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Initial Screening of Environmentally Sustainable Surface Pretreatments for Adhesive Bonding Applications

by Miriam S Siltan, David P Flanagan, Daniel C DeSchepper,
and Robert E Jensen

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Initial Screening of Environmentally Sustainable Surface Pretreatments for Adhesive Bonding Applications

by Miriam S Siltan

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14. ABSTRACT A methacrylate adhesive marketed for high-temperature applications was screened in combination with environmentally sustainable TT-C-490F surface pretreatments. Testing consisted of single-lap-joint testing using aluminum and steel specimens at room temperature along with hot/wet (H/W) conditioning (water immersion for 14 d at 63 °C) and in-situ at elevated temperature (ET) (71 °C). The methacrylate adhesive showed high initial bond strength and H/W durability but significant loss of strength retention at ET. The environmentally sustainable TT-C-490F Type IV inorganic pretreatments resulted in little to no loss of adhesive bond strength during H/W conditioning and their potential use as bonding pretreatments is worth further investigation.					
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1. Introduction

The greatest costs during the life-cycle of a Department of Defense (DOD) weapon system are those incurred in the operations and support phase, as shown in Fig. 1.¹ (Note: Figure 1 is illustrative versus quantitative. Adapted from “Operating and Support Cost-Estimating Guide”. Published by the Office of the Secretary of Defense, Cost Analysis Improvement Group, 1992.) With regards to adhesively bonded assemblies, these operating expenses generally result from failures in the field due to inferior pretreatments, the adhesive’s inability to withstand elevated temperature (ET), and/or moisture exposure conditions. Additionally, as environmental regulations force various chemicals from the commercial market, the pretreatments and adhesives containing regulatory banned constituents also become unavailable. This train of events leads to phase-out risks for ongoing maintenance of Army legacy platforms. One way to reduce life-cycle expenses is to focus on the environmental sustainability of the adhesive and surface pretreatment during initial research and development.

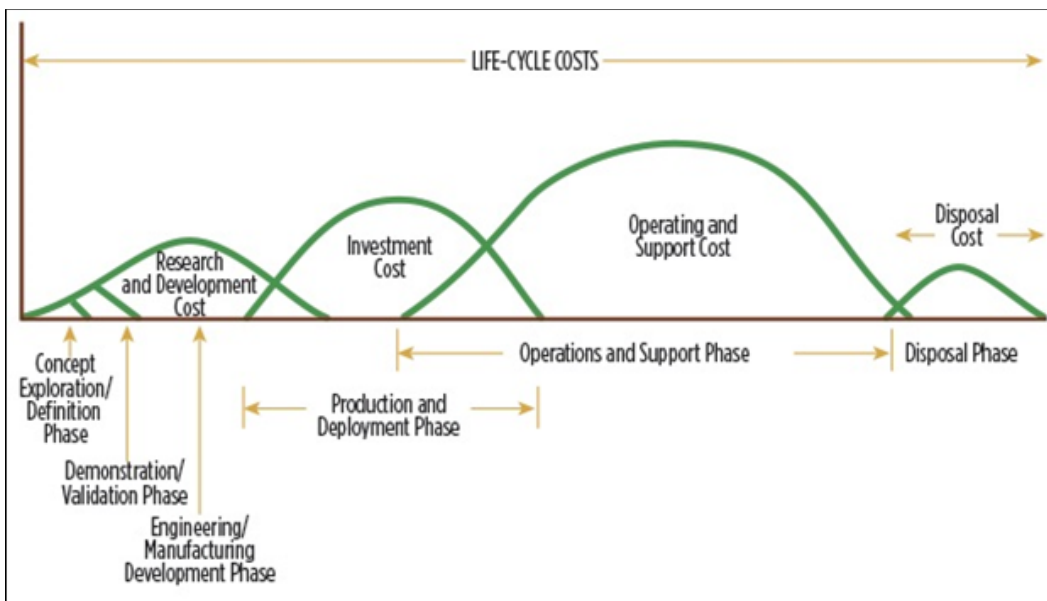


Fig. 1 Notional life-cycle costs for a DOD weapon system

The surface pretreatments utilized for this research are commercially available and qualified, or in the process of being qualified, under Federal Specification TT-C-490F,² as with Type II or IV hexavalent chromium-free chemical conversion coatings. A majority of TT-C-490F qualified pretreatments were originally formulated as zinc-based pretreatments to prevent corrosion on steel, not as bonding surface pretreatments. If these environmentally friendly TT-C-490F pretreatments prove compatible for adhesive bonding then they could potentially function simultaneously as both corrosion inhibitors and adhesion promoters.

SCI GRIP SG805, which is methacrylate based and marketed for high-temperature applications,³ was the adhesive examined for this study. This adhesive was tested against screening standards protocols outlined in ARL-ADHES-QA-001.00 rev 1.0⁴ and for compatibility with the environmentally sustainable TT-C-490F Type IV surface pretreatments. Testing consisted of single-lap-joint shear tensile testing using 2024 T3 aluminum and 1010 mild steel specimens at room temperature (RT), after 14 d of hot/wet (H/W) immersion at 63 °C, and in-situ at an ET of 71 °C. Single-lap-joints are widely studied in the literature and allow for minimal labor intensive standardized testing.⁵ Adhesives that retain 75% of their dry maximum strength (S_{max}) after environmental conditioning and during in-situ ET testing are of interest. The methacrylate adhesive showed promising high-initial bond strength and H/W durability but was unable to meet ET performance retention requirements. The use of environmentally sustainable TT-C-490F pretreatments resulted in little to no loss of adhesive bond strength during H/W conditioning and their potential use as bonding promoters for adhesives is worthy of further investigation.

2. Experimental

2.1 Materials and Environmental Assessment

SCI GRIP SG805 methacrylate adhesive was used for bonding the single-lap-joints. The TT-C-490F surface pretreatments included Bonderite M-NT 7400 (Henkel Corporation), SurTec 650 (CST-Surtec, Inc.), and ZIRCOBOND 4200 (PPG Industries, Inc.). While qualified for TT-C-490F, Oxysilan 9810/2 (Chemetall US, Inc.) was not included due to sample nonavailability at the commencement of the study. Additional environmentally sustainable surface pretreatments, as claimed by the manufacturers, included Bonderite M-NT 5700 (Henkel Corporation), XBOND 4000DM-DR (PPG Industries, Inc.), and AC-131 BB (3M Company). Grit blasting and an academic 3-aminopropyltrimethoxysilane (APS) coupling agent were used as performance comparisons with the TT-C-490F qualified and other commercial pretreatments.

Appendix A lists associated environmental restrictions matched against the constitutive chemical components of the adhesive and surface pretreatments using the restricted substances database feature of the US Army Research Laboratory’s Materials Selection and Analysis Tool (MSAT) database.⁶ The restricted substances database references chemical abstract service numbers reported in the manufacturer’s safety data sheets against current and pending environmental legislation in North America, Europe, and Asia. The highest substance rating returned was “caution”. None of the constitutive chemical components of the adhesive and surface pretreatments used for this study were reported as being “banned” by any current or pending regulations.

2.2 Joint Geometry

The single-lap-joints were fabricated and tested using American Society for Testing and Materials (ASTM) D1002-10 as the basis standard, schematically represented in Fig. 2.⁷

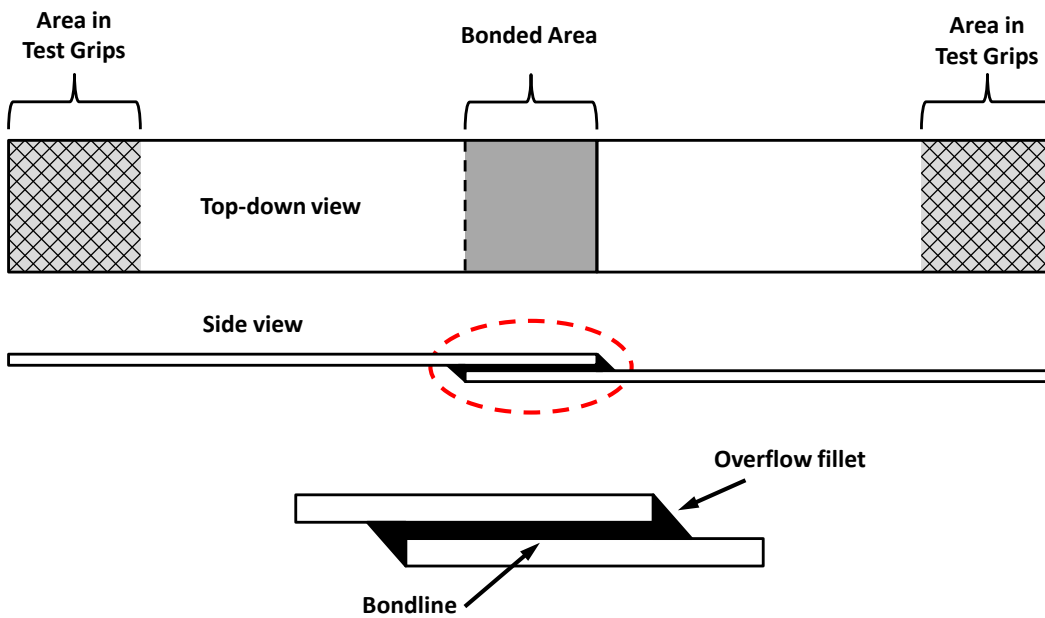


Fig. 2 Illustration of the adhesively bonded single-lap-joint test specimen configuration (refer to ASTM D1002-10 for dimensions)

Maximum strength (S_{\max}) is calculated by dividing the maximum load (P_{\max}) by the bonded area (A). The overlap length is 12.7 mm and the joint width is 25.4 mm.

$$S_{\max} = \frac{P_{\max}}{A}$$

Maximum strength and mode-of-failure represent the accepted standard reported outputs of single-lap-joint testing in both industry and academia.⁸

2.2.1 Surface Preparation

Preparation of the methacrylate adhesive bonded single-lap-joints followed procedures outlined in ARL-ADHES-QA-001.01 rev 2.2.⁹ The single-lap-joint substrates used 2024 T3 aluminum and 1010 steel coupons with an average thickness of approximately 1.60 and 1.52 mm, respectively. Sandpaper (180-grit aluminum oxide), an acetone wipe down, and an abrasive disc (3M Scotch-Brite Roloc, light grinding and blending disc, part number 60357) were used to initially remove oils and large deposits of oxide corrosion from the surfaces of the aluminum and steel. The coupons were then abrasive media blasted with clean and unused 60-grit aluminum oxide. From this point forward, the intended bonding surface of each coupon came into contact only with its corresponding coupon's grit-blasted bonding surface or air. After being blown off with a thin stream of nitrogen gas, the various TT-C-490F pretreatments were applied in accordance with their manufacturer's instructions. All of the pretreatments except for the APS and AC-131 BB sol-gel were applied by dip-coating the coupons for 2 min (Fig. 3), rinsed for 30 s with deionized water, and again blown off with a thin stream of nitrogen gas to produce a thin, uniform coating. The APS was also applied using the dip-coat technique; however, the coupons were blown off with nitrogen gas immediately after and then placed in an oven for 1 h at 65 °C. The AC-131 BB sol gel was brush applied to the surface intended for bonding, allowed to wet for 2 min, and then immediately blown off with nitrogen gas. All surface preparation was completed within 4 h of bonding.

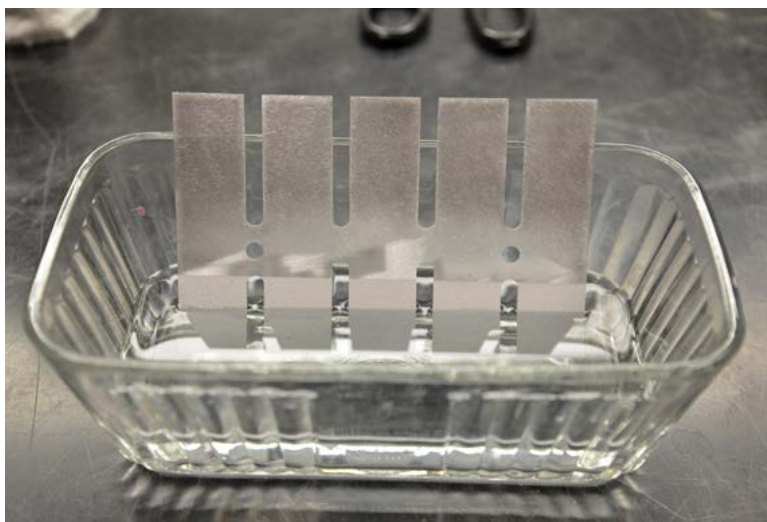


Fig. 3 Single-lap-joint coupons undergoing dip-coat pretreatment

2.2.2 Bonding

The methacrylate adhesive has a set time of 5 min, so it was imperative that the assembly components of the bonding tooling fixture were gathered beforehand to facilitate a quick-bonding process, as shown in Fig. 4. Bondline thickness and overlap dimension control were essential to reducing possible sources of experimental error. Spacer shims (0.762 mm) were used to set the bondline thickness. The tooling fixture is also equipped with alignment pins to set orientation and overlap length.⁹

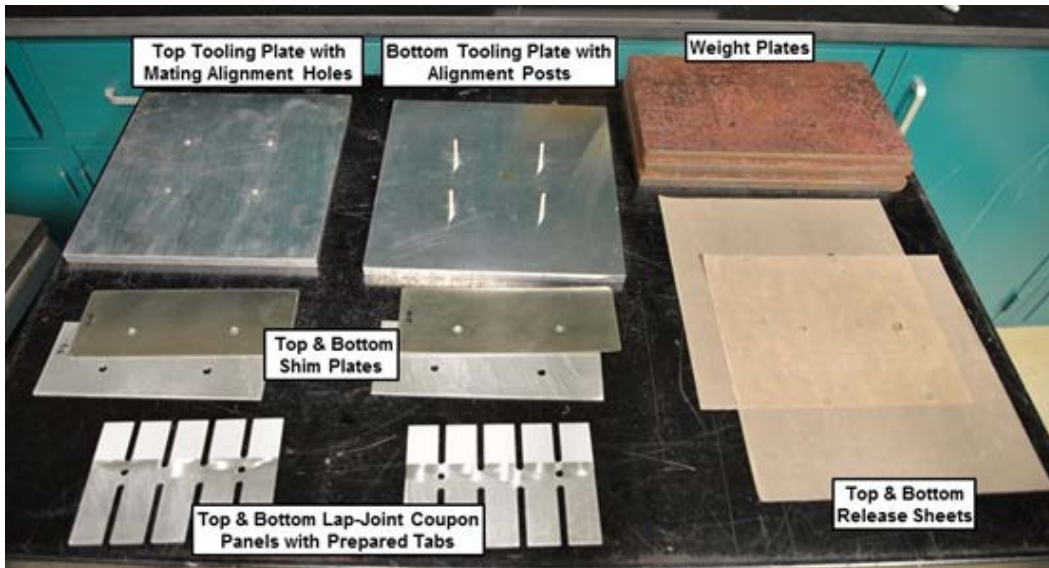


Fig. 4 Single-lap-joint bonding tooling fixture components

A pneumatic gun with a static-mixing head was used to dispense the 2-part adhesive with the required 10:1 ratio, per the manufacturer's recommendations.¹⁰ The methacrylate adhesive was applied to an area slightly larger than the overlap area of 12.7×25.4 mm at the front of each finger on the coupon. Bonding pressure was maintained by placing weights on top of the assembled tooling fixture to ensure intimate contact with the bondline thickness spacer shims while the adhesive was curing. Figure 5 shows the tooling fixture assembly stacking sequence. The cure cycle was 4 d at room temperature. In addition, the methacrylate adhesive was also cast into silicone molds and allowed to cure, to yield thin-rectangular specimen bars for use in dynamic mechanical analysis (DMA) testing.

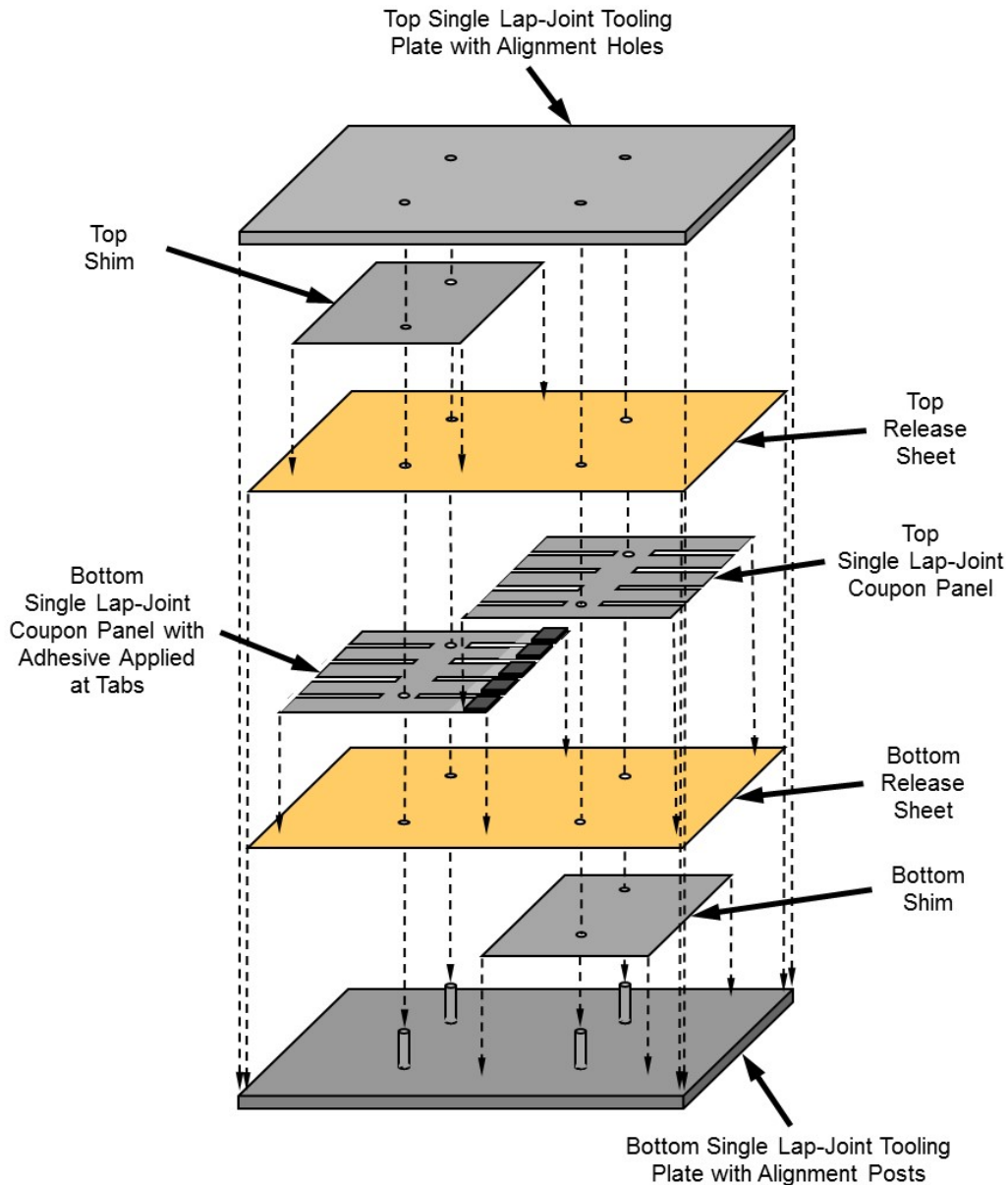


Fig. 5 Stacking assembly for fabrication of single-lap-joint panel

2.3 Testing

The MSAT database was used to collect the data following the workflow protocols outlined in ARL-TR-7696.¹¹

2.3.1 Mechanical Testing

The test procedure for performing the breaking strengths of single-lap-joint samples was conducted in accordance to paragraphs 9.1 and 9.2 of ASTM D1002-10.⁷ instrumented mechanical testing frame with a 25-kN load cell was used to ensure

that the breaking load of single-lap-joint samples fell between 15% and 85% of the cell's full-scale capacity. A crosshead speed of 1.27 mm/min was used with a pair of self-aligning grips that held the outer 25.4 mm of each end of the single-lap-joint test sample.

H/W-conditioned samples were fully immersed in deionized (DI) water for 14 d at 63 °C, prior to mechanical testing. Mechanical tests occurred on the same day as their removal from the heated bath. Once removed, samples were allowed to cool to RT and patted dry with paper towels prior to loading into the test frame. In-situ ET testing was conducted using a heated static oven that enclosed around the load frame, which was allowed to stabilize at a temperature of 71 °C, ± 3 °C for 45 min, prior to the start of mechanical testing. Before loading samples within the test grips, each single-lap-joint sample was allowed to rest at least 10 min at RT. Once placed within the grips, the thermocouple attached and the oven door closed and secured, the sample's temperature was then monitored for 10 min with a digital thermometer until the sample reached equilibrium with the oven temperature and was stable. H/W and ET conditions, which were recommended per ARL-ADHES-QA-001.00 rev 1.0,⁴ were based on test method guidelines provided by Military Standard-810G (MIL-STD-810G), *Environmental Engineering Considerations and Laboratory Tests*.^{12,13}

Upon completion of mechanical testing, failure surfaces of each broken single-lap-joint coupon sample were digitally imaged using a flatbed scanner (Hewlett-Packard OfficeJet D145) and archived in MSAT. Failed test coupons were then manually labeled with a MSAT identification (ID) code, and this information was then embedded within the image, preserving data integrity by eliminating file naming errors. Images were scanned at 300-dots per inch (dpi) resolution and saved as a tagged image file format (TIFF), which is a common minimum recommendation for photo archiving.¹⁴ After the fractured surfaces were visually inspected and examined, the tested coupons were assigned either an adhesive, cohesive, or mixed-mode (MM) type of failure designation. Load versus displacement plots and failure surface scans are provided in Appendix B.

2.3.2 Dynamic Mechanical Analysis

The small bulk adhesive rectangular sample bars made during the bonding process were sanded to uniform thickness using 60 grit followed by 180-grit aluminum oxide sandpaper. Thickness for single-cantilever DMA testing of the cured adhesive was between 2 and 4 mm. The samples were cut to a length of approximately 35 mm using a water saw from sections that were free of visible voiding defects. The thickness and width of a sample was measured using a telescoping micrometer before tightening it in the DMA clamps with a torque of

0.79 N·m. DMA was performed with a constant frequency of 1 Hz and a constant strain of 0.2%. The sample was equilibrated at 0 °C and held there for 10 min before being heated to 71 °C at a rate of 2 °C/min. Once this process was completed, the clamps were retightened, the length of the sample between the clamps was measured and recorded, and DMA was run a second time on the same sample. DMA testing yielded the storage modulus (E'), loss modulus (E''), and loss tangent ($\tan \delta$) of the methacrylate adhesive, allowing for determination of the adhesive's glass transition temperature (T_g).

3. Results

Sample sets were organized by substrate material, surface pretreatment, and conditioning method. Tables 1 and 2 provide a quantitative summary of tensile testing results along with associated modes of failure for the aluminum and steel single-lap-joints. Figures 6–9 show plots of maximum load and displacement at failure for the aluminum and steel joints. A load versus extension at complete failure plot is provided for each set, containing curves for all samples within that set. An example failure surface scan is included below each graph to show a representative sample for mode of failure confirmation. Complete records of test data and failure surface scans may be accessed at the National Institute of Standards and Technology Dspace repository site <http://hdl.handle.net/11256/937>. A summary of the data and supporting metadata descriptors are found in Appendix C.

Table 1 Aluminum 2024 T3, RT, H/W, and ET conditioning. Mode-of-failure: adhesive (ADH), cohesive (COH), and MM.

Surface pretreatment	RT conditioning			H/W conditioning			ET conditioning		
	S_{max} (MPa)	$d_{failure}$ (mm)	Mode-of-failure	S_{max} (MPa)	$d_{failure}$ (mm)	Mode-of-failure	S_{max} (MPa)	$d_{failure}$ (mm)	Mode-of-failure
Grit Blast	19.9 (+0.7)	1.59 (+0.16)	MM	18.8 (+0.8)	1.74 (+0.18)	MM	8.45 (+0.32)	2.40 (+0.35)	MM
3-APS	11.2 (+1.1)	0.72 (+0.03)	ADH	12.2 (+0.8)	1.01 (+0.08)	ADH	5.66 (+0.46)	3.49 (+0.75)	ADH
AC-131	20.6 (+0.2)	1.79 (+0.10)	MM	19.4 (+0.3)	2.14 (+0.09)	MM	Not tested	Not tested	NA
SurTec-650	20.0 (+0.2)	1.59 (+0.08)	MM	19.3 (+0.6)	1.92 (+0.12)	MM	Not tested	Not tested	NA
SerTec-5700	17.5 (+0.4)	1.34 (+0.20)	ADH/MM	19.6 (+0.1)	2.21 (+0.07)	ADH/MM	Not tested	Not tested	NA
SerTec-7400	17.9 (+0.9)	1.48 (+0.32)	ADH/MM	19.1 (+0.2)	1.95 (+0.11)	ADH/MM	Not tested	Not tested	NA
XBOND	20.1 (+0.8)	1.64 (+0.18)	ADH/MM	19.4 (+0.4)	1.91 (+0.13)	ADH/MM	Not tested	Not tested	NA
ZIRCOBOND	20.7 (+0.5)	1.82 (+0.09)	MM	19.5 (+0.7)	2.05 (+0.17)	MM	Not tested	Not tested	NA

NA = not applicable

Table 2 1010 mild steel, RT and H/W conditioning. ET conditioning not tested for steel samples. Mode-of-failure: ADH, COH, and MM.

Surface pretreatment	RT conditioning			H/W conditioning		
	S_{max} (MPa)	$d_{failure}$ (mm)	Mode-of-failure	S_{max} (MPa)	$d_{failure}$ (mm)	Mode-of-failure
AC-131	17.8 (±0.5)	1.51 (±0.10)	MM	18.7 (±0.4)	1.73 (+0.12)	MM
SurTec-650	21.6 (±0.6)	1.95 (±0.08)	MM	20.3 (±0.7)	2.02 (+0.14)	MM
SerTec-5700	20.9 (±0.5)	2.11 (+0.11)	MM	20.6 (±0.4)	2.02 (+0.08)	MM
SerTec-7400	20.9 (±0.4)	1.94 (+0.06)	MM	20.2 (±0.5)	1.98 (+0.05)	MM
XBOND	21.2 (±0.8)	1.89 (+0.07)	MM	20.5 (±0.3)	2.11 (+0.06)	MM
ZIRCOBOND	20.6 (±0.5)	1.82 (+0.22)	MM	19.4 (±0.6)	1.97 (+0.11)	MM

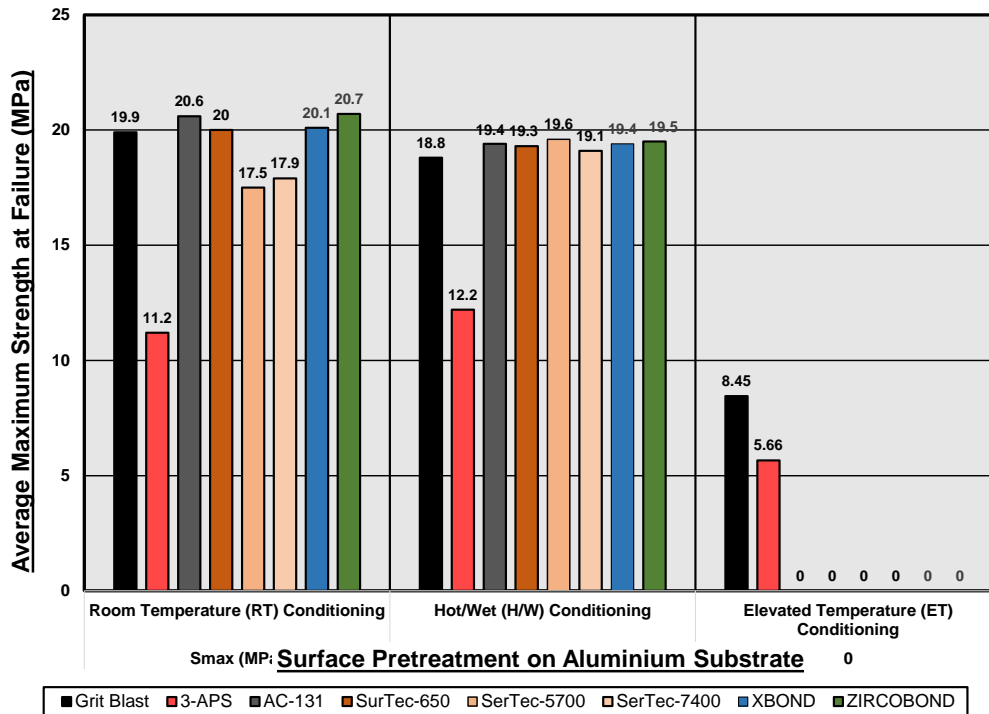


Fig. 6 Average maximum strength at failure (MPa) for aluminum 2024 T3, RT, H/W, and ET conditioning

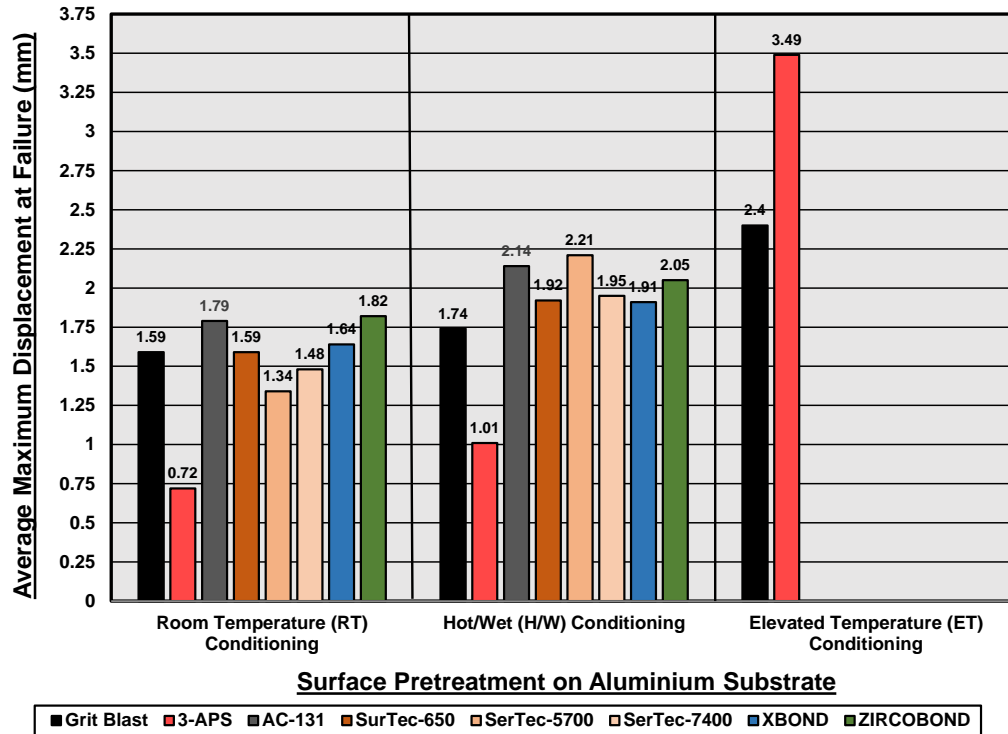


Fig. 7 Average maximum displacement at failure (mm) for aluminum 2024 T3, RT, H/W, and ET conditioning

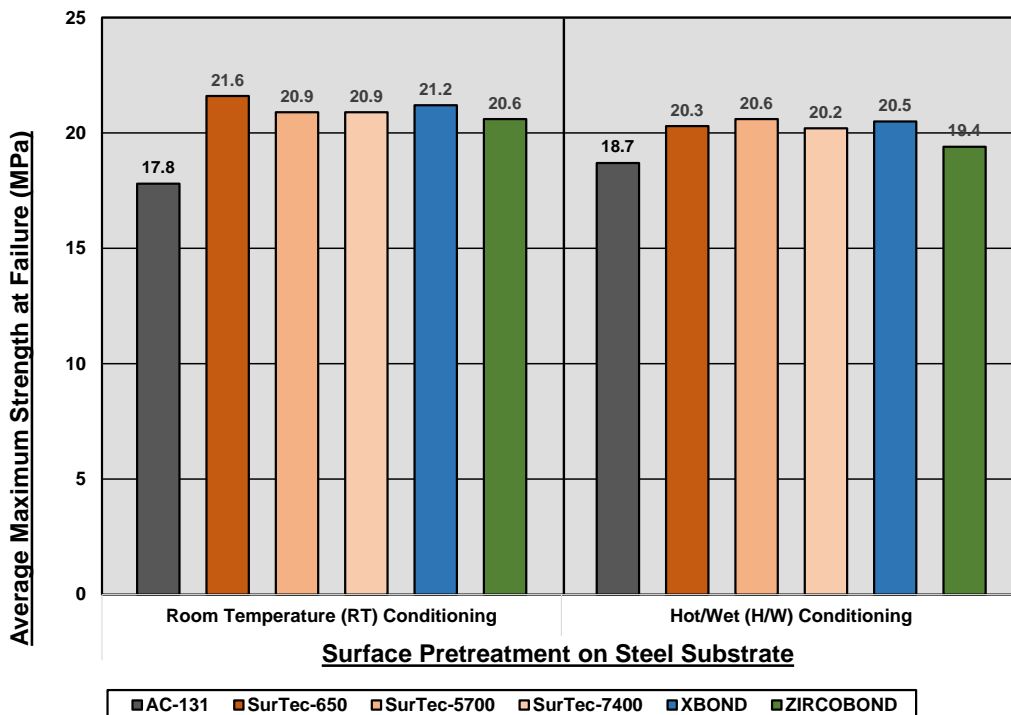


Fig. 8 Average maximum strength at failure (MPa) for 1010 mild steel, RT, and H/W conditioning. ET conditioning not tested for steel samples.

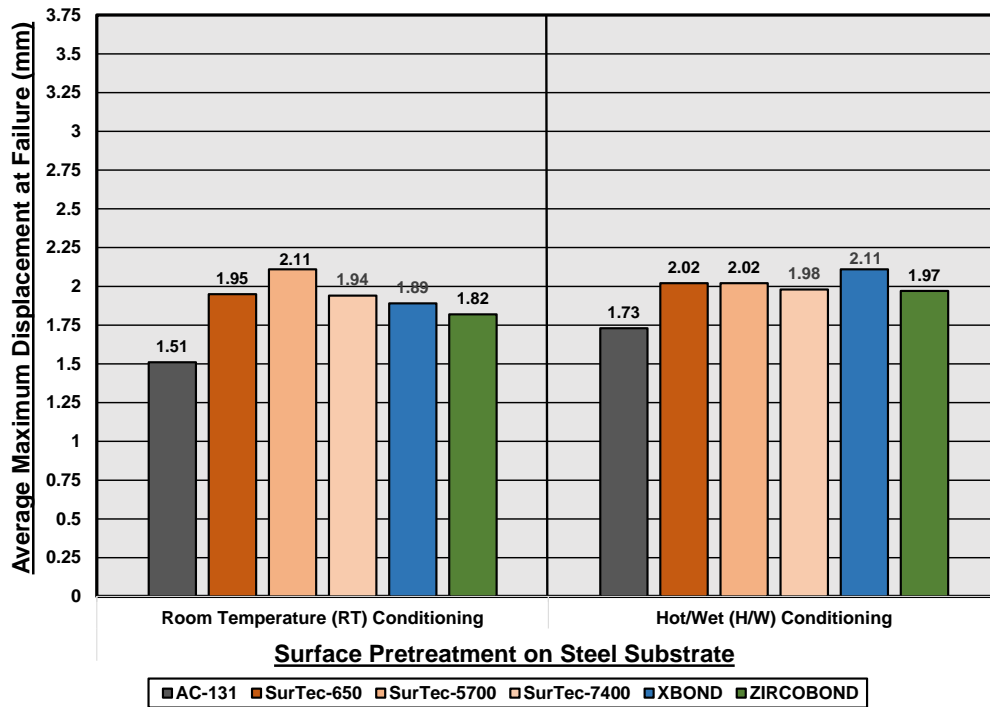


Fig. 9 Average maximum displacement at failure (mm) for 1010 mild steel, RT, and H/W conditioning. ET conditioning not tested for steel samples.

4. Discussion

The bonded single-lap-joints with the SG805 adhesive and sustainable TT-C-490F pretreatments yielded strengths of approximately 19 MPa. A majority of the aluminum lap joints, and all of the steel lap joints, had mixed-mode failure. At RT, the SG805 mechanical performance using the aluminum coupons with the grit-blasted surface preparation was comparable to the chemical pretreatments (Fig. 10), including similar failure modes as shown in Fig. 11. The grit-blasted samples also retained approximately 94% of their dry strength following H/W conditioning, as summarized in Table 3. The results for the grit-blasted samples are consistent with observations for methacrylate adhesive usage for dental applications, both with and without added silane coupling agent pretreatments, where good long-term wet durability is essential.¹⁵⁻¹⁷ Steel single-lap-joints with a grit-blast-only surface pretreatment were not tested.

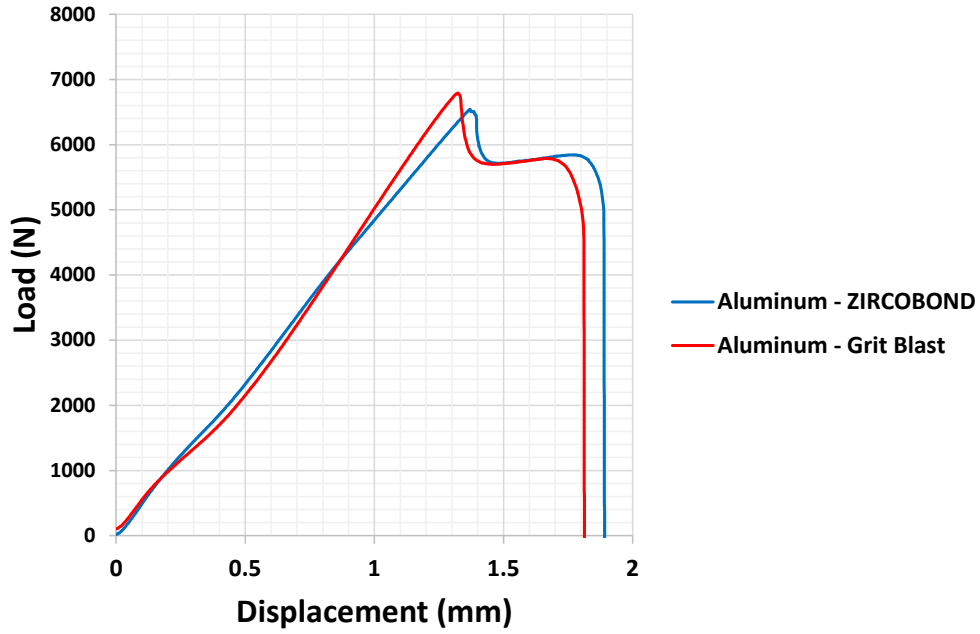


Fig. 10 Load vs. displacement response for aluminum single-lap-joints pretreated by grit blasting and with ZIRCOBOND TT-C-490F Type IV surface pretreatment

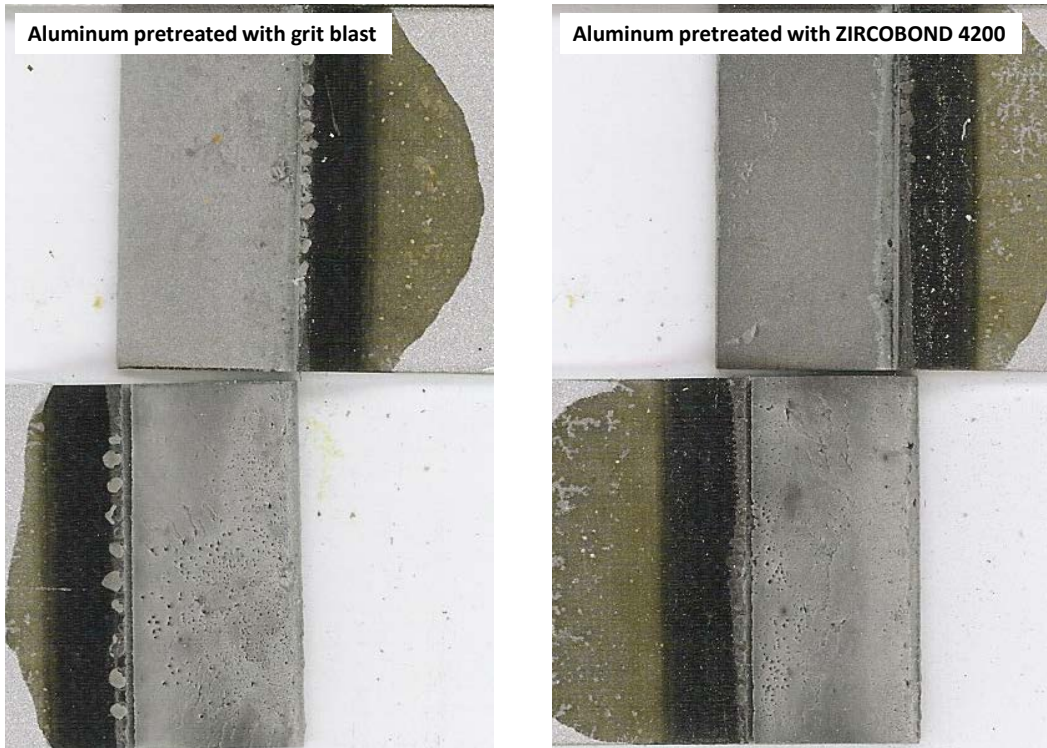


Fig. 11 Failure surfaces for aluminum single-lap-joint with grit-blasted surface (left) and ZIRCOBOND 4200 (right) pretreatments

The aluminum samples with the APS pretreatment had lower strengths and displacements to failure when compared with the TT-C-490F pretreatments as shown in Fig. 12. The amine functionality of the APS should be reactive toward the methacrylate groups of the adhesive through a Michael addition mechanism¹⁸ but was perhaps negated by the cure kinetics or other additives present in the adhesive, as seen by the adhesive mode-of-failure in Fig. 13. Methacrylate functional silane coupling agents have been shown to enhance adhesion with methacrylate-based adhesives and may show higher strengths with SG805.¹⁹

The steel single-lap-joints samples pretreated with AC-131 showed visual flash corrosion during its application. This phenomenon was not observed in any of the other pretreatments, and the corresponding load versus displacement response shown in Fig. 12 depicts lower peak properties than the TT-C-490F Type IV qualified pretreatments. The AC-131 pretreatment is a water-based system and was applied via brush wetting rather than the dip-coating technique used for the other chemical pretreatments. The brush-wetting method allows for air exposure and provides the corrosive environment necessary for an oxide layer to form. The failure surfaces of the steel joints treated with AC-131 showed adhesive mode-of-failure with evidence of the flash corrosion seen on the adhesive and steel adherend. The AC-131 provided comparable strengths when applied to the aluminum joints, as summarized in Table 1.

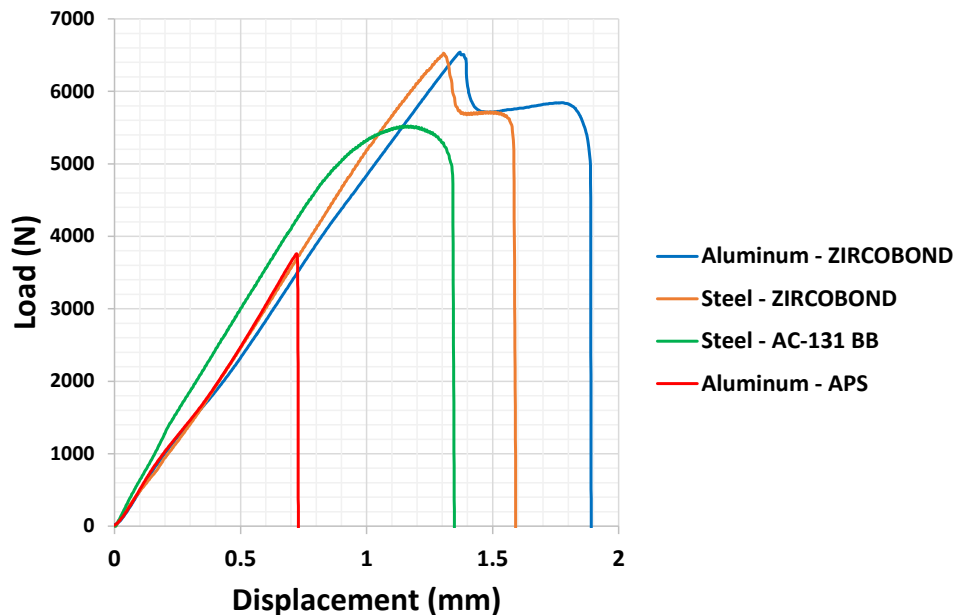


Fig. 12 Load vs. displacement response for aluminum single-lap-joints pretreated with APS, steel samples pretreated with AC-131, and steel and aluminum pretreated with ZIRCOBOND TT-C-490F Type IV surface pretreatment

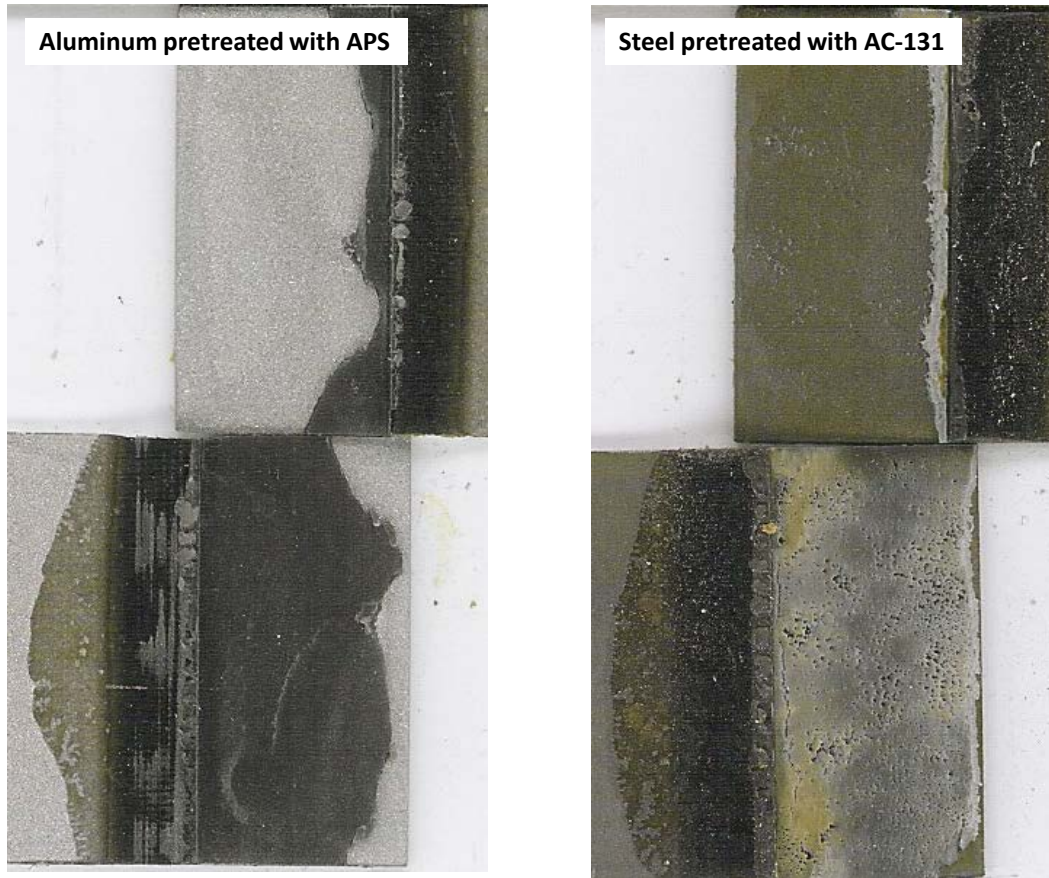


Fig. 13 Failure surfaces for aluminum single-lap-joint pretreated with APS (left) and steel pretreated with AC-131 (right)

Comparison within the aluminum and steel adherends shows similar, if not increased, performance of the SG805 methacrylate adhesive after H/W conditioning, as summarized in Table 3. The adhesive was likely undergoing additional postcure at the 63 °C temperature of the H/W conditioning, which can lead to an increase in strength. For an adhesive systems to pass the screening testing, it is required to retain 75% of its RT strength after H/W conditioning, which the SG805 methacrylate adhesive achieved.

Table 3 Average property changes after H/W and ET conditioning

Surface pretreatment	2024 T3 Aluminum				1010 mild steel	
	Average strength (S_{max}) retention after conditioning (%)		Average displacement ($d_{failure}$) change after conditioning (%)		Average strength (S_{max}) retention after conditioning (%)	Average displacement ($d_{failure}$) change after conditioning (%)
	H/W conditioning	(ET) conditioning	H/W conditioning	(ET) conditioning	H/W conditioning	
Grit Blast	94	42	9	51	NA	NA
3-APS	109	51	39	385	NA	NA
AC-131	106	NA	20	NA	105	15
SurTec-650	104	NA	21	NA	106	4
SerTec-5700	112	NA	65	NA	101	4
SerTec-7400	107	NA	32	NA	103	2
XBOND	103	NA	16	NA	103	12
ZIRCOBOND	106	NA	13	NA	106	8

Elevated temperature testing showed a significant decrease in bond performance, as shown in Fig. 14. The grit blasted single-lap-joint strength decreased from 19.9 MPa at RT to 8.45 MPa at 71 °C, for only a 42% retention in strength. Elevated temperature performance in the absence of moisture exposure is unlikely to degrade due to the surface pretreatment and is more reliant on the bulk adhesive properties. Although the lap joints pretreated with 3-APS displayed a high-average extension to failure of 3.49 mm, this was largely due to tensile failure of the adhesive after it had de-bonded from the substrate (Fig. 15). Elevated temperature testing was conducted at 71 °C, which is within the glass transition region of this adhesive, as shown in the storage and loss modulus DMA plot in Fig. 16. As the adhesive moves from the glassy to rubbery state, it becomes more ductile and is unable to sustain the high loads that are possible at lower temperatures.

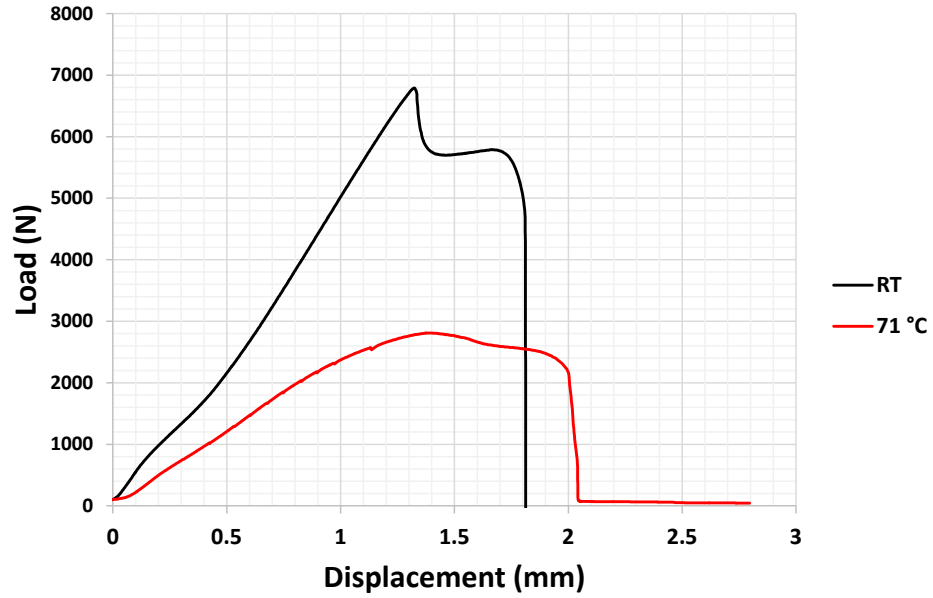


Fig. 14 Load vs. displacement response for grit-blasted aluminum single-lap-joints tested at RT and 71 °C

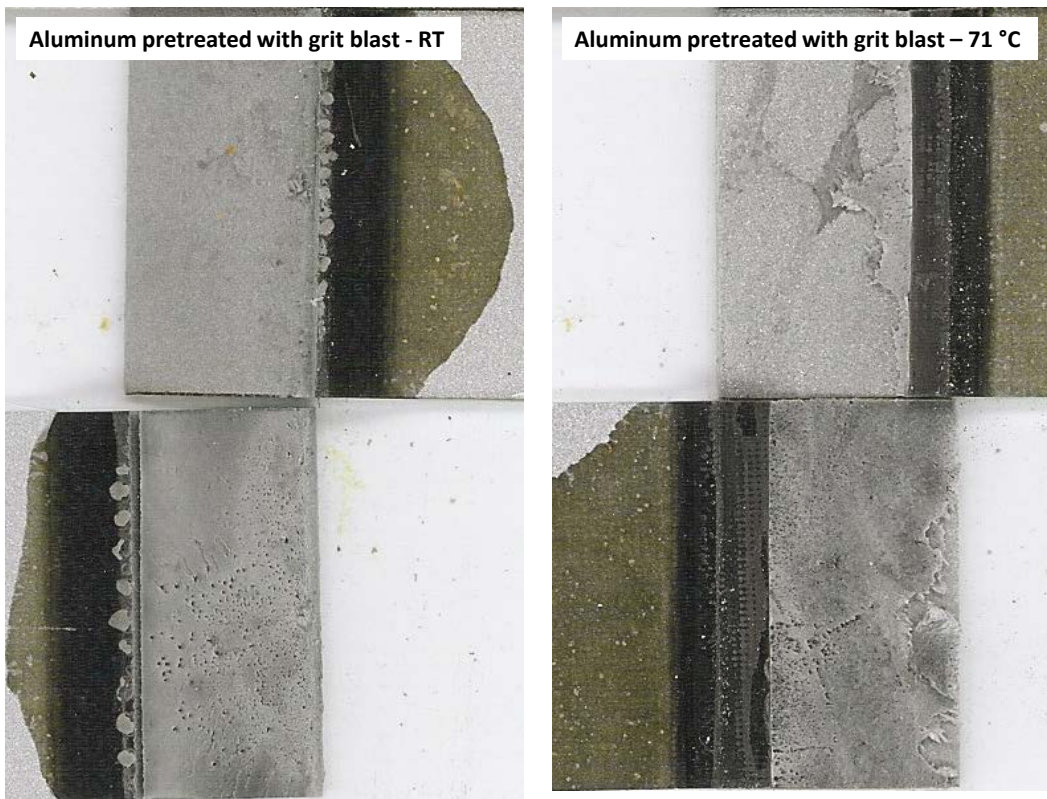


Fig. 15 Failure surfaces for grit-blasted aluminum single-lap-joints tested at RT (left) and 71°C (right)

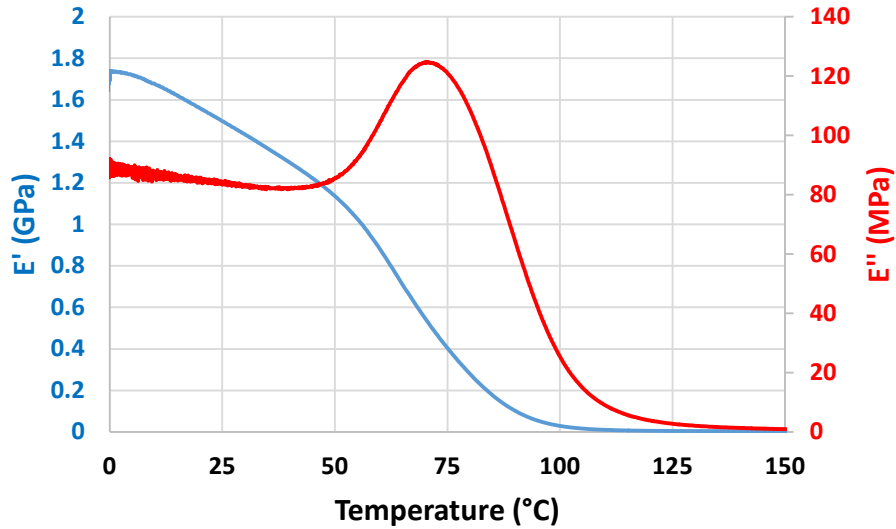


Fig. 16 Storage modulus (E') and loss modulus (E'') vs. temperature for SG805

5. Conclusions

SG805 was tested against ARL-ADHES-QA-001.00 rev 1.0 and for compatibility with the environmentally sustainable TT-C-490F surface pretreatments. Testing consisted of single-lap-joint shear tensile testing using aluminum and steel specimens with RT, H/W, and ET conditioning. The SG805 methacrylate adhesive was found to be suitable for RT and H/W conditions but showed excessive loss of strength at ET. At RT and after H/W conditioning the adhesive performed well on both aluminum and steel substrates, even when no chemical surface pretreatment was applied. Environmentally sustainable TT-C-490F Type IV inorganic pretreatments resulted in little to no loss of adhesive bond strength during H/W conditioning and their potential use as bonding pretreatments is worthy of further investigation. The TT-C-490F pretreatments offer the advantage of corrosion protection, which should be considered for bonding applications. Further research with the SG805 methacrylate adhesive is warranted for applications with lower service temperature requirements.

6. References

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**Appendix A. Environmental Restrictions Listed against the
Adhesive and Surface Pretreatments**

Adhesive or Surface Pretreatment	Substance Name	CAS Number	Substance Rating	Legislative Organization	Legislation Name	Legislation Rating
SG805 Adhesive	Methyl Methacrylate	80-62-6	Caution	US Environmental Protection Agency (EPA)	Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) hazardous substances	NA
				US EPA	Emergency Planning and Community Right-to-Know Act (EPCRA) Section 314, Toxic Chemicals	NA
				US EPA	EPA's List of Lists (LoL)	NA
				US EPA	National Emission Standards for Hazardous Air Pollutants (NESHAP) for Stationary Reciprocating Internal Combustion Engines-US Air Toxics Regulations	NA
				AeroSpace and Defence Industries Association of Europe - (ASD)	ASD-Standardization Declarable Substances List	Caution
				US EPA	EPCRA-Section 304, Extremely Hazardous Substances, Reportable Quality (RQ)	NA
				European Trade Union Confederation (ETUC)	ETUC Priority List	Caution
				European Chemical Agency (ECHA)	Community Rolling Action Plan (CoRAP) List	Caution
Bonderite M-NT 7400	Phosphoric Acid	7664-38-2	Caution	US EPA	CERCLA hazardous substances	NA
<ul style="list-style-type: none"> •TT-C-490F •TYPE IV, CLASS A, DEGREASE AND ABRASIVE BLAST and TYPE IV, CLASS B AND CLASS C 						
SurTec 650 ChromitAL	Zirconium Potassium Fluoride	16923-95-8	Caution	US EPA	CERCLA hazardous substances	NA
<ul style="list-style-type: none"> •TT-C-490F •TYPE IV, CLASS A, DEGREASE AND ABRASIVE BLAST 						
ZIRCOBOND 4200DM-DR	Cupric Nitrate	3251-23-8	Caution	US EPA	CERCLA hazardous substances	NA
<ul style="list-style-type: none"> •TT-C-490F •TYPE IV, CLASS A, DEGREASE AND ABRASIVE BLAST and TYPE IV, CLASS B AND CLASS C 						
Bonderite M-NT 5700	No hazardous ingredients claimed in manufacturer's safety data sheet.					
XBOND 4000DM-DR	No hazardous substance ratings associated with CAS numbers provided in manufacturer's safety data sheet.					

Adhesive or Surface Pretreatment	Substance Name	CAS Number	Substance Rating	Legislative Organization	Legislation Name	Legislation Rating
AC-131 BB	Acetic Acid	64-19-7	Caution	US EPA	CERCLA hazardous substances	NA
				US EPA	EPA's List of Lists (LoL)	NA
				ASD	ASD-STAN Declarable Substances List	Caution
				US EPA	EPCRA Section 304, Extremely Hazardous Substances, RQ	NA
AC-131 BB	Methanol	67-56-1	Caution	US EPA	CERCLA hazardous substances	NA
				US EPA	EPCRA Section 313, Toxic Chemicals	NA
				US EPA	The EPA's List of Lists (LoL)	NA
					NESHAP-US Air Toxics Regulations	NA
				US EPA	NESHAP for Stationary Reciprocating Internal Combustion Engines	NA
				ASD	ASD-STAN Declarable Substances List	Caution
				European Union (EU)	Directive 1996/82/EC (Seveso II) on the control of major-accident hazards involving dangerous substances	NA
				US EPA	EPCRA Section 304, Extremely Hazardous Substances, RQ	NA
				EU	Global Automotive Declarable Substance List (GADSL)	Caution
				ETUC	ETUC Priority List	Caution
				ECHA	Community Rolling Action Plan (CoRAP) List	Caution
APS	No hazardous substance ratings associated with CAS numbers provided in manufacturer's safety data sheet.					
Aluminum Oxide Grit Blast	CAS number 1344-24-1 list as "Caution" for fibrous forms only.					
OXSILAN 9810/2	Nitric Acid	7697-37-2	Caution	US EPA	CERCLA hazardous substances	NA
•TT-C-490F				US Department of Homeland Security (DHS)	Chemicals of Concern (CoC)	NA
•TYPE IV, CLASS A, DEGREASE AND ABRASIVE BLAST and TYPE IV, CLASS B AND CLASS C				US EPA	EPCRA Section 304, Extremely Hazardous Substances, Reportable Quality (RQ)	NA
				US EPA	EPCRA Section 313, Toxic Chemicals	NA
				US EPA	Clean Air Act Section 112(R): Accidental Release Prevention/Risk Management Plan Rule	NA
				US EPA	EPA's List of Lists (LoL)	NA
				ASD	ASD-STAN Declarable Substances List	Caution
				US EPA	EPCRA Section 302, Extremely Hazardous Substances, Threshold Planning Quantity (TPQ)	NA

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Appendix B. Load versus Displacement Curves and Failure Surface Scans

Complete records of test data and failure surface scans may be accessed at the National Institute of Standards and Technology Dspace repository site <http://hdl.handle.net/11256/937>.

B.1 Substrate = 2024 T3 Aluminum, Sample Conditioning = Room Temperature (RT)

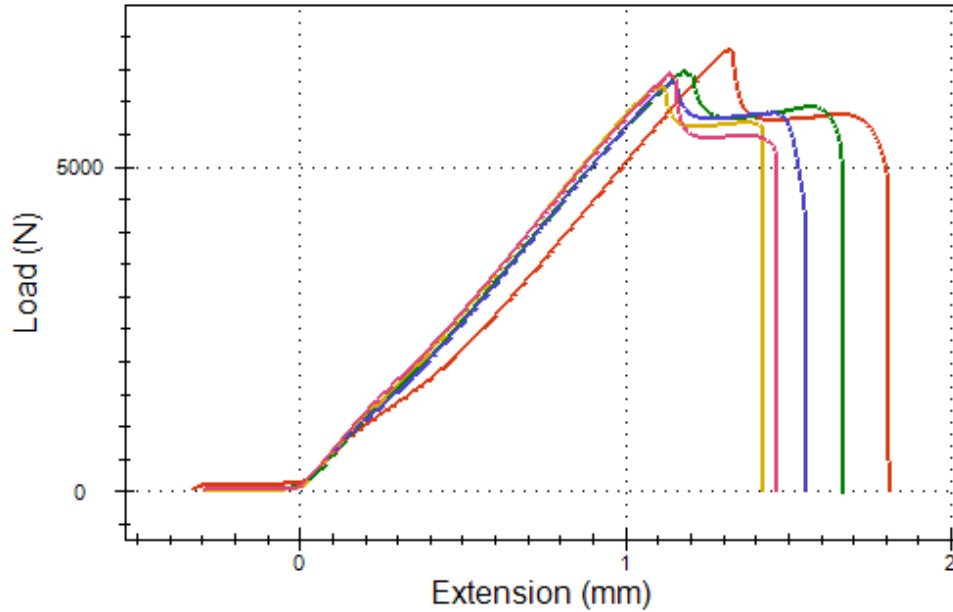


Fig. B-1 Load vs. extension (displacement) for single-lap-joints with aluminum oxide grit blast surface pretreatment. Materials Selection and Analysis Tool (MSAT) identifications (IDs) = 20150215–20150219, substrate = 2024 T3 aluminum, sample conditioning = RT.

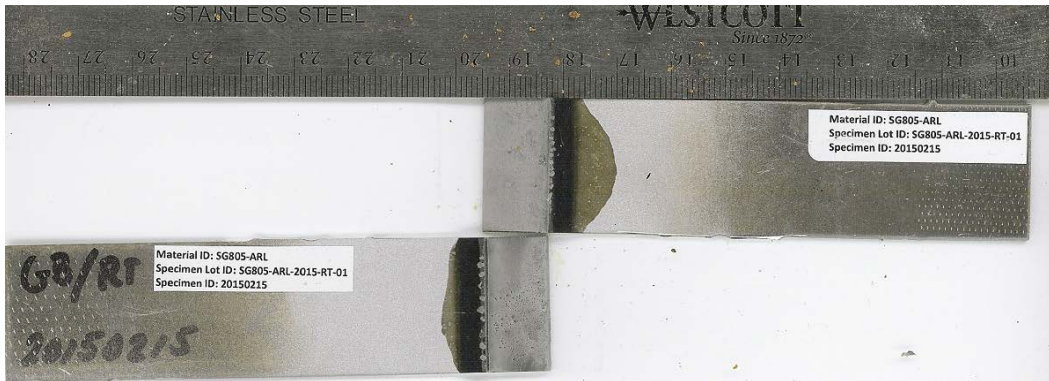


Fig. B-2 Failure surfaces for single-lap-joint with aluminum oxide grit blast surface pretreatment. MSAT ID = 20150215, substrate = 2024 T3 aluminum, and sample conditioning = RT. Mode-of-failure = mixed-mode (MM).

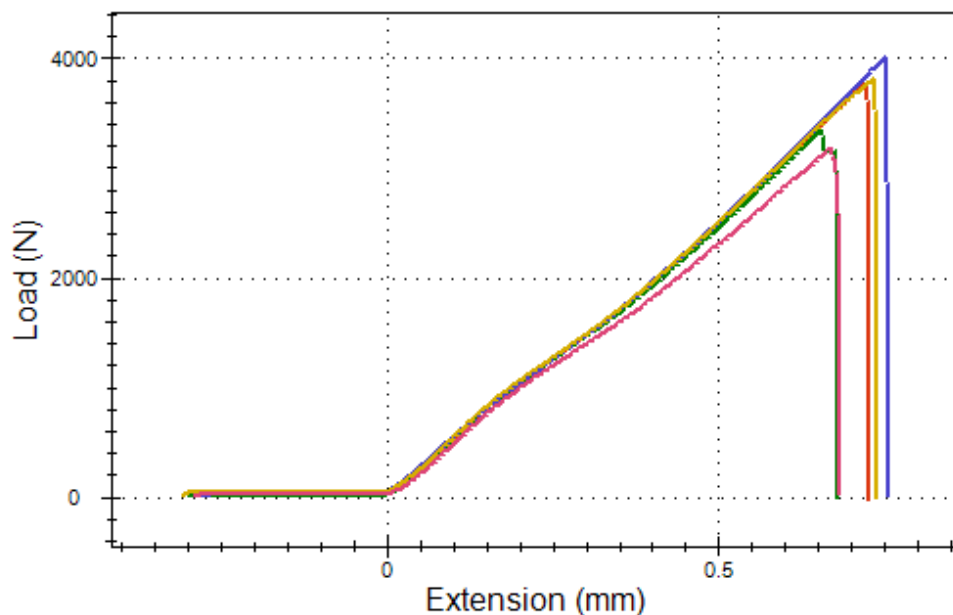


Fig. B-3 Load vs. extension (displacement) for single-lap-joints with 3-aminopropyltrimethoxysilane surface pretreatment. MSAT IDs = 20150225–20150229, substrate = 2024 T3 aluminum, and sample conditioning = RT.



Fig. B-4 Failure surfaces for single-lap-joint with 3-aminopropyltrimethoxysilane surface pretreatment. MSAT ID = 20150225, substrate = 2024 T3 aluminum, and sample conditioning = RT. Mode-of-failure = adhesive.

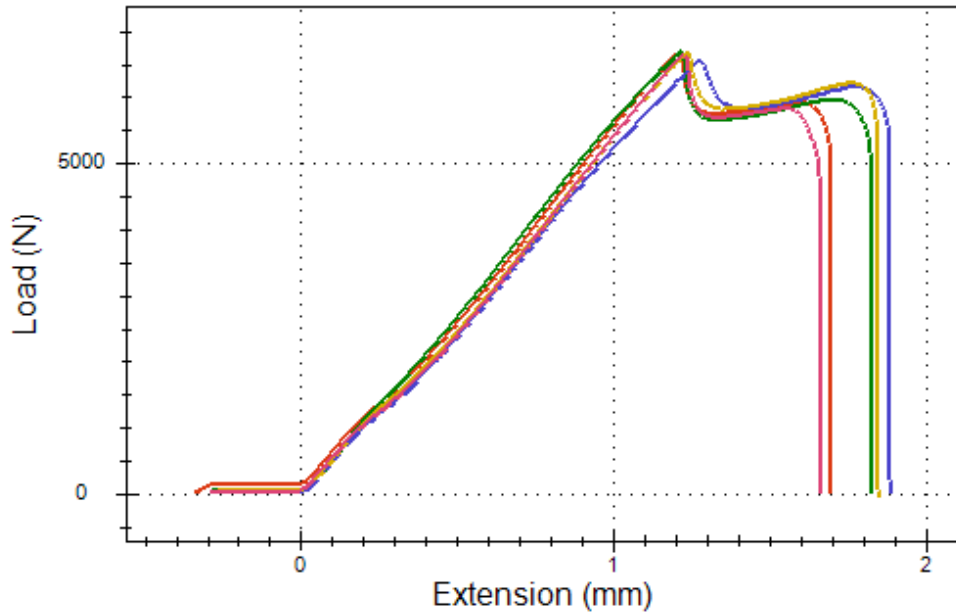


Fig. B-5 Load vs. extension (displacement) for single-lap-joints with AC-131 BB surface pretreatment. MSAT IDs = 20150235–20150239, substrate = 2024 T3 aluminum, and sample conditioning = RT.



Fig. B-6 Failure surfaces for single-lap-joint with AC-131 BB surface pretreatment. MSAT ID = 20150235, substrate = 2024 T3 aluminum, and sample conditioning = RT. Mode-of-failure = MM.

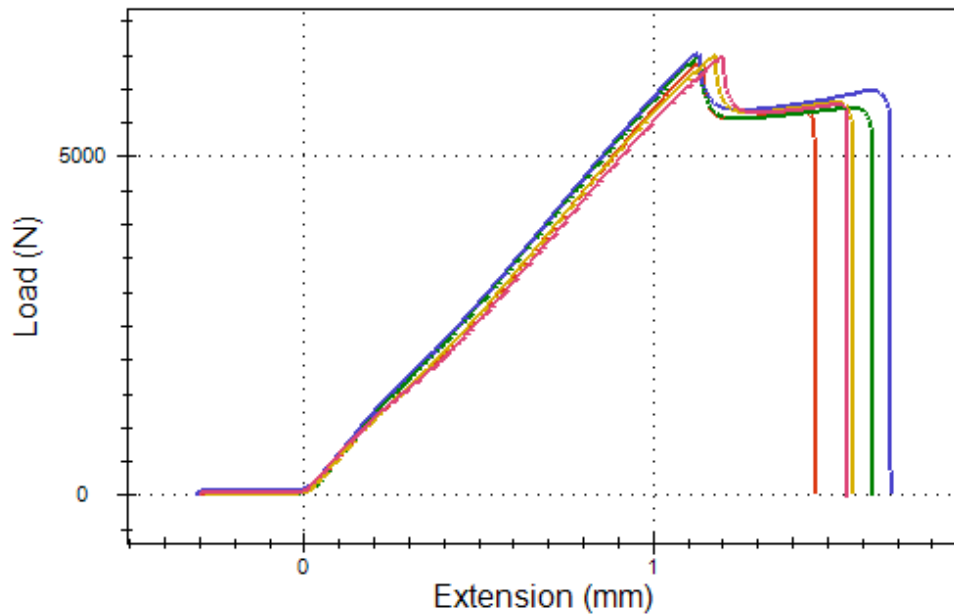


Fig. B-7 Load vs. extension (displacement) for single-lap-joints with SurTec 650 surface pretreatment. MSAT IDs = 20150245–20150249, substrate = 2024 T3 aluminum, and sample conditioning = RT.



Fig. B-8 Failure surfaces for single-lap-joint with SurTec 650 surface pretreatment. MSAT ID = 20150245, substrate = 2024 T3 aluminum, and sample conditioning = RT. Mode-of-failure = MM.

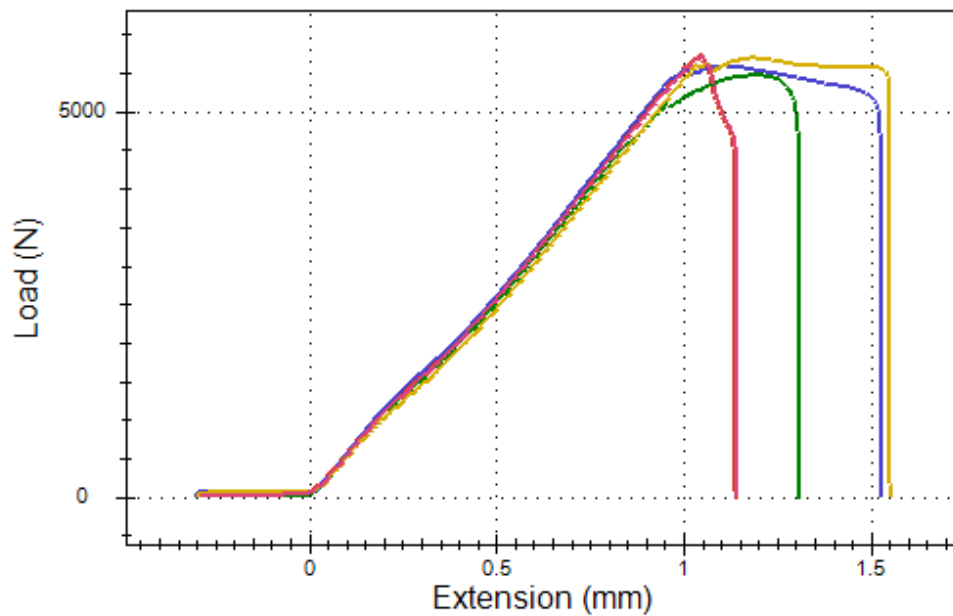


Fig. B-9 Load vs. extension (displacement) for single-lap-joints with Bonderite M-NT 5700 surface pretreatment. MSAT IDs = 20150255–20150259, substrate = 2024 T3 aluminum, and sample conditioning = RT.



Fig. B-10 Failure surfaces for single-lap-joint with Bonderite M-NT 5700 surface pretreatment. MSAT ID = 20150255, substrate = 2024 T3 aluminum, and sample conditioning = RT. Mode-of-failure = adhesive/ MM.

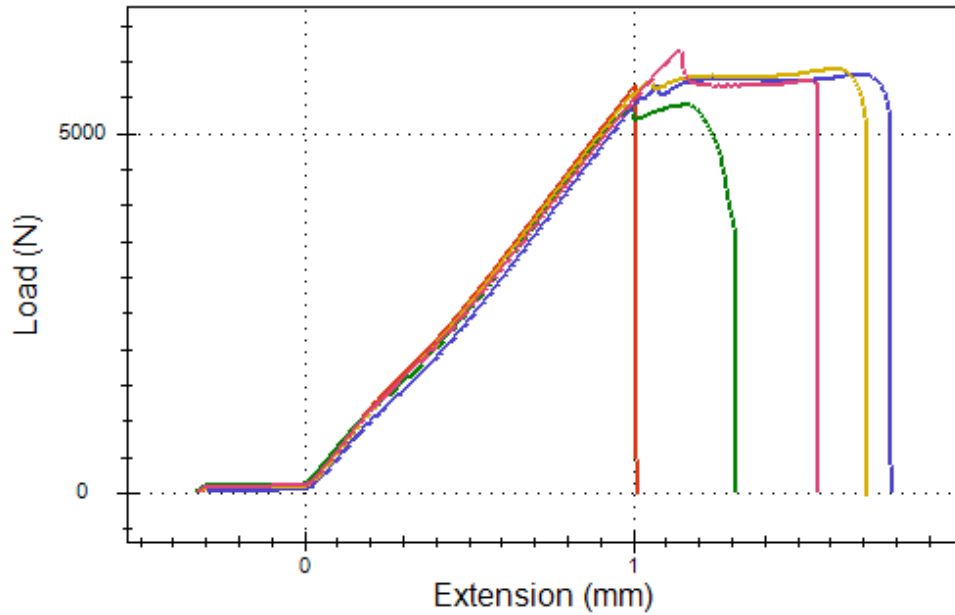


Fig. B-11 Load vs. extension (displacement) for single-lap-joints with Bonderite M-NT 7400 surface pretreatment. MSAT IDs = 20150265–20150269, substrate = 2024 T3 aluminum, and sample conditioning = RT.



Fig. B-12 Failure surfaces for single-lap-joint with Bonderite M-NT 7400 surface pretreatment. MSAT ID = 20150265, substrate = 2024 T3 aluminum, and sample conditioning = RT. Mode-of-failure = adhesive/ MM.

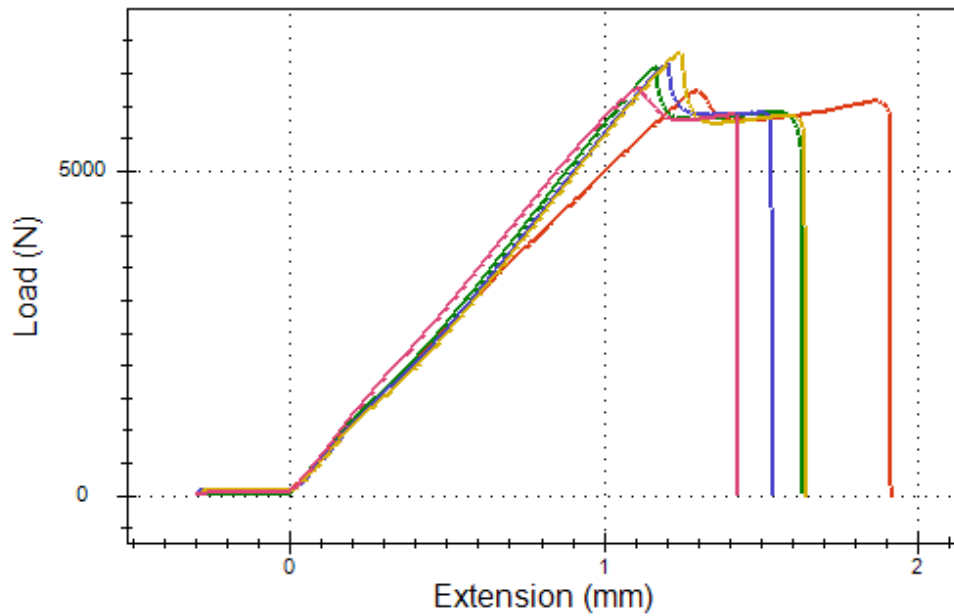


Fig. B-13 Load vs. extension (displacement) for single-lap-joints with XBOND 4000DM-DR surface pretreatment. MSAT IDs = 20150275–20150279, substrate = 2024 T3 aluminum, and sample conditioning = RT.



Fig. B-14 Failure surfaces for single-lap-joint with XBOND 4000DM-DR surface pretreatment. MSAT ID = 20150275, substrate = 2024 T3 aluminum, and sample conditioning = RT. Mode-of-failure = adhesive/ MM.

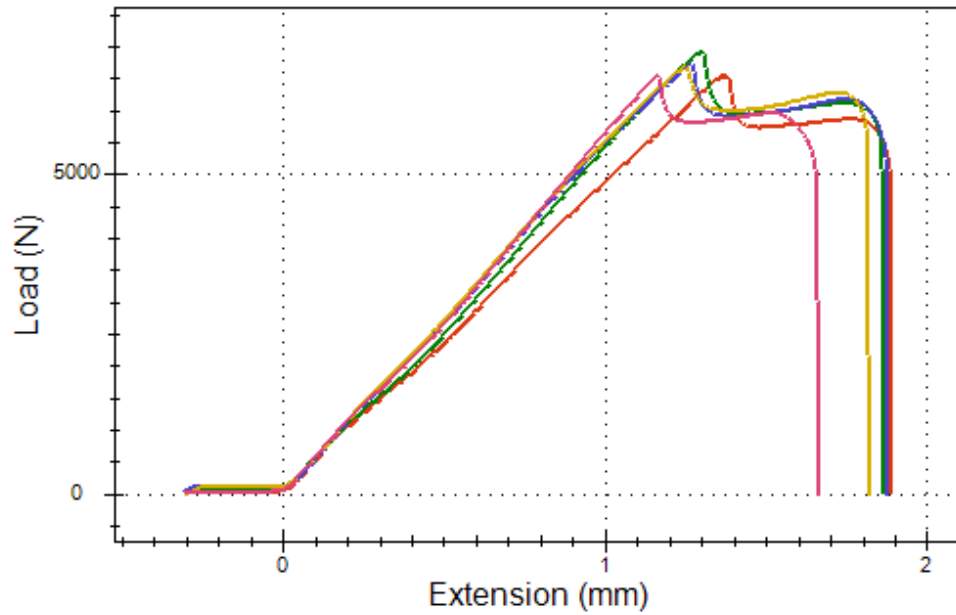


Fig. B-15 Load vs. extension (displacement) for single-lap-joints with ZIRCOBOND 4200 surface pretreatment. MSAT IDs = 20150285–20150289, substrate = 2024 T3 aluminum, and sample conditioning = RT.



Fig. B-16 Failure surfaces for single-lap-joint with ZIRCOBOND 4200 surface pretreatment. MSAT ID = 20150285, substrate = 2024 T3 aluminum, and sample conditioning = RT. Mode-of-failure = MM.

B.2 Substrate = 2024 T3 Aluminum, Sample Conditioning = Hot/Wet (H/W)

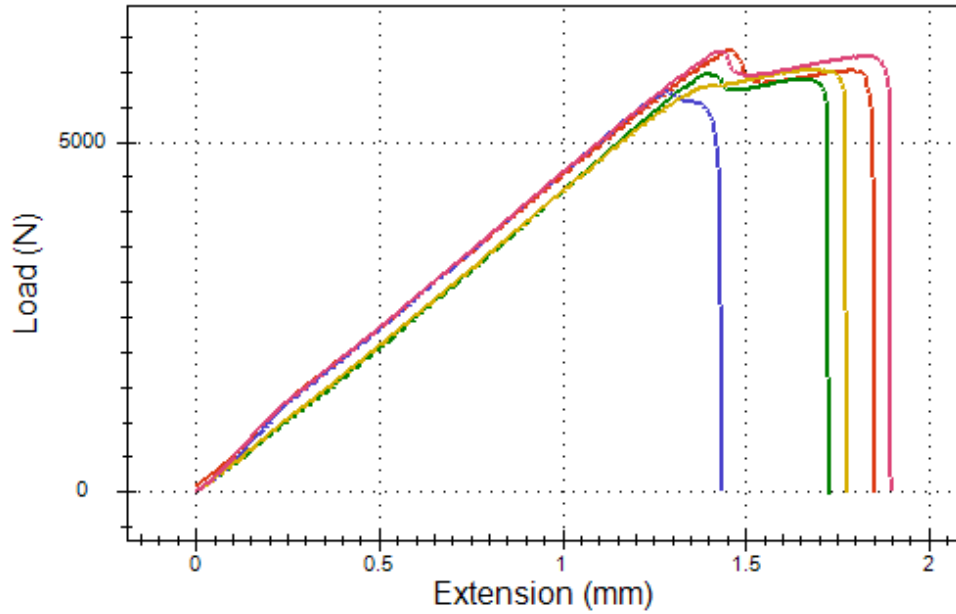


Fig. B-17 Load vs. extension (displacement) for single-lap-joints with aluminum oxide grit blast surface pretreatment. MSAT IDs = 20150220–20150224, substrate = 2024 T3 aluminum, and sample conditioning = H/W.



Fig. B-18 Failure surfaces for single-lap-joint with aluminum oxide grit blast surface pretreatment. MSAT ID = 20150222, substrate = 2024 T3 aluminum, and sample conditioning = H/W. Mode-of-failure = MM.

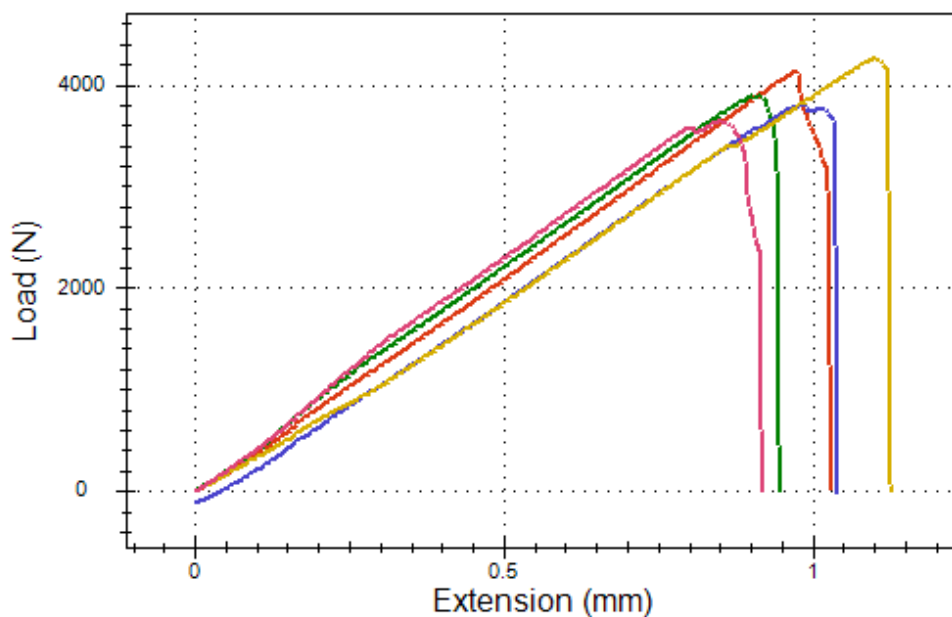


Fig. B-19 Load vs. extension (displacement) for single-lap-joints with 3-aminopropyltrimethoxysilane surface pretreatment. MSAT IDs = 20150230–20150234, substrate = 2024 T3 aluminum, and sample conditioning = H/W.

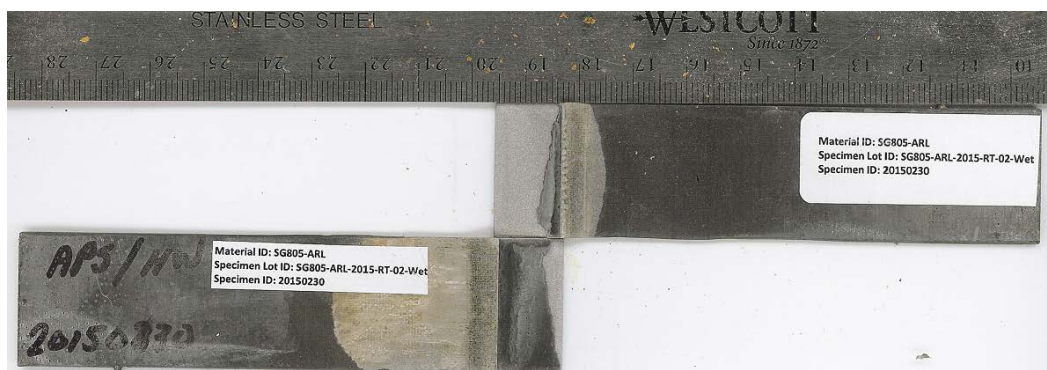


Fig. B-20 Failure surfaces for single-lap-joint with 3-aminopropyltrimethoxysilane surface pretreatment. MSAT ID = 20150230, substrate = 2024 T3 aluminum, and sample conditioning = H/W. Mode-of-failure = adhesive.

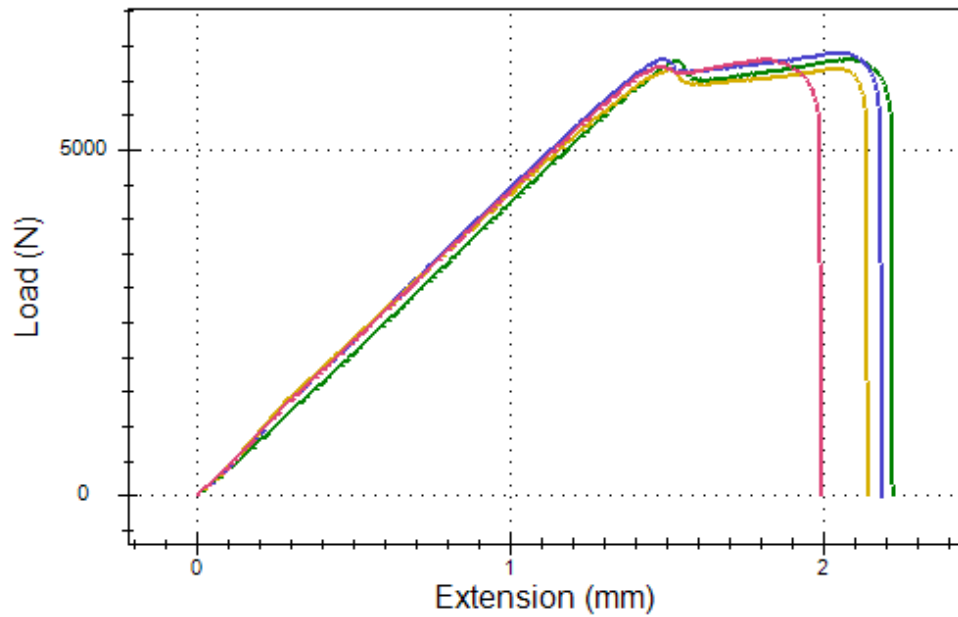


Fig. B-21 Load vs. extension (displacement) for single-lap-joints with AC-131 BB surface pretreatment. MSAT IDs = 20150240–20150244, substrate = 2024 T3 aluminum, and sample conditioning = H/W.



Fig. B-22 Failure surfaces for single-lap-joint with AC-131 BB surface pretreatment. MSAT ID = 20150240, substrate = 2024 T3 aluminum, and sample conditioning = H/W. Mode-of-failure = MM.

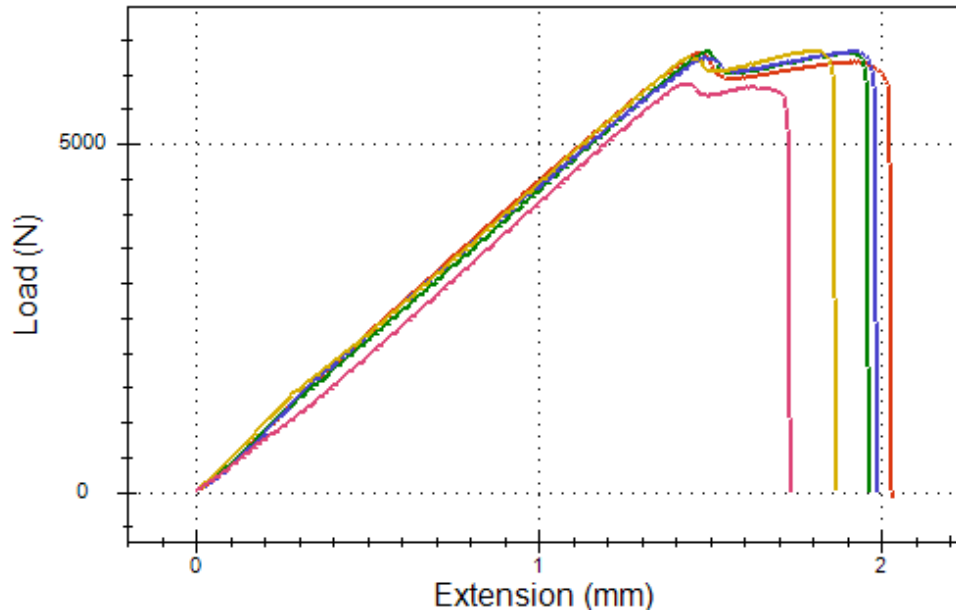


Fig. B-23 Load vs. extension (displacement) for single-lap-joints with SurTec 650 surface pretreatment. MSAT IDs = 20150250–20150254, substrate = 2024 T3 aluminum, and sample conditioning = H/W.



Fig. B-24 Failure surfaces for single-lap-joint with SurTec 650 surface pretreatment. MSAT ID = 20150250, substrate = 2024 T3 aluminum, and sample conditioning = H/W. Mode-of-failure = MM.

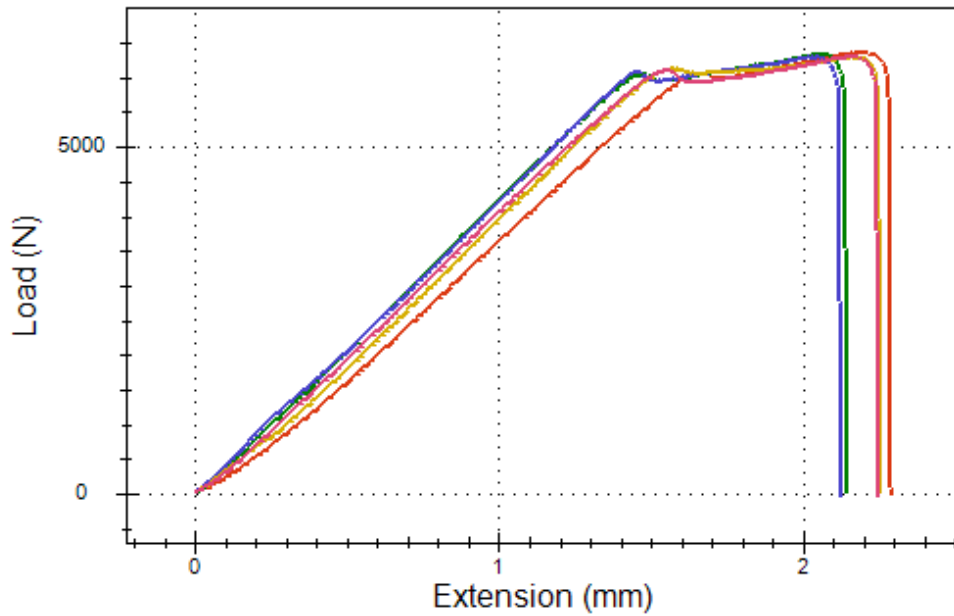


Fig. B-25 Load vs. extension (displacement) for single-lap-joints with Bonderite M-NT 5700 surface pretreatment. MSAT IDs = 20150260–20150264, substrate = 2024 T3 aluminum, and sample conditioning = H/W.



Fig. B-26 Failure surfaces for single-lap-joint with Bonderite M-NT 5700 surface pretreatment. MSAT ID = 20150260, substrate = 2024 T3 aluminum, and sample conditioning = H/W. Mode-of-failure = adhesive/ MM.

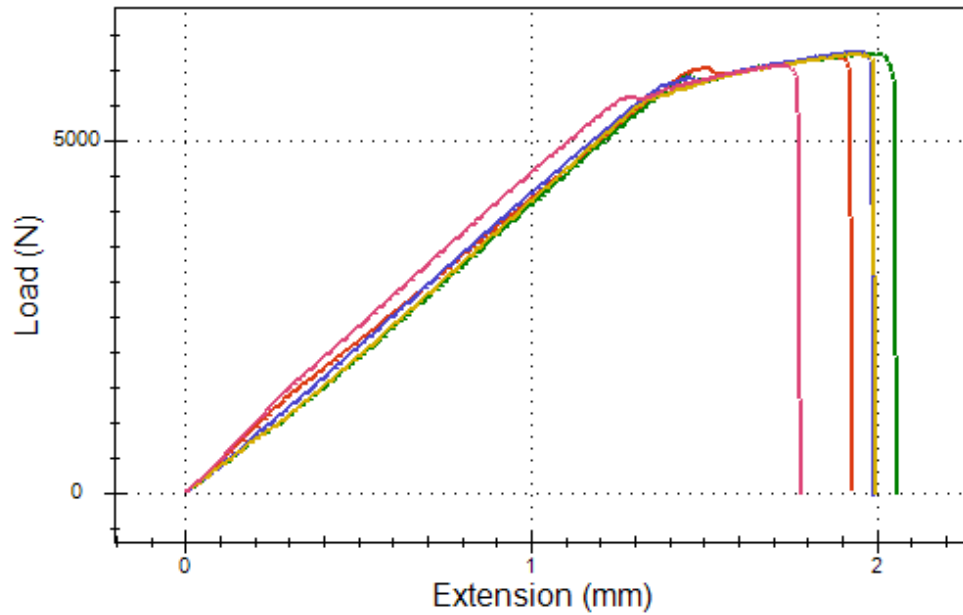


Fig. B-27 Load vs. extension (displacement) for single-lap-joints with Bonderite M-NT 7400 surface pretreatment. MSAT IDs = 20150270–20150274, substrate = 2024 T3 aluminum, and sample conditioning = H/W.



Fig. B-28 Failure surfaces for single-lap-joint with Bonderite M-NT 7400 surface pretreatment. MSAT ID = 20150270, substrate = 2024 T3 aluminum, and sample conditioning = H/W. Mode-of-failure = adhesive/ MM.

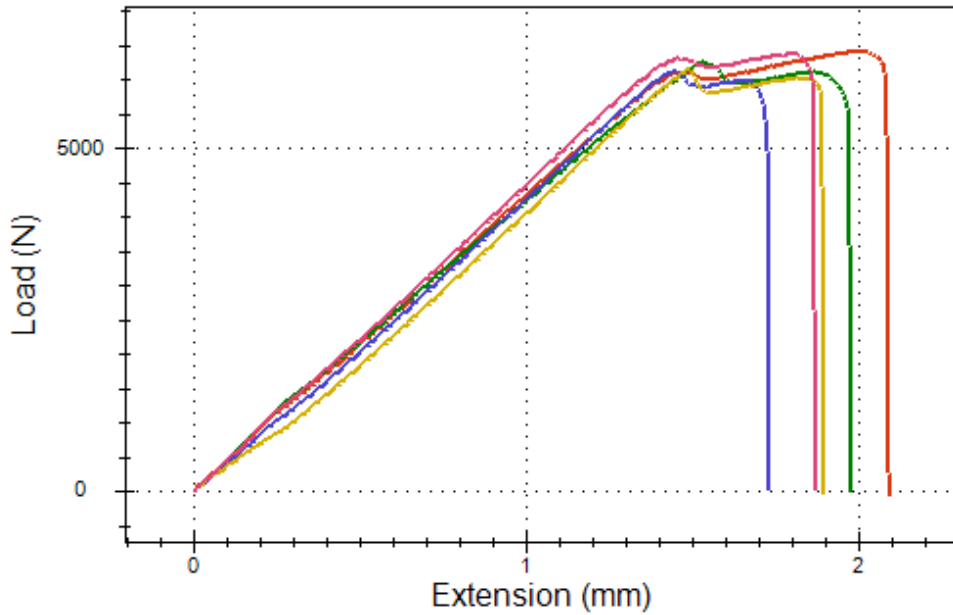


Fig. B-29 Load vs. extension (displacement) for single-lap-joints with XBOND 4000DM-DR surface pretreatment. MSAT IDs = 20150280–20150284, substrate = 2024 T3 aluminum, and sample conditioning = H/W.



Fig. B-30 Failure surfaces for single-lap-joint with XBOND 4000DM-DR surface pretreatment. MSAT ID = 20150280, substrate = 2024 T3 aluminum, and sample conditioning = H/W. Mode-of-failure = adhesive/ MM.

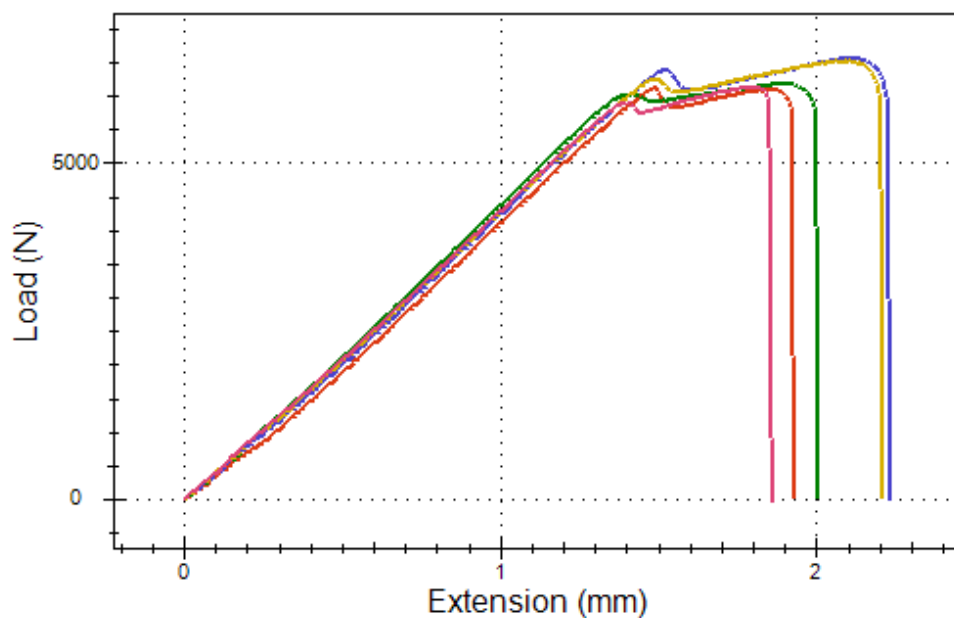


Fig. B-31 Load vs. extension (displacement) for single-lap-joints with ZIRCOBOND 4200 surface pretreatment. MSAT IDs = 20150290–20150294, substrate = 2024 T3 aluminum, and sample conditioning = H/W.



Fig. B-32 Failure surfaces for single-lap-joint with ZIRCOBOND 4200 surface pretreatment. MSAT ID = 20150290, substrate = 2024 T3 aluminum, and sample conditioning = H/W. Mode-of-failure = MM.

B.3 Substrate = 2024 T3 Aluminum, Test Conditions = Elevated Temperature (ET)

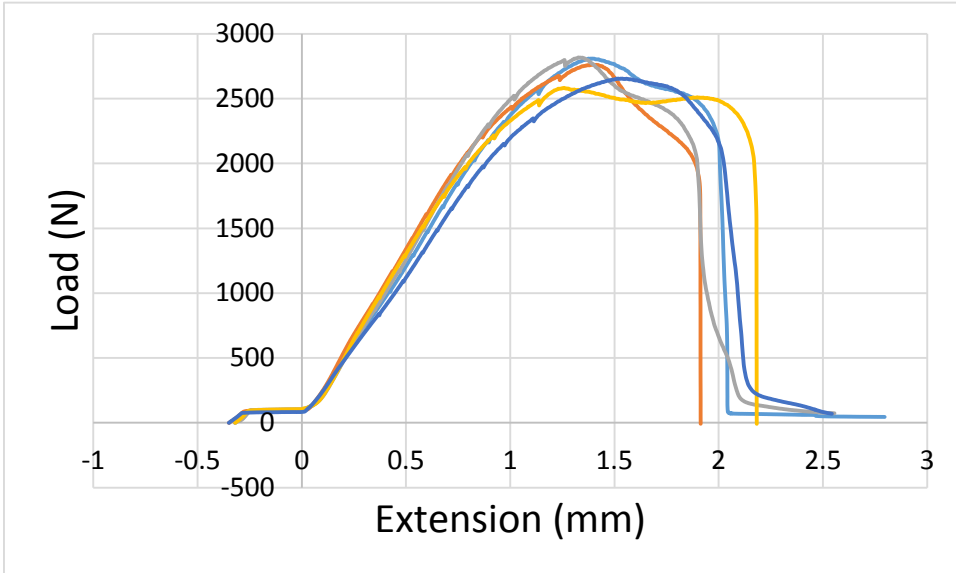


Fig. B-33 Load vs. extension (displacement) for single-lap-joints with aluminum oxide grit blast surface pretreatment. MSAT IDs = 20150871–20150875, substrate = 2024 T3 aluminum, and test conditions = ET.



Fig. B-34 Failure surfaces for single-lap-joint with aluminum oxide grit blast surface pretreatment. MSAT ID = 20150871, substrate = 2024 T3 aluminum, and test conditions = ET. Mode-of-failure = MM.

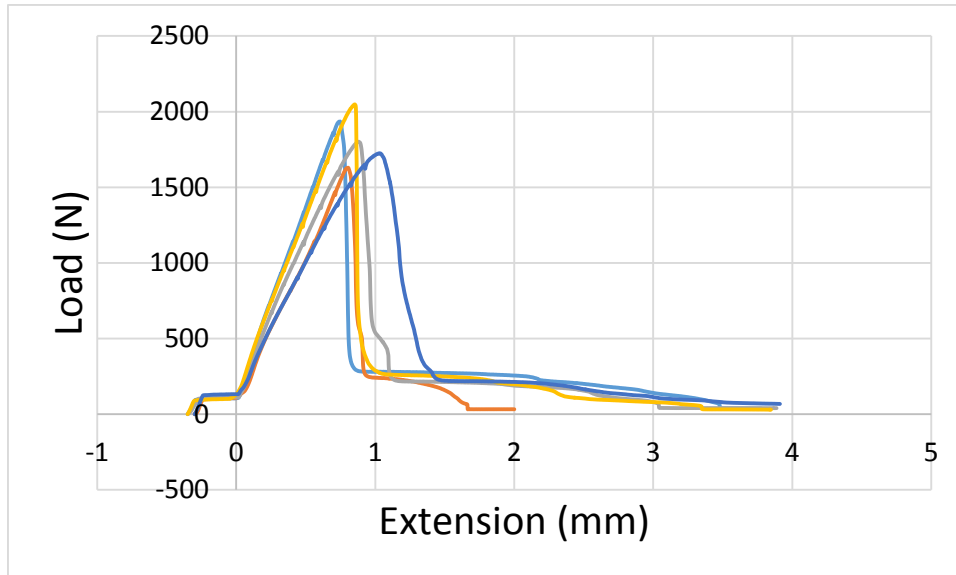


Fig. B-35 Load vs. extension (displacement) for single-lap-joints with 3-aminopropyltrimethoxysilane surface pretreatment. MSAT IDs = 20150388–20150392, substrate = 2024 T3 aluminum, and test conditions = ET.



Fig. B-36 Failure surfaces for single-lap-joint with 3-aminopropyltrimethoxysilane pretreatment. MSAT ID = 20150388, substrate = 2024 T3 aluminum, and test conditions = ET Mode-of-failure = adhesive.

B.4 Substrate = 1010 Steel, Sample Conditioning = RT

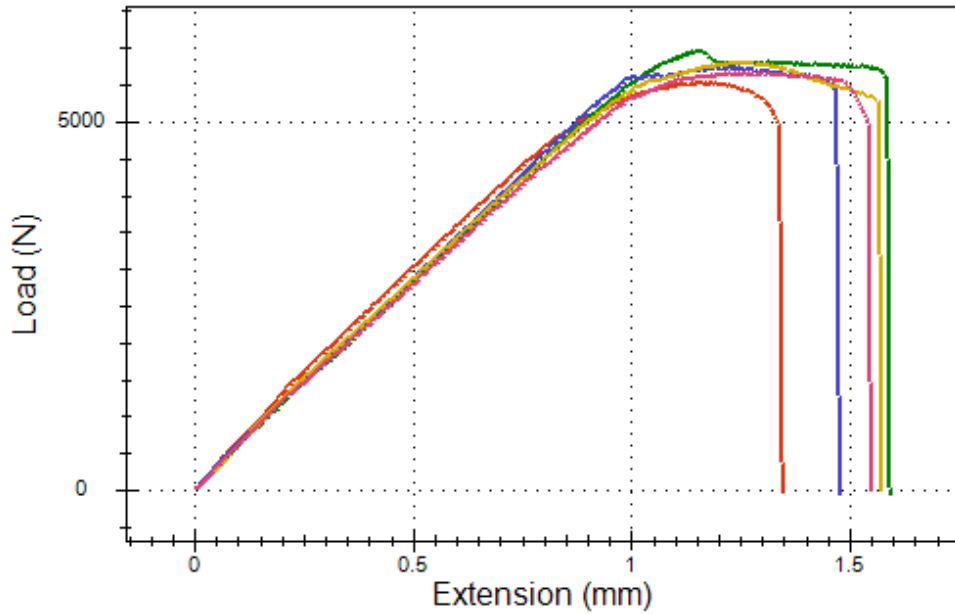


Fig. B-37 Load vs. extension (displacement) for single-lap-joints with AC-131 BB surface pretreatment. MSAT IDs = 20150438–20150442, substrate = 1010 steel, and sample conditioning = RT.



Fig. B-38 Failure surfaces for single-lap-joint with AC-131 BB surface pretreatment. MSAT ID = 20150438, substrate = 1010 steel, sample conditioning = RT. Mode-of-failure = MM.

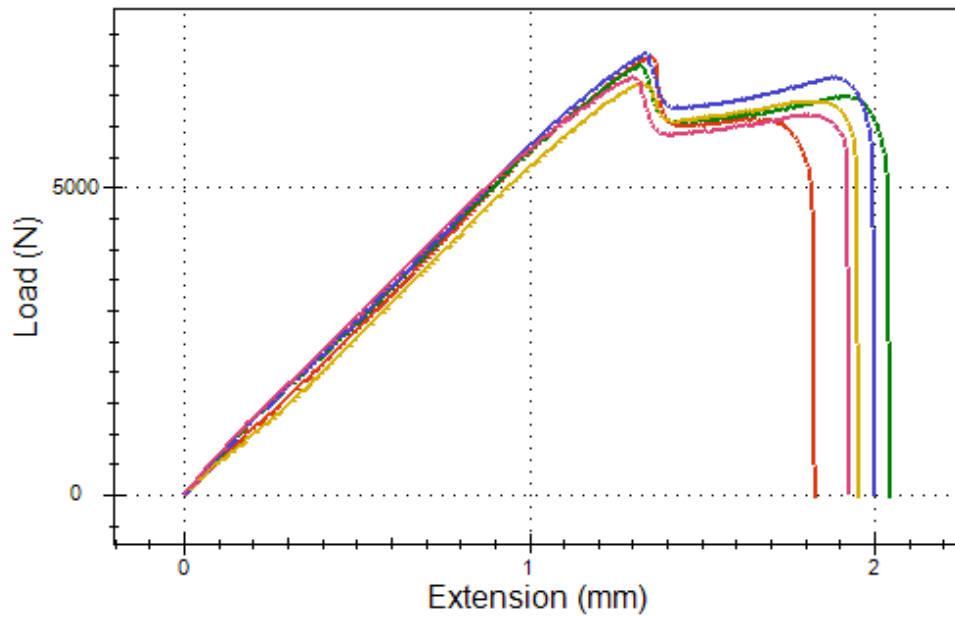


Fig. B-39 Load vs. extension (displacement) for single-lap-joints with SurTec 650 surface pretreatment. MSAT IDs = 20150448–20150452, substrate = 1010 steel, and sample conditioning = RT.



Fig. B-40 Failure surfaces for single-lap-joint with SurTec 650 surface pretreatment. MSAT ID = 20150448, substrate = 1010 steel, and sample conditioning = RT. Mode-of-failure = MM.

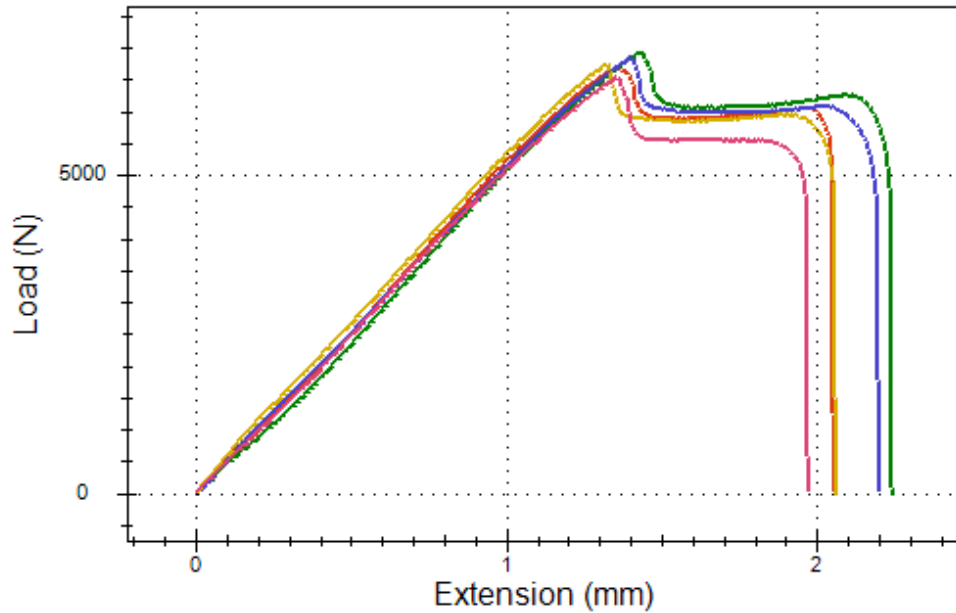


Fig. B-41 Load vs. extension (displacement) for single-lap-joints with Bonderite M-NT 5700 surface pretreatment. MSAT IDs = 20150458–20150462, substrate = 1010 steel, sample conditioning = RT.



Fig. B-42 Failure surfaces for single-lap-joint with Bonderite M-NT 5700 surface pretreatment. MSAT ID = 20150461, substrate = 1010 steel, and sample conditioning = RT. Mode-of-failure = MM.

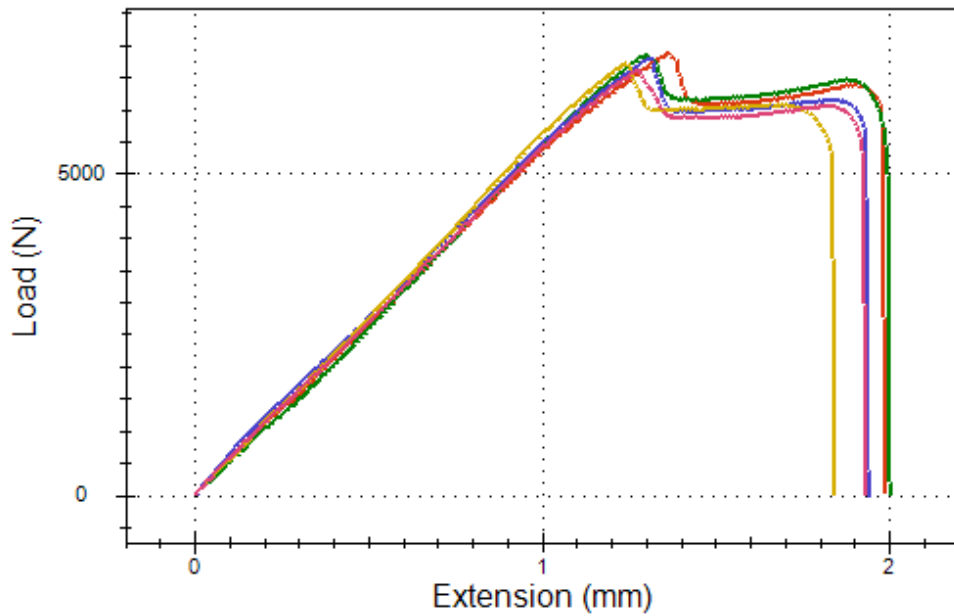


Fig. B-43 Load vs. extension (displacement) for single-lap-joints with Bonderite M-NT 7400 surface pretreatment. MSAT IDs = 20150468–20150472, substrate = 1010 steel, and sample conditioning = RT.

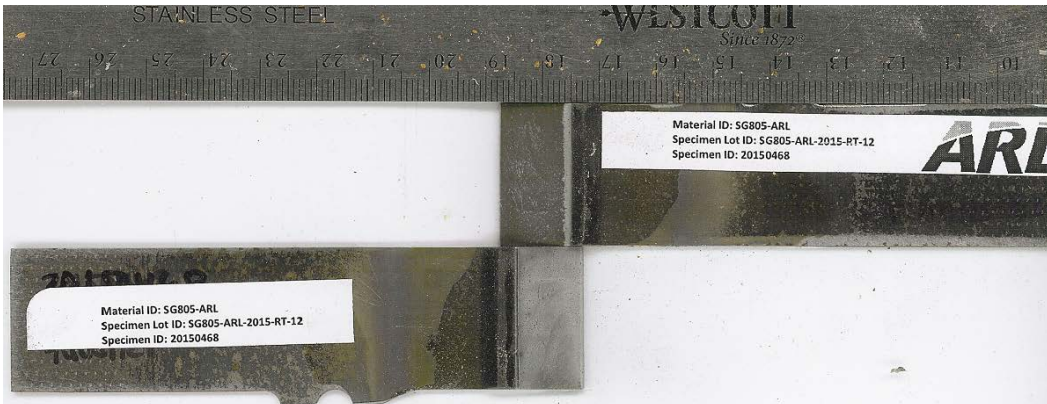


Fig. B-44 Failure surfaces for single-lap-joint with Bonderite M-NT 7400 surface pretreatment. MSAT ID = 20150468, substrate = 1010 steel, and sample conditioning = RT. Mode-of-failure = MM.

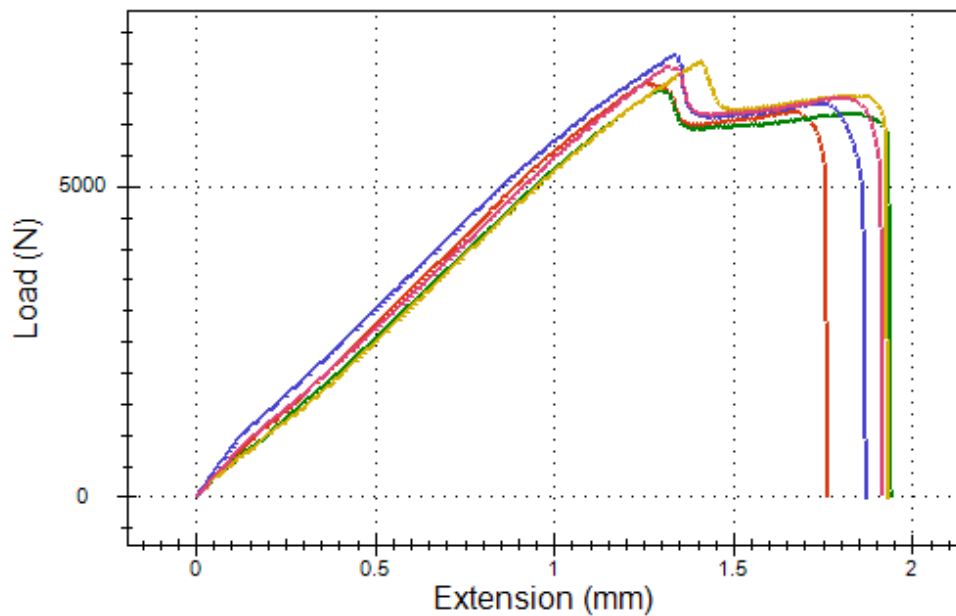


Fig. B-45 Load vs. extension (displacement) for single-lap-joints with XBOND 4000DM-DR surface pretreatment. MSAT IDs = 20150478–20150482, substrate = 1010 steel, and sample conditioning = RT.



Fig. B-46 Failure surfaces for single-lap-joint with XBOND 4000DM-DR surface pretreatment. MSAT ID = 20150478, substrate = 1010 steel, and sample conditioning = RT. Mode-of-failure = MM.

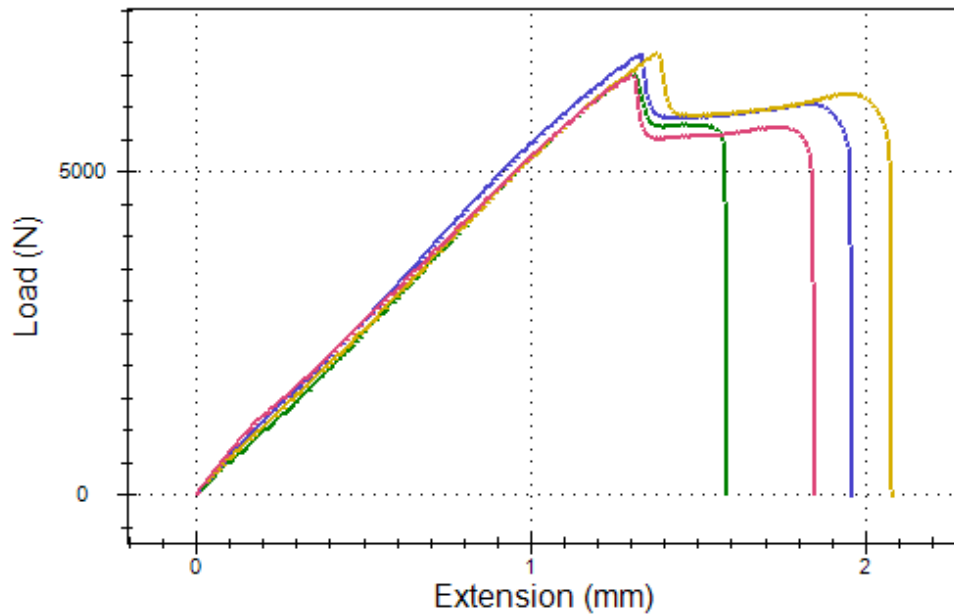


Fig. B-47 Load vs. extension (displacement) for single-lap-joints with ZIRCOBOND 4200 surface pretreatment. MSAT IDs = 20150488–20150492, substrate = 1010 steel, and sample conditioning = RT.



Fig. B-48 Failure surfaces for single-lap-joint with ZIRCOBOND 4200 surface pretreatment. MSAT ID = 20150492, substrate = 1010 steel, and sample conditioning = RT. Mode-of-failure = MM.

B.5 Substrate = 1010 Steel, Sample Conditioning = H/W

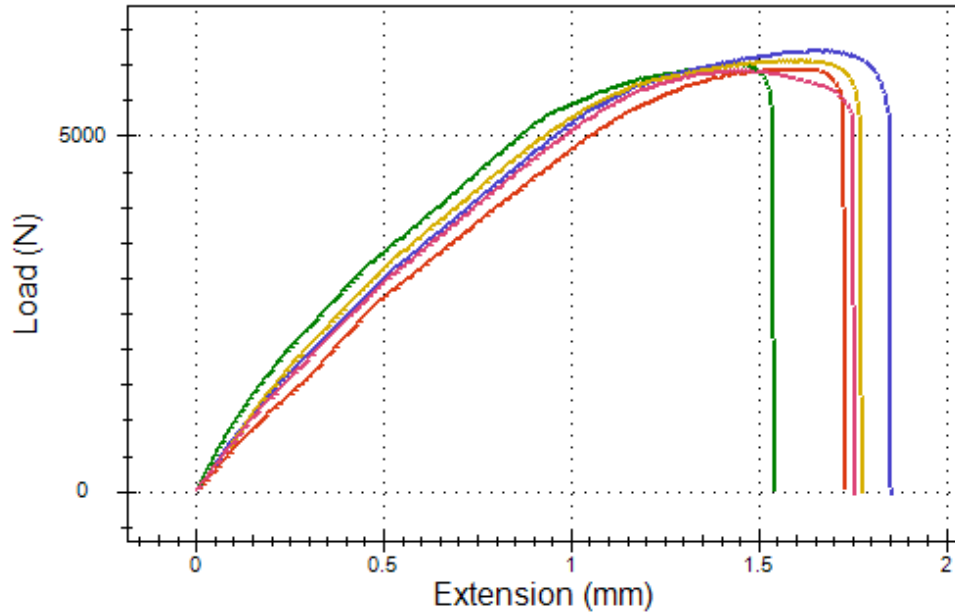


Fig. B-49 Load vs. extension (displacement) for single-lap-joints with AC-131 BB surface pretreatment. MSAT IDs = 20150443–20150447, substrate = 1010 steel, and sample conditioning = H/W.



Fig. B-50 Failure surfaces for single-lap-joint with AC-131 BB surface pretreatment. MSAT ID = 20150443, substrate = 1010 steel, and sample conditioning = H/W. Mode-of-failure = MM.

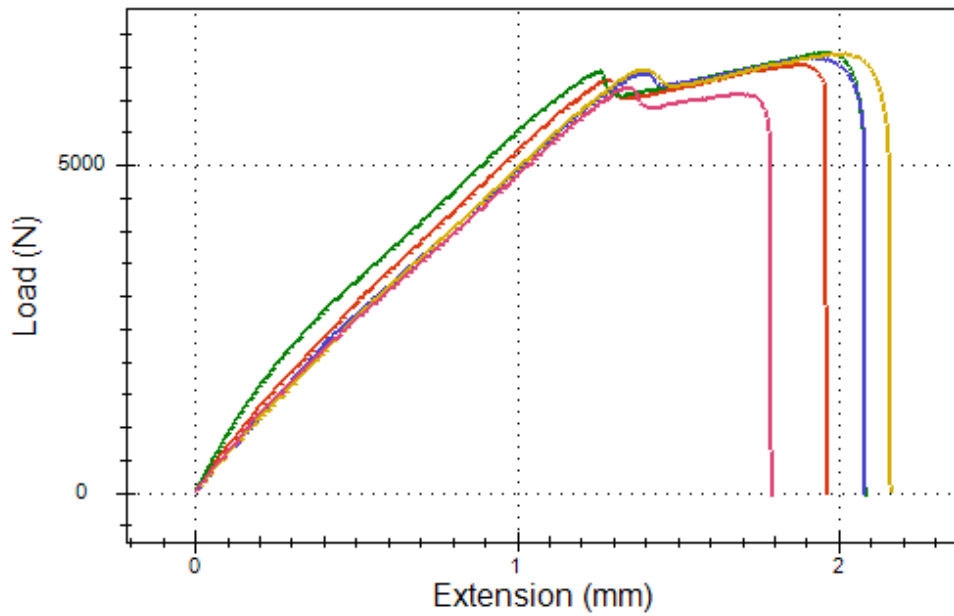


Fig. B-51 Load vs. extension (displacement) for single-lap-joints with SurTec 650 surface pretreatment. MSAT IDs = 20150453–20150457, substrate = 1010 steel, and sample conditioning = H/W.



Fig. B-52 Failure surfaces for single-lap-joint with SurTec 650 surface pretreatment. MSAT ID = 20150453, substrate = 1010 steel, and sample conditioning = H/W. Mode-of-failure = MM.

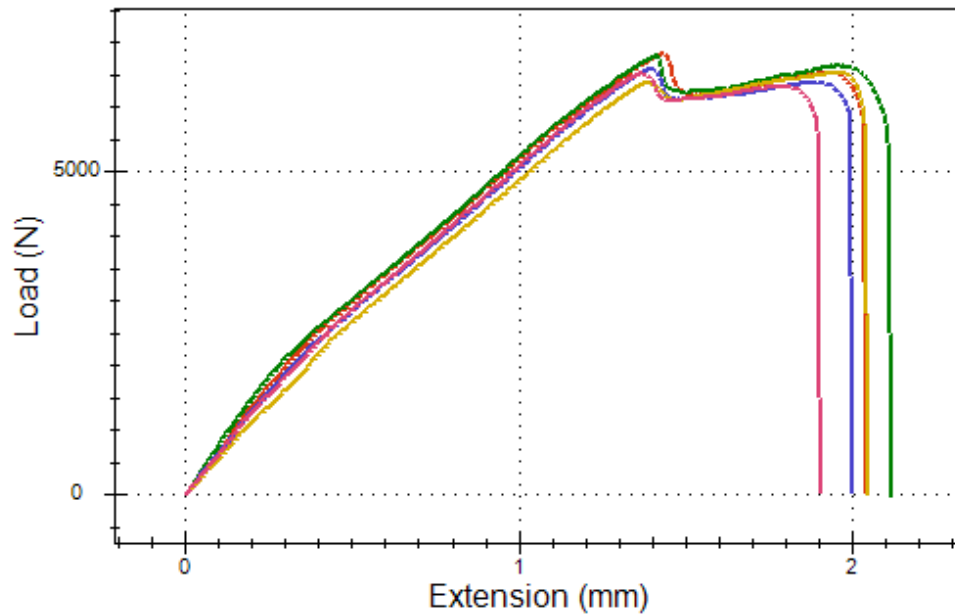


Fig. B-53 Load vs. extension (displacement) for single-lap-joints with Bonderite M-NT 5700 surface pretreatment. MSAT IDs = 20150463–20150467, substrate = 1010 steel, and sample conditioning = H/W.



Fig. B-54 Failure surfaces for single-lap-joint with Bonderite M-NT 5700 surface pretreatment. MSAT ID = 20150464, substrate = 1010 steel, and sample conditioning = H/W. Mode-of-failure = MM.

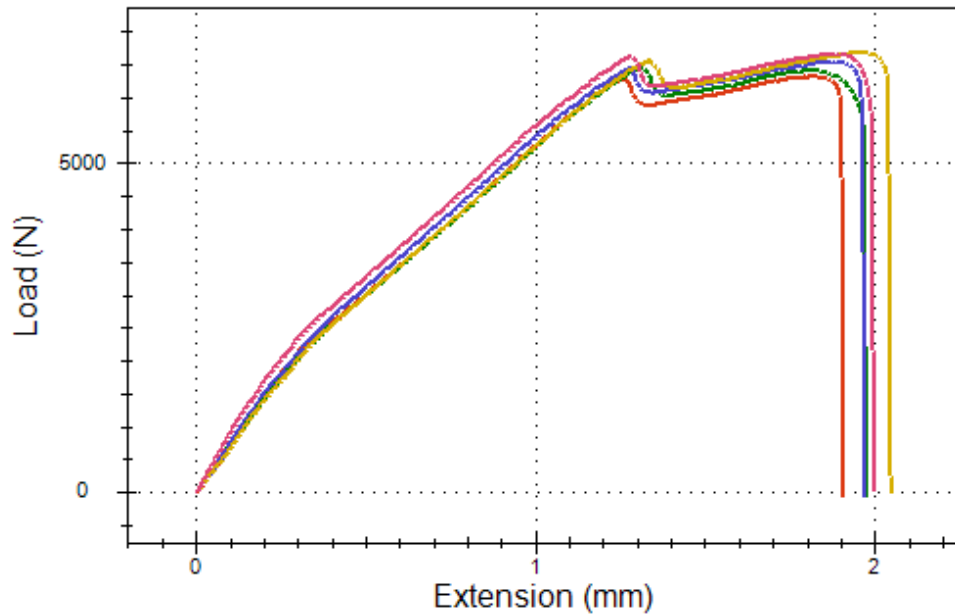


Fig. B-55 Load vs. extension (displacement) for single-lap-joints with Bonderite M-NT 7400 surface pretreatment. MSAT IDs = 20150473–20150477, substrate = 1010 steel, and sample conditioning = H/W.



Fig. B-56 Failure surfaces for single-lap-joint with Bonderite M-NT 7400 surface pretreatment. MSAT ID = 20150473, substrate = 1010 steel, and sample conditioning = H/W. Mode-of-failure = MM.

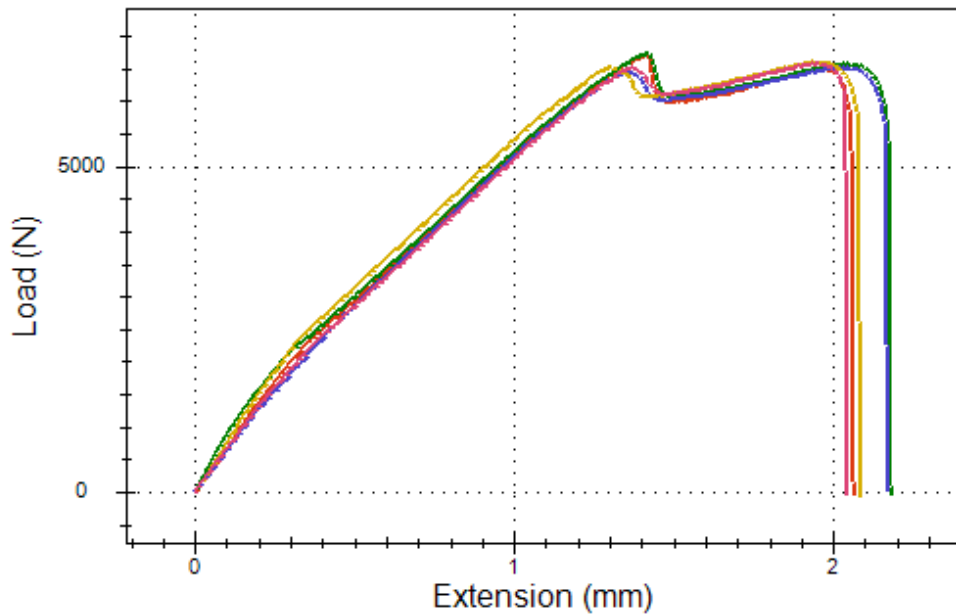


Fig. B-57 Load vs. extension (displacement) for single-lap-joints with XBOND 4000DM-DR surface pretreatment. MSAT IDs = 20150483–20150487, substrate = 1010 steel, and sample conditioning = H/W.



Fig. B-58 Failure surfaces for single-lap-joint with XBOND 4000DM-DR surface pretreatment. MSAT ID = 20150485, substrate = 1010 steel, and sample conditioning = H/W. Mode-of-failure = MM.

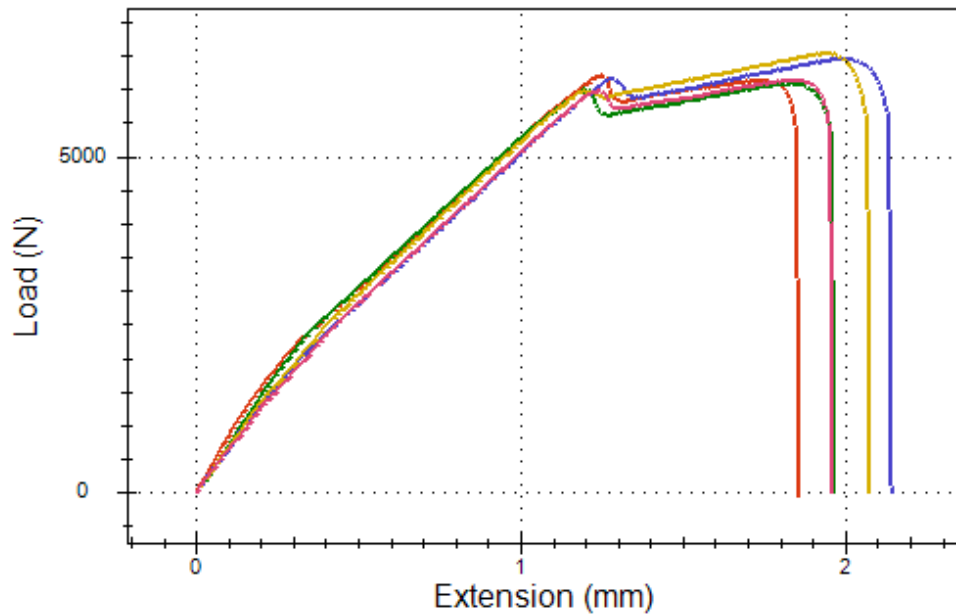


Fig. B-59 Load vs. extension (displacement) for single-lap-joints with ZIRCOBOND 4200 surface pretreatment. MSAT IDs = 20150493–20150497, substrate = 1010 steel, and sample conditioning = H/W.



Fig. B-60 Failure surfaces for single-lap-joint with ZIRCOBOND 4200 surface pretreatment. MSAT ID = 20150496, substrate = 1010 steel, and sample conditioning = H/W. Mode-of-failure = MM.

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Appendix C. Supporting Digital File Archive Index

Table C-1 provides the reader with a reference list and URL (uniform resource locator) links to experimental data and supporting metadata descriptors archived in the National Institute of Standards and Technology (NIST) DSpace repository <http://hdl.handle.net/11256/937>.

Table C-1 Supporting data collection

File name^a	Description
Aluminum joints	Size: 301.1 MB Format: MS Excel, TIFF, PDF Lab notes, load versus displacement data, and failure surface images
Steel joints	Size: 206.9 MB Format: MS Excel, TIFF, PDF Lab notes, load versus displacement data, and failure surface images
DMA	Size: 2.213 MB Format: MS Excel and csv text Dynamic mechanical analysis storage and loss modulus
Technical data sheets	Size: 5.910 MB Format: PDF Manufacturer technical and safety data sheets
Restricted substances	Size: 22.38 KB Format: MS Excel Currents and pending environmental legislations
Calibration certs	Size: 9.393 MB Format: PDF Calibration certificates for test equipment used

^aAbbreviated file name as it appears on NIST site.

List of Symbols, Abbreviations, and Acronyms

A	bonded area
ADH	adhesive mode-of-failure
APS	3-aminopropyltrimethoxysilane
ASTM	American Society for Testing and Materials
COH	cohesive mode-of-failure
cond.	conditioning
csv	comma-separated values
DMA	dynamic mechanical analysis
DOD	Department of Defense
dpi	dots per inch
E'	storage modulus
E''	loss modulus
ET	elevated temperature
ID	sample identification number
min	minute
mm	millimeter
MM	mixed-mode mode-of-failure
MPa	megapascal
MS	Microsoft
MSAT	Materials Selection and Analysis Tool
NIST	National Institute of Standards and Technology
P_{\max}	maximum load
PDF	portable document format
RT	room temperature
S_{\max}	maximum strength

T_g	glass transition temperature
$\tan \delta$	loss tangent
TIFF	tagged image file format
txt	text file format
URL	uniform resource locator
ZIP	archive file format

1 DEFENSE TECHNICAL
(PDF) INFORMATION CTR
DTIC OCA

2 DIRECTOR
(PDF) US ARMY RESEARCH LAB
RDRL CIO L
IMAL HRA MAIL & RECORDS
MGMT

1 GOVT PRINTG OFC
(PDF) A MALHOTRA

4 DIR USARL
(PDF) RDRL WMM C
D DESCHEPPER
D FLANAGAN
R JENSEN
J SNYDER

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