GENERAL SURVEY OF DATA ON THE
RELIABILITY OF METAL-BONDING
ADHESIVE PROCESSES

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UNITED STATES DEPARTMENT OF AGRICULTURE
FOREST SERVICE
In Cooperation with the University of Wisconsin
GENERAL SURVEY OF DATA ON THE RELIABILITY OF
METAL-BONDING ADHESIVE PROCESSES

By

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Summary

A survey was made among a number of representative aircraft fabricators and adhesive manufacturers to obtain information on the reliability of adhesive-bonding processes for metals based on the variability of lap-joint strength data from tests for adhesive acceptance, laboratory control, adhesive storage, and production control. Practically all tests were on standard lap-joint specimens at room temperatures. The range of the coefficient of variation for these metal-bonding tests was generally from 6.4 to 16.2 percent with most of the adhesive storage and production control data in the range above 10 percent. From the data obtained, there were no indications that the coefficient of variation for the strength data was greater for one type of adhesive than for another.

Introduction

During the past 10 years, there has been a great increase in the use of adhesives for structurally bonding metal components of aircraft. The quality of any adhesive bond will depend both on the adhesive and on the way in which it is used. When adhesives were first used for structural metal bonding, bond strengths were often erratic. With improved adhesive formulations

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1This report is one of a series (ANC-23, Item 56-3) prepared and distributed by the Forest Products Laboratory under U.S. Navy, Bureau of Aeronautics Nos. NAer 01628 and NAer 01593 and U.S. Air Force Nos. DO 33(616)33-20 and AF 18(600)102. Results here reported are preliminary and may be revised as additional data become available.

2Trade names are used in this report at the request of the ANC-23 Panel, and their use implies no endorsement of the products named by the U.S. Forest Products Laboratory or any other government agency.

3Maintained at Madison, Wis., in cooperation with the University of Wisconsin.
however, and more knowledge of the preparation of the metal surfaces and the proper control of bonding conditions, much better uniformity has been obtained in this bonding. There is, however, still variability in the strength of bonds made with the metal-bonding adhesives when used under generally well-controlled laboratory and under plant conditions that are believed to be rather uniformly controlled. The magnitudes of such variability in joint quality are not well established. These variations in joint strengths may be caused by minor variations in the metal, such as the uniformity of thickness, flatness of the sheet, smoothness of the cut edge, and variables in the preparation of the metal surface for bonding; in the adhesives, such as variations in composition and age, film thickness, and method of application and atmospheric condition under which the adhesive is applied and dried; in the time, temperature, and pressure conditions used for precuring and curing the adhesive bond; and in the conditioning, cutting, and testing of the joints.

To give designers of new aircraft components a more reliable basis for determining where adhesive bonding can be safely used in aircraft construction, it is desirable to develop more complete information on the magnitudes of variation in joint quality with different adhesive-bonding processes in actual use. The best way would be to design a series of experiments to study such variability under controlled conditions so that those variables contributing to undesirable variation in joint quality could be adequately identified. A study of this type however, would be complicated and involve considerable work under closely controlled conditions. Therefore the present partial survey was made to obtain preliminary information already available from various sources on the variability of joint strengths when these metal-bonding adhesives are used under repetitive conditions. The results are intended to give an overall indication of the strength variables of the metal-bonding processes as used in typical plant and laboratory conditions, rather than any comparison between individual adhesive processes.

Procedure

A survey was made among aircraft fabricators and adhesive manufacturers, principally in the western and southwestern areas to obtain samples of available lap-joint test data on the metal bonding processes. The data were selected to represent, as nearly as practical, the results of tests made under a single set of bonding conditions, but included, in most instances, the results obtained by using the same set of conditions for several months. The test data were obtained in most instances for standard 1/2-inch lap joints of 0.064-inch thick aluminum tested at room temperatures. There were, however, some fabricators that used other types of specimens, such as those with 1-inch laps or of magnesium alloy. Others had different conditions for their testing. Some sets of these other data were also obtained for comparison purposes.
The following organizations cooperated by supplying data that are incorporated in this survey.

Boeing Airplane Company, Seattle 14, Wash.
Chance Vought Aircraft, Inc., Dallas, Tex.
Convair Division, General Dynamics Corporation, Ft. Worth, Tex.
Convair Division, General Dynamics Corporation, San Diego 12, Calif.
Douglas Aircraft Company, Inc., Santa Monica, Calif.
Hexcel Products, Inc., Oakland, Calif.
Lockheed Aircraft Corporation, California Division, Burbank, Calif.
The Glenn L. Martin Company, Baltimore 3, Md.
McDonnell Aircraft Corporation, St. Louis 3, Mo.
Narmco Manufacturing Co., 930 Grape Street, San Diego, Calif.
Narmco Resins & Coating Co., Costa Mesa, Calif.
North American Aviation, Inc., International Airport, Los Angeles 45, Calif.
Northrop Aircraft, Inc., Northrop Field, Hawthorne, Calif.
Shell Development Co., Emeryville, Calif.
Sikorsky Aircraft Division, United Aircraft Corporation, Bridgeport, Conn.

The data obtained included summary graphs, test reports, and original workbook records, and represented adhesive acceptance tests, storage-life tests, and production and laboratory-control tests. The tests of adhesive acceptance, laboratory control, and storage life were usually made by laboratory personnel, while the production-control data were from control panels fabricated along with the production parts. The storage life tests, in most instances, were periodic tests made at 96-hour intervals during the use of the adhesive for production bonding.

These data were first summarized and tabulated on a survey summary sheet of the type shown in figure 1. On this sheet were entered the pertinent information on the source of data, type of adhesive, type of joint, bonding and test conditions, as well as the summary of the test data. Computations of the mean and coefficient of variation values as illustrated in figure 1 were then made at the Forest Products Laboratory for each set of data.

As there were numerous sets of data representing 100 to 2,000 individual tests, the data in these groups were summarized by indicating, for each set of data, the frequency distribution among various class intervals. Class intervals of either 100 or 200 pounds per square inch were used, the smaller intervals generally being used for those data with the smaller overall range.

The mean, standard deviation, and coefficient of variation values were determined from the class interval frequency distribution for each set of data by using the following statistical methods.

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1 The methods used in these analyses were taken from "Experimental Statistics for Engineers," by R. J. Hader, North Carolina State College, 1951.
The class interval data were arranged in increasing order as shown in figure 1. The midpoint values for each class interval were assigned, and all results in a given class interval were treated as though they had the same value, that of the interval midpoint. These midpoint values were coded to simplify the computations by arbitrarily assigning a coded value of 0 to the interval midpoint, \( A \), near the median value for the test data, and increasing or decreasing unit values, \( x_i \), to code the class interval midpoints on each side of this arbitrarily assigned origin as shown in the sample sheet. The mean value, \( \bar{X} \), for the group was then calculated as:

\[
\bar{X} = A + m \frac{\sum f_i x_i}{n}
\]

in which \( A \) is the value of the midpoint at an arbitrary origin, \( m \) the width of the class interval, \( f_i \) the frequency of observation in the \( i^{th} \) class interval, \( x_i \) the coded midpoint for the \( i^{th} \) class interval, and \( n \) the total number of observations.

For example, the calculation from the sample sheet would be:

\[
\bar{X} = 4,300 + \frac{200(-171)}{405} = 4,215 \text{ pounds per square inch}
\]

The standard deviation, \( S \), of the test values was next calculated as:

\[
S = m \sqrt{\frac{\sum f_i x_i^2 - (\sum f_i x_i)^2}{n - 1}}
\]

with the term \( \frac{(\sum f_i x_i)^2}{n} \) of this equation generally being small compared to the preceding term when the coded midpoint origin was properly selected to include the median value. For example, the calculation from the sample sheet would be:

\[
S = 200 \sqrt{2,735 - \frac{(-171)^2}{405}}
\]

\[
S = 513 \text{ pounds per square inch}
\]

The coefficient of variation in percent for the individual test values was then calculated as:

\[
C. V. = \frac{100 S}{\bar{X}}
\]
In some instances, only the average data for groups of tests were available. The average data were arranged into class intervals and calculated the same as individual test values, except that the standard deviation value obtained was for the group means. The standard deviation for the individual values was then estimated by multiplying the standard deviation for the group means by the square root of the number of values included in each group.

In two instances, the data were obtained in the form of biweekly mean and standard deviation values. The exact number represented by each biweekly value was not known, but the production was considered constant during the 1-year period. Therefore, the mean value, $\bar{X}$, for the entire set of data was obtained by averaging the biweekly mean values, and the mean value for the standard deviation, $S$, was obtained by averaging the biweekly variance values, $S^2$, and taking the square root of this average.

Data were obtained on most of the currently used types of metal-bonding adhesive processes. These adhesives have been classified in this work as:

- Neoprene-phenolic-nylon resin on nylon fabric
- Epoxy resin
- Nitrile-rubber-phenolic resin on unsupported tape
- Heat-resistant modified epoxy resin on glass fabric
- Polyvinyl-phenolic resin on glass fabric
- Polyvinyl powder and liquid phenolic resin

The trade names of the adhesives are included in the summary of results given in table 1. However, the performance of the different adhesives cannot be directly compared on the basis of the limited data presented in this report, since bonding was done by various laboratories under different conditions, for somewhat different purposes, and over a considerable period of time. Nor was there any assurance that the bonding was always done under the optimum conditions recommended by the adhesive manufacturer. Each fabricator, however, usually used a single set of standard conditions that were determined from earlier investigations to develop the best single set of conditions for his fabrication operation.

Results

The summary of the adhesive-bonded lap-joint data obtained in the survey among aircraft fabricators and adhesive manufacturers is given in table 1. These data are classified as to the type of adhesive, the source and type of test data (adhesive acceptance, storage, production control, and other conditions) and the type of test specimen and testing temperature used. Means, standard deviations, and coefficients of variations for shear-strength values are given for each set of data.
For this work, the coefficient of variation values for shear strength are of primary interest. These coefficients of variation values, for the data surveyed, range from 6.4 to 29.0 percent for aluminum lap-joints tested at room temperatures. Many of the higher coefficient values, however, were estimates based on groups of test data on panels for which individual test values were not available, and only averages were used in computations, and the estimates are not believed as accurate as estimates obtained from data for individual tests. Excluding these several high values based on averaged test data and also one other unusually high value of 20.7, the range of coefficient of variation was from 6.4 to 16.2 percent, with most of the adhesive storage and production control data showing coefficients of variation from 10.0 to 16.2 percent. Assuming normal distribution of the data, a coefficient of variation of 10 percent, for example, means that about 68 percent of the values are within plus or minus 10 percent of the mean value, 95 percent within 20 percent of the mean, and 99 percent within 30 percent of the mean.

The data on adhesive lap-joint tests at elevated temperatures, while very limited, indicated that these test values would also normally fall between 10.0 and 15.0 percent for the coefficient of variation. There were no indications from the data obtained that any one of the adhesive types differed greatly from the others in the degrees of variation in the adhesive-strength tests. There was not sufficient data that could be compared directly to indicate whether there were any important differences obtained in the uniformity of strength results by different plants using the same adhesive system.

Comparable data were not available on the coefficients of variation for tests made on welded or riveted joints. It is believed desirable, however, to continue investigations to improve the reliability of adhesive bonding of metals so that the results of all types of tests would show coefficients of variation at least as low as 6.0 to 10.0 percent, the lower range obtained in present analysis of these adhesive bond test data.
<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Type of plastic</th>
<th>Source of data</th>
<th>Type of data</th>
<th>Type of specimen</th>
<th>Number of specimens</th>
<th>Test type</th>
<th>Period of test</th>
<th>Lap-joint strength values</th>
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<td>Mechant</td>
<td>EPX</td>
<td>Plant A</td>
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<td>1/8&quot; lap, clad aluminum</td>
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<td>EPX</td>
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<td>2.663</td>
</tr>
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<td>Production control</td>
<td>1/8&quot; lap, clad aluminum</td>
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<td>70 to 80</td>
<td>490/490</td>
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</tr>
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<td>70 to 80</td>
<td>490/490</td>
<td>2.663</td>
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<td>Plant N</td>
<td>Production control</td>
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<td>70 to 80</td>
<td>490/490</td>
<td>2.663</td>
</tr>
<tr>
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<td>EPX</td>
<td>Plant O</td>
<td>Production control</td>
<td>1/8&quot; lap, clad aluminum</td>
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<td>490/490</td>
<td>2.663</td>
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<td>Production control</td>
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<td>Production control</td>
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</tr>
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<td>Plant S</td>
<td>Production control</td>
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<td>2.663</td>
</tr>
<tr>
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<td>Plant T</td>
<td>Production control</td>
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<td>70 to 80</td>
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</tr>
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<td>Plant U</td>
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<td>1/8&quot; lap, clad aluminum</td>
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<td>Plant V</td>
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<td>Plant Y</td>
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<td>15,200</td>
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<td>2.663</td>
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</table>
Figure 1.--Sample Survey Summary Sheet -- Reliability of Metal-Bonding Adhesive Processes

ADHESIVE PROCESS: Metlbond 4021
Bonded at Plant E
Date 2/20/54 to 1/6/56

SPECIMEN: 1/2 inch lap-joint of 0.064 inch clad 7075-T6 aluminum

BONDING PROCEDURE:
Material preparation
Washed in solvent thinner, 10-12 minutes at 145° F.
in a 3.5 - 12.5 - 30 sodium dichromate-sulfuric acid-
water, etch, rinse, and force-dry.

Adhesive spread: Method Brush coat No. of coats 1
Drying between coats
Precure: Air dry 4-16 hrs. Precure 15 min. at 180 ° F.
Cure: 40 min. at 300 ° F. pressure 100 psi.

Remarks:

TESTS: Test temperature -- 70-80° F. Testing speed -- 1,200-1,400 lbs. per min.
No. of panels 81
Specimens in each 5
Purpose of test Acceptance and storage data
Other sources of variation Date of bonding, and equipment variables.

SUMMARIZED RESULTS: \( \bar{X} = 4,215 \text{ psi} \quad S = 513 \text{ psi} \quad C. V. = 12.1 \text{ percent} \)

<table>
<thead>
<tr>
<th>Class interval</th>
<th>Midpoint ( X_i )</th>
<th>Coded ( f )</th>
<th>( f_{xi} )</th>
<th>( f_{xi}^2 )</th>
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<tr>
<td>3,000-3,199</td>
<td>3,100</td>
<td>-6</td>
<td>4</td>
<td>-24</td>
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<td>-5</td>
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<td>-80</td>
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<tr>
<td>3,400-3,599</td>
<td>3,500</td>
<td>-4</td>
<td>39</td>
<td>-156</td>
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<td>-3</td>
<td>46</td>
<td>-138</td>
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<td>-2</td>
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<td>-1</td>
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<td>-47</td>
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<tr>
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<td>56</td>
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<td>59</td>
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<td>49</td>
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<tr>
<td>Totals</td>
<td></td>
<td></td>
<td>405</td>
<td>-171</td>
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