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Testing Military-Grade Adhesive in Extreme Loading Conditions

by Daniel Deschepper, David Gray, Paul Moy, Timothy Walter, Robert Jensen, Marvin Pollum, Edward Hellerman, Joseph Kriley, Masa Nakajima, and Brian Rearick

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<p><u>Military performance requirements for adhesives have been traditionally derived to fulfill niche defense needs in harsh operational environments</u> with little consideration for dual-use commercial potential. The drawback with this approach is dwindling defense acquisition access to leading-edge commercially sustainable products, as the market drivers for purely non-defense applications are significantly larger. MIL-PRF-32662, <i>Adhesive, High-Loading Rate, for Structural and Armor Application</i>, is based on a decade of rigorous research efforts to statistically correlate the complex ballistic response of adhesively bonded armor assemblies to universally translatable and commercially relevant mechanical properties. These military performance thresholds were deliberately defined at just beyond state-of-the-art based on a statistical survey of commercially available adhesives. The end results are performance criterion for MIL-PRF-32662 that are difficult, but not impossible, to meet with the intention of attracting commercial interest. The objective of this experimental work is to quantify adhesive performance defined by “harsh military operational environments” against a suitable non-military harsh use condition. The result of this study shows a 32% increase in static load retention strength of an adhesive formulated to meet MIL-PRF-32662 Group 1 requirements when tested in the tensile butt joint geometry specified by Guinness World Records. These results offer further encouragement to explore the potential of military performance specifications as agents of change in leading technical advances in dual-use market sectors.</p>									
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Executive Summary

The strength of an adhesive formulated to meet bonding requirements for Army ground vehicles was tested using the tensile butt joint configuration specified by Guinness World Records.¹ This protocol was used to quantitatively compare the load bearing capacity of an adhesive meeting military requirements against a commercial benchmark of extreme performance. Results show that an adhesive meeting MIL-PRF-32662² Group 1 requirements readily sustains 22,680 kg (50,000 lb) of static loading for 1 h, for a 32% increase against the current world record. The results offer a potential quantitative measure for military-grade adhesive and show the utility of leveraging anticipatory military performance standards to lead additional material advancements.

¹ Heaviest weight lifted with glue (non-commercially available), Specific Guidelines Pack, Guinness World Records; 2023 [accessed 2024 May 23]. <https://www.guinnessworldrecords.com/world-records/heaviest-weight-lifted-with-glue>

² MIL-PRF-32662. Adhesive, high-loading rate, for structural and armor applications. Department of Defense (US); 2020 Aug 13.

1. Introduction

The term “military-grade” can have a variety of meanings that are perspective dependent. In 2014, Ford Motor Company emphasized the term heavily in advertising campaigns to garner consumer acceptance for the transition from steel to aluminum in the body of their flagship F150 model.¹ As cited by Ford, “Engineers selected these high-strength, military-grade aluminum alloys because of the metals’ unique ability to withstand tough customer demands.” From this point-of-view, military-grade implies superior performance. However, the bureaucratic and logistical barriers required for certification to military-grade acceptance levels per DOD performance requirements can also be perceived as impediments to innovation and the transition of fundamental science into tangible product.^{2,3} This is in-part due to the legacy age of many DOD performance standards dating to the 1950s and 1960s when the US military peaked in technology market share and was responsible for approximately two-thirds of domestic research and development (R&D) and one-third of global R&D.⁴ In 2023 the commercial private sector provides the overriding funding stream for technology development for primarily non-military applications.⁵ Since the “golden age” of DOD-derived performance specifications the interactive roles between requirements and innovation are now understood to be dependent on their timings to product life cycle, which is typically ignored universally across the materials domain.⁶⁻⁸ Traditional DOD adhesive specifications are measures of late life cycle quality assurance for low-risk bonding applications with long-term historical usage and well-understood design allowables.⁹ Military-grade certification for commercial industry can also imply a high degree of qualification effort for a limited defense market with marginal dual-use consumer return on investment. This is an unfavorable position for DOD, as commercial industry requires a sustainable business model to produce defense-relevant materials.

MIL-PRF-32662¹⁰ represents a forward-looking and “anticipatory” specification to deliberately coincide with the timing of emergent, and potentially lucrative, adhesive technologies by encouraging, high-risk/high-payoff commercial product innovation. The hypothesis of this approach is that defining DOD technology drivers in terms of industrially relevant performance metrics, with high non-DOD profit potential, will decrease transition time and increase DOD access to sustainable commercial products with leading state-of-the-art properties. Prior to the release of MIL-PRF-32662 in 2020, no adhesive, with bonded structural armor as the primary focus, had bridged the “valley of death” from basic research to qualified product. The technical challenge posited by Army ground vehicle applications is the need for concurrent high-strength and high-fracture toughness,

which is a difficult material property trade-space for adhesives. PPG Industries (PPG) was the first commercial adhesive manufacturer to meet MIL-PRF-32662¹⁰ Group 1 requirements with the development of their PR-2930 one-component epoxy adhesive. This adhesive targets the Army ground vehicle requirements put forth in MIL-PRF-32662 but is also formulated for compatibility with commercial automotive assembly line bonding processes. The next question to be answered is can the performance value of military-grade be quantified using a benchmark of an extreme testing condition referenced by the commercial sector?

The adhesive bonding joint configuration used for this study was a tensile butt joint with a 40 cm² surface area for bonding, which is specified by Guinness World Records.¹¹ The most recent record was set in 2019 to suspend a 17,200 kg (37,919 lb) construction vehicle at a height of 1 m for 1 h.¹¹ In this work, the world record hoist and static hold conditions were mimicked using a laboratory loading frame. Results show that an adhesive meeting MIL-PRF-32662 Group 1 requirements readily sustains 22,680 kg (50,000 lb) of static loading for 1 h, for a 32% increase against the current world record benchmark. The results offer a potential quantitative measure for military-grade and show the utility of leveraging anticipatory military performance standards to lead material advancements.

2. Protocol

2.1 Tensile Butt Joint Substrates

Adhesive bonding substrates were machined from annealed 4340 steel and cold-formed 7075 T651 aluminum. A tensile butt joint radius of 3.5 cm is relatively large for laboratory-scale testing and requires careful machining considerations to safely meet the gripping requirements. The cylindrical samples used for this study were tapered from 90 mm at the grip region to 70 mm at the bonding surface. The larger diameter of the grip region was required to accommodate internal female threads needed for coupling to a steel male shoulder type machinery eye bolt (63.5-mm diameter × 6.35-mm thread pitch). This bolt diameter is load rated for 289 kN (65,000 lbf), which exceeds the anticipated loading range of bonded butt joint assembly. A modified version of this bolt without the eyelet was used to enable gripping by the loading frame. Detailed dimensions of the tensile-butt joint substrates are provided as supplemental files:

(<https://materialsdata.nist.gov/handle/11256/1004>).

2.2 Surface Preparation

Surface preparation is a critical factor in providing maximum strength and durability of an adhesively bonded joint.¹² The following surface preparation protocols were developed by PPG for the steel and aluminum butt joint surfaces prior to application of the adhesive.

2.2.1 4340 Steel Surface Preparation

- 1) Solvent wipe with methyl ethyl ketone and acetone.
- 2) Immerse joining surface in room temperature deionized water for 2 min.
- 3) Immerse joining surface in ChemKleen 490MX alkaline cleaning solution (7.5 g/L concentration) for 2 min at 49 °C.
- 4) Immerse joining surface in room temperature deionized water for 2 min.
- 5) Dry at 70 °C for 30 min.
- 6) Note: Exploratory 4340 steel samples used to verify the loading frame setup were also prepared at the US Combat Capabilities Development Command (DEVCOM) Army Research Laboratory (ARL) by cleaning with 80-grit aluminum oxide blast media followed by an acetone solvent wipe. This surface pretreatment process is less labor intensive and will yield comparable dry strength performance. This pretreatment process is also known to be grossly inadequate for traditional epoxy-based adhesives undergoing long-term humidity exposure, which was not a concern during this study.

2.2.2 7075 T651 CF Aluminum Surface Preparation

- 1) Solvent wipe with methyl ethyl ketone and acetone.
- 2) Immerse joining surface in room temperature deionized water for 2 min.
- 3) Immerse joining surface in ChemKleen 490MX alkaline cleaning solution (7.5 g/L concentration) for 2 min at 49 °C.
- 4) Immerse joining surface in room temperature deionized water for 2 min.
- 5) Dry at 70 °C for 30 min.

2.3 Adhesive Application and Bonding

The adhesive, PR-2930 Low Viscosity Low Cure (PR-2930-LVLC), is a one-component, heat-cured, epoxy-based adhesive paste. PR-2930-LVLC is an

experimental variant of production PR-2930 that cures at lower temperatures. The weight of each substrate was collected prior to bonding. Three grams of adhesive paste were spread on one side of each butt joint substrate pair. The pair of substrates were then joined within an aligning and compressing fixture. The compression screw was tightened to a torque of 54 Nm. Adhesive squeeze out during the joining process was carefully collected and weighed approximately 1.9 g leaving a total of 1.1 g of adhesive within the butt joint bond line. The adhesive is designed to maintain a bond line thickness of approximately 250 μm through the inclusion of solid glass spacing spheres included as a component of the adhesive formulation package. Historical experimental validation of bond line thickness from single-lap joints previously fabricated by ARL using similar conditions shows this value at $181 \pm 21 \mu\text{m}$.

2.4 Curing Conditions

Thermocouples were fixed to the side of the metal butt joint substrates near the adhesive bond line and the entire fixtured assembly was placed in an oven, as shown in Fig. 1. The alignment fixture is a larger-scaled version taken from ISO 6922.¹³ The oven was then set to 140 °C and held isothermally for 3 h before turning the heat off and allowing the joints to slow-cool to room temperature overnight. The adhesive is formulated to fully cure at 140 °C in 1 h. The thermocouple measurements confirmed that 3 h at cure temperature was enough time to compensate for heating the thermal mass of the butt joint substrates and alignment fixture. The slow-cool minimizes residual stress in the joints. Additionally, a helical spring was used between the compression screw of the tooling fixture and the butt joints to compensate for mismatches in thermal expansion during the cure cycle. Joints were weighed after cure and the individual substrate weights were subtracted from the total weight to confirm approximately 1 g of cured adhesive joining each butt joint.

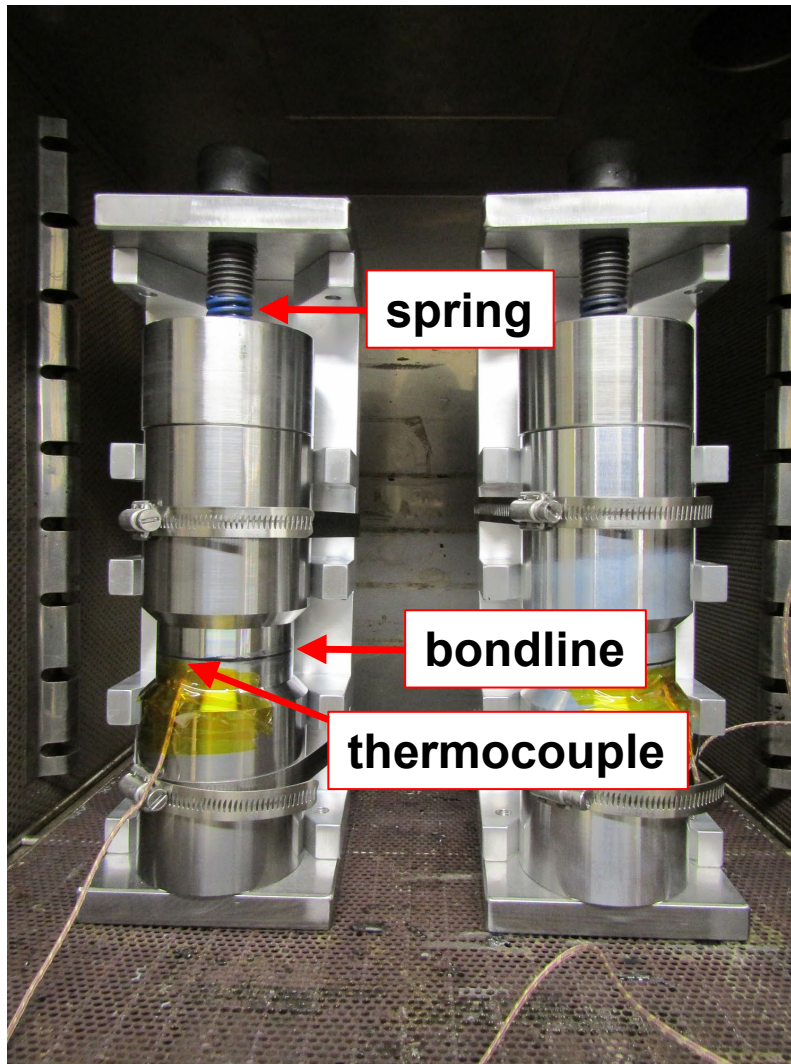


Fig. 1 Bonded tensile butt joints clamped in alignment fixtures undergoing oven cure at 140 °C. A thermocouple is placed at the bond line and a spring is used compensate for mismatches in thermal expansion.

2.5 Mechanical Testing

The peak strength and isostatic loading capacity of the adhesively bonded tensile butt joints was measured using a Tinius Olsen Model 1000Sl hydraulic test frame (Fig. 2) rated for a maximum load of 1000 kN (200,000 lbf). The system uses a pressure transducer to measure force and received a certified calibration during the experiments. The system is equipped with in-head pocket grips for attaching to cylindrical samples, as shown in Fig. 3. The loading rates used during testing were 148 N/s, 370 N/s, and 22.2 kN/s. The technical data sheet and calibration certificate for the testing frame are provided in the supplemental files: (<https://materialsdata.nist.gov/handle/11256/1004>).

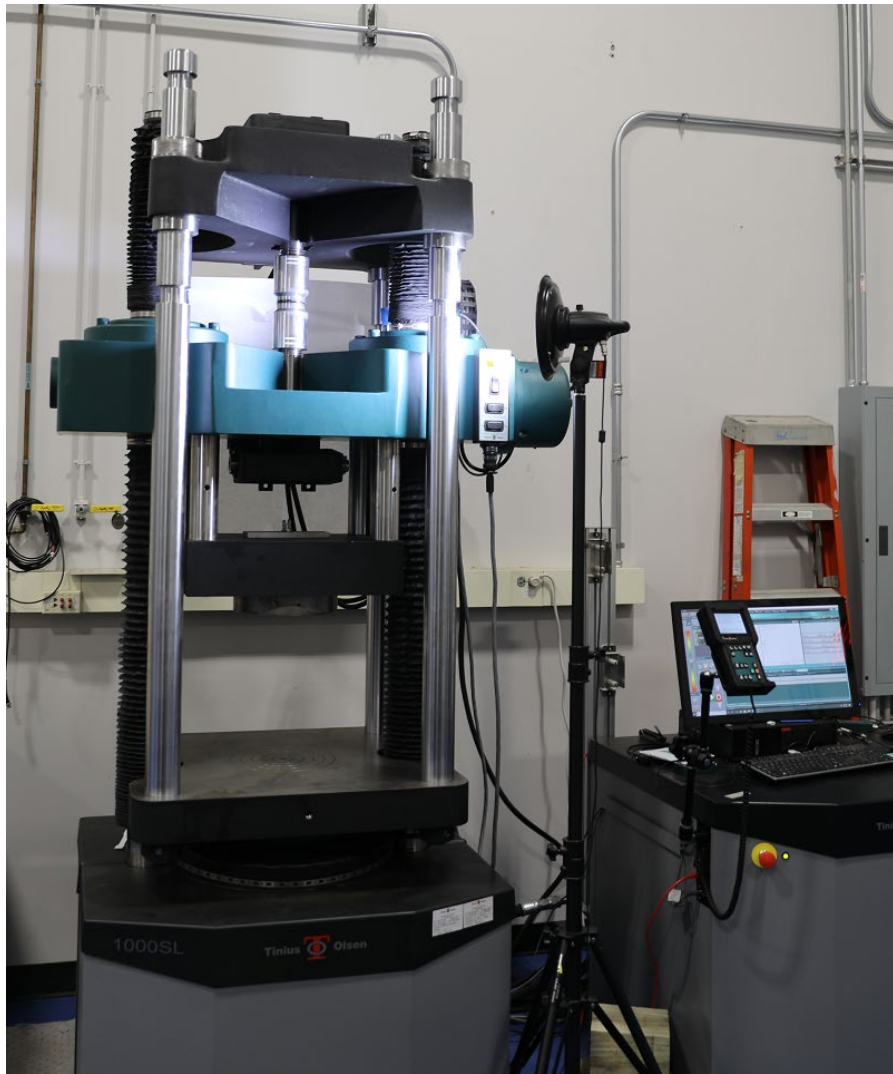


Fig. 2 Tinius Olsen Model 1000SI hydraulic test frame

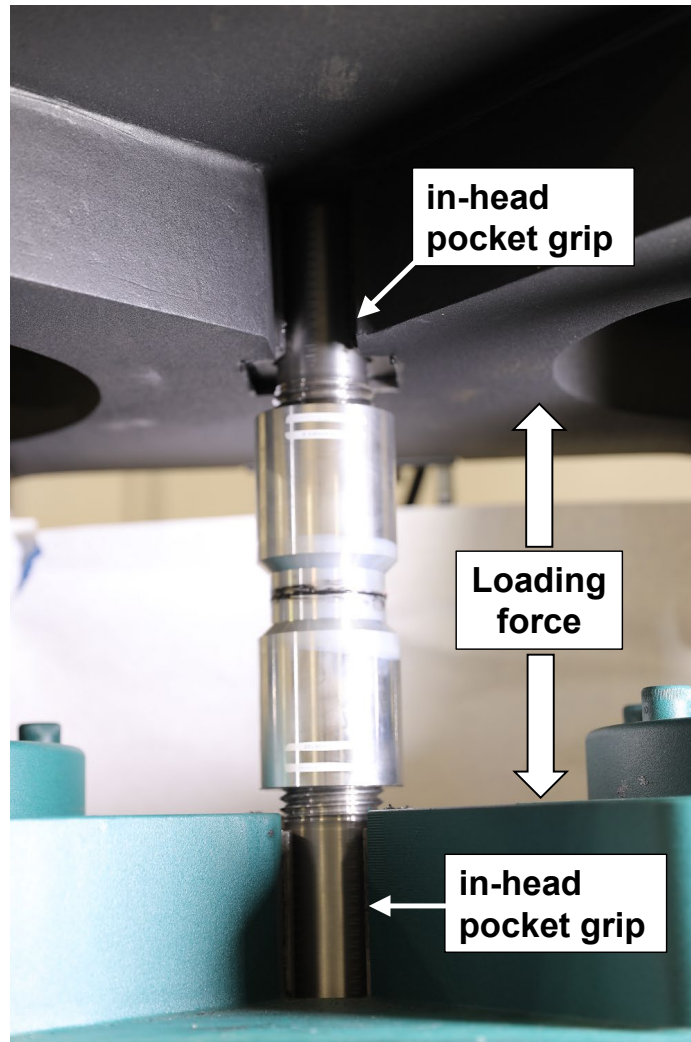


Fig. 3 In-head pocket grips used by the Tinius Olsen Model 1000SI hydraulic test frame

3. Representative Results and Discussion

Figure 4 shows representative results of the testing performed on 4340 steel and 7075 T651 aluminum tensile butt joints under different loading rates. The test conditions and results are summarized in the bullets that follow.

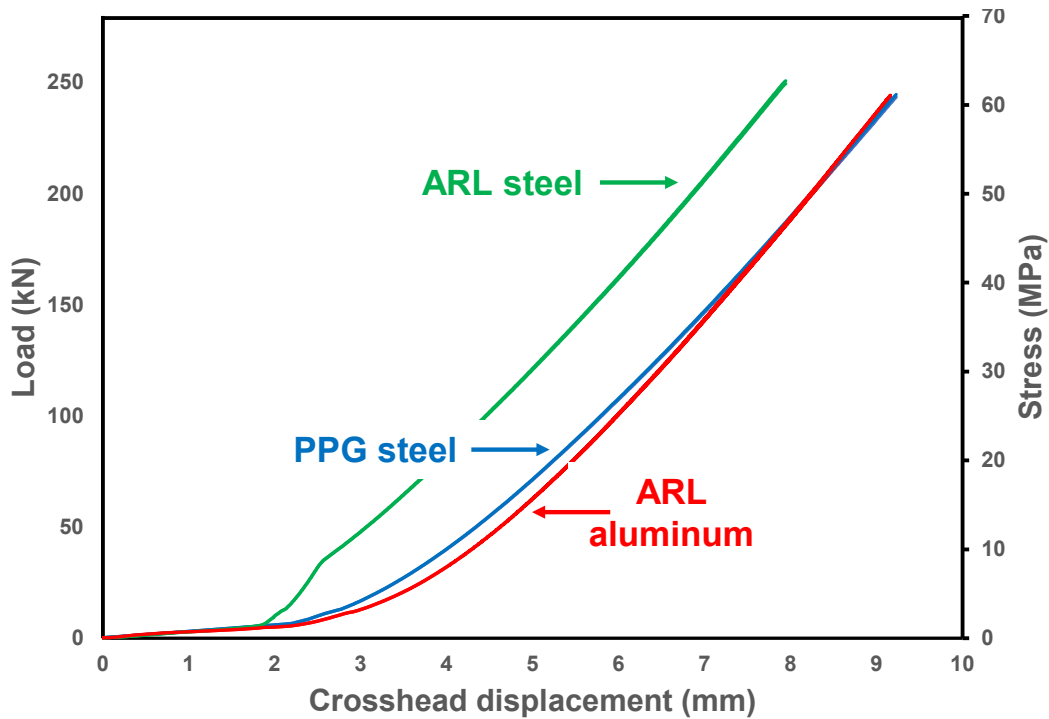


Fig. 4 Load vs. displacement response for bonded steel and aluminum tensile butt joints loaded at 148 N/s (2000 lbf/min)

- Loading rates: ARL aluminum = 148 N/s (2000 lbf/min), ARL steel = 370 N/s (5000 lbf/min), and PPG steel = 370 N/s (5000 lbf/min)
- Maximum strengths: ARL aluminum = 240.05 kN, 61.08 MPa; ARL steel = 249.55 kN, 62.71 MPa; and PPG steel = 243.45 kN, 61.18 MPa (average = 61.7 ± 0.9 MPa)
- Crosshead displacement is not calibrated for machine compliance or free play in the load-string between the internal machined threads in the joint and the external threads of the grip adapter.
 - Measurements taken using strain gauges are discussed later in this section.
- Results are very consistent and exceed the 43.77-MPa commercial world record benchmark for strength for this butt joint geometry.
- The failure surfaces are shown in Fig. 5.
- Mode-of-failure for the grit-blasted samples is cohesive. Mixed-mode failure (adhesive/cohesive) was visually observed for the chemically treated samples.

- Failure initiated at the edge of the sample due to mixed-mode loading conditions, but the overall strength of the joint was still comparable to samples that failed entirely cohesively.

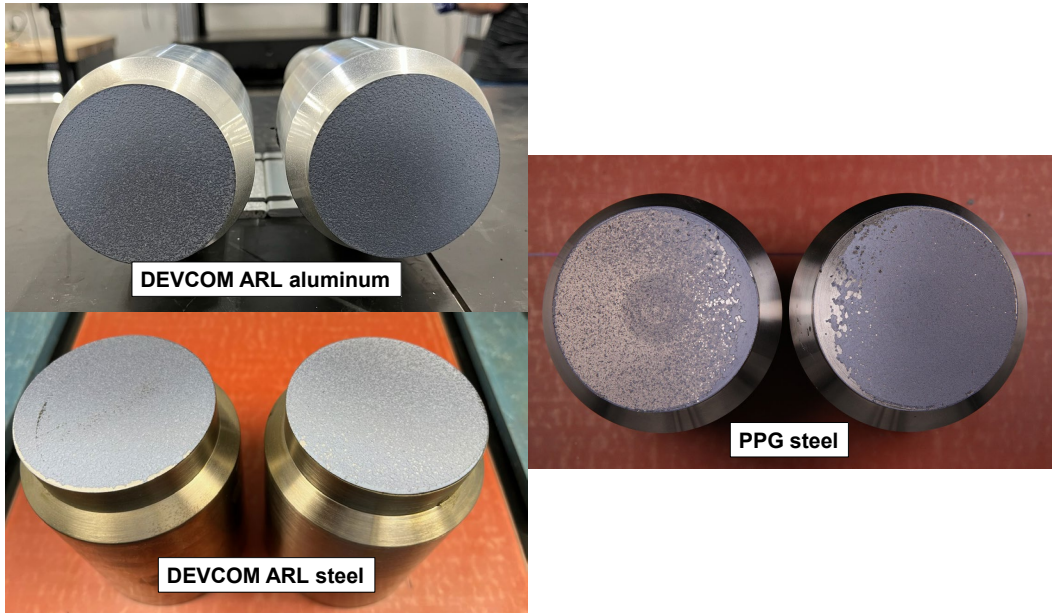


Fig. 5 Failure surfaces of the bonded steel and aluminum tensile butt joints referenced in Fig. 4

The 1-h isostatic load testing confirms that the MIL-PRF-32662¹⁰ Group 1 adhesive has improved strength and resistance to creep when compared to the benchmark commercial record loading of 17,200 kg. This can be seen in Fig. 6, where the MIL-PRF-32662 Group 1 samples maintain a higher load throughout the test duration than the reported commercial world record benchmark.

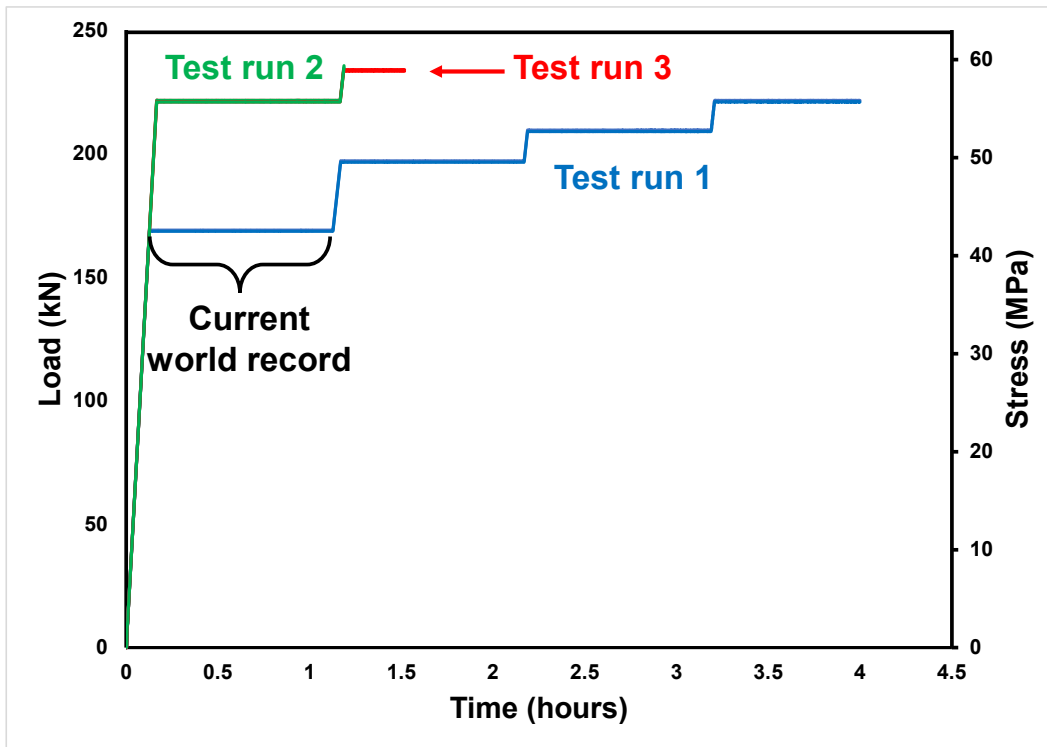


Fig. 6 Isostatic loading response with initial ramp rate at 370 N/s (5000 lbf/min)

Test conditions and results:

- Loading rates: all samples, initial = 370 N/s (5000 lbf/min), between hold segments = 185 N/s (2500 lbf/min)
- 1-h isostatic hold commercial benchmark for this butt joint geometry is at 42.5 MPa (170 kN, 17,200 kg, 37,919 lb).
- Test run 1
 - 1-h hold segment 1: 42.5 MPa (170 kN, 17,200 kg, 37,919 lb)
 - 1-h hold segment 2: 49.5 MPa (197 kN, 20,100 kg, 44,300 lb)
 - 1-h hold segment 3: 52.6 MPa (210 kN, 21,400 kg, 47,100 lb)
 - 1-h hold segment 4: 55.7 MPa (222 kN, 22,600 kg, 49,800 lb), failed 47.5 min after segment start
- Test run 2
 - 1-h hold segment 1: 55.7 MPa (222 kN, 22,600 kg, 49,800 lb)
 - 1-h hold segment 2: 58.8 MPa (234 kN, 22,600 kg, 52,600 lb), failed 20 min after segment start

- Test run 3
 - 1-h hold segment 1: 55.7 MPa (222 kN, 22,600 kg, 49,800 lb)
 - Loaded at 370 N/s (5000 lbf/min) to failure: 59.2 MPa (236 kN, 23,700 kg, 52,960 lb)
- Mixed-mode failure (adhesive/cohesive) was visually observed for the chemically treated samples.
- Similar mode-of-failure following the isostatic holds compared to the constant loading rate response.

The current Guinness World Records lift was performed using a construction crane, which likely loaded much faster than 370 N/s used to ramp to the previous 1-h isostatic holds. The Tinius Olsen Model 1000SI hydraulic test frame used for this study is capable of loading at 22.5 kN/s (5000 lbf/s). The hydraulic test frame uses a proportional integral derivative (PID) controller. PID controllers require tuning to maintain accuracy at the edge boundaries of the instrument's performance envelope.¹⁴ The PID controls of the hydraulic test frame were adjusted to both minimize the time required to equilibrate at the accelerated loading rate and to minimize load overshoot without oscillating during the static hold portions of the loading cycle. Figure 7 shows the loading rate versus time plots taken at 22.2 kN/min and 22.2 kN/s and experimental conditions and results follow.

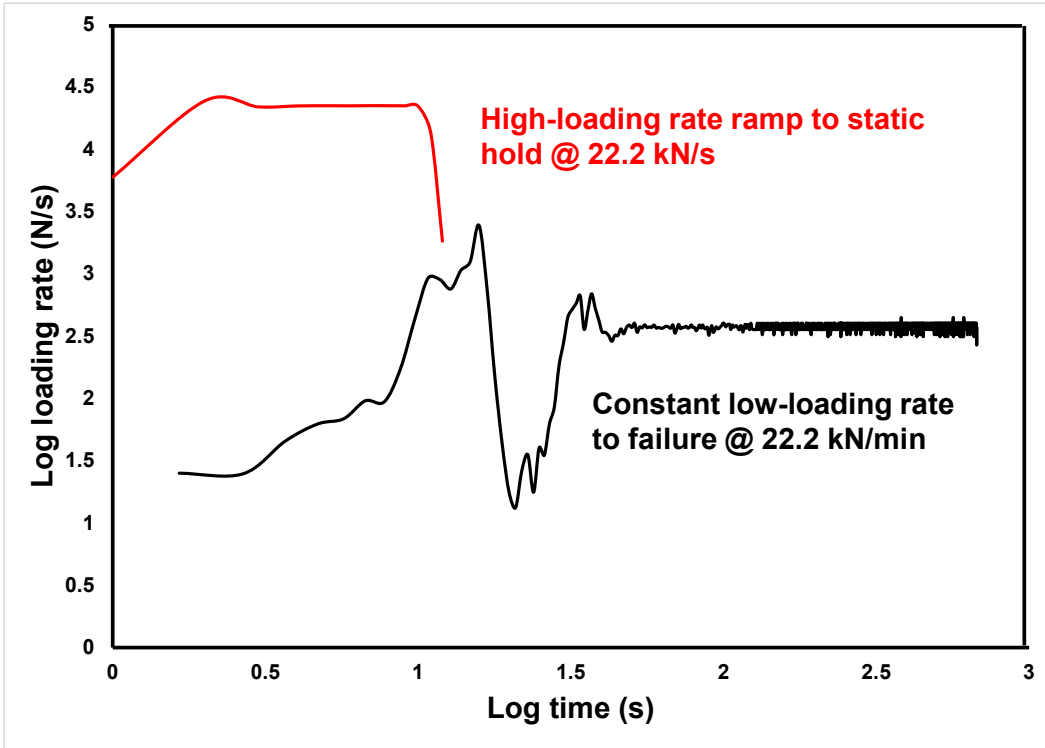


Fig. 7 Loading rate response with standard and adjusted PID settings to avoid significant load-overshoot at high rates

- The curve obtained at 22.2 kN/min was recorded during the constant loading of the ARL steel sample shown in Fig. 4. PID gains for the test frame were in the manufacturer’s standard settings of $P = 20$, $I = 2$, $D = 50$. With these settings the loading frame requires 45 s to reach a stable loading rate. This loading rate stabilization time is far too slow to mimic the world record conditions, which necessitated numerous iterative PID gain adjustments.
- Initial PID adjustments were performed by loading large diameter aluminum rod-stock at 22.2 kN/s (results not shown).
- Final PID adjustments were performed using a single steel bonded butt joint at 22.2 kN/s, as shown in Fig. 8. The sample was subjected to three loading cycles as follows:
 - Run 1
 - 22.2 kN/s to 111 kN (25,000 lbf), isostatic hold for 30 s, test was stopped and unloaded.
 - Run 2

- 22.2 kN/s to 222 kN (50,000 lbf) isostatic hold for 3 min, ramp to 244 kN (55,000 lbf), isostatic hold for 3 min, test was stopped and unloaded.
 - The PID controller exceeded the set-point load of 244 kN by 25 kN (5430 lbf).
 - The high loading rate PID gains were finalized at $P = 36$, $I = 11$, $D = 0.1$.
- Run 3
 - 22.2 kN/s to 222 kN (50,000 lbf) isostatic hold for 60 min, ramp to 267 kN (60,000 lbf), isostatic hold for approximately 4.75 min when the sample failed.
 - The loading rate versus time curve for this run is shown in Fig. 8. With these adjusted PID settings, the loading frame requires only 3 s to reach a stable loading rate with minimal load overshoot or load oscillation during the isostatic portion of the cycle.
- After adjusting the PID controller gains the hydraulic testing frame was more closely able to reproduce the expected world record lift conditions.
- A single bonded steel butt joint was used for the final PID adjustment iterations. The PR-2930-LVLC adhesive was loaded at high rates three times and still maintained an isostatic hold of 222 kN for 1 h, which exceeds the current world record by 32%.
- Failure was mixed-mode, initiated at an edge of the sample, as shown in Fig. 9.

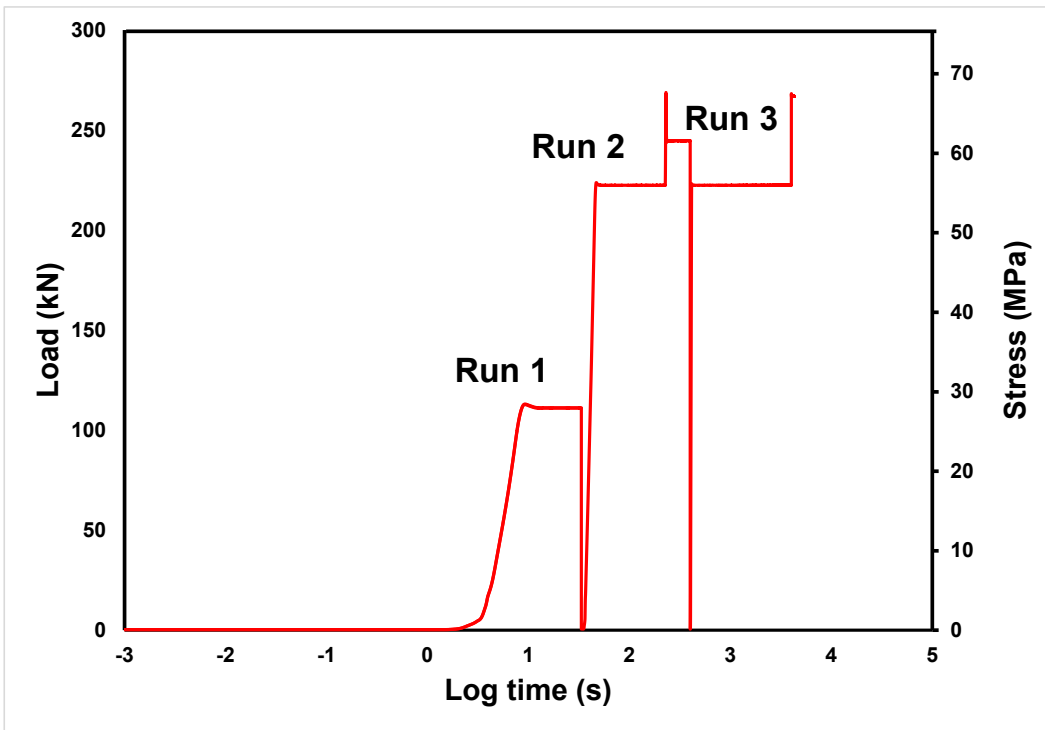


Fig. 8 Consecutive loading response curves for a single bonded steel butt joint used for iterative PID control adjustment

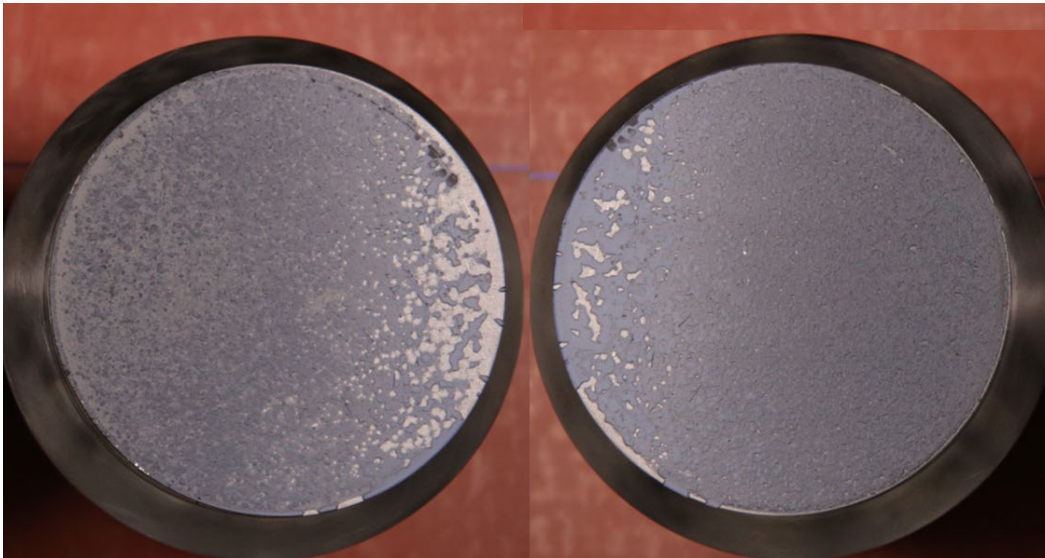


Fig. 9 Failure surface of the bonded steel butt joint used for PID control adjustment. Note: Image has been edited to orient the corresponding failure surface locations.

In the previous tests, displacement measurements were obtained directly from the crosshead, failing to consider machine compliance and the free play within the load-string between the internal machined threads at the joint and the external threads of

the grip adapter. Strain gauges, on the other hand, rectify these shortcomings by enabling precise measurement at the bond line itself. Figure 10 shows a steel bonded butt joint configured with strain gauges. Four Vishay Micro-Measurements CEA-06-500UW-350 strain gauges were affixed in 90° increments using a cyanoacrylate bonding adhesive. The gauge length is 12.7 mm.

Figure 10 also shows the inner machined threads and outer threaded rod inserts used to couple the butt joint to the hydraulic loading frame. The raw crosshead displacement is not calibrated for machine compliance or free play in the load-string between the internal machined threads in the joint and the external threads of the grip adapter. Most of the apparent displacement in the loading responses shown in Fig. 4 is due to these machined sample and fixture threads.

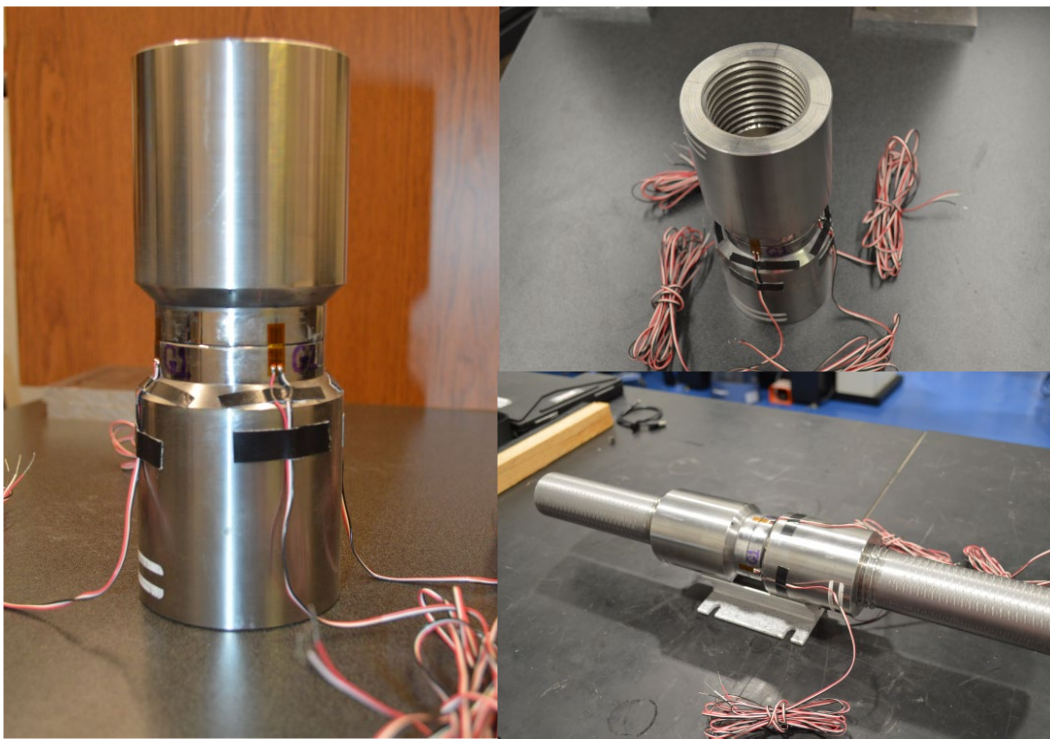


Fig. 10 Strain gauge configuration showing placement at the bond line in 90° intervals

Figure 11 shows the load versus time response for the strain-gauged sample. Experiment conditions:

- The high-loading rate PID gains were used at $P = 36$, $I = 11$, $D = 0.1$.
- 12-s ramp to 222 kN (50,000 lbf) at 22.2 kN/s to an isostatic hold for 60 min, 4.5-s ramp from 222 kN to failure at 312.3 kN (70,217.3 lbf, 78.5 MPa).

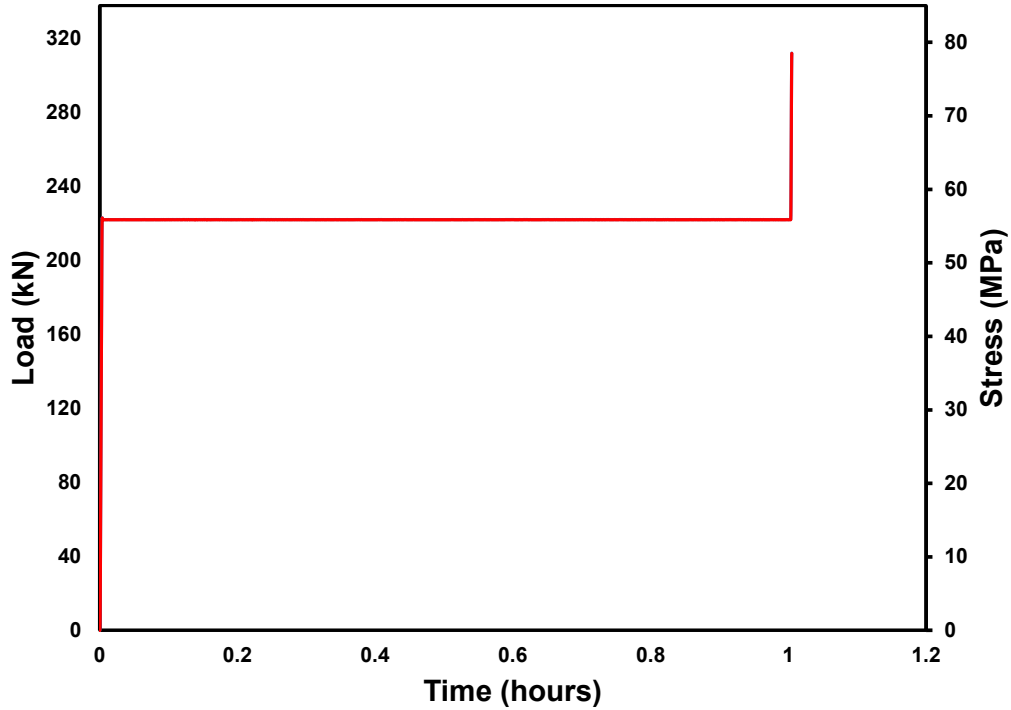


Fig. 11 Global loading response for the strain-gauged sample at 22.2 kN/s

Figure 12 shows the stress versus strain curves derived from the crosshead displacement and measured directly using the strain gauges. The strain measurements are uncorrected and based on the total length of the sample at 226 mm for the crosshead plot and 12.7 mm for the strain gauge plots.

Figure 12 also verifies that the machine compliance and free play in the load-string between the internal machined threads in the joint and the external threads of the grip adapter is substantial.

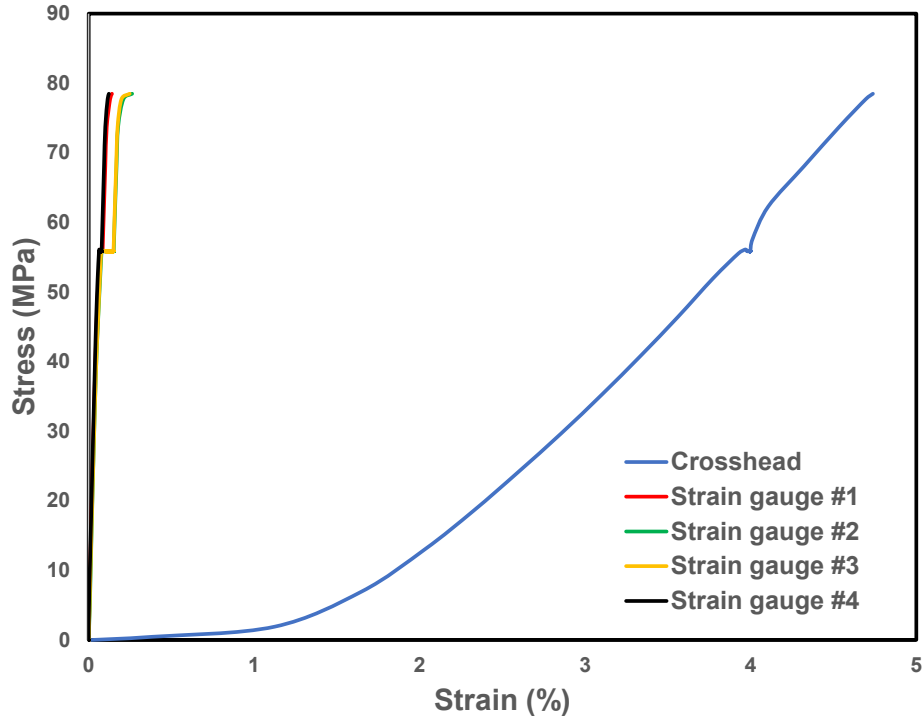


Fig. 12 Stress vs. strain curves derived from the crosshead displacement and measured directly using the strain gauges

Figure 13 shows the stress versus apparent strain at the adhesive bond line. The apparent strain at the bond line is derived from the following equations.¹⁵

$$E' = \frac{\sigma}{\varepsilon} = \frac{F}{A} \frac{t_a}{d - d_s}, \text{ where } d_s = \frac{F}{A E_s}$$

Hooke's law is used to determine the apparent modulus (E') at the bond line by considering the adhesive thickness (t_a). The displacement of the steel (d_s) is subtracted from the displacement of the strain gauge (d). The modulus of the steel (E_s) is 200 GPa.

The apparent modulus of the adhesive, based on linear fits of the stress–strain plots to 30 MPa are E' (GPa) strain gauge 1 = 2.94 GPa, strain gauge 2 = 2.21 GPa, strain gauge 3 = 2.29 GPa, strain gauge 4 = 2.95 GPa, average modulus = 2.60 ± 0.40 GPa. This agrees well with a commonly cited model value of structural adhesive modulus at 2.50 GPa.¹⁶

The decreased modulus recorded by strain gauges 2 and 3 indicates that this was an area of increased compliance in the bond line. This is likely attributed to an artifact of the longitudinal alignment during testing. Subsequent test runs alluded to the extreme sensitivity of the butt joint to localized edge stress concentrations due to slight bond line thickness variations. Interfacial corner failure due to mixed-mode loading has been predicted using finite element analysis.¹⁷

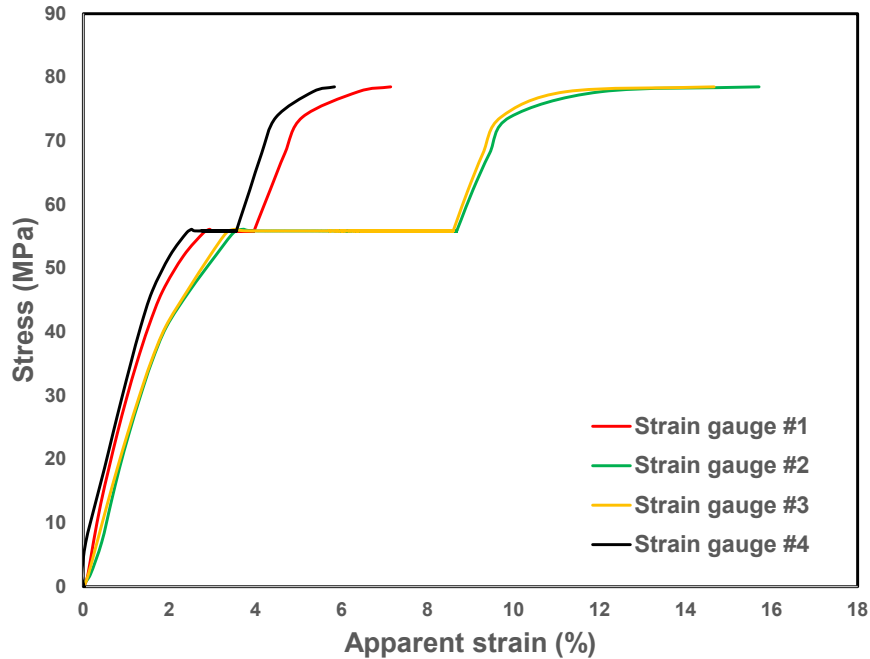


Fig. 13 Stress vs. the apparent strain of the butt joint at the adhesive bond line

Figure 14 shows the creep compliance curves of the butt joint at the adhesive bond line during isostatic loading:

- Creep compliance (J) is measured at constant stress by observing the resultant strain as a function of time.
- The creep compliance curves in Fig. 14 were derived from the isostatic hold at 222 kN (50,000 lbf) for 60 min.
- The creep compliance measured at strain gauges 2 and 3 is significantly greater than 1 and 4. This indicates that failure initiated between gauges 2 and 3.
- The creep compliance at strain gauge 2 and 3 is representative of typical epoxy adhesives at approximately 0.001 MPa^{-1} at short time durations and gradually increasing to 0.0015 MPa^{-1} as the test progressed.¹⁸
- The creep compliance at strain gauge 1 and 4 is lower than typical epoxy adhesives at approximately 0.0005 MPa^{-1} at short time durations, and it remained fairly constant as the test progressed. The creep compliance for Araldite LY564 is approximately 0.0009 MPa^{-1} measured at similar levels of stress and time duration.¹⁹
- Figure 15 confirms failure initiated between strain gauges 2 and 3.

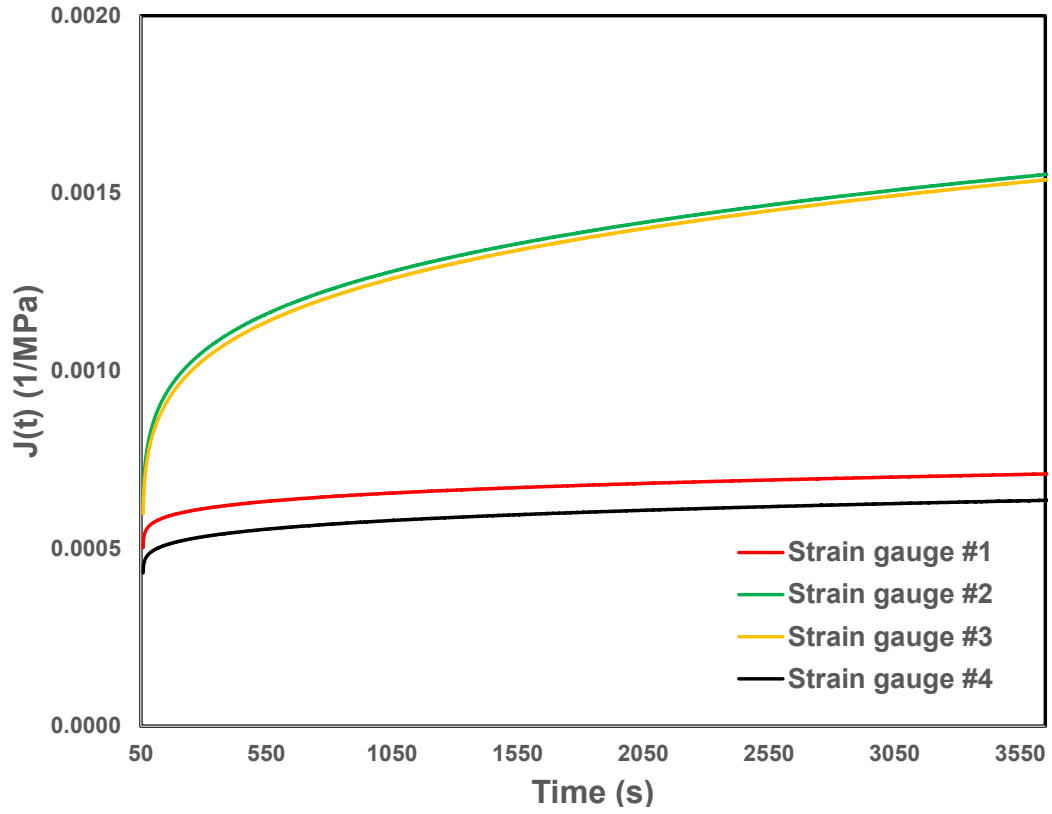


Fig. 14 Creep compliance of the butt joint at the adhesive bond line during isostatic loading

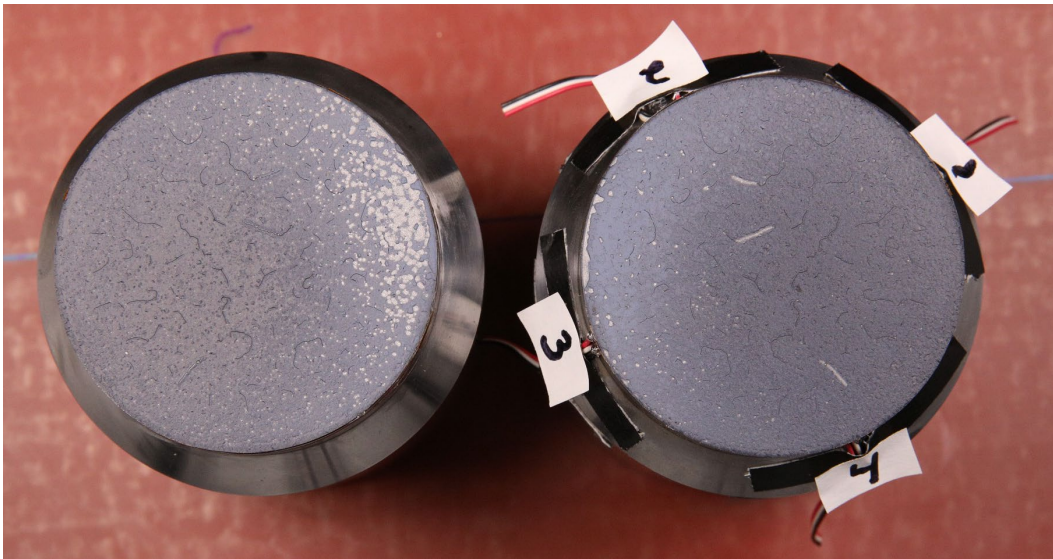


Fig. 15 Failure surfaces of the strain-gauged sample

4. Conclusion

The adhesive bonding joint configuration used for this study was a tensile butt joint with a 40 cm² surface area for bonding, which is specified by Guinness World Records. The following is a summary of the study results:

- Results show that an adhesive meeting MIL-PRF-32662¹⁰ Group 1 requirements readily sustains 22,680 kg (50,000 lb) of static loading for 1 h, for a 32% increase against the current world record benchmark (17,200 kg [37,919 lb]).
- Peak loads reached 31,850 kg (70,217 lb).
- Military-grade compares favorably against a commercial benchmark for extreme performance.
- Is the butt joint configuration specified by Guinness World Records¹¹ a worthy measurement of extreme performance?
 - The scaling factor of this joint is considerable and difficult. Typical academic investigations of butt joint performance are limited to surface areas of less than 1.7 cm².
 - Butt joints have limited utility for investigating adhesive constitutive properties, thus this joint configuration is not common for structural adhesive testing. Structural adhesives are more often screened using single-lap joints and rigorously characterized for fracture toughness using bonded double-cantilever beams. Butt joints are typically only used to characterize dental adhesives with very small bonding areas.^{20,21}
 - The alignment tooling and sample machining required for this scale is not trivial. Access to the high-capacity hydraulic loading frame available to DEVCOM ARL is also potentially limited.
 - Testing adhesive bond loads ranging from 222–312 kN is extreme for laboratory-scale testing. The loads achieved for this study were an order of magnitude greater than normal adhesive testing protocols.
 - Extensive laboratory testing of the adhesive is required as the high loads are dangerous for official Guinness World Record protocols and require safety considerations in the event of catastrophic failure to prevent injury, loss of life, and considerable property damage. This study demonstrates an unofficial breaking of the world record

using the relative safety of a laboratory loading frame. A “live” lift is required for official world record consideration.¹¹

- Due to the large bond area and high loads of the Guinness configuration, this joint will rapidly emphasize critical flaws in the adhesive bond line. From this perspective, the adhesive needs to have very high strength and fracture toughness to survive the 1-h isostatic load hold.
- When the bond line was correctly strain-gauged, the large butt joint configuration yielded adhesive constitutive properties that are consistent with values reported in the literature.
- Is the Guinness World Records butt joint configuration recommended to characterize constitutive properties of adhesives? No, there are many other standard testing configurations available to researchers that are much less labor-intensive and easier to derive rigorous constitutive properties from.
- Is Guinness World Records butt joint configuration a worthy method for testing extreme adhesive performance? Yes, as this is an intuitive joint loading configuration for non-adhesive subject matter experts to comprehend. Also, the isostatic loading experienced by this joint is so large in magnitude that critical flaws in the adhesive bond line will be rapidly exposed.

5. References

1. Ford uses high-strength steel plus high-strength aluminum alloy. Ford Motor Company; 2014 Jan 13. <https://media.ford.com/content/fordmedia/fna/us/en/news/2014/01/13/ford-uses-high-strength-steel-plus-high-strength--aluminum-alloy.html>.
2. Ortt JR, Egyedi TM. The effect of standards and regulation on radically new innovations. 2013 8th International Conference on Standardization and Innovation in Information Technology (SIIT); 2013. IEEE.
3. Hanseth O, Monteiro E, Hatling M. Developing information infrastructure: the tension between standardization and flexibility. *Sci Technol Hum Values*. 1996;21:407–426.
4. Sargent JF Jr, Gallo ME. The global research and development landscape and implications for the Department of Defense. Congressional Research Service; 2021 June 28. Report No.: R45403. <https://crsreports.congress.gov/product/pdf/download/R/R45403/R45403.pdf/>.
5. Secretary of Defense. Establishment of the Office of Strategic Capital. Memorandum. Department of Defense; 2022 Dec 1. <https://media.defense.gov/2022/Dec/01/2003123982/-1/-1/1/ESTABLISHMENT-OF-THE-OFFICE-OF-STRATEGIC-CAPITAL.PDF>
6. Egyedi M, Sherif MH. Standards dynamics through an innovation lens: next-generation Ethernet networks. *IEEE Communications Magazine*. 2010;48:166–171.
7. Krechmer K. Technical standards: foundations of the future. *StandardView*. 1996;4:4–8.
8. Horn PM. The changing nature of innovation. *Research Technology Management*. 2005;48:28–31.
9. AMS-A-25463. Adhesive, film form metallic sandwich construction. SAE International; 2013 July 16. Originated from MIL-A-25463, Amendment 2, 1961 Oct 19.
10. MIL-PRF-32662. Adhesive, high-loading rate, for structural and armor applications. Department of Defense (US); 2020 Aug 13.

11. Heaviest weight lifted with glue (non-commercially available). Specific Guidelines Pack. Guinness World Records; 2023 [accessed 2024 May 23]. <https://www.guinnessworldrecords.com/world-records/heaviest-weight-lifted-with-glue>.
12. Kinloch AJ. Adhesion and adhesive science and technology, Chapman and Hall; 1987.
13. ISO 6922. Adhesives – determination of tensile strength of butt joints. International Organization for Standardization; 1987.
14. Shah P, Agashe S. Review of fractional PID controller. *Mechatronics*. 2016;38:29–41.
15. Ochsner A, Gracio J. An evaluation of the elastic properties of an adhesive layer using the tensile-butt joint test: procedures and error estimates. *Int J Adhes Adhes*. 2007;27:129–135.
16. Adams RD, Coppendale J, Peppiatt NA. Stress analysis of axisymmetric butt joints loaded in torsion and tension. *J Strain Anal*. 1978;13:1–10.
17. Reedy ED. Connection between interface corner and interfacial fracture analysis of an adhesively-bonded butt joint. *Int J Solid Struct*. 2000;37:2429–2442.
18. Juliano TF, VanLandingham MR, Tweedie CA, Van Vliet KJ. Multiscale creep compliance of epoxy networks at elevated temperatures. *Exp Mech*. 2007;47:99–105.
19. Bakbak O, Birkan BE, Acar A, Colak O. Mechanical characterization of Araldite LY 564 epoxy: creep, relaxation, quasi-static compression and high strain rate behaviors. *Polym Bull*. 2022;79:2219–2235.
20. Neves AA, Coutinho E, Poitevin A, Van der Sloten J, Van Meerbeek B, Van Oosterwyck H. Influence of joint component mechanical properties and adhesive layer thickness on stress distribution in micro-tensile bond strength specimens. *Dental Materials*. 2009;25:4–12.
21. Kameyama A, Kato J, Yoshinari M, Kotoku Y, Akashi G, Hirai Y. Ultimate micro-tensile strength of dental adhesives cured at different light sources. *J Photopolymer Sci and Tech*. 2008;21:31–35.

List of Symbols, Abbreviations, and Acronyms

ARL	Army Research Laboratory
DEVCOM	US Army Combat Capabilities Development Command
DOD	Department of Defense
PID	proportional integral derivative
R&D	research and development