WADC Technical Report 53-126
Part 4

Elevated Temperature Resistant Modified Epoxide Resin Adhesives for Metals

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Materials Laboratory
Wright Air Development Center
Wright-Patterson Air Force Base, Ohio

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December 1, 1955 - November 30, 1956

SHELL

SHELL DEVELOPMENT COMPANY
EMERYVILLE, CALIFORNIA

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ELEVATED TEMPERATURE RESISTANT MODIFIED EPOXIDE RESIN ADHESIVES FOR METALS

Marguerite Naps
Shell Development Company

November, 1956

WRIGHT AIR DEVELOPMENT CENTER

NOTICES

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Contract No. AF 33(616)-3347
Project No. (7340)

Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared by Shell Development Company under USAF Contract No. AF 33(616)-3347. This contract was initiated under Project No. 7340, "Rubber, Plastics, and Composite Materials", Task No. 13401, "Structural Adhesives". The work was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. T. Martin acting as project engineer.

This report covers work conducted from December 1, 1955 through November 30, 1956.

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HIGH TEMPERATURE METAL TO METAL ADHESIVES

Introduction

This report summarizes the preparation and evaluation of heat resistant adhesives based on Experimental Resin X-131 and other experimental resins.

The principal task of this project was the development of a resinous adhesive which is useful for bonding corrosion resistant metals for service at 500°F.

Elevated temperature adhesives based on Experimental Resin X-131 (previously designated EPON X-15100) were investigated briefly under Contract AF 33(616)-2430 and described in WADC Technical Report 53-126, Part 3 (September, 1955). The adhesives showed promising performance at temperatures up to 500°F for bonding stainless steel. Further development work was undertaken under the current WADC Contract AF 33(616)-3347. The studies were directed primarily to techniques for improving the adhesion to metal surfaces, the extensibility and toughness of the resin, the thermal stability on long time aging, and the reproducibility of adhesive performance.

Exploratory investigations were also made of the utility of five new solid experimental resins and five new liquid experimental resins for the formulation of heat resistant adhesives for stainless steel. These resins are characterized, in general, by greater functionality compared with that of X-131 and commercial EPON resins.

Adhesives Based on Experimental Resin X-131 With No Modifying Resins

Dicyandiamide Cure

Experimental Resin X-131 cured with dicyandiamide and containing aluminum dust filler appeared to be the most promising heat resistant adhesive developed under Contract AF 33(616)-2430 [see WADC Technical Report 53-126, Part 3 (September, 1955)]. The adhesive was used in the form of a supported tape with glass fabric 112-Volan A as the carrier. An acetone solution of X-131 resin (60% solids) was blended with the curing agent dissolved in boiling solvent at a 1/2/3 weight ratio of dicyandiamide/water/acetone. The homogeneous solution was then stirred with finely divided aluminum dust (MD105). After concentration to about 85% solids by stirring and mild heating, the solvent dispersion was applied to the glass fabric with simple doctor knives. The impregnated fabric was dried for 15 minutes at 200°F to remove solvents.

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Maximum adhesive bond strength and heat resistance occurred with a loading of 100 phr[a] aluminum dust filler and 6 phr dicyandiamide (Formulation No. 1145). The adhesive was quite brittle, however, and the reproducibility of bond performance was poor. Adhesive shear strength for bonds to stainless steel was about 2000 psi at room temperature, 750 to 1000 psi at 500°F, and 600 psi after 200 hours at 500°F. The test results for fourteen batches of supported adhesive tape (previous and current work) follow:

Surface Treatment[d]	Tensile Shear Strength,[a] psi at 77°F Range	at 77°F Avg	at 500°F Range	at 500°F Avg	at 500°F After 200 hr at 500°F Range	at 500°F After 200 hr at 500°F Avg
Method A[b]	1605-2205	2010	420-1050	745	330-1035	590
Method B[c]	1650-2595	2195	710-1460	1080	300-905	620

a) Bonds to type 301 steel cured one-half hour at 330°F.
b) Used for 12 batches of adhesive tape in 14 tests.
c) Used for 6 batches of adhesive tape in 7 tests.
d) Refer to the section on metal treatment for a description of the methods used.

The effect of test temperature on the shear strength of X-131/dicyandiamide bonds to half-hard type 301 steel is shown in Figure 1 and Table 1. Illustrative data follow:

Test Temperature	Tensile Shear Strength, psi
-70°F	2525
77°F	2200
500°F	420
600°F	60

Deterioration in adhesive strength upon aging at 500°F was quite rapid. Loss in bond strength occurred on relatively short exposure periods of 3 to 48 hours. After longer periods of 168 to 224 hours, the adhesive strength increased to values about 70% of the original strength at 500°F. The effect of exposure time at 500°F upon the shear strength of bonds from Formulation No. 1145 is shown graphically in Figure 2. The experimental data are summarized in Table 2. Typical data follow:

a) In conformance with the practice used in all previous reports on heat resistant adhesive formulations, the abbreviation "phr" is used to designate the concentration of all types of ingredients as parts by weight per hundred parts of total polyepoxide resin.

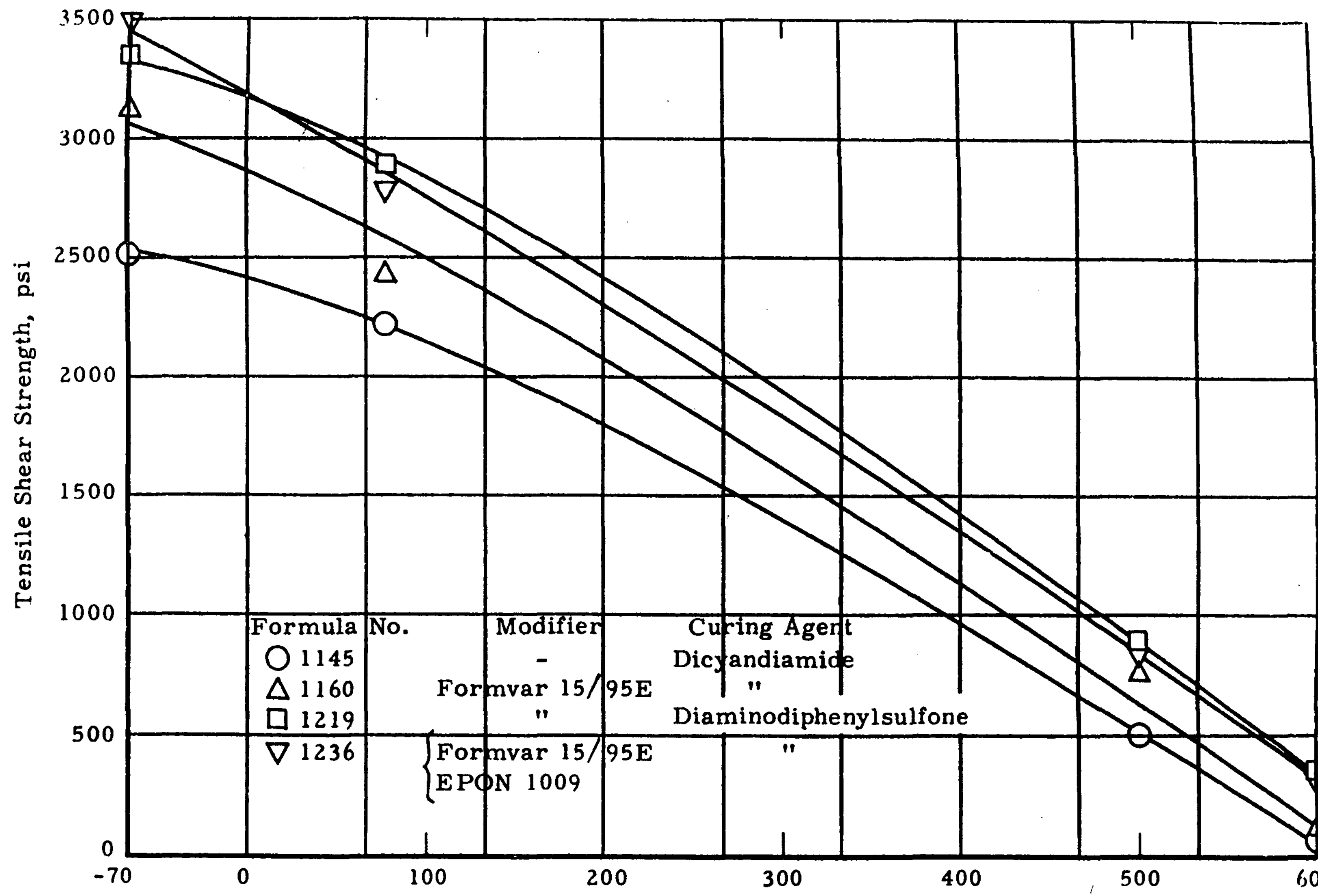

Figure 1. EFFECT OF TEST TEMPERATURE ON THE SHEAR STRENGTH OF X-131 ADHESIVE BONDS TO 301 STEEL

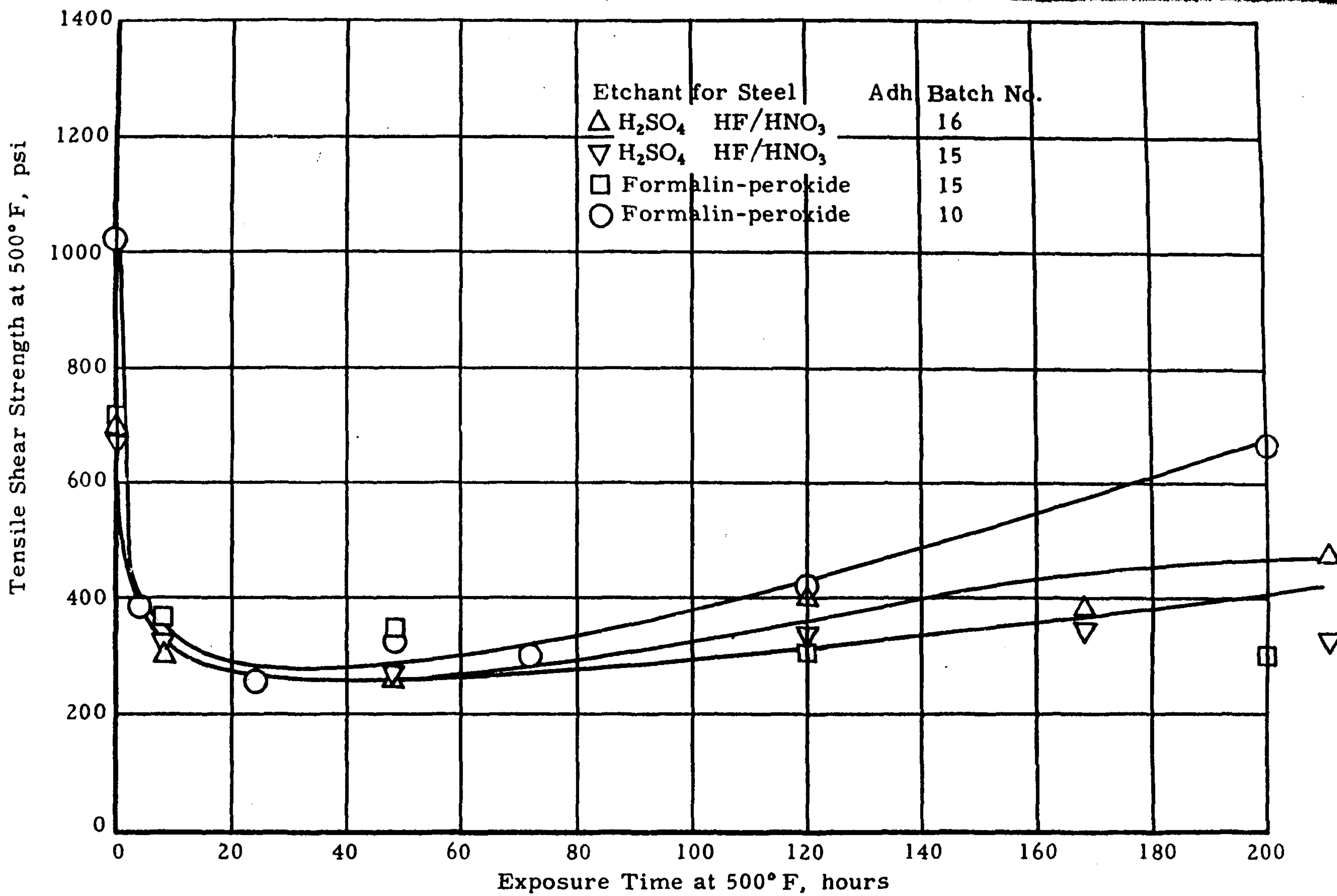

Figure 2. EFFECT OF EXPOSURE AT 500° F ON THE 500° F SHEAR STRENGTH OF X-131/DICYANDIAMIDE BONDS TO 301 STEEL

Formulation No. 1145 (by wt): 100 X-131 +100 aluminum dust + 6 dicyandiamide
Cure: one-half hour at 330° F

Exposure Time at 500°F, hr	Tensile Shear Strength, psi
0	790
8	350
48	260
168	370
200	525

Further development work and formulation studies of the X-131/dicyandiamide/aluminum dust adhesive were undertaken with the hope of improving 1) initial shear strength values at room temperature and 500°F, 2) heat resistance to aging at 500°F, and 3) reproducibility of bond performance. No significant improvement in shear strength or reproducibility was observed in the following experiments:

1. Postcuring X-131/dicyandiamide adhesive bonds for one to three hours at 400°F improved adhesive shear strength slightly at room temperature, 500°F and at 500°F after 200 hours' aging at 500°F (Table 3).

2. Postcuring X-131/dicyandiamide adhesive bonds for one to three hours at 500°F reduced adhesive shear strength at the three test conditions (Table 3).

3. Increasing the drying period of the supported adhesive tape from 15 minutes to two hours at 200°F increased the initial strength at room temperature slightly and the 500°F strength about 30%. Bond strength after 200 hours' exposure at 500°F was not altered appreciably, however (Table 4).

4. No significant change in adhesive shear strength was observed as the dicyandiamide concentration was increased from 6 to 14 phr (Table 5).

5. No significant change in adhesive shear strength was observed as the aluminum dust filler loading was increased from 75 to 200 phr with 6 phr dicyandiamide as the curing agent (Table 6).

6. Copper 8-quinolinolate (0.5 to 2.0 phr) had no apparent effect on the shear strength or on the heat resistance of X-131/dicyandiamide adhesives (Table 7).

Evaluation of Various Curing Agents

Four new compounds, benzimidazolyl guanidine, 2,6-diaminopyridine (Curing Agent E), 4,4',4"-triaminotriphenylmethane and mixed isomers of diaminodiphenylsulfone, were evaluated as curing agents for X-131 adhesives in comparison with the performance of dicyandiamide. All of these compounds are more soluble than dicyandiamide and can be handled more readily in acetone solutions of X-131 resin. The shelf life of the adhesive solutions and tapes with each of these compounds except diaminodiphenylsulfone was very limited, however. The adhesive bonds and shear strength test results for 100/100 X-131/aluminum dust tapes are summarized in Tables 8 and 9 (Curing Agent E), Table 10 (benzimidazolyl guanidine), Table 11 (diaminodiphenylsulfone) and Table 12 (4,4',4"-triaminotriphenylmethane).

Mediocre bond strength was obtained with all of the curing agents except diaminodiphenylsulfone. This compound was slightly superior to dicyandiamide in the development of strength at 500°F and markedly superior in strength retention on 500°F exposure. Figure 3 shows the effect of aging at 500°F on the 500°F shear strength of X-131 bonds cured with dicyandiamide and diaminodiphenylsulfone. Illustrative test data are given below for X-131 adhesive bonds cured one-half hour at 310 to 330°F with the new curing agents.

Curing Agent, phr	Tensile Shear Strength, psi		
	at 77°F	at 500°F	at 500°F After 200 hr at 500°F
6E	1875	1200	1000
13E	1665	425	285
10 benzimidazolyl guanidine	1175	530	595
20 " "	1425	110	415
23.5 4,4',4"-triaminotriphenylmethane	865	525	375
30 diaminodiphenylsulfone	2300	1090	885
6 dicyandiamide	2100	850	600

Adhesives Based on Experimental Resin X-131 With EPON 1009 and EPON 834

X-131/EPON 1009 Adhesive Tapes

EPON 1009 (molecular weight 4000) was used in combination with Experimental Resin X-131 with the hope of reducing the brittleness of X-131 adhesives. Supported adhesive tapes with glass fabric 112-Volan A were prepared with 90/10, 80/20 and 70/30 X-131/EPON 1009 resin ratios as follows:

1. Prepare a solution of X-131 in acetone (60% solids).

2. Dissolve the curing agent, dicyandiamide or diaminodiphenylsulfone, in the resin solution to form a homogeneous blend.

   a) Dicyandiamide must be added in boiling solvent at a 1/2/3 dicyandiamide/water/acetone weight ratio to achieve compatability.

   b) Diaminodiphenylsulfone dissolves in the X-131 resin solution. Heating must be avoided to prevent premature reaction.

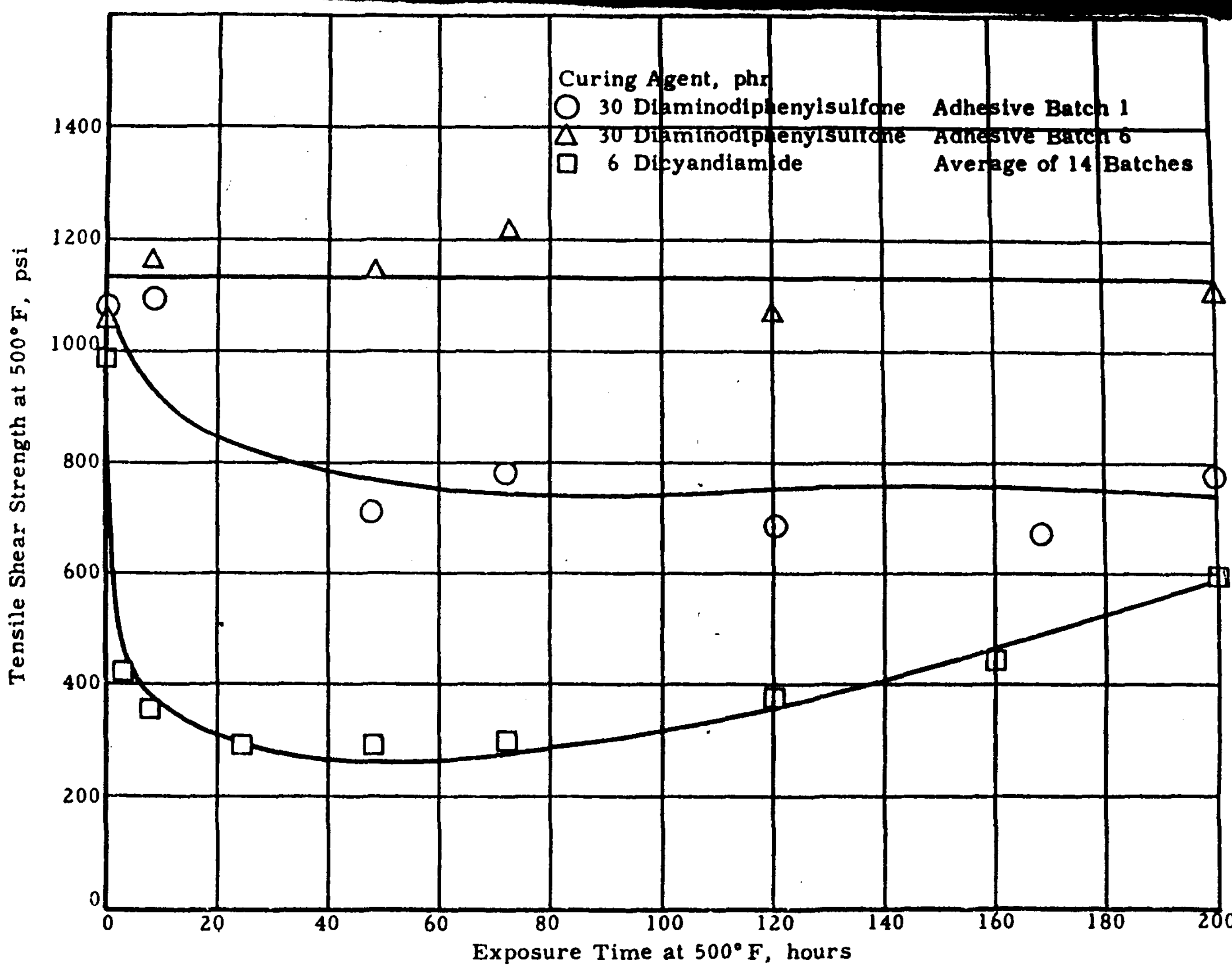

Figure 3. EFFECT OF EXPOSURE AT 500° F ON THE 500° F SHEAR STRENGTH OF BONDS TO 301 STEEL FROM X-131 ADHESIVES CURED WITH DICYANDIAMIDE AND DIAMINODIPHENYLSULFONE

Constant (by wt): 100 phr aluminum dust

Cure: one-half hour at 330° F

3. Add EPON 1009 solution (50% solids in acetone or methyl ethyl ketone).

4. Disperse finely divided aluminum dust (MD105) in the resin-curing agent solution.

5. Concentrate the solvent dispersion to about 80% solids by stirring at room temperature.

6. Apply the concentrated adhesive dispersion to glass fabric, 112-Volan A.

7. Dry the impregnated fabric about one hour at room temperature and 15 minutes at 200°F. Thickness of tape is 12-14 mils.

The effect of X-131/EPON 1009 resin ratio, in the range of 100/0 to 70/30, on the shear strength of adhesive bonds to type 301 steel is given in Table 13 for dicyandiamide cures and in Table 14 for diaminodiphenylsulfone cures. These data are also plotted in Figure 4. The limited test data, though somewhat erratic, indicated the following for adhesives cured with dicyandiamide or diaminodiphenylsulfone:

1. The brittleness of the X-131/EPON 1009 adhesives decreased with increasing EPON 1009 content. This behavior was reflected in the increased (20 to 30%) room temperature shear strength.

2. The shear strength at 500°F was reduced 20 to 30% by the replacement of 10 to 30 per cent of the X-131 resin with EPON 1009.

3. There was no appreciable difference in the thermal stability of adhesives with 100/0, 90/10, 80/20 and 70/30 X-131/EPON 1009 resin ratios (as judged by the 500°F shear strength data for specimens aged 200 hours at 500°F).

Typical test results for bonds to full hard type 301 steel cured with 6 phr dicyandiamide at contact pressure for one-half hour at 330°F follow:

	Tensile Shear Strength, psi		
X-131/EPON 1009, by wt	at 77°F	at 500°F	at 500°F After 200 hr at 500°F
100/0	1930	770	680
90/10	2225	755	635
80/20	2205	585	1235
70/30	2335	655	780

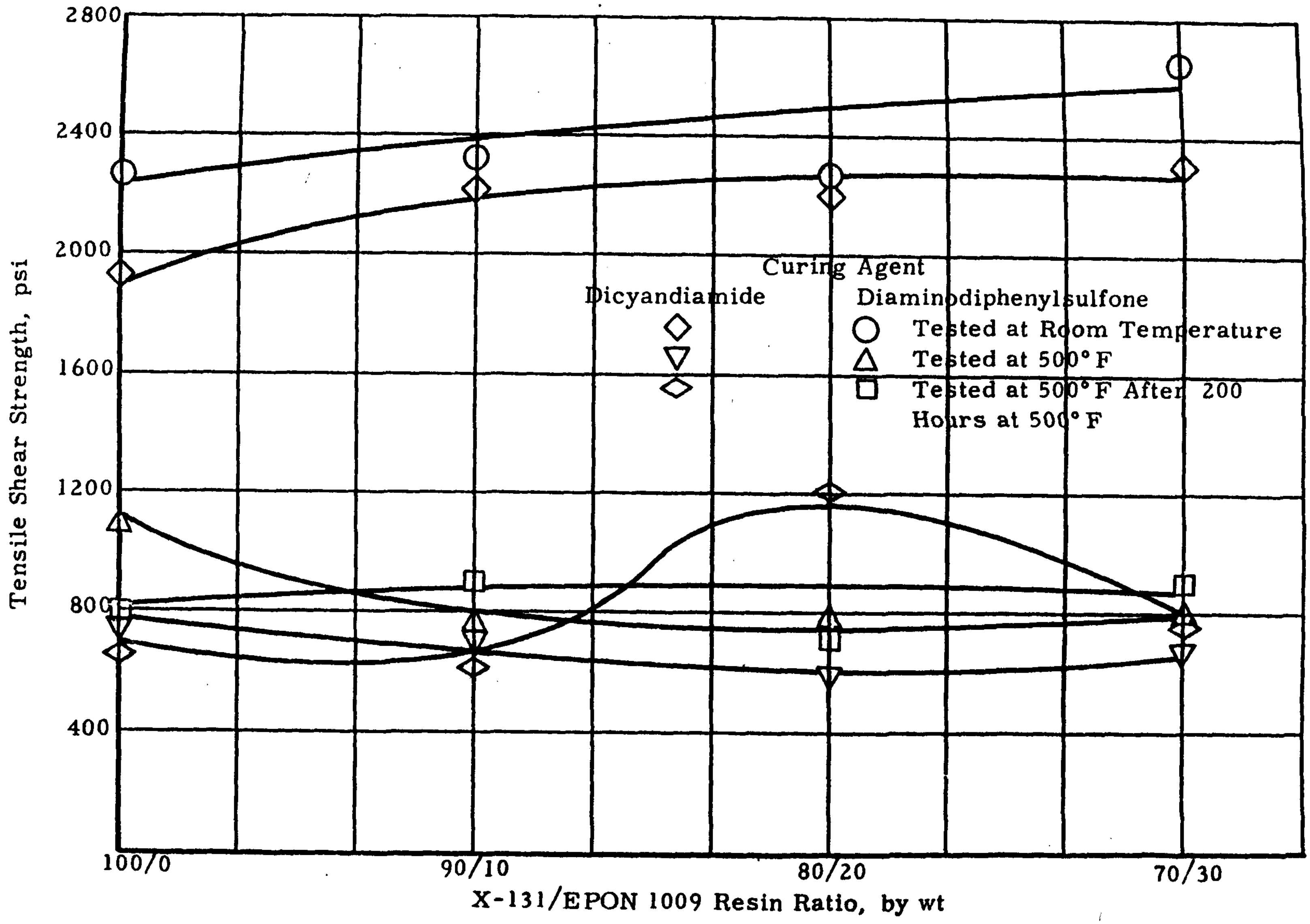

Figure 4. EFFECT OF X-131/EPON 1009 RATIO ON THE SHEAR STRENGTH OF BONDS TO 301 STEEL CURED WITH DICYANDIAMIDE AND DIAMINODIPHENYLSULFONE

Cure: one-half hour at 330° F

Illustrative data follow for bonds to half-hard type 301 steel cured at 100 psi with 30 to 21.5 phr diaminodiphenylsulfone for one-half hour at 240°F plus one-half hour at 330°F and postcured for two hours at 300°F.

X-131/EPON 1009, by wt	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F
100/0	2255	1075	780
90/10	2315	750	900
80/20	2255	775	700
70/30	2640	785	885

The effect of the drying time at 200°F applied to X-131/EPON 1009/dicyandiamide adhesive tapes on adhesive bond performance was also studied briefly (see Table 15). The initial strength at room temperature increased 25% (to 2700 psi) and the 500°F shear strength increased 80% (to 1025 psi) as the drying period was increased from 15 minutes to 120 minutes at 200°F. Shear strength at 500°F for bonds aged 200 hours at 500°F decreased (to 415 psi) progressively as the drying time at 200°F was increased.

Hot Melt Blends of X-131 and EPON 834

EPON 834, a viscous liquid epoxide resin, was used in combination with Experimental Resin X-131 (softening point 170°F) to improve the flow and wettability of the solid resin. Hot melt adhesives were prepared with X-131/EPON 834 resin ratios of 50/50, 60/40 and 70/30. Diaminodiphenylsulfone at a concentration of one amine hydrogen atom per epoxide group was the curing agent. The reinforcing filler, aluminum dust, was used at a loading of 100 parts by weight per hundred parts of total resin. Bonds to type 301 steel were assembled with glass cloth reinforcement and cured for one-half hour at 240°F plus one-half hour at 330°F at 100 psi. The test results are given in Table 16.

The X-131/EPON 834 adhesives were characterized by brittleness and mediocre heat resistance. The data follow:

X-131/EPON 834, by wt	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F
50/50	1560	375	550
60/40	1945	375	525
70/30	3215	110	445
100/0	2815	1140	830

Adhesives Based on Experimental Resin X-131 and Vinyl Resins

Experimental Resin X-131 was modified with high molecular weight vinyl resins of high softening point with the hope of increasing the extensibility of the adhesive bonds. The resins included 1) Formvar 15/95 E, polyvinyl formal, and 2) Gelva C-3-V-30, carboxylic-modified polyvinyl acetate.

X-131/Formvar 15/95 E Adhesive Tapes

Formvar 15/95 E (softening point 387°F) was dissolved in tetrahydrofuran and combined with acetone solutions of X-131 resin. Supported adhesive tapes with good bond performance were prepared with glass fabric (112-Volan A) as the carrier in the following manner:

1. Prepare a solution of X-131 resin in acetone (60% solids).
2. Dissolve the curing agent in the X-131 resin solution to form a homogeneous blend.
    a) Dicyandiamide must be added in boiling solvent at a 1/2/3 dicyandiamide/water/acetone weight ratio.
    b) Diaminodiphenylsulfone and acid-type curing agents evaluated in this work dissolve directly in the resin solutions at room temperature. Heating should be avoided to preclude premature cure.
3. Add Formvar 15/95 E solution (10% solids in tetrahydrofuran).
4. Disperse the filler (aluminum dust or asbestos fibers) in the resin-curing agent solution.
5. Concentrate the adhesive dispersion to a solids content of 58-60% by stirring at room temperature.
6. Apply the concentrated solvent dispersion of the adhesive to glass fabric (112-Volan A).
7. Dry the impregnated fabric about one hour at room temperature and 15 minutes at 200°F. Thickness of tape, 12-14 mils.

Formvar 15/95 E Concentration

The effect of the concentration of Formvar 15/95 E on the performance of X-131 adhesives was investigated with two curing agents, 1) dicyandiamide and 2) diaminodiphenylsulfone. X-131 adhesive tapes were prepared with 10, 15, 20, and 30 phr Formvar 15/95 E, 100 phr aluminum dust filler and 4 phr dicyandiamide. The addition of 10 phr Formvar 15/95 E improved the toughness and adhesive strength at room temperature with little or no sacrifice in bond strength after 200 hours' aging at 500°F. Further increases in room temperature shear strength were obtained with higher amounts (15 to 30 phr) of the vinyl resin, but the hot strength of the unaged bonds decreased rapidly. The test results are given in Table 17 and shown graphically in Figure 5. The data for bonds to full hard type 301 steel cured at contact pressure for one-half hour at 330°F and postcured three hours at 400°F follow:

Formvar 15/95 E, phr	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F
0	2305	1190	825
10	3085	975	805
15	3095	595	795
20	3710	500	1125
30	4200	375	665

The effect of X-131/Formvar 15/95 E resin ratio on adhesive bond performance was investigated for formulations cured with diaminodiphenylsulfone. This curing agent was used at the theoretical concentration (one amine hydrogen atom per epoxide group) based on the polyepoxide content of the resin blend. In two parallel series of experiments with 5/1, 3/1, 1/1 and 1/3 X-131/Formvar 15/95 E resin ratios the aluminum dust filler content was used at concentrations 1) equivalent to the polyepoxide resin content and 2) equivalent to the total resin content (polyepoxide plus vinyl resins). The complete test results are given in Table 18.

Poor elevated temperature strength at 500°F was obtained for formulations with 1/1 or 1/3 X-131/Formvar 15/95 E resin ratios. A ratio of 5/1 appeared to be about maximum for the development of any reasonable strength (640 psi) at 500°F. Formulations with 5/1 and 3/1 resin ratios also had good resistance to thermal degradation upon aging at 500°F. Poor reproducibility, which is characteristic of current X-131 adhesives, was, unfortunately, also observed for the X-131/Formvar 15/95 E blends. Figure 6 shows the effect of X-131/Formvar 15/95 E ratio (with 100 phr aluminum dust filler) on the shear strength of adhesive bonds at 1) room temperature, 2) 500°F, 3) 500°F after 8 hours at 500°F, and 4) 500°F after 200 hours at 500°F. The test results for bonds to half-hard type 301 steel cured at 100 psi with diaminodiphenylsulfone for one-half hour at 240°F plus one-half hour at 330°F follow:

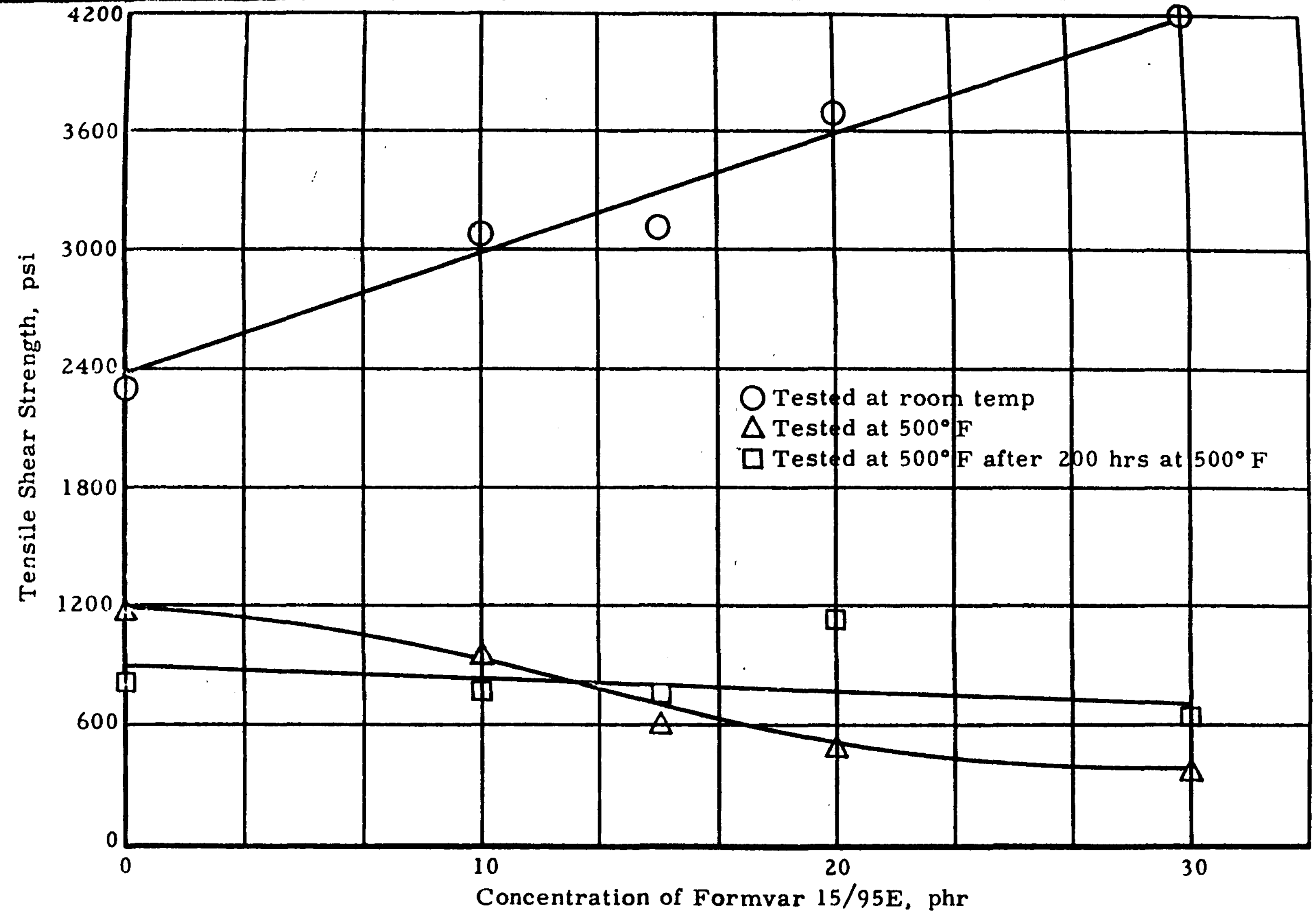

Figure 5. EFFECT OF FORMVAR 15/95E CONCENTRATION ON THE SHEAR STRENGTH OF X-131/DICYANDIAMIDE BONDS TO 301 STEEL

Cure: one-half hour at 330°F with 4 phr dicyandiamide

Postcure: 3 hours at 400°F

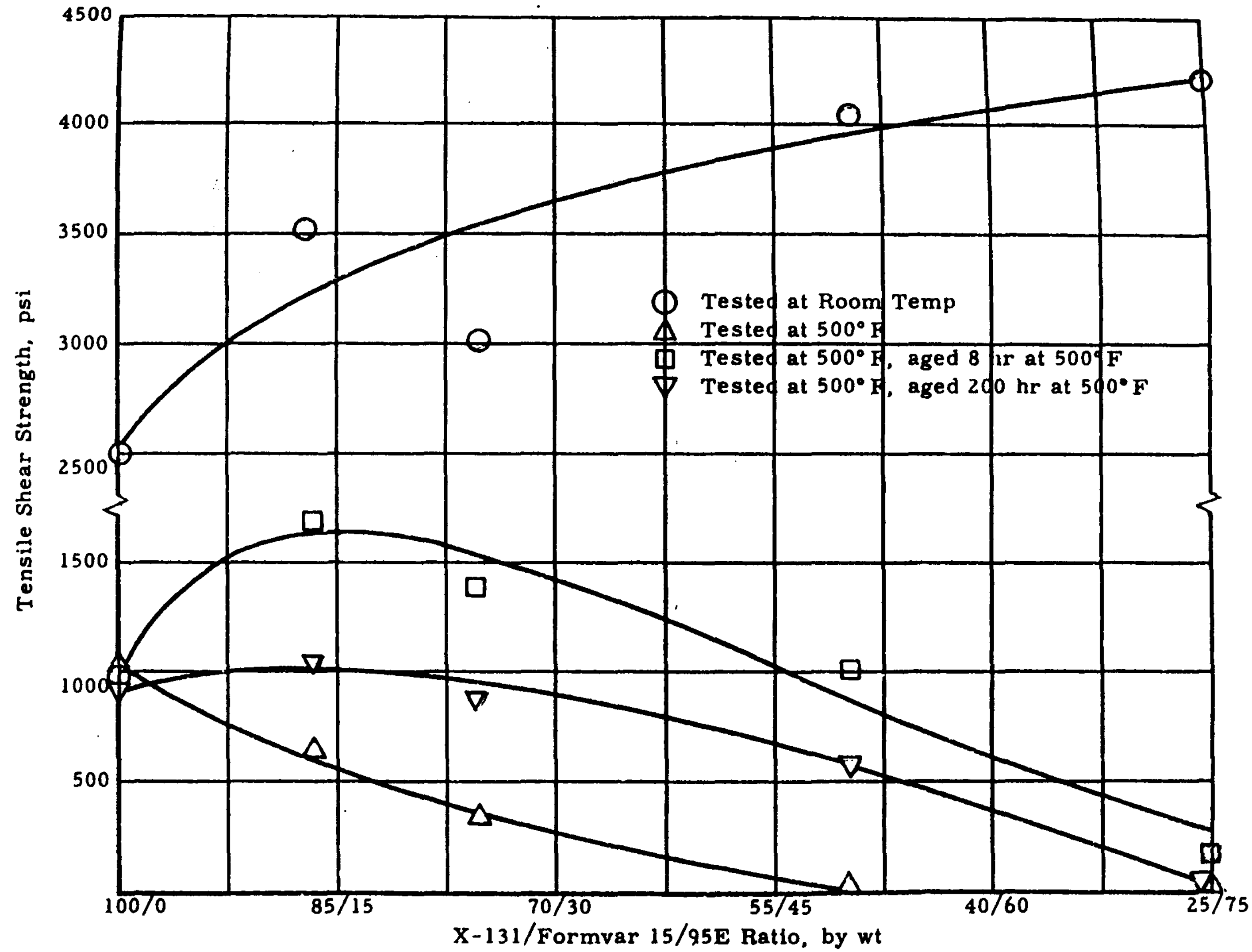

Figure 6. EFFECT OF FORMVAR 15/95E CONCENTRATION ON THE SHEAR STRENGTH OF X-131/DIAMINODIPHENYLSULFONE BONDS TO 301 STEEL

Wt Aluminum Dust Filler = Wt X-131 Resin

Cure at 100 psi: one-half hour at 240° F + one-half hour at 330° F

X-131/Formvar 15/95 E, by wt	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F
100/0	2420	1100	900
83.3/16.7	3555	640	1030
75/25	2895	345	850
50/50	4045	0	560
25/75	4210	0	0

An attempt to accelerate the cure of X-131/Formvar 15/95 E/diaminodiphenylsulfone adhesives with a boron trifluoride complex (Curing Agent BF_3-400) was unsuccessful. A reduction, rather than the desired increase in initial strength at 500°F was observed for an asbestos filled 5/1 X-131/Formvar 15/95 E adhesive which contained 1 phr BF_3-400 and the normal amount (30 phr) of diaminodiphenylsulfone (see Table 19). The adhesive shear strength at each test condition was 20 to 50% lower for asbestos-filled adhesives cured in the presence of BF_3-400.

Neutral asbestos fibers at a loading of 30 phr was equivalent to 100 phr aluminum dust in a single test of a 100/20 X-131/Formvar 15/95 E adhesive cured with diaminodiphenylsulfone (Table 20).

Bisphenol-A at a concentration of 2 to 10 phr was evaluated as a cure promoter for a 100/20 X-131/Formvar 15/95 E adhesive with curing agent diaminodiphenylsulfone. The test results are given in Table 21. Small amounts of Bisphenol-A (2 to 5 phr) appeared to have no significant effect on the initial 500°F shear strength. At higher concentrations (10 phr) a 60% reduction in hot strength was observed. Strength at 500°F after 200 hours' aging at 500°F appeared to be decreased about 30%. Pertinent data are given below for 100/20 X-131/Formvar 15/95 E bonds to half-hard type 301 steel cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F with diaminodiphenylsulfone in the presence of Bisphenol-A.

	Tensile Shear Strength, psi			
	Original		at 500°F After 500°F Aging for	
Bisphenol-A, phr	at 77°F	at 500°F	8 hr	200 hr
0	3605	1020	1505	950
5	4235	880	1285	690
10	4135	355	1080	675

Evaluation of Various Curing Agents

Diaminodiphenylsulfone appears to be the most promising curing agent for heat resistant 100/20 X-131/Formvar 15/95 E adhesive formulations. An exploratory study of other curing agent systems was undertaken in view of the slow rate of cure with the sulfone. The curing agents included 4,4',4"-triaminotriphenylmethane, melamine, boron trifluoride complexes of urea and ammonia, and BF_3-400, acid salts (magnesium perchlorate and zinc fluoborate) and hexahydrophthalic anhydride. The adhesive formulations are described in Table 12 (triaminotriphenylmethane), Table 22 (melamine), Table 23 (BF_3-400) and Table 24 (remaining compounds). Supported adhesive tapes were prepared with solvent solutions of 100/20 X-131/Formvar 15/95 E with neutral asbestos or aluminum dust fillers and 30 phr diaminodiphenylsulfone. Melamine and the acid salts were dispersed in the resin solutions; boron trifluoride complexes and hexahydrophthalic anhydride were dissolved. None of the compounds appeared to be as effective as diaminodiphenylsulfone (or dicyandiamide) for the production of heat resistant bonds. In addition. the thermal stability on long time aging at 500°F was mediocre or poor in the limited number of tests. Illustrative data for 100/20 X-131/Formvar 15/95 E adhesive bonds (cured one-half hour at 330°F) to half-hard type 301 steel follow:

Curing Agent, phr	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F
23.5 4,4',4"-triaminotriphenylmethane	1430	455	330
10 melamine	3830	245	645
3 BF_3-400	2405	515	420
3 BF_3-urea	2735	440	0
3 BF_3-ammonia	1635	50	0
3 $Mg(ClO_4)_2$	2125	420	0
9 $Zn(BF_4)_2 \cdot 7H_2O$	0	0	0
75 hexahydrophthalic anhydride	975	0	0

100/20/30 X-131/Formvar 15/95 E/Diaminodiphenylsulfone Adhesive (Formulation No. 1219)

One of the most promising heat resistant adhesive formulations was a glass fabric supported adhesive tape of the following composition (parts by wt): 100 X-131 + 20 Formvar 15/95 E + 100 aluminum dust + 30 diaminodiphenylsulfone. Bond performance was fairly reproducible in 16 tests of nine different batches of resin. Table 25 summarizes the shear strength values for bonds to type 301 steel cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F (with and without a postcure of two hours at 300-400°F). Typical data follow:

	No Postcure	Postcured 2 hr at 300-400°F
Tensile Shear Strength, psi		
At -70°F	-	3355
At room temperature	3750	2710
At 500°F, original	840	800
, after 500°F aging, 200 hr	745	850
At 600°F	-	235
Bend Strength, lb	156	110

The effect of test temperature on adhesive shear strength of Formulation No. 1219 in the range of -70 to 600°F is shown graphically in Figure 1.

Formulation No. 1219 showed good thermal stability on type 301 steel upon aging at 500°F. Figure 7 shows the effect of exposure time at 500°F on the 500°F adhesive shear strength of Formulation No. 1219 (average of four batches of adhesive tape) in comparison with the corresponding adhesive cured with dicyandiamide. Illustrative data taken from the tests results in Table 26 follow:

Exposure Time at 500°F, hr	Tensile Shear Strength at 500°F, psi Range	Avg of 4 Batches
0	640-1020	820
8	895-1690	1340
72	1155-1495	1335
200	630-1030	905

The effect of postcuring of Formulation No. 1219 bonds on the initial 500°F shear strength was studied in view of the improved strength (60% increase) of specimens tested after eight hours' aging at 500°F. Table 27 summarizes two series of experiments with oven postcures of 1) 2 to 8 hours at 300 to 500°F and 2) 2 to 32 hours at 270 to 400°F. The test results were unfortunately too erratic for interpretation. In general, however, postcures at moderate temperatures of 270 to 400°F had no consistent effect on the initial hot strength of the adhesive bonds. In all cases, however, embrittlement of the bond occurred as reflected in the decreased room temperature shear strength values. Data for bonds from adhesive batch No. 9 cured at 100 psi for one-half hour at 240°F and one-half hour at 330°F follow:

Postcure hr	Postcure °F	Tensile Shear Strength, psi at 77°F	Tensile Shear Strength, psi at 500°F
None		3630	715
16	270	3400	755
8	330	3225	835
2	400	2700	895
16	400	1860	1090

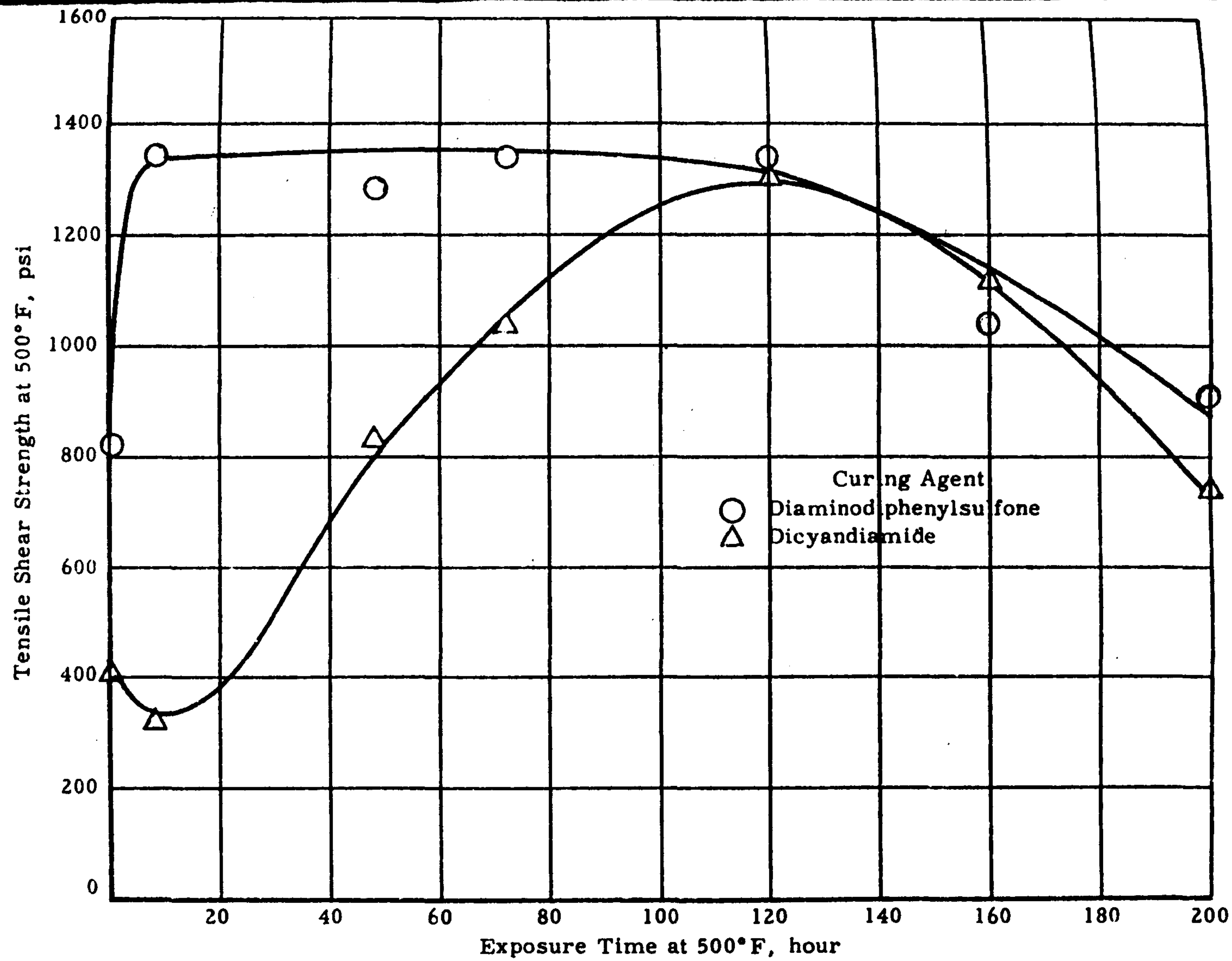

Figure 7. EFFECT OF EXPOSURE AT 500°F ON THE 500°F SHEAR STRENGTH OF 100/20 X-131/FORMVAR 15/95E BONDS TO 301 STEEL

Constant: 100 phr aluminum dust

Cure: one-half hour at 330°F

The embrittlement of the 100/20 X-131/Formvar 15/95 E adhesive on elevated temperature postcuring at 300 to 400°F was also reflected in the bend strength of the bonds. A limited number of test results (Table 28) showed a 35% decrease in bend strength for bonds postcured eight hours at 400°F. The data for bonds to type 301 steel cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F are plotted in Figure 8 and are also given below:

Postcure hr	Postcure °F	Bend Strength, lb
None		156
8	330	138
4	400	114
8	400	112

Adhesive test specimens described in this report were cut from type 301 steel panels with circular slit saws mounted on a milling machine. Several panels, however, were cut with a Do-All band saw in the investigation of postcured bonds from Formulation No. 1219. A comparison of the adhesive shear strength test results for the mill-sawed and band-sawed specimens (Table 29) indicated that the method of cutting had no appreciable effect on the initial strength at room temperature and at 500°F. The resistance of the specimens to prolonged aging at 500°F was reduced markedly, however, for bonds cut with the band saw. Thermal degradation of the adhesive was probably initiated at the edges of the specimens which were badly discolored on sawing. Shear strength at 500°F after 200 hours' exposure was 830 psi for the band-sawed bonds compared with a value of 1355 psi for corresponding mill-sawed bonds.

Gelva C-3-V-30

Gelva C-3-V-30, a high molecular weight, carboxylic modified polyvinyl acetate resin (softening point 356°F), was evaluated briefly as a flexibilizer for X-131 adhesives. It was hoped that no sacrifice in heat resistance would occur because of the possible coreaction of the resins through the carboxyl group. Supported adhesive tapes from acetone solutions of 90/10 to 70/30 X-131/Gelva C-3-V-30 containing 100 phr aluminum dust filler were cured with 1) diaminodiphenylsulfone (Table 30), 2) diaminodiphenylsulfone with a tertiary amine promoter (Table 31) and 3) dicyandiamide (Table 32).

The preliminary experiments indicated that Gelva C-3-V-30 was inferior to Formvar 15/95 E (polyvinyl formal) for the formation of thermally stable X-131 adhesives. The toughness and extensibility of adhesive bonds with 16.7 to 25 phr Gelva C-3-V-30 was improved, but strength at 500°F and resistance to aging at 500°F were reduced regardless of the curing agent used. Illustrative test results follow for bonds to 301 steel cured at 100 psi with diaminodiphenylsulfone for one-half hour at 240°F plus one-half hour at 330°F.

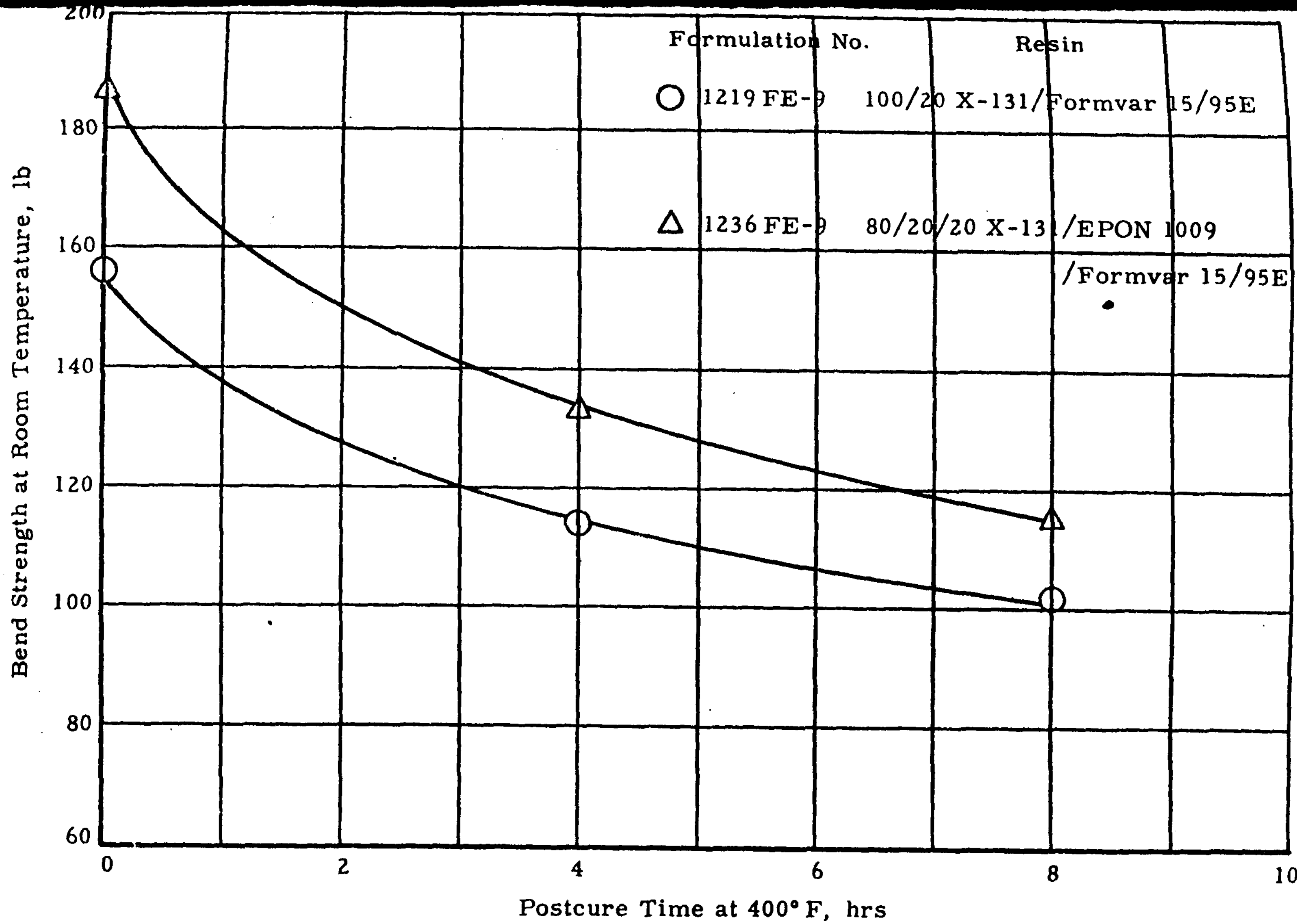

Figure 8. EFFECT OF POSTCURING AT 400° F ON THE BEND STRENGTH OF X-131/FORMVAR 15/95E ADHESIVE BONDS TO 301 STEEL

Cure at 100 psi: one-half hour at 240° F + one-half hour at 330° F with diaminodiphenylsulfone

Resin, Parts by wt	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F
100 X-131	2420	1060	1115
83.3/16.7 X-131/Gelva C-3-V-30	3340	785	320
83.3/16.7 X-131/Formvar 15/95 E	4015	1285	1095

Adhesives Based on 80/20/20 X-131/EPON 1009/Formvar 15/95 E

Good thermal stability on aging at 500°F was obtained in the initial tests of a combination of 80/20/20 X-131/EPON 1009/Formvar 15/95 E cured with 24.5 phr diaminodiphenylsulfone and containing 100 phr aluminum dust filler. The curing agent content was calculated on the basis of one amine hydrogen atom per epoxide group in the combined polyepoxide resins. An extensive evaluation was made with this formulation (No. 1236) in view of the excellent 500°F shear strength (1200 psi) of bonds to type 301 steel aged 200 hours at 500°F. Supported adhesive tapes with good bond performance were prepared with solvent dispersions of the adhesive according to the following procedure:

1. Prepare a solution of X-131 resin in acetone (60% solids content).
2. Dissolve diaminodiphenylsulfone in the X-131 solution at room temperature.
3. Add EPON 1009 solution (50% solids in acetone).
4. Add Formvar 15/95 E solution (10% solids in tetrahydrofuran).
5. Disperse aluminum dust (MD105) in the resin-curing agent solution.
6. Concentrate the adhesive dispersion to 58-60% by stirring at room temperature.
7. Apply the concentrated solvent dispersion of the adhesive to glass fabric (112-Volan A).
8. Dry the impregnated fabric about one hour at room temperature and 15 min at 200°F. Thickness of tape is 12-14 mils.

Bonds to Type 301 Steel

Adhesive bonds with Formulation No. 1236 were cured in a press at 100 psi for one-half hour at 240°F plus one-half hour at 330°F or only for one-half hour at 330°F. Some of the bonds were also postcured (oven) for periods of 2 to 4 hours at 300 to 400°F. The test results for 27 batches of adhesive tape are given in Tables 33 and 34. In general, postcuring decreased shear strength at room temperature (4000 to 2700 psi) and increased the hot strength from a relatively low value of about 530 psi to 900 psi at 500°F.

The strength retention of adhesive bonds from Formulation No. 1236 in a temperature range of -70 to 600°F is shown graphically in Figure 1. A sharp decrease in shear strength occurred as the test temperature was increased from 77 to 500°F. The data are summarized in Tables 33 and 34 and in Table 35 (600°F tests). Typical data for bonds cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F follow:

Test Temperature	Tensile Shear Strength, psi No Postcure	Tensile Shear Strength, psi Postcure 2-4 hr at 400°F
-70°F	3680	3480
77°F	3300-4000	2400-3400
500°F	200-645	625-870
600°F	330	200

The effect of elevated temperature aging of the unstressed adhesive bonds at 500°F and 600°F on tensile shear strength is given in Table 36. Some of these tests results are also plotted in Figures 9 and 10 (500°F exposure) and Figure 11 (600°F exposure). Illustrative data follow:

Aging Condition	Tensile Shear Strength, psi at 500°F	Tensile Shear Strength, psi at 600°F
8 hr at 500°F	1280	635
200 hr at 500°F	1360	545
400 hr at 500°F	520-1090	-
8 hr at 600°F	-	455
120 hr at 600°F	-	740

In a very limited study of long time load resistance at 500°F, adhesive bond failure occurred after 485 and 155 hours' aging at stresses of 400 and 500 psi, respectively. No evidence of creep was observed. Long time load test data are given in Table 37.

Reproducibility of the bond performance of adhesive Formulation No. 1236 was disappointing. Differences in various lots of X-131 resin appeared to be one of the factors which affected the hot strength and thermal stability of bonds at 500°F. No correlation between resin properties and bond performance could be made from the available data, however. The properties of various batches of X-131 resin are given in Table 38. A comparison of the performance of three batches of X-131 resin is given in Figure 12 (bonds without postcure) and Figure 13 (bonds postcured 2 to 4 hours at 400°F). The complete test results for Formulation No. 1236

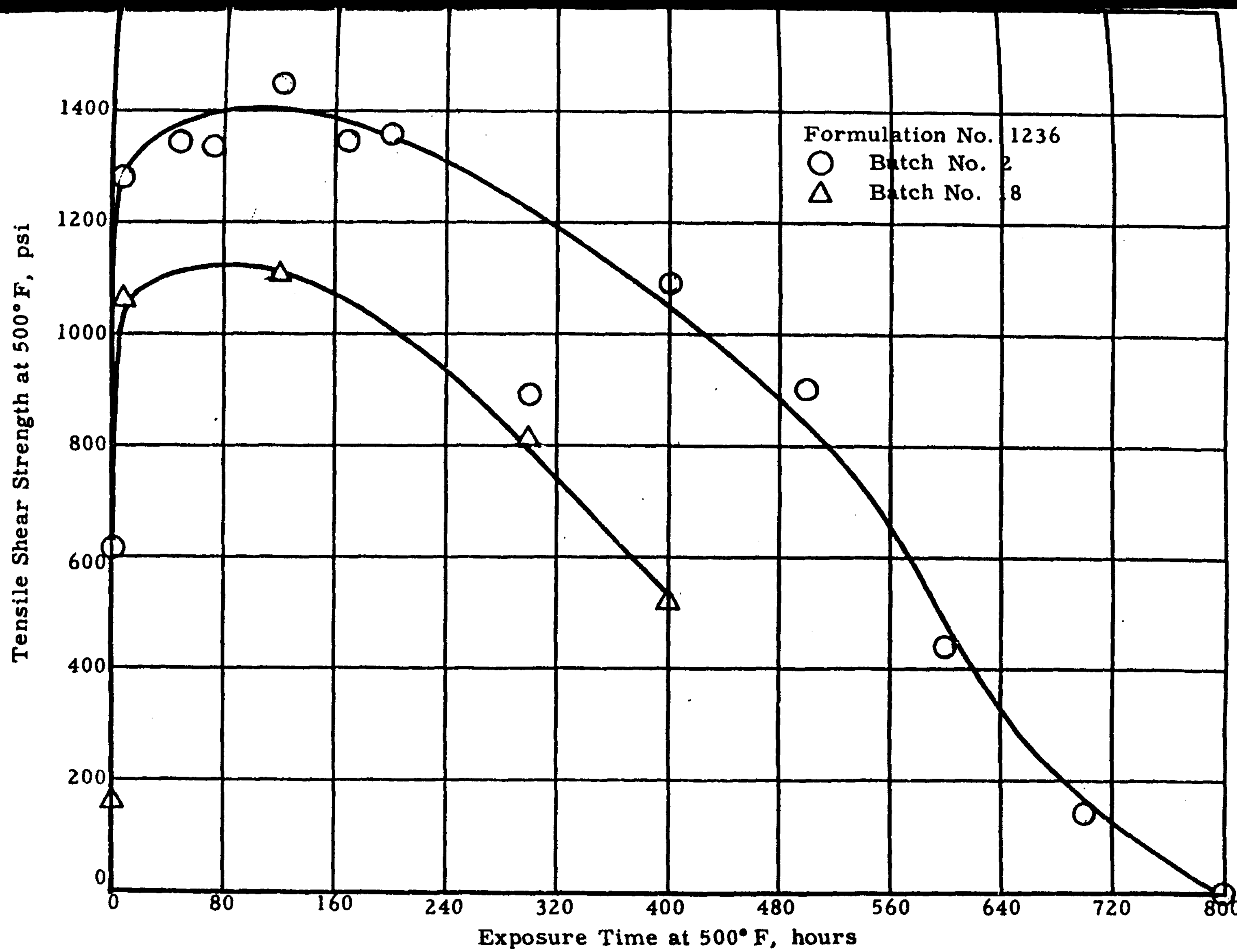

Figure 9. EFFECT OF AGING AT 500° F ON THE 500° F SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95E BONDS TO 301 STEEL

Cure at 100 psi: one-half hour at 240° F + one-half hour at 330° F

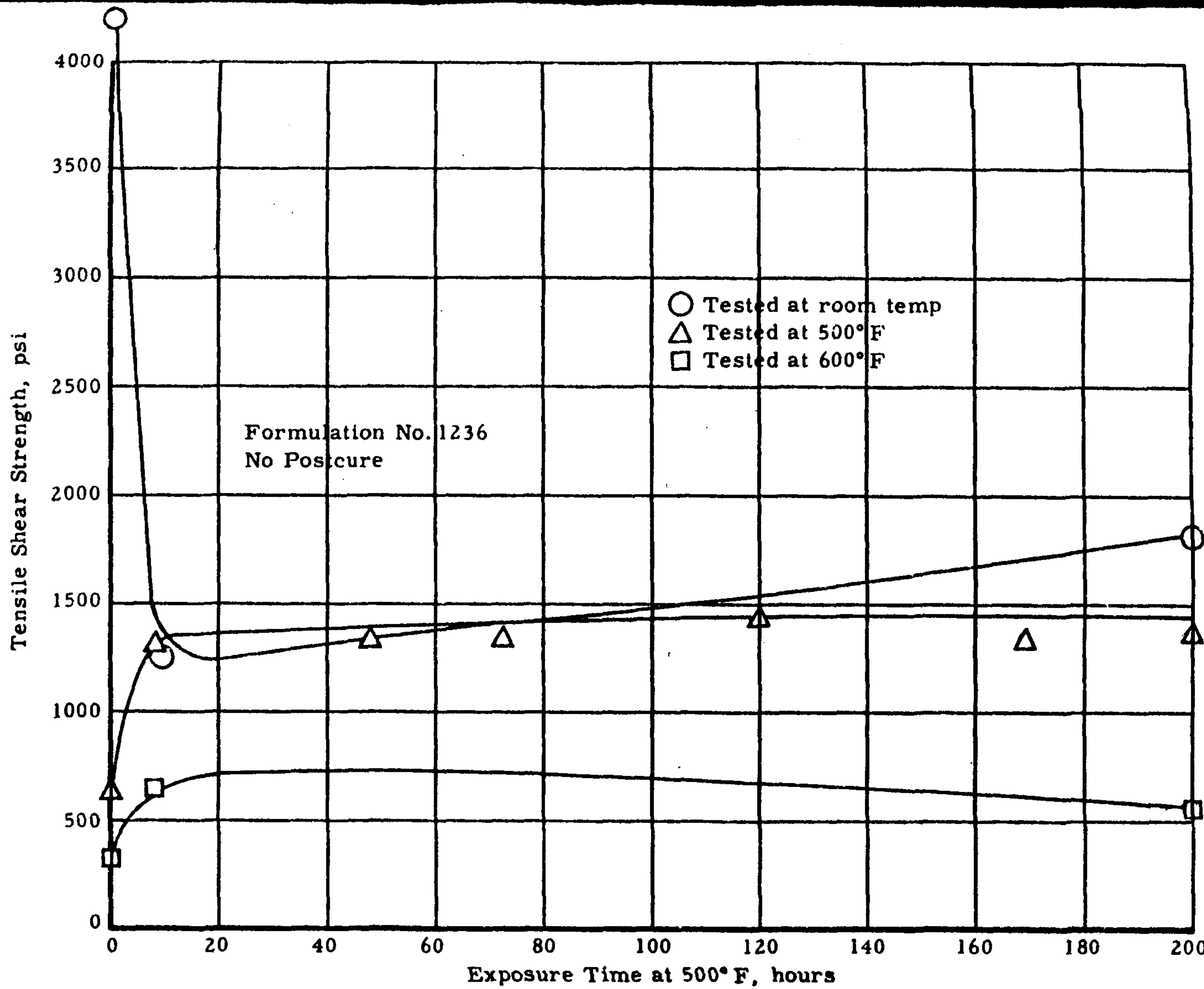

Figure 10. EFFECT OF AGING AT 500° F ON THE SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95E ADHESIVE BONDS TO 301 STEEL

Formulation, phr: 100 aluminum dust + 24.5 diaminodiphenylsulfone

Cure at 100 psi: one-half hour at 240° F + one-half hour at 330° F

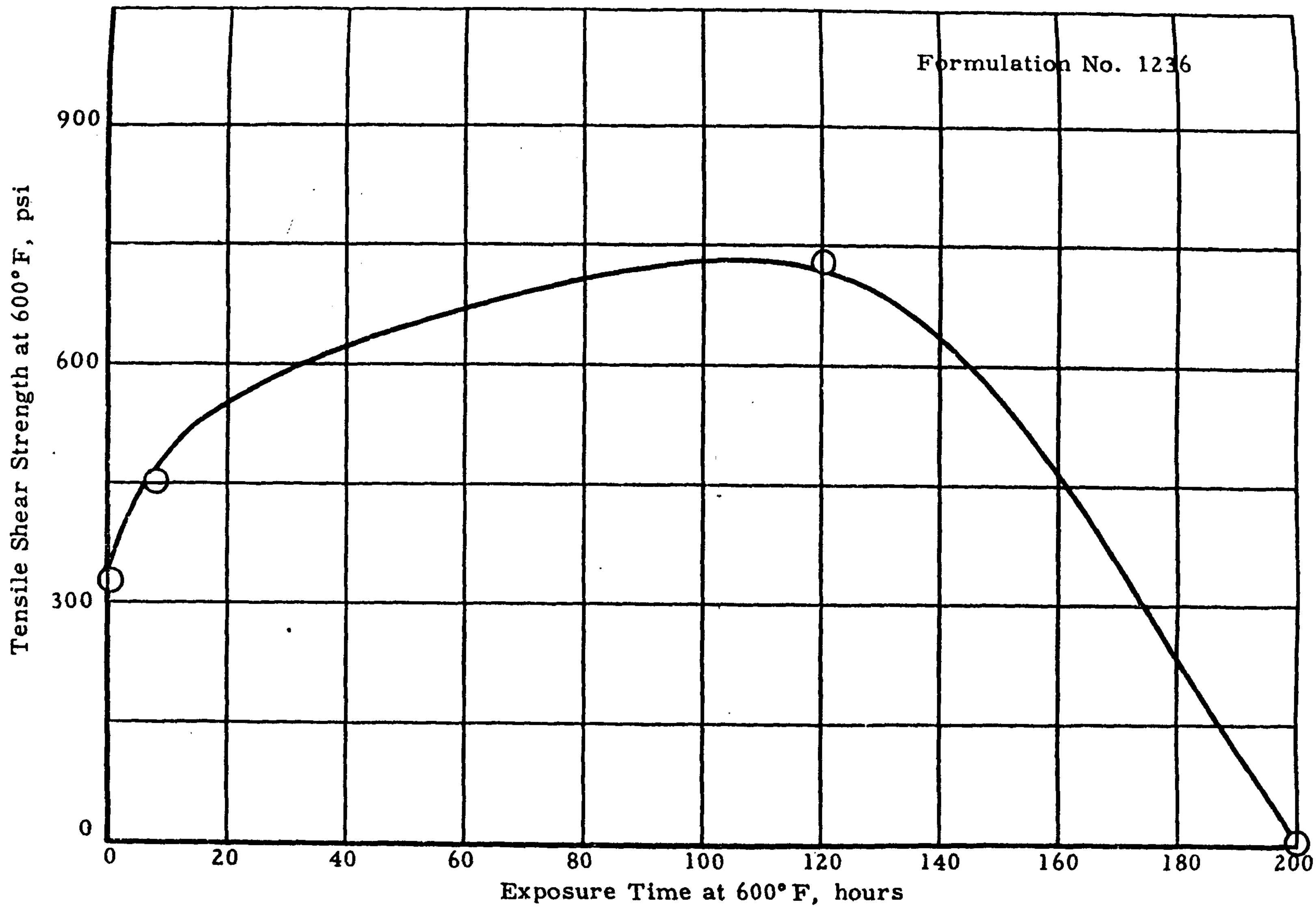

Figure 11. EFFECT OF AGING AT 600°F ON THE 600°F SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95E BONDS TO 301 STEEL

Cure at 100 psi: one-half hour at 240°F + one-half hour at 330°F

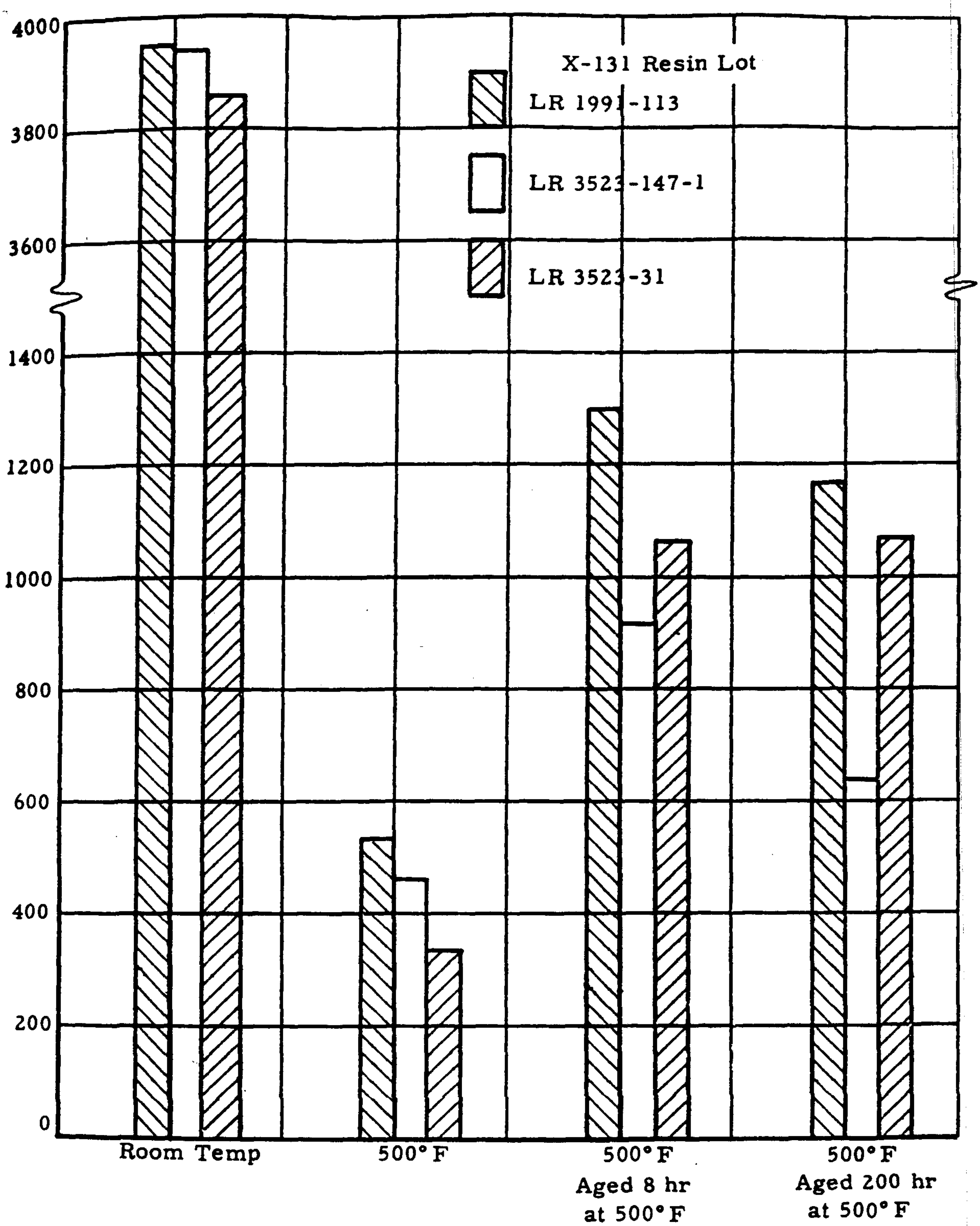

Figure 12. COMPARISON OF THE SHEAR STRENGTH OF BONDS TO 301 STEEL FROM FORMULATION NO. 1236 PREPARED WITH VARIOUS LOTS OF X-131 RESIN (NO POSTCURE)

Cure: one-half hour at 330° F at 100 psi

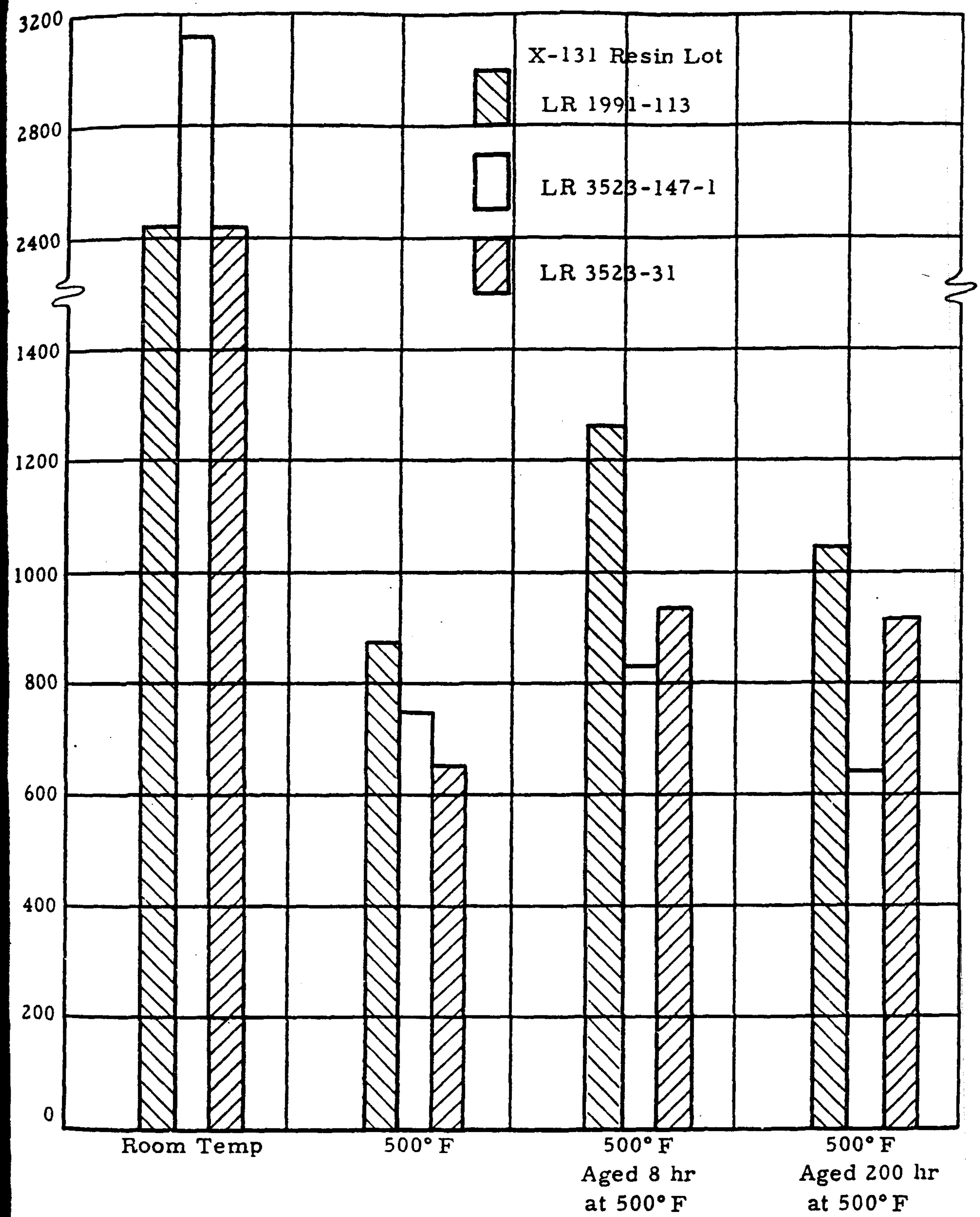

Figure 13. COMPARISON OF THE SHEAR STRENGTH OF BONDS TO 301 STEEL FROM FORMULATION NO. 1236 PREPARED WITH VARIOUS LOTS OF X-131 RESIN (POSTCURED 2 TO 4 HR AT 400° F)

Cure: one-half hour at 330° F at 100 psi prepared with various X-131 resin lots are summarized in Table 39. The average shear strength values for bonds prepared with 5 and 6 batches of adhesive tape cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F are the following:

Tensile Shear Strength, psi	X-131 Resin Lot LR1991-113	X-131 Resin Lot LR3523-147-1
At 77°F	3950	3940
At 500°F	530	460
At 500°F after 8 hr at 500°F	1295	910
At 500°F after 200 hr at 500°F	1165	635

Corresponding test data for bonds postcured 2 to 4 hours at 400°F follow:

Tensile Shear Strength, psi	X-131 Resin Lot LR1991-113	X-131 Resin Lot LR3523-147-1
At 77°F	2420	3110
At 500°F	870	745
At 500°F after 8 hr at 500°F	1265	745
At 500°F after 200 hr at 500°F	1040	640

As shown in the above examples, postcuring Formulation No. 1236 bonds for 2 to 4 hours at 400°F decreased room temperature strength 25 to 40% and increased the 500°F strength 60 to 70%. Attempts to find postcuring conditions which would develop greater initial strength at 500°F were unsuccessful. The experimental results are given in Table 40. Illustrative data for bonds cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F follow:

Postcure hr	Postcure °F	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F
None		3900	495	680
16	270	4025	610	535
4	330	3700	735	715
8	400	3015	1065	805
16	400	2935	875	445

The embrittlement of adhesive bonds from Formulation No. 1236 upon postcuring at elevated temperature is also reflected in the decrease in the bend strength of the bonds. The effect of postcuring on bend strength is shown in Figure 8 and Table 28. Pertinent data follow:

Postcure hr	Postcure °F	Bend Strength, lb
None		186
8	330	149
4	400	133
8	400	115
8	500	48

There was no significant difference in the joint strength of Formulation No. 1236 to type 301 steel which was prepared for bonding with four different surface treatments. These treatments included 1) sulfuric acid followed by hydrofluoric/nitric acid, 2) formalin-peroxide solution, 3) formalin-peroxide solution followed by passivation with chromic acid and 4) chromic/sulfuric acid. Shear strength was 4000 psi at room temperature, about 600 psi at 500°F, and 1200 psi at 500°F for bonds aged 200 hours at 500°F for each of the treatments. The test results are given in Table 41. Figure 14 shows the effect of aging time at 500°F on the 500°F shear strength of bonds to 301 steel prepared with various surface treatments.

No appreciable difference in joint strength was observed in a single experiment in which glass fabric carrier was replaced with stainless steel woven wire (see Table 42).

Erratic bond performance precluded a valid evaluation of the salt spray resistance of Formulation No. 1236 bonds to type 301 steel. Shear strength retention was 50 to 87% of the unexposed "control" specimens after 30 days' salt spray exposure in a limited number of tests. The data are given in Table 43.

Exploratory studies were made of the effect of process variables in adhesive tape and adhesive bond preparation in order to increase adhesive strength and to reduce the wide scatter of strength values of Formulation No. 1236. These experiments included 1) use of methylal in the adhesive resin solution in place of a portion of the ketone solvent, 2) changes in the cure cycle, 3) postcuring in the press directly after cure, and 4) use of X-131 resinous primers. No appreciable improvement in shear strength or in the repeatability of adhesive performance was observed in a limited study of these factors. The test results are given in Table 44 (tape prepared with methylal), Table 45 (cure conditions) and Table 46 (X-131 primers).

The storage stability of adhesive Formulation No. 1236 at room temperature was not determined. A few spot checks of aged tape indicated that the performance of the adhesive may be altered appreciably by room temperature storage. Low strength values, especially for the 500°F tests, were obtained with stored tape (see Table 47). The results of a single experiment with Formulation No. 1236 bonds to 301 steel cured at 100 psi

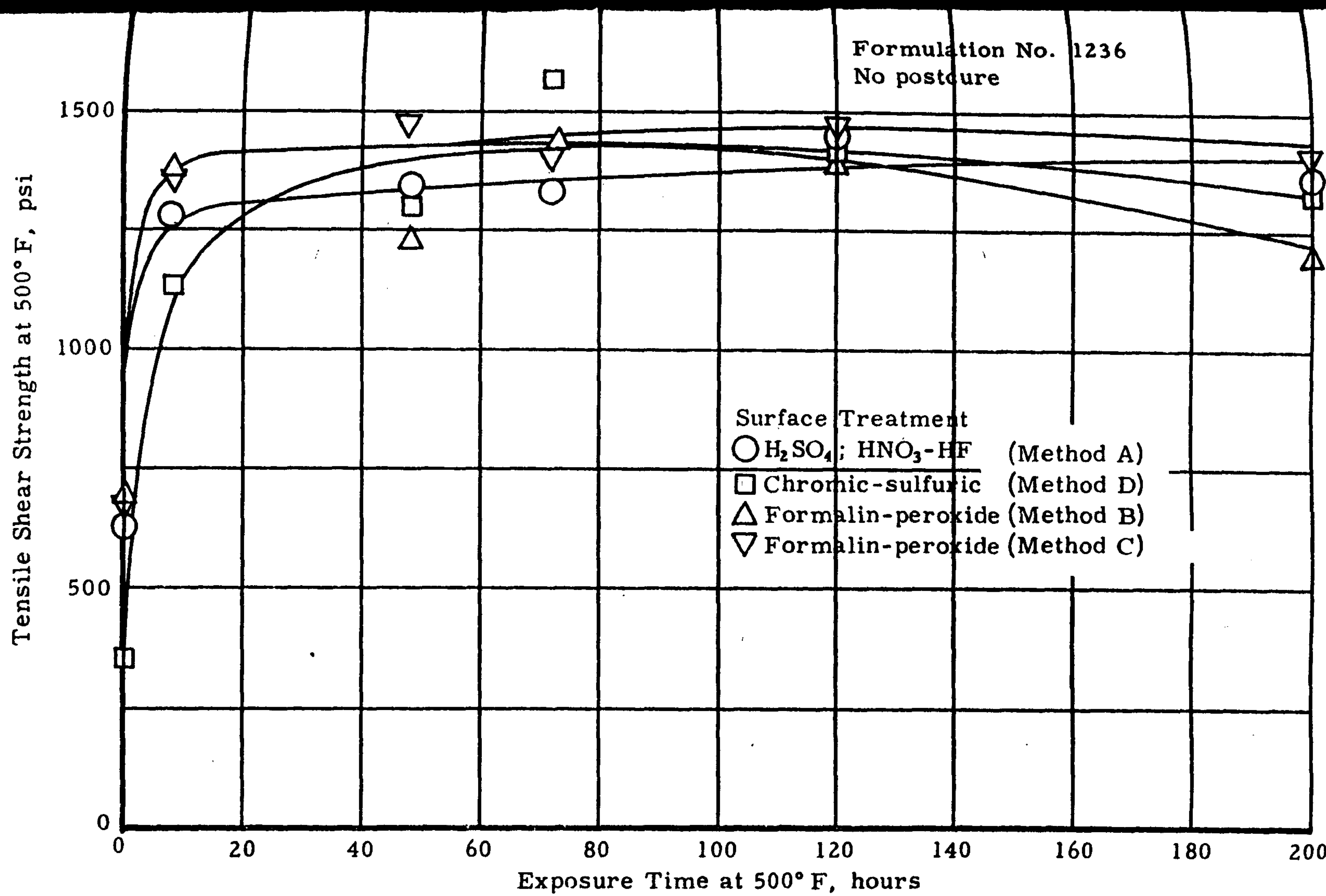

Figure 14. EFFECT OF AGING AT 500° F ON THE 500° F SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95E BONDS TO 301 STEEL WITH VARIOUS SURFACE TREATMENTS

Formulation, phr: 100 aluminum dust + 24.5 diaminodiphenylsulfone
Cure at 100 psi: one-half hour at 240° F + one-half hour at 330° F

for one-half hour at 240°F and one-half hour at 330°F follow:

Tensile Shear Strength, psi	Age of Adhesive Tape at Room Temperature	
	Fresh	8.2 Months
At 77°F	4095	3380
At 500°F	675	305
At 500°F after 8 hr at 500°F	1390	1105
At 500°F after 200 hr at 500°F	1410	200

Bonds to Type 17-7 PH Steel and Titanium

A preliminary evaluation of the strength of Formulation No. 1236 bonds to type 17-7 PH steel showed that the joint strength to this metal is equivalent to the strength to type 301 steel. No significant difference in behavior was noted in a single test of the bond strength for 17-7 PH which was heat treated in air or in helium. The test data for experiments with 17-7 PH steel are summarized in Table 48. The effect of aging at 500°F on the 500°F shear strength of bonds to type 17-7 PH steel is shown graphically in Figure 15. Illustrative data for bonds cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F and postcured two hours at 400°F follow:

Type of Steel	Tensile Shear Strength, psi		
	at 77°F	at 500°F	at 500°F After 200 hr at 500°F
17-7 PH (heat treated in air)	3500	915	885
17-7 PH (heat treated in helium)	3200	810	955
301	2205	585	705

The adhesive bond strength of Formulation No. 1236 to C-110 M titanium alloy treated with chromic-sulfuric acid was mediocre (see Table 49). Shear strength values for bonds cured at 100 psi for one-half hour at 330°F were as follows: at room temperature 2275 psi; at 500°F, about 200 psi; at 500° after 200 hours' aging at 500°F, about 400 psi.

Formulation Studies of Adhesives Based on a Blend of 80/20/20 X-131/EPON 1009/Formvar 15/95 E

The effect of diaminodiphenylsulfone concentration on the bond strength of the 80/20/20 X-131/EPON 1009/Formvar 15/95 E was studied briefly for formulations containing 100 and 120 phr aluminum dust as the reinforcing filler. Supported adhesive tapes were prepared with 12 to 29 phr of diaminodiphenylsulfone. Bonds to type 301 steel were cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F. Some of the bonds were also postcured for two and four hours at 400°F. Shear strength test results at room temperature, at 500°F, and at 500°F after 200 hours' exposure

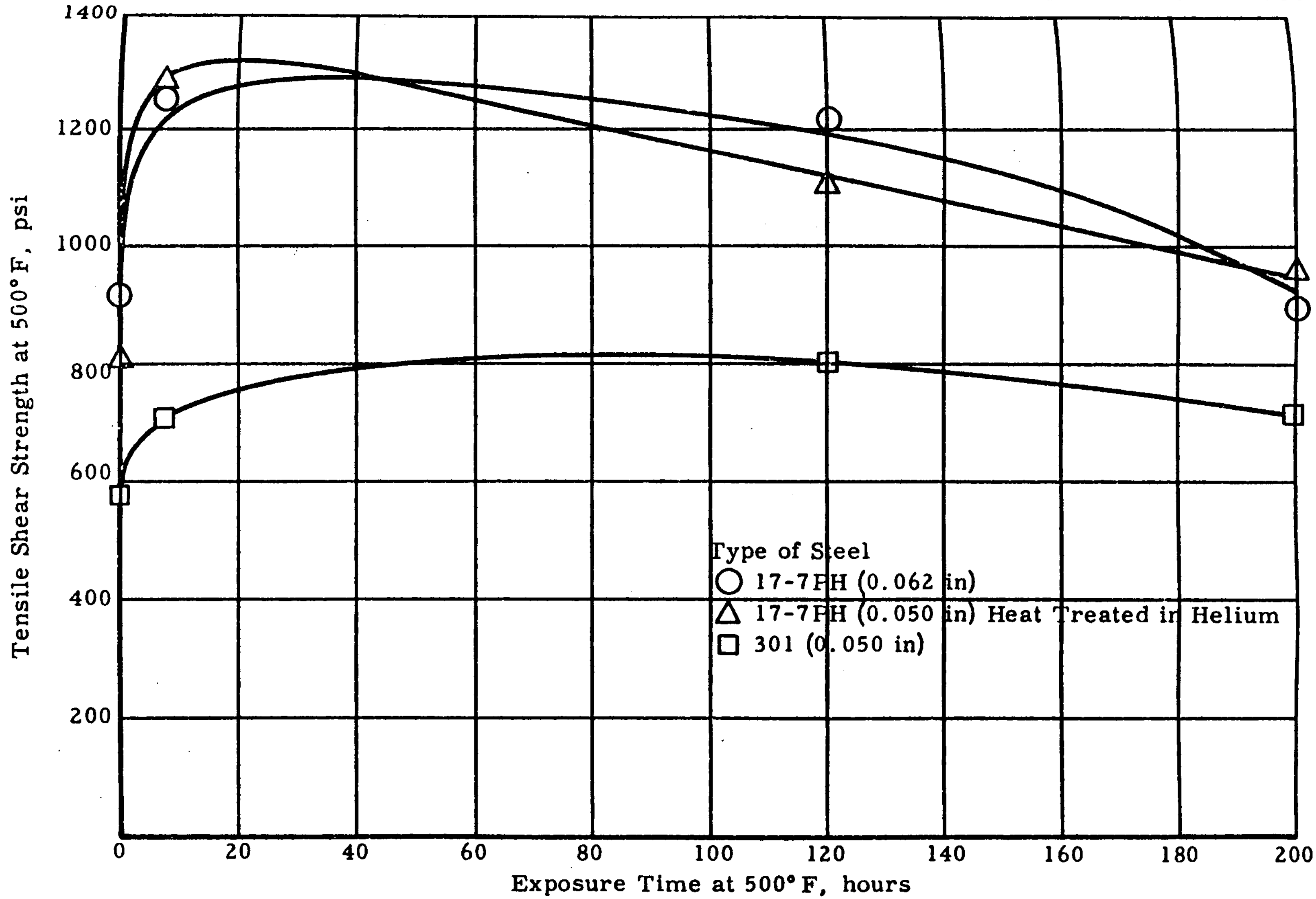

Figure 15. EFFECT OF AGING AT 500°F ON THE 500°F SHEAR STRENGTH OF BONDS FROM FORMULATION NO. 1236 TO TYPE 17-7PH STEEL

Cure at 100 psi: one-half hour at 330°F

Postcure: two hours at 400°F

at 500°F are summarized in Table 50 (100 phr filler) and Table 51 (120 phr filler).

The limited test data indicated that maximum joint strength and strength retention on 500°F aging was obtained with 24 to 29 phr diaminodiphenylsulfone. The application of a postcure increased the initial strength of the bonds with 12 to 20 phr diaminodiphenylsulfone; the values were less than with the higher concentrations of the sulfone, however. The shear strength test results are plotted in Figure 16 and are also summarized below:

Adhesive Bonds Without Postcure

Diaminodiphenylsulfone, phr	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F
12	1195	0	325
16	1030	0	325
20	2010	335	1045
24.5	4095	675	1410
29	4310	1010	1325

Adhesive Bonds Postcured 4 Hours at 400°F

Diaminodiphenylsulfone, phr	Tensile Shear Strength, psi at 77°F	at 500°F
12	1605	340
16	1980	595
20	2610	1025
24.5	2370	940

Bisphenol-A (2 to 10 phr) was added to Formulation No. 1236 with the hope of increasing the rate of cure of the resins and consequently of increasing the hot strength of the unaged bonds. The limited data (Table 52) indicated that small amounts of this additive (2 to 5 phr) increased 500°F shear strength slightly; higher concentrations of Bisphenol-A (8 to 10 phr) decreased the hot strength. The erratic test results precluded a valid evaluation of the effect of the additive on the thermal stability of the adhesive, although it appeared that resistance to aging at 500°F was reduced with 8 to 10 phr Bisphenol-A. Shear strength data follow for bonds to 301 steel cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F:

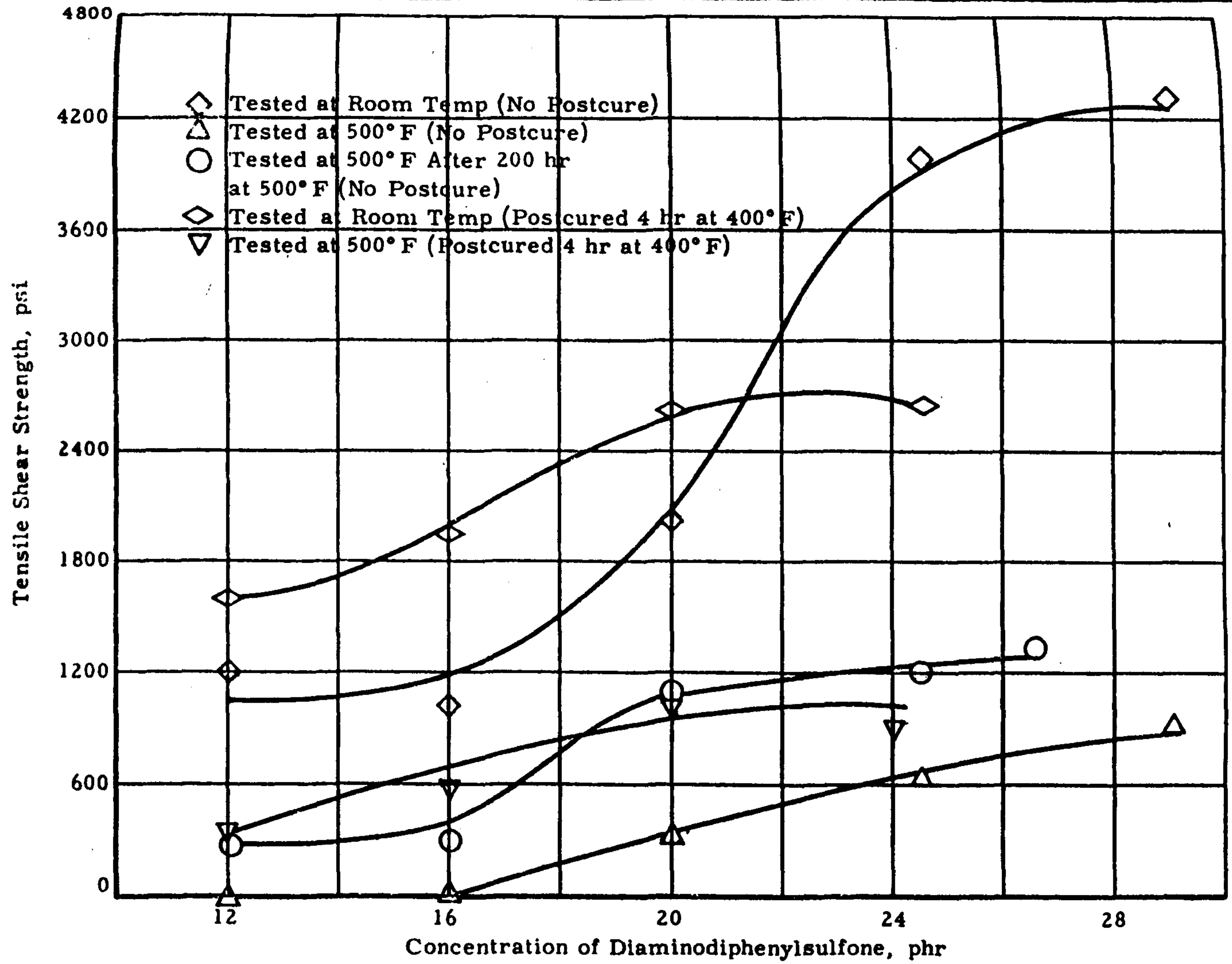

Figure 16. EFFECT OF THE CONCENTRATION OF DIAMINODIPHENYLSULFONE ON THE SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95E BONDS TO 301 STEEL

Constant: 100 phr aluminum dust

Cure at 100 psi: one-half hour at 240° F + one-half hour at 330° F

Bisphenol-A, phr	Tensile Shear Strength, psi at 77°F	at 500°F
0	4155	645
2	4205	775
5	4140	785
8	4215	420
10	4065	375

The utility of several other curing agents for a blend of 80/20/20 X-131/EPON 1009/Formvar 15/95 E was studied briefly. The compounds included 1) poorly soluble, latent curing agents, dicyandiamide and melamine, 2) 4,4',4"-triaminotriphenylmethane, and 3) phthalic acid. Solvent spread supported adhesive tapes were prepared in the usual manner except for dicyandiamide and melamine which were dispersed in the resin solutions. The formulations are described in Table 12 (triaminotriphenylmethane), Table 24 (phthalic acid), Table 53 (dicyandiamide) and Table 54 (melamine). The hot strength of bonds cured with each of these compounds was very poor (0 to 385 psi at 500°F). Bonds with dicyandiamide and melamine developed further strength on exposure to 500°F, indicating a very slow rate of cure. The heat resistance of bonds cured with 4,4',4"-triaminotriphenylmethane and phthalic acid was mediocre. Illustrative data for bonds to 301 steel cured at 100 psi for one-half hour at 240°F plus one-half hour at 330°F follow:

	Curing Agent, phr	Tensile Shear Strength, psi at 77°F	at 500°F	at 500°F After 200 hr at 500°F 8 hr	200 hr
6	dicyandiamide	2590	0	910	930
10	melamine[a]	2660	135	465	1055
19	4,4',4"-triaminotriphenylmethane	3035	385	835	475
47.5	phthalic acid[a]	3925	130	-	305
24.5	diaminodiphenylsulfone	3950	530	1265	1160

a) Bonds postcured 3 hours at 330°F.

Adhesives Based on New Experimental Resins

An exploratory study was made of adhesive formulations based on ten new experimental resins. These resins are characterized, in general, by greater functionality when compared with commercial EPON resins and Experimental Resin X-131. In addition, four of the new resins are liquids of very low viscosity. It was expected that the high degree of functionality and flow of the resins would result in high adhesion and good thermal resistance in metal bonding.

The new experimental resins included the following: 1) solids similar in physical properties to X-131 (modified X-131 resin, X-141, LR2047-76, LR2047-98, and LR2047-126) and 2) low viscosity liquids (X-401-3, X-701, X-801-3 and LR2047-83 and LR2047-199). The solid resins were used in tape form; the liquid resins were used in paste adhesives or as reactive diluents for X-131 resin.

Experimental, modified X-131 resin was evaluated in solvent spread supported adhesive tapes with and without Formvar 15/95 E resin and EPON 1009. The molecular weight, softening point and hydroxyl content of the modified X-131 resin are greater than the corresponding values for the unmodified resin. The properties of various batches of modified X-131 resin are given in Table 55. Adhesive bonds cured with dicyandiamide and diaminodiphenylsulfone are described in Table 56 and Table 57, respectively. The adhesives were characterized by poor wettability and very poor flow. The improvement in adhesion which was anticipated with the higher hydroxyl content resin was therefore not realized. Test data are cited below for adhesives formulated with 20 phr Formvar 15/95 E and cured at 100 psi with diaminodiphenylsulfone for one-half hour at 240°F plus one-half hour at 330°F and postcured two hours at 300°F.

	X-131 Resin	
Tensile Shear Strength, psi	Modified	Unmodified
At room temperature	3520	3615
At 500°F	465	770
At 500°F after aging 8 hr at 500°F	1225	1260
At 500°F after aging 72 hr at 500°F	655	1290
At 500°F after aging 200 hr at 500°F	1000	630

Adhesive formulations based on the other solid experimental resins cured with diaminodiphenylsulfone are given in Table 58 (X-141), Table 59 (LR2047-76), Table 60 (LR2047-98) and Table 61 (LR2047-126). The adhesive shear strengths of bonds to type 301 steel were similar to corresponding X-131 resin bonds in brittleness (mediocre room temperature strength) and in heat resistance. Modification with 20 phr Formvar 15/95 E improved the toughness of the bonds with little or no sacrifice in thermal stability on aging, but decreased initial strength at 500°F. Adhesive shear strength at 600°F (see Table 62) was about double that for the X-131 resin. A comparison of the bond performance of aluminum dust filled adhesives containing no flexibilizing agent follows. These adhesives were cured with diaminodiphenylsulfone at 100 psi for one-half hour at 240°F plus one-half hour at 330°F.

Resin	Tensile Shear Strength, psi at 77°F	at 500°F	at 600°F	at 500°F After 200 hr at 500°F
X-131	2200	1100	200	900
X-141	2435	750	460	700
LR2047-76	1705	1045	-	540
LR2047-98	2040	1225	495	985
LR2047-126	1940	1330	465	315

Paste adhesives containing aluminum dust or glass fibers were formulated with the new liquid experimental resins as follows: 1) X-401-3 and X-801-3 with a blend of pyromellitic dianhydride and maleic anhydride (Tables 63 and 64), 2) X-801-3 with diaminodiphenylsulfone (Table 65) and 3) LR2047-83 with diaminodiphenylsulfone (Table 59). The preliminary evaluation of these adhesives for steel bonding indicated that the experimental resins are characterized by brittleness and a slow rate of cure. The shear strength values at 500°F and at 600°F (Table 62) were inferior to those of corresponding bonds from X-131 resin. Illustrative data for adhesives cured with diaminodiphenylsulfone follow:

Resin	Tensile Shear Strength, psi at 77°F	at 500°F	at 600°F
LR2047-83	2045	245	-
X-801-3	2080	200	135
X-131	2200	1100	200

No promising formulations were found in an exploratory study of paste adhesives cured with a combination of pyromellitic dianhydride and maleic anhydride. Shear strength at 500°F was poor (200 to 600 psi). Some of the adhesive bonds increased in hot strength to about 1000 psi after 200 hours' exposure at 500°F.

Exploratory studies were also made of the utility of the liquid experimental resins as reactive diluents for X-131 resin. Hot melt adhesives formulated with 90/10 to 50/50 X-131/diluent ratios and cured with diaminodiphenylsulfone or dicyandiamide are given in Table 65 (X-801-3), Table 66 (X-401-3), Table 67 (X-701-3) and Table 68 (LR2047-199). The best adhesive bond strength was obtained with experimental resins X-801-3 and LR2047-199. Blends containing 50% of these diluents had shear strength values of 2000 to 3000 psi at room temperature and 950 psi at 500°F after 200 hours aging at 500°F.

Procedures for Metal Treatment and Adhesive Bond Preparation

Surface Treatments for Metals

Stainless Steel. Type 301 stainless steel (thickness 0.050 in.) was used in most of the evaluation work described in this report. Metal with full hard temper was used in the form of strips 1.0 in. x 4.0 in.; metal with half-hard temper was used in the form of panels 4.0 in. x 10.5 in.

The entire panel or strip was degreased with trichloroethylene before the application of any chemical treatment. The metal was warmed to 150°F before chemical treatment to maintain good temperature control of the bath. Eighteen panels (or an equivalent area of strips) were immersed to a depth of one inch in about one liter of the treatment bath. The pickling solutions were not re-used; the alkaline detergent solution was used for three or four applications.

Heat treated type 17-7 PH steel (thickness 0.062 in. and 0.050 in.) with vapor blast finish and C-110 M titanium alloy (thickness 0.050 in.) were used for adhesive bonding in a limited number of experiments. These metals were treated in the same manner employed for the preparation of stainless steel.

The metals were prepared for etching, as follows:

1. Vapor degreased with trichloroethylene.

2. Cleaned with alkaline detergent solution for 10 minutes at 160-180°F. Composition of solution: 3 oz sodium metasilicate, 1.5 oz tetrasodium pyrophosphate, 1.5 oz sodium hydroxide and 0.5 oz Nacconol NR in one gallon water.

3. Rinsed thoroughly in tap water and dried for about 5 minutes at 160-200°F.

The cleaned metal was treated according to the following methods. The treated metal was then rinsed thoroughly in tap water and dried for 5 to 10 min in a 200°F oven.

Method A (sulfuric acid followed by a hydrofluoric/nitric acid "bright dip").

Solution 1.

Sulfuric acid (sp grav 1.84)	190 grams
Water (distilled)	895 "
Nacconol NR	1 "
Operating conditions	10 min at 150-160°F

Solution 2.

Nitric acid (sp grav 1.42)	200 grams
Hydrofluoric acid (48%)	50 "
Water (distilled)	815 "
Operating conditions	10 min at room temp

The metal was rinsed thoroughly in tap water before immersion in Solution 2.

Various modifications of Solution 1, Method A were tried in order to obtain greater uniformity in the appearance of the etched metal. The treatments are as follows:

Method	Operating Temperature, °F	Surfactant
A	150-160	0.5% Nacconol NR
A-M_1	190-195	0.5% Nacconol NR
A-M_2	195-200	None
A-M_3	160-170	0.5% Pluronic L62
A-M_4	160-170	0.25% Pluronic L62
A-M_5	160-170	None

Method B, formalin-peroxide solution (FPL etch).

Hydrochloric acid (sp grav 1.18)	500 grams
Hydrogen peroxide (30%)	20 "
Formalin (aqueous 37%)	100 "
Water (distilled)	450 "
Operating conditions	10 min at 140-150°F

Method C (formalin-peroxide solution followed by passivation with chromic/sulfuric acid).

Solution 1. Method B followed by rinsing in tap water.

Solution 2.

Sodium dichromate dihydrate	33 grams
Sulfuric acid (sp grav 1.84)	65 "
Water (distilled)	1122 "
Operating conditions	10 min at 160-170°F

Method D (Franklin Institute WS-4).

Saturated $Na_2Cr_2O_7$ solution	35 ml
Sulfuric acid (sp grav 1.84)	1000 ml
Operating conditions	15 min at 130-150°F

Aluminum. Clad 2024-T3 aluminum alloy (thickness 0.064 in.) conformed to federal specification QQA-362. The metal was vapor degreased with trichloroethylene and etched with chromic/sulfuric acid, Solution 2, Method C. The panels (4.0 in. x 13.0 in.) were rinsed thoroughly in tap water and dried for about 5 minutes at 160-200°F.

Adhesive Bond Preparation

Adhesive bonds described in this report were single one-half inch lap joints 1.0 in. x 7.5 in. Type 301 stainless steel (0.050 in. thickness, 2B finish) which conformed to specification MIL-S-5059A (ASG) or MIL-S-5059 was used in all of the work. Bonds from full hard metal were assembled with 1.0 x 4.0 strips and cured at contact pressure for one-half hour at 330°F. Panels 4.0 in. x 10.5 in. of half-hard metal were bonded in a hydraulic press at a pressure of 100 psi for one-half hour at 330°F or with a two-stage cure cycle of one-half hour at 240°F plus one-half hour at 330°F. The glue lines were heated to 240°F in 2 to 3 minutes, maintained at this temperature for 30 minutes and then heated rapidly to 330°F and held for 30 minutes. The bonds were cooled rapidly in the press (2 to 3 minutes). Postcuring was done in a forced-draft oven except for a few experiments in which the bonds were postcured in the press directly after the cure cycle. Seven or eight test specimens one inch in width were cut from a panel with a circular staggered-tooth slit saw mounted on a milling machine.

Table 1. EFFECT OF TEST TEMPERATURE ON THE SHEAR STRENGTH OF X-131/DICYANDIAMIDE BONDS TO TYPE 301 STEEL

Formulation No. 1145 (parts by wt): 100 X-131 + 6 dicyandiamide + 100 aluminum dust.

Cure at 100 psi: one-half hour at 240°F plus one-half hour at 330°F.

Panel No.[a]	$1145FM_3$-18	$1145FM_4$-18
Tensile Shear Strength,[b] psi		
At -70°F	2525	-
At room temperature	2205[c]	2180
At 500°F, original	420	600
, after 200 hours at 500°F	680	-
At 600°F	-	60
Bend strength at room temp,[b] lb	110	-

a) Bonds from solvent spread supported adhesive tapes with glass fabric (112-Volan A) dried 15 minutes at 200°F.

b) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A-M_3 and A-M_4.

c) Average of six values.

Table 2. EFFECT OF EXPOSURE TIME AT 500°F ON THE SHEAR STRENGTH OF X-131/DICYANDIAMIDE BONDS TO TYPE 301 STEEL

Cure (contact pressure): one-half hour at 330°F.

Panel No.[a]	1145JM-16	1145JM-15	1145FC-15	1145JFs-10	1145JM-17
Surface Preparation	A	A	C	B	A
Adhesive Batch No.	16	15	15	10	17
Tensile Shear Strength,[b]					
At room temperature	1825	1730	1650	2105	2055
At 500°F	685	665	710	1025	855
At 500°F after 500°F aging, 3 hr	-	-	-	380	-
8	305	315	365	-	365
24	-	-	-	255	-
48	255	260	345	325	115
72	-	-	-	300	285
120	405	335	305	415	115
168	375	345	-	-	385
200	-	-	300	670	440
231	475	330	-	-	-
500	115	215	-	-	-

a) Solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Formulation (by wt): 100 X-131 (lot No. 1991-113) + 100 aluminum dust + 6 dicyandiamide.

b) Average of three values for one-half inch lap joints to full hard type 301 steel (thickness 0.050 in.).

Table 3. EFFECT OF POSTCURING ON THE SHEAR STRENGTH OF X-131/DICYANDIAMIDE BONDS TO TYPE 301 STEEL

Cure (contact pressure): one-half hour at 330°F.

Specimen No.[a]	Dicyan-diamide, phr	Postcure		Tensile Shear Strength,[b] psi		
				Original		Aged 200 hr at 500°F
		°F	hr	77°F	500°F	500°F
1085JF-3	4	400	0	2310	755	730
-PC1	4	400	1	2510	1065	825
-PC3	4	400	3	2305	1190	825
$1085JF_2$-4	4	500	0	2320	545	620
-PC1	4	500	1	1715	660	530
-PC3	4	500	3	1815	215	560
$1145JF_2$-8	6	500	0	2505	1050	905
-PC1	6	500	1	2135	640	770
-PC3	6	500	3	1665	605	655

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 minutes at 200°F. Formulation (by wt): 100 X-131 (lot No. LR1991-113) + 100 aluminum dust.

b) Average of three values for bonds to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method B.

Table 4. EFFECT OF DRYING TIME AT 200°F OF X-131/DICYANDIAMIDE TAPES ON THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL

Cure (contact pressure): one-half hour at 330°F.

Specimen No.[a] 1145JM$_2$-	Drying Time, Min at 200°F	Tensile Shear Strength,[b] psi		
		Original		Aged 200 hr at 500°F
		77°F	500°F	500°F
12	15	1940	815	425
12-30	30	1875	920	445
12-60	60	2115	1015	445
12-120	120	2350	1130	465
13	15	1935	595	410
13-30	30	1940	575	350
13-60	60	2270	695	375
13-120	120	2595	835	415

a) Bonds from solvent spread supported adhesive tapes with glass fabric 112-Volan A. Formulation (by wt): 100 X-131 (lot No. LR1991-113) + 100 aluminum dust + 6 dicyandiamide.
b) Average of three values for bonds to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A-M$_2$.

Table 5. EFFECT OF DICYANDIAMIDE CONCENTRATION ON THE SHEAR STRENGTH OF X-131 BONDS TO TYPE 301 STEEL

Cure (contact pressure): one-half hour at 330°F.

Specimen No.[a]	1145JM-17	1146JM-2	1203JM	1204JM	1205JM
Dicyandiamide, phr	6	8	10	12	14
Tensile Shear Strength,[b] psi					
At room temperature	2055	1935	1815	1915	1665
At 500°F	845	585	525	535	60
At 500°F after 500°F aging, 8 hr	365	385	460	475	595
48	115	95	195	505	595
72	285	245	525	555	540
120	115	380	620	1025	535
168	385	320	320	795	630
200	460	350	455	790	295

a) Aqueous-acetone spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients (by wt): 100 X-131 (lot No. LR1991-113) + 100 aluminum dust.

b) Average of three values for bonds to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A.

Table 6. EFFECT OF ALUMINUM FILLER CONCENTRATION ON THE SHEAR STRENGTH OF X-131/DICYANDIAMIDE BONDS TO TYPE 301 STEEL

Cure (contact pressure): one-half hour at 330°F.

Specimen No.[a]	Aluminum Dust, phr	Dicy[b] phr	Tensile Shear Strength,[c] psi							
			Original		At 500°F After 500°F Aging for					
			77°F	500°F	8 hr	48 hr	72 hr	120 hr	168 hr	200 hr
1192JM$_2$	75	4	1650	535	-	-	-	-	-	250
1171JM$_2$-2	75	6	2095	645	-	-	-	-	-	525
1085JM$_2$-5	100	4	2275	670	-	-	-	-	-	670
1145JM$_2$-11	100	6	2080	670	-	-	-	-	-	565
1145JM-17[d]	100	6	2055	845	365	115	285	115	385	460
1193JM$_2$	125	4	2355	585	-	-	-	-	-	880
1194JM$_2$	125	6	2125	765	-	-	-	-	-	980
1206JM[d]	125	6	1730	590	180	160	205	260	285	340
1207JM[d]	150	6	1755	585	95	205	280	470	420	465
1208JM[d]	175	6	1685	570	435	365	360	430	580	610
1209JM[d]	200	6	1925	675	380	375	245	430	455	685

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 minutes at 200°F.

b) "Dicy" is dicyandiamide. This curing agent was dissolved in an aqueous-acetone solution of X-131 resin (lot No. LR1991-113).

c) Average of three values for one-half inch lap joints to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A-M$_2$.

d) Surface treatment: Method A.

Table 7. EFFECT OF COPPER 8-QUINOLINOLATE ON THE SHEAR STRENGTH OF X-131/DICYANDIAMIDE BONDS TO TYPE 301 STEEL

Cure (contact pressure): one-half hour at 330°F.

Specimen No.[a]	1145JM-17	1210JM	1211JM	1212JM
Copper 8-Quinolinolate, phr	0	0.5	1.0	2.0
Tensile Shear Strength,[b] psi				
At room temperature	2055	1455	1260	1675
At 500°F	845	510	485	415
At 500°F after 500°F aging, 8 hr	365	260	335	220
48	115	270	305	280
72	285	310	240	315
120	115	345	305	440
168	385	400	305	270
200	460	425	505	505

a) Solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients (by wt): 100 X-131 (lot No. LR1991-113) + 100 aluminum dust + 6 dicyandiamide.

b) Average of three values for bonds to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A.

Table 8. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131 ADHESIVES WITH 4-6 PHR CURING AGENT E

Cure: one-half hour at 310°F at contact pressure.

Specimen No.[a]	Curing Agent E, phr	Postcure at 400°F, hr	Tensile Shear Strength,[b] psi		
			Original		Aged 200 hr at 500°F
			77°F	500°F	500°F
$1186IF_3$	4	0	525	0	-
-PC1	4	1	2335	305	810
-PC3	4	3	1845	700	685
$1187IF_3$	6	0	2195	485	1145
-PC1	6	1	1930	930	1025
-PC3	6	3	1875	1200	1000

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 minutes at 200°F. Formulation (by wt): 100 X-131 (lot No. 1991-113) + 100 aluminum dust.

b) Average of three values for bonds to full hard, type 301 steel (thickness 0.050 in.). Surface treatment: Method B.

Table 9. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131 ADHESIVES WITH 13 PHR CURING AGENT E

Cure at 100 psi: one-half hour at 240°F plus one-half hour at 310-330°F.
Postcure (oven): three hours at 300°F.

		1232EM$_8$-P33	1229EM$_4$-P33[b)]	1231FM$_4$-P33[c)]
Panel No.[a)]		1232EM$_8$-P33	1229EM$_4$-P33[b)]	1231FM$_4$-P33[c)]
Surface Treatment Method		A-M$_8$	A-M$_4$	A-M$_4$
Cure, min/°F		30/310	30/330	30/330
Type of Adhesive[d)]		Hot Melt	Tape	Tape
Formvar 15/95E, phr		None	None	20
Tensile Shear Strength,[e)] psi				
At room temperature		1670	1285	1905
At 500°F, original		565	485	215
At 500°F, after 500°F aging,	24 hr	210	200	370
	48	515	210	515
	72	0[f)]	250[f)]	400[f)]
	120	145	215	455
	200	370	315	165

a) Constant ingredients, phr: 100 aluminum dust + 13 Curing Agent E (2,6-diaminopyridine).
b) Test results for panel No. 1229EM$_8$-P33 cured one-half hour at 310°F: Shear strength at room temperature, 1660 psi (avg of eight values).
c) Test results for panel No. 1231EM$_8$-P33 cured one-half hour at 310°F: Shear strength at room temperature, 1900 psi (avg of eight values).
d) Adhesive bonds prepared with glass fabric 112-Volan A. Adhesive tapes were prepared from solvent dispersions of the adhesive and then dried 15 minutes at 200°F.
e) Average of two or three values for one-half inch lap-joints to half-hard type 301 steel (thickness 0.050 in.).
f) One value.

Table 10. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131 ADHESIVES CURED WITH BENZIMIDAZOLYL GUANIDINE

Cure (contact pressure): one-half hour at 330°F

Formulation No.[a]	1200JM	1202FM[b]	1145JM-16
Curing Agent, phr	10 BIG[c]	20 BIG[c]	6 dicy[c]
Tensile Shear Strength,[d] psi			
At room temperature	1175	1425	1825
At 500°F, original	530	110	685
At 500°F, after 500°F aging, 8 hr	300	135	305
48	540	95	255
72	510	240	-
120	610	340	405
168	715	420	375
200	595	415	475

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 minutes at 200°F. Formulation (parts by wt): 100 X-131 (lot No. LR1991-113) + 100 aluminum dust.
b) Cure: One-half hour at 240°F plus one-half hour at 330°F.
c) "Dicy" is dicyandiamide. "BIG" is benzimidazolyl guandine.
d) Average of three values for one-half inch lap joints to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A.

Table 11. SHEAR STRENGTH OF X-131/DIAMINODIPHENYLSULFONE BONDS TO TYPE 301 STEEL

Formulation (parts by wt): 100 X-131 + 100 aluminum dust + 30 diaminodiphenylsulfone.
Cure at 100 psi: one-half hour at 240°F plus one-half hour at 330°F.

Panel No.[a]	1201JM-P23[b]	1201FE-3[c]	1201FE-4	1201FE-5	1201FE-6
X-131 Resin lot No. LR-	1991-113	1991-113	3523-147-1	3523-115	1991-113
Tensile Shear Strength,[d] psi					
At room temperature	2255	1840[e]	2815[e]	2140[e]	2420
At 500°F, original	1075	905	1140	1280	1060
At 500°F, after 500°F aging, 8 hr	1095	-	-	-	1165[e]
48	710	-	-	-	1150[e]
72	785	-	-	-	1220[e]
120	690	-	-	-	1075[e]
168	670	-	-	-	-
200	780	870	830	835	1115[e]
At 600°F, original	-	-	195	-	-

a) Bonds from solvent spread supported adhesive tapes with glass fabric (112-Volan A) dried 15 minutes at 200°F.
b) Bonds to full-hard type 301 steel cured at contact pressure for one-half hour at 330°F and postcured two hours at 300°F. Surface treatment: Method A.
c) Adhesive tape stored for two months at room temperature before testing.
d) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
e) Average of two values.

Table 12. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131 ADHESIVES CURED WITH 4,4',4"-TRIAMINOTRIPHENYLMETHANE

Cure: one-half hour at 330°F and 100 psi

		1326JE	1327JE	1328JE
Panel No.[a]		1326JE	1327JE	1328JE
X-131/EPON 1009 Ratio,[b] by wt		100/0	100/0	80/20
Formvar 15/95 E, phr		-	20	20
Triaminotriphenylmethane, phr		23.5	23.5	19
Tensile Shear Strength,[c] psi				
At room temperature		865	1430	3035
At 500°F, original		525	455	385
At 500°F, after 500°F aging,	8 hr	605	420	835
	120	730	510	795
	200	375	330	475

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 minutes at 200°F. Constant ingredient, by wt: 100 aluminum dust.
b) X-131 lot No. LR3523-64.
c) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Average of two values.

Table 13. EFFECT OF X-131/EPON 1009 RESIN RATIO ON THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL CURED WITH DICYANDIAMIDE

Cure (contact pressure): one-half hour at 330°F with 6 phr dicyandiamide.

Specimen No.[a]	X-131[b]/ EPON 1009	Surface Treatment Method	Tensile Shear Strength,[c] psi		
			Original		Aged 200 hr at 500°F
			77°F	500°F	500°F
1145JF[d]	100/0	B	2290	1145	675
1195JF_4-2	90/10	B	2865	1045	830
1196JF_4-2	80/20	B	2785	1020	1075
1197JF_4-2	70/30	B	2455	855	880
1145JM[e]	100/0	A	1930	770	680
1195JM_2-2	90/10	A-M_2	2225	755	635
1196JM_2-2	80/20	A-M_2	2205	585	1235
1197JM_2-2	70/30	A-M_2	2335	655	780

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients, phr: 100 aluminum dust + 6 dicyandiamide.
b) X-131 resin lot No. LR1991-113.
c) Average of three values for bonds to full hard type 301 steel (thickness 0.050 in.).
d) Test results given are the averages for six adhesive batches.
e) Test results given are the averages for eight adhesive batches.

Table 14. EFFECT OF X-131/EPON 1009 RESIN RATIO ON THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL CURED WITH DIAMINODIPHENYLSULFONE

Cure at 100 psi: one-half hour at 240°F plus one-half hour at 330°F
Postcure (oven): two hours at 300°F

Panel No.[a]		1201JM-P23[c]	1233FM$_3$-P23	1234FM$_3$-P23	1235FM$_3$-P23
X-131/EPON 1009 Ratio, by wt		100/0	90/10	80/20	70/30
Diaminodiphenylsulfone, phr		30	27	24.5	21.5
Tensile Shear Strength,[b] psi					
At room temperature		2255	2315	2255	2640
At 500°F		1075	750	775	785
At 500°F after 500°F aging,[d]	8 hr	1095	855	550	590
	48	710	420	500	580
	72	785	760	760	610
	120	690	720	760	590
	200	780	900	700	885

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust. X-131 lot No. 1991-113.
b) Average of three values for 0.5 in lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A-M$_3$.
c) Bonds to full hard type 301 steel (thickness 0.050 in.) cured at contact pressure. Average of three values for all tests. Surface treatment: Method A.
d) Average of two values.

Table 15. EFFECT OF DRYING TIME AT 200°F OF X-131/ EPON 1009/DICYANDIAMIDE TAPES ON THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL

Cure (contact pressure): one-half hour at 330°F

Specimen No.[a]	X-131/EPON 1009 Resin Ratio[b]	Drying Time, Min at 200°F	Tensile Shear Strength,[c] psi		
			Original		Aged 200 hr at 500°F
			77°F	500°F	500°F
1195JM$_2$-2	90/10	15	2225	755	635
-30	"	30	2435	895	605
-60	"	60	2730	865	395
-120	"	120	2745	935	415
1196JM$_2$-2	80/20	15	2205	585	1235
-30	"	30	2235	635	960
-60	"	60	2690	745	435
-120	"	120	2700	1025	415
1197JM$_2$-2	70/30	15	2335	655	780
-30	"	30	2575	585	370
-60	"	60	2500	735	260
-120	"	120	2405	770	460

a) Bonds from solvent spread supported adhesive tapes with glass fabric 112-Volan A. Constant ingredients, phr: 100 aluminum dust + 6 dicyandiamide.

b) X-131 resin lot No. LR1991-113.

c) Average of three values for bonds to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A-M$_2$.

Table 16. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131/ EPON 834 HOT MELT ADHESIVES CURED WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi for one-half hour
at 240°F plus one-half hour at 330°F

Panel No.[a]	1298FE	1299FE	1300FE	1201FE-4[b]
X-131/EPON 834 Ratio,[c] by wt	50/50	60/40	70/30	100/0
Diaminodiphenylsulfone, phr	25	26	27	30
Tensile Shear Strength,[d] psi				
At room temperature	1560	1945	3215	2815[e]
At 500°F, original	375	375	110	1140
, after 500°F aging, 8 hr	0	120	55[e]	-
, 120	555[e]	570[f]	625[e]	-
, 200	550	525	445	830

a) Bonds prepared with hot melt adhesives and glass cloth 112-Volan A reinforcement. Constant ingredient: 100 phr aluminum dust (MD201).
b) Solvent spread tape dried 15 min at 200°F.
c) Experimental resin X-131, lot No. LR3523-147-1.
d) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
e) Average of two values.
f) One value.

Table 17. EFFECT OF FORMVAR 15/95 E CONCENTRATION ON THE SHEAR STRENGTH OF X-131/DICYANDIAMIDE BONDS TO TYPE 301 STEEL

Cure (contact pressure): one-half hour at 330°F
Postcure: three hours at 400°F

Specimen No.[a]	Formvar 15/95 E, phr	Tensile Shear Strength,[b] psi		
		Original		Aged 200 hr at 500°F
		77°F	500°F	500°F
1085JF-3PC3	0	2305	1190	825
1182JF$_2$-PC3	10	3085	975	805
1183JF$_2$-PC3	15	3095	595	795
1184JF$_2$-PC3	20	3710	500	1125
1185JF$_2$-PC3	30	4200	375	665

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients (by wt): 100 X-131 + 100 aluminum dust + 4 dicyandiamide.
b) Average of three values for bonds to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method B.

Table 18. EFFECT OF X-131/FORMVAR 15/95 E RESIN RATIO ON THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL CURED WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]		1219FF-5	1248FF	1249FF	1250FF
X-131/Formvar 15/95 E Ratio, by wt		83.3/16.7	75/25	50/50	25/75
Al Dust/Total Resin Ratio, by wt		5/6	3/4	1/2	1/4
Tensile Shear Strength,[b] psi					
At room temperature		3555	2895	4045	4210
At 500°F, original		640	345	0	0
, after 500°F aging,[c]	8 hr	1690	1385	1010	160
	48	1545	1500	1100	95
	72	1390	1425	790	60
	120	1490	1200	855	110
	200	1030	850	560	0
Panel No.[a]		1251FF	1252FF	1253FF	1254FF
X-131/Formvar 15/95 E Ratio, by wt		83.3/16.7	75/25	50/50	25/75
Al Dust/Total Resin Ratio, by wt		1/1	1/1	1/1	1/1
Tensile Shear Strength,[b] psi					
At room temperature		4015	3725	3665	2070
At 500°F, original		1285	1265	0	0
, after 500°F aging,[c]	8 hr	1530	1490	620	465
	48	1490	1605	460	120
	72	1405	1215	400	0
	120	1230	1265	500	0
	200	1095	925	100	0

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 30 phr diaminodiphenylsulfone. X-131 resin lot No. LR1991-113.

b) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method B.

c) Average of two values for all aged bonds.

Table 19. EFFECT OF BF_3-400 ON THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM 100/20 X-131/Formvar 15/95 E ADHESIVES CURED WITH DIAMINODIPHENYLSULFONE

Cure at 100 psi: one-half hour at 240°F plus one-half hour at 330°F.
Postcure (oven): two hours at 300°F.

Panel No.[a]	$1237FM_4$-P23	$1238FM_4$-P23
Curing Promoter,[b] phr	1 BF_3-400	None
Tensile Shear Strength,[c] psi		
At room temperature	2285	2655
At 500°F, original	430	680
, after 500°F aging, 8 hr[d]	695	1165
, 48[d]	725	1010
, 72[d]	855	1315
, 120[d]	460	1010
, 200	285	830

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F.
b) Constant ingredients (parts by wt): 100 X-131 (lot No. LR1991-113) + 20 Formvar 15/95 + 30 neutral TC asbestos + 30 diaminodiphenylsulfone.
c) Average of three values for one-half inch lap joints to half-hard type 301 steel; specimens cut with Do-All band saw. Surface treatment: Method A-M_4.
d) Average of two values.

Table 20. COMPARISON OF THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM 100/20 X-131/FORMVAR 15/95 E ADHESIVES WITH ASBESTOS AND ALUMINUM FILLERS

Cure at 100 psi: one-half hour at 240°F
plus one-half hour at 330°F.
Postcure (oven): two hours at 300°F.

Panel No.[a] Filler,[b] phr		1238FM$_4$-P23 30 TC Asbestos	1219FM$_4$-4P23 100 Al Dust
Tensile Shear Strength,[c] psi			
At room temperature		2655	2740[d]
At 500°F, original		680	670[e]
, after 500°F aging,	8 hr	1165	-
	, 48	1010[f]	-
	, 72	1315[f]	-
	, 120	1010[f]	-
	, 200	830	830

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F.
b) Constant ingredients (parts by wt): 100 X-131 (lot No. LR1991-113) + 20 Formvar 15/95 E + 30 diaminodiphenylsulfone.
c) Average of three values for one-half inch lap joints to half-hard type 301 steel; specimens cut with Do-All band saw. Surface treatment: Method A-M$_4$.
d) Average of nine values.
e) Average of six values.
f) Average of two values.

Table 21. EFFECT OF BISPHENOL-A ON THE SHEAR STRENGTH OF 100/20 X-131/FORMVAR 15/95 E BONDS TO TYPE 301 STEEL

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]	Bisphenol A, phr	Tensile Shear Strength,[b] psi						
		Original		At 500°F After 500°F Aging For[c]				
		77°F	500°F	8 hr	48 hr	72 hr	120 hr	200 hr
1219FE-6	0	3605	1020	1505	1415	1495	1115	950
1260FE	2	3785	545	1520	1025	760	660	625
1220FE-2	5	4235	880	1285	1270	920	1050	690
1261FE	8	4010	530	1520	1320	1155	1275	610
1262FE	10	4135	355	1080	1010	880	425	675

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients (parts by wt): 100 X-131 (lot No. LR1991-113) + 20 Formvar 15/95 E + 100 aluminum dust + 30 diaminodiphenylsulfone.
b) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
c) Average of two values for all aged bonds.

Table 22. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM 100/20 X-131/FORMVAR 15/95 E ADHESIVES CURED WITH MELAMINE

Press cure at 100 psi with 10 phr dispersed melamine

Panel No.[a]	1268FE-P333	1268FE-P3334	1276JE-2P34	1276PE-2P34
X-131 Resin Lot No. LR-	1991-113	1991-113	3523-147-1	3523-147-1
Cure Temperature, min/°F	30/240 + 30/330	30/240 + 30/330	30/330	30/80-400[b]
Postcure, Method	Oven	Oven	Oven	Press
, hr/°F	3/330	3/330 + 2/400	3/400	3/400
Tensile Shear Strength,[c] psi				
At room temperature	2845	2940	-	3830
At 500°F, original	525	755	-	245
, after 500°F aging, 8 hr	-	-	480[d]	-
, 200	1075	1075	385[d]	645

a) Bonds from solvent spread supported adhesive tapes with glass fabric (112-Volan A) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust.
b) Time-temperature cycle: 80 to 300°F in 15 min; 300 to 400°F in 10 min.
c) Average of two to three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Average of 14 or 15 values; range 0 to 610 psi.

Table 23. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131 ADHESIVES CURED WITH CURING AGENT BF_3-400

Cure: one-half hour at 330°F

Panel No.[a]	1199JM	$1221FM_3$[b]
Hardness of Steel	Full Hard	Half-Hard
Cure Pressure	Contact	25 psi
X-131/EPON 1009 Ratio, by wt	80/20	100/0
Formvar 15/95 E, phr	0	20
Tensile Shear Strength,[c] psi		
At room temperature	1165	2405
At 500°F, original	545[d]	515
, after 500°F aging, 8 hr	355	395[e]
, 48	160	370[e]
, 72	390	320[e]
, 120	305	650[e]
, 168	555	-
, 200	450	420[e]

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients, phr: 3 BF_3-400 + 30 TC neutral asbestos. X-131 resin lot No. LR1991-113.

b) Bonds precured one-half hour at 240°F at 25 psi. Surface treatment: Method A-M_3.

c) Average of three values for one-half inch lap joints to type 301 steel (thickness 0.050 in.). Surface treatment: Method A.

d) Shear strength for bonds precured one-half hour at 240°F: 395 psi at 500°F.

e) Average of two values.

Table 24. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM 100/20 X-131/FORMVAR 15/95 E ADHESIVES CURED WITH ACID-TYPE CURING AGENTS

Press cure at 100 psi for one-half hour
at 240°F plus one-half hour at 330°F

Panel No.[a]	EPON Resins, Parts by wt	Curing Agent, phr	Filler, phr	Tensile Shear Strength, psi[b] Original 77°F	Original 500°F	Aged 200 hr At 500°F 500°F[c]
1242FF	100 X-131	3 Magnesium perchlorate	100 Al	2125	420	0[b]
1245FF	"	9 Zinc fluoborate heptahydrate	" "	broke on handling		
$1244FM_4$[d]	"	3 $BF_3 \cdot$ ammonia	" "	1635	50	0
1243FF	"	3 $BF_3 \cdot$ urea	" "	855	0	45
1247FF-P333[e]	"	" "	30 TC Asbestos	2735	440	0
$1240FM_4$-P333[e]	"	75 Hexahydrophthalic anhydride[f]	"	975	0	0
$1239FM_4$-P333[e]	" g)	" " f)	"	490	0	0
1246FF-P333[e]	80/20 X-131/1009	47.5 Phthalic acid	"	3925[c]	130	305[b]
1267FF-P333[e]	"	" " " f)	"	1965	55	285

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient, phr: 20 Formvar 15/95 E. Experimental resin X-131, lot No. LR1991-113.
b) Average of three values for bonds to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method B.
c) Average of two values.
d) Surface preparation: Method $A-M_4$.
e) Bonds postcured for 3 hours at 330°F.
f) Formulation contained 2 phr dimethyl ethanolamine as a cure promoter.
g) This formulation without Formvar 15/95 E resin.

Table 25. SHEAR STRENGTH OF 100/20 X-131/FORMVAR 15/95 E BONDS TO TYPE 301 STEEL (FORMULATION NO. 1219)

Formulation (parts by wt): 100 X-131 + 20 Formvar 15/95 E
+ 100 aluminum dust + 30 diaminodiphenylsulfone
Cure at 100 psi: one-half hour at 240°F plus one-half
hour at 330°F

Panel No.[a] 1219-	Surface Treatment	Adhesive Batch No.	X-131 Lot No. LR	Postcure hr/°F	Tensile Shear Strength,[b] psi: Original 77°F	Original 500°F	Aged 200 hr at 500°F: 500°F
JM_1-P23[c]	A-M_1	1	1991-113	2/300	2720	850	1015
FM_3-2P23	A-M_3	2	3523-64	2/300	3615	770	630[d]
FM_4-2P23	A-M_4	2	3523-64	2/400	3295	610	-
FM_3-3P24	A-M_3	3	1991-113	2/400	2480[f]	900	975
FM_4-3P24	A-M_4	3	1991-113	2/400	1865[d]	990	1225[d]
FM_4-4P23[e]	A-M_4	4	1991-113	2/300	2740[f]	670[f]	830
FM_4-4P23[e]	A-M_4	4	1991-113	2/300	2725[f]	635[f]	1355
FM_4-4P24	A-M_4	4	1991-113	2/400	1655	695	1020
FF-4P23	B	4	1991-113	2/300	3205	835	820
-4P24	B	4	1991-113	2/400	2380	935	900
FF-5	B	5	1991-113	--	3555	640	1030[d]
FE-6	C	6	1991-113	--	3605	1020	950[d]
-7	C	7	3523-147-1	--	3870[d]	905	415
-8	C	8	3523-115	--	4080[d]	930	555
-9	C	9	3523-147-1	--	3630[d]	715	765
FE-9P233	C	9	3523-147-1	2/330	3160[d]	780	675
-9P24	C	9	3523-147-1	2/400	2700[d]	895	700

a) Bonds from solvent spread supported tape with glass fabric (112-Volan A) dried 15 min at 200°F.
b) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.).
c) Bonds to full hard type 301 steel cured at contact pressure for one-half hour at 330°F.
d) Average of two values.
e) Specimens cut with Do-All band saw.
f) Average of five to nine values.

Table 26. EFFECT OF 500°F EXPOSURE ON THE SHEAR STRENGTH OF 100/20 X-131/FORMVAR 15/95 E BONDS TO TYPE 301 STEEL (FORMULATION NO. 1219)

Cure at 100 psi: one-half hour at 240°F
plus one-half hour at 330°F

Panel No.[a] 1219-		JM_1-P23[b]	FM_8-2P23[c]	FF-5	FE-6
Surface Treatment		A-M_1	A-M_8	B	C
Adhesive Batch No.		1	2	5	6
Postcure, hr/300°F		2	2	0	0
Tensile Shear Strength,[d] psi					
At room temperature		2720	3615	3555	3605
At 500°F, original		850	770	640	1020
At 500°F, after 500°F aging,	8 hr	895	1260	1690	1505
	48	920	1215	1545	1415
	72	1155	1290[e]	1390	1495
	120	1355	1380	1490	1115
	168	1035	-	-	-
	200	1015	630	1030	950

a) Bonds from solvent spread supported adhesive tape with glass fabric (112-Volan A) dried 15 min at 200°F. Formulation (by wt): 100 X-131 (lot No. LR1991-113) + 20 Formvar 15/95 E + 100 aluminum dust + 30 diaminodiphenylsulfone.
b) Bonds to full hard type 301 steel cured at contact pressure for one-half hour at 330°F.
c) X-131 resin lot No. LR3523-64.
d) Average of two or three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.).
e) One value.

Table 27. EFFECT OF POSTCURE ON THE SHEAR STRENGTH OF 100/20 X-131/FORMVAR 15/95 E BONDS TO TYPE 301 STEEL (FORMULATION NO. 1219)

Press cure at 100 psi for one-half hour at 240°F
plus one-half hour at 330°F

Panel No.[a] 1219	Surface Treatment	Adhesive Batch No.	Postcure (oven) hr	Postcure (oven) °F	Tensile Shear Strength,[b] psi Original 77°F	Tensile Shear Strength,[b] psi Original 500°F	Tensile Shear Strength,[b] psi Aged 200 hr at 500°F 500°F
FM_4-4P23	A-M_4	4[c]	2	300	2740	670	825
-4P43	"	"	4	"	2325	395	890
-4P83	"	"	8	"	2275	540	800
-4P24	"	"	2	400	1655	695	1020
-4P44	"	"	4	"	1535	670	1025
-4P84	"	4[c]	8	400	1560	720	1035
-4P½5	"	"	0.5	500	1705	685	585
-4P15	"	"	1	"	1340	775	715
-4P25	"	"	2	"	1675	740	1035
-4P45	"	"	4	"	975	485	795
FF-4P23	B	4[c]	2	300	3205	835	820
-4P83	"	"	8	"	2900	825	465
-4P24	"	"	2	400	2380	935	900
-4P84	"	"	8	"	2280	1055	935
-4P½5	"	"	0.5	500	2130	385	960
-4P45	"	"	4	"	735	840[e]	765
FE-9	C	9[d]	0	-	3630[e]	715	765
-9P427	"	"	4	270	3805[e]	740	615
-9P1627	"	"	16	"	3400[e]	755	505
-9P3227	"	"	32	"	3335[e]	640	480
-9P233	"	"	2	330	3160[e]	780	675
-9P433	C	9[d]	4	330	3470[e]	845	685
-9P833	"	"	8	"	3225[e]	835	550
-9P24	"	"	2	400	2700[e]	895	700
-9P84	"	"	8	"	2460[e]	1225	460
-9P164	"	"	16	"	1860[e]	1090	410

a) Bonds from solvent spread supported adhesive tape with glass fabric (112-Volan A) dried 15 min at 200°F. Formulation (by wt): 100 X-131 + 20 Formvar 15/95 E + 100 aluminum dust + 30 diaminodiphenylsulfone.
b) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.).
c) Experimental resin X-131, lot No. LR1991-113.
d) Experimental resin X-131, lot No. LR3523-147-1.
e) Average of two values.

Table 28. BEND STRENGTH OF X-131 ADHESIVE BONDS TO TYPE 301 STEEL

Cure at 100 psi: one-half hour at 330°F

Panel No.[a]	X-131/EPON 1009 Ratio, by wt	Formvar 15/95 E, phr	Curing Agent,[b] phr	Postcure, hr/°F	Bend Strength, lb Range	Bend Strength, lb Avg[c]	Shear Strength at 77°F,[c] psi
1145FM$_3$-18	100/0	-	6 dicy	None	106-116	110	2215[d]
1160FM$_3$-4P24	"	20	"	2/400	98-107	102	2240[d]
1219FM$_3$-3P24	"	"	30 DADPS	"	103-109	105	2480[d]
1219FE-9	"	"	" "	None	145-163	156	3630[e]
-9P833	"	"	" "	8/330	103-156	138	3225[e]
1219FE-9P44	100/0	20	30 DADPS	4/400	103-133	114	-
-9P84	"	"	" "	8/400	97-106	102	2460
1236FM$_3$-2P23	80/20	"	24.5 DADPS	2/300	117-123	120	2780[d]
1236FM$_4$-2P85	"	"	" "	8/500	42-57	48	1245
1236FE-9	"	"	" "	None	181-198	186	3900[e]
1236FE-9P833	80/20	20	24.5 DADPS	8/330	135-170	149	3560[e]
-9P44	"	"	" "	4/400	126-139	133	-
-9P84	"	"	" "	8/400	98-146	115	3015[e]
-10P44	"	"	" "	4/400	108-198	150[f]	2845
1236JE-19PaP44	"	"	" "	"	91-101	94	1870
1236JE-20P44	80/20	20	24.5 DADPS	4/400	68-81	75	2135
-20Pb$_2$P44	"	"	" "	"	57-96	80	1575
-20Pc$_1$P44	"	"	" "	"	81-94	88	1985
-20Pc$_2$P44	"	"	" "	"	91-101	91	2015

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust.

b) "Dicy" is dicyandiamide; "DADPS" is diaminodiphenylsulfone.

c) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.).

d) Average of six values.

e) Average of two values.

f) Average of four values.

Table 29. COMPARISON OF THE SHEAR STRENGTH OF TYPE 301 STEEL ADHESIVE SPECIMENS CUT WITH A DO-ALL BAND SAW AND A MILL SAW

Formulation No. 1219 (100/20 X-131/Formvar 15/95 E)
Cure at 100 psi: one-half hour at 240°F plus one-half hour at 330°F.
Postcure (oven): two hours at 300°F.

Panel No.[a]	$F1219FM_4$-4P23	$1219FM_4$-P23
Method of Cutting Specimens	Do-All Band Saw	Mill Saw
Tensile Shear Strength,[b] psi		
At room temp, average	2740	2725
, range	2530-2960	2180-2940
, No. of specimens	9	8
At 500°F, average	670	635
, range	440-850	430-740
, No. of specimens	6	5
At 500°F after 200 hr at 500°F, average	830	1355
, range	760-900	1260-1450
, No. of specimens	3	3

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Formulation No. 1236, batch 4 (parts by wt): 100 X-131 (lot No. LR1991-113) + 20 Formvar 15/95 E + 100 aluminum dust + 30 diaminodiphenylsulfone.
b) Shear strength for one-half inch lap joints, one inch in width, to half-hard, type 301 steel (thickness 0.050 in.). Surface treatment: Method A-M_4.

Table 30. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131/ GELVA C-3-V-30 ADHESIVES CURED WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi for one-half hour at 240°F
plus one-half hour at 330°F

Panel No.[a]	1201FE-6	1255FE	1256FE
X-131/Gelva C-3-V-30[b] Resin Ratio, by wt	100/0	83.3/16.7	75/25
Diaminodiphenylsulfone, phr	30	25	22.5
Tensile Shear Strength,[c] psi			
At room temperature	2420	3340	3715
At 500°F, original	1060	785	510
, after 500°F aging,[d] 8 hr	1165	495	545
, 48	1150	460	640
, 72	1220	665	430
, 120	1075	580	380
, 200	1115	320	0

a) Bonds from solvent spread supported tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient, by wt: 100 aluminum dust filler.
b) X-131 lot No.: LR-1991-113. Gelva C-3-V-30 is a carboxylic modified polyvinyl acetate resin.
c) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Average of two values for all aged specimens.

Table 31. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131/ GELVA C-3-V-30 ADHESIVES CURED WITH DIAMINODIPHENYLSULFONE AND BENZYLDIMETHYLAMINE PROMOTER

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a)]	X-131/ Gelva C-3-V-30 Weight Ratio[b)]	Curing Agent[c)] DADPS, by wt	Benzyl-dimethyl-amine, by wt	Postcure at 400°F, hr	Tensile Shear Strength,[d)] psi		
					Original		Aged 8 hr at 500°F
					77°F	500°F	500°F[e)]
1301FE	90/10	26	0.45	0	3130	890	865
-P24	"	"	"	2	1890	920	935
1304FE	83.3/16.7	24	.41	0	2320	420	240
-P24	"	"	"	2	3125	585	365
1255JE-2[e)]	"	"	"	0	3240[f)]	385[f)]	1035
1302FE	80/20	23	.40	0	1890	260	220
-P24	"	"	"	2	2030	525	270
1303FE	70/30	20.5	.35	0	2290	160	185
-P24	"	"	"	2	2630	225	200

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Solids content of adhesive dispersion used for coating: 75-80%w. Constant ingredient (by wt): 100 aluminum dust filler.
b) X-131 batch No. LR3523-147-1. Gelva C-3-V-30 is a carboxylic modified polyvinyl acetate resin.
c) DADPS is diaminodiphenylsulfone.
d) Average of two values for bonds to type 301 steel (1/2 H), thickness 0.050 in. Surface treatment: Method C.
e) Cure: one-half hour at 330°F. Shear strength at 500°F after 500°F aging: 120 hours, 1355 psi: 200 hours, 605 psi.
f) Average of three values.

Table 32. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131/GELVA C-3-V-30 ADHESIVES CURED WITH DICYANDIAMIDE

Press cure at 100 psi for one-half hour at 330°F

Panel No.[a)]	1145JM-17[b)]	1307JE	1308JE
X-131/Gelva C-3-V-30 Ratio, by wt	100/0	83.3/16.7	70/30
Tensile Shear Strength,[c)] psi			
At room temperature	2055	3005	3565
At 500°F, original	845	615	545
, after 500°F aging, 8 hr	365	395	425
, 120	115	965[d)]	895[d)]
, 200	460	490	45

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients (by wt): 6 dicyandiamide + 100 aluminum dust.
b) Bonds to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A.
c) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Average of two values.

Table 33. SUMMARY OF THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM FORMULATION NO. 1236, BATCHES 1 THROUGH 13

Formulation No. 1236 (parts by wt): 80 X-131 + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24.5 diaminodiphenylsulfone. Press cure at 100 psi for one-half hour at 330°F.

Panel No.[a] 1236-	Surface Treatment	X-131 Lot No. LR-	Adhesive Batch No.	Precure at 240°F, hr	Postcure at 400°F, hr	Tensile Shear Strength,[b] psi: Original 77°F	Original 500°F	At 500°F After 500°F Aging: 8 hr	120 hr	200 hr
FM_3-P24	A-M_3	1991-113	1	1/2	2	2780[c]	905	-	-	1145
FM_4-P24	A-M_4	"	"	"	"	2440	865	-	-	1350
-2	"	"	2	"	0	4200[c]	615	1280	1450	1360
-2P24	"	"	"	"	2	2370	940	-	-	-
FD-3	D	"	3	"	0	3875	350	1135	1425	1220
FE-3	C	1991-113	3	1/2	0	4095	675	1390	1440	1410
FF-3	B	"	"	"	"	4015	695	1370	1370	1190
FE-5	C	"	5	"	"	4155	685	1330	835	645
-6	"	3523-147-1	6	"	"	4155	645	-	-	535
-7	"	"	7	"	"	4020	750	-	-	660
FE-8	C	3523-147-1	8	1/2	0	4275	630	735	-	670
-8P24	"	"	"	"	2	3335	825	970	-	840
-8P44	"	"	"	"	4	3405	830	935	-	595
-9	"	"	9	"	0	3900	495	-	-	680
-9P24	"	"	"	"	2	3435	835	-	-	675
FE-10P44	C	3523-147-1	10	1/2	4	2765[c]	895	800	-	660[d]
FD-10P44	D	"	"	"	"	3410	-	-	-	-
FM_4-10P44	A-M_4	"	"	"	"	1710	-	-	-	-
-11P34	"	"	11	"	3	1570	-	-	-	-
FD-11P34	D	"	"	"	"	3310	-	-	-	-
FE-11P34	C	3523-147-1	11	1/2	3	2645	-	-	-	-
PE-11P34	"	"	"	"	"	2750	585	735	775	645
FE-12	"	"	12	"	0	3870	460	985	735	765
-13	"	"	13	"	"	3870	390	-	-	455
-13P34	"	"	"	"	3	2850	425	-	-	535
JE-13	C	3523-147-1	13	0	0	3310	235	1050	-	505
-13P44	"	"	"	"	4	2620	710	790	-	745

a) Bonds from solvent spread supported adhesive tapes (thickness 12-14 mils) with glass fabric (112-Volan A) dried 15 min at 200°F.

b) Average of two to four values for one-half inch lap joints to type 301 steel (thickness 0.050 in.).

c) Average of six values.

d) Exposure period 248 hr.

Table 34. SUMMARY OF THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM FORMULATION NO. 1236, BATCHES 14 THROUGH 27

Formulation No. 1236 (parts by wt): 80 X-131 + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24.5 diaminodiphenylsulfone. Press cure at 100 psi for one-half hour at 330°F.

Panel No.[a] 1236-	Surface Treatment	X-131 Lot No. LR-	Adhesive Batch No.	Precure at 240°F, hr	Postcure at 400°F, hr	Tensile Shear Strength,[b] psi				
						Original		At 500°F After 500°F Aging		
						77°F	500°F	8 hr	120 hr	200 hr
JE-14	C	1991-113	14	0	0	3635[c]	315[c]	1200	1530	1240[c]
-14P24	"	"	"	"	2	2610	870[c]	1290[c]	1460[c]	945[c]
-15	"	3523-64	15	"	0	3955	340	1075	1325	975
-15P24	"	"	"	"	2	3465	685	910	835	735
-16	"	3523-147-1	16	"	0	4200	350	965	995	825
JE-16P24	C	3523-147-1	16	0	2	3635	840	725	595	620
-18[d]	"	3523-64	18	"	0	2330	165	1070	1110	-
JM$_5$-18[d]	A-M$_5$	"	"	"	"	3715	170	820	865	-
JE-19P44[d]	C	3523-31	19	"	4	2465	635	925	990	900
JE-20	"	"	20	"	0	4030	170	940	960	1105
JE-20P44	C	3523-31	20	0	4	2730[c]	590[c]	805[c]	1020	990[c]
FE-20	"	"	"	1/2	0	4190[c]	280[c]	970	1190	980[c]
JE-21	"	3466-106	21	0	0	3795	180	1035	1200	935
-21P24	"	"	"	"	2	3085	665	945	880	760
-22	"	3466-109	22	"	0	915	0	945	575	295
JE-22P24	C	3466-109	22	0	2	2665	485	920	765	625
JM$_5$-23P24	A-M$_5$	3523-31	23	"	"	2205	585	705	790	705
JE-24	C	"	24	"	0	1010	0	1065	750	490
FE-24	"	"	"	1/2	"	3885	245	-	-	1310
-24P24	"	"	"	"	2	2230	760	1100	1145	1090
FE-25	C	1991-113	25	1/2	0	3325	345	1235	1115	975
-25P24	"	"	"	"	2	2265	770	1215	1110	800
-26	"	3523-31	26	"	0	3785	335	1090	1120	895
-26P24	"	"	"	"	2	2315	580	825	1055	805
-27	"	"	27	"	0	3370	615	1235	1220	1035
-27P24	"	"	"	"	2	2195	625	1200	1120	865

a) Bonds prepared with solvent spread supported adhesive tapes (thickness 12-14 mils) with glass fabric (112-Volan A) dried 15 min at 200°F.
b) Average of two or three values for one-half inch lap joints to type 301 steel (thickness 0.050 in.).
c) Average of five or six values.
d) Tape dried 30 min at 200°F; thickness 26 mils.

Table 35. SUMMARY OF THE 600°F SHEAR STRENGTH OF BONDS TO TYPES 301 AND 17-7 PH STEEL AND TO TITANIUM FROM X-131 RESIN ADHESIVES

Cure at 100 psi: one-half hour at 330°F.

Panel No.[a]	X-131/EPON 1009 Ratio, by wt	Formvar 15/95 E, phr	Curing Agent,[b] phr	Metal	Postcure,[c] hr/°F	Tensile Shear Strength at 600°F, psi Range	Avg[d]
1201FE-4	100/0	-	30 DADPS	301	-	0-330	195
1219FM$_4$-4P24	"	20	" "	"	2/400	0-200	125
-4P43	"	"	" "	"	4/300	170-200	185
-2P23	"	"	" "	"	2/300	310-380	335
1286FE	"	"	35 "	"	-	140-210	175
-P34	"	"	" "	"	3/400	0-200	95
1236FM$_4$-2	80/20	20	24 DADPS	301	-	250-380	330
"	"	"	" "	"	8/500	520-690	635
"	"	"	" "	"	200/500	520-580	445
-JE-14	"	"	" "	"	-	270-350	320
"	"	"	" "	"	8/600	420-490	455
1236JE-14	80/20	20	24 DADPS	301	120/600	660-790	740
"	"	"	" "	"	200/600	0	0
-FE-24P24	"	"	" "	"	2/400	200-310	245
-JE-20P44	"	"	" "	"	4/400	140-270	185
1287FE	"	"	29 "	"	-	360-400	375
1236JD-20	80/20	20	24 DADPS	Titanium	-	0	0
-20P44	"	"	" "	"	4/400	0-60	30
-JM$_5$-23P24(H)	"	"	" "	17-7 PH	2/400	410-520	470
1145FM$_4$-18	100/0	-	6 Dicy	301	-	40-70	55
1160FM$_4$-2	"	20	" "	"	-	80-110	95
1275FE-P34	100/0	-	10 Melamine	301	3/400	0-140	115
1276FE-P34	"	20	" "	"	"	60-120	90
-PE-P34	"	"	" "	"	"	0-200	140
1277PE-P34	80/20	"	" "	"	"	0-170	165
607FM$_4$-834	(Adhesive 422)			"	-	1650-1900	1810

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust. Details of cure conditions and metal treatments used are given in tables which summarize X-131 formulations cured with 1) DADPS, 2) dicyandiamide or, 3) melamine.

b) "DADPS" is diaminodiphenylsulfone. "Dicy" is dicyandiamide.

c) The data include tests results for specimens aged for prolonged periods at 500°F and 600°F. The aging is designated as a "postcure" to simplify construction of the table.

d) Average of three values for one-half inch lap joints. Thickness of metal adherends: 0.050 in.

Table 36. EFFECT OF ELEVATED TEMPERATURE AGING ON THE SHEAR STRENGTH OF FORMULATION NO. 1236 BONDS TO TYPE 301 STEEL

Formulation (by wt): 80 X-131 + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24.5 diaminodiphenyl-sulfone

Cure at 100 psi: one-half hour at 330°F

Panel No.[a]		1236FM$_4$-2[b]	1236JE-14	1236JE-18
Adhesive Batch No.		2	14	18[c]
X-131 Resin Lot No. LR-		1991-113	1991-113	3523-64
Tensile Shear Strength,[d] psi				
At room temp, original		4215	3970	2330
, after 500°F aging,	8 hr	1245	-	-
	, 200	1800	-	-
At -70°F, original		3680	-	-
, after 500°F aging,	8 hr	1480	-	-
At 500°F, original		620	320	165
, after 500°F aging,	8 hr	1280	1335	1070
	, 48	1345	-	-
	, 72	1335	-	-
	, 120	1450	1530	1110
	, 168	1345	-	-
	, 200	1360	1350	-
	, 300	890	-	815
	, 400	1090	-	520
	, 500	905	-	-
	, 600	440	-	-
	, 700	145	-	-
	, 800	0	-	-
At 600°F, original		330	320	-
, after 500°F aging,	8 hr	635	-	-
	, 200	545	-	-
, after 600°F aging,	8	-	455	-
	, 120	-	740	-
	, 200	-	0	-

a) Bonds prepared with solvent spread adhesive tapes (thickness 12 mils) with glass fabric 112-Volan A dried 15 min at 200°F.
b) Bonds precured for one-half hour at 100 psi and 240°F. Surface treatment: Method A-M$_4$.
c) Adhesive tape dried 30 min at 200°F; thickness 26 mils.
d) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.

Table 37. LONG TIME LOAD STRENGTH AT 500° F OF X-131 ADHESIVE BONDS TO TYPE 301 STEEL

Press cure at 100 psi for one-half hour
at 240°F + one-half hour at 330°F

Exp No.	Specimen No.[a]	X-131/EPON 1009/ Formvar 15/95 E Ratio,[b] by wt	Postcure at 400°F, hr	Load, psi	Time to Failure, hr[c]
11	1201FE-4	100/0/0	0	240	215.4 (no break)
12[d]	1219FM$_4$-4P44	100/0/20	4	240	215.4 (no break)
1[d]	"	"	"	400	25.3
18[d]	-4P84	"	8	400	280.8 (no break)
26[d]	"	"	"	500	257.2 (no break)
27[d]	"	"	"	600	208.6
22	1236FE-10P44	80/20/20	4	400	484.2 (no break)
16	"	"	"	500	9.6
23	"	"	"	500	155.8
17	"	"	"	600	55.4

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients: 100 phr aluminum dust + 24.5 to 30 phr diaminodiphenyl sulfone. Bonds were one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
b) X-131 resin lot No. LR3523-147-1.
c) No evidence of creep was observed.
d) Surface treatment used with Formulation No. 1219: Method A-M$_4$. X-131 resin lot No. LR1991-113.

Table 38. PROPERTIES OF EXPERIMENTAL RESIN X-131

Resin Lot No. LR-	Epoxy, eq/100 g	Hydroxyl, eq/100 g	Chlorine, %w	Mol Wt	Durran Softening Point, °F
1991-113	0.485	0.113	0.57	667	170
3523-20	.485	.092	.77	677	160
-31	.502	.086	.56	663	155
-64	.489	.102	.70	721	170
-115	.466	.143	1.23	823	174
-147-1	.464	.130	1.29	696	160
3466-106[a)]	.465	.092	1.31	640	156
-109[a)]	.499	.048	.47	732	176

a) Laboratory scale preparation.

Table 39. COMPARISON OF THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM FORMULATION NO. 1236 PREPARED WITH VARIOUS X-131 RESIN LOTS

Supported adhesive tape with 112-Volan A glass fabric (parts by wt): 80 X-131 + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24 diaminodiphenylsulfone
Press cure at 100 psi for one-half hour at 330°F[a)]

X-131 Resin Lot No. LR-	Number of Adhesive Batches	Number of Experiments	Postcure 2-4 hr at 400°F	Tensile Shear Strength,[b)] psi at 77°F Range	Avg	at 500°F Range	Avg	At 500°F After 8 hr at 500°F Range	Avg	At 500°F After 200 hr at 500°F Range	Avg
1991-113	5	7	None	3325-4215	3950	320-695	530	1135-1390	1295	645-1410	1165
3523-147-1	6	7	"	3310-4275	3940	235-645	460	735-1050	910[c)]	455-825	635
3523-64	2	3	"	2330-3955	3335	165-340	225	820-1075	990	865-1000	940
3523-31	4	5	"	3370-4190	3850	170-615	330	940-1235	1060[c)]	895-1310	1065
1991-113	4	6	Yes	1940-2780	2420	770-940	870	1215-1335	1265[d)]	790-1350	1040[e)]
3523-147-1	6	8	"	2620-3435	3110	425-895	745	725-935	745[f)]	535-840	640
3523-64	1	1	"	-	3465	490-685	565[d)]	-	910	-	735
3523-31	6	7	"	1830-3130	2340	550-760	625	705-1200	930	705-1090	905

a) Some of the bonds were precured at 100 psi for one-half hour at 240°F.
b) Two or usually three specimens were tested at each condition in each experiment.
c) Average for four experiments.
d) Average for three experiments.
e) Average for five experiments.
f) Average for six experiments.

Table 40. EFFECT OF POSTCURE ON THE SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E ADHESIVE BONDS TO TYPE 301 STEEL (FORMULATION NO. 1236)[a]

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Postcure (oven)		Tensile Shear Strength, psi[b]		
		Original		Aged 200 hr at 500°F
hr	°F	at 77°F[c]	at 500°F	at 500°F
None	-	3900	495	680
4	270	4250	585	650
16	"	4025	610	535
32	"	3860	670	530
2	330	3670	675	875
4	"	3700	735	715
8	"	3560	775	640
2	400	3435	835	675
8	"	3015	1065	805
16	"	2935	875	445

a) Formulation No. 1236, batch No. 9 (parts by wt): 80 X-131 (lot No. LR 3523-147-1) + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24.5 diaminodiphenylsulfone. Bonds prepared from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F.

b) Average of three values for one-half inch lap joints to half-hard type 301 steel. Surface treatment: Method C.

c) Average of two values.

Table 41. EFFECT OF METAL SURFACE TREATMENT ON THE SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E BONDS TO TYPE 301 STEEL

Formulation No. 1236
Cure at 100 psi: one-half hour at
240°F plus one-half hour at 330°F

Panel No.[a]	$1236FM_4$-2[e]	1236FD-3	1236FE-3	1236FF-3
Surface Treatment[b]	A-M_2	D	C	B
Tensile Shear Strength,[c] psi				
At room temperature	4215	3875	4095	4015
At 500°F	620	350	675	695
At 500°F[d] after				
500°F aging, 8 hr	1280	1135	1390	1370
48	1345	1330	1480	1240
72	1335	1570	1400	1420
120	1450	1425	1440	1370
200	1360	1320	1410	1190

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 minutes at 200°F. Formulation No. 1236, Batch No. 3 (by wt): 80 X-131 (lot No. LR1991-113) + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24.5 diaminodiphenylsulfone.
b) See the last section of this report for description of methods of surface preparation.
c) Average of three values for one-half inch lap joints to half-hard, type 301 steel (thickness 0.050 in.).
d) Average of two values except for Panel No. $1236FM_4$-2 where three specimens were tested for each test condition.
e) Formulation No. 1236, Batch No. 2 was used for bonding.

Table 42. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM AN 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E ADHESIVE TAPE WITH STAINLESS STEEL CARRIER

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]	1236FF-3	1236FF-4
Formulation No. 1236, Batch No.	3	4
Type of Carrier	Glass Cloth 112-Volan A	Stainless Steel[b]
Tensile Shear Strength,[c] psi		
At room temperature[d]	4015	3500
At 500°F, original[d]	695	305
, after 500°F aging, 8 hr	1370	1105
48	1240	1265
72	1420	1295
120	1370	905
200	1190	955

a) Bonds from solvent spread supported tape dried 15 min at 200°F. Formulation (by wt): 80 X-131 (lot No. LR1991-113) + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24.5 diaminodiphenylsulfone.
b) Woven wire, type 304, 30 mesh; wire diameter 0.0065 in.
c) Average of two values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method B.
d) Average of three values.

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Table 43. EFFECT OF SALT SPRAY EXPOSURE ON THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM FORMULATION NO. 1236

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a] 1236	Adhesive[b] Batch No.	Surface Treatment	Postcure at 400°F, hr	Tensile Shear Strength at Room Temp, psi			
				Original		After 30 Days' Salt Spray	
				Range	Avg[c]	Range	Avg[d]
1) FE-10P44	10	Method C (FPL) formalin-peroxide followed by passivation	4	2400-2560	2480	2540-2920	2715
2) "	"		"	2920-2930	2925	1800-2370	2110
1) FE-11P34	11		3	1960-3190	2570	980-1560	1255
2) "	"		"	2510-3120	2815	960-1850	1395
1) FD-10P44	10	Method D (Franklin Institute) chromic-sulfuric acids	4	2390-3490	2940	1610-2000	1850
2) "	"		"	3040-3890	3465	1420-1980	1670
1) FD-11P34	11		3	3250-3400	3325	2140-2700	2430
2) "	"		"	3240-3350	3295	2140-2640	2465
1) FM_4-10P44	10	Method A-M_4 sulfuric; HNO_3-HF	4	1480-1660	1570	760-1340	975
2) "	"		"	1740-1960	1850	880-1390	1180
1) FM_4-11P34	11		3	1530-1740	1635	650-920	790
2) "	"		"	1410-1590	1500	840-1260	1060

a) Bonded panels (10.5 x 8.0 in.) with one-half inch lap-joints; half-hard type 301 steel (thickness 0.050 in.).

b) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Formulation No. 1236 (parts by wt): 80 X-131 (lot No. LR-3523-147-1) + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24 diaminodiphenyl sulfone.

c) Average of two values.

d) Average of five values.

Table 44. COMPARISON OF THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM FORMULATION NO. 1236 ADHESIVE TAPES PREPARED WITH METHYLAL AND ACETONE

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]	1236FE-12	1236FE-8
Methylal, parts by wt	53	0
Acetone, parts by wt	25	53
1/9 MEK/THF,[b] parts by wt	200	200
Tensile Shear Strength, psi[c]		
At room temperature	3870	4275
At 500°F, original	460	630
, after 500°F aging, 8 hr	985	735
, 72	920[d]	-
, 120	1140[d]	-
, 200	765	670

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried one hour at room temperature and 15 min at 200°F. Formulation, parts by wt: 80 X-131 (lot No. LR3523-147-1) + 20 EPON 1009 + 20 Formvar 15/95 E + 24 diaminodiphenylsulfone + 100 aluminum dust. Solvent concentration based on 80/20/20 X-131/EPON 1009/Formvar 15/95 E.

b) MEK is methyl ethyl ketone; THF is tetrahydrofuran.

c) Average of three values for bonds to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.

d) One value.

Table 45. EFFECT OF METHOD OF BOND PREPARATION ON THE SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL

Panel No.	1236PE-11P34		1236PE-11P335		1236FE-10F44	
Adhesive batch no.	11		11		10	
Cure at 100 psi	75 to 400°F in 30 min		75 to 350°F in 30 min		30 min at 240°F 30 min at 330°F	
Postcure, hr/°F	Press - 3/400		Press - 3/350		Oven - 4/400	
Tensile shear strength,b) psi	Range	Avg	Range	Avg	Range	Avg
At room temp	2230-3100	2750	3560-3880	3665	2110-3410	2845
At 500°F, original	500-660	585	580-740	655	700-1000	895
, aged at 500°F, 8 hr	650-850	735	680-790	745	720-890	800
, 72 hrc)	500-740	615	730-1120	925	-	-
, 120 hrc)	660-890	775	360-670	515	-	-
, 200 hr	610-680	645	550-700	605	-	-
, 248 hr	-	-		-	650-670	660

a) Bonds from solvent spread supported adhesive tapes (112-Volan A glass fabric) dried 15 min at 200°F. Formulation, parts by wt: 80 X-131 (lot no. 3523-147-1) + 20 EPON 1009 + 20 Formvar 15/95 E + 24 diaminodiphenyl sulfone + 100 aluminum dust.
b) Test results for three bonded specimens to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
c) Test results for two specimens.

Table 46. EFFECT OF X-131 RESINOUS PRIMERS ON THE SHEAR STRENGTH OF FORMULATION NO. 1236 BONDS TO TYPE 301 STEEL

Press cure at 100 psi: one-half hour at 330°F
Postcure (oven): four hours at 400°F

Panel No.[a]	Adhesive Batch No.	Primer Solution Composition, Parts by Weight: % Solids in Acetone	X-131	EPON 1009	DADPS[b]	Primer Drying Conditions, 1 hr Air Dry Min/°F	Tensile Shear Strength,[c] psi: Original 77°F	Original 500°F	At 500°F After 500°F Aging for 8 hr	200 hr	Bend Strength,[c] lb
1236JE-19PaP44	19[d]	0.1	100	-	30	10/167	1870	645	875	860[e]	94
-19P44	"	-	-	-	-	-	2465	635	925	900	-
1236JE-20PbP44	20[f]	0.01	100	-	31	15/200	1575	545	775	840[e]	79
-20Pc1P44	"	"	80	20	25	-	1985	650	810	920[e]	87
-20Pc2P44	"	"	"	"	"	15/200	2015	585	800	835[e]	91
-20P44	"	-	-	-	-	-	1830	550	715	905[e]	75

a) Bonds from solvent spread supported adhesive tape with glass fabric carrier (grade 112-Volan A). Formulation No. 1236, parts by weight: 80 X-131 (lot No. LR 3523-31) + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 25 diaminodiphenylsulfone.
b) DADPS is diaminodiphenylsulfone.
c) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Adhesive tape thickness: 26 mils.
e) Average of two values.
f) Adhesive tape thickness: 12 mils.

Table 47. ROOM TEMPERATURE STORAGE STABILITY OF X-131 SUPPORTED ADHESIVE TAPES WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi for one-half hour
at 240°F plus one-half hour at 330°F

Panel No.[a]	X-131/EPON 1009 Ratio, by wt	Formvar 15/95 E, phr	Postcure hr/°F	Age of Tape, Month	Tensile Shear Strength,[b] psi				
					Original		At 500°F After 500°F Aging		
					77°F	500°F	8 hr	120 hr	200 hr
1201JM-P23[c]	100/0	0	2/300	Fresh	2255	1075	-	-	780
-FM3-P23	"	"	"	2.4	1410	585	-	-	535[d]
1219JM1-P23[c]	100/0	20	2/300	Fresh	2720	850	-	-	1015
-FM4-P23	"	"	"	1.6	2825	495	-	-	665[d]
1236FE-3[e]	80/20	20	0	Fresh	4095	675	1390[d]	1440[d]	1410[d]
-3b[e]	"	"	"	8.2	3380	305	1105	535[d]	200
-FM3-P24	"	"	2/400	Fresh	2780	905	-	-	1145
-FM4-P24	"	"	"	0.3	2440	865	-	-	1350[d]

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients, phr: 100 aluminum dust + 30 diaminodiphenylsulfone.
b) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A.
c) Bonds prepared with full hard type 301 steel. Cure (contact pressure): one-half hour at 330°F.
d) Average of two values.
e) Surface treatment: Method C.

Table 48. SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E ADHESIVE BONDS TO TYPE 17-7 PH STEEL (FORMULATION NO. 1236)

Press cure at 100 psi for one-half hour at 330°F

Panel No.[a] 1236-	Steel[b]		Surface Etchant	Postcure[c] at 400°F, hr	Tensile Shear Strength,[d] psi				
					Original		At 500°F After 500°F Aging		
	Type	Thickness, in.			77°F	500°F	8 hr	120 hr	200 hr
F-13[e]	17-7 PH	0.062	None	0	3880	270	-	-	515
-13P34[e]	"	"	"	3	2370	535	-	-	0
J-13P34	"	"	"	3	2520	610	1005	205	285
-14P34	"	"	"	3	-	815	1070	-	460
JE-18	301	.050	C	0	2330	165	1070	1110	-
JE-18	17-7 PH	.062	C	0	4425	230	1095	1340	-
-18P84	"	"	"	8	-	575	-	-	-
JM$_5$-18	"	"	A-M$_5$	0	3815	180	1105	1060	
-18P84	"	"	"	8	-	685	-	-	-
-18	301	.050	"	0	3715	170	820	865	-
JM$_5$-23P24	17-7 PH	.062	A-M$_5$	2	3505	915	1250	1220	885
-23P24(H)[f]	17-7 PH (Heat treated in helium)	.050	"	2	3205	810	1280	1100	955
-23(H)	" (Heat treated in helium)	"	"	0	2505	255	-	-	730
-23P24	301	"	"	2	2205	585	700	790	705

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Formulation No. 1236, batches 13, 14, 18 and 23 (parts by wt): 80 X-131 + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24 diaminodiphenylsulfone.

b) Armco 17-7 PH steel, condition TH 1050, with vapor blast finish. Half-hard type 301 steel with 2B finish.

c) Oven postcure at 400°F (no pressure).

d) Average of two or three values for single one-half inch lap joints.

e) Bonds "precured" at 100 psi for one-half hour at 240°F.

f) Shear strength at 600°F: 470 psi.

Table 49. SHEAR STRENGTH OF BONDS TO TITANIUM (C-110 M ALLOY) FROM 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E ADHESIVE (FORMULATION NO. 1236)

Press cure at 100 psi: one-half hour at 330°F

Panel No.[a] Metal (thickness 0.050 in.) Postcure, hr/°F		1236JD-20 Titanium[b] -	1236JD-20P44 Titanium[b] 4/400	1236JE20-P44 301 Steel[c] 4/400
Tensile Shear Strength,[d] psi				
At room temperature		2275	770[e]	1830
At -70°F		1465	625	-
At 500°F, original		165	215	550
, after 500°F aging,	8 hr	695	335	715
	, 48	525	355	-
	, 120	565	265	-
	, 168	365	295	-
	, 200	395	385	905[e]
Bend Strength, lb		114	35	-

a) Formulation No. 1236, batch No. 20 (parts by wt): 80 X-131 (lot No. LR3523-31) + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 25 diaminodiphenylsulfone. Bonds prepared with solvent spread supported tape (112-Volan A) dried 15 min at 200°F.
b) C-110 M titanium alloy. Surface treatment: Method D.
c) Conforms to MIL-S-5059; Micro Rold, half-hard, condition C, composition D. Surface treatment: Method C.
d) Average of three values for one-half inch lap joints.
e) Average of two values.

Table 50. *EFFECT OF DIAMINODIPHENYLSULFONE CONCENTRATION ON THE SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E BONDS TO TYPE 301 STEEL*

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]	Postcure at 400°F, hr	DADPS,[b] phr	Tensile Shear Strength,[c] psi		
			Original		Aged 200 hr at 500°F
			77°F	500°F	500°F
1257FE	0	12	1195	0	325
-P24	2	"	2780[d]	70	-
-P44	4	"	1605	340	-
1258FE	0	16	1030	0	325
-P24	2	"	1370[d]	350	-
-P44	4	"	1980	595	-
1259FE	0	20	2010	335	1045
-P24	2	"	3380[d]	865	-
-P44	4	"	2610	1025	-
1236FE-5	0	24.5	4155	645	645
-3	0	"	4095	675	1410
-9[e]	0	"	3900	495	680
-P24[e]	2	"	3435	835	675
1269FE	0	29	4310	1010	1325
1287FE[e]	0	"	4100	535	635[f]

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust filler. X-131 resin lot No. LR1991-113.
b) DADPS is diaminodiphenylsulfone.
c) Average of two or three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) One value.
e) X-131 resin lot No. LR3523-147-1.
f) Exposure period at 500°F was 248 hr.

Table 51. EFFECT OF ALUMINUM FILLER AND DIAMINODIPHENYLSULFONE CONCENTRATIONS ON THE SHEAR STRENGTH OF X-131/FORMVAR 15/95 E BONDS TO TYPE 301 STEEL

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]	X-131/ EPON 1009 Ratio, by wt	X-131 Lot No. LR	DADPS,[b] phr	Al Dust, phr	Tensile Shear Strength, psi[c]					
					Original		At 500°F After 500°F Aging for			
					77°F	500°F	8 hr	120 hr[d]	200 hr	248 hr
1219FF-5[e]	100/0	1991-113	30	100	3555	640	1690[d]	1490	1030[d]	-
1219FE-9	"	3523-147-1	"	"	3630[d]	715	-	-	765	-
1286FE	"	"	35.5	"	3830	740	995	955[f]	-	630
1288FE	"	"	"	120	3855	645	1015	1060	775[d]	-
1251FF[e]	"	1991-113	30	"	4015	1285	1530[d]	1230	1095[d]	-
1251FE-2	"	3523-31	"	"	2755	255	1370	710	445	-
1236FE-5	80/20	1991-113	24.5	100	4155	645	1330	835	645[d]	-
1236FE-20	"	3523-31	"	"	4250	245	970	1190	1065	-
1236FE-9	"	3523-147-1	"	"	3900[d]	495	-	-	680	-
1332FE	"	3523-31	"	120	3830	305	1115	1170	1135	-
1269FE	"	1991-113	29	100	4310[d]	1010	-	-	1325	-
1287FE	"	3523-147-1	"	"	4100	535	855	720[f]	-	635
1289FE	"	"	"	120	4050	780	775	520	670	-

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 20 parts by weight Formvar 15/95 E.
b) "DADPS" is the curing agent diaminodiphenylsulfone.
c) Average of three values for bonds to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Average of two values.
e) Surface treatment: Method B.
f) Exposure period at 500°F was 139 hr.

Table 52. EFFECT OF BISPHENOL-A ON THE SHEAR STRENGTH OF 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E BONDS TO TYPE 301 STEEL

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]	Bisphenol-A phr	Tensile Shear Strength, psi[b]						
		Original		At 500°F After 500°F Aging For[c]				
		77°F	500°F	8 hr	48 hr	72 hr	120 hr	200 hr
1236FE-5	0	4155	645	1330	1120	950	835	645
1263FE	2	4205	775	1485	1230	1155	850	845
1264FE	5	4140	785	1365	1115	890	1130	1025
1265FE	8	4215	420	900	780	535	540	300
1266FE	10	4065	375	1275	605	820	735	460

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 minutes at 200°F. Constant ingredients (parts by wt): 80 X-131 (lot No. LR1991-113) + 20 EPON 1009 + 20 Formvar 15/95 E + 100 aluminum dust + 24.5 diaminodiphenylsulfone.
b) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
c) Average of two values for all aged bonds.

Table 53. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E ADHESIVES CURED WITH DICYANDIAMIDE DISPERSED IN THE RESIN SOLUTION

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a] Postcure (oven), hr/°F		1272FE None	1272FE-P34 3/400
Tensile Shear Strength,[b] psi			
At room temperature		2590	3790[c]
At 500°F, original		0	200
, after 500°F aging,[d]	8 hr	910	
,	48	690	-
,	72	1175	-
,	120	885	-
,	200	930	

a) Bonds from solvent spread supported adhesive tape (glass cloth 112-Volan A) dried 15 min at 200°F. Formulation, phr: 100 aluminum dust + 6 dicyandiamide. Dicyandiamide not dissolved but dispersed by trituration with the resin solutions.

b) Average of three to four values for bonds to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.

c) Average of seven values.

d) Average of two values for all aged specimens.

Table 54. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM 80/20/20 X-131/EPON 1009/FORMVAR 15/95 E ADHESIVES CURED WITH MELAMINE

Press cure at 100 psi with dispersed melamine

Panel No.[a]	1273FE-P333[b]	1277FE-P34	1277PE-P34	1277JE-2P34	1277PE-2P34
Melamine, phr	10	8.3	8.3	8.3	8.3
Cure Temperature, min/°F	30/240 + 30/330	30/240 + 30/330	30/80-400[c]	30/330	30/80-400[c]
Postcure, Method	Oven	Oven	Press	Oven	Press
, hr/°F	3/330	3/400	3/400	3/400	3/400
Tensile Shear Strength,[d] psi					
At room temperature	2660	1540	3315	-	3705
At 500°F, original	135	270	325	-	380
, after 500°F aging, 8 hr	465	460	555	435[e]	-
, 200	1055	140	650[f]	325[e]	700

a) Bonds from solvent spread supported adhesive tapes with glass fabric (112-Volan A) dried 15 min at 200°F. Constant ingredient, 100 phr aluminum dust. X-131 resin lot No. LR3523-147-1.
b) X-131 resin lot No. LR1991-113.
c) Time-temperature cycle: 80 to 300°F in 15 min; 300 to 400°F in 10 min.
d) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
e) Average of 10 values; range 0 to 900 psi.
f) Exposure period 248 hours at 500°F. Shear strength at 500°F after 139 hr at 500°F: 825 psi.

Table 55. IDENTIFICATION AND PROPERTIES OF EXPERIMENTAL MODIFIED X-131 RESIN

Resin Lot No. LR	Batch Scale	Type	Code	Epoxy, eq/100 g	Hydroxyl eq/100 g	Mol Wt	Durran Softening Point, °F
1991-113	Pilot Plant	Unmodified	A	0.485	0.113	667	170
3523-20	" "	"	B	.485	.092	677	160
-64	" "	"	C	.489	.102	721	170
2224-31A	Laboratory	Modified B	D	.341	.21	1140	214
-31B	"	"	E	.325	.21	1500	325
3490-49	"	"	F	-	-	-	215
3687-6	"	Modified C	G	.377	.174	977	226

Table 56. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM EXPERIMENTAL MODIFIED X-131 RESIN CURED WITH DICYANDIAMIDE

Press cure at 100 psi for one-half hour
at 240°F plus one-half hour at 330°F

Panel No.[a]	1217JM[b]	$1218JM_1$-P23[b]	$1224FM_3$	$1226FM_3$
Modified X-131 Resin, Batch No.[c]	E	D	G	F
Type 301 Steel	Full Hard	Full Hard	Half-Hard	Half-Hard
Postcure (oven), hr/°F	None	2/300	None	None
Dicyandiamide, phr	6	4.5	4.5	6
Formvar 15/95 E, phr	0	0	20	20
Tensile Shear Strength,[d] psi				
At room temperature	1280	1665	3525	1995
At 500°F, original	400	635	320	0
, after 500°F aging, 8 hr	230	340	135[e]	30[e]
, 48	175	390	415[e]	415[e]
, 72	195	430	475[e]	415[e]
, 120	225	595	720[e]	530[e]
, 168	325	615	-	-
, 200	305	645	365[e]	250[e]

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust.
b) Bonds cured at contact pressure for one-half hour at 330°F.
c) See Table 55 for identification of the experimental resin.
d) Average of three values for one-half inch lap joints to type 301 steel (thickness 0.050 in.). Surface treatment: Method A.
e) Average of two values.

Table 57. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM EXPERIMENTAL MODIFIED X-131 RESIN CURED WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi for one-half hour at 330°F

Panel No.[a]	$1225FM_3$-P23[b]	$1219FM_3$-2P23[b]	1309JE	1309JE-P24	1236JE-15	1236JE-15P34
X-131 Resin[c]	Modified(G)	Unmodified(C)	Modified(G)	Modified(G)	Unmodified(C)	Unmodified(C)
X-131/EPON 1009 Ratio, by wt	100/0	100/0	80/20	80/20	80/20	80/20
Diaminodiphenylsulfone, phr	23	30	19	19	24	24
Postcure (oven), hr/°F	2/300	2/300	0	2/400	0	2/400
Tensile Shear Strength,[d] psi						
At room temperature	3520	3615	4050	3815	3955	3465
At 500°F, original	465	770	105	645	340	685
, after 500°F aging, 8 hr	1225[e]	1260[e]	1300	655	1075	910
, 48	975[e]	1215[e]	-	-	-	-
, 72	655[e]	1290[f]	-	-	-	-
, 120	1240[e]	1380[e]	1140[e]	850[e]	1325[e]	835[e]
, 200	1000[e]	630[e]	700	750	975	735

a) Bonds from solvent spread supported adhesive tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients, phr: 20 Formvar 15/95 E + 100 aluminum dust.
b) Bonds were precured one-half hour at 240°F at 100 psi. Surface treatment of metal: Method A-M_3.
c) See Table 55 for identification of the experimental resin.
d) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
e) Average of two values.
f) One value.

Table 58. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM EXPERIMENTAL RESIN X-141 CURED WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi for one-half hour
at 240°F plus one-half hour at 330°F

	Tensile Shear Strength,[a] psi		
Panel No.[b]	1282FE	1283FE	1284FE
X-141/EPON 1009 Resin Ratio, by weight	100/0	100/0	80/20
Formvar 15/95 E, phr	0	20	20
Diaminodiphenylsulfone, phr	33	33	27
At room temperature	2435	3270	3045
At 500°F, original	750	815	185
, after 500°F aging, 8 hr	845	865	895
, 139[c]	920	1180	880
, 248[c]	515	770	385
At 600°F	460	475	125

a) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.

b) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust.

c) Average of two values.

Table 59. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM EXPERIMENTAL RESINS LR2047-76 AND LR2047-83 CURED WITH DIAMINODIPHENYLSULFONE

Cure at 100 psi: one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]		1270FE	1201FE-6	1271FE
Resin		LR2047-76	X-131 LR1991-113	LR2047-83
Diaminodiphenylsulfone, phr		38	30	31
Adhesive Form		Tape[b]	Tape[b]	Hot Melt
Tensile Shear Strength,[c] psi				
At room temperature		1705	2420	2045
At 500°F, original		1045	1060	245[e]
, after 500°F aging,[d]	8 hr	1080	1165	495
,	48	1480	1150	200
,	72	1295	1220	355
,	120	890	1075	220
,	200	540	1115	0

a) Bonds with glass fabric, 112-Volan A. Constant: 100 phr aluminum dust filler.
b) Solvent spread tape dried 15 min at 200°F.
c) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Average of two values for all aged specimens.

Table 60. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM EXPERIMENTAL RESIN LR2047-98 CURED WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.[a]		1278FE	1278FE-P44	1285FE	1285FE-P24
LR2047-98/EPON 1009 Ratio, by wt		100/0	100/0	80/20	80/20
Formvar 15/95 E, phr		0	0	20	20
Diaminodiphenylsulfone, phr		30	30	24	24
Postcure, hr/°F		None	4/400	None	2/400
Tensile Shear Strength,[b] psi					
At room temperature		2040	1230	3800	2635
At 500°F, original		1225	890	945	905
, after 500°F aging,	8 hr	820	-	1155	1155
	, 139[c]	955	-	350	730
	, 248[c]	985	480	840	420
At 600°F		495	-	400	445

a) Bonds from solvent spread supported adhesive tapes (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust.

b) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.

c) Average of two values.

Table 61. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM EXPERIMENTAL RESIN LR2047-126 CURED WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi: 30 min from 80°F to 400°F plus 3 hours at 400°F

Panel No.a)	LR2047-126 /EPON 1009, by wt	Formvar 15/95 E, phr	DADPS,b) phr	Tensile Shear Strength,c) psi				
				Original			At 500°F After 500°F Aging	
				77°F	500°F	600°F	8 hr	200 hr
1295PE-P34	100/0	0	35	1940	1330	465	1460	315
1293PE-P34	"	20	"	3010	285	320d)	1280	395
-2P34	"	"	"	2725	265	-	1245	390
1294PE-P34	80/20	"	28	3180	295	510	1040	935
-2P34	"	"	"	2420	210	-	1025	775

a) Bonds from solvent spread supported tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredient: 100 phr aluminum dust.
b) "DADPS" is diaminodiphenylsulfone.
c) Average of two or three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Range of values: 140 to 660 psi.

Table 62. SUMMARY OF THE 600°F SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM NEW EXPERIMENTAL RESINS

Cure at 100 psi: one-half hour at 330°F with diaminodiphenylsulfone.a)

Panel No.b)	Resins, by wt				Formvar 15/95 E, phr	Postcure, hr/°F	Tensile Shear Strength at 600°F, psi	
	Experimental		X-131	EPON 1009			Range	Avgc)
1279JE-P333	100	X-801-3	0	0	-	3/330	90-170	135
1280JE-P333	"	"	"	"	20	"	0-110	55
-P333HM	"	"	"	"	"	"	0-75	50
1281JE-P333	80	"	"	20	"	"	60-80	65
1290PE-P34	10	"	72	18	-	3/400	30-100	65
1291PE-P34	40	X-801-3	48	12	-	3/400	130-170	150
1292PE-P34	60	"	32	8	-	"	0-80	40
1282FE	100	X-141	0	0	-	-	370-540	460
1283FE	"	"	"	"	20	-	444-540	475
1284FE	80	"	"	20	"	-	110-160	125
1278FE	100	LR2047-98	0	0	-	-	430-580	495
1285FE	80	"	"	20	20	-	340-430	400
-P24	"	"	"	"	"	2/400	420-490	445
1293PE-P34	100	LR2047-126	"	"	"	3/400	140-660	320
1294PE-P34	80	"	"	"	"	"	460-600	510
1295PE-P34	100	LR2047-126	0	0	-	3/400	300-580	465
1329JE-2	10	LR2047-199	90	"	-	-	120-270	170
1330JE-2	30	"	70	"	-	-	50-90	65
1331JE-2	50	"	50	"	-	-	60-180	105
607JM5-1112	(Adhesive 422)				-	-	1530-1670	1615

a) The curing agent was used at a concentration calculated on the basis of one amine hydrogen per epoxide group. Filler was 100 phr aluminum dust.

b) Bonds prepared with glass fabric (112-Volan A). Details of bond preparation including adhesive form, cure and complete formulations are given in the tables which summarize the formulations containing the experimental resins.

c) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.

Table 63. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM RESINS CURED WITH COMBINATIONS OF PYROMELLITIC DIANHYDRIDE AND MALEIC ANHYDRIDE

Panel No.[a]	Resins, Parts by Weight		Formvar 15/95 E, phr	Filler,[b] phr	Curing Agent,[c] phr		Cure hr/°F	Tensile Shear Strength,[d] psi				
								Original		At 500°F After 500°F Aging		
	Experimental	EPON 1009			PMDA	MA		77°F	500°F	8 hr	120 hr	200 hr
1318E-2	80 X-801-3	20	-	100 Al dust 5 Cab-O-Sil	34	31	16/320	1815	610	805	935	845
1320E[e]	100 X-801-3	-	20	100 Al dust	39	35	"	1680	580	1015	435	365
1322JE	100 X-801-3	-	"	10 glass fibers	"	"	0.5/330	2515	205	765	665	345
1325JE	100 X-801-3	-	-	10 glass fibers 10 Cab-O-Sil	"	"	"	2540	175	725	390	0
1319E[e]	100 X-401-3	-	20	100 Al dust	36	22	16/320	2740	290	-	-	585
1317E-2	80 X-401-3	20	-	100 Al dust 5 Cab-O-Sil	31	28	"	2580	460	710	845	1010
1321JE	100 X-401-3	-	20	10 glass fibers	36	32	0.5/330	3385	210	780	1225	945
1324JE	100 X-401-3	-	-	10 glass fibers 10 Cab-O-Sil	"	"	"	2860	180	400	555	655
1323JE	100 VCDE[f]	-	20	10 glass fibers	66	60	"	1540	0	0	0	0

a) Bonds prepared with paste adhesives.
b) Cab-O-Sil is colloidal silica (Godfrey L. Cabot, Inc.).
c) PMDA is pyromellitic dianhydride (du Pont Co.); MA is maleic anhydride. PMDA was dispersed in a solution of the resins and MA.
d) Average of three values for one-half inch lap joints to half-hard type 301 steel. Surface treatment: Method C.
e) The adhesive contained 1 phr phosphoric acid as a cure promoter.
f) VCDE is vinylcyclohexene dioxide (Carbide and Carbon Chemical Corp.).

Table 64. SHEAR STRENGTH OF BONDS TO ALUMINUM FROM RESINS CURED WITH COMBINATIONS OF PYROMELLITIC DIANHYDRIDE AND MALEIC ANHYDRIDE

Panel No.[a]	Resins, Parts by wt		Cab-O-Sil[b] Filler, by wt	Curing Agent, phr[c]		Type of Adhesive	Tensile Shear[d] Strength After 16 hr at 350°F, psi	
		EPON 1009		PMDA	MA		at 77°F	at 500°F
1310	100 X-131	-	0	20	18	Tape	915	115
1314[e]	100 X-131	-	0	20	18	Tape	1195	200
1315[e]	80 X-131	20	0	16.5	15	Tape	1235	215
1311	100 EPON 828	-	10	23	20	Paste	1135	340
1316	80 EPON 828	20	5	21	19	Paste	1435	90
1312	100 X-401-3	-	10	36	32	Paste	1090	0
1317	80 X-401-3	20	5	31	28	Paste	1355	300
1313	100 X-801-3	-	10	39	35	Paste	885	365
1318	80 X-801-3	20	5	34	31	Paste	1335	345

a) Bonds to 0.064 in. clad aluminum alloy 2024-T3 with glass cloth reinforcement (grade 112-Volan A). Constant ingredient (parts by wt): 100 aluminum dust filler.
b) Colloidal silica (Godfrey L. Cabot, Inc.).
c) PMDA is pyromellitic dianhydride (du Pont Co.); MA is maleic anhydride. PMDA was dispersed in a solution of the resins and MA.
d) Average of three values for one-half inch lap-joints. Surface treatment: chromic-sulfuric acid etch.
e) Formulation also contained 20 phr Formvar 15/95 E.

Table 65. SHEAR STRENGTH OF EXPERIMENTAL RESIN X-801-3 ADHESIVE BONDS TO TYPE 301 STEEL CURED WITH DIAMINODIPHENYLSULFONE

Press cure at 100 psi for one-half hour at 330°F

Panel No.[a)]	Resin, Parts by wt			Formvar 15/95 E, phr	DADPS,[c)] phr	Postcure at 330°F, hr	Adhesive Form	Tensile Shear Strength,[d)] psi				
	X-801-3[b)]	X-131	EPON 1009					Original			At 500°F After 500°F Aging	
								77°F[e)]	500°F	600°F	8 hr	200 hr
1279JE-P333[f)]	100	0	0	0	56	3	Paste	2080	200	135	835	440
-FE-P433[f)]	"	"	"	"	"	4	"	2025	225	-	-	285[h)]
1274FE-P333	"[g)]	"	"	20	59	3	Hot Melt	2680	700	-	1110	610[e)]
-2P433	"[g)]	"	"	"	"	4	" "	2570	210	-	-	105[h)]
1280JE-P333	"	"	"	"	56	3	Paste	4150	0	55	1215	875
1280JE-P333	100	0	0	20	56	3	Hot Melt	950	0	50	140	220
-FE-2P433	"	"	"	"	"	4	" "	2850	65	-	-	110[h)]
1281JE-P333	80	"	20	"	48	3	Paste	2245	0	65	815	675[h)]
1290PE-P34[i)]	10	72	18	0	31	3	Hot Melt	2005	795	65	290	675
1291PE-P34[i)]	40	48	12	"	39	3	" "	2170	755	150	775	925
1292PE-P34[i)]	50	40	10	"	42	3	" "	2035	730	40	715	435

a) Bonds prepared with glass fabric (112-Volan A) inserted between adhesive coated panels. Constant: 100 phr aluminum dust (MD201).
b) Laboratory preparation LR2102-77.
c) "DADPS" is diaminodiphenylsulfone.
d) Average of three values for half-inch lap joints to type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
e) Average of two values.
f) Adhesive recipe also contained 8 phr silica (Cab-O-Sil).
g) Laboratory preparation LR3571-82-5.
h) Exposure period 248 hr at 500°F.
i) Postcured in the press at 100 psi and 400°F.

Table 66. SHEAR STRENGTH OF X-131/X-401-3 ADHESIVE BONDS TO TYPE 301 STEEL

Press cure at 100 psi for one-half hour at 240°F plus one-half hour at 330°F

Panel No.a)	X-131 Batchb)	X-131/X-401-3 Resin Ratio	Curing Agent,c) phr	Tensile Shear Strength,d) psi Original 77°F	Original 500°F	Aged 200 hr at 500°F 500°F
1222FM$_3$	A	85/15	6 dicy	2665	105	525
1222FM$_3$-3	B	"	" "	2690	85	475e)
1222FM$_3$-2	C	"	" "	2540	65	230
1160FM$_3$-4	C	100/0f)	" "	3635	385	610
1223FM$_3$	G	85/15g)	4.5 dicy	2965	275	170e)
1228FM$_3$	F	85/15	4.5 dicy	3115	165	225e)
1230FM$_3$-P23h)	A	"	30 DADPS	3410	330	350e)
1230FM$_3$-2P23h)	C	"	" "	2025	30	230e)
1219FM$_3$-2P23h)	C	100/0g)	" "	3615	770	630e)

a) Bonded with hot melt cast unsupported adhesive tapes; constant ingredients, phr: 20 Formvar 15/95 E + 60 aluminum dust.
b) See Table 55 for identification of resin batches.
c) "Dicy" is dicyandiamide; "DADPS" is diaminodiphenylsulfone.
d) Average of three values for 0.5 in. lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method A-M$_3$.
e) Average of two values.
f) Solvent spread supported tape (112-Volan A glass fabric) with 100 phr aluminum dust.
g) Adhesive contained 10 phr Formvar 15/95 E.
h) Bonds postcured two hours at 300°F.

Table 67. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM X-131/X-701 ADHESIVES CURED WITH DICYANDIAMIDE

Cure (contact pressure): one-half hour at 330°F

Specimen No.[a]	X-131/X-701[b] Resin Ratio	Tensile Shear Strength,[c] psi		
		Original		Aged 200 hr at 500°F
		77°F	500°F	500°F
1145JM[d]	100/0	1930	770	680
$1172JM_1$	90/10	2055	565	305
$1173JM_1$	80/20	2285	560	470
$1174JM_1$	70/30	2220	425	420

a) Bonds from hot melt impregnated supported adhesive tapes with grade 112-Volan A glass fabric. Constant ingredients: 100 phr aluminum dust (MD201) + 6 phr dicyandiamide.
b) X-131 resin lot No. LR1991-113.
c) Average of three values for bonds to full hard type 301 steel (thickness 0.050 in.). Surface treatment: Method $A\text{-}M_1$.
d) Solvent spread supported adhesive tape. The test results given are the averages for eight adhesive batches.

Table 68. SHEAR STRENGTH OF BONDS TO TYPE 301 STEEL FROM BLENDS OF X-131 RESIN AND EXPERIMENTAL RESIN LR2047-199

Cure at 100 psi: one-half hour at 330°F

Panel No.[a]	1329JE	1329JE-2	1330JE	1330JE-2	1331JE	1331JE-2
X-131/LR2047-199 ratio,[b] by wt	90/10	90/10	70/30	70/30	50/50	50/50
Adhesive batch No.	1	2	1	2	1	2
Diaminodiphenylsulfone, phr	36.5	36.5	49	49	61	61
Tensile Shear Strength,[c] psi						
At room temperature	2225	2970	2860	3140	2905	3700
At 500°F, original	580	555	695	415	605	585
, after 500°F aging, 8 hr	625	555[d]	535	560[d]	1040	845[d]
, 120	400	-	285	-	935	-
, 159	355[d]	-	395[d]	-	945[d]	-
, 200	-	255	-	375	-	980
At 600°F	-	170	-	65	-	105

a) Bonds from paste adhesives with glass fabric (112-Volan A) inserted in the glue line. Constant ingredient, by wt: 100 aluminum dust (MD 201).
b) X-131 resin lot No. LR3523-64.
c) Average of three values for one-half inch lap joints to half-hard type 301 steel (thickness 0.050 in.). Surface treatment: Method C.
d) Average of two values.

Table 69. COMPARISON OF THE SHEAR STRENGTH OF X-131 ADHESIVE BONDS TO CLAD ALUMINUM ALLOY AND TYPE 301 STEEL

Panel No.[a)]	Formvar 15/95 E, phr	Curing Agent,[b)] phr	Variable, phr	Metal[c)]	Tensile Shear Strength[d)] Original 77°F	Original 500°F	Aged 200 hr At 500°F 500°F
1145J-16	-	6 dicy	-	Alclad	1475	695	625
1145JM-16	-	" "	-	301, FH	1825	685	475
1205J	-	14 dicy	-	Alclad	1565	460	790
1205JM	-	" "	-	301, FH	1665	60	295
1209J	-	6 dicy	200 Al dust	Alclad	1350	770	595
1209JM	-	" "	" " "	301, FH	1925	675	685
1160J-2	20	6 dicy	-	Alclad	2025	620	865
1160J-2P23[e)]	"	" "	-	"	1755	570	895
1160JM$_1$-2P23[c)]	"	" "	-	301, FH	2310	640	710
935J-2	15	10 dicy	10 TAT-0-212[g)]	Alclad	1650	500	310
935JM$_3$-2	"	" "	Hot melt	301, 1/2H	1935	80	305
1218J	-	4.5 dicy	-	Alclad	1525	555	545
1218J-P23[e)f)]	-	" "	-	"	1425	465	635
1218JM$_1$-P23[e)f)]	-	" "	-	301, FH	1665	635	645
1223F-P23[e)]	10	4.5 dicy		Alclad	1845	225	120
1223F[g)]	"	" "		"	2530	410	275
1223FM$_3$[g)]	"	" "	60 Al dust	301, 1/2H	2965	275	170
1222F-P23[e)]	20	6 dicy	Hot melt	Alclad	2745	400	625
1222F[h)]	"	" "		"	2245	380	590
1222FM$_3$[h)]	"	" "		301, 1/2H	2665	105	525
1219J	20	30 DADPS	-	Alclad	835	555	700
1219J-P23[e)]	"	" "	-	"	2135	605	915
1219JM$_1$-P23[e)]	"	" "	-	301, FH	2720	850	1015
1220J	20	30 DADPS	5 Bisphenol A	Alclad	2615	545	830
1220J-P23[e)]	"	" "	" "	"	2455	685	950
1220FE-2	"	" "	" "	301, 1/2H	4235	880	690
1236J-13[i)]	20	24 DADPS		Alclad	2315	225	710
1236JE-13[i)]	"	" "		301, 1/2H	3310	235	505

a) Bonds from solvent spread supported tape (glass fabric 112-Volan A) dried 15 min at 200°F. Constant ingredients (parts by wt): 100 X-131 + 100 aluminum dust. Cure: One-half hour at 330°F.
b) "Dicy" is dicyandiamide; "DADPS" is diaminodiphenylsulfone.
c) Aluminum alloy clad 2024-T3, thickness 0.064 inch; type 301 steel, thickness 0.050 inch.
d) Average of three values for one-half inch lap joints.
e) Bonds postcured two hours at 300°F.
f) Adhesive contained experimental modified X-131 (D).
g) TAT-0-212 is triethanolamine titanate N-oleate (du Pont).
h) Adhesive based on a blend of 85/15 modified X-131 (G)/X-401-3.
i) Resin base: 80/20 X-131/EPON 1009.

Table 70. IDENTITY AND SOURCE OF MATERIALS EVALUATED IN HIGH TEMPERATURE ADHESIVE FORMULATIONS

Material	Description	Source
Aluminum dust, atomized MD105	99% passes 325 mesh	Metals Disintegrating Co., Inc.
MD201	65% passes 325 mesh	" " " "
Asbestos, neutral TC	Short fiber	Powhatan Mining Co.
BF_3-400	Curing agent for epoxy resins. Boron trifluoride complex.	Shell Chemical Corporation
Cab-O-Sil	Colloidal silica	Godfrey L. Cabot, Inc.
Cupric 8-Quinolinolate	-	Fisher Chemical Corporation
Curing Agent E	2,5-Diaminopyridine	Shell Chemical Corporation
Diaminodiphenylsulfone	4,4'- or mixed isomers	Merck and Company
Dicyandiamide	mp 209°C	American Cyanamid Company
EPON 828	Liquid epoxy resin, mp 40-50°F	Shell Chemical Corporation
EPON 834	Liquid epoxy resin, mp 68-82°F	" " "
EPON 1009	Solid epoxy resin, mp 295-310°F	" " "
Formvar 15/95 E	Polyvinyl formal	Shawinigan Resins Corporation
Gelva C-3-V-30	Carboxylic modified vinyl acetate polymer	Shawinigan Resins Corporation
Glass fibers (milled)	1/32 inch	Ferro Corporation, Fiber Glass Division
Glass fabric, 112-Volan A	Style 112 with Volan A finish; thickness 2.5 to 3.0 mils	Coast Mfg and Supply Company
Nacconol NR	Anionic surfactant	Allied Chemical and Dye Corporation National Aniline Division
Pluronic L62	Non-ionic surfactant	Wyandotte Chemicals Corporation
Pyromellitic dianhydride	mp 286°C	du Pont
TAT-O-212	Triethanolamine titanate N-oleate	du Pont

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JULIUS HYMAN AND COMPANY
DENVER, COLORADO

File No. 573-1
31 August 1954

LABORATORY MEMORANDUM

(183)

TO: JGB-TGM, CCM, GHM, JRM

SUBJECT: PREPARATION OF LOW VISCOSITY EPON ADHESIVE VI

SUMMARY

EPON Adhesive VI is a paste-like, proprietary compound used as a bonding agent, particularly in the aircraft industry. It is prepared by simple mixing of EPON 834 resin, Vinylite AYAF (polyvinyl acetate resin), asbestos, allyl glycidyl ether (AGE), TiO_2, and Acetamine Orange (a dye). With the transfer of its production from Shell Development Co., Emeryville to Denver a marked increase in the viscosity of the final product has occurred. This in turn has caused customer complaints because of the increased difficulty in handling the material. Consequently, an investigation of the cause for this change was undertaken.

Laboratory piloting of Adhesive VI manufacture has indicated that some, as yet unknown, property of the EPON 834 resin itself is the main variable affecting the viscosity of the final adhesive. Traces of alkalinity introduced with the asbestos also increase the viscosity significantly. Removal of this alkali (with resulting reduction in adhesive viscosity) was accomplished in the laboratory by a water wash but this is not considered practical for plant operation.

Comparison of the Denver and Emeryville methods for producing adhesives shows that the Emeryville method employing shorter mixing time definitely results in a lower viscosity product.

However, it is concluded that the combined use of the Emeryville process and alkali-free asbestos will not always balance the unknown, undesirable property of certain lots of EPON 834.

It is recommended that a sufficient quantity of Denver EPON 834 stocks (1 or 2 drums) be transferred to Emeryville to permit Shell Development to make a plant batch of Adhesive VI in their equipment and by their technique. Further action would await the results of this test.

INTRODUCTION

The initial manufacture of EPON Adhesive VI was carried out by Shell Development Co., Emeryville. They developed the formula and supplied Market Development quantities of this material until the details of its manufacture were finalized. Customer acceptance, and consequently specifications, were based on the material as produced by Emeryville. After transfer of its manufacture to Denver it was found that the Denver product was more viscous than the Emeryville material and exceeded the specification for this property (maximum of 1 MM centipoises at 77°F). Meetings were held between interested parties from Head Office, Denver, Shell Development Company (Emeryville), and the San Francisco Marketing Office to discuss the problem. The meeting resulted in an extensive pilot program at Denver. This report summarizes the results of the work to date on Adhesive VI.

EXPERIMENTAL

All samples of EPON 834 used in this work were obtained from Houston either as normal production or special laboratory samples. These included the EPON from normal Denver plant stocks.

All other materials were from Denver plant stocks: Allyl glycidyl ether (AGE), asbestos, titanium dioxide, Vinylite AYAF, and Acetamine Orange (dye).

The original Emeryville procedure for preparing Adhesive VI has been modified in the plant to fit more closely the operating situation at Denver. Both methods are described in the Appendix. Laboratory preparation of adhesive used a three-quart Read-Standard sigma blade blender, closely duplicating the plant blender of the same type and manufacturer. Where pre-mixing was suggested (Emeryville procedure), laboratory glassware was used.

Viscosity was measured on a Brookfield Viscometer, Model HBF. All samples were placed in a 77°F water bath for a minimum of 16 hours before testing. This time interval was found adequate for attaining temperature equilibrium.

Alkalinity measurements on the EPON resin were made by diluting 50 g of the EPON with 150 ml of acetone and pouring into 200 ml of distilled water. The resultant water-acetone phase was titrated with 0.02 N H_2SO_4 to pH 7 and the alkali calculated as ppm NaOH. The alkalinity of the asbestos floats was determined by shaking 25 grams asbestos with 100 ml water, and filtering and washing with another 100 ml water under suction. The combined filtrate and washings were checked for pH, then titrated to pH 7.0 with 0.02 N H_2SO_4.

Infrared scans of three samples of EPON (Lots 3B1, 4B159, 6B153) were made on the Model 21, Perkin-Elmer Spectrophotometer after dilution with CCl_4 to facilitate handling.

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A method was devised for determining AGE content of the product since it was felt that loss of this, its most volatile component, could be responsible for high viscosities. Using a pilot batch of Adhesive VI which had been very carefully prepared to contain the correct amount of AGE (10.8%w) a small amount of the adhesive was spread on a small glass plate in a layer about 1/64 inch thick, weighed to determine the sample size, and placed in a vacuum oven to measure loss in weight. After investigating various conditions it was determined that a loss in weight equivalent to the AGE content occurred after one hour at 120°C and 10 mm Hg. Applying this test to plant batches of Adhesive VI with high viscosities, about 10.2%w volatiles were removed. It was concluded that no significant loss of AGE takes place during plant production of the adhesive.

Table I shows the results obtained with the two different mixing procedures and with variations in the AGE content. It is apparent that the Emeryville process with shorter mixing time produces a lower viscosity adhesive than does the Denver process. However, the Emeryville process (using our raw materials) did not make an adhesive with a viscosity below 1 MM CP @ 77°F. Although in some cases adhesive viscosity shows anomalous behavior with high AGE content it is obvious that a minimum of 15%w AGE would be needed (compared to 10.8%w called for in the formula) to reduce the viscosity below 1 MM CP.

Tensile shear strength of adhesive with high AGE content (Batch P-17) was 3515 psi at 77°F and 1725 psi at 180°F. These values pass shear specifications and are in line with shear values of adhesive containing the normal amount of AGE.

Table II deals with the effect of alkalinity introduced with the asbestos. Normal asbestos washed with neutral water will produce a wash water of pH 8-11. The removal of these traces of alkali reduced the resultant adhesive viscosity from 1.8 to about 1.3 MM CP. The addition of 2.5%w water to the formula was even more effective, reducing viscosity to 0.8 MM CP. The effect of water on product performance is not known. The addition of 0.1%w acetic acid (HAc) appeared detrimental to viscosity while 1%w water was ineffective.

Table III illustrates the variation of adhesive viscosity with the EPON resin used. From these data it is concluded that traces of alkali in the resin and the apparent resin viscosity have no bearing on the final adhesive viscosity. However some unknown property of the resin appears to have a significant effect on adhesive viscosity. Samples of the EPON resin (Lots 381, 48159, 68153) were scanned in the infrared but were found to give identical spectra. Table IV lists the analytical data obtained by Houston and forwarded with their resin samples[1].

It is to be pointed out that Run P-25 using EPON 834 supplied from Emeryville stocks did make a specification viscosity adhesive (0.96 MM CP).

Table V shows how viscosity measurements of Adhesive VI tend to increase with storage time. The prime example is Emeryville Batch 2604-7 which increased from 0.89 to 1.40 MM CP over a period of ten weeks. The Emeryville laboratory has been advised of this and will investigate other samples of their production.

All samples of adhesive examined appear to "sweat out" a reddish liquid on the surface of entrapped bubbles. Batches P-14, P-16, P-18, P-19, P-20, P-21, P-22, and P-25 were stirred by hand prior to the six day viscosity measurements and, as shown in Table V, the viscosity was reduced.

DISCUSSION AND CONCLUSION

Based on the data contained in this report it is concluded that the Emeryville process does make a lower viscosity Adhesive VI than does the Denver method. Even though no specification adhesive has been made from resins on hand, other than that supplied by Emeryville, it is evident that Denver production should be made with the shortest time-temperature process to lower adhesive viscosity as much as possible.

The addition of other components such as water, additional AGE, etc. is not recommended, even though they do lower viscosity, because of possible effects on adhesive performance.

Although traces of alkalinity in the asbestos are detrimental to the viscosity of the final product, water washing of the asbestos is not considered practical for plant operation. Further, the supplier (Johns-Manville) states that no other grade of asbestos (referring to alkali content) is made by them. Other sources of asbestos will be investigated.

The measurement of viscosity in this manner (Brookfield Viscometer) is obviously not a precise test. Since these adhesives are pseudo-plastic the actual results obtained are not true viscosity. However, as long as the torque (measured in terms of the viscosity scale) which results during the test is repeatable and correlates with customer acceptance, the test is satisfactory for evaluating the adhesive. The present test appears to be repeatable to within about ± 15%.

FUTURE WORK

It is recommended that one or two drums of Denver EPON 834 stocks be transferred to Emeryville to permit Shell Development to make a plant batch of Adhesive VI in their equipment and by their method. An observer from Denver should be present. This resin would represent material which, when used at Denver, both in the plant and laboratory, failed to make specification adhesive. Should this resin produce high viscosity adhesive at Emeryville further discussions with Houston would be in order.

Data by: S. Zevin, J. A. Streich, R. M. Berry
Reference: Tech. Record Vol. 168, pp 50-53, Vol. 189, pp 3, 10-13, 15, 18
Vol. 169, pp 71, 72
1) Houston letter to Head Office, Mfg.-Oper., of July 19, 1954 on EPON 834 - Adhesive VI.

SZ/FMB/baj

Approved: F. R. Bolenbaker
E. M. Lando

CC: Shell Development Co., Denver
D. L. Yabroff
K. E. Marple (2)

APPENDIX

EPON ADHESIVE VI MANUFACTURE

DENVER OPERATING PROCEDURE (REVISED)

At least 24 hours before a batch of Adhesive VI is to be mixed, two drums of EPON 834 should be placed in the hot box to liquefy the resin. The adhesive is prepared by mixing by weight in the blender as follows:

1. Allyl glycidyl ether (AGE), 177 lb.
2. Acetamine orange, 147 grams
3. Vinylite AYAF, 94 lb.
4. Titanox, 8 lb 2 oz. Mix ten minutes, turn on steam, and add
5. EPON 834, 500 lb (1 drum).
6. Asbestos floats, 353 lb. This should be added in 100 lb increments, seeing that each addition is mixed in before adding more.
7. When asbestos has almost all been pulled into the mix, stop the blender, scrape down the walls to the point where the blades reach, mix again for a few minutes to be sure all the asbestos has been worked in, then add
8. EPON 834, 500 lb. Allow to mix overnight, reversing the direction of agitation occasionally. Inspect to see that no unmixed resin remains on top. If there is such, work it in with the scraper by hand.
9. When batch is ready, bolt down the blender lid, set up the scale and pour the adhesive into cans as specified. Containers will be 1-gallon, holding 8 pounds, or 5-gallon, holding 40 pounds net. Label each can with designation "VI" and batch number. About at the middle of the batch, take two 1-pint samples properly labeled, which are delivered to the laboratory.
10. Toward the end of the packaging, it will be necessary to run the blender in the proper direction to push out the bulk of the adhesive. The last of the contents will have to be scraped out. If another batch of the same adhesive is to be made, the blender need not be scraped clean. If another type of adhesive will be made next, scrape out as much of this batch as possible.

APPENDIX

EPON ADHESIVE VI MANUFACTURE

EMERYVILLE OPERATING PROCEDURE

Formula

		%w
85	parts EPON 834	61.28
15	parts AGE	10.80
8	parts AYAF	5.80
30	parts Asbestos	21.60
0.7	parts Titanium Dioxide	0.50
0.03	parts Acetamine Orange	0.02
		100.00

Mixing

The polyvinyl acetate (AYAF) is dissolved in AGE by stirring it in a tank at 50°C for 4-10 hr. The solution is made to contain 15 parts AGE and 8 parts AYAF and to this solution is added about 0.03 parts dye.

The Read mixer is charged with 85 parts EPON 834 and this is heated to about 70°C by steam. Add 30 parts asbestos floats and 0.7 parts titanium dioxide and the total mixed for 2 hr at 70-75°C.

To the 115 parts of EPON-Asbestos is added the 23 parts of AGE-AYAF solution and blending continued for 30 minutes.

TABLE I

EFFECT OF MIXING METHOD AND AGE CONTENT ON ADHESIVE VI VISCOSITY

(EPON 834 - 0.77 MM CP @ 77°F)

Batch No.	Mixing Method	Formula	Viscosity, MM CP @ 77°F	Remarks
P-3	Denver	Standard	2.69, 2.88	See Appendix for details of mixing method
P-4	Denver	Standard	2.94, 2.75	
P-5	Emeryville	Standard	2.18, 2.24	See Appendix for details of mixing method
P-6	Emeryville	Standard	1.86, 1.92	
P-7a	Emeryville	Standard	1.79	
P-7b	Emeryville	AGE raised to 12.2%w	1.73, 1.79	Made from P-7a by adding AGE and re-mixing
P-8a	Emeryville	AGE raised to 12.2%w	1.31	AGE incorporated in original mix
P-8b	Emeryville	AGE raised to 13.5%w	1.28, 1.28	Made from P-8a by adding AGE and re-mixing
P-13a	Emeryville	AGE - 14%	1.79, 1.79, 1.82	AGE in original mix
P-17	Emeryville	AGE - 15%	1.12, 1.09	AGE in original mix; tensile shear strength at 77°F - 3515 psi, at 180°F - 1725 psi.
P-9a	Emeryville	AGE - 13%	1.22	AGE in original mix; tensile shear strength at 77°F - 3515 psi, at 180°F - 1725 psi.
P-9b	Emeryville	AGE - 15%	1.15, 1.60	Made from P-9a by adding AGE and re-mixing.

TABLE II

EFFECT OF ASBESTOS ON ADHESIVE VI

Batch No.	Components Mixed	Temp, °C	Viscosity, MM CP @ 77°F	Remarks
P-11a	EPON, Asbestos, TiO_2	80-90	12.0	Mixing time - 10 minutes
P-12a	EPON, Asbestos, TiO_2	30-32	10.8	Mixing time - 10 minutes
P-11b	All	80-90	1.79	Additional mixing time - 5 minutes
P-12b	All	30-32	2.02, 2.11	Additional mixing time - 5 minutes
P-14	All	80-90	1.38, 1.41	Asbestos and washed in lab to reduce pH to 6.5-7.5
P-16	All	80-90	1.24, 1.29	Repeat of P-14
P-23	All	80-90	2.56, 2.62	0.1%w HAc added
P-24	All	60-90	0.77, 0.80	2.5%w H_2O added
P-26	All	80-90	2.05, 2.11	1%w H_2O added
P-27	All	80-90	1.70, 2.18	Repeat of P-26

TABLE III

EFFECT OF EPON 834 ON ADHESIVE VI

Batch No.	EPON Used: Lot No.	EPON Used: NaOH, ppm	EPON Used: Viscosity MM CP @ 77°F	Adhesive Viscosity MM CP @ 77°F	Remarks
2604-7	A-03-8	-	- - -	0.89[a)]	Normal Emeryville production Batch 2604-7
- - -	381	3	0.77	2.73	Normal Denver plant production
P-18	AM149	2	0.69	1.49	Lab prepared, Emeryville method
P-19	784	18	1.92	1.12	Lab prepared, Emeryville method
P-20	783	8	17.2	1.11	Lab prepared, Emeryville method
P-21	6M152	7	0.28	1.28	Lab prepared, Emeryville method
P-22	6M153	2	0.15	1.67	Lab prepared, Emeryville method
P-25	AM159	2	0.76	0.96	Lab prepared adhesive from EPON 834 supplied via Emeryville

a) This measurement of viscosity made by Emeryville; viscosity measured 1.09 MM CP when received at Denver and 1.48 MM CP after 10 weeks storage

TABLE IV

PROPERTIES OF EPON 834 USED FOR MAKING ADHESIVE VI

Lot No.	4B149	7B3	7B4	6B152[a)]	6B153[b)]	6B15[c)]
Viscosity, Gardner	A_2	A_1	A_{1+}	A_2	A_2	A_{2+}
Color, Gardner	5	1	2	1	3	2-3
WPE	251	237	248	230	227	243
Water, %w	0.10	0.01	0.01	0.01	0.01	0.03
Chloride, %w	0.81	0.27	0.21	0.18	0.53	0.25

a) 45%w NaOH used for secondary caustic addition

b) Modified EPON 828 process

c) Prepared via evaporative cooling during reaction

NOTE: These data supplied by Houston Laboratory; see their ltr. of July 19, 1954 to H.O., Mfg.-Oper.

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TABLE V

VARIATION OF VISCOSITY MEASUREMENTS

Batch No.	Viscosity (MM CP @ 77°F), at Time After Mixing 1 day	2 days	3 days	6 days	12 days	10 weeks
P-9a	1.22	1.22	1.22	1.60	- -	- -
P-9b	1.15	1.01	1.17	1.28	- -	- -
P-7a	1.79	- -	1.54	- -	- -	- -
P-7b	1.76	- -	1.76	- -	- -	- -
P-14	1.40	1.65	- -	1.33	- -	- -
P-16	1.27	- -	- -	1.22	1.24	- -
P-17	- -	- -	1.04	- -	1.28	- -
P-24	0.79	- -	- -	- -	0.91	- -
P-18	1.49	1.92	- -	1.89	2.02	- -
P-19	1.12	1.59	1.71	1.55	1.63	- -
P-20	1.11	1.27	1.41	1.30	1.47	- -
P-21	1.28	1.41	- -	1.40	1.36	- -
P-22	1.67	1.44	- -	1.43	1.91	- -
P-25	0.96	- -	1.23	1.12	1.27	- -
2604-7 a)	0.89 a)	- -	- -	1.09	- -	1.40

a) Plant adhesive prepared and measured at Emeryville.