 indicated that this shortened time period would be satisfactory. The conversion in this reaction increased to 84% (142g.). A reaction may take place between the epoxy and the solids in the reaction flask to decrease the conversion. This would result in an increased amount of residue on the distillation step. Further studies would have to be undertaken to prove this.

• Reactions # 33 - 36, 38 —These reactions were run with a 150g. charge and conversions ranged from 88 to 91 percent.

• Reactions # 37, 39 - 46 —The charge of bis(butenyl)carborane was increased to 225g. and the amount of reagents were increased proportionately. (74.2 ml of H_2O_2, 373 ml. of trifluoroacetic anhydride and 750g. of sodium carbonate). A 22 hour reflux period gave a 96% conversion (243g.). The high conversion is probably due to a nearly optimized reaction procedure and decreased physical loss of material on the distillation step. The detailed procedure is given in the following section.

Optimized Procedure

i. Phase I — A two liter, 3 neck, round bottom flask containing a magnetic stirring bar was charged with 800 ml of methylene chloride and chilled with an ice-water bath. Hydrogen peroxide (74.2 ml of 90%, 2.70 moles) was added carefully to the flask and the mixture was stirred for five minutes. Then 373 ml (2.70 moles) of trifluoroacetic anhydride was placed in a dropping funnel and added dropwise over a 45 minute period. Stirring was continued for an additional seven minutes. A nitrogen flow was maintained over the mixture during this period. Hydrogen peroxide is insoluble in methylene chloride. At the end of Phase I, the solution is homogeneous indicating the complete formation of trifluoroperacetic acid.

ii. Phase II — A five liter, 3 neck, round botto[illegible] flask, was equipped with a condenser, mechanical stirrer and a solid addition [illegible]nnel. The flask was charged with 715 g. (6.75 moles) of Baker grade sodium c[illegible]bonate (98% guaranteed by the vendor to pass through a No. 325 mesh screen), 22[illegible] (0.900 moles) of bis(butenyl)-carborane dissolved in 1000 ml of methylene chlo[illegible], and an additional 1200 ml. of methylene chloride. The solid addition funnel [illegible]as replaced with a one liter dropping funnel. The mixture was stirred for five [illegible]moles at room temperature, cooled with an ice water bath and the nitrogen flo[illegible] initiated. The solution from Phase I was placed in the dropping funnel and add[illegible] over a four hour period. The ice water bath was removed, and stirring continue[illegible]r two hours without application of external heat. Reflux was then initiated with a[illegible]m oil bath. During the first 30 minutes, reflux was quite vigorous but gradually[illegible]subsided. Reflux was continued for an additional 19 to 22 hours. The contents we[illegible]hecked with starch-iodide paper and Ph paper. The flask was cooled to roo[illegible]mperature with the aid of an ice water bath, and the mixture was Büchner filte[illegible] using a No. six funnel and No. 1 filter paper. The solids were washed with a[illegible] 800 ml of fresh methylene chloride to remove any retained product. The sli[illegible]y cloudy solution was then gravity filtered through No. 588 paper. The solut[illegible] was Rincoed at about 60-70°C until solvent was completely removed. Infrared a[illegible] V.P.C. analyses were recorded.

The crude product from above was fractionally dis[illegible]lled under vacuum (.02-05mm). The heating tape on the head was held at a varia[illegible]tting of 59 volts. A first fraction (about 5 grams) was usually removed at 170-180°C[illegible]gh temperature. The temperature was increased and the main fraction was removed a[illegible] temperature of 190-228°C. Infrared and V.P.C. analyses were recorded on this[illegible]raction. In a typical reaction 243g. of pure product, representing a conversion o[illegible]% starting material to desired product is obtained. V.P.C. analysis indicated an[illegible]purity level of <2% of alkenyl derivatives.

3.5.5 HYDROXYTRIFLUOPOACETATE OF BIS(EPOXYBUTYL)CARBORANE

A reaction was undertaken to prepare and characterize the trifluoroacetate adduct by eliminating sodium carbonate from the reaction procedure. Trifluoroacetic anhydride (16.9 ml, 0.120 moles) was added with stirring over a 15 minute period to a solution of 80 ml of methylene chloride and 3.2 ml (0.12 moles) of hydrogen peroxide maintained at ice water temperature. The resulting solution was stirred for an additional seven minutes and then added over a 30 minute period to a solution of 10 g (0.040 moles) of bis(butenyl)carborane dissolved in 140 ml. of methylene chloride. The resulting solution was stirred for 30 minutes at room temperature and then refluxed for one hour. After this period, a test with starch iodide paper was positive, indicating the presence of peroxy compounds. Sodium carbonate (31.8g.) was then added and the mixture refluxed for an additional hour. A test with starch iodide paper was negative. The mixture was filtered to remove insoluble salts, and methylene chloride was removed from the filtrate by a Rinco evaporation. An infrared spectrum of the semi-liquid product (12.2g.) as a smear between KBr plates contained a strong band at 1780cm^{-1} characteristic of the carbonyl stretch of $R_f\overset{O}{\overset{\|}{C}}-$. There were no sharp bands to indicate the presence of epoxy groups. The material was only slightly soluble in CCl_4 but the spectrum that was obtained contained a sharp band at 3620cm^{-1} and a broad band in the 3400 - 3600cm^{-1} region. A gas chromatographic analysis showed the absence of bis(epoxybutyl)carborane. Several materials were detected with retention times shorter than that of bis(epoxybutyl)carborane.

3.6 DIFFERENTIAL THERMAL ANALYSIS

Differential thermal analysis was used to study the curing characteristics and to obtain information on the thermooxidative stability of the bis(epoxybutyl)carborane, 43 phr G-50 hardener, 50 phr aluminum system.

3.6.1 CURING CHARACTERISTICS

The composition studied was that used for the assembly of Series 44-77 and contained 29 phr asbestos. Samples were taken at various stages of the processing procedure. A nitrogen atmosphere was used for all these analyses. Results are as follows.

- Mechanical Mixture of Components — The thermogram is shown in Figure 4. The endotherm at 75°C corresponds to the melting point of the epoxy monomer. The exotherm that peaks at 230°C and the shoulder at 280°C are assumed to be due to the curing reaction. The exotherm at 405°C is apparently due to thermal decomposition and appears in the other three thermograms described below.

- Sample After B-Stage — The thermogram is shown in Figure 5. The endotherm at 75°C corresponding to the melting point has decreased. The exotherm at 230°C corresponding to curing is still present. The exotherm (shoulder) at 280°C has been shifted slightly to 295°C.

- Sample After Cure-Assembly — The thermogram is shown in Figure 6. The endotherm at 75°C has now disappeared. The curing exotherm at 230°C has decreased and the exotherm at 295°C has now become more apparent.

- Sample After Post-Cure — The thermogram is shown in Figure 7. Both curing exotherms at 230 and 295°C have now disappeared and have been replaced by a slight, but reproducible endotherm at 275°C.

These results show that this cure-assembly post-cure cycle results in complete cure, at least within the limits of detection by DTA.

3.6.2 THERMOOXIDATIVE DEGRADATION

No attempt was made to thoroughly evaluate the thermooxidative stability of the adhesive system. In order to obtain preliminary information, DTA thermograms were compared in air and nitrogen of the formulation used for Series 44-77. This formulation did not contain asbestos filler. Results are listed below.

- Nitrogen — This thermogram is shown in Figure 8. It is similar to that obtained for the asbestos filled sytem (Figure 7, Series 44-82). The exotherm for decomposition starts about 325°C and peaks at 410°C. A slight endotherm is apparent at 275°C.

- Air — This thermogram is shown in Figure 9. In air, the exotherm starts a little sooner than in nitrogen (275°C versus 325°C) and peaks at about the same temperature - 410°C. The shape of the curve is generally the same. However, the portion of the exotherm beyond 410°C is difficult to reproduce. A second exotherm has been observed at about 425°C in some DTA scans. The appearance of this exotherm is dependent on sample size and air flow rate. It may represent physical movement of the sample. A slight endotherm at 275°C is apparent.

In order to obtain more definitive data on the thermal stability of this system, TGA and IGA data should be obtained. The appearance of a slight endotherm at 275°C in all the cured systems may be significant. This endotherm may represent a transition temperature of some kind and may account for the sharp fall-off in lap-shear strength above 500°F. Additional studies would have to be undertaken to determine if this is the case.

SAMPLE: Series 44-82
Mechanical Mixture
Bis(epoxybutyl)carborane
43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

SIZE 3.674 mg.
REF.
PROGRAM MODE
RATE 10 °C/MIN, START 23 °C

ATM. N₂ Flow

	T	ΔT
SCALE SETTING	50 °C/IN	0.1 °C/IN

RUN NO. 70-355
DATE 7/1/70
OPERATOR W. Benko

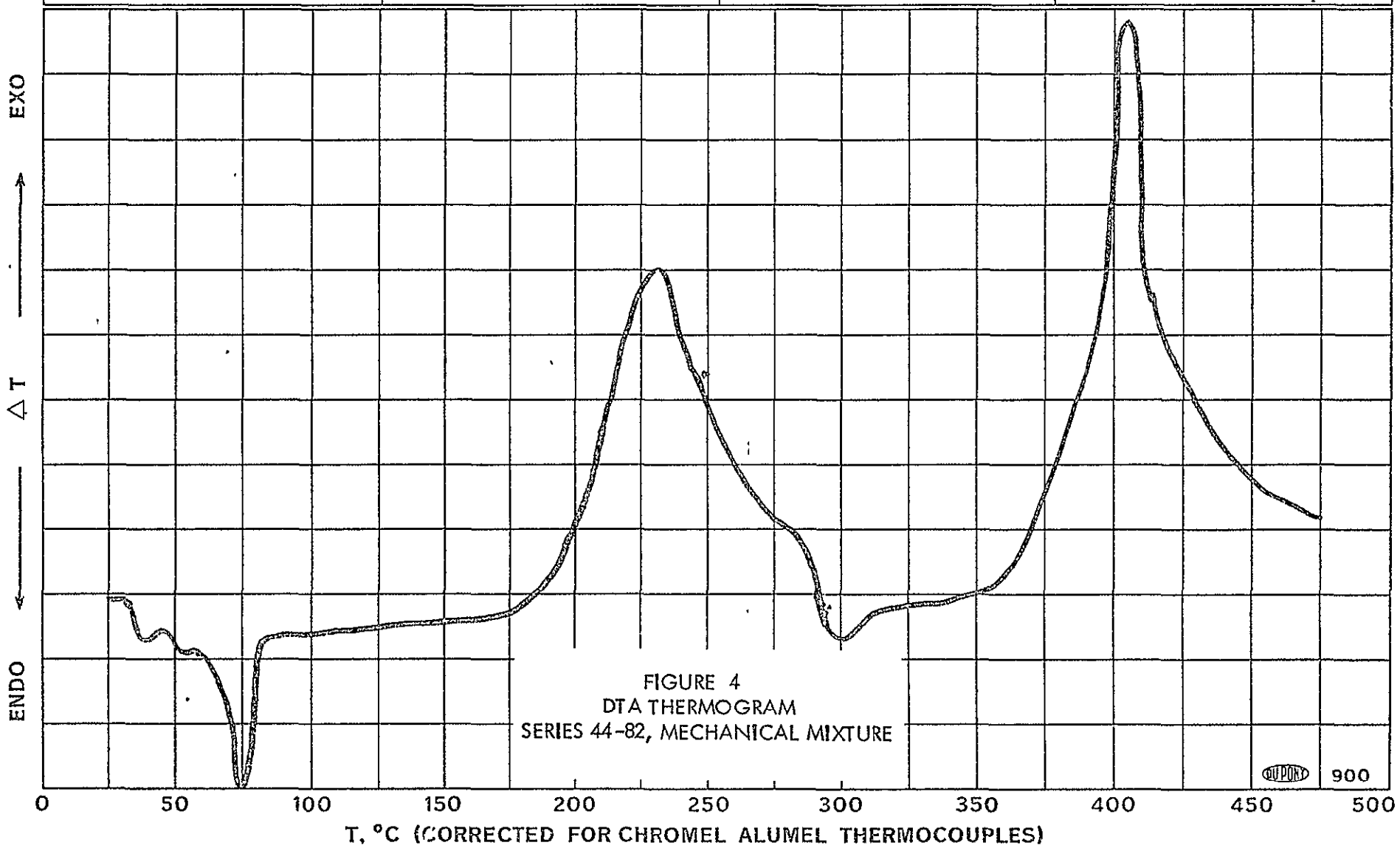


FIGURE 4
DTA THERMOGRAM
SERIES 44-82, MECHANICAL MIXTURE

SAMPLE: Series 44-82 B-Staged 55 Minutes (293-302°F) Bis(epoxybutyl)carborane 43 phr G-50 Hardener 50 phr Aluminum 29 phr Asbestos	SIZE 3.227 mg. REF. PROGRAM MODE RATE 10 °C/MIN, START 23 °C	ATM. N_2 Flow T: 50 °C/IN; ΔT: 0.1 °C/IN SCALE SETTING	RUN NO. 70-356 DATE 7/1/70 OPERATOR W. Benko

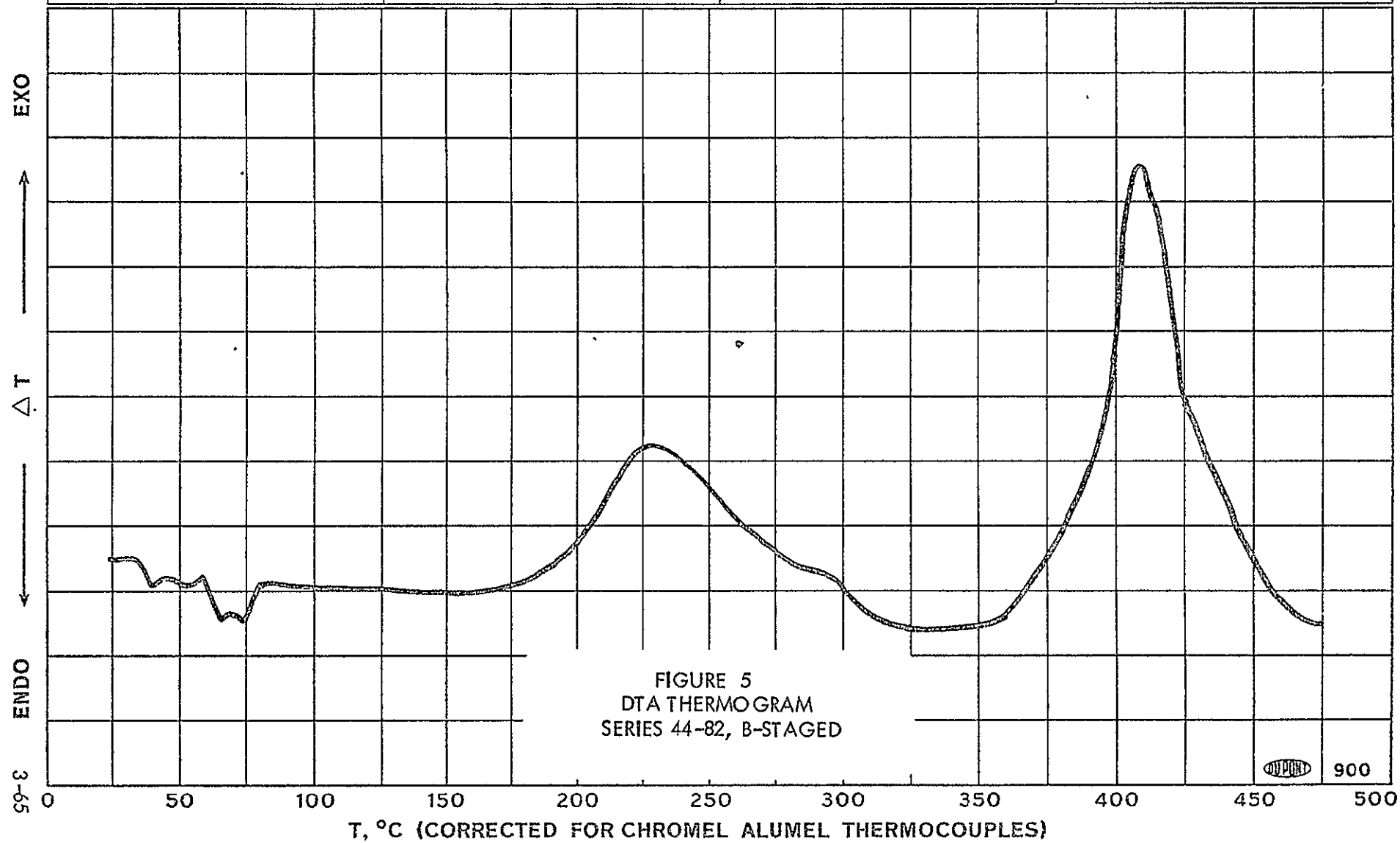


FIGURE 5
DTA THERMOGRAM
SERIES 44-82, B-STAGED

SAMPLE: Series 44-82
After Cure-Assembly Cycle
Bis(epoxybutyl)carborane
43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

SIZE 3.300 mg.

REF.

PROGRAM MODE

RATE 10 °C/MIN, START 23 °C

ATM. N_2 Flow

	T	ΔT
SCALE SETTING	50 °C/IN	0.1 °C/IN

RUN NO. 70-357

DATE 7/1/70

OPERATOR W. Benko

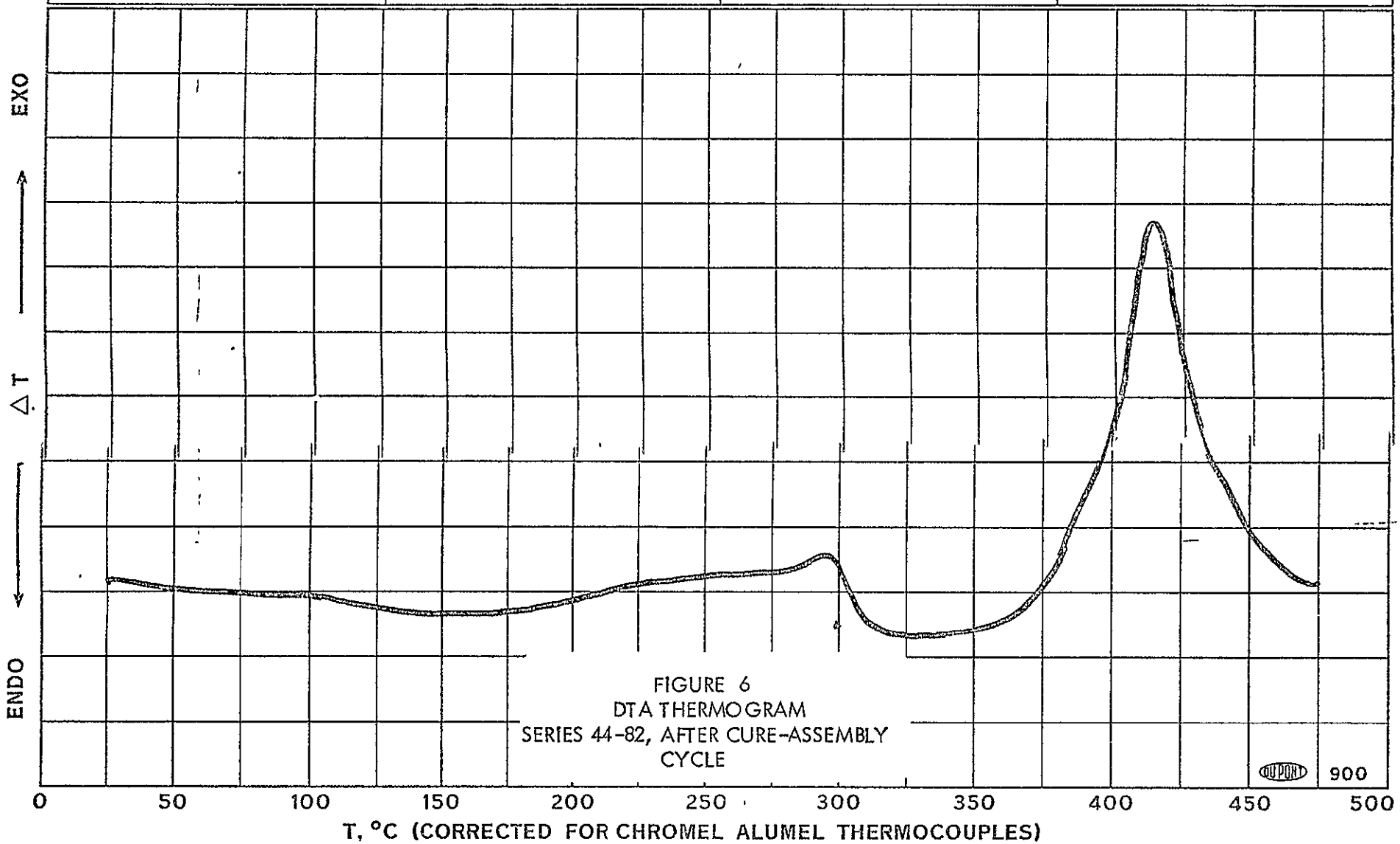


FIGURE 6
DTA THERMOGRAM
SERIES 44-82, AFTER CURE-ASSEMBLY CYCLE

50 phr Aluminum
29 phr Asbestos

PROGRAM MODE

RATE 10 °C/MIN, START 23 °C

SCALE SETTING 50 °C | 0.1 IN

RUN NO. TC-355

DATE

OPERATOR W. Benko

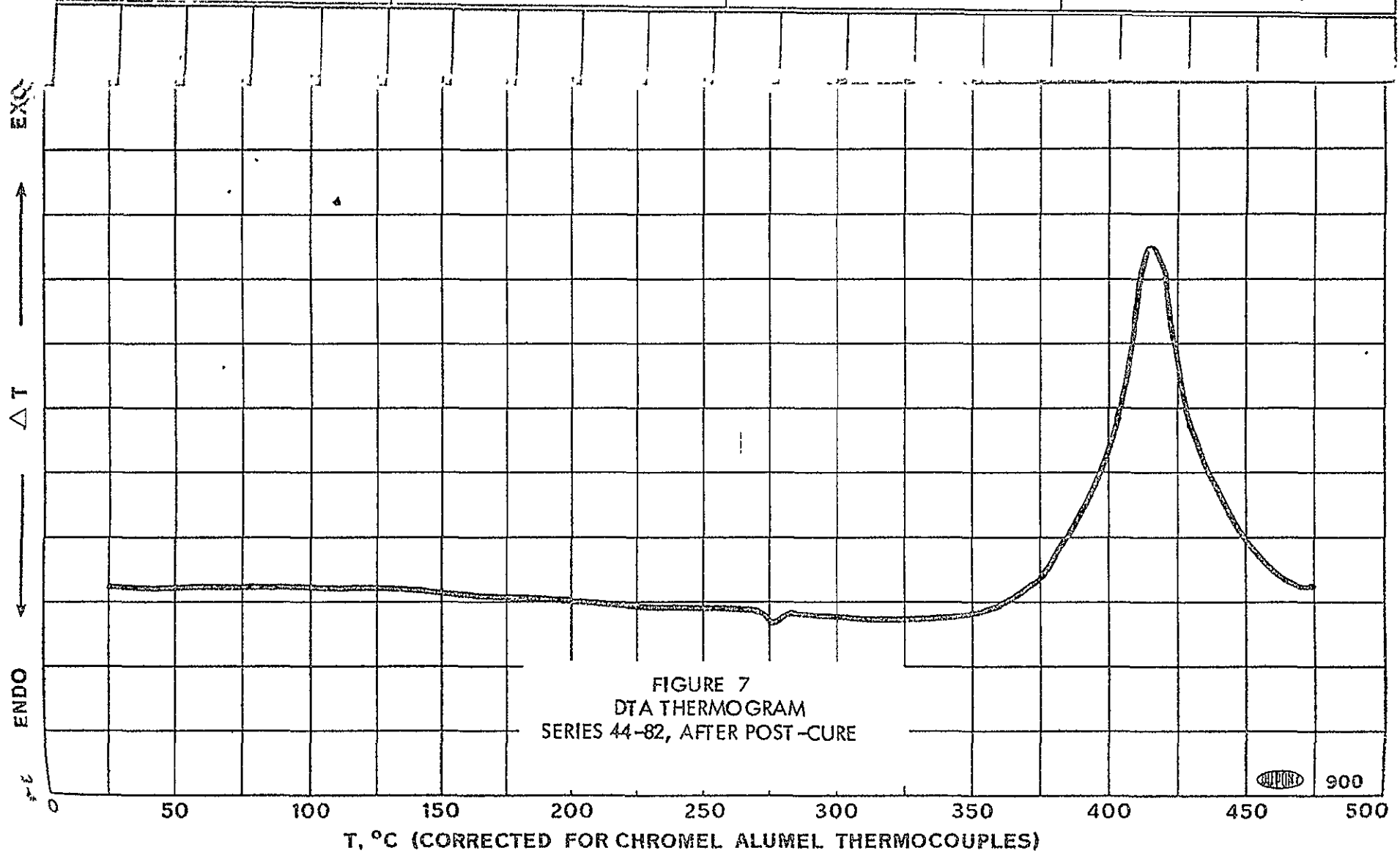


FIGURE 7
DTA THERMOGRAM
SERIES 44-82, AFTER POST-CURE

SAMPLE: Series 44-77
Bis(epoxycyclohexyl) adipate
43 phr [illegible]
50 phr Aluminum
Completely cured

REF.

PROGRAM MODE

RATE 10 °C/MIN, START 23 °C

ATM. N_2 Flow

	T	ΔT
SCALE SETTING	50 °C/IN	0.1 °C/IN

RUN NO. 70-411

DATE 11/13/70

OPERATOR W. Benko

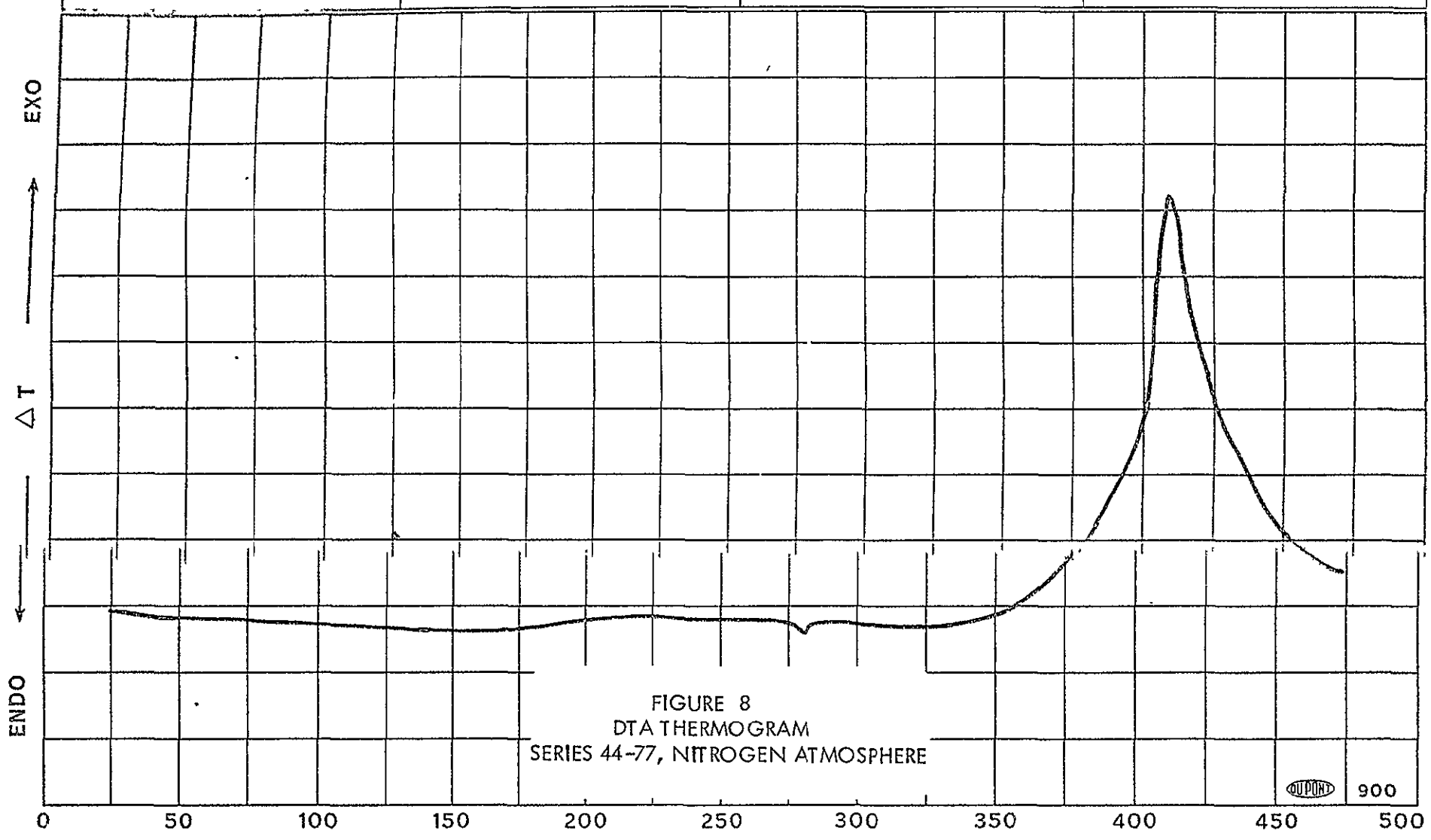


FIGURE 8
DTA THERMOGRAM
SERIES 44-77, NITROGEN ATMOSPHERE

SAMPLE: Series 44-77 Bis(epoxybutyl)carborane 43 phr G-50 Hardener 50 phr Aluminum Completely cured	SIZE 2.117 mg. REF. PROGRAM MODE RATE 10 °C/MIN, START 23 °C	ATM. Air Flow T: 50 °C/IN; ΔT: 0.1 °C/IN SCALE SETTING	RUN NO. 70-412 DATE 11/13/70 OPERATOR W. Benko

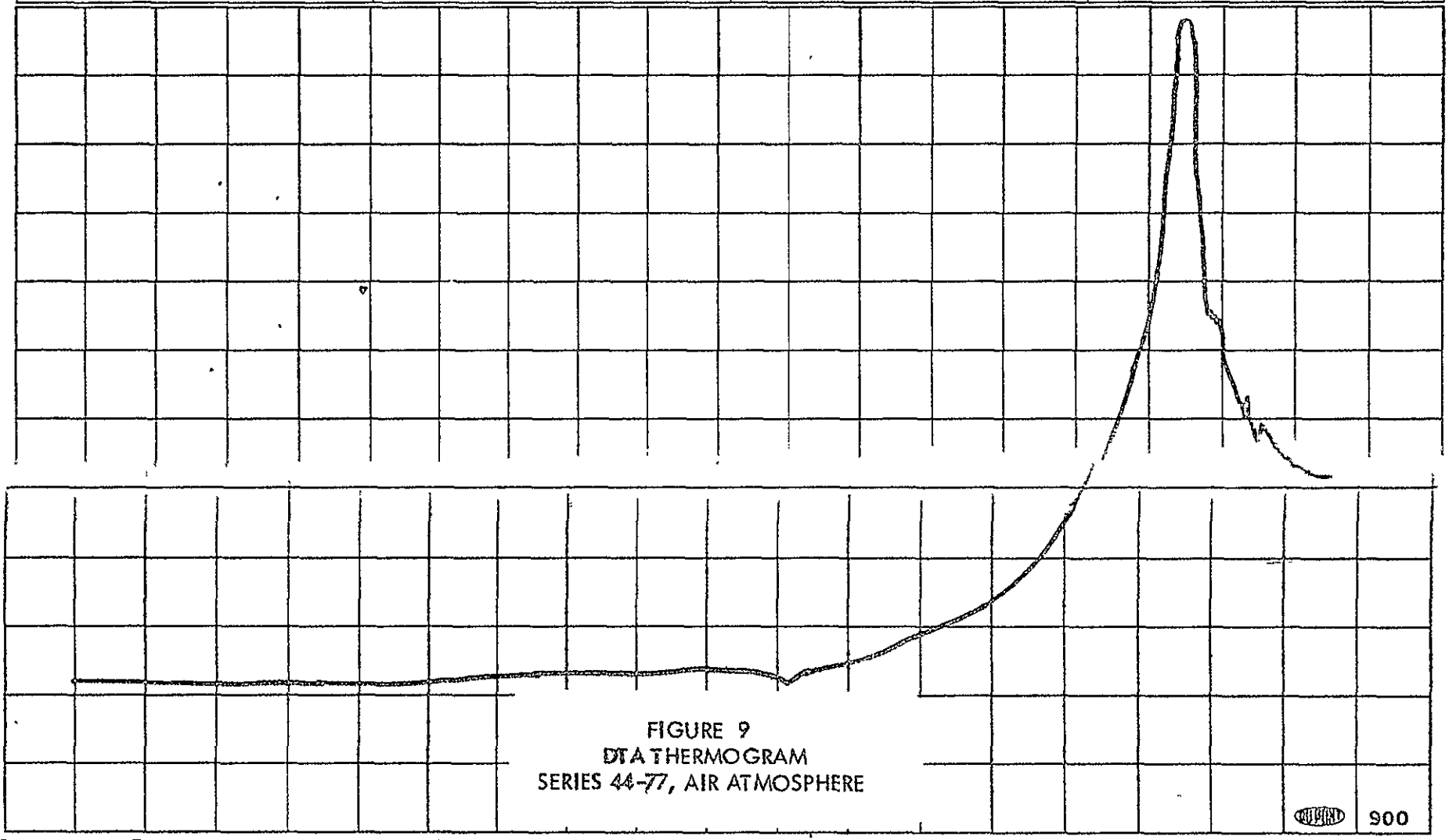


FIGURE 9
DTA THERMOGRAM
SERIES 44-77, AIR ATMOSPHERE

4. RECOMMENDATIONS

This program has resulted in the development of a [illegible] high temperature stable adhesive system based on the monomer, bis(epoxybutyl) [illegible] adduct. This monomer can be cured with conventional catalytic curing agents and [illegible] hardeners without the evolution of volatile products. Adhesive bonded joints can be assembled under conditions of temperature, pressure and time which are applicable to [illegible] additional epoxy processing technology. The B-staged adhesive can be applied to substrates by hot-melt or carrier tape techniques. Excellent lap-shear strengths on stainless steel [illegible] have been obtained in air atmospheres over the temperature range -320 to [illegible]°F. It was not necessary to add anti-oxidants in order to achieve these results. The [illegible] sandwich bonding capability (stainless steel core and face sheets) has been demonstrated.

Although the results obtained to date are very encouraging, certain other studies must be conducted in order to achieve design data.

1. The reason for the fall-off in lap shear strength above 500°F must be determined. Results obtained thus far indicate that a [illegible] temperature may have been exceeded. If this is the case, then a change in formulation (for example, a different hardener) may be required to improve bond strength above 500°F.

2. Lap-shear strength at 500°F decreases [illegible] aging at 500°F in air on steel substrates. This may be due to the [illegible] degradation of the adhesive catalyzed by the substrate, a problem encountered with other epoxy-based adhesives. This effect should be investigated more closely. [illegible] of anti-oxidants may improve the resistance of the adhesive bond [illegible].

3. A major limiting factor to the [illegible] of honeycomb properties is the brittleness of the adhesive. This could [illegible] by the incorporation of a

flexibilizing unit into the epoxyalkylcarborane monomer structure or by a blend with a high-temperature - stable polymer system.

4. Some improvement in the flow control of the adhesive during the cure-assembly of test specimens is required. This may be accomplished by replacing the asbestos with Cabosil or Alon-C.

Others have proposed that the carborane cage structure exerts a stabilizing effect (by electronic or steric influences) on polymer structures in which it is incorporated. The following additional information should be accumulated to determine the extent of this influence on adhesive systems.

1. The thermal properties of the cured adhesive (TGA, DTA and IGA) should be measured and compared to those of adhesives based on model epoxy compounds which do not contain carborane in the monomer structure.

2. Lap-shear strength of the cured model epoxy compounds should be measured and compared to those of the cured epoxyalkylcarborane monomer.

3. The mechanism of thermal and thermooxidative degradation should be determined (by mass spectrometry, pyrolytic infrared spectroscopy, and pyrolytic gas chromatography).

The long-range potential of this system is dependent on the further characterization of adhesive properties and on the availability of quantities of raw material needed for the synthesis of the epoxy monomer. Not only is the assurance of a long-range source of supply needed but it must be made available at a reasonable cost. This raw material is decaborane, a compound of boron and hydrogen which was originally produced in some quantity for a high energy fuels program. This material is no longer being produced and several R and D

investigations, including this program, have utilized existing stocks. Other promising polymer systems that incorporate carborane in the structure have been reported recently. If enough useful products are developed, then decaborane may become available at a reasonable price. However, until that time, the use of carborane based polymers, including the adhesive developed on this program, is expected to be limited to those applications where cost is not a major determining factor.

5. REFERENCES

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Lap-Shear Strength (Series 44-1)

Bis(epoxybutyl)carborane - 47.8 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-1-1	←No Test			→
44-1-2	500	.003	1640	cohesive
44-1-3	500*	.005	570	adh(coh)
44-1-4	500	.004	1000	cohesive
44-1-5	75	.050	1660	adhesive

Composition - 1.98 g. epoxy + 0.95g hardener
B-stage, 291 - 302°F for 95 minutes
Carrier - 112 Volan A glass cloth
Outgassed - 20 minutes at 248°F
Cure Assembly, 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
Post Cure - *24 hours at 500°F
Substrate - 17/7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-2)

Bis(epoxybutyl)carborane - 48.5 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-2-1	75	.051	2300	adh(coh)
44-2-2	500	.002	800	adh(coh)
44-2-3	500*	.006	1300	coh-ad
44-2-4	500*	Broke in Jaws	---	---
44-2-5	75	.003	1860	---
44-2-6	500	.008	860	coh-ad
44-2-7	500*	Broke in Jaws	---	---
44-2-8	500*	Broke in Jaws	---	---
44-2-9	500*	.006	750	adh(coh)
44-2-10	75	.009	1830	adhesive

Composition - 3.03g. epoxy + 1.47g. hardener
B-stage, 293-304°F for 60 minutes
Carrier - 112 Volan A glass cloth
Outgassed - 60 minutes at 248°F
Cure Assembly - 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
*Post Cure - 24 hours at 500°F
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-3)

Bis(epoxybutyl)carborane - 50 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-3-1	75	.004	1700	---
44-3-2	500	.006	560	adhesive
44-3-3	500	.005	550	adhesive
44-3-4	75	.003	1820	---
44-3-5	500	.004	610	adhesive

Composition - 2.0g. epoxy + 1.0g. hardener
B-stage, 284 - 295°F for 30 minutes followed by 30 minutes at the same temperature under vacuum
Carrier - 112 Volan A glass cloth
Outgassed - 60 minutes at 248°F
Cure Assembly, 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
Post Cure - None
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-4)

Bis(epoxybutyl)carborane - 49.2 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-4-1	75	.008	560	adhesive
44-4-2	75	.022	740	adhesive
44-4-3	75	.015	1240	adhesive
44-4-4	75	.013	1330	adhesive
44-4-5	75	.008	1570	adhesive

Composition - 2.02g. epoxy + 0.99g. hardener
B-stage, 297° - 302°F for 30 minutes followed by 30 minutes at the same temperature under vacuum
Carrier - 112 Volan A glass cloth
Outgassed - 75 minutes at 248°F
Cure Assembly - 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
Post Cure - None
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-5)

Bis(epoxybutyl)carborane - 47.5 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-5-1	75	.008	2080	---
44-5-2	500	.005	530	adhesive
44-5-3	500*	Broke in Jaws	---	---
44-5-4	500*	Broke in Jaws	---	---
44-5-5	75	.008	2060	---
44-5-6	500	.005	800	adh(coh)
44-5-7	500*	Broke in Jaws	---	---
44-5-8	500*	Broke in Jaws	---	---
44-5-9	500	.003	690	adh(coh)
44-5-10	75	.003	2080	---

Composition - 3.33g. epoxy + 1.57g. hardener
B-stage, 302°F for 30 minutes followed by 35 minures additional at the same temperature under vacuum
Outgassed - 60 minutes 248°F
Cure Assembly, - 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
*Post Cure - 24 hours at 500°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-6)

Bis(epoxybutyl)carborane - 48.5 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-6-1	75	.005	1690	adhesive
44-6-2	75*	.004	1270	adhesive
44-6-3	500	.004	600	adhesive
44-6-4	500*	.004	650	adhesive
44-6-5	500	.005	860	adh(coh)
44-6-6	75	.005	1990	cohesive
44-6-7	75*	.005	1400	adhesive
44-6-8	500*	.006	830	coh-ad
44-6-9	500	.006	720	adh(coh)
44-6-10	75	.003	2530	coh-ad

Composition - 3.51g. epoxy + 1.79g. hardener
B-stage, 302 - 311°F for 30 minutes followed by 35 minutes additional at the same temperature under vacuum
Outgassed - 60 minutes at 248°F
Cure Assembly - 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
* Post Cure - 2 hours at 500°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-7)

Bis(epoxybutyl)carborane - 8.2 phr monoepoxybutylcarborane
48.5 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-7-1	75	.004	2320	cohesive
44-7-2	75*	.004	1420	adhesive
44-7-3	500	.004	640	adhesive
44-7-4	500*	.004	600	coh(ad)
44-7-5	500	.004	600	adhesive
44-7-6	75	.005	2000	adhesive
44-7-7	75*	.005	1280	adhesive
44-7-8	500*	.004	250	adhesive
44-7-9	500	.003	320	adhesive

Composition - 3.53g. bis(epoxybutyl) - + 0.29g. mono(epoxybutyl)carborane + 1.72g. hardener
B-stage, 275 - 296°F for 70 minutes. The mixture was very fluid at the end of this period.
Carrier - 112 Volan A glass cloth
Outgassed - 60 minutes at 248°F
Cure Assembly - 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
*Post Cure - 2 hours at 500°F
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-8)

Bis(epoxybutyl)carborane - 10 phr monoepoxybutylcarborane
47.5 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-8-1	75	.006	1550	adh(coh)
44-8-2	75*	.006	1100	adhesive
44-8-3	500	.005	710	adh(coh)
44-8-4	500*	.006	390	coh(adh)
44-8-5	500	.004	490	adhesive
44-8-6	75	.006	1500	adhesive
44-8-7	75*	.005	1040	adhesive
44-8-8	500*	.006	400	adhesive
44-8-9	500	.006	520	adh(coh)
44-8-10	75	.004	1640	adhesive

Composition - 3.60g. bis(epoxybutyl) - + 0.[illegible]g. mono(epoxybutyl)carborane + 1.71g. hardener
Postage, 293° - 302°F for 45 minutes followed by 45 minutes additional at the same temperature under vacuum
Outgassed - 60 minutes at 248°F
Cure Assembly - 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
* Post Cure - 2 hours at 500°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-9)

Bis(epoxybutyl)carborane + 40.5 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-9-1	75	.010	1360	adh(coh)
44-9-2	75*	.008	980	adhesive
44-9-3	500	.007	1060	adh(coh)
44-9-4	500*	.006	1560	adh(coh)
44-9-5	500	.006	1470	adh(coh)
44-9-6	75	.003	2300	adh(coh)
44-9-7	75*	.006	1460	adhesive
44-9-8	500*	.006	1590	adh(coh)
44-9-9	500	.005	1100	adh(coh)
44-9-10	75	.004	2120	coh(adh)

Composition - 4.03g. epoxy + 1.63g. hardener
B-stage, 305 - 307°F for 40 minutes followed by 35 minutes additional at the same temperature under vacuum
Outgassed - 60 minutes at 248°F
Cure Assembly - 352 to 392°F, 120 psi, 2 1/2 hours, Carver Press
* Post Cure - 2 hours at 500°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-10)

Bis(epoxybutyl)carborane - 40.3 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-10-1	75	.008	1914	adhesive
44-10-2	75*	.013	580	adhesive
44-10-3	500	.010	1050	adh-coh
44-10-4	500*	.010	1020	adhesive
44-10-5	500	.007	1304	coh(adh)
44-10-6	75	.020	1340	adhesive
44-10-7	75*	.111	380	adhesive
44-10-8	500*	.011	800	adhesive
44-10-9	500	.016	1000	adh(coh)
44-10-10	75	.012	1540	adhesive

Composition - 4.08g. epoxy + 1.65g. hardener
B-stage, 309°F - 316°F for 70 minutes
Carrier - 112 Volan A glass Cloth
Outgassed - 60 minutes at 248°F
Cure Assembly - 352 to 392°F, 60 psi, 2 1/2 hours, Carver Press
* Post Cure - 2 hours at 500°F
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure 1
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-II)

Bis(epoxybutyl)carborane - 40.8 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-II-1	75	.009	1780	adhesive
44-II-2	75*	.017	734	adhesive
44-II-3	500	.017	940	coh(adh)
44-II-4	500*	.019	750	adhesive
44-II-5	500	.010	1350	coh(adh)
44-II-6	75	.009	1740	adhesive
44-II-7	75*	.014	794	adhesive
44-II-8	500*	.007	1410	adhesive
44-II-9	500	.007	1190	coh-adh
44-II-10	75	.006	1940	adhesive

Composition - 4.04g. epoxy + 1.65g. hardener
B-stage, 315-322°F for 80 minutes
Outgassed - 60 minutes at 248°F
Cure Assembly - 352 to 392°F, 60 psi, 2 1/2 hours
* Post Cure - 2 hours at 500°F
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-12)

Bis(epoxybutyl)carborane 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-12-1	75	.007	2160	adhesive
44-12-2	75*	.110	886	adhesive
44-12-3	500	.006	1030	adh(coh)
44-12-4	500*	.007	1010	adhesive
44-12-5	500	.006	800	adh(coh)
44-12-6	75	.006	2000	adhesive
44-12-7	75*	.008	946	adhesive
44-12-8	500*	.007	1204	adhesive
44-12-9	500	.007	590	adhesive
44-12-10	75	.006	1780	adhesive

Composition - 4.05g. epoxy + 1.41g. hardener
B-stage, 298 - 309°F for 17 minutes followed by 50 minutes additional at the same temperature under vacuum
Outgassed - 60 minutes at 248°F
Cure Assembly, 352 to 392°F, 60 psi, 2 1/2 hours, Carver Press
Post Cure - 2 hours at 500°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual speciments

Lap-Shear Strength (Series 44-13)

Bis(epoxybutyl)carborane - 36.2 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-13-1	75	.006	2060	adhesive
44-13-2	75*	.009	520	adhesive
44-13-3	500	.004	780	adhesive
44-13-4	500*	.005	710	adhesive
44-13-5	500	.006	580	adhesive
44-13-6	75	.009	2100	adhesive
44-13-7	75*	.008	560	adhesive
44-13-8	500*	.008	1400	adh(coh)
44-13-9	500	.117	184	adhesive
44-13-10	75	.008	2060	adhesive

Composition - 3.99g. epoxy + 1.45g. hardener
B-stage, 298 - 309°F for 25 minutes followed by 45 minutes additional at the same temperature under vacuum.
Outgassed - 60 minutes at 248°F
Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press
* Post Cure - 2 hours at 500°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless steel
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-14)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-14-1	75	.005	2130	adh-coh
44-142	75*	.007	970	adh(coh)
44-143	500	.012	470	adh-coh
44-14-4	500*	.006	1130	adh(coh)
44-14-5	500	.004	650	adh-coh
44-14-6	75	.008	2150	adh-coh
44-14-7	75*	.009	1000	adhesive
44-148	500*	.008	1570	adh-coh
44-149	500	.010	1100	adh-coh
44-1410	75	.008	2260	adh-coh

Composition - 3.99g. epoxy + 1.37g. hardener
B-stage, 298 - 309°F for 70 minutes
Outgassed - 60 minutes at 248°F
Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press
* Post Cure - 64 1/2 hours at 400°F
Substrate - 17/7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-15)

Bis(epoxybutyl)carborane – 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-15-1	75	.007	2120	adh-coh
44-15-2	75*	.008	950	adh(coh)
44-15-3	500	.005	840	adh-coh
44-15-4	500*	.008	1300	adh(coh)
44-15-5	500	.005	720	adh-coh
44-15-6	75	.009	2120	adh-coh
44-15-7	75*	.008	1020	adh(coh)
44-15-8	500*	.004	1440	adh(coh)
44-15-9	500	.006	1080	adh-coh
44-15-10	75	.022	1600	adh-coh

Composition - 3.99g. epoxy + 1.37g. hardener
B-stage, 295 - 306°F for 70 minutes
Carrier - 112 Volan A glass cloth
Outgassed - 60 minutes at 248°F
Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press
* Post Cure - 64 1/2 hours at 400°F
Substrate - 17/7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-16)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
[redacted]-16-1	75	.005	2360	adh(coh)
44-16-2	75*	.008	840	adhesive
44-16-3	500	.005	950	adh(coh)
44-16-4	500*	.006	900	adhesive
44-16-5	500	.007	1200	adh-coh
44-16-6	75	.005	2560	adh(coh)
44-16-7	75*	.005	1140	adhesive
44-16-8	500*	.006	820	adhesive
44-16-9	500	.004	750	adh-coh
44-16-10	75	.005	2320	adh-coh

Composition - 4.00g. epoxy + 1.38g. Hardener
[redacted]-stage, 311°F for 50 minutes followed by 20 minutes additional at the same temperature under vacuum.
Outgassed - 60 minutes at 248°F
Cure Assembly - 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press
* Post Cure - 64 1/2 hours at 400°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-17)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-17-1	75	.008	2530	adh-coh
44-17-2	75*	.003	680	adh(coh)
44-17-3	500	.004	800	adh-coh
44-17-4	500*	.004	1550	adh(coh)
44-17-5	500	.004	780	adh-coh
44-17-6	75	.004	2480	cohesive
44-17-7	75*	.004	1660	adh-coh
44-17-8	500*	.010	1180	adh-coh
44-17-9	500	.005	360	cohesive
44-17-10	75	.005	2330	cohesive

Composition - 4.00g. epoxy + 1.38g. Hardener
B-stage, 311°F for 50 minutes followed by 20 minutes additional at the same temperature under vacuum
Outgassed - 60 minutes at 248°F
Cure Assembly - 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press
*Post Cure - 64 1/2 hours at 400°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-18)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-18-1	75	.006	2100	adh-coh
44-18-2	75	.006	1950	adh-coh
44-18-3	75	.004	2100	adh-coh
44-18-4	75	.005	2080	adh-coh
44-18-5	75	.006	2100	adh-coh

Composition - 2.99g. epoxy + 1.04g. hardener
B-stage, 270 - 275°F for 90 minutes and an additional 20 minutes slowly raising the temperature to 315°F
Outgassed - 60 minutes at 248°F
Cure Assembly 302 - 315°F, 60 psi, 2 1/2 hours, Carver Press
Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-19)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-19-1	75	.004	2080	adh-coh
44-19-2	75*	.004	1580	adh-coh
44-19-3	500	.005	550	adhesive
44-19-4	500*	.010	1260	adh-coh
44-19-5	500	.006	490	adh-coh
44-19-6	75	.003	2000	adh-coh
44-19-7	75*	.006	1380	adh-coh
44-19-8	500*	.007	1790	coh(ad)
44-19-9	500	.003	1210	adh-coh
44-19-10	75	.005	2160	adh-coh

Composition - 3.96g. epoxy + 1.30g. hardener
B-stage, 262-270°F for 55 minutes followed by 50 minutes additional at 270 - 302°F. under vacuum
Outgassed - 60 minutes at 248°F
Cure Assebmly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press
*Post Cure - 64 hours at 400°F.
'Carrier - 112 Volan A glass cloth
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strengrh (Series 44-20)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-20-1	75	.004	2120	adh-coh
44-20-2	75*	.005	1440	adh-coh
44-20-3	500	.004	610	adh-coh
44-20-4	500*	.007	1740	adh-coh
44-20-5	500	.005	680	adh-coh
44-20-6	75	.003	2400	adh-coh
44-20-7	75*	.003	1480	adh(coh)
44-20-8	500*	.005	1640	adhesive
44-20-9	500	.006	840	adhesive
44-20-10	75	.007	200	adh-coh

Composition - 4.00g. epoxy + 1.39g. hardener
B-stage, 279 - 293°F for 45 minutes followed by 80 minutes additional at 275-311°F under vacuum.
Carrier - 112 Volan A glass cloth
Outgassed - 60 minutes at 248°F
Cure Assembly, 302-351°F, 60 psi, 2 1/2 hours, Carver Press
*Post Cure - 64 hours of 400°F
Substrate - 17/7 PH Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-21)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-21-1	75	.003	1960	adh-coh
44-21-2	75*	.003	1620	adh-coh
44-21-3	500	.003	520	cohesive
44-21-4	500*	.005	1470	adhesive
44-21-5	500	.004	1060	cohesive
44-21-6	75	.005	1910	adh-coh
44-21-7	75*	.004	1160	adhesive
44-21-8	500*	.006	1260	adhesive
44-21-9	500	.010	680	adh-coh
44-21-10	75	.006	1920	adh-coh

Composition - 4.03 g. epoxy + 1.35 g. hardener

B-stage, 282 - 285°F for 45 minutes followed by 80 minutes additional at 280 - 291°F under vacuum.
Outgassed - 60 minutes at 248°F
Cure Assembly, 302-351°F, 60 psi, 2 1/2 hours, Carver Press
*Post Cure - 64 hours at 400°F
Carrier - 112 Volan A glass cloth
Substrate - 17/7 Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-22)

Bis(epoxybutyl)carborane - 33 phr. G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-22-1	75	.004	2040	adh-coh
44-22-2	75*	.005	1460	adhesive
44-22-3	500	.003	890	adhesive
44-22-4	500*	.003	1860	adh(coh)
44-22-5	500	.005	740	adhesive
44-22-6	75	.003	1870	adh-coh
44-22-7	75*	.006	1150	adh(coh)
44-22-8	500*	.007	1560	adhesive
44-22-9	500	.004	230	adhesive
44-22-10	75	.004	1970	adh-coh

Composition - 4.02g. epoxy + 1.39g. hardener
B-stage, 280 - 282°F for 45 minutes followed by 80 minutes additional at 279-291°F under vacuum
Outgassed - 60 minutes at 248°F
Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press
Post Cure - 64 hours at 400°F
Carrier - 112 Volan A glass cloth
Substrate- 17/7 Stainless steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Sheat Strength (Series 44-23)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-23-1	75	.004	1870	Adhesive
44-23-2	75*	.006	1460	Adhesive
44-23-3	500*	.005	1500	Adhesive
44-23-4	500	.007	410	Adh-Coh
44-23-5	500	.007	480	Adh-Coh
44-23-6	75	.006	1800	Adhesive
44-23-7	75*	.006	1450	Adh(Coh)
44-23-8	500*	.006	1000	Adhesive
44-23-9	500	.007	460	Adh-Coh
44-23-10	500*	.006	1980	Cohesive

Composition - 4.05 epoxy & 1.42 g. hardener

B-stage, 275 - 296°F for 50 minutes followed by 75 minutes additional at 294 - 298°F under vacuum

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 116 hours at 397°F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-24)

Bis(epoxybutyl)carborane - 33 phr - G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-24-1	75	.005	1950	Adhesive
44-24-2	75*	.006	1220	Adh(Coh)
44-24-3	500	.005	770	Adh-Coh
44-24-4	500*	.007	1410	Adhesive
44-24-5	500	.006	680	Adh-Coh
44-24-6	75	.006	2300	Adhesive
44-24-7	75*	.006	1320	Adhesive
44-24-8	500*	.006	1100	Adhesive
44-24-9	500	.006	540	Adh-Coh
44-24-10	500*	.006	1240	Adhesive

Composition - 4.01 g. epoxy & 1.39 g. hardener

B-stage, 271 - 289^{0}F for 50 minutes followed by 75 minutes additional at 285 - 293^{0}F under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302 - 351^{0}F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 116 hours at 397^{0}F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-25)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-25-1	75	.003	1760	Adh(Coh)
44-25-2	75*	.003	1580	Adh-Coh
44-25-3	500	.004	550	Adhesive
44-25-4	500*	.003	790	Adhesive
44-25-5	500	.005	450	Adhesive
44-25-6	75	.004	2440	Cohesive
44-25-7	75*	.005	2120	Adh-Coh
44-25-8	500*	.004	1020	Adhesive
44-25-9	500	.006	550	Adhesive
44-25-10	500*	.003	610	Adh-Coh

Composition - 3.99 g. epoxy & 1.38 g. hardener

B-stage, 280 - 287^{0}F for 50 minutes followed by 75 minutes additional at 285 - 298^{0}F under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302 -351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 64 hours at 332^{0}F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual Specimens

Lap-Shear Strength (Series 44-26)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-26-1	75	.005	2080	Adh(Coh)
44-26-2	75*	.006	1520	Adhesive
44-26-3	500	.005	450	Adhesive
44-26-4	500*	.009	1020	Adh-Coh
44-26-5	500	.007	480	Adh-Coh
44-26-6	75	.006	2160	Adh(Coh)
44-26-7	75*	.006	2140	Adh(Coh)
44-26-8	500*	.009	840	Adh(Coh)
44-26-9	500	.011	380	Adh(Coh)
44-26-10	500*	.009	1040	Adh-Coh

Composition - 4.03 g. epoxy & 1.40 g. hardener

B-stage, 280^{0}F for 50 minutes followed by 75 minutes additional at 282 - 306^{0}F under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302 - 351^{0}F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 64 hours at 332^{0}F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-27)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-27-1	75	.006	2380	Adh-Coh
44-27-2	75*	.006	2430	Cohesive
44-27-3	500	.006	740	Adhesive
44-27-4	500*	.005	1160	Adhesive
44-27-5	500	.006	980	Adhesive
44-27-6	75	.007	1680	Adh-Coh
44-27-7	75*	.006	1840	Adh(Coh)
44-27-8	500*	.005	790	Adh-Coh
44-27-9	500	.007	400	Adhesive
44-27-10	500*	.005	800	Adh-Coh

Composition - 4.02 g. epoxy & 1.37 g. hardener

B-stage, 253 - 280^{0}F for 130 minutes followed by 20 minutes additional at 271 - 275^{0}F under vacuum

Outgassed - 45 minutes at 248^{0}F

Cure Assembly, 244 - 282^{0}F, 30 psi, 22 hours, Carver Press

* Post Cure - 64 hours at 338^{0}F

Carrier - 112 Volan A glass cloth

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

Application - Hot melt

Individual specimens

Lap-Shear Strength (Series 44-28)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-28-1	75*	.008	1420	Adhesive
44-28-2	75*	.007	820	Adhesive
44-28-3	500*	.005	1320	Adhesive
44-28-4	500*	.005	1220	Adhesive
44-28-5	500*	.005	1620	Adhesive
44-28-6	75*	.007	1340	Adhesive
44-28-7	75*	.007	1300	Adhesive
44-28-8	500*	.006	1680	Adhesive
44-28-9	500*	.005	1290	Adhesive
44-28-10	75*	.008	1380	Adhesive

Composition - 4.02 g. epoxy & 1.36 g. hardener

B-stage, 270 - 280°F for 65 minutes followed by 45 minutes additional at 270 - 291°F under vacuum

Outgassed - 30 minutes at 248°F

Cure Assembly, 244 - 282°F, 30 psi, 5 hours, Carver Press

*Post Cure - 7 hours at 356°F followed by 72 1/2 hours additional at 400°F

Carrier - 112 Volan A glass cloth

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

Application - Hot melt

Individual specimens

Lap-Shear Strength (Series 44-29)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44- 29-1	75	.005	560	Adh-Coh
44- 29-2	75*	.005	1920	Adhesive
44- 29-3	500	.005	100	Cohesive
44- 29-4	500*	.005	1360	Adh-Coh
44- 29-5	500	.005	150	Cohesive
44- 29-6	Broke during assembly			
44- 29-7	75*	.006	1750	Adhesive
44- 29-8	500*	.006	1100	Adh-Coh
44- 29-9	500	.005	100	Cohesive
44- 29-10	500*	.006	1430	Adh-Coh
44- 29-11	75	.006	220	Adh-Coh
44- 29-12	500	.007	80	Cohesive
44- 29-13	500*	.005	910	Adhesive
44- 29-14	75	.006	310	Cohesive
44- 29-15	75*	.005	2100	Adhesive

Composition - Individual specimens: 5.00 g. epoxy & 1.73 g. hardener

B-stage: 280 - 288^{0}F for 80 minutes followed by 55 minutes additonal at 291 - 302^{0}F under vacuum

Outgassed - 30 minutes at 248^{0}F

Cure Assembly: 244 - 282^{0}F, 30 psi, 3 hours, Carver Press

*Post Cure - 7 hours at 302^{0}F followed by 72 1/2 hours at 392^{0}C

Carrier - 112 Volan A glass cloth

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

Application - Hot melt

Lap-Shear Strength (Series 44-30)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
100 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-30-1	500*	.006	1260	Adhesive
44-30-2	75	.007	2700	Adh-Coh
44-30-3	500*	.006	1380	Adhesive
44-30-4	75	.007	2920	Adh-Coh
44-30-5	500*	.006	1250	Adhesive

Composition - 2.01 g. epoxy & 0.67 g. hardener & 2.00 g. aluminum powder

B-stage, 269 - 284^{0}F for 30 minutes, add aluminum powder over a 10 minute period, heat for 70 minutes additional at 291 - 294^{0}F under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 305 - 345^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 116 hours at 392^{0}F

Application - Hot melt

Carrier - 112 Volan A glass cloth

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

Individual specimens

Lap-Shear Strength (Series 44-31)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
100 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44- 31-1	75	.007	2580	Cohesive
44- 31-2	75*	.005	1540	Adhesive
44- 31-3	500	.010	830	Adh-Coh
44- 31-4	500*	.009	1320	Adh(Coh)
44- 31-5	500	.010	610	Adhesive
44- 31-6	75	.007	2880	Cohesive
44- 31-7	75*	.005	1780	Adh(Coh)
44- 31-8	500*	.011	1240	Adhesive
44- 31-9	500	.010	530	Adhesive
44- 31-10	500*	.010	1450	Adhesive

Composition - 2.97 g. epoxy & 1.02 g. hardener & 2.99 g. aluminum powder

B-stage, 269 - 284^{0}F for 50 minutes, add aluminum powder over a 5 minute period, heat for 70 minutes additional at 284 - 285^{0}F under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 307 - 350^{0}F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 116 hours at 392^{0}F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-32)

Bis(epoxybutyl)carborane - 25 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44- 32-1	75	.007	1930	Cohesive
44- 32-2	75*	.005	2360	Adh-Coh
44- 32-3	500	.005	430	Cohesive
44- 32-4	500*	.006	920	Adh(Coh)
44- 32-5	500	.005	600	Adh-Coh
44- 32-6	75	.006	1000	Cohesive
44- 32-7	75*	.005	2200	Adh-Coh
44- 32-8	500*	.006	400	Adh-Coh
44- 32-9	500	.006	150	Cohesive
44- 32-10	500*	.007	700	Adh-Coh
44- 32-11	75	.007	2740	Cohesive
44- 32-12	500	.005	120	Cohesive
44- 32-13	75*	.005	2640	Adh-Coh
44- 32-14	75	.007	2420	Cohesive
44- 32-15	75*	.005	2240	Adhesive

Composition - Individual specimens: 5.04 g. epoxy & 1.27 g. Hardener

B-stage: 287 - 302^{0}F for 30 minutes followed by 90 minutes additional at 291 - 298^{0}F under vacuum

Outgassed - Cure Assembly: 60 minutes at 248^{0}F

Post Cure: 307 - 350 ^{0}F, 60 psi, 2 1/2 hours, Carver Press; 64 1/2 hours at 392^{0}F

Carrier - 112 Volan A glass cloth

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

Application - Hot melt

Lap-Shear Strength (Series 44-33)

[illegible](epoxybutyl)carborane - , 33 phr G-50 Hardener

[illegible]	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
[illegible]	75	.006	2120	Adh-Coh
[illegible]	75*	.004	2420	Adh-Coh
[illegible]	500	.006	440	Cohesive
[illegible]	500*	.006	1620	Adh-Coh
[illegible]	500	.005	410	Cohesive
[illegible]	75	.006	2360	Cohesive
[illegible]	75*	.005	2560	Adh-Coh
[illegible]	500*	.006	1210	Adh-Coh
[illegible]	500	.006	340	Cohesive
[illegible]	500*	.006	990	Coh-Ad

[illegible] - 4.02 g. epoxy & 1.38 g. hardener

[illegible] - 280 - 291°F for 50 minutes followed by 75 minures additional at 280 - 285°F under vacuum

[illegible] - 60 minutes at 248°F

[illegible] Assembly - 307 - 350 °F, 60 psi, 2 1/2 hours, Carver Press

[illegible] - 64 1/2 hours at 392°F

[illegible] - 112 Volan A glass cloth

[illegible] - 17-7 PH Stainless Steel

[illegible] - Procedure I

[illegible] - Hot melt

[illegible] specimens

Lap-Shear Strength (Series 44-34)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
100 phr aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-34-1	75	.014	2000	adhesive
44-34-2	75*	--	740	adhesive
44-34-3	500	.009	440	adh(coh)
44-34-4	500*	.010	1380	adhesive
44-34-5	500	.010	420	adh(coh)
44-34-6	75	.011	3020	adhesive
44-34-7	75*	--	740	adhesive
44-34-8	500*	.011	1250	adhesive
44-34-9	500	.009	530	adhesive
44-34-10	500*	.011	1180	adhesive

Composition - 3.01 g. epoxy and 1.02 g. hardener and 3.02 g. aluminum powder

B-stage, 277 - 280^0 F for 50 minutes add aluminum powder over a 5 minute period, heat for additional 65 minutes at 277 - 284^{0}F under vacuum.

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 307 - 350^{0}F, 60 psi , 2 1/2 hours, Carver Press

*Post Cure - 216 hours at 392^{0}F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-35)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
100 phr aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-35-1	75	.010	2680	adh(coh)
44-35-2	75*	--	1090	adhesive
44-35-3	500	.008	400	adh(coh)
44-35-4	500*	.011	1260	adhesive
44-35-5	500	.012	520	adhesive
44-35-6	75	.011	2700	adh(coh
44-35-7	75*	--	1150	adhesive
44-35-8	500*	.011	1200	adhesive
44-35-9	500	.011	770	cohesive
44-35-10	500*	.011	1120	adhesive

Composition - 2.99 g. epoxy and 1.01 g. hardener and 3.02 g. aluminum powder

B-stage 280 - 288^{0}F for 55 minutes, add aluminum powder over a 5 minute period, hear for an additional 65 minutes at the same temperature under vacuum.

Outgassed - 60 minutes at 248^{0}F.

Cure Assembly,. 307 - 350^{0}F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 120 hours at 482^{0}F.

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-36)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-36-1	75	.006	1920	adh(coh)
44-36-2	75*	.007	2060	adh-coh
44-36-3	500	.007	620	adhesive
44-36-4	500*	.006	1350	adh-coh
44-36-5	500	.006	620	adh(coh)
44-36-6	75	.006	2320	cohesive
44-36-7	75*	.006	1960	adh-coh
44-36-8	500*	.006	1340	adh(coh)
44-36-9	500	.006	590	adh-coh
44-36-10	500*	.006	1360	adh-coh

Composition - 4.01 g. epoxy and 1.37 g. hardener

B-stage, 284^{0}F for 50 minutes followed by 70 minutes at 282 - 285 ^{0}F under vacuum.

Outgassed - 60 minutes at 248^{0}F
Cure Assembly, 307 - 350^{0}F, 60 psi, 2 1/2 hours, Carver Press
*Post Cure - 116 hours at 392^{0}F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-37)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-37-1	75	.005	2540	cohesive
44-37-2	75*	.006	2220	adh-coh
44-37-3	500	.011	310	adh-coh
44-37-4	500*	.005	910	adh-coh
44-37-5	500	.012	240	cohesive
44-37-6	75	.008	2140	cohesive
44-37-7	75*	.005	2420	adh-coh
44-37-8	500*	.005	1130	cohesive
44-37-9	500	.010	480	adh-coh
44-37-10	500*	.009	1100	cohesive

Composition - 3.99 g. epoxy and 1.38 g. hardener

B-stage, 285 - 287^{0}F for 50 minutes followed by 70 minutes at 287 - 289^{0}F under vacuum.

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 307 - 350^{0}F, 60 psi, 2 1/2 hours, Carver Press

Post Cure - 116 hours at 392^{0}F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-38)

Bis(epoxybutyl)carborane - 50 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44- 38-1	75	.018	1210	adh(coh)
44- 38-2	75*	.011	1500	adh(coh
44- 38-3	500	.012	610	adh(coh)
44- 38-4	500*	.011	950	adh(coh)
44- 38-5	500	.017	1150	cohesive
44- 38-6	broke in jaws			
44- 38-7	75*	.014	300	cohesive
44- 38-8	broke in jaws			
44- 38-9	500	.018	160	cohesive
44- 38-10	500*	.009	320	cohesive

Composition - 3.98 g. epoxy and 1.98 g. hardener

B-stage, 271 - 280^{0}F for 55 minutes followed by 70 minutes under vacuum at the same temperarure

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 307 - 350^{0}F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 116 hours at 392^{0}F

Carrier - 112 Volan A glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-39)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
100 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-39-1	75	.007	3090	adh (coh)
44-39-2	75*	.006	940	adhesive
44-39-3	500*	.008	1860	coh(adh)
44-39-4	500*	.007	1850	cohesive
44-39-5	75*	.006	2220	adh(coh)
44-39-6	75	.0105	2120	adh-coh
44-39-7	75*	.0065	1860	adhesive
44-39-8	500*	.008	1060	adhesive
44-39-9	75	.006	3260	coh(adh)
44-39-10	500*	.007	1730	adh-coh

Composition - 2.99 g. epoxy and 0.98 g. hardener and 2.95 g. aluminum

B-stage, 280 - 284^{0}F for 55 minutes, add aluminum and heat at 280 - 284^{0}F under vacuum for 80 minutes additional.

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 305 - 345 ^{0}F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 64 hours at 392^{0}F

Carrier - 112 heat cleaned glass cloth
Substrate - 17-7,PH Stainless Steel
Cleaning - Procedure I
Applicarion - Hor melt
Individual specimens

Lap-Shear Strength (Series 44-40)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-40-1	75	.007	2960	adh(coh)
44-40-2	75*	.006	2340	adhesive
44-40-3	500*	.006	1710	adh-coh
44-40-4	500*	.007	2040	adh-coh
44-40-5	75*	.007	2300	adhesive
44-40-6	75	.006	3320	coh(adh)
44-40-7	75*	.006	2360	adhesive
44-40-8	500*	.008	1070	adhesive
44-40-9	75	.006	3200	coh-adh
44-40-10	500*	.008	1270	adhesive

Composition - 3.01 g. epoxy and 0.99 g. hardener and 1.49 g. aluminum

B-stage - 280 - 297 ^{0}F for 55 minutes, add aluminum and heat at 275 - 288 ^{0}F for 65 minutes additional

Outgassed - 60 minutes at 248^{0}F

Cure Assembly - 305 - 345^{0}F, 60 psi, 2 1/2 hours, Carver Press

Post Cure - 64 hours at 392^{0}F

Carrier - 112 heat cleaned glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-41)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-41-1	75	.006	3220	adh-coh
44-41-2	75*	.006	2520	adh(coh)
44-41-3	500*	.008	990	adhesive
44-41-4	500*	.007	790	adhesive
44-41-5	75*	.007	2340	adh(coh)
44-41-6	75	.007	3040	coh(ad)
44-41-7	75*	.008	2040	adhesive
44-41-8	500*	.009	1160	adh(coh)
44-41-9	75	.010	2100	adh(coh)
44-41-10	500*	.009	1480	adh(coh)

Composition - 2.99 g. epoxy and 1.28 hardener

B-stage 273 - 275^{0}F for 30 minutes followed by 55 minutes additional at 275 - 293^{0}F under vacuum.

Outgassed - 60 minutes at 248^{0}F

Cure Assembly 305 - 345^{0}F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 64 hours at 392^{0}F

Carrier - 112 heat cleaned glass cloth
Substrate - 17-7 PH Stainless Steel, 0.05 in.
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-42)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-42-1	75	.010	2780	adh-coh
44-42-2	75*	.008	1600	adhesive
44-42-3	500*	.007	1780	adh(coh)
44-42-4	500*	.008	2180	adhesive
44-42-5	75*	.007	940	adhesive
44-42-6	75	.010	2480	cohesive
44-42-7	75*	.007	1560	adhesive
44-42-8	500*	.006	1200	adhesive
44-42-9	75	.009	2960	cohesive
44-42-10	500*	.007	1410	adhesive

Composition - 2.99 g. epoxy and 0.99 g. hardener and 1.51 g. aluminum

B-stage, 298 - 302°F for 55 minutes, add aluminum, heat for 60 minutes additional at 275 - 293°F under vacuum

Outgassed - 60 minutes at 248°F, 60 psi, 2 1/2 hours, Carver Press

Cure Assembly, 305 - 345°F, 60 psi, 2 1/2 hours

*Post Cure - 4 hours at 500°F.

Carrier - 112 heat cleaned glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-43)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
100 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-43-1	75	.009	2700	adh-coh
44-43-2	75*	.008	1600	adhesive
44-43-3	500*	.009	1250	adhesive
44-43-4	500*	.008	1810	adhesive
44-43-5	75*	.009	1780	adhesive
44-43-6	75	.009	1640	adh-coh
44-43-7	75*	.009	1180	adhesive
44-43-8	500*	.009	1020	adhesive
44-43-9	75	.009	3040	coh(adh)
44-43-10	500*	.009	1080	adhesive

Composition - 3.00 g. epoxy and 1.00 g. hardener and 2.99 g. aluminum

B-stage, 293^{0}F for 55 minutes, add aluminum, heat at 293^{0}F under vacuum for 60 minutes

Outgassed - 60 minutes at 248^{0}F
Cure Assembly, 305 - 345^{0}F, 60 psi, 2 1/2 hours.
*Post Cure - 4 hours at 500^{0}F

Carrier - 112 heat cleaned glass cloth
Substrate - 17-7 PH Stainless Steel
Cleaning - Procedure I
Application - Hot melt
Individual specimens

Lap-Shear Strength (Series 44-44)

Bis(epoxybutyl)carborane — 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-44-1	75	0.005	2700	adh - coh
44-44-2	75*	0.007	1840	adhesive
44-44-3	500*	0.006	2020	adh - coh
44-44-4	500*	0.007	1630	adhesive
44-44-5	75*	0.007	1550	adhesive
44-44-6	75	0.006	2460	cohesive
44-44-7	75*	0.007	1790	adh - coh
44-44-8	500*	0.007	2080	adh - coh
44-44-9	75	0.006	2880	cohesive
44-44-10	500*	0.005	1440	adhesive

Composition — 2.99 g epoxy + 0.98 g hardener + 1.49g aluminum

B-Stage, 284°F for 60 minutes, add aluminum, heat at 280°F for additional 70 minutes under vacuum

Outgassed — 60 minutes at 248°F

Cure Assembly, 305 - 245°F, 60 psi, 2 1/2 hours, Carver Press

Post Cure* — 4 hours at 482°F

Carrier — 112 heat cleaned glass cloth

Substrate — 17-7 PH Stainless Steel

Cleaning — Procedure II

Application - Hot melt

Individual specimens

Lap-Shear Strength (Series 44-45)

Bis(epoxybutyl)carborane — 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-45-1	75	0.006	2550	adh - coh
44-45-2	75*	0.006	1560	adh - coh
44-45-3	500*	0.007	1330	adhesive
44-45-4	500*	0.006	1710	adhesive
44-45-5	75*	0.006	1470	adhesive
44-45-6	75	0.006	2380	adh - coh
44-45-7	75*	0.006	1180	adhesive
44-45-8	500*	0.007	1220	adhesive
44-45-9	75	0.006	2620	adh - coh
44-45-10	500*	0.006	1130	adhesive

Composition — 2.99 g epoxy +0.98g hardener + 1.49g aluminum

B-Stage, 284°F for 60 minutes, add aluminum, heat for 60 minutes at 284°F under vacuum

Outgassed — 60 minutes at 248°F

Cure Assembly, 305 - 345°F, 60 psi, 2 1/2 hours, Carver Press

Post Cure* — 4 hours ar 482°F

Carrier — 112 heat cleaned glass cloth

Substrate — 17-7 PH Stainless Steel

Cleaning: Procedure III

Application - Hot melt

Individual specimens

Lap-Shear Strength (Series 44-46)

Bis(epoxybutyl)carborane — 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-46-1	75	0.005	2700	adh - coh
44-46-2	75*	0.006	2000	adhesive
44-46-3	500*	0.007	1640	adhesive
44-46-4	500*	0.005	1620	adhesive
44-46-5	75*	0.005	1930	adhesive
44-46-6	75	0.005	3180	cohesive
44-46-7	75*	0.005	1830	adhesive
44-46-8	500*	0.007	1000	adhesive
44-46-9	75	0.006	3240	cohesive
44-46-10	500*	0.005	1060	adhesive

Composition — 2.99 g epoxy + 0.98g hardener + 1.49g aluminum

B-Stage, 284°F for 60 minutes, add aluminum, heat for additional 60 minutes under vacuum at 284°F

Outgassed — 60 minutes at 248°F

Cure Assembly, 305 - 345°F, 60 psi, 2 1/2 hours, Carver Press

Post Cure* — 4 hours ar 482°F

Carrier — 112 heat cleaned glass cloth

Substrate — 17-7 PH Stainless Steel

Cleaning- Procedure I

Application - Hot melt

Individual specimens

Lap-Shear Strength (Series 44-47)

Bis(epoxybutyl)caiborane — 50 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-47-1	75	0.007	2740	coh - adh
44-47-2	75*	0.007	2160	adhesive
44-47-3	500*	0.006	1000	adhesive
44-47-4	500*	0.006	1060	adh - coh
44-47-5	75*	0.007	1900	adhesive
44-47-6	75	0.006	2260	adhesive
44-47-7	75*	0.007	2040	adh - coh
44-47-8	500*	0.006	1200	coh - coh
44-47-9	75	0.007	3200	coh - adh
44-47-10	500*	0.006	950	adhesive

Composition — 4.00 g epoxy + 1.99 g hardener + 1.99 g aluminum

B-Stage, 248°F for 60 minutes, add aluminum, heat for 40 minutes at 284°F under vacuum

Outgassed — 60 minutes at 248°F

Cure Assembly, 305 - 345°F, 60 psi, 2 1/2 hours, Carver Press

Post Cure* — 4 hours at 482°F

Carrier — 112 heat cleaned glass cloth

Substrate — 17-7 PH Stainless Steel

Cleaning - Procedure I

Application - Hot melt

Individual specimens

Lap-Shear Strength (Series 44-52)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-52-1	75	.004	1830	Adh - Coh
44-52-2	75*	.005	1400	Adhesive
44-52-3	500*	.006	1090	Adhesive
44-52-4	500*	.007	1140	Adhesive
44-52-5	75*	.005	1780	Adhesive
44-52-6	75	.005	2440	Adh - Coh
44-52-7	75*	.005	1520	Adhesive
44-52-8	500*	.006	1320	Adhesive
44-52-9	75	.004	2850	Adh - Coh
44-52-10	500*	.006	1420	Adhesive

Composition - 3.00 g epoxy + 1.00 g hardener + 1.50 g aluminum

B-stage, 270 - 289°F for 60 minutes, add aluminum, heat at 282 - 302°F for 60 minutes additional under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi, 2-1/2 hours, Carver Press

*Post Cure - 4 hours at 482°F

Carrier - 112 heat cleaned glass cloth

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

From Partially Machined Panels

Application - Hot melt

Lap-Shear Strength (Series 44-53)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-53-1	75	.004	2800	Adh - Coh
44-53-2	75*	.005	1640	Adhesive
44-53-3	500*	.008	1840	Adhesive
44-53-4	500*	.007	2100	Adhesive
44-53-5	75*	.005	1820	Adhesive
44-53-6	75	.004	2880	Adh - Coh
44-53-7	75*	.005	1780	Adhesive
44-53-8	500*	.008	1510	Adhesive
44-53-9	75	.004	2640	Adh-Coh
44-53-10	500*	.007	1210	Adhesive

Composition - 2.99 g epoxy + 1.00 g hardener + 1.51 g aluminum

B-stage, 275 - 287°F for 60 minutes, add aluminum, heat at 284 - 302°F for 70 minutes additional under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi, 2-1/2 hours, Carver Press

*Post Cure - 4 hours at 482°F

Carrier - 112 heat cleaned glass cloth

Substracte - 17-7 PH Stainless Steel

Cleaning - Procedure I

From Completely Machined Panels

Application - Hot melt

Lap-Shear Strength (Series 44-54)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-54-1	75	.005	2480	Adh - Coh
44-54-2	75*	.005	1500	Adhesive
44-54-3	500*	.007	2000	Adhesive
44-54-4	500*	.007	310	Adhesive
44-54-5	75*	.006	1510	Adhesive
44-54-6	75	.005	2560	Adh - Coh
44-54-7	75*	.005	1490	Adhesive
44-54-8	500*	.007	1670	Adhesive
44-54-9	75	.005	2580	Adh - Coh
44-54-10	500*	.008	1440	Adhesive

Composition - 3.00 g epoxy + 1.00 g hardener + 1.51 g aluminum

B-stage, 277 - 291°F for 60 minutes, add aluminum, heat at 275 - 293°F for 60 minutes additional under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi, 2-1/2 hours, Carver Press

*Post Cure - 4 hours at 482°F

Carrier - 112 heat cleaned glass cloth

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

From Partially Machined Panels With Saw Cuts

Application - Hot melt

Lap-Shear Strength (Series 44-55)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-55-1	75	.005	3380	Cohesive
44-55-2	75*	.005	2300	Adh (Coh)
44-55-3	500*	.006	2040	Adh-Coh
44-55-4	500*	.006	2200	Adh-Coh
44-55-5	75*	.006	2180	Adh(Coh)
44-55-6	75	.005	2720	Cohesive
44-55-7	75*	.005	2220	Adh(Coh)
44-55-8	500*	.005	2280	Coh-Adh
44-55-9	75	.004	2600	Cohesive
44-55-10	500*	.005	2080	Coh-Adh

Composition - 3.01g epoxy + 1.01g hardener + 1.49 aluminum

B-Stage, 280 - 300°F for 60 minutes, add aluminum, heat at 275-298°F for 60 minutes additional under vacuum

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 470°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at ~338°F

Substrate: 17-7 PH Stainless Steel

Cleaning: Procedure II

From completely machined panels

Application - Hot melt

Lap-Shear Strength (Series 44-56)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-56-1	75	.006	2600	Coh-Adh
44-56-2	75*	.005	1480	Adhesive
44-56-3	500*	.004	1500	Adhesive
44-56-4	500*	.005	1560	Adhesive
44-56-5	75*	.006	1260	Adhesive
44-56-6	75	.005	2520	Coh-Adh
44-56-7	75*	.005	1080	Adhesive
44-56-8	500*	.005	1580	Adhesive
44-56-9	75	.005	2680	Cohesive
44-56-10	500*	.005	1480	Adhesive

Composition - 3.02g epoxy + 0.99 hardener + 1.47 aluminum

B-Stage, 266 - 300°F for 60 minutes, add aluminum, heat at 275 - 293°F for 60 minutes under vacuum

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 470°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at ~ 338°F

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

From completely machined panels

Application - Hot melt

Lap-Shear Strength (Series 44-57)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-57-1	75	.005	2660	Cohesive
44-57-2	75*	.006	2000	Adh (Coh)
44-57-3	500*	.006	1890	Adh-Coh
44-57-4	500*	.005	1700	Adhesive
44-57-5	75*	.006	2080	Adh (Coh)
44-57-6	75	.005	2760	Cohesive
44-57-7	75*	.006	1920	Adh (Coh)
44-57-8	500*	.006	1780	Adhesive
44-57-9	75	.005	2900	Cohesive
44-57-10	500*	.006	1540	Adhesive

Composition - 3.02g epoxy + 0.99g hardener + 1.50g aluminum

B-Stage, 273 - 293°F for 60 minutes, add aluminum, heat at 275 - 293°F for 75 minutes additional under vacuum

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 4 hours at 470°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at ~ 338°F

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure II

From completely machined panels

Application - Hot melt

Lap-Shear Strength (Series 44-58)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-58-1	75	.005	3280	Cohesive
44-58-2	75*	.006	1440	Adhesive
44-58-3	500*	.005	1900	Adhesive
44-58-4	500*	.006	1720	Adhesive
44-58-5	75*	.005	1280	Adhesive
44-58-6	75	.005	2600	Cohesive
44-58-7	75*	.005	1600	Adhesive
44-58-8	500*	.005	990	Adhesive
44-58-9	75	.005	2550	Cohesive
44-58-10	500*	.006	1120	Adhesive

Composition - 3.01g epoxy + 1.01g hardener + 1.52g aluminum

B-Stage, 271 - 287°F for 60 minutes, add aluminum, heat at 275 - 293°F for 60 minutes under vacuum

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi , 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 470°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at ~338°F

Substrate - 17-7 PH Stainless Steel

Cleaning - Procedure I

From completely machined panels

Application - Hot melt

Lap-Shear Strength (Series 44-59)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-59-1	75	.005	3010	Cohesive
44-59-2	75*	.005	2400	Adh-Coh
44-59-3	500*	.005	2320	Cohesive
44-59-4	500*	.005	2340	Cohesive
44-59-5	75*	.005	2240	Adh-Coh
44-59-6	75	.006	3100	Cohesive
44-59-7	75*	.004	2300	Adh-Coh
44-59-8	500*	.005	1920	Coh (Adh)
44-59-9	75	.005	2950	Cohesive
44-59-10	500*	.005	1720	Coh (Adh)

Composition - 3.00g epoxy + 1.00g hardener + 1.51 aluminum

B-Stage, 275 - 287°F for 60 minutes, add aluminum, heat at 293 - 298°F for 60 minutes additional under vacuum

Outgassed - 60 minutes at 248°F

Cure Assembly, 302 - 351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 470°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at ~ 338°F

Cleaning: Procedure II

From completely machined panels

Subsrrate: 17/7 PH Stainless Steel

Application - Hot melt

Lap-Shear Strength (Series 44-60)

Bis(epoxybutyl)carborane – 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-60-1	75	.006	2770	Cohesive
44-60-2	75*	.006	2340	Adh-Coh
44-60-3	500*	.005	2000	Coh (Adh)
44-60-4	500*	.005	2120	Coh (Adh)
44-60-5	75*	.005	2160	Adh (Coh)
44-60-6	75	.005	2520	Cohesive
44-60-7	75*	.005	2460	Adh-Coh
44-60-8	500*	.005	1470	Coh (Adh)
44-60-9	75	.005	2550	Cohesive
44-60-10	500*	.005	1470	Coh (Adh)

Composition – 3.01g epoxy + 0.99g hardener + 1/49g aluminum

B-Stage, 275 – 293°F for 60 minutes, add aluminum, heat at 293 – 298°F for 60 minutes additional under vacuum

Outgassed – 60 minutes at 248°F

Cure Assembly, 302 – 351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure – 4 hours at 470°F, gravity convection oven

Carrier – 112 heat cleaned glass cloth, pretreated at ~338°F

Substrate – 17-7 PH Stainless Steel

Cleaning – Procedure II

From completely machined panels

Application – Hot melt

Lap-Shear Strength (Series 44-61)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-61-1	75	.005	2540	Cohesive
44-61-2	75*	.006	2240	Cohesive
44-61-3	500*	.007	2340	Cohesive
44-61-4	500*	.005	2000	Coh (Adh)
44-61-5	75*	.005	2080	Cohesive
44-61-6	75	.006	2820	Cohesive
44-61-7	75*	.006	2280	Cohesive
44-61-8	500*	.006	1840	Coh (Adh)
44-61-9	75	.006	2710	Cohesive
44-61-10	500*	.006	1860	Coh (Adh)

Composition - 3.00g. epoxy + 1.00g. hardener + 1.52g. aluminum

B-stage, 289-298^{0}F for 60 minutes, add aluminum, heat at 277-293^{0}F for 60 minutes additional under vacuum.

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302-351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 460±2^{0}F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at ~338^{0}F

Substrate - 17-7 PH stainless steel

Cleaning - MEK, Procedure II

From completely machined panels

Application - Hot melt

Lap-Shear Strength (Series 44-62)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-62-1	75	.006	2480	Cohesive
44-62-2	75*	.005	2240	Cohesive
44-62-3	500*	.006	2440	Coh-Adh
44-62-4	500*	.005	2350	Coh (Adh)
44-62-5	75*	.005	2160	Cohesive
44-62-6	75	.005	3220	Cohesive
44-62-7	75*	.005	2300	Cohesive
44-62-8	500*	.006	1930	Cohesive
44-62-9	75	.006	2520	Cohesive
44-62-10	500*	.005	2050	Cohesive

Composition - 3.01g. epoxy + 0.99g. hardener + 1.50g. aluminum

B-stage, 289-302^{0}F for 60 minutes, add aluminum, heat at 266-293^{0}F for 60 minutes additional under vacuum.

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302-351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 460 $\pm$ 2^{0}F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at ~750^{0}F

Substrate - 17-7 PH stainless steel

Cleaning - MEK, Procedure II

From completely machined panels

Application - Hot melt

Lap-Shear Strength (Series 44-63)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-63-1	75	.006	3040	Cohesive
44-63-2	75*	.007	860	Adhesive
44-63-3	500*	.007	430	Adhesive
44-63-4	500*	.007	240	Adhesive
44-63-5	75*	.008	720	Adhesive
44-63-6	75	.006	3030	Cohesive
44-63-7	75*	.008	380	Adhesive
44-63-8	500*	.006	800	Adhesive
44-63-9	75	.006	2800	Cohesive
44-63-10	500*	.007	910	Adhesive

Composition - 3.01g. epoxy + 0.99g. hardener + 1.51g. aluminum

B-stage, 271-293^{0}F for 60 minutes, add aluminum, heat at 275-289^{0}F for 60 minutes additional under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly - 302-351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 392^{0}F followed by 4 hours at 505-520^{0}F, forced draft oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750^{0}F

Substrate - 17-7 PH stainless steel, 0.063 in.

Cleaning - MEK, Procedure II

Individual specimens

Application - Hot melt

Lap-Shear Strength (Series 44-64)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-64-1	75	.008	2900	Cohesive
44-64-2	75*	.009	750	Adhesive
44-64-3	500*	.007	380	Adhesive
44-64-4	500*	.007	390	Adhesive
44-64-5	75*	.008	1100	Adhesive
44-64-6	75	.008	2520	Cohesive
44-64-7	75*	.008	1130	Adhesive
44-64-8	500*	.006	930	Adhesive
44-64-9	75	.007	2960	Cohesive
44-64-10	500*	.007	950	Adhesive

Composition - 2.99g. epoxy + 0.99g. hardener + 1.50g. hardener

B-stage, 264-288^{0}F for 60 minutes, add aluminum, heat at 287-293^{0}F for 60 minutes additional under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302-351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 505-520^{0}F, forced draft oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750^{0}F

Substrate - 17-7 PH stainless steel, 0.063 in

Cleaning - MEK, Procedure II

Individual specimens

Application - Hot melt

Lap-Shear Strength (Series 44-65)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-65-1	75	.008	2940	Cohesive
44-65-2	75*	.007	1670	Adhesive
44-65-3	500*	.008	1400	Coh-Adh
44-65-4	500*	.009	1470	Adh(Coh)
44-65-5	75*	.008	1620	Adhesive
44-65-6	75	.009	2280	Cohesive
44-65-7	75*	.010	2160	Adh(Coh)
44-65-8	500*	.009	2020	Adh(Coh)
44-65-9	75	.006	2880	Cohesive
44-65-10	500*	.008	1480	Adhesive

Composition - 2.99g. epoxy + 1.01g. hardener + 1.50g. aluminum

B-stage, 266-294^{0}F for 60 minutes, add aluminum, heat at 293-298^{0}F for 60 minutes under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302-351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 453-467^{0}F, forced draft oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750^{0}F

Substrate - 17-7 PH stainless steel, 0.063 in.

Cleaning - MEK, Procedure II

Individually assembled specimens

Application - Hot melt

Lap-Shear Strength (Series 44-66)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-66-1	75	.006	2600	Cohesive
44-66-2	75*	.008	2780	Adh-Coh
44-66-3	500*	.009	1450	Adh-Coh
44-66-4	500*	.008	1650	Adh-Coh
44-66-5	75*	.008	2460	Adh-Coh
44-66-6	75	.006	3640	Cohesive
44-66-7	75*	.007	2660	Adh-Coh
44-66-8	500*	.007	2080	Coh-Adh
44-66-9	75	.007	2540	Cohesive
44-66-10	500*	.008	1710	Adh-Coh

Composition - 3.01g. epoxy + 1.00g. hardener + 1.50g. aluminum

B-stage, 269-307^{0}F for 60 minutes, add aluminum, heat at 284-302^{0}F for 60 minutes additional under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302-351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 458-462^{0}F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750^{0}F

Substrate - 17-7 PH stainless steel, 0.063 in.

Cleaning - MEK, Procedure II

Individually Assembled Specimens

Application - Hot melt

Lap-Shear Strength (Series 44-67)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-67-1	75	.008	2730	Cohesive
44-67-2	75*	.008	2180	Adh(Coh)
44-67-3	500*	.008	1540	Adh(Coh)
44-67-4	500*	.007	1680	Adh(Coh)
44-67-5	75*	.008	1740	Adhesive
44-67-6	75	.007	3660	Cohesive
44-67-7	75*	.010	1680	Adhesive
44-67-8	500*	.008	1300	Adhesive
44-67-9	75	.008	3200	Cohesive
44-67-10	500*	.008	2000	Adh(Coh)

Composition - 3.00g. epoxy + 1.00g. hardener + 150g. aluminum

B-stage, 293-302^{0}F for 60 minutes, add aluminum, heat at 280-293^{0}F for 60 minutes under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302-351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 453-467^{0}F, forced draft oven

Carrier - 112 heat cleaned glass cloth, pretreatment at 750^{0}F

Substrate - 17-7 PH stainless steel, 0.063 in.

Cleaning - MEK, benzene, Procedure II

Individually assembled specimens

Application - Hot melt

Lap-Shear Strength (Series 44-68)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-68-1	75	.008	2940	Cohesive
44-68-2	75*	.008	2640	Adh(Coh)
44-68-3	500*	.008	1970	Coh(Adh)
44-68-4	500*	.007	2310	Cohesive
44-68-5	75*	.007	2720	Adh-Coh
44-68-6	75	.007	3380	Cohesive
44-68-7	75*	.008	2460	Adh-Coh
44-68-8	500*	.008	2150	Cohesive
44-68-9	75	.008	2660	Cohesive
44-68-10	500*	.008	2010	Cohesive

Composition - 3.00g. epoxy + 1.00g. hardener + 1.50g. aluminum

B-stage, 276-298^{0}F for 60 minutes, add aluminum, heat at 284-294^{0}F for 60 minutes under vacuum

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 302-351^{0}F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 458-462^{0}F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750^{0}F

Substrate - 17-7 PH stainless steel, 0.063 in.

Cleaning - MEK, benzene, Procedure II

Individually assembled specimens

Application - Hot melt

Lap-Shear Strength (Series 44-69)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-69-1	75	0.009	3080	cohesive
44-69-2	75*	0.008	2780	adhesive
44-69-3	500*	0.008	2140	cohesive
44-69-4	500*	0.008	1700	cohesive
44-69-5	75*	0.009	2600	adhesive
44-69-6	75	0.008	3080	cohesive
44-69-7	75*	0.009	2300	adhesive
44-69-8	500*	0.009	2120	coh (adh)
44-69-9	75	0.009	2920	cohesive
44-69-10	500*	0.008	2080	coh (adh)

Composition - 3.01g. epoxy + 1.01g. hardener + 1.49g. aluminum

B-stage, 296-302°F for 60 minutes, add aluminum, heat at 287-293°F for 60 minutes under vacuum

Outgassed - 60 minutes at 248°F

Cure Assembly, 302-351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 468-472°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual Specimens

Application - Hot melt

Lap-Shear Strength (Series 44-70)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strenght psi	Type of Failure
44-70-1	75	0.008	2870	cohesive
44-70-2	75*	0.008	2420	adhesive
44-70-3	500*	0.008	1620	adh (coh)
44-70-4	500*	0.008	2210	coh (adh)
44-70-5	75*	0.008	2360	adhesive
44-70-6	75	0.008	3280	cohesive
44-70-7	75*	0.009	2340	adhesive
44-70-8	500*	0.007	1940	cohesive
44-70-9	75	0.008	3480	cohesive
44-70-10	500*	0.008	2210	cohesive

Composition - 3.00g. epoxy + 1.00g. hardener + 1.50g. aluminum

B-stage, 258-293°F for 60 minutes, add aluminum, heat at 275-302°F for 60 minutes under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302-351°F, 60 psi, $2\frac{1}{2}$ hours, Carver Press

* Post Cure - 4 hours at 468-472°F, gravity convection oven

Carrier - 112 heat-cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual Specimens

Application - Hot melt

Lap-Shear Strength (Series 44-71)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-71-1	75	0.008	2560	cohesive
44-71-2	75*	0.008	2340	adhesive
44-71-3	500*	0.006	2200	cohesive
44-71-4	500*	0.006	2120	cohesive
44-71-5	75*	0.010	2240	adhesive
44-71-6	75	0.007	3150	cohesive
44-71-7	75*	0.009	2460	adhesive
44-71-8	500*	0.007	2480	cohesive
44-71-9	75	0.008	3200	cohesive
44-71-10	500*	0.006	2290	cohesive

Composition - 3.01g. epoxy + 1.29g. hardener + 1.50g. aluminum

B-stage, 266-302°F for 60 minutes, add aluminum, heat at 287-302°F for 60 minutes under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302-351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 468-472°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual Specimens

Application - Hot melt

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Lap-Shear Strength (Series 44-72)

Bis(epoxybutyl)carborane - 33 phr G-50 Hardener
50 phr Aluminum
10 phr bis(epoxypentyl)carborane

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-72-1	75	0.008	3200	cohesive
44-72-2	75*	0.009	2400	adhesive
44-72-3	500*	0.008	1740	cohesive
44-72-4	500*	0.007	1400	cohesive
44-72-5	75*	0.009	2420	adhesive
44-72-6	75	0.009	3030	cohesive
44-72-7	75*	0.009	2350	adhesive
44-72-8	500*	0.008	1760	cohesive
44-72-9	75	0.009	3000	cohesive
44-72-10	500*	0.008	1940	cohesive

Composition - 3.00g. bis(epoxybutyl)carborane + 0.30g. bis(epoxypentyl)carborane + 1.00g. hardener + 1.49g. aluminum

B-stage, 275-298°F for 60 minutes, add aluminum, heat at 269-296°F for 60 minutes under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302-351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 468-472°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual Specimens

Application - Hot melt

Lap-Shear Strength (Series 44-73)

Bis(epoxybutyl)carborane - 33 phr G-50
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-73-1	75	.008	2900	Cohesive
44-73-2	75*	.008	2380	Adhesive
44-73-3	500*	.006	2220	Adh-Coh
44-73-4		Poor Specimen		
44-73-5	75*	.008	2180	Adhesive
44-73-6	75	.007	2840	Cohesive
44-73-7	75*	.009	2300	Adhesive
44-73-8	500*	.008	2000	Adh-Coh
44-73-9	75	.010	3320	Cohesive
44-73-10	500*	.009	1500	Adhesive

Composition - 3.01g. epoxy + 1.00g. hardener + 1.50g. aluminum

B-stage, 280-302°F for 85 minutes, add aluminum, heat at 266-295°F for 35 minutes under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302-351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 462-468°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel. 0.063 in.

Cleaning - Procedure II

Individual Specimens

Application - Hot melt

Lap-Shear Strength (Series 44-74)

Bis(epoxybutyl)carborane - 43 phr G-50
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-74-1		Poor Specimen		
44-74-2	75*	.009	2290	Adhesive
44-74-3	500* (10'hold)	.008	2320	Cohesive
44-74-4	500* (30'hold)	.007	2630	Adh-Coh
44-74-5	75*	.006	2250	Adhesive
44-74-6	75	.006	3070	Cohesive
44-74-7	75*	.006	2100	Adhesive
44-74-8	500* (30'hold)	.007	2360	Adhesive
44-74-9	75	.009	2880	Cohesive
44-74-10		Poor Specimen		

Composition - 3.01g. epoxy + 1.29g. hardener + 1.51g. aluminum

B-stage, 258-293°F for 60 minutes, add aluminum, heat at 291-298°F for 60 minutes under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302-351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 462-468°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual Specimens

Application - Hot melt

Lap-Shear Strength (Series 44-75)

Bis(epoxybutyl)carborane - 47.5 phr G-50
50 phr Aluminum
10 phr Bis(epoxypentyl)carborane

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-75-1	75	.007	3520	Cohesive
44-75-2	75*	.008	2120	Adh(Coh)
44-75-3	500* (10'hold)	.009	970	Adhesive
44-75-4	500* (30'hold)	.007	1700	Adh-Coh
44-75-5	75*	.007	2120	Adhesive
44-75-6	75	.008	2940	Cohesive
44-75-7	75*	.008	2220	Adhesive
44-75-8	500* (10'hold)	.008	1260	Adhesive
44-75-9	75	.008	3240	Cohesive
44-75-10	500* (30'hold)	.007	1900	Adh-Coh

Composition - 2.99g. bis(epoxybutyl)carborane + 0.31g. bis(epoxypentyl)carborane + 1.43g. hardener + 1.50g. aluminum

B-stage, 289-307°F for 60 minutes, add aluminum, heat at 266-293°F for 60 minutes under vacuum.

Outgassed - 60 minutes at 248°F

Cure Assembly, 302-351°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 462-468°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual specimens

Application - Hot melt

Lap-Shear Strength (Series 44-77)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-77-1	75	.006	3120	Cohesive
44-77-2	75*	.006	2700	Coh (Adh)
44-77-3	500* (10' hold)	.008	2500	Cohesive
44-77-4	75*	.006	2600	Coh (Adh)
44-77-5	500* (10' hold)	.006	1720	Cohesive

Composition - 3.01g. epoxy + 1.29g. hardener + 1.50g. aluminum

B-stage, 257-298°F for 60 minutes, add aluminum, heat at 277-298°F for 60 minutes additional under vacuum.

Outgassed - 60 minutes at 248°F.

Cure Assembly, 302-350°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 435-493°F, gravity convection oven

* Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

From completely machined panels

Application - Hot melt

Lap-Shear Strength (Series 44-79)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-79-1	75	.008	1310	Cohesive
44-79-2	75*	.008	1780	Adh (Coh)
44-79-3	500* (10' hold)	.011	670	Adhesive
44-79-4	500* (10' hold)	.010	1320	Adh (Coh)
44-79-5		Poor Specimen		
44-79-6	75*	.008	2000	Adh (Coh)
44-79-7	75*	.010	1920	Adh-Coh
44-79-8	500* (10' hold)	.010	1870	Adh-Coh
44-79-9	500* (30' hold)	.011	1760	Coh-Adh
44-79-10	500* (30' hold)	.010	1150	Coh)Adh

Composition - 3.01g. epoxy + 1.27g. hardener + 1.49g. aluminum

B-stage, 262-293°F for 60 minutes, add aluminum, heat at 284-298°F for 60 minutes under vacuum.

Outgassed - No

Cure Assembly, 340-360°F, 14 psi, 2 1/2 hours (Vacuum bag)

* Post Cure - 4 hours at 435-493°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual Specimens

Applicarion - Hot melt

Lap-Shear Strength (Series 44-80)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-80-1	75	.008	1910	Cohesive
44-80-2	75*	.007	2520	Coh-Adh
44-80-3	500* (10' hold)	.008	1780	Cohesive
44-80-4	500* (10' hold)	.007	1880	Cohesive
44-80-5	75*	.008	2880	Cohesive
44-80-6	75*	.007	2700	Cohesive
44-80-7	75*	.008	2400	Cohesive
44-80-8	Poor Specimen			
44-80-9	500* (30' hold)	.008	830	Cohesive
44-80-10	Poor Specimen			

Composition - 3.00g. epoxy + 1.28g. hardener + 1.49g. aluminum

B-stage, 273-289°F for 60 minutes, add aluminum, heat at 278-295°F for 60 minutes under vacuum

Outgassed - No

Cure Assembly, 340-360°F, 14 psi, 2 1/2 hours (Vacuum bag)

* Post Cure - 4 hours at 435-493°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual Specimens

Application - Hot melt

Lap-Shear Strength (Series 44-81)

Bis(epoxybutyl)carborane - 43-phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-81-1	75	.006	1320	Cohesive
44-81-2	75*	.009	2580	Cohesive
44-81-3	500* (10' hold)	.008	1840	Cohesive
44-81-4	500* (10' hold)	.008	1630	Cohesive
44-81-5		Poor Specimen		
44-81-6	75*	.008	2450	Cohesive
44-81-7	75*	.009	2380	Coh (Adh)
44-81-8	500* (10' hold)	.007	1650	Cohesive
44-81-9	500* (30' hold)	.008	2130	Cohesive
44-81-10	500* (30' hold)	.007	2080	Cohesive

Composition - 2.99g. epoxy + 1.29g. hardener + 1.49g. aluminum

B-stage, 280-296°F for 60 minutes, add aluminum, heat at 282-296°F for 60 minutes under vacuum.

Outgassed - No

Cure Assembly, 340-360°F, 14 psi, 2 1/2 hours (Vacuum bag)

* Post Cure - 4 hours at 435-493°F, gravity convection oven

Carrier - 112 hear cleaned glass cloth, pretreated at 750°F

Substrate - 17-7 PH Stainless Steel, 0.063 in.

Cleaning - Procedure II

Individual specimens

Application - Hot melt

Lap-Shear Strength (Series 44-82)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

Specimen Number	Test Temperature °F *	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-82-1	75	.006	2630	Cohesive
44-82-2	500 (10' hold)	.006	930	Cohesive
44-82-3	500 (30' hold)	.007	1000	Coh (Adh).
44-82-4	75	.006	2720	Cohesive
44-82-5	500 (10' hold)	.005	940	Coh (Adh)
44-82-6	75	.006	2480	Coh (Adh)
44-82-7	500 (10' hold)	.005	830	Cohesive
44-82-8	500 (30' hold)	.007	980	Cohesive
44-82-9	75	.006	2400	Coh(Adh)
44-82-10	500 (10' hold)	.005	900	Coh (Adh)

Composition - 20.94g. epoxy + 8.59g. hardener + 9.96g. aluminum + 5.77g. asbestos

B-stage, 293-302°F for 15 minutes (clear), add aluminum and asbestos, heat at 293-302°F with occasional vacuum for 40 minutes additional.

Outgassed - one hour at 212°F on glass cloth.

Cure Assembly, 340-360°F, 14 psi, 2½ hours (vacuum bag)

Post Cure, 4 hours at 435-495°F, gravity convection oven all specimens

Carrier - 112 heat cleaned glass cloth, pretreared at 750°F for one hour

Cleaning - Procedure II

Completely machined panels

Substrate: 17-7 PH Stainless steel

Application - Tape

Lap-Shear Strength (Series 44-83)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

Specimen Number	Test Temperature °F *	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-83-1	75	.006	2780	Cohesive
44-83-2	500 (10' hold)	.006	930	Cohesive
44-83-3	500 (30' hold)	.007	1110	Coh (Adh)
44-83-4	75	.007	2840	Cohesive
44-83-5	500 (10' hold)	.006	830	Cohesive
44-83-6	75	.007	2500	Cohesive
44-83-7	500 (10' hold)	.006	900	Coh (Adh)
44-83-8	500 (30' hold)	.007	1050	Cohesive
44-83-9	75	.007	2940	Cohesive
44-83-10	500 (10' hold)	.006	1040	Cohesive

See Series 44-82 for conditions of assembly, cure, and post-cure.

Lap-Shear Strength (Series 44-84)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
36 phr Asbestos

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-84-1	500 (10' hold)		2060	Cohesive
44-84-2	500 (10' hold)		2480	Cohesive
44-84-3	492 (10' hold)		2860	Cohesive
44-84-4	500 (10' hold)	.005 to	2240	Cohesive
44-84-5	500 (30' hold)	.010	2420	Cohesive
44-84-6	500 (30' hold)		2340	Cohesive
44-84-7	500 (30' hold)		1980	Cohesive
44-84-8	500 (30' hold)		2000	Cohesive
44-84-9	550 (10' hold)		970	Adh (Coh)
44-84-10	584 (10' hold)		270	Adhesive

Composition - 40.00g. epoxy + 17.20g. hardener + 20.00g. aluminum + 14.40g. asbestos

B-stage, 293-302°F for 15 minutes (clear), add aluminum and asbestos, heat at 293-302°F with occasional vacuum for 40 minutes additional

Outgassed - one hour at 212°F on glass cloth

Cure Assembly, 307-351°F, 60 psi, 2 ½ hours (Carver Press)

* Post Cure - 4 hours at 435-495°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F for one hour

Cleaning - Procedure II

Completely machined panels

Substrate - 17/7 PH Stainless Steel

Application - Tape

Lap-Shear Strength (Series 44-85)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
36 phr Asbestos

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
44-85-1	500 (10' hold)	.005	1970	Cohesive
44-85-2	500 (10' hold)	.005	2360	Cohesive
44-85-3	500 (60' hold)	.006	2360	Cohesive
44-85-4	550 (10' hold)	.005	970	Adh (Coh)
44-85-5	500 (30' hold)	.005	2130	Cohesive

Composition - See Series 84

B-stage - See Series 84

Outgassed - No

Cure Assembly, < 340-360°F at 0-15 psi for 100 minutes, 340-360°F at 60 psi absolute for 2 hours (vacuum bag plus back-fill)

Post Cure - 4 hours at 435-495°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, preheated at 750°F for one hour

Cleaning - Procedure II

Completely machined panels

Substrate - 17/7 PH Stainless Steel

Application - Tape

Lap-Shear Strength (Series 44-86)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

Specimen Number	Test Temperature °F	Post-Cure Time (Hours)	Failure Strength* psi	Type of Failure
44-86-1	500 (10' hold)	one	1840	Coh-Adh
44-86-2	500 (10' hold)	two	1600	" "
44-86-3	500 (10' hold)	two	1700	" "
44-86-4	500 (10' hold)	one	1720	" "
44-86-5	500 (30' hold)	four	1720	" "
44-86-6	500 (10' hold)	one	2020	" "
44-86-7	500 (10' hold)	four	1980	" "
44-86-8	500 (10' hold)	two	1820	" "
44-86-9	500 (10' hold)	four	1840	" "
44-86-10	500 (10' hold)	four	1770	" "

Composition - 10.0g. epoxy + 4.3g. hardener + 5.0g. aluminum + 2.9g. asbestos

B-stage, 293-311°F for 15 minutes (clear), add aluminum and asbestos, heat at 293 - 302°F with occasional vacuum for 40 minutes additional.

Outgassed - No

Cure Assembly, 307-351°F, 60 psi, 2½ hours (Carver Press)

Post Cure, 435-495°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750° for one hour

Cleaning - Procedure II

Completely machined panels

* Glue line thickness ranged from 0.008 to 0.010 inches

Substrate - 17/7 PH Stainless Steel

Application - Tape

Lap-Shear Strength (Series 44-87)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

Specimen Number	Test Temperature °F	Aging Time at 500°F (Hours)	Failure Strength* psi	Type of Failure
44-87-1	499 (10' hold)	160	570	Adhesive
44-87-2	499 (60' hold)	None	1500	Adhesive
44-87-3	75	None	3210	Cohesive
44-87-4	499 (10' hold)	None	2430	Cohesive
44-87-5	499 (30' hold)	None	2300	Cohesive
44-87-6	499 (10' hold)	160	772	Adhesive
44-87-7	75	None	3020	Cohesive
44-87-8	499 (10' hold)	None	2300	Cohesive
44-87-9	498 (10' hold)	None	2620	Cohesive
44-87-10	499 (10' hold)	160	550	Adhesive

Composition - 10.0g. epoxy + 4.3g. hardener + 5.0g. aluminum + 2.9g. asbestos

B-stage - Blend epoxy, aluminum and asbestos for 15 minutes at 293-311°F with vacuum, add hardener, heat at 293-311°F with vacuum for 55 minutes additional.

Outgassed - No

Cure Assembly, 307-351°F, 60 psi, 2 ½ hours (Carver Press)

Post Cure - one hour, 435-495°F, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F for one hour

Cleaning - Procedure II

Completely machined panels

* Glue line thickness ranged from 0.008 to 0.010 inches

Substrate - 17/7 PH Stainless Steel

Application - Tape

Lap-Shear Strength (Series 44-88)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum Powder
29 phr Asbestos Powder

Specimen Number	Test Temperature °F	Aging Time at 500°F (Hours)	Failure Strength psi	Degree of Cohesive Failure
44-88-1	501 (10' hold)	0	1780	85%
44-88-2	502 (120' hold)	0	2230	85%
44-88-3	500 (10' hold)	2	2140	100%
44-88-4	500 (10' hold)	4	2420	80%
44-88-5	504 (60' hold)	0	2140	90%
44-88-6	500 (10' hold)	0	1810	85%
44-88-7	504 (10' hold)	8	2180	50%
44-88-8	499 (10' hold)	16	1180	20%
44-88-9	498 (10' hold)	40	860	20%
44-88-10	500 (10' hold)	66	800	10%

Composition - 9.98g. epoxy + 4.30g. hardener + 5.03 aluminum + 2.88g. asbestos

B-stage, 273-289°F for 55 minutes with continuous outgassing

Outgassed - No

Cure Assembly - 340-360°F, 60 psi, Carver Press, 2½ hours

Post Cure - 1 hour at 435-493°F gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F for one hour

Cleaning - Procedure II

Completely machined specimens

Substrate - 17/7 PH Stainless Steel

Application - Tape

Lap-Shear Strength (Series 44-89)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
39 phr Asbestos

Specimen Number	Test Temperature °F	Aging Time at 500°F (Hours)	Failure Strength psi	Degree of Cohesive Failure
44-89-1	500 (10' hold)	0	1660	90%
44-89-2	499 (10' hold)	120	740	5%
44-89-3	500 (10' hold)	2	2080	100%
44-89-4	500 (10' hold)	4	2020	50%
44-89-5	500 (10' hold)	120	740	5%
44-89-6	501 (30' hold)	0	1820	90%
44-89-7	500 (10' hold)	8	2060	30%
44-89-8	499 (10' hold)	16	1420	20%
44-89-9	499 (10' hold)	40	900	20%
44-89-10	500 (10' hold)	66	830	10%

See Series 44-88 for conditions of assembly, cure and post-cure.

Lap-Shear Strength (Series 44-90)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

Specimen Number	Test Temperature °F	Post Cure Time, Hours	Failure Strength psi	Type of Failure
44-90-1	500	one	2080	Cohesive
44-90-2	500	one	2180	Cohesive
44-90-3	500	one	2100	Cohesive
44-90-4	500	one	2220	Cohesive
44-90-5	500	one	1910	Cohesive
44-90-6	500	four	2100	Cohesive
44-90-7	500	four	2100	Cohesive
44-90-8	500	four	2180	Cohesive
44-90-9	500	four	2340	Cohesive
44-90-10	500	four	2220	Cohesive

Composition - 10.0g. epoxy + 4.39 hardener + 5.0g. aluminum +2.9g. asbestos

B-stage, 293-311°F for 15 min (resin + aluminum + asbestos) add hardener, stir under vacuum for 55 min at 293-311°F.

Outgassed - No

Cure Assembly, 307-351°F, 60 psi, 2½ hrs (Carver Press)

Post Cure - 435-495°F gravity convection, time as shown

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F for one hour

Cleaning - Procedure II

Individual Specimens

Glue line ranged from 0.005 to 0.020 inches

Substrate: 17/7 PH Stainless Steel

Application - Tape

Lap-Shear Strength (Series 44-91)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

Specimen Number	Test Temperature °F	B-stage Time (min)	Failure Strength psi	Type Failure
44-91-1	500	55	2180	Cohesive
44-91-2	500	55	2490	Cohesive
44-91-3	500	55	2340	Cohesive
44-91-4	500	55	2080	Cohesive
44-91-5	500	55	1980	Cohesive
44-91-6	500	70	2320	Cohesive
44-91-7	500	70	1940	Cohesive
44-91-8	500	70	1750	Cohesive
44-91-9	500	70	1920	Cohesive
44-91-10	500	70	2300	Cohesive

Composition - 60.0g. epoxy + 25.8g. hardener + 17.4g. asbestos + 30.0g. aluminum

B-stage, for specimens 1-5 hear at 290-311°F for 55 min with continuous stirring under vacuum
for specimens 6-10 " " " " " 70 min " " " " "

Outgassed - No

Cure Assembly - 307-351°F, 60 psi, 2½ hrs, Carver Press

Post Cure - 1 hr at 435-465°F, gravity convection oven

Carrier - 112 heat-cleaned glass cloth, pretreated at 750°F for one hour

Cleaning - Procedure II

Individual Specimens

* Glue line ranged from 0.005-0.020 inches

Substrate: 17/7 PH Stainless Steel

Application - Tape

Series 44-92

Composition - 100g. epoxy + 43.0g. hardener + 50g. aluminum + 29g. asbestos

B-stote - Blend epoxy, aluminum and asbestos for 10 minutes at 293-311°F with vacuum, add hardener, heat at 293-311°F with vacuum for 55 minutes additional

Outgassed - No

Cure Assembly, 307-351°F, 60 psi 2½ hours, (Carver Press)

Post Cure, 435-465°F, one hour, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F for one hour

Cleaning - Procedure II

Completely machined panels

Substrate: 17/7 PH Stainless Steel

Application - Tape

Series 44-92 (continued)

Lap Shear Strength at - 320°F

Post-Cured

Specimen No.	Failure Strength psi	Type of Failure	Day of Assembly
49	4040	10% cohesive	5
24	3980	"	3
18	2940	"	2
7	2880	"	1
36	2300	"	4

Non Post-Cured

1	2940	10% cohesive	1
12	2900	"	2
55	2700	"	6
43	2500	"	5
30	1300	"	3

Series 44-92 (continued)

Lap Shear Strength at - 65°F

Post - Cured

Specimen No.	Failure Strength psi	Type of Failure	Day of Assembly
25	2480	25% cohesive	3
19	3300	"	2
8	3220	"	1
50	3040	"	5
37	Broke during assembly		

Non Post-Cured

13	2840	25% cohesive	2
44	2760	"	5
2	2700	"	1
56	2080	"	6
31	2040	"	4

Series 44-92 (continued)

Lap Shear Strength at 75°F

Post-Cured

Specimen No.	Failure Strength psi	Type of Failure	Day of Assembly
51	3060	85% cohesive	6
20	2920	"	2
19	2860	"	2
26	2740	"	3
38	Broke during assembly		

Non Post-Cured

3	2960	85% cohesive	1
14	2940	"	2
57	2800	"	6
32	2600	"	4
45	2500	"	5

Series 44-92 (continued)

Lap Shear Strength at 350°F

Post-Cured

Specimen No.	Failure Strength psi	Type of Failure	Day of Assembly
10	3100	55% cohesive	1
21	2940	75% "	3
39	2860	55% - "	4
27	2830	75% "	3
52	2800	75% "	6

Non Post-Cured

4*	3300	80% cohesive	1
15	2460	"	2
58	2040	"	6
33	1920	"	4
46	980	"	5

* 12 minutes to equilibrate as compared to 5 for others,

Series 44-92 (continued)

Lap Shear Strength at 450°F

Post-Cured

Specimen No.	Failure Strength psi	Type of Failure	Day of Assembly
11	2960	80% cohesive	2
22	2800	"	3
53	2630	"	6
40	2600	"	4
28	2380	"	3

Non Post-Cured

5*	2860	80% cohesive	1
59	860	"	6
34	820	"	4
16	820	"	2
47	680	"	5

* Longer equilibration time (12 minutes versus 8 minutes for others) before 10 minute hold

Series 44-92 (continued)

Lap Shear Strength at 500°F

Post-Cured

Specimen No.	Failure Strength psi	Type of Failure	Day of Assembly
6	2460 (60' hold)	80% cohesive	1
17	2230	80% "	2
41	2010	50% "	5
48	1860	50% "	5
35	1810	50% "	4
54	1740	50% "	6
29	1660 (60' hold)	25% "	3
23	1570	50% "	3
42	1380	50% "	5
60	1320	50% "	6

Lap-Shear Strength (Series 44-93)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum
29 phr Asbestos

Specimen Number	Test Temperature °F	Aging Time Hours (500°F)	Failure Strength* psi	Type of Failure
44-93-1	500	2	1500	50% Cohesive
44-93-2	500	4	1300	50% Cohesive
44-93-3	500	8	760	25% Cohesive
44-93-4	500	16	600	10% Cohesive
44-93-5	500	30	560	10% Cohesive

Composition and B-stage - Impregnated cloth as for Series 44-92

Outgassed - No

Cure Assembly, 307-351°F, 60 psi, 2 ½ hours (Carver Press)

Post Cure, 435-465°F, one hour, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750°F for one hour

Cleaning - Procedure I

Completely machined panels

* Glue line thickness ranged from 0.008 to 0.010 inches

Substrate: 17/7 PH Stainless Steel

Application - Tape

Lap-Shear Strength (Series 44-94)

Bis(epoxybutyl)carborane - 43 phr G-50 Hardener
50 phr Aluminum

Specimen Number	Test Temperature °F	Aging Time Hours (500°F)	Failure Strength* psi	Type of Failure
44-94-1	500	16	960	10% Cohesive
44-94-2	500	30	860	10% Cohesive
44-94-3	500	2	1780	80% Cohesive
44-94-4	500	4	1500	50% Cohesive
44-94-5	500	8	1230	50% Cohesive

Composition - 10.0g. epoxy + 4.30g. hardener + 5.00g. aluminum

B-stage, Blend epoxy and aluminum for 10 minutes of 293-311°F with vacuum, add hardener, heat at 293-311°F with vacuum for 55 minutes additional

Outgassed - No

Cure Assembly, 307-351°F, 60 psi, 2½ hours (Carver Press)

Post Cure, 435-465°F, one hour, gravity convection oven

Carrier - 112 heat cleaned glass cloth, pretreated at 750° for one hour

Completely machined panels

* Glue line thickness ranged from 0.008 to 0.010 inches

Substrate: 17/7 PH Stainless Steel

Application - Tape

Cleaning - Procedure II

APPENDIX B
LAP SHEAR TEST RESULTS
OTHER BIS(EPOXYALKYL)CARBORANES

Lap-Shear Strength (Series 46-I)

Epoxybutylepoxyhexylcarborane — 39.6 phr G-50 Hardener

Specimen Number	Test Temperature °F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
46-I -1	75	~.006	3500	cohesive
46-I -2	75*	.006	2100	adhesive
46-I -3	500*	.005	110	adhesive
46-I -4	500*	.006	120	adhesive
46-I -5	75*	.006	1880	adhesive
46-I -6	75	.006	3280	cohesive
46-I -7	75*	.006	1960	adhesive
46-I -8	500*	.007	290	adhesive
46-I -9	75	.005	3080	adh-coh
46-I -10	500*	.006	350	adhesive

Composition - 3.13 g. epoxy and 1.25 g. hardener

B-stage, 293 - 302°F for 110 minutes and 60 minutes additional at 312 - 338°F under vacuum

Outgassed - 60 minutes at 248°F

Cure Assembly, 305 - 345°F, 60 psi, 2 1/2 hours, Carver Press

* Post Cure - 4 hours at 500°F

Carrier - 112 heat cleaned glass cloth

Substrate - 17-7 PH stainless steel

Cleaning - Procedure I

Application - Hot melt

Individual specimens

Lap-Shear Strength (Series 55-I)

Bis(epoxypentyl)carborane - 39.8 phr G- Hardener

Specimen Number	Test Temperature ^{0}F	Glue Line Thickness (in)	Failure Strength psi	Type of Failure
55-I-I	75	.004	3650	cohesive
55-I-2	75*	.006	2080	adhesive
55-I-3	500*	.006	380	adhesive
55-I-4	500*	.006	350	adhesive
55-I-5	75*	.007	2020	adhesive
55-I-6	75	.004	3660	coh(adh)
55-I-7	75*	.007	1800	adhesive
55-I-8	500*	.007	730	adhesive
55-I-9	75	.004	3800	coh(adh)
55-I-10	500*	.006	1360	adh(coh)

Composition - 3.02 g. epoxy and 1.20 g. hardener

B-stage, 278 - 298^{0}F for 75 minutes followed by 70 minutes additional at 311-320^{0}F under vacuum.

Outgassed - 60 minutes at 248^{0}F

Cure Assembly, 305 - 345^{0}F, 60 psi, 2 1/2 hours, Carver Press

*Post Cure - 64 hours at 400^{0}F

Carrier - 112 heat cleaned glass cloth

Substrate - 17-7 PH stainless steel

Cleaning - Procedure I

Application - Hot melt

Individual specimens