

STRUCTURAL ADHESIVE WITH COMBINED HIGH STRENGTH AND DUCTILITY FOR MULTIMATERIAL JOINING AND GALVANIC ISOLATION

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ABSTRACT

A newly developed structural adhesive demonstrates a unique combination of high strength (43 ± 2 MPa) and displacement (4.7 ± 1.2 mm) in aluminum lap joint testing. Bulk material characterization of the prototype adhesive reveals its extreme ductility, with nearly 80% shear strain before failure and a 2.5-fold increase in strain energy density as compared to commercial structural adhesives. The prototype adhesive is found to maintain 67 to 82% of its initial strength under extreme environmental conditions, including at high temperatures (71°C), after high humidity (63°C hot water soak, 2 weeks), and after corrosive conditions (B117 salt spray, 1000 hours). The prototype structural adhesive is shown to also generate high strength bonds with multiple substrates, including steel, carbon fiber, and mixed material joints, while also providing galvanic isolation.

INTRODUCTION

Lightweighting efforts have introduced a variety of new materials into vehicle construction, bringing about new challenges for material joining. Structural adhesives offer an attractive option for bonding these new materials with sufficient strength, while also limiting galvanic corrosion between dissimilar materials.^{1,2} As structural adhesives are increasingly implemented into vehicle designs, it is necessary to improve both their strength and ductility to ensure dependable performance.

Typically, in order to gain the required ductility in a structural adhesive joint, it is necessary to sacrifice some of the adhesive strength and environmental resistance. New technologies have recently facilitated the development of a structural

adhesive exhibiting a unique combination of high strength and ductility with environmental resistance. The performance of this adhesive in bonding aluminum and multimaterial joints will be discussed along with its ability to galvanically isolate lightweighting materials for superior corrosion performance.

EXPERIMENTAL

Adhesive lap joints were prepared and tested according to ASTM D1002-10. The 2024-T3 aluminum substrate (AA2024, 1.6 mm thick) was grit blasted with 54 grit aluminum oxide and cleaned with a PPG proprietary alkaline cleaner prior to bonding. The hot-dip galvanized steel (HDG, 0.81 mm thick) was used as received (oiled) and cleaned with a PPG proprietary

alkaline cleaner prior to bonding. The carbon fiber reinforced plastic (CFRP, 0.81 mm thick) was wiped with isopropyl alcohol prior to bonding. The adhesive bondline thickness was controlled through the addition of spherical glass beads (0.25 mm diameter) at 1% by weight. Lap joint specimens were cured using a three-step bake cycle of 90°C for 60 minutes, a 1°C/min ramp to 160°C, and a hold at 160°C for 90 minutes. Specimens were tested at a pull rate of 1.3 mm/min.

Environmental resistance testing of the AA2024 adhesive lap joints was tested through several methods. Performance at high temperature was measured using an Instron fitted with an environmental chamber. Each specimen was equilibrated at 71°C for 15 minutes prior to testing.³ Water resistance was measured by immersing lap shear specimens in a water bath at 63°C for 14 days prior to testing.³ Corrosion resistance was measured by placing lap shear specimens into a B117 salt spray cabinet for 1000 hours prior to testing. This testing was performed with bare AA2024 specimens and with lap shear specimens coated with a Type IV two-component epoxy primer (primed). All samples were tested within 30 minutes of removal from either the hot water bath or salt spray cabinet.

Thick adherend shear test specimens were prepared and tested in accordance with ISO 11003-2. Stepped adherends were machined from 2024-T3 aluminum, which was grit blasted with 54 grit aluminum oxide and cleaned with a PPG proprietary alkaline cleaner prior to bonding. The overlap length (5 mm) and the bondline thickness (0.62 mm) were controlled by curing the specimens within a machined fixture. The fixture was placed in a 170°C oven for 3 hours to cure adhesive. Specimens were loaded onto an Instron and fitted with a D5656 averaging extensometer from Epsilon Technology Corporation. A pull rate of 0.5 mm/min was used.

Multimaterial galvanic assemblies were prepared using cold rolled steel (CRS), 2024-T3 aluminum,

and carbon fiber substrates (all 0.81 mm thick). Substrates were joined using combinations of ¼” stainless steel, electrocoated steel, cadmium coated steel, and nylon fasteners as well as structural adhesive paste. Assemblies were baked according to the cycle above in order to cure the structural adhesive. Assemblies were coated with a Type IV two-component epoxy primer and allowed to cure for one week prior to corrosion testing. Assemblies were qualitatively analyzed after 1000 hours in B117 salt spray.

RESULTS AND DISCUSSION

Single Lap Joint Testing

Figure 1 presents the lap joint performance of the newly developed structural adhesive on AA2024 versus that of a standard commercial structural adhesive. Compared to the commercial structural adhesive, the prototype adhesive exhibits a nearly 50% increase in lap shear strength and more than 100% increase in lap shear displacement before failure. This combination of lap shear strength and displacement is exceptional in comparison to the numerous commercial adhesives measured under the same experimental conditions.⁴

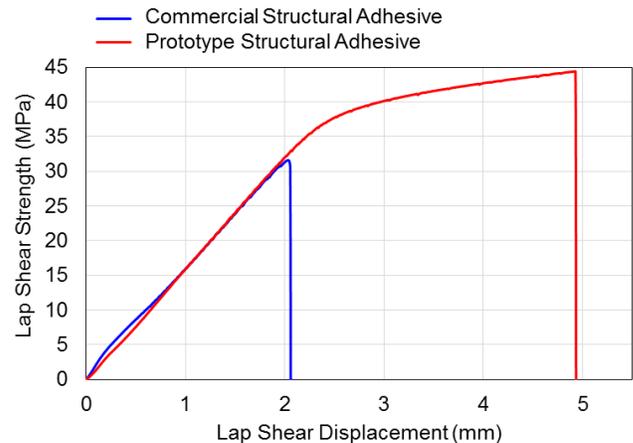


Figure 1: Plot of lap shear strength versus displacement at failure for a commercial structural adhesive as compared to the newly developed prototype structural adhesive.

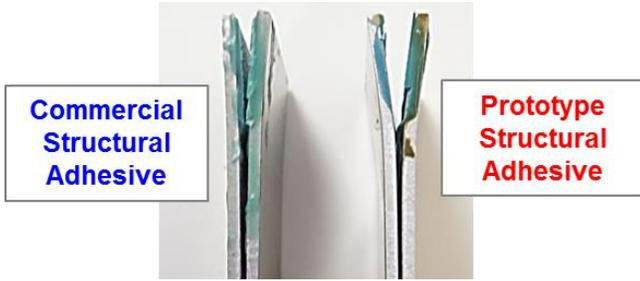


Figure 2: A comparison of the AA2024 substrate deformation after failure of structural adhesive lap joints.

Figure 2 presents images of the lap shear specimens after failure. The lap joints prepared with the prototype structural adhesive clearly undergo a higher degree of substrate deformation during lap joint testing. This raises the question as to whether the large increase in lap shear displacement observed is due yielding within the substrate or within the adhesive.

Bulk Material Properties

To answer the question of substrate yielding versus adhesive yielding, bulk shear properties of the adhesives were measured using a thick adherend shear test. This test method bonds aluminum adherends that are 6 mm thick and additionally employs an extensometer fitted around the adhesive bondline that is used to measure and correct for any slight deformation of the substrate during testing.

Figure 3 presents the corrected shear stress versus shear strain curves for the two structural adhesives. As observed in the simple lap shear test above, the prototype structural adhesive exhibits and increased maximum shear stress combined with a significant increase in shear strain before failure. The shear strain represents the ductility of the adhesive, or its ability to dissipate energy under shear stress. The area under the stress-strain curve represents the strain energy density of the adhesive, which is 2.5-fold greater for the prototype structural adhesive as compared to the commercial one (26.6 MPa versus 10.2 MPa, respectively). These results validate the results of

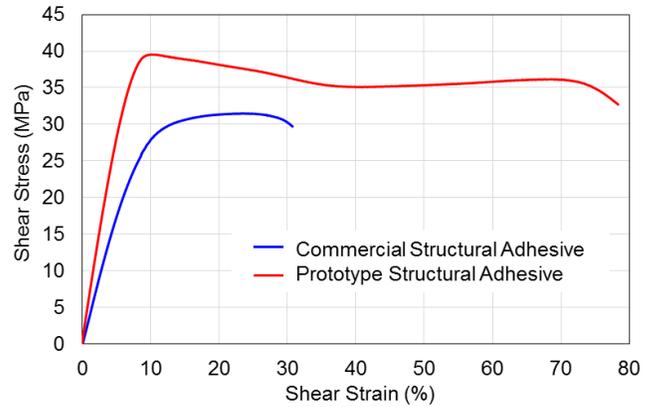


Figure 3: Stress-strain curves from thick adherend shear test (ISO 11003-2).

the simple lap shear test and demonstrate that the observed increase in displacement is indeed due to the increased ductility of the adhesive and its much greater strain energy density.

Environmental Resistance

Typically, extremely ductile adhesives (as observed in Figure 3) exhibit reduced environmental resistance due to greater flexibility and/or lower crosslink density of the polymer matrix. Therefore, the performance of the prototype adhesive was measured under several extreme environmental conditions (Table 1).

Table 1. Retention of adhesive lap joint strength at elevated test temperature and following a variety of environmental conditionings. All tested on grit blasted AA2024 substrate.

Environmental Conditioning	Test Temp.	Lap Shear Strength (MPa)	Strength Retention (%)
None	25°C	43.0 ± 2.5	-
None	71°C	35.4 ± 0.6	82.4
63°C water soak 14 days	25°C	33.5 ± 2.4	77.9
Salt spray (bare) 1000 hours	25°C	29.1 ± 3.2	67.6
Salt spray (primed) 1000 hours	25°C	35.4 ± 2.3	82.3

To investigate the adhesive strength at elevated temperatures, lap joint specimens were equilibrated at 71°C (160°F) before mechanical testing. The prototype adhesive retained 82% of the lap shear strength compared to room temperature testing. This strength retention at elevated temperatures is much higher than that measured for other ductile adhesives tested under the same conditions.⁵

Humidity and corrosion resistance testing was performed by conditioning the lap joint specimens, as specified in Table 1, prior to mechanical testing. In each case, the prototype adhesive retained a significant percentage of its initial lap shear strength. Additionally, in the more realistic case, where the lap joint specimen was coated with primer prior to corrosion testing, the adhesive retained an even greater percentage of its initial strength. Importantly, regardless of the environmental conditioning, the prototype adhesive maintained the same cohesive failure mode with no significant adhesive failure observed.

Adhesion to Various Substrates

All the above tests were performed on 2024-T3 aluminum substrate. However, in current and future vehicle designs a variety of substrates will be employed, including aluminum, steel, and carbon fiber. Table 2 and Figure 4 demonstrate the performance of the prototype adhesive in bonding these substrates.

On oily HDG substrate, the prototype adhesive bond caused significant necking within the steel substrate prior to adhesive failure (Figure 4). Cleaning the HDG prior to bonding slightly increased the lap shear strength, enough to cause the steel substrate to fail before the adhesive bond.

Substrate failure was also observed in the CFRP and mixed material joints. As noted in Table 2, substrate failure invalidates the numerical strength and displacement values, therefore these results should be regarded as qualitative. It is interesting to note that in the pure CFRP joint the failure

Table 2. Performance of prototype adhesive on hot-dip galvanized steel (HDG), carbon fiber reinforced plastic (CFRP), and in mixed material joints.

Substrate	Lap Shear Strength (MPa)	Displacement (mm)	Failure Mode
Oiled HDG	15.6 ± 0.4	15.8 ± 2.9	Adhesive
Cleaned HDG	16.3 ± 0.2	40.8 ± 0.8	Substrate*
CFRP	23.6 ± 1.9	5.2 ± 0.2	Substrate*
Mixed AA2024/CFRP	19.8 ± 1.5	3.1 ± 0.3	Substrate*

*Substrate failure invalidates the lap shear strength and displacement values, therefore these results are qualitative

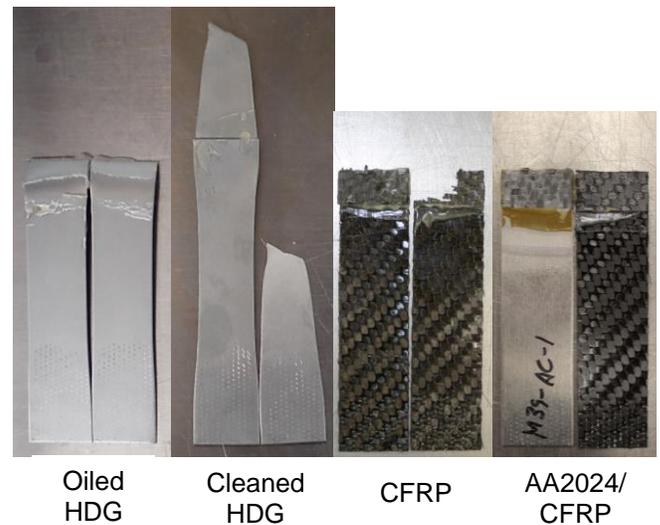


Figure 4: Representative adhesive lap joints after testing. Substrates include oiled and cleaned hot-dip galvanized steel (HDG), carbon fiber reinforced plastic (CFRP), and 2024-T3 aluminum (AA2024).

occurred through the entire substrate, tearing the carbon fibers, while in the mixed material joint, failure occurred via delamination of the bonded CFRP layer. Clearly, further work is needed to quantitatively characterize the performance of the prototype adhesive on these substrates and in mixed material joints. Due to the high strength of the new adhesive, these tests will require thicker metal substrates and stronger carbon fiber composites than those typically used for testing.

Galvanic Isolation

It is well known that adhesives can provide galvanic isolation in mixed material systems.² In order to investigate this ability for the prototype structural adhesive, it was used to bond multimaterial galvanic assemblies. These assemblies are modified from those used by researchers at NAVAIR and AMCOM,⁶ and allow for simultaneous investigation of multiple material interfaces and fastener types, including adhesives.

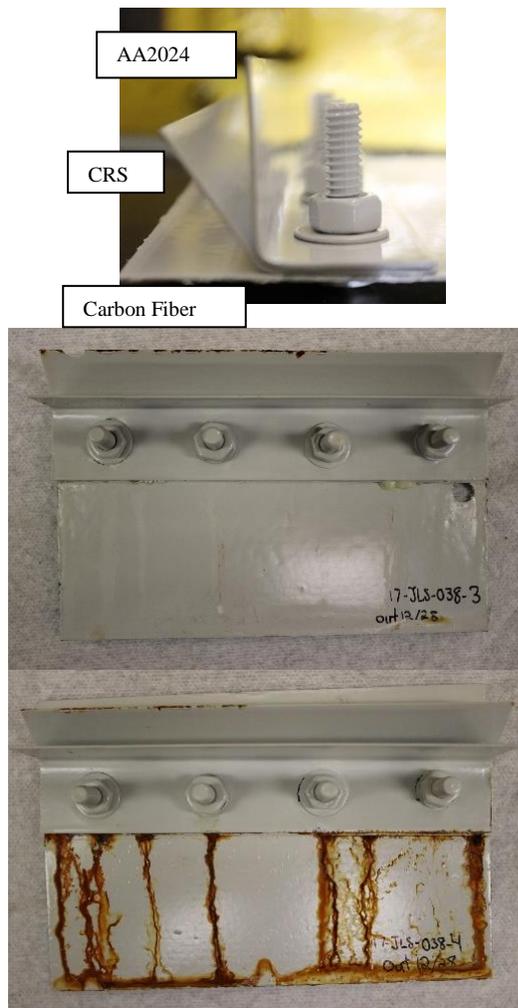


Figure 5: Setup (top) of multimaterial galvanic assemblies implementing 2024-T3 aluminum (AA20204), cold rolled steel (CRS), and carbon fiber. Images show galvanic assemblies joined with (middle) and without (bottom) structural adhesive after 1000 hours in B117 salt spray.

It is immediately apparent from Figure 5 that the use of the prototype adhesive within these multimaterial assemblies significantly reduces the extent of corrosion, as may be expected. Interestingly, the adhesive appears to provide galvanic isolation even though the assemblies are connected through with metallic fasteners. Figure 6 shows the extensive corrosion of the AA2024 and CRS when not galvanically isolated using adhesive. The assembly bonded with adhesive could not be separated for further investigation.



Figure 6: Galvanic assembly without adhesive disassembled after 1000 hours B117 salt spray to investigate substrate corrosion.

CONCLUSIONS

The collective mechanical and corrosion testing of the prototype structural adhesive demonstrate the unique combination of high strength, high ductility, and high environmental resistance. Preliminary qualitative results in bonding and protecting multimaterial systems are promising, and future work will focus on additional quantitative assessments of the prototype adhesive performance in these areas.

ACKNOWLEDGMENTS

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REFERENCES

- [1] A.J. Kinloch, Adhesion and Adhesives: Science and Technology, 1987, Springer Science & Business, London.
- [2] R.D. Adams, Adhesive Bonding - Science, Technology and Applications, 2005, Woodhead Publishing, Cambridge.
- [3] R.E. Jensen, et al. Test Method, Group Assignment, and Categorization Guide for High-Loading-Rate Applications – Preparation and Testing of Single Lap Joints, 2016, ARL-SR-0356.
- [4] R.E. Jensen, et al. Adhesives: Test Method, Group Assignment, and Categorization Guide for High-Loading-Rate Applications – History and Rationale, 2017, ARL-SR-0371.
- [5] R.E. Jensen, et al. Single-Lap-Joint Screening of Hysol EA 9309NA Epoxy Adhesive, 2017, ARL-TR-8011.
- [6] J. Russell, Comprehensive Evaluation and Transition of Non-Chromate Primers within DoD, ASETSDefense 2012: Workshop on Sustainable Surface Engineering for Aerospace and Defense.