MASTER

ADHESIVE TORSIONAL CREEP/FAILURE STUDY

George Mills

QUALITY DIVISION

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ABSTRACT

This report describes the test method and results of the creep/failure behavior of three adhesive formulations with a torsional load applied.

The load of a predetermined value was applied to the sample (Fig. 1) via a torque wrench/holding frame apparatus (Fig. 2), and was held until the sample failed.

The adhesive formulations were tested after cure in two modes (ambient and elevated temperature) and at four temperature levels (23, 50, 100 and 140 C).

INTRODUCTION

On several occasions, it has been observed that subassembly components, bonded with L-315/Polyol/FeAA and allowed to cure at ambient temperature for 24 hours, have slipped at the bonded interface when subjected to a low but constant shear force. The shear force was imparted by component weight when the bonded subassemblies were at rest in an unlevel attitude, equator up, in a bowl fixture. In this position, the bonded interface is near the load bearing end with the greater mass above the interface, hence, the imparted shear force.

This undesirable rest method has since been corrected but the fact remained that L-315/Polyol/FeAA, after 24 hours cure, allowed the components to slip. This was discussed with D. L. Seaton (LLL) and he thought this worthy of investigation and suggested in a letter (Seaton to Purcell, dated April 29, 1975) that a torsional creep/failure study be conducted of this adhesive formulation and two additional formulations that were being considered by LLL. As a result of the conversation and consequent letter, a test was conceived and a matrix constructed.

Production associated adhesive bond samples are prepared by bonding a 2.5 cm aluminum cube to the approximate center of a 7.6 cm square aluminum plate. A weight (approximately 2.3 kg) is applied to the cube and the sample is allowed to cure at ambient temperature for 24 hours. After the required cure time, the extruded glue fillet is removed and the sample is torqued to ultimate failure load.

This method was chosen as the general model for the creep/failure study—the specific method is detailed in this report.

DISCUSSION

ALUMINUM SAMPLE PREPARATION

The 6061-T6 aluminum plates and cubes were machined from new material to a 32 finish on their mating faces. After machining, the metal was degreased in an ultrasonic Freon bath. Following the bath, the mating

faces were lightly smoothed with 600-grit wet/dry sandpaper. At this time, a random sample of 10 sets (plates and cubes) of metal was selected for dimensional verification which consisted of measuring the flatness of the mating surfaces (≤ 0.003 cm) and the area of the cube mating face (6.45 ± 0.13 square cm). After the dimensional integrity of the random samples was verified, all the metal parts were subjected to an acetone wash followed by a distilled water rinse. No water film break was allowed; if a break was observed, the acetone wash/distilled water rinse was repeated, as necessary, until no water film break was apparent. The metal parts were then dried at 100 C for 24 hours. The cleaned metal parts were handled with white cotton gloves and wrapped in Kimwipes to prevent contamination.

ADHESIVE PREPARATION

The three adhesive types tested were:

- A. L-315/Polyol/Ferric Acetylacetonate
- B. L-315/Polyol/0.03 DABCO
- C. LW520/Eutectic

The mix ratio of adhesives A and B was 100 pbw resin and 9 pbw curing agent; the mix ratio of adhesive C was 100 pbw resin and 7.5 pbw curing agent.

The resins and curing agents were heated for an hour at 75 C, degassed at 5 mm Hg for 30 minutes, then mixed. The mixture was stirred with a glass rod for 3 minutes. All mixing and application was in an area with the humidity controlled to 18% or less (corrected to 21 C).

BOND PROCEDURE

A thin, uniform coating of adhesive was brushed on the cube face. The cube and plate were mated and a weight (approximately 2.3 kg) was applied to the cube. The sample was allowed to cure for 24 hours at ambient temperature. After this ambient cure, the adhesive fillet was carefully trimmed away. The thickness of adhesive at the interface (glue gap) ranged from 0.001 to 0.004 cm.

Following the ambient cure, those samples selected for elevated temperature cure and test were "force cured" at 100 C for four hours.

TEST MATRIX

Two major test groups were evaluated: temperature cured and tested, and time cured at ambient and tested.

Ninety samples were prepared for the temperature cured and tested group consisting of 30 each of adhesives A, B, and C.

A total of 135 samples were prepared for the time cured and tested group, i.e. 45 each of adhesives A, B, and C.

The matrixes of tests are shown in Tables I and II.

Table I. Temperature Cured and Tested

Adhesive	Test Temperature			
	23 C	50 C	100 C	150 C
A	T5	T5	T5/C5	T5/C5
B	T5	T5	T5/C5	T5/C5
C	T5	T5	T5/C5	T5/C5

T = Test Sample; C = Control; Number = Quantity.

Table II. Time Cured and Tested

Time Tested (months)	Test Temperature		
	23 C	50 C	150 C
1	A5, B5, C5	A5, B5, C5	A5, B5, C5
2	A5, B5, C5	A5, B5, C5,	A5, B5, C5
3	A5, B5, C5	A5, B5, C5	A5, B5, C5

A, B, C = Adhesive Types; Number = Quantity.

The test samples prepared for the temperature cured and tested group were bonded in quantities of 10 from one adhesive mixture; three mixtures of each adhesive were required to bond the 30 samples. The test samples and controls (when required), representing one adhesive type, were bonded from the same adhesive mix. The test samples prepared for the time cured and tested group were bonded in quantities of 15 from one adhesive mix.

TEST DESCRIPTION

By placing similar test samples in temperature conditioning ovens and monitoring their temperature rise with a potentiometer, the soak time required for the samples to reach test temperature was determined. These

soak times were 15 minutes for 50 C, 20 minutes for 100 C and 30 minutes for 150 C. During actual testing, the torque fixture was conditioned a minimum of four hours before any samples were inserted in the oven to allow the large steel "heat sink" to reach test temperature.

The test samples were placed in the oven in groups of five. After the required "soak" period, the test fixture and one sample were removed from the oven and tested.

The test consisted of torquing the sample to a predetermined load and timing with a stop watch. The test was terminated at sample failure or five minutes, whichever occurred first. Five minutes was chosen as the termination point for two reasons: (1) the sample under test was slowly cooling, and (2) the samples remaining in the oven were exposed to a longer heating time. If the sample did not fail in five minutes, this was noted and the sample was torqued to ultimate failure.

Control samples, when required, were exposed to temperature conditioning along with their companion samples. At the end of the conditioning time, the control samples were removed from the oven, allowed to cool to ambient temperature, and tested.

TEST RESULTS

Because of the wide range of failure times, the median failure time was chosen as the most realistic result representative of the test (shown in Tables III through XIV). Also reported is the failure time range. Plots of the median failure times are shown in Figs. 3 and 4.

CONCLUSIONS

Adhesives A and B, when cured, are hard and unyielding at ambient temperature. Therefore, their failures at 23 C and 50 C were catastrophic. They tended to become softer at elevated temperatures. At elevated temperatures (100 C and 150 C), the softened adhesive failures were preceded by creep of from 0.6 to 1.7 N·m. The failures observed in these two adhesive types were in the adhesive.

Adhesive C remained elastic throughout the test temperature range. Failure in this material was preceded by creep of from 1.1 to 3.4 N·m, particularly at elevated temperatures. After failure, the adhesive could be cleanly peeled in sheets from the metal and would elongate approximately 50% before tearing. The failures observed in this adhesive were bonding failures; the adhesive had no real affinity for the metal.

Repeated exposure to elevated temperatures (as in the 100 C and 150 C control samples compared with the 23 C tests) appears to have a degrading effect on the strength of the adhesives.

The test samples that were "force cured" at 100 C for four hours exhibited greater resistance to torsional failure than the samples cured at ambient temperature for one and two months. The samples cured at ambient temperature for four months compared reasonably closely in torsional failure times with the "force cured" samples. Hence, the inference can be drawn that the degree of adhesion cured in four hours at 100 C is approximately equal to four months at 23 C.

Except for the unexplained failure of adhesive C in the two months, 150 C test, adhesive C exhibited greater resistance to torsional failure than adhesive A or B. Also, the fact that it retains its elasticity throughout the test temperature ranges could make its use attractive in applications in which assemblies containing materials of differing coefficient of thermal expansion (CTE) were subject to relatively wide temperature ranges.

Adhesive B appears to have greater torsional strength than adhesive A initially when cured and tested at 23 C. At 150 C, however, there is virtually no difference in torsional strengths of the two.

Fig. 1. Adhesive Formulation Sample

Fig. 2. Torque Wrench/Holding Frame Apparatus

Table III. Force Cured and Tested at 23 C

(Applied Torque, 56.5 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	248	93/300
B	75	64/300
C	94	85/300

Table IV. Force Cured and Tested at 50 C

(Applied Torque, 22.6 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	191	105/225
B	35	28/150
C*	26	IF/300

**Adhesive C was tested at 56.5 N·m torque. It would not fail at 22.6 N·m torque.*

IF = Immediate Failure

Fig. V. Force Cured and Tested at 100 C (Test Sample)

(Applied Torque, 11.3 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	IF	IF/30
B	5	2/42
C*	48	3/95

**Adhesive C was tested at 33.9 N·m torque. It would not fail at 11.3 N·m torque.*

IF = Immediate Failure

Table VI. Force Cured (Reheated to 100 C) Cooled and Tested at 23 C (Control Sample)

(Applied Torque, 56.5 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	60	43/196
B	35	26/102
C	59	13/280

Table VII. Force Cured and Tested at 150 C
(Test Sample)

(Applied Torque, 11.3 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	IF	IF/87
B	IF	IF/180
C*	48	3/95

**Adhesive C was tested at 33.9 N·m torque. It would not fail at 11.3 N·m.*

IF = Immediate Failure

Table VIII. Force Cured (Reheated to 150 C) Cooled and
Tested at 23 C (Control Sample)

(Applied Torque, 56.5 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	180	38/300
B	148	6/300
C	60	13/290

Table IX. Cured 1 Month at Ambient Temperature
and Tested at 23 C

(Applied Torque, 56.5 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	44	36/300
B	50	44/300
C	1	IF

IF = Immediate Failure

Table X. Cured 1 Month at Ambient Temperature
and Tested at 50 C

(Applied Torque, 22.6 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	70	30/164
B	137	8/300
C	165	18/175

Table XI. Cured 1 Month at Ambient Temperature
and Tested at 150 C

(Applied Torque, 11.3 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	IF	IF/3
B	137	8/300
C*	300	24.9/33.9 N·m

IF = Immediate Failure

**Adhesive C was tested at 16.9 N·m. The adhesive refused to fail at this torque; failure was induced at the values indicated in "Range."*

Table XII. Cured 2 Months at Ambient Temperature
and Tested at 23 C

(Applied Torque, 56.5 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	21	6/125
B	216	50/300
C	21	7/165

Table XIII. Cured 2 Months at Ambient Temperature
and Tested at 50 C

(Applied Torque, 22.6 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	105	4/205
B	151	14/300
C*	255	71/270

**Adhesive C was tested at 33.9 N·m, it refused to fail at 22.6 N·m.*

Table XIV. Cured 2 Months at Ambient Temperature
and Tested at 150 C

(Applied Torque, 11.3 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	255	80/300
B	3	IF/195
C	See Comment	

IF = Immediate Failure

The blocks spun on the cubes at about 9.0 N·m torque during the adhesive tests. The adhesive appeared to have reverted to a semi-cured state. All five samples exhibited this condition. The 10 other samples, assembled from the same adhesive mixture, appeared normal when tested at their respective temperatures (23 and 50 C) thus ruling out a complete mixture poisoning.

Contamination of the bonding face(s) of one sample or even two of the group of five is conceivable; however, since 15 samples were assembled at the same time from the same adhesive mixture and the samples were randomly assigned to a test group after assembly, it is highly improbable that the faces were contaminated.

The samples were set aside with the adhered faces still mated and allowed to cool for 24 hours. After cooling, the samples were torqued to failure; all were in excess of 67.8 N·m.

Table XV. Cured 4 Months at Ambient Temperature
and Tested at 23 C

(Applied Torque, 56.5 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	40	4/300
B	160	100/300
C	225	105/300

Table XVI. Cured 4 Months at Ambient and Tested at 50 C

(Applied Torque, 22.6 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	186	12/300
B	195	97/300
C	300	1F/300

NOTE: Two of the five cubes of the adhesive C sample were cocked producing low test results.

Table XVII. Cured 4 Months at Ambient Temperature
and Tested at 150 C

(Applied Torque, 11.3 N·m)

<u>Adhesive Type</u>	<u>Median Time (sec)</u>	<u>Range (sec)</u>
A	12	IF/300
B	13	12/300
C*	300	IF/300

**Adhesive C refused to fail at 11.3 N·m. Consequently, these
samples were torqued to 16.9 N·m.*

IF = Immediate Failure

Fig. 3. Force Cured and Tested

*See comment following Table XIV.

Fig. 4. Time Cured and Tested