NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2717

EFFECT OF TEMPERATURES FROM -70° TO 600° F ON STRENGTH OF ADHESIVE-BONDED LAP SHEAR SPECIMENS OF CLAD 24S-T3 ALUMINUM ALLOY AND OF COTTON- AND GLASS-FABRIC PLASTIC LAMINATES

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Forest Products Laboratory

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SUMMARY

The performance of 14 commercial adhesives at temperatures from -70° to 600° F was evaluated in lap shear specimens of clad 24S-T3 aluminum alloy to itself and that of 7 commercial adhesives at -70° to 250° F in lap joints of cotton-fabric-phenolic laminate to itself, of glass-fabric-polyester laminate to itself, and in joints of each of these laminates to clad aluminum. One hot-setting tape adhesive was found to be significantly superior to all others in lap-joint specimens of aluminum alloy to itself and that of 7 commercial adhesives at -70° to 250° F mercial adhesives evaluated at -70° to 250° F in lap shear specimens of plastic laminates bonded to themselves and to aluminum had only fair resistance to stressing immediately upon reaching 250° F. The adhesives generally performed adequately in the various joints at -70° F.

INTRODUCTION

In the manufacture of aircraft, the bonding of panels of sandwich constructions of aluminum to various core materials and the bonding of aluminum to itself by means of adhesives offer several important advantages over other methods of fastening. Similar techniques for adhesive bonding of structural plastic laminates are also important. A number of adhesives have been formulated for use in bonding metals, and some of these adhesives have also been used for bonding plastic laminates. Other adhesives have been formulated particularly for bonding these laminates. Adhesives of these types have generally proved to be satisfactory under ordinary service conditions. In modern aircraft, however, such bonds may be exposed to very low temperatures in arctic service or at high altitudes or to very high temperatures that may occur at high speeds or in certain parts of the aircraft. Limited available information indicated that some of the current adhesives were not adequate for high-temperature service.

The work for which the results are here presented was undertaken in order to evaluate more extensively the performance in aluminum lap joints of a number of representative commercial metal-bonding adhesives at temperatures from -70° to 600° F, both when tested immediately at these temperatures and when tested after several days of exposure at temperatures from 160° to 450° F. Such performance data for these commercial adhesives were desired as a background on which to base new formulations of adhesives which might be adequate for aircraft use. Inasmuch as the field of adhesives for bonding structural parts is in a state of constant flux, as is evidenced by the frequent appearance of new commercial adhesives, as well as new or modified bonding procedures, the limited results reported herein should be viewed as representing only the relative efficiency of the adhesives when tested under the conditions specified. They are not intended for use as a basis on which to select adhesives for aircraft use.

The work herein reported was done in two separate series of tests. A first group of adhesives was systematically evaluated at a complete series of temperature conditions. A second series of adhesives was then evaluated on a much more limited scale by screening them at elevated-temperature conditions that had been found to be quite critical for the first group of adhesives. Only those adhesives considered to show unusual promise at high temperatures in these screening tests were then evaluated more thoroughly over a larger range of temperature conditions.

Because of the importance of certain high-strength plastic laminates in modern aircraft it was also desirable to make some preliminary investigations of the performance both at an elevated and at a low temperature of some of the better adhesives used in bonding these laminates to themselves. Only those adhesives that showed promising performance in these tests were further evaluated under the same conditions in bonds of these laminates to clad aluminum alloy.

This investigation was conducted at the Forest Products Laboratory under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

PROCEDURE

Adhesives

The following commercial adhesives, identified in this report by the letters preceding the descriptions given below, were evaluated in aluminum-to-aluminum joints. A code sheet identifying the adhesives by trade names or supplier is attached to the report as appendix A.

A high-temperature-setting formulation of a thermosetting resin and synthetic rubber (believed to be neoprene)

B two-stage process; primary adhesive was A and secondary adhesive was an acid-catalyzed, intermediate-temperature-setting, phenol-resin adhesive

C high-temperature-setting modified vinyl-resin formulation

D high-temperature-setting modified vinyl-resin formulation

E high-temperature-setting, two-component, modified vinyl-resin formulation

F high-temperature-setting modified vinyl-resin formulation

G high-temperature-setting formulation of a thermosetting resin and synthetic rubber (believed to be butadiene-acrylonitrile)

H high-temperature-setting silicone resin

I high-temperature-setting adhesive assumed to be a formulation of a phenol resin and a polyvinyl-butyral resin

J high-temperature-setting adhesive in powder form assumed to be a formulation based on epichlorohydrin resins

K high-temperature-setting adhesive assumed to be of a modified polyvinyl-butyral base

L room-temperature-setting adhesive assumed to be a formulation based on epichlorohydrin resins

M room-temperature-setting adhesive assumed to be a formulation based on epichlorohydrin resins

N high-temperature-setting tape adhesive assumed to be a formulation of phenol resin and synthetic rubber (probably butadiene-acrylonitrile)

In addition to adhesives D and M, the following adhesives were used in plastic-to-plastic and in plastic-to-aluminum joints.

O room-temperature-setting resorcinol resin

P room-temperature-setting polyester-resin formulation with benzoyl-peroxide catalyst and a commercial promoter

Q room-temperature-setting adhesive assumed to be a formulation based on epichlorohydrin resins

R high-temperature-setting polyester-resin formulation with benzoyl-peroxide catalyst

S room-temperature-setting furane-resin formulation

Test Panels and Specimens

Aluminum-to-aluminum joints.- Test panels of aluminum-to-aluminum joints were prepared by bonding two sheets of 0.064-inch-thick clad 24S-T3 aluminum alloy, each 5 by 8 inches in area, with an overlap of 0.5 inch along the 8-inch length. The panel and test specimen are shown in figure 1. Six specimens, each 1 inch wide and 9.5 inches long, were cut from each panel with a special power saw in such a manner that overheating or mechanical damage to the specimen was reduced to a minimum.

Plastic-to-plastic and plastic-to-aluminum joints.- Lap shear specimens of four types were prepared. These were:

(1) Type A: Grade L cotton-fabric-phenolic laminate to itself

(2) Type B: Glass-fabric (181-114)-polyester laminate to itself

(3) Type C: 24S-T3 clad aluminum to grade L cotton-fabric-phenolic laminate

(4) Type D: 24S-T3 clad aluminum to glass-fabric (181-114)-polyester laminate

Panels for specimens of types A and B were prepared by bonding two sheets of the laminate, each 1/8 inch thick and $1\frac{7}{8}$ by $10\frac{1}{2}$ inches in area, with a 0.5-inch lap along the $10\frac{1}{2}$-inch side, as shown in figure 2. Eight specimens, each 1 inch wide and $3\frac{1}{4}$ inches long as shown, were cut from each of these panels with a special power saw in such a manner that mechanical damage and overheating of the specimen were reduced to a minimum.

Panels for specimens of types C and D were prepared by bonding strips of 0.032-inch-thick 24S-T3 clad aluminum alloy, each $1\frac{7}{8}$ by $10\frac{1}{2}$ inches in area, with a strip of 0.032-inch-thick plastic laminate 1/2 inch by $10\frac{1}{2}$ inches in area, inserted between the sheets of aluminum so that each edge of the aluminum overlapped the laminate, as shown in figure 3. Eight specimens, each 1 inch wide and $3\frac{1}{4}$ inches long, were cut from each of these panels with a special power saw, as for types A and B.

Preparation of Surfaces for Adhesive Bonding

Aluminum.- Lettering and surface grease were first removed from the aluminum sheets by wiping with a benzene-soaked cloth. The sheets were then immersed for 5 minutes at 140° to 160° F in a bath of the following composition:

Ingredient	Parts by weight
Sodium dichromate	1
Sulfuric acid (concentrated)	10
Water	30

After removal from this bath the sheets were rinsed thoroughly in cold water, then in hot water, and then allowed to air-dry.

Plastic laminates.- Plastic laminates were prepared by light sanding with medium-fine emery cloth and then wiped with a clean cloth saturated with ethyl acetate.

Procedures for Bonding

The conditions used for bonding the aluminum-to-aluminum joints are given in table 1, and the conditions for bonding plastic laminates to themselves and to aluminum are given in table 2. The adhesive was always spread within 4 hours after cleaning the metal or plastic laminate. After their removal from pressure all panels were conditioned for 7 days at 80° F and 30-percent relative humidity before individual test specimens were cut from them as previously described.

In bonding aluminum to aluminum with adhesives A to N the bonding conditions were within the range recommended by the manufacturer, with the exception of adhesive H, which was primarily intended as a metal primer used in combination with other adhesives. The manufacturer's recommendations for bonding conditions for adhesive H therefore were not available. This silicone resin was used experimentally as an adhesive in this study because of the reputation of silicones as heat-resistant materials.

Specific recommendations were not usually made by the adhesive manufacturers for use of their adhesives in bonding aluminum to plastic laminates or plastic laminates to themselves. However, the adhesives used in bonding these materials were used following the manufacturers' general recommendations for the adhesives, with the exception of adhesive P, which was used with special promoters in an attempt to formulate an adhesive capable of curing at normal room temperatures.

Testing Procedure

Aluminum-to-aluminum specimens.- The aluminum-to-aluminum specimens were tested in tension shear in a universal-type testing machine. At -70° and at 80° F the specimens were held in self-alining wedge grips. At the other temperatures the load was applied through special slotted grips by means of pins 5/16 inch in diameter, which were inserted through holes (3/8 in. in diam.) drilled 3/4 inch from each end of the specimen. These grips were self-alining. The latter method permitted rapid changing of specimens in the test machine and, therefore, reduced temperature fluctuations because the test-chamber door was open for shorter periods of time. This method of loading with pins could not be used at -70° to 80° F because at these temperatures the specimens failed at the holes where the pins were inserted. At the higher temperatures the adhesive bonds were not strong enough to reach this level of strength of the metal. The load was applied at the rate of 600 pounds per minute until failure occurred.

After failure, each broken specimen was examined and the percentages of failure that occurred in cohesion in the bonding agent and in adhesion of the bonding agent to the metal were noted.

Plastic-to-plastic and plastic-to-aluminum specimens.- The plastic-to-plastic and plastic-to-aluminum specimens were tested in tension shear in a universal-type testing machine. The specimens were held by grips of the plywood-testing type with the jaws offset so that eccentricity was reduced to a minimum. Loading was at a rate of 600 pounds per minute until failure occurred. After failure, each specimen was examined and the percentages of failure that occurred in the laminate or in adhesion to the laminate or to the aluminum were noted.

Number of Test Panels and Specimens

Aluminum-to-aluminum joints.- The number of aluminum-to-aluminum test panels prepared depended on the number of tests to be made. For the series of tests with adhesives A to G and with adhesive N, 17 to 20 panels were prepared so that representative groups of six specimens could be tested under each of the test conditions shown for these adhesives in table 3. With adhesives H to M, which were given screening tests but were not included in complete evaluation tests, three panels were prepared and provided similar representative groups of six specimens for preliminary tests. Later, additional panels were prepared with the same adhesives under the same conditions and tested under other conditions. In such cases an extra control group of six specimens was always retested at 80° F. The results of tests of these control groups were in close agreement, except as noted in the tables.

Plastic-to-plastic and plastic-to-aluminum joints.- Three panels were prepared for specimens of types A, B, C, and D with each adhesive and each plastic. Each panel provided eight test specimens. The 24 specimens cut from these panels were then sorted into three representative groups of eight each for the various tests described in the section entitled "Test Conditions."

Test Conditions

Aluminum-to-aluminum joints.- In a complete series of tests, groups of six aluminum-to-aluminum specimens were tested at each of the following conditions:

(1) Test 1: Tested immediately upon reaching equilibrium with each of the following temperatures: -70°, 80°, 140°, 160°, 200°, 250°, 350°, 450°, and 600° F

(2) Test 2: Tested after exposure for 192 hours (8 days) at each of the following temperatures and while at these temperatures: 160°, 250°, 350°, and 450° F

(3) Test 3: Tested at room temperature after 192 hours (8 days) at each of the following temperatures: 160°, 250°, 350°, and 450° F

(4) Test 4: Tested at -70°, 80°, or 400° F after exposure to three repeating cycles, each consisting of 3 hours at -70° F followed by 21 hours at 400° F

Temperatures were checked frequently with thermocouples.

For screening tests, adhesives were first evaluated by testing at 80° F, at 450° F after 192 hours at this temperature, and at 80° F after 192 hours at 450° F.

Plastic-to-plastic and plastic-to-aluminum joints.- One group of eight plastic-to-plastic specimens of each plastic (types A and B) was tested at 80° F. Adhesives that were considered promising on either plastic in these tests at 80° F were evaluated further by testing one of the other groups of specimens at -70° F and the other at 250° F as soon as the specimens had reached equilibrium at these temperatures. Adhesives that showed promise in these tests at -70° and at 250° F, and which on the basis of other work were shown to be suitable for bonding aluminum, were further investigated for bonding the same plastic laminates to clad aluminum alloy by preparing specimens of types C and D. These were tested at 80° and at -70° and 250° F immediately after reaching equilibrium at these temperatures.

RESULTS

Aluminum-to-Aluminum Joints

Results of the tests of the various commercial adhesives in aluminum-to-aluminum joints are given in table 3. These results are the average of a number of tests as shown in the table. Table 4 shows the maximum and minimum joint strengths obtained in these tests.

Most of the adhesives had satisfactory strength when tested initially at 80° F. Exceptions were adhesives G and H. These were probably incompletely cured under the curing conditions used, since there was evidence to indicate that their over-all joint performance had improved somewhat more than that of some of the other adhesives when subsequently heated at 450° F in the 192-hour exposures. As presently formulated, however, adhesives G and H could not be considered to be practical adhesives for bonding aluminum to itself for high-strength joints.

Only adhesives G and N were considered to have promise for service at extreme high-temperature conditions, in that these adhesives were the only ones evaluated that had reasonably good strength after 192 hours' exposure at 450° F when tested both at 450° F and at 80° F (439 and 486 lb per 0.5 sq in. average strength, respectively, for G and N at 450° F, and 605 and 606 lb per 0.5 sq in. average strength, when tested at 80° F after exposure at 450° F). Adhesives E and H had average strengths of 608 and 592 pounds per 0.5 square inch, respectively, when tested at 80° F after 192 hours at 450° F, but they retained only 161 and 100 pounds per 0.5 square inch, respectively, when tested at 450° F after this exposure. Other adhesives gave lower values in both tests, always being lower in strength when tested at 450° F than when tested at 80° F after this heat exposure. Adhesive N was the best in performance at 600° F when joints were tested immediately upon reaching equilibrium at this temperature, having an average strength of 114 pounds per 0.5 square inch, but this strength is probably of little significance in a consideration of structural applications.

All adhesives lost significant proportions of their initial strength at 80° F when they were tested immediately after reaching equilibrium at temperatures of 160° F or higher. The magnitude of such loss in strength for each adhesive generally increased as the temperature of test increased except for adhesives B, C, and D. Adhesives A, B, and N were superior at the intermediate test temperatures, retaining considerably higher strengths at 250° F or higher than did any of the other adhesives tested immediately at these temperatures. It should be noted that these three adhesives were presumably formulated of phenol resins and synthetic rubbers and that other adhesives, believed to be composed primarily of phenol resins and thermoplastic vinyl resins, such as C, D, E, and F, had definitely lower strengths when tested immediately at 250° F or higher. Adhesive G, also believed to be a synthetic-rubber formulation, had low resistance to stressing at 250° F or higher. Adhesives K, L, and M, the latter two being room-temperature-setting epichlorohydrin-resin formulations, had the lowest resistance to stressing at 140° and 200° F and were the least heat resistant of all adhesives evaluated in this study.

The relative behavior of several adhesives when tested immediately at 250°, 350°, or 450° F, and at either 80° F or these same temperatures after 192 hours at these elevated temperatures, is interesting and may disclose valuable information on the characteristics of certain resin formulations. The 192 hours' exposure to 250° and 350° F improved the strength of the joints made with adhesives C, F, and G when they were tested at 80° F following the exposure at elevated temperature, and for adhesive D when tested at 80° F after exposure at 250° F only. In some cases this improvement was significant, as was that for adhesive G, which was previously noted to be slow in initial curing. Joints made with this adhesive increased in average strength from 778 (initial value) to 1635 pounds per 0.5 square inch when tested at 80° F after the 192-hour exposure at 350° F. Under the same conditions, joints made with adhesive C increased in average strength from 1781 to 2495 pounds per 0.5 square inch, and those made with adhesive F, from 1182 to 1720 pounds per 0.5 square inch. Other adhesives, such as A, D, and N, lost a significant amount of strength because of this 192-hour exposure at 350° F. The improvement in strength noted in adhesives C, F, and G was probably due, at least in part, to further cure or cross-linking than that which resulted under the initial curing conditions noted in table 1. Such further cure was also indicated by the increases in strength noted between the results of tests of specimens upon first reaching equilibrium at 250° and 350° F and by the results of tests at these elevated temperatures after 192 hours of exposure at these same temperatures. Joints made with adhesive B, for example, had an initial average strength of 498 pounds per 0.5 square inch when tested upon first reaching 350° F and an average strength of 722 pounds per 0.5 square inch when tested at 350° F after 192 hours at this temperature. Adhesives C, D, E, F, G, and N showed similar increases. More striking increases were noted at 250° F. Joints made with adhesive D increased in average strength from 93 to 919 pounds per 0.5 square inch after the 192-hour exposure at this temperature. Somewhat smaller increases were also noted for adhesives B, C, E, F, and N. Another striking example was noted for adhesive G at 450° F, where the average initial strength at 450° F of 46 pounds per 0.5 square inch increased to 439 pounds per 0.5 square inch after 192 hours at this temperature. Increases in strength at 450° F were also noted for adhesives C, D, E, H, and N, although, with the exception of adhesive N, the improvements were probably of little practical significance. The strength of joints tested at the elevated temperature after the 192 hours of exposure was, in general, considerably lower than when tests were made at 80° F after the same exposure.

The performance of adhesives A and B was, in general, different from that of the other adhesives in the 192-hour exposure at elevated temperatures. Joints made with adhesive A, in particular, had lower strengths after the 192-hour exposure at 250°, 350°, and 450° F than similar joints tested upon reaching these temperatures. Joints made with adhesives A and B both lost essentially all strength after the 192-hour exposure at 450° F when tested at 450° F, and they regained none of this strength when cooled to 80° F after this exposure. It should be noted that these two adhesives, A and B, had the highest average strengths of all adhesives evaluated (475 and 510 lb per 0.5 sq in., respectively) when tested upon coming to equilibrium with 450° F, but that when tested either at 80° or at 450° F after 192 hours at 450° F, these same adhesives had the poorest performance.

These data, then, show interesting differences in both the initial thermostability of adhesives at elevated temperatures, probably because of variations in thermoplasticity of the resins involved, and in the resistance to thermal degradation of these adhesives after a period of 8 days at these same elevated temperatures. Loss of strength in the long exposure may be caused either by actual chemical degradation of the resins or by further curing of the resin beyond an optimum point that results in glue lines with excessively high strains that produce failure at lower levels when stressed in the testing procedure used in the present study. Adhesive N is believed to represent the most successful compromise between an adhesive having sufficient strength after initial cure, one having sufficient resistance to thermal softening over a range of elevated temperatures, and one having resistance to further degradation as a result of exposure at these elevated temperatures for several days.

Joints tested immediately upon reaching equilibrium at -70° F generally had lower strengths than those tested at 80° F. Exceptions were noted for adhesives A and G. The former had an average strength of 1530 pounds per 0.5 square inch at 80° F and of 2813 pounds per 0.5 square inch at -70° F and the latter had strengths of 778 and 1493 pounds per 0.5 square inch, respectively, under the same conditions. Adhesives E and N showed decreases of only 11 and 7 percent, respectively, when tested at -70° F, while adhesives B, C, D, and F lost from 32 to 60 percent of their average strength at 80° F when tested at -70° F. The joints were always much stronger at -70° F than at 250° F or higher, either when tested upon reaching the temperature (test 1) or after 192-hour exposures to such elevated temperatures (test 2). Performance of these adhesives in such aluminum-to-aluminum joints at -70° F did not appear to be so serious a limitation to their utilization as was their performance at the elevated temperatures.

The cyclic exposure at -70° and 400° F (test 4) was not so severe as the 192-hour exposure at 350° or 450° F (tests 2 and 3). Results in test 1 at -70° F indicate that the part of the cycle involving the low temperature probably had less effect on joint performance than did the portion at 400° F. When the specimens were tested at 80° F after the three cycles strength values were generally higher than when specimens were tested after exposure for 192 hours at 350° F (test 2) and were generally lower than when tested at 80° F after 192 hours at 350° F (test 3). Tests made at 80° F after the cyclic exposure generally gave higher strength values than those obtained in tests at 80° F after heating for 192 hours at 450° F (test 3). Adhesive B had the highest strength (558 lb per 0.5 sq in.) when tested at 400° F after cyclic exposure.

Plastic-to-Plastic and Plastic-to-Aluminum Joints

The results of tests of plastic-to-plastic and plastic-to-aluminum joints are given in tables 5 and 6. The averages of the results of a number of tests are given in table 5 and the maximum and minimum joint strengths obtained in these tests are given in table 6.

Adhesives O, P, and R gave average strengths of 322, 327, and 421 pounds per 0.5 square inch, respectively, in phenolic laminate specimens at 80° F and were not considered to be sufficiently promising to justify further investigation in this work. Adhesive O was also not considered promising enough for bonding glass-fabric laminates to justify further evaluation. Adhesives D, M, Q, and S gave initial average strengths at

80° F of 639 to 648 pounds per 0.5 square inch in the phenolic laminate and resulted in large amounts of plastic failure. Adhesive D was the most promising at 250° F, retaining an average strength of 414 pounds per 0.5 square inch, and adhesive S was next with 276 pounds per 0.5 square inch; adhesives M and Q, in turn, retained 188 and 118 pounds per 0.5 square inch, respectively, at 250° F. All four adhesives retained all of their initial strength at 80° F when tested at -70° F in phenolic laminate joints.

In joints of glass-fabric laminate to itself, adhesive D gave considerably higher initial average strength at 80° F (1100 lb per 0.5 sq in.) than it did in phenolic laminate joints, mainly because of less failure in the stronger glass-fabric plastic laminate. Strength results with adhesives M and Q in glass-laminate specimens were about the same at 80° F as they were in phenolic laminate specimens, and similar joints made with adhesive S were somewhat lower in average strength at 80° F (420 lb per 0.5 sq in.) than for this adhesive in phenolic laminate joints (639 lb per 0.5 sq in.). Performances of these adhesives at 250° F in the glass-fabric laminates were quite similar to those in the phenolic laminates, with strength values for adhesives D and S being somewhat lower and the strength value for adhesive M being somewhat higher on glass-fabric laminates. Adhesive D, the only one used in aluminum-to-aluminum joints and tested immediately after reaching equilibrium at 250° F, had an average strength of 93 pounds per 0.5 square inch in metal-to-metal joints at 250° F, as previously mentioned, as compared with 414 and 303 pounds per 0.5 square inch in the plastic-to-plastic joints of the two laminates in the present study. All adhesives in the present study of glass-fabric-laminate joints retained essentially all of their initial strength at 80° F when tested at -70° F except D, and the strength of the joints with adhesive D at -70° F was still 862 pounds per 0.5 square inch.

Only adhesives D, M, and Q were considered to be of sufficient promise to be evaluated in plastic-laminate-to-aluminum joints, since previous experience in other studies had indicated that adhesives P, R, and S were not promising for bonding to aluminum. Adhesives D and Q produced initial joints with both laminates to aluminum that were approximately equal in strength at 80° F, whereas adhesive M was somewhat inferior. The relative performance of these adhesives at 250° F was somewhat similar to that of the same adhesives in plastic-to-plastic joints, with adhesive D being considerably superior to adhesives M and Q. Performance of these same adhesives in plastic-to-metal joints at -70° F was distinctly poorer than that for these same adhesives at 80° F, or than that in plastic-to-plastic joints at -70° F. Failures were largely in adhesion to the metal or to the plastic, which may have been caused by excessive differential stresses exerted in the glue lines between the dissimilar materials at this low temperature. Adhesive D was the best of the three adhesives at -70° F, retaining 60 and 84 percent, respectively, of the average strength at 80° F in the joints of the phenolic laminate to aluminum and of the glass-fabric laminate to aluminum.

Adhesive D, a hot-setting formulation, was thus the most promising adhesive for bonding both plastic laminates to themselves and to aluminum where resistance to stressing at both -70° and 250° F is important. The two room-temperature-setting formulations M and Q were inferior to D in resistance to softening at 250° F and, to a lesser extent, to resistance to stressing at -70° F in the plastic-to-metal joints.

SUMMARY OF RESULTS

The following results are derived from an investigation of the effect of temperatures from -70° to 600° F on the bond strength of lap shear specimens of clad 23S-T3 aluminum alloy and of cotton- and glass-fabric plastic laminates.

1. The most promising commercial adhesive tested, from the standpoint of resistance to temperatures of 450° F or higher, was adhesive N, a hot-setting tape adhesive assumed to be a formulation of phenol resin and synthetic rubber, probably butadiene-acrylonitrile. None of the other adhesives tested was considered to be as promising for applications where resistance to temperatures of 450° F or higher is required, although adhesive G, a slow-curing adhesive, also believed to be a formulation of phenol resin and a synthetic rubber, had promising resistance to prolonged exposure at high temperatures. Adhesives A and B, containing another type of synthetic rubber, were not promising for high-temperature service.

2. Adhesive D, a high-temperature-setting, modified vinyl-resin formulation, was the only adhesive investigated that had reasonably good adhesion in aluminum-to-aluminum, in glass-cloth-phenolic laminates to themselves, and in laminate-to-aluminum joints and which had any reasonably good resistance to temperatures as high as 250° F.

3. Of the adhesives found to be suitable for bonding phenolic-cotton-fabric or polyester-glass-fabric laminates to themselves, none had any outstanding resistance to thermal softening when tested upon reaching 250° F.

4. The shear strength of lap-type specimens of aluminum or plastic laminates bonded with the various adhesives in this study was considerably less affected by stressing at -70° F than by stressing at 250° F or higher.

Forest Products Laboratory
Madison, Wisc., January 26, 1951

4. The shear strength of lap-type specimens of alumimum or plastic laminates bonded with the various adhesives in this study was considerably less affected by stressing at -70° F than by stressing at 250° F or higher.

Forest Products Laboratory
Madison, Wisc., January 26, 1951

APPENDIX

CODE SHEET IDENTIFYING ADHESIVES USED IN INVESTIGATION

The trade names or suppliers of the adhesives used in the present investigation are as follows:

A Cycleweld C-3
B Cycleweld C-3 with Durez 13297
C Cycleweld 55-10
D Plycozite 117C
E Redux E
F Bakelite XC16320
G Pliobond M-20
H GE 12513
I adhesive supplied by B. B. Chemical Co.
J Araldite Type 1
K Plastilock 500
L adhesive L-1372 of the Specialty Resins Co.
M adhesive L-1358F of the Specialty Resins Co.
N Plastilock 601 Tape
O Amberlite PR-115
P Selectron 5003, with 1.6 percent benzoyl peroxide and 3 percent promoter RT-1
Q Cycleweld C-11
R Selectron 5003, with 0.8 percent benzoyl peroxide
S Resin X-2

TABLE 1

CONDITIONS USED IN BONDING LAP SHEAR TEST SPECIMENS

OF CLAD 24S-T3 ALUMINUM ALLOY

Adhesive	Method of applying adhesive	Open assembly at room conditions after last coat (hr)	Precuring conditions after open assembly		Bonding pressure (psi)	Curing conditions	
			Temperature (°F)	Time (min)		Temperature (°F)	Time (min)
A	Spray	40 to 64	----	--	250	350	25
B	(a)	24	----	--	250	225	60
C	Spray	48 to 68	200	45	150	300	30
D	Spray	68	[b]200	45	250	300	15
E	(c)	40 to 44	----	--	250	300	10
F	Web	20	----	--	250	300	25
G	Brush	16 to 24	200	10	250	350	25
H	Brush	8	320	20	200	400	192
I	Brush	Overnight	350	12	250	350	10
J	[d]Sprinkled	[e]None	----	--	5	360	120
K	Brush	1	325	15	250	325	30
L	Brush	None	----	--	5	75	Overnight
M	Brush	None	----	--	5	75	Overnight
N	One layer of tape	None	----	--	250	400	15

[a]Primary adhesive was applied by spray and secondary adhesive, by brush. Primary adhesive was assembled and cured as for adhesive A, except that no pressure was used during curing in open assembly.

[b]Followed by precuring in closed assembly at 300° F for 9 min.

[c]One component was applied by brush and the other, by dusting while first component was wet.

[d]On metal heated to 200° F.

[e]Metal only cooled to room temperature.

TABLE 2

CONDITIONS USED IN BONDING LAP SHEAR TEST SPECIMENS OF PLASTIC LAMINATES TO THEMSELVES AND TO CLAD 24S-T3 ALUMINUM ALLOY

Adhesive	Method of applying adhesive	Assembly conditions	Precuring conditions	Bonding pressure (psi)	Curing conditions	
					Temperature (°F)	Time (hr)
D	Spray	16 hr at 75° F plus 45 min at 200° F	9 min at 300° F	150	300	1/4
M	Brush	Immediate assembly	No precure	5	75	16
O	Brush	5 min open plus 10 min closed	No precure	150	75	16
P	Brush	Immediate assembly	No precure	150	75	16
Q	Brush	Immediate assembly	No precure	5	75	16
R	Brush	$3\frac{1}{2}$ hr open	No precure	150	250	1
S	Brush	5 min open plus 10 min closed	No precure	5	75	16

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TABLE 3

AVERAGE RESULTS OF TESTS OF LAP SHEAR SPECIMENS OF CLAD 24S-T3 ALUMINUM ALLOY BONDED TO ITSELF WITH SEVERAL COMMERCIAL ADHESIVES AND TESTED IMMEDIATELY AND AFTER AGING AT VARIOUS TEMPERATURES

[Each value is average of results of tests on six specimens except in test 3 for adhesives A to G, where each value is average of results of tests on two specimens. Where no values are given, no tests were made.]

Adhesive	Test 1: Specimens tested immediately after reaching (a)																	
	-70° F		80° F		140° F (b)		160° F		200° F (b)		250° F		350° F		450° F		600° F	
	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)
A	2813	100	1530	57	1165	22	1005	38	927	16	830	42	653	22	475	35	33	100
B	822	99	1359	0	1139	0	870	0	958	0	586	0	498	0	510	0	76	14
C	770	100	1781	18	1991	84	743	82	1140	39	342	45	139	42	63	35	26	32
D	830	100	2082	48	2187	26	894	12	1180	7	93	0	26	0	10	0	12	0
E	1732	3	1939	23	1629	39	1103	17	623	7	250	0	129	0	50	0	32	0
F	798	96	1182	0	640	92	464	0	375	86	162	0	65	7	39	10	10	17
G	1493	91	778	74	----	--	312	87	----	---	176	83	110	38	46	1	---	---
H	----	---	631	0	----	--	----	--	----	---	---	--	---	--	20	0	---	---
I	----	---	2480	67	1408	30	----	--	366	100	---	--	---	--	---	--	---	---
J	----	---	2297	4	2053	5	----	--	1923	57	---	--	---	--	---	--	---	---
K	----	---	1619	69	401	14	----	--	69	83	---	--	---	--	---	--	---	---
L	----	---	1353	88	259	98	----	--	90	98	---	--	---	--	---	--	---	---
M	----	---	1668	0	498	85	----	--	130	95	---	--	---	--	---	--	---	---
N	1633	94	1756	11	1079	9	1020	6	756	14	666	8	462	29	269	76	114	86

[a]Data here reported for adhesives C, D, E, and F tested immediately at temperatures of 80°, 160°, 250°, 350°, 450°, and 600° F were from tests conducted jointly by the NACA and under a project in cooperation with ANC-23 Panel on Aircraft Design Criteria.

[b]Tests at 140° and 200° F were made on a separate series of specimens made in the same way as the original series and in some cases with different samples of adhesives. Control tests made at 80° F on this second series gave results generally in good agreement with similar control tests in the original series. Exceptions were with adhesive C, in which the control values in the second series averaged 2604 lb/0.5 sq in. strength and 72-percent adhesion failure, and with adhesive M, in which the control values for the series tested at 140° and 200° F averaged 961 lb/sq in. strength and 25-percent adhesion failure.

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TABLE 3.- Concluded

AVERAGE RESULTS OF TESTS OF LAP SHEAR SPECIMENS OF CLAD 24S-T3 ALUMINUM ALLOY BONDED TO ITSELF

WITH SEVERAL COMMERCIAL ADHESIVES AND TESTED IMMEDIATELY AND AFTER

AGING AT VARIOUS TEMPERATURES - Concluded

Adhesive	Test 2: Specimens tested at temperatures shown below after 192 hr at such temperatures								Test 3: Specimens tested at 80° F after exposure for 192 hr at -								Test 4: Specimens exposed to three repeating cycles of 3 hr at -70° F followed by 21 hr at 400° F and then tested at -					
	160° F		250° F		350° F		450° F		160° F		250° F		350° F		450° F		-70° F		80° F		400° F	
	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Adhesion failure (percent)
A	1014	29	682	16	557	97	0	100	1236	5	783	85	704	100	0	100	56	100	104	100	56	96
B	----	---	888	1	722	83	6	100	----	--	1185	0	1158	98	0	100	675	100	838	93	558	98
C	918	100	736	96	304	[illegible]	104	58	1942	90	2310	95	2495	50	345	30	794	43	1270	80	128	58
D	1244	4	919	10	320	9	46	0	2155	40	2375	90	1255	10	115	0	376	0	266	0	100	4
E	1574	10	929	0	287	0	161	0	1535	28	1515	90	1285	52	608	8	916	9	690	6	172	0
F	830	12	763	24	282	18	39	16	1802	0	2215	10	1720	10	413	10	959	15	1017	3	206	4
G	----	---	----	--	314	0	439	17	----	--	----	--	1635	0	605	0	---	---	1792	0	256	8
H	----	---	----	--	----	--	100	23	----	--	----	--	----	---	592	0	---	---	----	---	---	--
I	----	---	----	--	----	--	62	15	----	--	----	--	----	---	c0	---	---	---	----	---	---	--
J	----	---	----	--	----	--	105	7	----	--	----	--	----	---	c0	---	---	---	----	---	---	--
K	----	---	----	--	----	--	66	9	----	--	----	--	----	---	c0	---	---	---	----	---	---	--
L	----	---	----	--	----	--	75	50	----	--	----	--	----	---	c0	---	---	---	----	---	---	--
M	----	---	----	--	----	--	73	28	----	--	----	--	----	---	c0	---	---	---	----	---	---	--
N	1207	5	1006	24	742	93	486	89	1896	10	1679	48	1479	94	606	89	---	---	----	---	---	--

[c]Joint specimens fell apart while being placed in test grips.

TABLE 4

MINIMUM AND MAXIMUM STRENGTH VALUES OF TESTS OF LAP SHEAR SPECIMENS OF
CLAD 24S-T3 ALUMINUM ALLOY BONDED TO ITSELF WITH SEVERAL
COMMERCIAL ADHESIVES AND TESTED IMMEDIATELY AND
AFTER AGING AT VARIOUS TEMPERATURES

Adhesive	Minimum and maximum strength values (lb/0.5 sq in.)																			
	Test 1: Specimens tested immediately after reaching -									Test 2: Specimens tested at temperatures shown below after 192 hr at such temperatures				Test 3: Specimens tested at 80° F after exposure for 192 hr at -				Test 4: Specimens exposed to three repeating cycles of 3 hr at -70° F followed by 21 hr at 400° F and then tested at -		
	-70° F	80° F	140° F	160° F	200° F	250° F	350° F	450° F	600° F	160° F	250° F	350° F	450° F	160° F	250° F	350° F	450° F	-70° F	80° F	400° F
A	2570-3060	1332-1760	1130-1225	912-1140	900- 950	760-945	607-687	445-497	27- 44	870-1087	610- 735	460-635	------	1165-1307	775- 790	697- 710	------	0- 145	50- 132	0- 85
B	650-1180	1210-1540	980-1295	815-1010	780-1025	420-690	412-662	469-540	37-114	------	700- 925	672-780	0- 25	------	1170-1200	1110-1205	------	560-1000	765- 965	530-580
C	700- 830	1680-1960	1815-2060	622- 927	1115-1205	275-427	90-170	44- 79	16- 31	625-1145	592- 875	350-435	0-160	1770-2115	2300-2320	2350-2640	285-405	510-1100	750-1650	67-152
D	740-1120	1800-2350	2100-2280	435-1470	969-1640	60-127	20- 36	8- 12	2- 17	707-1695	470-1385	147-510	27- 67	2060-2250	2330-2420	350-2160	105-125	175- 885	195- 482	55-140
E	1530-1870	1850-2010	1595-1705	1010-1160	585- 645	207-295	100-155	38- 59	24- 38	1390-1725	872-1002	250-335	115-207	1525-1545	1480-1552	1230-1340	567-650	385-1245	480- 890	145-205
F	700- 920	982-1335	582- 695	285- 550	350- 400	135-185	56- 76	19- 54	7- 12	745-1145	585- 960	212-440	12- 75	1575-2030	2205-2225	1540-1900	345-482	845-1140	832-1512	75-185
G	1150-1780	607- 957	------	107- 465	------	50-265	62-145	35- 55	------	------	------	255-360	355-505	------	------	255- 360	355-505	------	1640-1905	215-292
H	------	550- 712	------	------	------	------	------	10- 30	------	------	------	------	60-125	------	------	------	160-825	------	------	------
I	------	2206-2578	1275-1485	------	200- 430	------	------	------	------	------	------	------	25- 85	------	------	------	------	------	------	------
J	------	1575-2460	1645-2590	------	1770-2170	------	------	------	------	------	------	------	90-130	------	------	------	------	------	------	------
K	------	1465-1792	320- 470	------	60- 85	------	------	------	------	------	------	------	0-120	------	------	------	------	------	------	------
L	------	1166-1448	185- 305	------	80- 100	------	------	------	------	------	------	------	0-117	------	------	------	------	------	------	------
M	------	1535-1817	295- 690	------	85- 180	------	------	------	------	------	------	------	0-160	------	------	------	------	------	------	------
N	1010-2365	1585-1925	925-1320	955-1120	675- 895	560-735	310-580	235-305	70-155	1005-1400	900-1085	635-800	452-532	1730-2045	1440-1880	1380-1570	575-640	------	------	------

TABLE 5

AVERAGE RESULTS OF TESTS OF LAP SHEAR SPECIMENS OF PLASTIC LAMINATES BONDED TO THEMSELVES AND OF PLASTIC LAMINATES BONDED TO CLAD ALUMINUM WITH SEVERAL COMMERCIAL ADHESIVES AND TESTED AT -70°, 80°, AND 250° F IMMEDIATELY UPON REACHING EQUILIBRIUM AT THESE TEMPERATURES

[Each value is average of results of tests on eight specimens. Failure not recorded was usually cohesion failure in adhesive. Where no values are given, no tests were made.]

Adhesive	Type A[1]: Grade L cotton-fabric-phenolic laminate tested at -									Type B[2]: Glass-fabric (181-114)-polyester laminate tested at -								
	-70° F			80° F			250° F			-70° F			80° F			250° F		
	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent) (3)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent) (3)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)
D	685	100	5	640	100	0	414	0	66	862	18	82	1100	13	79	303	0	41
N	691	100	0	648	100	0	188	0	29	815	25	75	684	6	94	287	0	37
O	---	---	-	322	0	100	---	-	--	---	--	---	419	2	97	---	--	---
P	---	---	-	327	0	100	---	-	--	421	0	100	466	0	100	218	0	100
Q	657	100	0	640	100	0	118	0	5	899	49	51	642	8	92	119	1	22
R	---	---	-	421	0	100	---	-	--	761	32	68	790	20	80	333	13	22
S	700	100	0	639	75	25	276	0	49	546	5	82	420	5	92	143	5	88

[1]Type A specimen consisted of two 1- by $1\frac{7}{8}$-in. pieces of 1/8-in.-thick grade L cotton-fabric-phenolic laminate lap-jointed for 0.5 in.
[2]Type B specimen was the same as type A except that the laminate used was 1/8-in.-thick glass-fabric (181-114)-polyester laminate.
[3]Where 100-percent plastic failures are shown, failures were mainly in tension.

TABLE 5.- Concluded

AVERAGE RESULTS OF TESTS OF LAP SHEAR SPECIMENS OF PLASTIC LAMINATES BONDED TO THEMSELVES AND OF PLASTIC LAMINATES BONDED TO CLAD ALUMINUM WITH SEVERAL COMMERCIAL ADHESIVES AND TESTED AT -70°, 80°, AND 250° F IMMEDIATELY UPON REACHING EQUILIBRIUM AT THESE TEMPERATURES - Concluded

Adhesive	Type C[4]: 24S-T3 clad aluminum to grade L cotton-fabric-phenolic laminate tested at									Type D[5]: 24S-T3 clad aluminum to glass-fabric (181-114)-polyester laminate tested at								
	-70° F			80° F			250° F			-70° F			80° F			250° F		
	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)	Average shear strength (lb/0.5 sq in.)	Plastic failure (percent)	Adhesion failure (percent)
D	248	1	94	415	5	91	151	0	100	335	25	63	402	53	45	231	5	80
M	140	0	89	285	3	89	91	0	82	202	1	89	352	4	85	49	0	34
O	---	-	---	---	-	---	---	-	---	---	--	--	---	--	--	---	-	--
P	---	-	---	---	-	---	---	-	---	---	--	--	---	--	--	---	-	--
Q	186	0	100	498	0	100	41	0	96	231	5	87	550	10	90	33	0	86
R	---	-	---	---	-	---	---	-	---	---	--	--	---	--	--	---	-	--
S	---	-	---	---	-	---	---	-	---	---	--	--	---	--	--	---	-	--

[4]Type C specimen consisted of two 1- by $1\frac{7}{8}$- in. pieces of 0.032-in. 24S-T3 clad aluminum alloy overlapped for 0.5 in. with a shim of 0.032-in. grade L cotton-fabric-phenolic laminate in the lap area.

[5]Type D specimen was the same as type C specimens except that the shim in the lap area was of 0.032-in. glass-fabric (181-114)-polyester laminate.

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TABLE 6

MINIMUM AND MAXIMUM STRENGTH VALUES OF TESTS OF LAP SHEAR SPECIMENS OF PLASTIC LAMINATES BONDED TO THEMSELVES AND OF PLASTIC LAMINATES BONDED TO CLAD ALUMINUM WITH SEVERAL COMMERCIAL ADHESIVES AND TESTED AT -70°, 80°, AND 250° F IMMEDIATELY UPON REACHING EQUILIBRIUM AT THESE TEMPERATURES

Adhesive	Minimum and maximum strength values (lb/0.5 sq in.)											
	Type A[1]: Grade L cotton-fabric-phenolic laminate tested at -			Type B[2]: Glass-fabric (181-114)-polyester laminate tested at -			Type C[3]: 24S-T3 clad aluminum to grade L cotton-fabric-phenolic laminate tested at -			Type D[4]: 24S-T3 clad aluminum to glass-fabric (181-114)-polyester laminate tested at -		
	-70° F	80° F	250° F	-70° F	80° F	250° F	-70° F	80° F	250° F	-70° F	80° F	250° F
D	632-740	615-655	250-485	800-912	935-1320	175-460	235-267	360-472	135-185	287-385	370-442	185-335
M	672-727	615-690	170-205	487-957	385- 870	212-330	120-155	245-330	75-100	155-240	310-425	45- 57
O	-------	280-360	-------	-------	365- 455	-------	-------	-------	-------	-------	-------	-------
P	-------	280-385	-------	365-445	400- 525	200-250	-------	-------	-------	-------	-------	-------
Q	622-708	595-670	95-130	825-986	600- 712	80-150	165-225	378-702	25- 65	172-274	444-704	20- 45
R	-------	365-490	-------	640-852	740- 835	265-420	-------	-------	-------	-------	-------	-------
S	645-748	600-680	195-365	405-660	315- 485	120-160	-------	-------	-------	-------	-------	-------

[1]Type A specimen consisted of two 1- by $1\frac{7}{8}$- in. pieces of 1/8-in.-thick grade L cotton-fabric-phenolic laminate lap-jointed for 0.5 in.

[2]Type B specimen was the same as type A except that the laminate used was 1/8-in.-thick glass-fabric (181-114)-polyester laminate.

[3]Type C specimen consisted of two 1- by $1\frac{7}{8}$ -in. pieces of 0.032-in. 24S-T3 clad aluminum alloy overlapped for 0.5 in. with a shim of 0.032-in. grade L cotton-fabric-phenolic laminate in the lap area.

[4]Type D specimen was the same as type C specimens except that the shim in the lap area was of 0.032-in. glass-fabric (181-114)-polyester laminate.

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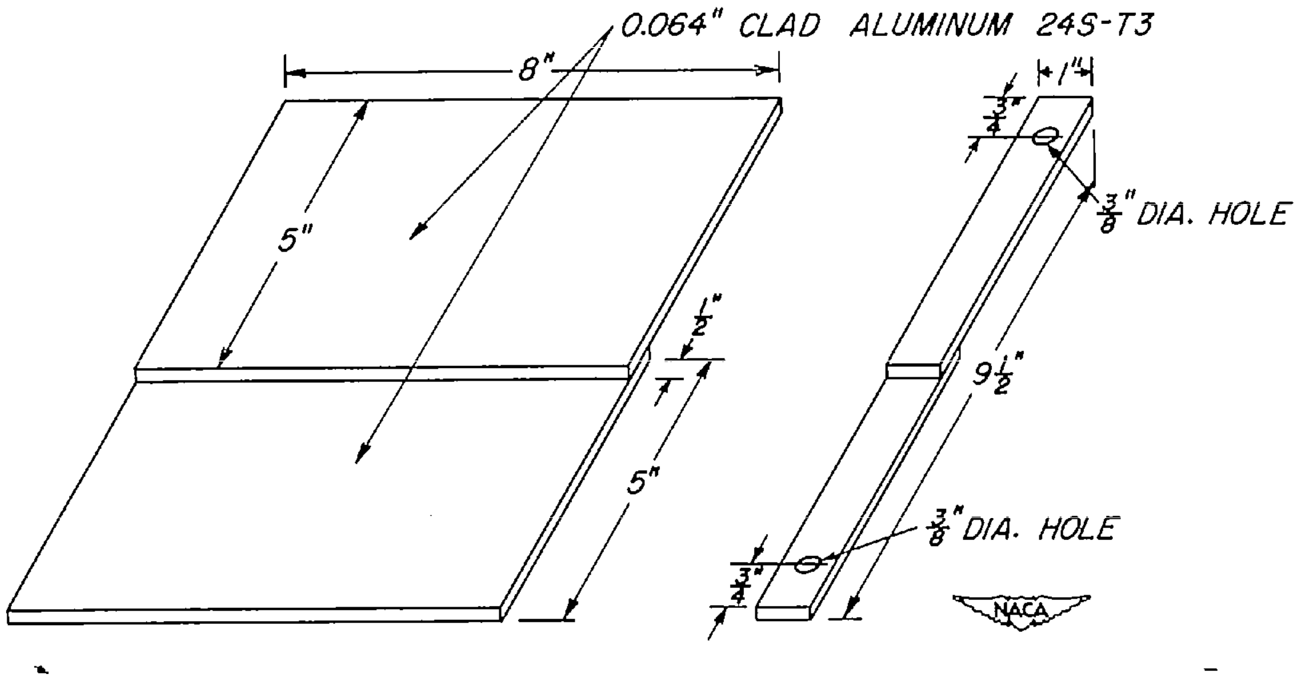

Figure 1.- Test panel and test specimen for evaluation of adhesives in joints of clad aluminum to itself.

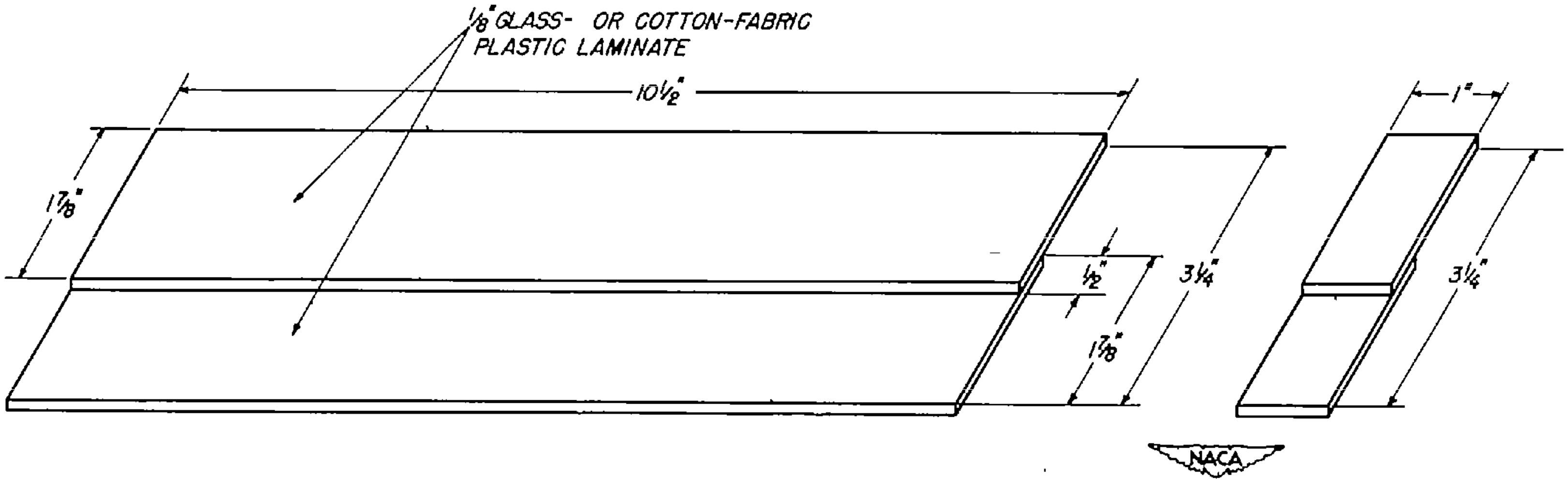

Figure 2.- Test panel and test specimen for evaluation of adhesives in joints of plastic laminate to itself.

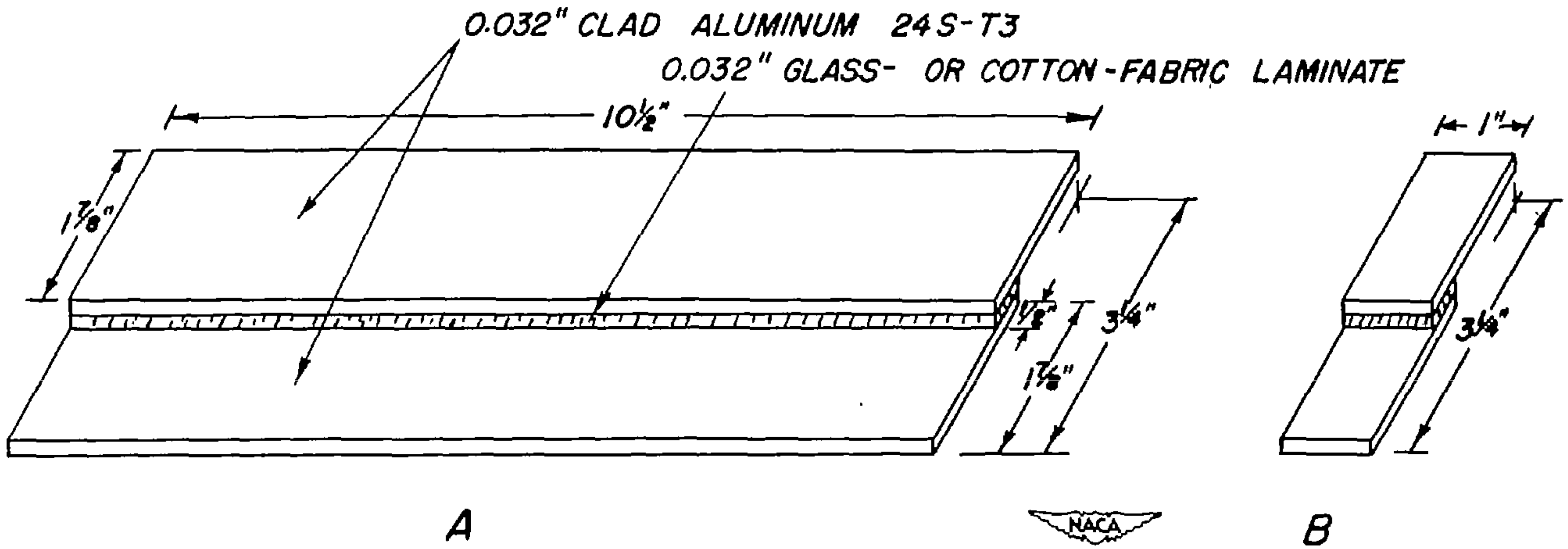

Figure 3.- Test panel and test specimen for evaluation of adhesives in joints of plastic laminates to clad aluminum.

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