Structural Adhesives—Characteristics and Application

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Introduction

Probably the most significant development in the field of adhesives is that of structural metal adhesives which have found such widespread use in the aircraft and missile industry. When used properly in lightweight structures, structural adhesives make possible bonded metal assemblies in which the metal is stressed beyond its elastic limit thereby achieving a 100% bond. In other words the use of a structural adhesive makes it possible to take advantage of the total strength of the metal since fastening is accomplished to every increment of the metal surface. The data in Table I show what can be accomplished by this type of bond in terms of metal stress and deformation.

The purpose of this discussion is to describe briefly some characteristics of structural adhesives. One application will also be described along with the reasons for making the choice of adhesive which was finally used.

Structural Adhesives Characteristics

Structural adhesives can be divided roughly into two broad classifications. One classification is the hard, relatively brittle type with high shear and tensile strengths combined with low peel strengths. Epoxy resin adhesives are typical of this class.

The second classification consists of a combination of a hard, brittle material such as an epoxy or phenolic resin with a thermoplastic or rubbery material such as a vinyl resin, a rubber, or a polyamide. This class has high shear and tensile strengths accompanied by high peel strength.

An epoxy adhesive is usually a solventless fluid or a thixotropic paste with separate curing agent which requires only contact pressure during assembly and cure. The cure temperature may vary from room temperature to 350°F. depending on the type of hardener or curing agent used to effect a cure. Epoxy adhesives are divided into three groups on the basis of cure temperature in specification MIL-A-8623.

The tensile adhesion properties of some epoxy adhesives meeting MIL-A-8623 type II are shown in Table II. These are all filled epoxy adhesives cured with diethylaminopropylamine. Shear properties per ASTM D1002 and T peel properties are shown in Table III. Surface treatment used is the sulfuric acid-dichromate etch per MIL-A-9067 unless other treatments are described. Tensile adhesion strengths are high even after exposure to humidity and thermal cycling. Shear strengths are good but probably suffer from the fact that the loads caused deformation in the metal with resultant peel components. The peel strengths are only a few pounds per inch.

The high-peel strength type of adhesive is usually available as a single component solution or film. The film may be used with or without a primer depending on the ability of the film to wet the adherend surface when it is activated by heat and pressure. Other reasons for using a primer may be to protect the adherend surface prior to bonding and to provide a tacky surface to facilitate adhesiveadherend assembly for complicated parts. This type of adhesive usually requires temperatures of 300 to 350°F. and pressures up to 200 psi for 60 to 90 min. to achieve a proper cure. The shear and T peel properties of some of these adhesives are shown in Table IV. The data show that those two-part adhesives which contain a rubber or a thermoplastic component, such as polyvinyl butyral or a polyamide, have a relatively high peel strength (72.0 lb./in.).

Application

The design for our application required that a thin gage (0.020-0.025 in.) aluminum cylinder be bonded to a heavy aluminum cap using a lap joint, as shown in Figure 1. The bond would have to

 TABLE I

 Applied Load vs. Stress and Elongation in a Structural

 Bond^{a,b}

| | Ap- | | | | | Elon- | Elon- |
|----------|-------|-------|----------------|-------|----------------|-------|-------|
| | plied | Bond | Bond | Sheet | Sheet | ga- | ga- |
| | load, | area, | stress, | area, | stress, | tion, | tion, |
| No. | lb. | in.² | \mathbf{psi} | in.² | \mathbf{psi} | in. | % |
| 1 | 3040 | 0.75 | 4050 | 0.050 | 60,800 | | |
| 2 | 2940 | 0.75 | 3920 | 0.050 | 58,800 | | |
| 3 | 3040 | 0.75 | 4050 | 0.050 | 60,800 | | |
| 4 | 2990 | 0.75 | 3990 | 0.050 | 59,800 | | |
| Av. | 3000 | 0.75 | 4000 | 0.050 | 60,000 | 0.360 | 5.75 |
| 6 | 3230 | 1.00 | 3230 | 0.050 | 64,600 | | |
| 7 | 3280 | 1.00 | 3280 | 0.050 | 65,600 | | |
| 8 | 3210 | 1.00 | 3210 | 0.050 | 64,200 | | |
| 9 | 3140 | 1.00 | 3140 | 0.050 | 62,800 | | |
| Av. | 3215 | 1.00 | 3215 | 0.050 | 64,300 | 0.53 | 18.85 |
| 11 | 3400 | 1.50 | 2266 | 0.050 | 68,000 | | |
| 12 | 3295 | 1.50 | 2196 | 0.050 | 65,900 | | |
| 13 | 3345 | 1.50 | 2230 | 0.050 | 66,900 | | |
| 14 | 3345 | 1.50 | 2230 | 0.050 | 66,900 | | |
| Av. | 3346 | 1.50 | 2230 | 0.050 | 66,920 | 0.845 | 12.1 |

^a The specimen was a double lap shear type prepared from 2024-T3 aluminum strip 0.025 in. thick by 1 in. wide. The aluminum surface was treated with a sulfuric acid-dichromate solution per MIL-A-9067.

 $^{\rm b}$ The adhesive was a polyvinyl butyral phenolic cured at 350°F. for 1 hr. at 50 psi pressure.

^o Rate of head travel was 0.050 ipm.

support a permanent shear stress of approximately 230 psi. The cure temperature was limited to 140°F. although the bond itself would be exposed to 165°F. under load for short periods after being cured.

Since the thin skin was cylindrical and the load was parallel to the axis of the cylinder, it was reasoned that the aluminum cylinder would tend to neck in¹ so that any existing forces however slight, other than shear, would put the bond in compression thereby minimizing the possibility of any peel component.

TABLE II Tensile Adhesion Strength (psi) of Epoxy Adhesives Cured with Diethylaminopropylamine^a

| Conditioning ^b | Spec. No. | Epoxy No. 1 | Epoxy No. 2 | Epoxy No. 3 | Epoxy No. 4 |
|----------------------------|--------------|----------------|----------------|----------------|----------------|
| Controls | 1 | 5400 | 6200 | 7500 | 5540 |
| | 2 | 6600 | 7500 | 7520 | 6000 |
| | 3 | 7380 | 6560 | 7400 | 7100 |
| | 4 | 7020 | 5380° | 6720 | 7080 |
| | 5 | 7820 | 4620 | 7220 | 6720 |
| | Av. | 6840 | 6050 | 7270 | 6480 |
| Specimens given | 1 | 7470 | 8340 | 8020 | 5660 |
| 24 hr. cycle | 2 | 8340 | 6040 | 8550 | 7310 |
| -65°F. 8 hr. | 3 | 7520 | 7010 | 9100 | 7190 |
| +165°F. 16 hr | . 4 | 8530 | 6620 | 8790 | 7330 |
| 4 cycles | 5 | 7410 | 8600 | 9310 | 7330 |
| | Av. | 7850 | 7320 | 8550 | 6960 |
| SCEL ^a humidity | 1 | 6360 | 6180 | 6780 | 6360 |
| cycle 20 days | 2 | 5100 | 5940 | 7200 | 6900 |
| | 3 | 6220 | 5000 | 7640 | 6400 |
| | 4 | 6460 | 6880 | 7820 | 6620 |
| | 5 | 6200 | 6160 | 7460 | 6760 |
| | Av. | 6060 | 6030 | 7380 | 6600 |

^a Specimens consisted of aluminum (6061-T6) tensile plugs $1^{1}/_{2}$ in. long, 1.129 in. in diameter. They were abraded and wiped with clean acetone before being coated with the adhesive and assembled in a jig under sufficient spring pressure to maintain contact during cure. Rate of head travel during test was 0.050 ipm.

^b All specimens cured $2^{1}/_{2}$ -4 hr. at 165°F.

^o Specimen did not break. Threads stripped out at load of 5380 lb.

^d Šignal Corps Electronics Lab.

The lack of any appreciable peel forces meant that an epoxy adhesive could be used. With this in mind it was decided that the method which would insure a positive fill in the bond area was to inject the epoxy resin into the bond area after the mating surfaces were assembled. A circumferential groove was machined into the heavier adherend surface to

| Tensile Shear and T Peel Strengths of Epoxy No. 2 with Aluminum | | | | | | | | |
|---|---|---|---|---|--|--|--|--|
| | Controls | After thermal cycling | SCEL humidity | 30 days at 165°F. | | | | |
| Tensile shear psi per ASTM D1002 | $2850 (5^{a}) (2610-3060^{b})$ | $3202 (5^{a}) (3150-3260^{b})$ | 2630 (5 ^a) (2560-2690 ^b) | 3260 (5 ^a) (2980–3430 ^b) | | | | |
| T peel,° lb./in. | $2.0(5^{a})$ (1.3-2.5 ^b) | $2.8(5^{a})$ (1.7-4.6 ^b) | $2.3 (5^{a})$ (1.6-3.6 ^b) | $3.7 (5^{a}) (1.7-8.8^{b})$ | | | | |

 TABLE III

 Fensile Shear and T Peel Strengths of Epoxy No. 2 with Aluminu

* Number of specimens tested.

^b Minimum and maximum values of specimens tested.

^o The T peel specimen consisted of 1 in. wide strips of 0.025 in. thick aluminum 5 in. long bonded together for 4 in. of the length. They were pulled at 10 ipm of head travel.

| | Average values ^a for the following conditions | | | | | | |
|---|---|------------------------|----------------------------|-----------------------|---------------------------|-------------------------------------|--|
| Adhesives ^b type | Test method | Controls, Av. | After 2 hr. 350°F., av. | —65 to 165°F., av. | 50 hr. salt spray, av. | 20 days SCEL humidity, av. | |
| Polyvinyl butyral phenolic | T peel ^o Tensile shear ^d | 28.8 (5) 3378 (5) | 6.1(5) 3572(5) | 32.8 (6) 3358 (5) | 25.0 (5) 3020 (5) | 38.3 (6) 3092 (5) | |
| Nylon epoxy No. 1 | T peel Tensile shear | 49.7 (6) 4666 (5) | 45.7 (6) 4990 (5) | 58.2 (6) 4476 (5) | 34.8 (6) 4336 (5) | 56.5 (6) 3790 (5) | |
| Epoxy phenolic | T peel Tensile shear | 4.0 (5) 2144 (5) | 4.0 (6) 2310 (5) | 4.7 (6) 2132 (6) | 4.0 (6) 2000 (5) | 5.0 (6) 2136 (5) | |
| Epoxy/nitrile rubber phenolic (2 phase) | T peel Tensile shear | $15.6(5)\ 2438(5)$ | 6.0(5) 2428(5) | 12.4 (5) 2328 (5) | 14.0 (5) 2100 (5) | 16.5 (5) 2184 (5) | |
| Nitrile rubber phenolic | T peel Tensile shear | $39.8(5)\ 2416(5)$ | 23.8(5) 3614(5) | 44.8 (5) 2282 (5) | 41.4 (5) 1878 (5) | 44.2 (4) 1358 (5) | |
| Epoxy nylon No. 2 | T peel Tensile shear | $96.0\ (5)\ 4558\ (5)$ | 51.0 (4) 4342 (5) | 33.0 (5) 5248 (5) | 87.0 (5) 4720 (5) | 38.4 (5) 2124 (5) | |
| Adhesives type | $\begin{array}{c} {\bf Test} \\ {\bf method} \end{array}$ | 200°F., Av. | 250°F., Av. | 300°F., Av. | | | |
| Epoxy nylon No. 1 | T peel, lb. Tensile shear, psi | 73.6 (5) 1748 (5) | 40.2 (5) 476 (5) | 14.2 (5) 218 (5) | <u></u> | | |
| Epoxy nylon No. 2 | T peel, lb. Tensile shear, psi | 62.0 (5) 2768 (5) | 12.2 (5) 1348 (5) | 7.4 (5) 700 (5) | | | |

 TABLE IV

 Evaluation of Different Types of Adhesives

* Figures in parentheses represent number of specimens tested.

^b All adhesives cured per manufacturers recommendations.

• T peel specimen-see footnote c, Table III.

^d Tensile shear specimen per ASTM D'1002.

lead the adhesive around (Fig. 1). There was enough clearance between adherend surfaces so that the adhesive tended to fill in the whole lap area. The thixotropic epoxy adhesive was injected into the bond interface by means of a series of holes equally spaced around the cylinder. Since it was thixotropic it did not tend to flow out of the bond area.

The adhesive used for this application was epoxy adhesive No. 1, a filled epoxy resin cured with diethylaminopropylamine per MIL-A-8623 type II. Strength properties of this type of adhesive to aluminum are shown in Tables II and III.

On the basis of the foregoing reasons a full-scale experiment was made. The cylindrical 6061-T6 aluminum skin 0.022-0.025 in. thick was bonded to the 7075-T6 aluminum cap with a 0.600 \pm 0.010 in. lap joint. The surfaces to be bonded were sandblasted.

When the unit was tested to destruction, axially in tension, the aluminum skin failed at approximately 900 lb. load/in. of overlap or at a 1400-psi stress in the bond. In other words the ultimate strength of 6061 aluminum (35,000 psi) was exceeded.

In order to test the bond more fully it was decided to make the cylindrical skin from 7075-T6 aluminum with a yield strength of 70,000 psi. Two types of mechanical joints were evaluated along with the adhesive bonded joints.

One of the mechanical joints consisted of a groove cut into the heavier aluminum section with the other section being rolled into the groove and bound with music wire. The other joint was made by using No. 6-32 machine screws approximately 1.4 in. apart. These were compared to two bonded units using epoxy adhesive No. 1. The results are shown in Table V. The mechanical joints

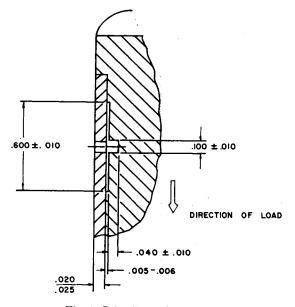


Fig. 1. Injection seal application.

failed at 180 lb./in. of joint for the screw joint with the metal tearing at the screw holes. The rolled-in. joint failed at a much lower value. The two bonded joints failed at 1300 and 960 lb./in. of joint which in terms of stress in the aluminum skin is 56,500 and 41,700 psi, respectively.

Since the unit was required to support a permanent load in actual operation and might conceivably be exposed to 165° F., a 24-hr. test was run at $160 \pm 5^{\circ}$ F. with a bond stress of 300 psi. Epoxy adhesive No. 1 failed this test after 6 hr. Since it was known that this adhesive was slightly flexibilized, tests were made comparing this adhesive to another epoxy (epoxy adhesive No. 5) which was considered to have better high temperature properties but which could still be cured at 140° F. The results of this comparison are shown in Table VI.

The variables evaluated in Table VI for two different epoxy adhesives were the cleaning method and elevated temperature strengths. While test results at 250° F. are not necessarily representative of operation at 165° F., these results do give added assurance of the greater heat resistance of epoxy adhesive No. 5.

Compression shear specimens were used to evaluate these variables because it was felt that this type of specimen was relatively independent of the peel forces to which tensile shear specimens are subject.

While the dichromate treatment seemed to give slightly better results at 165 and 250°F., the greater convenience of being able to protect certain areas

TABLE V Relative Strengths of Mechanical and Bonded Joints^{a, b}

| Fastening method | Failing load per in. of joint, lb. | Joint stress at failure, psi | Stress in Al skin, psi |
|--------------------|---|------------------------------------|------------------------------|
| Rolled into groove | | | |
| and bound with | | | |
| music wire | 88 | 150 | 3,800 |
| Machine screws 1.4 | | | |
| in. apart | 180 | 300 | 7,800 |
| Bonded joint No. 1 | 1,300 | 2,150 | 56,000 |
| Bonded joint No. 2 | 960 | 1,600 | 42,000 |

• These specimens were bonded with epoxy adhesive No. 1. • The surfaces to be bonded were sandblasted.

TABLE VI Effect of Surface Treatment and Test Temperatures on

| Epoxy | Cure time, hr. | Temp. cured, °F. | Treatment | Temp., tested, °F. | Psi |
|-------------|----------------------|------------------------|------------|--------------------------|-------------|
| Epoxy No. 1 | 16 | 140 | Sandblast | 165 | 2660 |
| Epoxy No. 1 | 16 | 140 | Sandblast | 165 | 2680 |
| Epoxy No. 1 | 16 | 140 | Sandblast | 165 | 2790 |
| Av. | | | | | 2710 |
| Epoxy No. 5 | 16 | 140 | Sandblast | 165 | 3280 |
| Epoxy No. 5 | 16 | 140 | Sandblast | 165 | 2710 |
| Epoxy No. 5 | 16 | 140 | Sandblast | 165 | 3385 |
| Av. | | | | | 3120 |
| Epoxy No. 1 | 16 | 140 | Sandblast | 250 | 190 |
| Epoxy No. 1 | 16 | 140 | Sandblast | 250 | 205 |
| Epoxy No. 1 | 16 | 140 | Sandblast | 250 | 175 |
| Av. | - | | | | 190 |
| Epoxy No. 5 | 16 | 140 | Sandblast | 250 | 495 |
| Epoxy No. 5 | 16 | 140 | Sandblast | 250 | 390 |
| Epoxy No. 5 | 16 | 140 | Sandblast | 250 | 400 |
| Epoxy No. 5 | 16 | 14Ò | Sandblast | 250 | 300 |
| Av. | | 1 | | | 396 |
| Epoxy No. 1 | 16 | 140 | Dichromate | 165 | 2850 |
| Epoxy No. 1 | 16 | 140 | Dichromate | 165 | 3140 |
| Epoxy No. 1 | 16 | 140 | Dichromate | 165 | 3070 |
| Av. | | | | | 3020 |
| Epoxy No. 5 | 16 | 140 | Dichromate | 165 | 342Ö |
| Epoxy No. 5 | 16 | 140 | Dichromate | 165 | 3790 |
| Epoxy No. 5 | 16 | 140 | Dichromate | 165 | 3070 |
| Ay. | | | | | 3430 |
| Epoxy No. 1 | 16 | 140 | Dichromate | 250 | 175 |
| Epoxy No. 1 | 16 | 140 | Dichromate | 250 | 195 |
| Epoxy No. 1 | 16 | 140 | Dichromate | 250 | 140 |
| Av. | | | | | 170 |
| Epoxy No. 5 | 16 | 140 | Dichromate | 250 | 520 |
| Epoxy No. 5 | 16 | 140 | Dichromate | 250 | 670 |
| | - | | | | |

140

140

16

16

Epoxy No. 5

Epoxy No. 5

Av.

250

250

Dichromate

Dichromate

530

545

566

| Specimen | | Length | Initial bond stress, . psi | Failure Load | | | |
|----------|--------------------|---------|-------------------------------------|--------------|--------------|---------------------------|--|
| No. | Environment | of test | | Bond stress | Metal stress | Joint details | |
| 1 | -65°F. | 24 hr. | 300 | 1350 | 34,500 | Sandblasted surface | |
| 2 | SCEL salt spray | 50 hr. | 300 | 720 | 18,500 | Dichromate etched surface | |
| 3 | SCEL humidity | 20 days | 300 | 1900 | 48,500 | Dichromate etched surface | |
| 4 | SCEL thermal shock | 28 hr. | 300 | 1700 | 43,500 | Sandblasted surface | |
| 5 | SCEL humidity | 20 days | 300 | 2550 | 65,000 | Sandblasted surface | |
| 6 | SCEL salt spray | 50 hr. | 300 | 2350 | 60,000 | Sandblasted surface | |
| 7 | Comb.ª | a | 300 | 900 | 22,900 | Sandblasted surface | |
| 8 | Comb.ª | | 300 | 1800 | 47,000 | Sandblasted surface | |

 TABLE VII

 Results of Joint Environmental Tests—Static Tests of Bonded Joints

* Specimens Nos. 7 and 8 were subject to a combination of environments as follows: 24 hr. at 160°F., 24 hr. at -65°F., 20 days in SCEL humidity, 28 hr. in SCEL thermal shock, and 50 hr. in SCEL salt spray.

by use of masking, led to the choice of sandblast as the cleaning method.

The epoxy adhesive No. 5 was chosen for subsequent evaluation because of the elevated temperature requirement. The elevated temperature test, 24 hr. at $160 \pm 5^{\circ}$ F. with a 300-psi bond stress was repeated on a representative unit using epoxy adhesive No. 5. The unit passed this successfully and was then tested to destruction at ambient temperature. It failed at the bond with a bond stress of 1200 psi.

Subsequent test results on representative units are shown in Table VII. Except for two low values for specimen Nos. 2 and 7, the failing loads were satisfactory. The low value for specimen No. 7 was attributed to the fact that the bond area was not completely filled with adhesive. Specimen No. 5 failed in the metal skin at a metal stress of approximately 65,000 psi.

While the bond evaluation was made on 7075-T6 aluminum in the thin section, the final design for production used 6061-T6 aluminum because of its greater ductility and ease of production.

Conclusions

On the basis of these results this design was accepted for production units. The design minimized peel forces making possible the use of an epoxy adhesive with the following advantages.

The cure temperature was kept within an acceptable limit.

Since a filled solventless epoxy adhesive has practically negligible shrinkage and can still be handled like a fluid, it was possible to inject the adhesive into the bond area after the unit was assembled, thus minimizing the possibility of starved areas due to wiping action. Assuming that the same strength bond was obtained with the two different aluminum alloys, a greater degree of assurance in the strength of the bonded joints was made possible by substituting the stronger aluminum alloy 7075-T6 for the 6061-T6 aluminum alloy actually used in the end item.

Reference

1. Popov, E. P., *Mechanics of Materials*, Prentice-Hall, Englewood Cliffs, N. J., p. 27.

Synopsis

Structural adhesives are one of the more important developments in the adhesives field because they make possible bonded assemblies in lightweight structures which take full advantage of the strengths of the materials used. Structural adhesives can be divided into two classes. One class consists of hard brittle resins typified by unmodified epoxy adhesives which have high shear and tensile strengths but low peel strength. The second class consists of a combination of resins resulting in a more ductile type of adhesive which has high peel strength along with high shear and tensile strength. One application is described here in which an epoxy adhesive is used in a design which minimizes peel forces and which causes failure of the bonded assembly in the metal. The evaluation of this design showed it to be successful.

Résumé

Les adhésifs structurés se situent parmi les développements les plus importants dans le domaine des adhésifs parce qu'ils rendent possible l'existence d'ensembles liés dans des structures légères qui profitent pleinement de la solidité des matériaux utilisés. Les adhésifs structurés peuvent être divisés en deux classes. Une classe consiste en résines dures et cassantes caractérisées par des adhésifs époxy non modifiés qui ont de grandes résistances au cisaillement et à la traction mais une faible résistance à l'écaillement. La seconde classe consiste en une combinaison de résines donnant un type d'adhésif plus ductile qui a une grande résistance à l'écaillement de même qu'une grande résistance au cisaillement et à la traction. On décrit ici une application dans laquelle un adhésif époxy est utilisé afin de minimiser les forces d'écaillement et d'assurer l'adhésion de l'ensemble au métal. Cette technique s'est avérée favorable.

Zusammenfassung

Strukturklebestoffe bilden eine der bedeutenderen Entwicklungen im Bereich der Klebestoffe, da sie Verbindungen in Leichtgewichtsstrukturen möglich machen, welche die Festigkeit der verwendeten Materialien voll ausnützen. Strukturklebestoffe können in zwei Klassen eingeteilt werden. Die eine Klasse besteht aus harten, spröden Harzen vom Typ der nichtmodifizierten Epoxyklebestoffe, die eine hohe Scher- und Zugfestigkeit, aber niedrige Ablösungsfestigkeit besitzen. Die zweite Klasse besteht aus einer Harzkombination, die zu einem duktileren Klebstofftyp mit hoher Ablösungsfestigkeit und gleichzeitig hoher Scher- und Zugfestigkeit führt. Es wird eine Anwendung beschrieben, bei welcher ein Epoxyklebestoff in einer Anordnung verwendet wird, die die Ablösungskräfte möglichst klein macht und zu einen Bruch der Verklebung in Metall führt. Diese Anordnung wurde mit Erfolg verwendet.