

DEVELOPMENT OF ADHESIVES WITH IMPROVED HEAT RESISTANCE IN BONDS OF STAINLESS STEEL

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DEVELOPMENT OF ADHESIVES WITH IMPROVED HEAT

RESISTANCE IN BONDS OF STAINLESS STEEL¹

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Summary

Adhesives containing combinations of polyamide resin with melamine compounds, epoxy resins with vinyl anhydride copolymers, and inorganic fillers were studied as heat-resistant bonding agents for stainless steel.

Attempts to improve the strength of the polyamide-melamine type adhesive at 550° F. were unsuccessful.

Studies were made of the heat-resistant properties of eight different epoxy resins modified with an ethyl acrylate-maleic anhydride copolymer and with maleic anhydride. The most promising of these were adhesive formulations of epoxy resins which were glycidyl ethers of either bisphenol A or tetra hydroxy phenyl ethane and contained aluminum powder and arsenic pentoxide fillers impregnated into asbestos cloth.

¹This is the final report of an investigation made in cooperation with the Department of the Navy, Bureau of Naval Weapons, under Order No. NAer 01967.

²Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

Introduction

The objective of this project was to develop heat-resistant adhesives for bonding stainless steel, based on (a) polymers of polyamide combined with melamine compounds, and (b) vinyl copolymers, containing reactive anhydride side groups, combined with epoxy resins.

The phenol-epoxy type of adhesive, generally recognized as one of the most heat-resistant organic adhesives for metal bonding, possesses high tensile shear strength at elevated temperatures when tested both immediately and after relatively long periods of aging at temperatures up to 600° F. in bonds of aluminum. One serious limitation of this type of adhesive, however, is the low resistance to thermal aging on bonds of stainless steel at temperatures of 500° F. and above.

Some results of the work at the U.S. Forest Products Laboratory for the National Advisory Committee on Aeronautics³ showed that thermal aging resistance of an adhesive bond was related to the chemical structure of the polymer and the type of metal adherend. Thus phenol, epoxy, and phenol-epoxy type adhesives had good thermal aging properties in bonds of aluminum but were poor in bonds of stainless steel. Phenol-nitrile rubber and silicone-type adhesives, however, showed better thermal aging resistance in bonds of stainless steel than in bonds of aluminum.

This study also revealed that the addition of certain inorganic compounds to a phenol-epoxy type of adhesive improved the resistance of the adhesive to heat aging on stainless steel. Some of these inorganic materials were compounds of manganese, arsenic, tin, and antimony. Later work by Janis and others⁴ on temperature-resistant adhesives for steel showed very promising results with a silicone-epoxy-phenolic adhesive system cured with arsenic pentoxide. A similar increase in resistance to thermal degradation and increased joint strength were obtained with antimony trioxide in a phenol-epoxy adhesive and straight epoxy-resin adhesive in bonds of aluminum.⁵

³Black, J. M., and Blomquist, R. F. Development of Metal Bonding Adhesive With Improved Heat Resistance. WADC Tech. Report 56-650. November 1956.

⁴Janis, E. C., Boram, W. R., Riel, F. J., and Sussman, S. E. Research and Development on Elevated Temperature-Resistant Structural Metal-to-Metal Adhesives. WADC Tech. Report 59-11. May 1959.

⁵Black, J. M., and Blomquist, R. F. Relationship of Metal Surfaces to Heat-Aging Properties of Adhesive Bonds. NACA TN 4287. September 1958.

Results of some further work⁶ for NASA at the Forest Products Laboratory involved a study to relate the polymer structure of the base component of the adhesive to thermal degradation in bonds of aluminum and of stainless steel. This study was essentially a screening of a variety of basic polymers for resistance to degradation at 550° F. and did not include investigations into formulation variables of catalysts, fillers, or crosslinking agents directed toward development of an improved heat-resistant adhesive. The results of this work revealed some polymer systems which possessed a relatively high degree of resistance to aging at 550° F. in bonds of stainless steel. These promising materials were polyamide resins, butadiene copolymers, combinations of phenol resin with butadiene copolymers, melamine resin with polyamides, and a combination of a maleic anhydride-vinyl copolymer with epoxy resins.

The present study was undertaken to develop further the melamine-polyamide and the vinyl anhydride copolymer-epoxy resin combinations into acceptable heat-resistant adhesive formulations for bonding of stainless steel. Investigations of curing mechanisms, both organic and inorganic in nature, which would increase strength at elevated temperature were to be considered.

Experimental Procedure

Polymer and Adhesive Materials

The various polymers employed in this study are designated by code numbers in the test and tables of the report. They were dissolved in appropriate solvents and were applied to the metal surfaces as solutions or suspensions or impregnated into a woven asbestos cloth and applied as tape adhesive. The asbestos cloth was type 28-PO-47, made by Raybestos-Manhattan, Inc., and was sized by immersion in A-1100, an organic silane solution made by Union Carbide and Carbon, and dried for 30 minutes at 300° F.

The adhesive formulations of polyamide and melamine were dissolved in methyl alcohol, isopropyl alcohol, and water systems to have a solids content of about 20 percent by weight. Heating and refluxing of the materials was employed to aid in dissolving the polymers. Solvents were removed before bonding by oven-drying specimens at 200° F. The information on drying conditions, bonding temperatures, bonding time, and pressure is given in table 1 for the polyamide-based adhesives.

⁶Black, J. M., and Blomquist, R. F. Relationship of Polymer Structure to Thermal Deterioration of Adhesive Bonds in Metal Joints. NASA TN D-108. August 1959.

In the epoxy resin-vinyl anhydride copolymer adhesive formulations, the polymer materials were dissolved in methyl ethyl ketone with refluxing to a solids content of about 10 percent. To aid in the solution of the copolymer, it was reduced in the presence of methyl ethyl ketone to small particles in a Waring blender before refluxing. The aluminum dust and arsenic pentoxide also were mixed into the polymer solution by means of the Waring blender.

In making the asbestos cloth tape modifications of the adhesives, four heavy coats of adhesive solutions were applied to the cloth by brush and dried for 10 minutes at 160° F. between coats and a final drying period of 10 minutes at 200° F. after the last coat. Only one layer of the tape adhesive was used in bonding. In bonding with the solution-type adhesive, two coats of the epoxy resin-copolymer solution were applied to each metal surface. Each coat was dried for 10 minutes at 200° F. before pressing. All bonds of epoxy-resin formulations were cured at 400° F. for 60 minutes at a bonding pressure of 400 pounds per square inch.

The aluminum filler employed in this study was aluminum dust, Alcoa 123 from the Aluminum Company of America. It was used as obtained. The arsenic pentoxide, chemically pure, was dried at 200° F. and kept in a dessicator until used.

Specimen Preparation and Tests

The tensile shear strength of 0.5 -inch lap-joint specimens of 16-gage, Type 316, 18-8 annealed stainless steel was employed as the means of evaluating the bonding properties of the various formulations. Joints were tested at 80° F. and at 550° F. both before and after aging at 550° F. to determine the resistance of the adhesive bonds to heat.

Test specimens were prepared by bonding 1- by 3.5-inch coupons into a lap-joint specimen with a 0.5-inch overlap. This resulted in specimens 1 inch wide and 6-1/2 inches long. Preparation of individual specimens was done to eliminate the problem of bond failure or partial failure during cutting of specimens from a bonded panel. The annealed steel surfaces were prepared for bonding by first immersing them in a solution of 100 grams of concentrated hydrochloric acid, 4 grams of 30 percent hydrogen peroxide, 20 grams of 40 percent formalin solution, and 90 grams of water at 150° F. for 10 minutes. After the surfaces were rinsed in tap water, they were immersed for 5 minutes at 150° F. in a solution consisting of 10 grams of concentrated sulfuric acid, 1 gram of sodium dichromate, and 30 grams of water. Surfaces were then rinsed in distilled water and air dried.

Twelve lap-joint shear specimens were prepared for each adhesive formulation. Three specimens were tested for each condition of test at 80° and 550° F. before and after heat aging. Test specimens were loaded in tension in self-aligning grips at the rate of 600 pounds per 0.5 square inch per minute. Load was applied in the tests at the elevated temperature 5 minutes after the specimen came to the test temperature as determined by thermocouple measurements.

Temperature during heat aging was controlled within $\pm 3^\circ$ F. in special electrically heated ovens.

DISCUSSION

Studies on Polyamide-Melamine Formulations

The results of tensile lap-shear tests are shown in table 1. The polyamide (PA-1) employed in these studies was an alcohol-soluble type of nylon and was combined with varying amounts of diallyl melamine, melamine-formaldehyde resin (MF-1), and curing agents paraformaldehyde, maleic anhydride, and triethylamine. Paraformaldehyde was included in the adhesive compositions in an attempt to produce methylol groups on the diallyl melamine which could react with the amino hydrogens of the polyamide to produce cross linkage. In addition, there was also the possibility that the allyl groups could cross link with each other after aging at high temperature or would be free to form a strong bond to the steel surface. The triethylamine was considered to be a catalyst for the methylolation reaction. Maleic anhydride also could conceivably form a cross link between methylol groups and reactive amino hydrogen, and the unsaturated bonds would be potentially reactive with the allyl groups.

The formulations with diallyl melamine were rather similar in strength properties regardless of amount of paraformaldehyde, triethylamine, and maleic anhydride. Initial tensile shear strength at 80° F. ranged from about 1,700 to 2,600 pounds per square inch and, after aging 3 hours at 550° F., ranged between 400 and 700 pounds per square inch. These formulations had no strength at 550° F. either immediately or after 3 hours at 550° F., indicating that little or no cross linkage had occurred.

Formulations combining a melamine-formaldehyde resin (MF-1) with the polyamide showed some strength at 550° F. as increased amounts of the melamine resin were used. Shear strength at 80° F., however, was decreased

as the melamine resin was increased, possibly indicating excessive embrittlement of the adhesive. Bond strength at 80° F. after heat aging was also low, presumably because of the low resistance of the melamine resin to thermal degradation and further embrittlement.

When melamine compounds were deleted from the formulations and the polyamide was combined with maleic anhydride or paraformaldehyde either separately or together, the strength of bonds at 80° F. was improved by heating for 3 hours at 550° F., but no strength was obtained at 550° F.

Because of the unsuccessful attempts to develop bond strength at elevated temperature with these formulations, further work on the polyamide-melamine combinations was discontinued.

Studies of the Epoxy-Resin-Vinyl Copolymer Formulations

In the epoxy resin-vinyl copolymer series of formulations, various epoxy resins were combined with a vinyl copolymer (C-1) reported to be composed 95 percent of ethyl acrylate and 5 percent of maleic anhydride. An anhydride equivalent weight of 2,000 grams was assumed for this copolymer in the formulation studies. These combinations could conceivably produce cross linkage through reaction of the epoxide and anhydride groups. In many formulations, additional molecular maleic anhydride was added to the formulation to attain a particular ratio of epoxy equivalents to anhydride equivalents. The epoxy resins employed varied in chemical structure. Resins of the bisphenol A, tetra glycidyl ether of hydroxyl phenyl ethane, Novalac epoxy, dicyclopentadiene dioxide, dicyclodiepoxycarboxylate, silicone-epoxy, and unsaturated polyolefin types were employed.

Formulations with Epoxy Resin, E-1

Epoxy resin E-1 was a diglycidyl ether of bisphenol A with a reported epoxide equivalent weight of 486 grams. The results of the tensile shear tests of lap joints at 80° and 550° F. are shown in table 2 for the different formulations.

For solution-type adhesive formulations of E-1 resin with copolymer and copolymer supplemented with maleic anhydride, the strength of bonds at 550° F. after 48 hours at 550° F. varied from 20 to 150 pounds per square inch. The ratio of epoxy equivalent to anhydride equivalent in these formulations had no marked effect on joint strength at 550° F. At a ratio of about 1:1 of epoxide to anhydride equivalents, formulations with a combination of copolymer and molecular maleic anhydride (adhesive No. 30B, table 2) were

higher in strength after aging at 550° F. than a formulation with copolymer alone (adhesive No. 49, table 2) indicating the plasticizing effect of the large amount of copolymer in relation to the amount of epoxy resin. The addition of a polyhydric alcohol, glycerol, as initiator of the reaction between anhydride and epoxide groups seemed to have no beneficial effect (adhesives Nos. 37C, 43, and 30C, table 2).

The most striking improvement in strength of bonds at the elevated temperature after aging 48 hours at 550° F. was obtained when the asbestos tape modification with aluminum powder and arsenic pentoxide fillers was employed. The effectiveness of the aluminum powder and arsenic pentoxide fillers as curing agents is shown from the results obtained with solution-type adhesives Nos. 39A, 39B, and 35B, which were modifications without filler, and with adhesives Nos. 39C, 39D, and 35D, which were modifications with the aluminum and arsenic pentoxide in tape form.

Shear strength of the tape formulas at 550° F. was increased also as the amount of anhydride employed was decreased. Adhesive tape-filler formulations in which the anhydride content was 0.27 equivalent or less to 1 mol of the epoxide (adhesives Nos. 39D, 42, and 39C, table 2) were higher than adhesives Nos. 35D and 23C. The formulation without anhydride (adhesive No. 52) was slightly higher in strength after aging at 550° F. than any of the other formulations, indicating no apparent advantage in the use of anhydride compounds in the cure of the epoxy resin. All formulations of epoxy resin were characterized by low strength at 550° F. after 3 hours at 550° F. This was apparent for both the solvent and tape-filler modifications, indicating the cure associated with the aluminum and arsenic pentoxide was a relatively slow process and developed only after longer aging at 550° F.

Strength properties of the more promising type adhesives Nos. 39C, 39D, and 52 at 80° and 550° F. after aging for 100 and 200 hours at 550° F. are shown in table 3. These results show that only adhesive No. 52 without copolymer or maleic anhydride modification retained any appreciable strength at 550° F. (610 pounds per square inch). The results are significant in that the use of aluminum and arsenic pentoxide as fillers serve not only to cross link the epoxy resin but also to retard its thermal degradation in bonds of stainless steel. Adhesive tape No. 52 had a strength of 2,532 pounds per square inch at 80° F. after 3 hours at 550° F. (table 2), but was lower after 100 hours at 550° F. (1,152 pounds per square inch, table 3), and after 200 hours at 550° F. had a tensile shear strength of 786 pounds per square inch at 80° F.

The shear strength of the adhesive bonds at 80° F. for similar formulations was generally higher after aging for 3 hours at 550° F. for the solution type than for the corresponding filler-tape modifications.

Formulations With Epoxy Resin E-2

Epoxy resin E-2 was a tetra glycidyl ether of tetrakis (hydroxyl phenyl) ethane with an epoxide equivalent weight of 218 grams. Results of tensile shear tests are shown in table 4.

Solution-type formulations of epoxy resin with copolymer or with copolymer and maleic anhydride had shear strengths below 200 pounds per square inch at 550° F. after aging 48 hours at 550° F. Joint strength at 550° F. was decreased as the ratio of epoxide to anhydride was increased, for example, from 1 to 0.175 in formulation 36A to a ratio of 1 to 1.05 in formulation 31B (table 4). The addition of glycerol as a cure initiator in formulations 32B3 and 32B4 produced higher bond strength at 550° F. than similar formulations, adhesives Nos. 32B2 and 36B, without glycerol.

Adhesive tape modifications with aluminum and arsenic pentoxide filler were considerably higher in strength at 550° F. than the solution-type formulations. All tape formulations had a tensile shear strength exceeding 400 pounds per square inch at 550° F. after aging 48 hours. Shear strength after aging for 100 and 200 hours at 550° F. (table 3) were usually lower than after 48 hours at 550° F., particularly for adhesive No. 31C (table 4) which had a high amount of anhydride in the formulation. Bond strengths at room temperature after aging 100 and 200 hours were significantly lower than after shorter periods of heat aging.

Adhesive tape No. 51 (table 4) a formulation of the epoxy resin without anhydride modification, had the highest strength at 550° F. of the E-2 resin formulations with a strength of 794 pounds per square inch after 48 hours at 550° F. However, joint strength of this adhesive at 80° and 550° F. after aging for 100 and 200 hours (table 3) was much lower.

In adhesives 58 through 58-3, the amount of arsenic pentoxide was varied from 8 to 32 percent. Joint strength at 550° F. after aging 48 hours was increased to 732 pounds per square inch but strength after 3 hours' aging was decreased as the amount of arsenic pentoxide was increased. These results tended to indicate that the arsenic pentoxide may have retarded the rate of cure, but contributed to the resistance of the adhesive to thermal deterioration and greater degrees of cross-linking with higher shear strength when cured adequately by longer aging at 550° F. The strength of lap joints at room temperature was not changed appreciably by increasing the amount of arsenic pentoxide in the formulation. The increased amounts of arsenic pentoxide had no marked beneficial effect on resistance to long-time heat aging, as seen from the results of shear tests at 550° F. after aging for 100 and 200 hours at 550° F. (table 3).

Formulations with Epoxy Resin, E-3

Epoxy resin E-3 was a diglycidyl ether of bisphenol A with a reported epoxide equivalent weight of 193 grams. The results of tensile shear tests of adhesive formulations of this resin are shown in table 5. Bonds of the various formulations were low in strength at 550° F. after aging for 48 hours at 550° F. No appreciable increase in strength was obtained when tape modification with aluminum and arsenic pentoxide filler was used.

Formulations with Epoxy Resin, E-4

Epoxy resin E-4 was an epoxy Novalac-type resin with an epoxy equivalent weight of 176 grams. Test results in table 6 show that shear strength of the solution type adhesive at 550° F. after 48 hours at 550° F. was increased moderately as smaller amounts of anhydride copolymer and maleic anhydride were used. The most marked increase in strength at 550° F. after 48 hours was obtained with the tape modification (adhesive No. 55) with aluminum and arsenic pentoxide filler. The joint strength at 80° and 550° F. of bonds made with this adhesive (No. 55), however, were decreased by aging for 100 or 200 hours at 550° F. (table 3).

Formulations with Epoxy Resin, E-5

Epoxy resin E-5 was a silicone-epoxy resin with an epoxy equivalent weight of 174 grams. The tensile shear tests of various formulations with the adhesive, shown in table 7, were low at both room temperature and 550° F. except for adhesive formulation No. 54. Adhesive No. 54 was a tape modification with aluminum and arsenic pentoxide filler and had shear strengths of about 1,000 pounds per square inch at 80° F. and about 400 pounds per square inch at 550° F. after 48 hours at 550° F. The shear strength of bonds made with adhesive No. 54 after aging for 100 and 200 hours at 550° F., table 3, was appreciably lower than after 48 hours of aging. The marked increase in strength with use of aluminum and arsenic pentoxide in the formulation No. 54 suggests that further study of these materials with epoxy resin E-5 may result in even better strength properties and resistance to heat aging.

Formulations with E-6, E-7, and E-8 Resins

Results of bonding with adhesive formulations of epoxy resins E-6, E-7, and E-8 are shown in table 8. Resin E-6 was a dicyclopentadiene dioxide epoxy monomer with a reported epoxy equivalent weight of about 82 grams. Resin E-7 was a dicyclodiepoxy carboxylate monomer with an epoxy equivalent

weight of about 140 grams. Resin E-8 was an unsaturated polyolefin with an epoxy equivalent weight of about 145 grams.

An adhesive tape formulation of resin E-6 with copolymer, maleic anhydride, glycerol, aluminum, and arsenic pentoxide, (adhesive No. 47B, table 8) had higher strength at 80° and 550° F. after aging 48 hours at 550° F. than the solution-type formulation, No. 47. An adhesive tape-filler formulation, No. 57, without maleic anhydride and glycerol was low in strength at elevated temperatures. A tape-filler formulation, No. 53, of resin E-6 alone without anhydride or glycerol modification had no strength and fell apart when removed from the press after a cure of 60 minutes at 400° F. The most promising of these E-6 formulations, No. 47B, when aged for 100 and 200 hours at 550° F. and tested, had practically no strength (table 3).

Adhesive formulations of resins E-7 and E-8 (table 7) even with aluminum and arsenic pentoxide filler were low in strength at 550° F. after heating for 48 hours at 550° F.

Conclusions

Adhesive formulations of polyamide resins combined with various melamine-type compounds are not considered promising materials for heat-resistant adhesives for bonding of metal. The polyamide resin was shown to have high resistance to thermal degradation in bonds of steel, but attempts to develop cross linkage and strength at elevated temperatures with the melamine components were unsuccessful.

Epoxy resins combined with a vinyl-type copolymer of ethyl acrylate and maleic anhydride and with the chemical maleic anhydride were found to have promise as heat-resistant adhesives for stainless steel. Some cross linkage was presumably formed as joint strengths approaching 200 pounds per square inch at 550° F. were obtained after joints had aged for 48 hours at 550° F. Epoxies of the allyl glycidyl ether type of bisphenol A (E-1 and E-3), tetrakis (hydroxyl phenyl) ethane (E-2), and phenol Novalac resin (E-4) were most promising.

A significant increase in bond strength at 550° F. was obtained when certain of the epoxy resin-anhydride formulations containing 100 percent of aluminum powder (Alcoa 123) and 16 percent of arsenic pentoxide, based on the weight of resin components, were formed into a tape adhesive of asbestos cloth 28-PO-47 sized with A-1100 silicone resin. The use of these inorganic components, aluminum, and arsenic pentoxide, increased the bond strength to 400 pounds per square inch and higher at 550° F. Greater amounts of arsenic

pentoxide up to 32 percent decreased the strength of bonds tested at 550° F. after having aged for only 3 hours at 550° F. but caused an increase in strength in bonds aged for 48 hours at 550° F. Presence of arsenic pentoxide thus appeared to retard the initial rate of cure but contributed to a higher degree of cure and cross linkage and greater resistance to thermal degradation when adhesives were heat aged for longer periods of time. The most promising epoxy resins in these tape-adhesive formulations were the glycidyl ether types, resins E-1 and E-2. A strength of about 400 pounds per square inch at 550° F. was retained after 200 hours' aging at 550° F. with the formulation No. 58-1 containing resin E-2, vinyl anhydride copolymer, aluminum, and arsenic pentoxide impregnated into an asbestos cloth tape.

High strength at 550° F. was obtained also with certain epoxy resins, without anhydride modification, with the aluminum and arsenic pentoxide filler in the asbestos tape form. The most promising of these formulations was adhesive No. 52 with resin E-1, 100 percent of aluminum dust, and 16 percent of arsenic pentoxide impregnated into asbestos cloth which had a strength of 610 pounds per square inch at 550° F. after aging 200 hours at 550° F.

The rather remarkable results with arsenic pentoxide as a curing agent and degradation inhibitor for epoxy resins emphasizes the need for further research on this element and its compounds for use in adhesives for metal. Similar elements which exhibit properties of both the metals and nonmetals or are amphoteric, such as antimony, tin, zinc, and aluminum would be of interest also.

Table 1.--Tensile shear test values of stainless steel lap joints bonded with various formulations of polyamide resin and melamine compounds

Adhesive composition ¹		Bonding conditions ²		Average shear test values at	
PA-1: DAM	Para: MF-1	MA	TEA	Number: Precure: Pressure:	80° F.: 550° F.: 550° F. aged ³
Mol	Mol	Mol	Mol	Min.	P.s.i.: P.s.i.: P.s.i.: P.s.i.: P.s.i.
1 : 0.5	:	:	:	30	50 : 1,712 : 686 : 0 : 0
1 : .5	1	:	:	30	50 : 2,572 : 532 : 0 : 0
1 : .5	2	:	:	30	50 : 2,506 : 486 : 0 : 0
1 : .5	2	:	0.145	30	50 : 2,600 : 586 : 0 : 0
1 : .5	1	0.5	:	30	200 : 1,948 : 450 : : 0
1 : 1	2	:	:	60	100 : 2,192 : 410 : 0 : 0
1 : 1	2	:	.29	30	200* : 2,546 : 724 : 0 : 0
1 : 1	4	:	.145	60	100 : 2,036 : 650 : 0 : 0
1 :	0.5	:	:	60	200 : 3,532 : 1,222 : 0 : 0
1 :	.75	:	:	30	200 : 2,760 : 666 : 40 : 0
1 :	1.5	:	:	30	400 : 752 : 550 : 92 : 34
1 :	:	.5	:	30	50 : 286 : 1,496 : 0 : 0
1 :	1	.5	:	30	200 : 280 : 1,140 : 0 : 0
1 :	1	:	:	15	50* : 100 : 586 : 0 : 0

¹Molecular weight of the polyamide resin (PA-1) was taken as 113 grams and of the melamine resin (MF-1) as 258 grams. Other abbreviations denote DAM for diallyl melamine, para for para-formaldehyde, MA for maleic anhydride, and TEA for triethylamine.

²Bonds cured at 320° F. for 6 minutes, except those with an asterisk, which were cured at 400° F. for 60 minutes.

³Aged 3 hours at 550° F. before testing.

Table 2.--Results of tensile shear tests of lap joints of stainless steel bonded with various formulations of epoxy resin, E-1

Adhesive No.	Adhesive composition ¹				Tape ²	E/A ratio ³	Shear strength		Shear strength	
	E-1	C-1	MA	Gly	Al	As ₂ O ₅		at 80° F.	at 550° F.	
										No : Aged 3 : Aged 48 : hours at: hours at: hours at: : aging : hours at: hours at: : 550° F.: 550° F.: 550° F.
								P.s.i.	P.s.i.	P.s.i.
39A	1	0.18					1/0.18	2,412	2,322	150
39C	1	.18					1/0.18	1,486	1,586	164
35A	1	.27				100	1/0.27	2,746	2,466	186
42	1	.27				100	1/0.27	1,424	1,740	152
39B	1	.18	0.09				1/0.27	1,372	2,042	90
39D	1	.18	.09			100	1/0.27	1,406	1,280	136
38B	1	.27	.13				1/0.40	2,272	2,180	102
30A	1	.54					1/0.54	1,590	1,910	118
37B	1	.27	.27				1/0.54	1,711	1,540	98
37C	1	.27	.27	0.27			1/0.54	1,992	712	112
35B	1	.27	.54				1/0.81	1,416	602	120
43	1	.27	.54	.40			1/0.81	1,282	1,772	146
35D	1	.27	.54			100	1/0.81	1,492	750	32
49	1	1.0					1/0.1	1,082	1,550	100
30B	1	.54	.54				1/1.08	682	1,400	62
30C	1	.54	.54	.54			1/1.08	1,200	528	82
23C	1	.54	1.08			100	1/1.62	930	1,262	102
52	1.0					100	1/0	1,426	2,532	44

¹E-1 column denotes the number of epoxy equivalent weights of epoxy resin, E-1; C-1 column denotes the number of anhydride equivalent weights of copolymer, C-1; MA column denotes the number of anhydride equivalent weights of maleic anhydride; and Gly column denotes the number of hydroxyl equivalent weights of glycerol. The percent figures in the Al column denote the percent by weight of aluminum powder, Alcoa 123, and those in the column headed As₂O₅ of arsenic pentoxide based on the weight of the dry polymer solids.

²This column denotes that asbestos cloth was used where indicated by symbol X to form a tape-type adhesive.

³The ratio of epoxide equivalents to anhydride equivalents.

Table 3.--Results of tensile shear tests of lap joints of stainless steel bonded with various asbestos tape formulations of epoxy resins after aging for 100 and 200 hours at 550° F.

Adhesive: No.	Adhesive composition	E/A 1	Aged 100 hours	Aged 200 hours
		ratio	and tested at	and tested at
	Epoxy: C-1 : MA : As ₂ O ₅			
	resin:		80° F.: 550° F.:	80° F.: 550° F.
		Percent:	P.s.i. : P.s.i.:	P.s.i. : P.s.i.
Epoxy E-1:				
39C	1 : 0.18 : .175 : .35	16	1/0.18 : 232	198 : 146
39D	1 : .18 : 0.09 : .35	16	1/0.27 : 40	30 : 46
52	1 : .18 : .18 : .35	16	1/0 : 1,152	644 : 786
Epoxy E-2:				
36C	1 : .175 : .35 : .70	16	1/0.175 : 332	386 : 282
36D	1 : .175 : .35 : .70	16	1/0.505 : 426	440 : 280
31C	1 : .35 : .70 : .25	16	1/1.05 : 180	126 : 176
58	1 : .25 : .25 : .25	8	1/0.25 : 382	406 : 312
58-1	1 : .25 : .25 : .25	16	1/0.25 : 436	330 : 412
58-2	1 : .25 : .25 : .25	24	1/0.25 : 386	376 : 356
58-3	1 : .25 : .25 : .25	32	1/0.25 : 360	228 : 346
51	1 : .25 : .25 : .25	16	1/0 : 622	462 : 402
Epoxy E-4:				
55	1 : .25 : .25 : .25	16	1/0.25 : 426	240 : 306
Epoxy E-5:				
54	1 : .25 : .25 : .25	16	1/0.25 : 120	144 : 96
Epoxy E-6:				
47B	1 : .1 : .3 : .3	16	1/0.25 : 20	0 : 14

Ratio of epoxide equivalents to anhydride equivalents in adhesive formulations.

Table 4.--Results of tensile shear tests of lap joints of stainless steel bonded with various formulations of epoxy resin, E-2

Adhesive: No.	Adhesive composition ¹					Tape ² ratio ³	Shear strength		No : Aged 3 : Aged 48 aging : hours at: hours at: hours at : 550° F.: 550° F.: 550° F.
	E-2	C-1	MA	Gly	Al	As ₂ O ₅	at 80° F.	at 550° F.	
					Percent		P.s.i.	P.s.i.	P.s.i.
36A	1.0	0.175				1/0.175	2,202	1,212	104
36C	1.0	.175			100	16	1,002	1,792	210
32B2	1.0	.125	0.125			1/0.25	1,884	2,046	196
32B3	1.0	.125	.125	0.125		1/0.25	1,606	1,960	196
32B4	1.0	.125	.125	.125	100	16	1,106	1,584	314
32A	1.0	.35				1/0.35	1,190	702	32
32B1	1.0	.25	.25			1/0.50	972	1,532	140
36B	1.0	.175	.35			1/0.505	1,670	700	50
36D	1.0	.175	.35		100	16	1,052	1,472	208
44	1.0	.175	.35	.212		1/0.505	1,380	540	146
32B	1.0	.35	.37			1/0.72	772	590	5
31B	1.0	.35	.70			1/1.05	1,096	516	32
31C	1.0	.35	.70		100	16	1,012	1,332	172
51	1.0				100	16	1,356	830	384
58	1.0	.25			100	8	1,080	1,286	214
58-1	1.0	.25			100	16	950	1,236	160
58-2	1.0	.25			100	24	952	1,096	172
58-3	1.0	.25			100	32	1,082	1,222	66

¹E-2 column denotes the number of epoxy equivalent weights of epoxy resin, E-2; C-1 column denotes the number of anhydride equivalent weights of copolymer, C-1; MA column denotes the number of anhydride equivalent weights of maleic anhydride; and Gly column denotes the number of hydroxyl equivalent weights of glycerol. The percent figures in the Al column denote the percent by weight of aluminum powder, Alcoa 123, and those in the column headed As₂O₅ of arsenic pentoxide based on the weight of the dry polymer solids.

²This column denotes that asbestos cloth was used where indicated by symbol X to form a tape-type adhesive.

³The ratio of epoxide equivalents to anhydride equivalents.

Table 5.--Results of tensile shear tests of lap joints of stainless steel bonded with various formulations of epoxy resin, E-3

Adhesive:	Adhesive composition ¹						Tape ²	E/A : Shear strength : Shear strength
No.	E-3	C-1	MA	Gly	Al	As ²⁰ 5	ratio ³ : at 80° F. : at 550° F.	
	:	:	:	:	:	:	:	
	:	:	:	:	:	:	No : Aged 3 : Aged 3 : Aged 48	
	:	:	:	:	:	:	: hours at: hours at : hours at	
	:	:	:	:	:	:	: 550° F.: 550° F. : 550° F.	
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¹E-3 column denotes the number of epoxy equivalent weights of epoxy resin, E-3; C-1 column denotes the number of anhydride equivalent weights of copolymer, C-1; MA column denotes the number of anhydride equivalent weights of maleic anhydride; and Gly column denotes the number of hydroxyl equivalent weights of glycerol. The percent figures in the Al column denote the percent by weight of aluminum powder, Alcoa 123, and those in the column headed As₂O₅ of arsenic pentoxide based on the weight of the dry polymer solids.

²This column denotes that asbestos cloth was used where indicated by symbol X to form a tape-type adhesive.

³The ratio of epoxide equivalents to anhydride equivalents.

Table 6.--Results of tensile shear tests of lap joints of stainless steel bonded with various formulations of epoxy resin, E-4

Adhesive: No.	Adhesive composition ¹						Tape ² :	E/A ratio ³	Shear strength :		Shear strength at 550° F.
	E-4	C-1	MA	Gly	Al	As ₂ O ₅			at 80° F.	No : Aged 3 : Aged 3 : Aged 48 aging : hours at: hours at: hours at: : 550° F.: 550° F.: 550° F.:	

¹E-4 column denotes the number of epoxy equivalent weights of epoxy resin, E-4; C-1 column denotes the number of anhydride equivalent weights of copolymer, C-1; MA column denotes the number of anhydride equivalent weights of maleic anhydride; and Gly column denotes the number of hydroxyl equivalent weights of glycerol. The percent figures in the Al column denote the percent by weight of aluminum powder, Alcoa 123, and those in the column headed As₂O₅ of arsenic pentoxide based on the weight of the dry polymer solids.

²This column denotes that asbestos cloth was used where indicated by symbol X to form a tape-type adhesive.

³The ratio of epoxide equivalents to anhydride equivalents.

Table 7.--Results of tensile shear tests of lap joints of stainless steel bonded with various formulations of epoxy resin, E-5

Adhesive: No.	Adhesive composition ¹				Tape ²	E/A ratio ³	Shear strength	
	E-5	C-1	MA	Gly			at 80° F.	at 550° F.
				As205			No	Aged 3 : Aged 48
							aging	hours at: hours at
							550° F.	550° F.: 550° F.
							P.s.i.	P.s.i.
							P.s.i.	P.s.i.
50-2	1.0	0.25				1/0.25	106	670
54	1.0	.25			X	1/0.25	990	1,036
50-3	1.0	.25		0.125		1/0.25	126	542
50-1	1.0	.3				1/0.3	140	546
50	1.0	.5				1/0.5	272	586

LE-5 column denotes the number of epoxy equivalent weights of epoxy resin, E-5; C-1 column denotes the number of anhydride equivalent weights of copolymer, C-1; MA column denotes the number of anhydride equivalent weights of maleic anhydride; and Gly column denotes the number of hydroxyl equivalent weights of glycerol. The percent figures in the Al column denote the percent by weight of aluminum powder, Alcoa 123, and those in the column headed As2O5 of arsenic pentoxide based on the weight of the dry polymer solids.

²This column denotes that asbestos cloth was used where indicated by symbol X to form a tape-type adhesive.

³The ratio of epoxide equivalents to anhydride equivalents.

Table 8.--Results of tensile shear tests of lap joints of stainless steel bonded with various formulations of epoxy resin. E-6, E-7, and E-8

Adhesive: No.	Adhesive composition ¹					Tape ² :	E/A ratio ³	Shear strength :	
	C-1	MA	Gly	A1	As205			at 80° F.	at 550° F.
Epoxy E-6								P.s.i.	P.s.i.
53	1.0					100	X	1/0	0
57	1.0	0.25				100	X	1/0.25	730
47	1.0	.1	0.3	0.2				1/0.4	1,930
47B	1.0	.1	.3	.2		100	X	1/0.4	940
Epoxy E-7:									
48	1.0	.1	.3	.2		100	X	1/0.4	992
Epoxy E-8:									
59	1.0	.25						1/0.25	370
59-1	1.0	.25				100	X	1/0.25	632

Epoxy resin column denotes the number of epoxy equivalent weights of epoxy resin, E-6, E-7, and E-8; C-1 column denotes the number of anhydride equivalent weights of copolymer, C-1; MA column denotes the number of anhydride equivalent weights of maleic anhydride; and Gly column denotes the number of hydroxyl equivalent weights of glycerol. The percent figures in the Al column denote the percent by weight of aluminum powder, Alcoa 123, and those in the column headed As₂O₅ of arsenic pentoxide based on the weight of the dry polymer solids.

²This column denotes that asbestos cloth was used where indicated by symbol X to form a tape-type adhesive.

³The ratio of epoxide equivalents to anhydride equivalents.

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