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ROYAL AIRCRAFT ESTABLISHMENT

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TECHNICAL NOTE No: CHEM 1168

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COMPARATIVE TESTS ON REDUX AND ARALDITE METAL ADHESIVES

by

J.H.LEIGH and Mrs. J. SAVAGE

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[redacted]

U.D.C. No. 668.395

Technical Note Chem. 1168

March, 1952

ROYAL AIRCRAFT ESTABLISHMENT, FARNBOROUGH

Comparative Tests on Redux and Araldite Metal Adhesives

by

J.H. Leigh

and

(Mrs) J. Savage

RAE Ref: Chem.2004/3 and 2007/9/64

SUMMARY

The tests were carried out to evaluate Redux K6 and Araldite Type 1 as adhesives for aluminium alloys under the various conditions likely to be encountered during the life of bonded structures. Lap joint test specimens were made and supplied by Percival Aircraft Ltd., who also shared the testing with Aluminium Laboratories Ltd., and Chemistry Dept., R.A.E. The following results are given:- shear strength at -60°, 20° and 100°C, effect of double curing, effect of gauge on shear strength, effect of ageing at room temperature and 70°C, corrosion, effect of immersion in various aircraft fluids, effect of cyclic temperature and humidity conditions and fatigue.

LIST OF CONTENTS

		Page
1	Introduction	3
2	Description of materials	3
3	Description of test specimens	3
4	Specimen manufacture	4
5	Description of tests	5
6	Summary of results	8
7	Discussion of results	12
8	Conolusions	13
	References	14
	Advance Distribution	14

LIST OF TABLES

	Table
Summary of Results	I
Control Tensile Results	II

LIST OF ILLUSTRATIONS

	Figure
Fatigue curves - Araldite	1
Fatigue curves - Redux	2

1 Introduction

The work described in this report was started because of the encouraging results obtained on small scale preliminary tests on samples of Araldite adhesive obtained from Ciba Ltd., of Basle. Considerable interest was shown in the material by aircraft manufacturers and the extent of its possible application was increased with its production in this country by Aero Research Ltd. In view of the fact that a full scale evaluation was required it was decided that the information obtained should be comparative with Redux liquid K.6, this latter adhesive was a new development of Redux liquid E on which most of the previous work had been done.

The tests have been confined to simple lap joints. Further work is proceeding to extend the evaluation of the materials when used in both simple and complex structures.

2 Description of the materials

Adhesive (a) Redux liquid K.6 phenolic resin and 30 - 50 mesh polyvinyl formal powder, supplied by Aero Research Ltd.

(b) Araldite Type 1 epoxide resin - natural - in rod form, supplied by Aero Research Ltd.

Metal Aluminium coated aluminium alloy of thickness 20 SWG to Specification D.T.D.610B.

3 Description of the test specimens

All the test specimens were made and supplied by Percival Aircraft, Ltd., under Contract No. 6/Stores/4515/CB 43/C.

(i) Dimensions

The standard shear type of test specimen was used. This consisted of 20 SWG metal strips $4\frac{1}{2}$" long and 1" wide bonded together to give a $\frac{1}{2}$" overlap. Special specimens were supplied for the fatigue tests, the cyclic exposure tests and the effect of strip thickness tests. These specimens are described in the appropriate sections of this report.

(ii) Metal pre-treatment

A preliminary investigation was made by Percival Aircraft Ltd., to determine the most suitable pre-treatment for the metal prior to bonding, the types of surface treatment being:-

(a) Chrome-sulphuric and pickling to D.T.D.915A

(b) Chromic acid anodising to D.T.D.910

(c) Sulphuric acid anodising to D.T.D.910

For all types, the metal was precleaned by trichlorethylene degreasing.

Following analysis of the results, it was decided that chromic acid anodising should be used for all test specimen material. Two types of surface treatment were used for the fatigue test specimen, chromic acid anodising and D.T.D.915A pickling.

4 Specimen manufacture

(i) The test specimens were cut from 6' x 3' and 6' x 4' sheets parallel to the 6' dimensions. The sheet was guillotined to 1" wide strips, each strip being numbered as it was cut and these were then cut into $4\frac{1}{2}$" lengths and again numbered and lettered. Thus each half of a joint bore a corresponding number and consecutive letter which indicated its location within the parent sheet.

In addition to the strips cut for the joint test specimens, 6 control tensile specimens were cut at random from the 6' x 1" strips in the form of 9" x 1" blanks, each being numbered and lettered using the same coding system. The results of these control tensile tests are given in Table 2.

(ii) Redux bonding technique

Since the curing temperature and times for Araldite bonding corresponded with the artificial ageing treatment required to convert D.T.D.610B to D.T.D.546, it was decided that all the material used for Redux bonding should be given this heat treatment. This ensured comparable material properties for each type of joint. The specification mechanical properties are shown in Table 2 with the tensile test results.

The metal test strips were marked off to give the $\frac{1}{2}$" overlap and a thin film of liquid resin K.6 was applied with a brush. This portion was dipped into the powder and the surplus shaken off. The opposite halves of the joint were mated and placed on a brass sheet. Twenty joints were loaded on the sheet at a time and the whole was placed in an electrically heated press with thermostatic and manual control, and subjected to a pressure of 200 lb per sq inch for 25 minutes at 143 ± 3°C.

At the end of the curing period, the temperature was decreased to 90°C by forced draught and the joints were removed and air cooled to room temperature.

(iii) Redux control tests

Three specimens were taken at random from each press loading of twenty and tested. The conditions for the release of the batch were that:-

(a) All three specimens had a shear strength of not less than 1500 lb. (Specification D.T.D.775),

(b) If one of the three specimens gave a failing load below 1500 lb then three further specimens were tested and providing that these three were satisfactory then the batch was released.

If any batch failed to satisfy these requirements it was discarded.

(iv) Araldite bonding technique

The $\frac{1}{2}$" overlap portion of each strip was heated to approximately 100°C on an electric hot plate and a film of Araldite was applied by rubbing the rod over the joint area. The two halves of the joint were mated and placed in a jig which located the specimen both lengthwise and laterally. Ten joints were placed in each jig and a weight was applied to give a nominal joint pressure of 6 lb per sq inch. Six jigs were used and these were all placed in a Wild-Barfield air-circulating oven, thermostatically controlled at 180 - 185°C, for three hours. An additional quarter of an hour was allowed for heating the jigs and the specimens. At the end of the curing period the jigs were removed and allowed to cool to room temperature.

(v) Araldite control tests

A procedure was adopted similar to that described for the Redux controls, except that six specimens were tested from each batch and a failure of more than one specimen to attain a minimum shear strength of 1500 lb resulted in the batch being discarded.

5 Description of tests

Ten specimen joints made with each adhesive were used for each test. This number was selected following the work described in Chemistry Note No.1043 on the statistical analysis of lap joint strength test results[1].

The following tests are described in this section:-

(i) Effect of temperature at -60°C and 100°C

(ii) Effect of metal sheet thickness 14, 16 and 24 SWG

(iii) Effect of double curing

(Work done by Percival Aircraft Ltd., and covered by their report No.578 - 1 Dec. 1950).

(iv) Effect of cyclic conditions of temperature and humidity

(v) Effect of fatigue after 20,000,000 cycles

(Work done by Aluminium Laboratories, Ltd., under contract 6/Gen/1006/CF.14B and covered by their report No, B-IR-186-50-27 - 15.12.50),

(vi) Effect of immersion in various aircraft fluids.

(vii) Effect of exposure under corrosive conditions

(viii) Effect of ageing at 20°C and 70°C

(Work done by Chemistry Department, R.A.E.)

The test procedures for the above were as follows:-

(i) -60°C The test specimen was surrounded by an asbestos lined muff packed with solid carbon dioxide and the ends were gripped in the jaws of the testing machine. The temperature was indicated by a toluene thermometer placed adjacent to the specimen. In order to ensure that the specimen was in equilibrium with the container temperature, it was allowed to remain in the apparatus for ten minutes before applying the load.

100°C The test specimen was surrounded by an asbestos lagged muff and hot air was injected until the specimen was in equilibrium with the container temperature. The temperature was indicated by means of a thermocouple fixed to the face of the specimen with Bostik Pressure Plastic.

Additional tests were done on Araldite bonded specimens to determine the effect of a longer period of exposure at this temperature, the specimens being subjected to 100°C for 30 minutes after attaining thermal equilibrium.

(ii) The effect of metal thickness on the shear strength of the specimen joints was determined by preparing the standard ½" overlap but with metal strips of the following thicknesses:- 14 SWG (0.080"), 16 SWG (0.064") and 24 SWG (0.022"). These specimens were tested at room temperature.

(iii) The effect of double curing or re-heating on the shear strength of previously made specimens was determined by repeating the appropriate curing cycle as described for the two adhesives. This test was included in the programme because re-heating is a condition which may be unavoidable in the production of a complex structure.

(iv) The effect of cyclic exposure tests on shear strength was determined, the conditions being:-

(a) exposure of specimen joints for 5, 10 and 20 cycles to ordinary air at room temperature, raised to 90°C and maintained for 3 hours, followed by cooling under a forced draught; a complete cycle took about 24 hours.

(b) Exposure of specimen joints for 10 and 20 cycles to moist air at 70°C and maintained for 3 hours, followed by natural cooling to room temperature; a complete cycle took about 24 hours. The test specimens were manufactured in the same manner as the standard specimens but were ¾" wide. These were ultimately machined to ½" width and ¼" holes were drilled 4" apart on the centre line.

For the hot/dry exposure cycles (a), the specimens were suspended in an oven and the temperature was raised to 90°C and maintained for 3 hours. The specimens were then removed from the oven and cooled by compressed air.

For the hot/wet exposure cycles (b), the specimens were suspended over water in a vessel placed in the oven, the vessel communicating with the outside air by means of a long glass tube passing through the oven wall. In this way, a large excess of water was always present in the vessel. A temperature of 70°C was maintained for 3 hours, followed by natural cooling to room temperature.

During both types of exposure, temperatures were measured by means of a thermometer suspended near the specimen. Readings were initially checked against thermocouples attached to the specimens.

(v) The fatigue test specimens were manufactured in the same manner as the standard specimens but were ¾" wide. These were ultimately machined to ½" width and ¼" holes were drilled 4" apart on the centre line.

The fatigue tests were done on R.A.E. direct stress machines and the ½" wide specimens were nominally in shear and the loading was such that the fluctuating stresses were applied as defined by $P(1 \pm \frac{1}{2})$.

The centrifugal force of an out-of balance bob-weight provided the fluctuating load, so that this varied as the square of the speed. The machines were driven by synchronous A.C. motors. Mains variation was checked for some weeks prior to the tests. The frequency was measured against 50 cycles obtained by sub-division of the Droitwich 200,000 cycle carrier wave. This method of checking was found to be quicker and more accurate than measuring the speed of the machine, although cross-checks on the speed were made.

(vi) Test specimens were immersed in various aircraft fluids, this being done by suspension so that the joint area was completely covered throughout the period of the test. All the fluids were at room temperature and shear strength was determined after a period of 1 month and 6 months immersion, with the exception of methylene dichloride and paint remover from which specimens were tested after 1 day and 10 days,

The following fluids were used:-

(a) High-aromatic fuel to Specification R.D.E/F 75

(b) Engine lubricating oil to Specification D.E.D.2472

(c) Engine coolant to Specification D.T.D.779
(30% ethylene glycol and 70% water)

(d) De-icing fluid to Specification D.T.D.406A

(e) Methylene dichloride

(f) Paint remover to Specification D.T.D.226

(g) Distilled water

(vii) Test specimens were subjected to intermittent seawater spray, three times a day, five days a week for twelve months. The specimens were suspended under outdoor conditions but with roof and three sides cover. Control specimens were tested before and after the twelve months period and effect on shear strength was determined.

(viii) The effect of ageing on shear strength was determined on test specimens stored at room temperature and at 70°C. The specimens were stored in closed glass containers and a batch was removed every three months for strength tests. For the ageing at 70°C the glass container was kept in a thermostatically controlled oven.

For the ageing at room temperature, results are given for a total period of 1 year 3 months whereas for the ageing at 70°C, results are given for a total period of 2 years 6 months. This difference between the two ageing periods is due to non-availability of specimens at the time of commencing the tests.

SUMMARY OF RESULTS

TABLE I

Type of Test	Redux K.6			Araldite Type 1		
	Failing Load - lb					
	Max.	Min.	Mean	Max.	Min.	Mean
Effect of temperature						
-60°C	A.2315	1645	1945	2100	1680	1904
100°C	A.423	160	248	2030	925	1504
Repeat 100°C	A. -	-	-	1900	1730	1824
30 minutes 100°C	A. -	-	-	1970	1513	1783
Effect of sheet thickness						
0.080" - 14 SWG	A.2710	2100	2340	2300	1650	1970
0.064" - 16 SWG	A.2700	1600	2060	2500	1650	2142
0.022" - 24 SWG	A.1400	1380	>1394	1420	1300	>1388
Effect of double cure	A.2200	1699	1875	2220	1800	1994
Effect of cyclic conditions						
Hot/dry 5 cycles	P.2290	1735	>2023	2040	1780	1918
" 10 cycles	P.2310	1915	>2119	2060	1595	1872
" 20 "	P.2330	1800	>2073	2060	1665	1810
" 5 "	A.2330	1670	>2002	1880	1370	1682
" 10 "	A.2060	1630	>1899	1985	1200	1580
" 20 "	A.2210	1015	1786	2280	1235	1851
Hot/wet 10 cycles	P.2155	1555	>2029	1820	1350	1605
" 20 "	P.2095	1660	>1906	1660	1145	1449
" 10 "	A.2120	1670	1871	-	-	-
" 20 "	A.2120	1235	1699	2280	1810	2073
" (Repeat) 20 "	A. -	-	-	2090	1420	1852
Effect of aircraft fluids						
High aromatic fuel						
1 month	A.2144	1767	1938	2188	1955	2004
6 months	A.2040	1730	1927	2042	1760	1849
Engine lubricating oil						
1 month	A.2226	1533	>2062	2213	1732	>2001
6 months	A.2240	1612	2003	2076	1376	1783
Engine coolant						
1 month	A.2148	1472	1856	2070	1470	1783
6 months	A.2240	1400	1834	1812	1258	1658
De-icing fluid						
1 month	A.2078	1230	1634	2083	1506	1829
6 months	A.2790	1474	1884	1956	1700	1848

SUMMARY OF RESULTS (contd)

TABLE I

Type of Test	Redux K.6			Araldite Type 1		
	Failing Load - lb					
	Max.	Min.	Mean	Max.	Min.	Mean
Effect of aircraft fluids (contd)						
Methylene dichloride						
1 day	A.1548	622	1203	2040	1782	1952
10 days	A. 200	120	138	1953	1571	1808
Paint remover						
1 day	A.2060	1027	1671	2010	1530	1825
10 days	A.1916	1116	1492	2053	1693	1822
Distilled water						
1 month	A.2076	1590	1866	2136	1556	1901
6 months	A.2140	1624	1890	1867	1528	1880
Effect of corrosive conditions	A.1758	1026	1318	1796	1272	1548
Effect of ageing						
20°C - 3 months	A.2460	1194	2024	2132	1606	1841
" 6 "	A.2272	1730	2033	2177	1666	1995
" 9 "	A.2430	1088	>1943	2084	1400	1829
" 12 "	A.2076	1208	>1838	1816	1644	1732
" 15 "	A.2160	1564	1945	1672	1476	>1609
70°C - 3 months	A.2014	1556	1793	1987	1700	1811
" 6 "	A.2003	1582	1884	2064	1450	1763
" 9 "	A.2132	1282	1748	2034	1442	1795
" 12 "	A.2084	1572	1868	2094	1788	1800
" 15 "	A.1965	1540	1804	2062	1454	>1700
" 18 "	A.2154	1320	1840	1971	1204	1670
" 21 "	A.2069	1300	>1726	2092	1540	1876
" 24 "	A.1962	1204	1647	2036	1552	1734
" 27 "	A.1920	1388	1726	2076	1316	1660
" 30 "	A.2040	1432	1701	2008	1480	1658
Fatigue 20,000,000 cycles	lb. load per joint P.125 ± 62 A.120 ± 60			lb. load per joint 140 ± 70 135 ± 67		

A = Anodised

P = Pickled

> = At least one specimen broke in the sheet

TABLE II

Control Tensile Results

Sheet No.	Test Piece No.	Condition	Ult. Tensile Stress Tons/Sq. in.	0.1% Proof Stress Tons/sq. in.	Elongation % (2" G.L.)
1	1AB	DTD.610 B	28.6	18.15	11.0
	46RS		27.3	18.70	14.0
	23GH		27.2	18.65	18.0
	22RS	DTD.546	27.7	23.00	9.0
	44AB		27.6	22.60	9.5
	28LM		28.1	23.65	9.5
2	103AB	DTD.610 B	28.4	18:74	21.5
	114GH		28.53	19.3	19.5
	149RS		29.38	19.4	22.0
	127CD	DTD.546	28.7	24.32	8.5
	137NP		28.05	23.95	8.0
	143AB		28.27	24.10	9.0
3	150AB	DTD.610 B	29.2	19.67	19.0
	181JK		28.35	19.55	22.0
	198RS		28.73	19.35	19.0
	162GH	DTD.546	28.36	24.25	9.5
	190CD		28.55	23.85	8.0
	174NP		28.55	24.10	9.0
4	318NP	DTD.610 B	29.40	20.25	17;0
	330AB		26.40	17.6	19.0
	335RS		27.20	18.1	19.0
	302CD	DTD.546	26.20	22.90	8.0
	312GH		28.05	24.25	9.0
5	401A		28.7	24.0	9.5
	404S		27.9	24.8	8.0
	413H	DTD.546	27.5	22.5	10.0
	420P	(3 for	28.5	24.5	10.0
	434K	corrosion)	29.0	24.0	7.0
	448S		28.0	23.7	8.0
6	450B		28.1	19.3	14.5
	461K		28.1	18.8	15.0
	472P		27.1	18.5	15.0
	480D	DTD.610	29.1	19.3	12.5
	488H		27.8	19.2	18.5
	497S		29.1	18.9	21.5

TABLE II (Contd)

Control Tensile Results

Sheet No.	Test Piece No.	Condition	Ult. Tensile Stress Tons/sq. in.	0.1% Proof Stress Tons/sq. in.	Elongation % (2" G.L.)
7	601AB 629JK 640RS	DTD.546	27.8 28.8 28.2	23.8 25.3 24.2	9.0 9.0 12.0
	609JK 634LM 647GH	DTD.610	27.2 27.2 26.8	17.6 18.0 17.8	22.0 19.0 22.0
8 (Corrosion)	2RS 3EF 7AB	DTD.546	27.1 26.8 26.2	22.6 23.0 22.2	8.0 8.0 9.0
9	14 SWG	DTD.610 B DTD.546	28.1 26.6	17.4 21.9	19.5 10.0
10	16 SWG	DTD.610 B DTD.546 B	28.8 25.3	19.65 21.6	16.0 10.0
11	24 SWG	DTD.610 B DTD.546 B	25.5 27.1	17.8 21.8	9.0 10.0

The Specification mechanical properties for DTD.610 B and DTD.546, are:-

	Ult. Tensile Stress Tons/sq.in	0.1% Proof Stress Tons/sq.in	Elongation %
DTD.610 B	25.0	15.0	15.0
DTD.546	27.0	21.0	8.0

7 Discussion of results

(i) Effect of temperature

Temperatures in the range of -60°C to 100°C have little or no effect on the shear strength of Araldite joints. No reduction in strength was found after maintaining a temperature of 100°C for 30 minutes.

At 100°C Redux joints gave a decrease in shear strength of about 87% of room temperature strength. Redux joints were unaffected at -60°C.

(ii) Effect of metal sheet thickness

As would be expected, the apparent shear strength of both Redux and Araldite joints was increased with increase of metal thickness. This effect was more marked with the Redux joints, and may be caused by the bonding pressure being higher than that used for Araldite joints.

When the joints made with 24 SWG metal were tested, 80% of the failures were in the metal.

(iii) Effect of double cure

Re-curing or double cure does not affect the initial shear strength of either Redux or Araldite joints.

(iv) Effect of cyclic conditions of temperature and humidity

Redux joints, both pickled and anodised, were only slightly reduced in shear strength after exposure to hot/dry and hot/wet cycles. For both cycles, the anodised joints gave lower figures than the pickled joints and the hot/wet cycles produced the greater loss of strength. The maximum loss was 11% for the anodised Redux joints after exposure to 20 cycles of hot/wet conditions.

The results shown in the summary for Araldite specimens are in some instances for tests which have been repeated, the original results having indicated that the manufacturing technique may have been at fault.

Araldite joints, both pickled and anodised, were slightly reduced in shear strength after exposure to the hot/dry conditions. Exposure to 20 cycles produced a loss of 12% for the pickled specimens and 13% for the anodised specimens.

The hot/wet conditions affected the shear strength of pickled Araldite joints to a greater degree, ~~the loss~~ being 29% after 20 cycles. Anodised Araldite joints sustained a slight loss in strength, this being only 3 to 14%.

(v) Fatigue

The endurance limits for 20 million cycles are in the descending order, Araldite pickled, Araldite anodised, Redux pickled and Redux anodised. All failures except one were in the metal sheet. The thickness of each sheet was checked for each group of joints, and found to be similar, so that the difference in endurance limit is still present when expressing the curves in terms of stress on the sheet.

From the results obtained, it is concluded that the fatigue properties of both Redux and Araldite are satisfactory for the gauge of sheet used for the specimens. The fatigue curves are shown in Figs. 1 and 2. The work is to be repeated using a thicker sheet for the specimens.

(vi) Effect of immersion in various aircraft fluids

The results obtained after specimens had been immersed in various fluids for varying periods were satisfactory, with the exception of Redux specimens after immersion in methylene dichloride and paint remover. The loss in shear strength was greater after immersion in methylene dichloride than in paint remover. In both tests, the loss was proportional to increase in immersion period.

(vii) Effect of exposure under corrosive conditions

Both Redux and Araldite specimens sustained a loss in shear strength after exposure to corrosive conditions. Redux joints were affected to a greater degree, the scatter being more marked due to metal corrosion within the joint area. Evidence of metal corrosion was visible within the Araldite joint area in some instances but was slight.

(viii) Effect of ageing

Both Redux and Araldite specimens sustained a slight loss in mean shear strength after ageing at room temperature and at 70°C. This loss was almost negligible and could be attributed to normal scatter.

8 Conclusions

Reference to the results shown in Table I indicates that the difference between maximum and minimum failing loads is appreciable throughout the range of tests. This scatter is not an unusual occurrence in the testing of adhesives but some of the results obtained in this programme can be considered as exceptional. It is reasonable to assume that instances of wide variation in results are not entirely due to test conditions, but to inconsistency of manufacturing technique. It is unfortunate that substandard specimens cannot be identified except by testing to failure and that this has not usually been done until after considerable periods of subjection to test conditions.

It has been generally accepted that a minimum failing load of 1500 lb is reasonable for evaluating standard test specimens bonded with Redux and Araldite. If the results are judged by this criterion, then it will be seen that, in general, there is little difference between the two adhesives. This conclusion can be applied to the assessment of results obtained from most of the test conditions although the shear strength of Redux specimens is reduced in some instances. This reduction results from subjection to a temperature of 100°C, immersion in methylene dichloride or paint remover and storage under corrosive conditions. Araldite specimens can be considered as satisfactory under all the test conditions.

REFERENCES

No.	Author	Title etc.
1	H.W. Hall	Araldite metal to metal synthetic resin adhesive. Tech Note No. Chem.1043, March 1948.

Attached

Figs. 1 and 2 CH.1252 and 1253.

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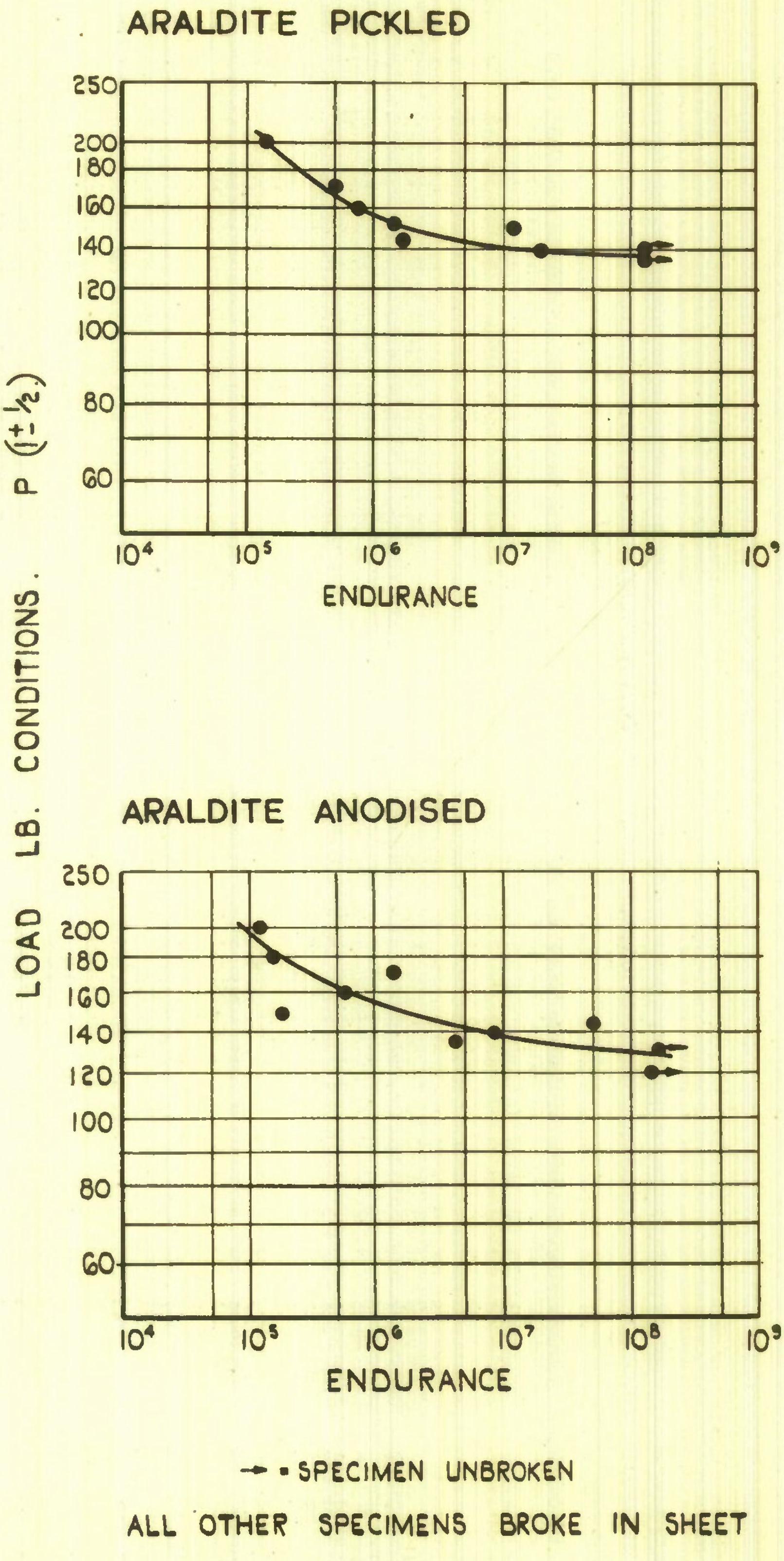
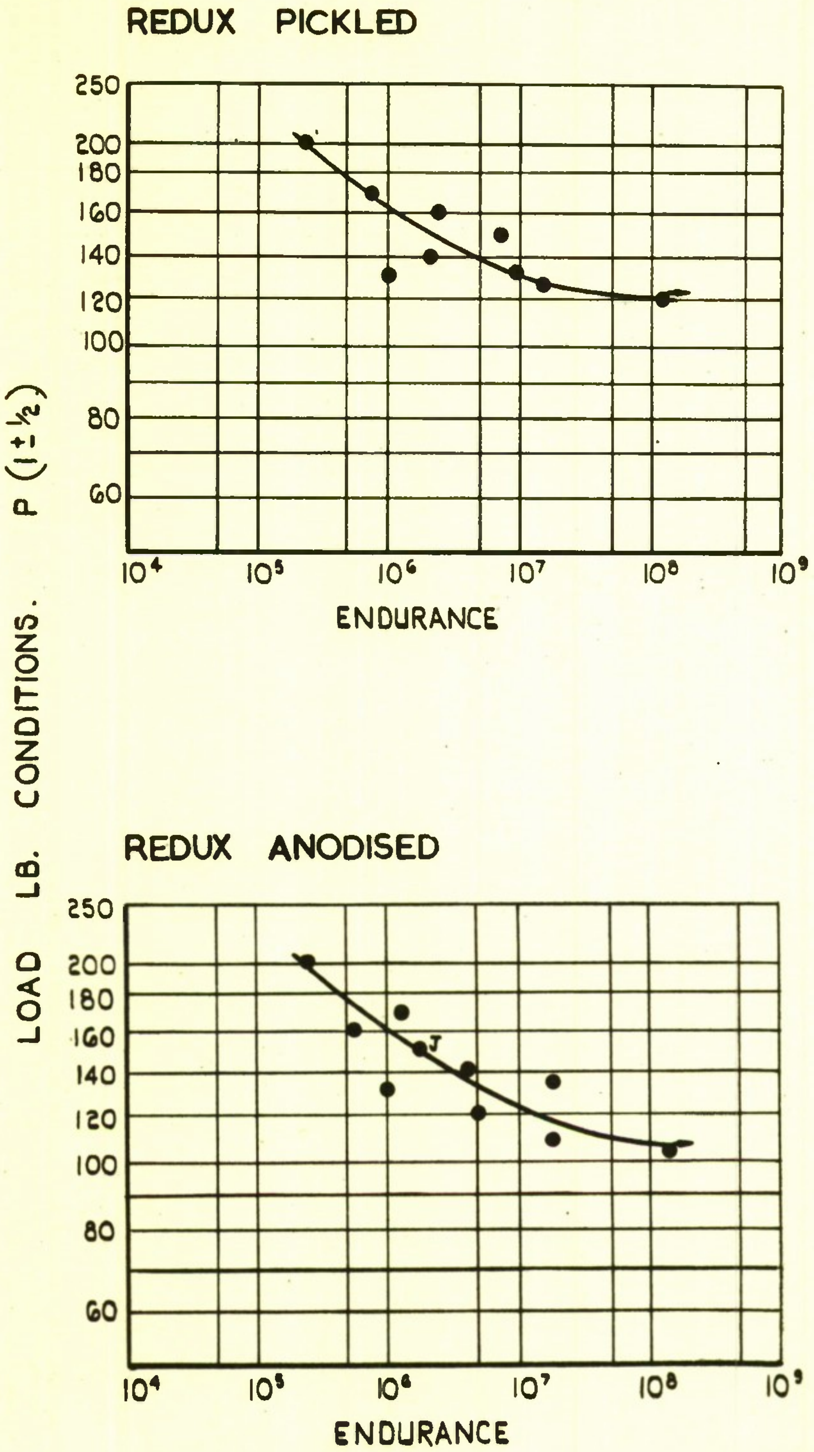


SINGLE LAP JOINTS – ½" OVERLAP – ½" WIDTH

FIG. 1 FATIGUE S/N CURVES – 20G. DTD. 546 B



→ = SPECIMEN UNBROKEN

J = SPECIMEN BROKE IN JOINT

ALL OTHER SPECIMENS BROKE IN SHEET

SINGLE LAP JOINTS – ½" OVERLAP – ½" WIDTH

FIG.2 FATIGUE S/N CURVES — 20 G DTD. 546 B

[dstl]

Information Centre
Knowledge Services
[dstl] *Porton Down,*
Salisbury
Wilts
SP4 0JQ
Tel: 01980-613753
Fax 01980-613970

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Covering dates 1952
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