

**ARL-TR-7696** • JUNE 2016



# Multivariate Analysis of High Through-Put Adhesively Bonded Single Lap Joints: Experimental and Workflow Protocols

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

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by Robert E Jensen, Daniel C DeSchepper, and David P Flanagan Weapons and Materials Research Directorate (ARL)

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1. REPORT DATE (	(DD-MM-YYYY)	2. REPORT TYPE			3. DATES COVERED (From - To)
June 2016		Final			May 2012–March 2016
4. TITLE AND SUB	TITLE				5a. CONTRACT NUMBER
Multivariate A	nalysis of High T	hrough-Put Adhesi	vely Bonded Sing	gle Lap	
Joints: Experin	nental and working	low Protocols			5b. GRANT NUMBER
					5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)	on Danial C Dasa	hoppor and David	<b>P</b> Elanagan		5d. PROJECT NUMBER
Robert E Jense	in, Daniel C Dese	hepper, and David	1 Tanagan		5e. TASK NUMBER
					5f. WORK UNIT NUMBER
7. PERFORMING (	ORGANIZATION NAME	E(S) AND ADDRESS(ES)			8. PERFORMING ORGANIZATION REPORT NUMBER
US Army Rese	earch Laboratory	., .,			
ATTN: RDRL	-WMM-C				ARL-TR-7696
Aberdeen Prov	ving Ground, MD	21005-5069			
9. SPONSORING/I	MONITORING AGENC	Y NAME(S) AND ADDRE	SS(ES)		10. SPONSOR/MONITOR'S ACRONYM(S)
					11. SPONSOR/MONITOR'S REPORT NUMBER(S)
12. DISTRIBUTION	N/AVAILABILITY STATE	MENT			
Approved for	public release; dis	tribution is unlimite	ed.		
13. SUPPLEMENT	ARY NOTES				
14. ABSTRACT					
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for supplemen	tal release of all r	aw and analyzed e	xperimental data	, along with a	associated metadata, in downloadable format
that is included	d with this report.				
15. SUBJECT TERM	٨S				
single lap joint	t, relational databa	ase, informatics, wo	orkflow, adhesive		
16. SECURITY CLA	SSIFICATION OF:		17. LIMITATION OF	18. NUMBER OF	19a. NAME OF RESPONSIBLE PERSON
			ABSTRACT	PAGES	Robert E Jensen
a. REPORT	D. ABSTRACT	C. THIS PAGE	TIT	38	19D. IELEPHONE NUMBER (Include area code)
Unclassified	Unclassified	Unclassified	00	50	410-306-1910

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## Acknowledgments

This research was supported in part by the US Army Education Outreach - Gains in the Education of Math and Science (GEMS) Program at the US Army Research Laboratory (ARL) and was administered by the George Washington University. Support was also provided by the Science and Engineering Apprentice Program (SEAP), which was administered by the George Washington University. The efforts of Dr Sandra Young through the Education Outreach Office (ARL) are also acknowledged.

The Materials Selection and Analysis Tool (MSAT) workflow and schema development was greatly assisted by the Materials and Processing Technical Information System Team at NASA's Marshall Space Flight Center by Mr Benjamin Henrie, Mr Paul Wimberley, Mr Bill Elliot, Mr Dennis Griffin, and Ms Teresa Miller. Collaboration with NASA was supported through an interagency Space Act Agreement.

Curatorial protocols for capturing the mechanical testing results into MSAT were developed at ARL by Ms Wendy Kosik-Chaney and Mr Jonathan Kaufman. Dynamic mechanical analysis was performed by Dr Ian McAninch. Mr Kaufman and Dr McAninch were supported in part by an appointment to the Research Participation Program at ARL administered by the Oak Ridge Institute for Science and Education (ORISE) through an interagency agreement between the US Department of Energy and ARL.

Student intern assistance during the GEMS sessions and follow-on mechanical testing were provided by Mr Edmond Elburn (SEAP), Mr Andres Bujanda, Mr Jose Baeza (College Qualified Leaders [CQL] program), and Mr Daniel Perez (CQL). Follow-on mechanical testing was also performed by Mr Nathan Jensen (ARL Science, Technology, Engineering, and Mathematics Outreach, Bring Your Child to Work Day). Mechanical testing setup was assisted by Mr David Gray.

Single-lap-joint fixtures and test coupon panels were fabricated under the guidance of Mr Bobby Hall (ARL Experimental Fabrication Shop) and Mr Robert Bruce (ARL Wood Model/Metal Fabrication Shop).

Mr Wayne Ziegler (ARL) is also recognized for overseeing the cyber-infrastructure of MSAT and establishing the web-based linkages needed for direct server application of MI: Toolbox, which greatly reduced analysis time.

Appreciation is also expressed to Dr Randy Mrozek (ARL) and Mr Ian Beringer (ORISE) for supporting our efforts to quantify failure surfaces using the 3-D microscope.

The authors also thank Air Products and Chemicals, Inc., for supplying the CEP100 epoxy adhesive under cooperative agreement W911NF-08-2-0062.

The ARL Technical Publishing office is acknowledged for assistance with editing and formatting this report.

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## 1. Introduction

The adhesively bonded single lap joint was used to study the effects of coupon thickness, bondline thickness, surface preparation, presence of an overflow fillet, and adhesive type. This joint configuration is convenient for screening armor adhesives<sup>1</sup> and is widely studied in academia and industry.<sup>2,3</sup> Fabrication of 960 single-lap-joint samples was completed by Gains in the Education of Mathematics and Science (GEMS) high school and middle school students as part of the US Army Research Laboratory's (ARL) involvement with Science, Technology, Engineering, and Mathematics (STEM) youth outreach. ARL technicians prepared an additional 245 controls. The GEMS program was leveraged to probe alternative workflow protocols revolving around a relational database and to generate sample sets large enough for subsequent multivariate analysis, which will be discussed in a separate paper. The purpose of this report is to carry the experimental and workflow protocols in detail and to briefly discuss the assembly quality of the GEMS student samples.

Active links in this report are provided to the reader for full access to relevant experimental data and supporting metadata descriptors. Digital resources are housed on the National Institute of Standards and Technology (NIST) DSpace repository and a complete file listing can be found in the Appendix.

## 2. Experimental

The single lap joints were fabricated and tested using ASTM D1002- $10^4$  as the basis standard, schematically represented in Fig. 1. The joint geometry has a lengthy history of study in the academic literature, dating to the 1930s.<sup>5</sup>



Fig. 1 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).

$$S_{\max} = \frac{P_{\max}}{A}.$$
 (1)

The maximum strength and mode of failure represent the accepted standard reported outputs of single-lap-joint testing in both industry and academia.<sup>6,7</sup>

#### 2.1 GEMS Considerations

#### 2.1.1 Adhesive Selection

The adhesives used were a peroxide-cured methacrylate (SG300, SCIGRIP Americas) and an amine-cured epoxy (CEP100, Air Products and Chemicals, Inc.). These adhesives populate varying regions of Group II and Group III maximum single-lap-joint strength (S<sub>max</sub>) versus displacement at complete failure (d<sub>failure</sub>), as defined by <u>ARL-ADHES-QA-001.00 rev 1.0</u> (Fig. 2).<sup>1</sup> This variation in properties between SG300 and CEP100 proved insightful for subsequent multivariate statistical analysis. The adhesives have glass transition temperatures (T<sub>g</sub>'s) of –25 °C and 82 °C for SG300 and CEP100, respectively. Both adhesives provided working pot lifetimes and handling characteristics that accommodated the GEMS program educational requirements, student experience, and scheduling constraint of 75 min/student session.



Fig. 2 Adhesive groups based upon  $S_{\text{max}}$  and  $d_{\text{failure}}$  single-lap-joint performance at room temperature (dry conditioning as defined by ARL-ADHES-QA-001.00 rev 1.0)

#### 2.1.2 Joint Assembly and Quality Control

Bondline thickness and overlap dimension control were essential to reducing possible sources of experimental error. To minimize assembly variation, a singlelap-joint tooling fixture was used, as shown in Fig. 3. The tooling fixture is equipped with alignment pins to set orientation and overlap length. Spacer shims are also used to set a predetermined and constant bondline thickness. A total of 48 single-lap-joint tooling fixtures were available, with 12 in use for each individual GEMS session. Each GEMS participant was able to fabricate his or her own individual set of single-lap-joint coupons, which enhanced the overall sample set size needed for multivariate analysis and the hands-on laboratory experience for the students. The single-lap-joint tooling fixture is further described in ARL-ADHES-QA-001.01 rev 2.2.8 Other research groups have discussed tooling fixtures for aligning single lap joints using a more complex design that allows for variable overlap lengths.<sup>9</sup> The test matrix systematically varied adhesive type, coupon thickness, bondline thickness, overflow fillet, and surface pretreatment. Each test matrix set was performed independently by 2 students to minimize data integrity loss due to inadvertent mishandling during preparation.



Fig. 3 Single-lap-joint tooling fixtures were used to ensure accurate joint overlap and alignment. Spacer shims controlled the bondline thickness.

## 2.1.3 Metadata Verification

The GEMS students provided a critical check and balance by completing handwritten "travel sheets" to verify metadata parameters, such as sample identification (ID), adhesive and sample preparation information, coupon and bondline thickness, and laboratory temperature and relative humidity. The GEMS students' handwritten travel sheets were scanned and stored electronically in ARL's Material Selection and Analysis Tool (MSAT) relational database for subsequent cross-referencing validation, which proved useful for auditing and culling data input errors. The GEMS students also hand-wrote their MSAT sample IDs on the nonbonded sides of their single-lap-joint coupons using a permanent ink marker.

Including the 245 ARL control samples that were bonded, a total of 1,205 singlelap-joint coupons were prescribed as input in the <u>testing matrix</u>. A total of (5) 6th graders, (23) 7th graders, (36) 8th graders, (30) 9th graders, (17) 10th graders, (11) 11th graders, (1) 12th grader, 21 teacher assistants, and 17 SEAP student assistants participated in the bonding experiments. Each GEMS participant fabricated his or her own set of 5 single lap joints as prescribed in the test matrix. A total of 1,202 single-lap-joint test results, and associated metadata, were captured in MSAT. The overall data capture efficiency was 99.8% using a materials informatics workflow approach.\*

## 2.2 Materials Informatics Workflow Approach

The materials informatics workflow approach, as shown in Fig. 4, requires up-front consideration and continual permeation in and out of a relational database during the application of experimental methods, collection of data, and analysis of the results. A detailed schematic of the specific workflow process from the method application through analysis used for the GEMS program is shown in Fig. 5 and described in the following sections.

<sup>&</sup>lt;sup>\*</sup>Missing mechanical testing results and failure surfaces for samples 20120152, 20120333, and 20120457. Missing failure surfaces for sample 20120299.

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Fig. 4 Schematic of materials informatics workflow approach showing a relational database center of operation

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Fig. 5 Flowchart summary of the overall materials informatics workflow progression used to fabricate, test, and analyze single lap joints

### 2.2.1 Single-Lap-Joint Bonding

The single-lap-joint bonding subroutine of the workflow process (Fig. 6) was devised to maximize high through-put sample production and minimize sample tracking errors. The experimental parameters varied in the <u>test matrix</u> included surface pretreatment, bondline thickness, coupon thickness, presence or absence of an overflow fillet, and adhesive type, as shown in Table 1. These parameters are commonly taken into account in academic studies of the single lap joints.<sup>2,3,10,11</sup> Aluminum alloy (2024 T3) was used as the single-lap-joint coupon material for all tests.



Fig. 6 Single-lap-joint bonding subroutine of the workflow process

	Option					
Parameter –	1	2	3	4		
Surface pretreatment	Solvent wipe	Silane coupling agent				
Bond-line thickness	0.127 mm (0.005 in)	0.381 mm (0.015 in)	0.762 mm (0.030 in)	1.14 mm (0.045 in)		
Coupon thickness	1.14 mm (0.045 in)	1.52 mm (0.060 in)	2.29 mm (0.090 in)	-		
Overflow fillet	Yes	No				
Adhesive type	SG300	CEP100				

 Table 1
 Single-lap-joint experimental parameters

Each test matrix sample was preassigned an 8-digit sample ID using the "MSAT Unique Specimen ID" generator. This feature was built into MSAT based directly upon similar functionality in NASA's Materials and Processing Technical Information System (MAPTIS). The Unique Specimen ID generator prevents duplication of sample names, which is critical to carrying large sets of data through a database. The sample IDs are assigned sequentially by the generator function.

The GEMS students' <u>handwritten travel sheets</u> were used to force a manual human confirmation of the prescribed test matrix conditions to the MSAT ID and to capture pertinent experimental metadata that required immediate documentation (temperature and humidity).<sup>12</sup> The GEMS students noticed approximately 5 errors with their preassembled single-lap-joint fabrication kits, such as incorrect shim spacer or coupon thickness, that were immediately corrected by ARL personnel prior to bonding. Capture of <u>repetitive metadata</u> (adhesive application, cure conditions, mechanical testing conditions, etc.) was completed via batch processing imports into MSAT using GRANTA MI – MI: Toolbox.<sup>13</sup>

#### 2.2.2 Sample Curing and Preparation for Mechanical Testing

Sample curing and preparation for mechanical testing (Fig. 7) were performed by ARL personnel. While samples could be cured immediately following the GEMS bonding sessions, sample preparation for mechanical testing required more time and could not be fully completed during the same week. During the first week's GEMS session, a minimal number of single lap joints were prepped to serve as teaching samples for their mechanical testing session. Once the first week's GEMS session was completed, there was adequate time to prepare enough samples for mechanical testing in the remaining 3 sessions. Sample preparation involved separating individual single-lap-joint coupons, finishing rough edges, removing fillets if required, and measuring bondline thickness as described in ARL-SR-0356.<sup>8</sup> Bondline thickness travel sheets were converted to spreadsheet format and uploaded in MSAT.



Fig. 7 Sample curing and preparation for mechanical testing subroutine of the workflow process

## 2.2.3 Mechanical Testing

Figure 8 shows the experimental workflow used for mechanical testing of the single lap joints. Mechanical testing for the GEMS sessions was preceded by a short and informal "white board" discussion to cover the minimal basics of tensile strength measurements and load frame operation. Each GEMS student was able to carry out supervised testing of a minimum of one single lap joint, including mounting the sample. The remaining sample coupons were tested by summer intern students and ARL technicians. The testing supervisor was assigned as the operator in MSAT.



Fig. 8 Mechanical testing subroutine of the workflow process

Mechanical testing was conducted using various test frames and load cells (Instron, Norwood, MA). <u>Test frames, load cells, operators, calibration dates, and calibration certificates were traced to individual single-lap-joint samples using MSAT</u>. A total of 7 test operators, 3 load frames, and 5 load cells were used to complete the GEMS single-lap-joint mechanical testing.

Electronic capture of the load versus displacement data into MSAT was facilitated using formatted output files from the mechanical testing frame. Individual load versus displacement results were exported in <u>comma-separated values (csv) format</u>. An experimental metadata summary for the set of 5 single lap joints was also captured in the testing script at the mechanical testing frame and exported in <u>text file format</u>. The csv and txt file exports used the MSAT unique sample ID to ensure the pedigree of the test results. The csv and txt file formats were also scripted for compatibility with the GRANTA MI base operating software and the schema format used by MSAT. The export files were compressed into ZIP archive file format and emailed as attachments to the MAPTIS team at NASA's Marshall Space Flight Center. The mechanical test results were then uncompressed and uploaded in MSAT using MI: Toolbox by NASA.

#### 2.2.4 Mechanical Testing Analysis

The mechanical testing results were converted into load versus displacement and load versus time plots in MSAT. The load versus displacement data were then exported from MSAT into a Microsoft Excel spreadsheet for <u>semi-automated analysis</u> using MI: Toolbox, following the process subroutine shown in Fig. 9. The analysis spreadsheet allows the assignment of test validity, the option to exclude the results from the test summary, and the option to calculate derivative plots. The derivative curves were investigated for applicability in determining yielding behavior.<sup>14</sup> The analysis spreadsheet was developed jointly by ARL (analysis considerations) and NASA's Marshall Space Flight Center (GRANTA MI compatible export, parameter, and import options).



Fig. 9 Mechanical testing analysis subroutine of the workflow process

The primary consideration in the analysis of the single lap joint was in balancing the efficiency of the subroutine process while maintaining "human-in-the-loop" validity assessment. The analysis spreadsheet is written to provide automated measures of strength, displacement, and area under the curve. However, human judgment is required to assign test validity, which could only be ascertained from experience in cases where the load versus displacement curve appeared irregular. All test data, whether valid or invalid, were retained in MSAT. Completed <u>analysis</u> spreadsheets<sup>\*</sup> were imported back into MSAT using MI: Toolbox.

#### 2.2.5 Mode-of-Failure Analysis

Upon completion of mechanical testing, the <u>failure surfaces</u> of the broken singlelap-joint coupons were digitally imaged using a flatbed scanner (HP OfficeJet D145) and archived in MSAT, as shown in Fig. 10. The failed test coupons were

<sup>\*</sup> Note: The <u>derivative option</u> was not used in the analysis because of time considerations and preliminary statistical analysis showing minimal correlations.

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manually labeled with the MSAT ID to embed this information within the image, which preserved data integrity by eliminating file-naming errors. Images were scanned at 300 dots per inch (dpi) resolution and saved in tagged image file format (TIFF), which is a common minimum recommendation for photo archiving.<sup>15</sup> Test coupons were visually assigned either an adhesive, cohesive, or mixed-mode of failure.



Fig. 10 Mode-of-failure analysis subroutine of the workflow process

#### 2.2.6 Data Export and Multivariate Analysis

Pedigreed raw and analyzed GEMS single-lap-joint data were exported from MSAT as <u>categorical and numerical output</u>, as shown schematically in Fig. 11. Data exported included metadata related to processing, quality, and analysis. <u>Multivariate analysis</u> was performed using JMP Statistical Discovery 11.2.0 (SAS Institute Inc.).



Fig. 11 Formatted output and multivariate statistical analysis subroutine of the workflow process

### 3. Results and Discussion

#### 3.1 Joint Quality and Failure Mode Analysis

The quantitative failure mode analysis of the single lap joints was both challenging and time consuming for the sample set size presented in this research. While mechanical test results can be efficiently exported using well-defined x versus y data in a kilobyte file size range, image capture represents the opposite. For this research each individual set of failed bonding surfaces was captured digitally using a flatbed scanner at 300 dpi resolution (TIFF). The samples were scanned with affixed labels to allow for independent identification from within the image, rather than relying on the accuracy of the file name. The combination of manually scanning and the larger megabyte-sized files resulted in extensive processing times for uploading into MSAT. Once uploaded, we quickly realized that the scanned failure surfaces appeared slightly different to the human eye with samples in hand, most likely due to very slight mismatches in scanner calibration and image resolution. Efforts to resolve the disparity between the digital images and the human eye were attempted using a calibrated 3-D microscope,<sup>†</sup> but this route led to unmanageable file sizes and processing time requirements. Despite these image processing challenges, analysis was required to provide categorical mode-of-failure inputs for subsequent multivariate statistical analysis of the mechanical testing results to ensure representative comparability.

The categorical assignment of mode-of-failure descriptions was arrived at by approaching the problem from the historical social and behavioral sciences origins of multivariate statistical analysis, "where it was used to understand people's judgements of the similarity of items in a set".<sup>16</sup> The visible modes-of-failure for both the CEP100 and SG300 adhesives were consistent enough to be graded using a <u>simple questionnaire</u>. The physical samples of the failed single lap joints were laid out for visible inspection of the entire ensemble by the primary author at the same time, as shown in Fig. 12. The mode of failure was assigned a categorical description of "adhesive", "cohesive", or "mixed mode". Fillet and adhesive fill quality were graded using a wording scale ranging from "very good", "good", "fair", "poor", to "very poor", following guidelines for rating opinions.<sup>17</sup> The subjective nature of these categorical mode-of-failure assignments seemed to be lessened by the high number of samples. Moreover, the assignment process was easily accomplished during a single working shift. These categorical assignments were required to ensure quantitative multivariate analysis for samples with

<sup>&</sup>lt;sup>†</sup>The 3-D imaging was taken using a Keyence Corporation VK-X2000 3-D and profile measurement laser microscope. ISO 25178 surface texture measurements were taken using VK Analyzer, version 3.3.0.0.

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consistent modes of failure and processing quality. Representative individual samples are shown in Figs. 13–16. The physical samples have also been archived and are available for secondary inspection.



Fig. 12 Entire SG300 sample set presented for categorical assignment of mode of failure, fillet quality, and adhesive fill quality



Fig. 13 CEP100 sample showing a "very good" fillet with a "very good" adhesive fill



Fig. 14 CEP100 sample showing a "very poor" fillet with a "very good" adhesive fill



Fig. 15 SG300 sample showing a "very good" fillet with a "very good" adhesive fill



Fig. 16 SG300 sample showing a "very poor" fillet with a "very poor" adhesive fill

The mode of failure for the CEP100 epoxy was adhesive regardless of surface pretreatment conditions. The mode of failure for the SG300 adhesive was predominantly mixed-mode for the solvent-cleaned surfaces and cohesive for the silane coupling agent pretreatment. The SG300 cohesive failures were consistent with descriptions of "failure close to the interface"<sup>18</sup> or "thin layer cohesive failure".<sup>10,19</sup> Noticeable differences were observed in the adhesive fill and fillet quality between the student- and ARL technician–prepared samples, with increased laboratory experience trending toward decreased counts of "fair", "poor", and "very

poor" fillet and fill probabilities, as shown in Table 2. The student SG300 samples showed a higher probability of a "very good" fill than the ARL Technician samples, due to the presence of single pin-hole voids degrading the assignment to "good", which could be due to a wide range of test and operator conditions. Only single lap joints with "very good" fill and fillet quality were considered for the multivariate analysis of the mechanical testing.

Dating	Adhesive fil	l probability	Fillet probability		
Kating	<b>CEP100</b>	SG300	<b>CEP100</b>	SG300	
	Student 0.77	Student 0.81	Student 0.41	Student 0.60	
Very good	Technician 0.97	Technician 0.29	Technician 0.80	Technician 0.91	
Card	Student 0.10	Student 0.08	Student 0.09	Student 0.07	
G000	Technician 0.02	Technician 0.65	Technician 0.05	Technician 0.06	
	Student 0.06	Student 0.07	Student 0.26	Student 0.15	
Fair	Technician 0.02	Technician 0.06	Technician 0.15	Technician 0.03	
_	Student 0.03	Student 0.03	Student 0.10	Student 0.07	
Poor	Technician 0	Technician 0	Technician 0	Technician 0	
	Student 0.04	Student < 0.01	Student 0.14	Student 0.09	
Very poor	Technician 0	Technician 0	Technician 0	Technician 0	

 Table 2
 Survey questionnaire results for adhesive fill and fillet quality

Both adhesives yielded approximately 400 samples with a "very good" fill and 140 samples with a "very good" fillet, with exact sample counts shown in Tables 3 and 4.

Table 3 Maximum strength and displacement at maximum load as a function of observed adhesive fill quality, including number of samples (N). Standard deviations are shown in parenthesis.

A dla asimo	Adhesive fill quality						
Adnesive	Very good	Good	Fair	Poor	Very poor		
	19.2 (± 7.0) MPa	16.9 (± 6.4) MPa	12.3 (± 6.1) MPa	9.2 (± 5.3) MPa	4.7 (± 8.6) MPa		
CEP100	1.6 (± 0.6) mm	1.7 (± 0.8) mm	1.2 (± 0.6) mm	1.1 (± 0.7) mm	$0.6 (\pm 0.4) \text{ mm}$		
	N = 484	N = 48	N = 33	N = 16	N = 19		
	12.2 (± 2.7) MPa	10.0 (± 2.5) MPa	9.9 (± 1.8) MPa	7.5 (± 1.7) MPa	4.6 (± 2.5) MPa		
SG300	1.8 (± 0.6) mm	1.7 (± 0.5) mm	1.7 (± 0.5) mm	1.6 (± 0.4) mm	$1.2 (\pm 0.1) \text{ mm}$		
	N = 407	N = 125	N = 48	N = 19	N = 3		

Table 4 summarizes the effect of fillet quality to maximum strength and displacement at maximum load, while maintaining the adhesive fill quality at "very good". For both the CEP100 and SG300 adhesives, there appears to be a slight decrease in performance when the fillet quality is either "poor" or "very poor".

Maximum strength has been reported for composite joggle lap joints with controlled overflow fillets.<sup>17</sup> These results indicate that at least a minimum "fair" fillet is sufficient to reduce the known stress concentrations at the edge boundaries of the overlap area.<sup>6,10</sup>

Table 4	Maximum strength an	id displacement at	t maximum load as	s a function	of observed
fillet qua	lity, including number	of samples (N). Sta	ndard deviations a	re shown in p	parentheses.

Adhesive fill	Fillet quality					
quality	Very good	Good	Fair	Poor	Very poor	
Very good	19.7 (± 7.4) MPa	20.7 (± 6.4) MPa	23.5 (± 7.5) MPa	15.8 (± 8.6) MPa	15.6 (± 8.6) MPa	
CEP100	1.8 (± 0.6) mm	1.6 (± 0.5) mm	1.7 (± 0.5) mm	1.2 (± 0.7) mm	1.3 (± 0.8) mm	
	N = 147	N = 22	N = 64	N = 10	N = 11	
Very good	12.7 (± 2.4) MPa	13.3 (± 1.6) MPa	12.0 (± 1.2) MPa	10.1 (± 2.1) MPa	10.0 (± 4.5) MPa	
SG300	1.9 (± 0.6) mm	1.8 (± 0.3) mm	1.9 (± 0.3) mm	1.4 (± 0.6) mm	1.4 (± 0.5) mm	
~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~	N = 143	N = 16	N = 25	N = 13	N = 19	

#### 3.2 Student and ARL Control Sample Comparisons

The GEMS students and ARL technician control samples directly overlapped for 240 samples (480 total) where bondline thickness was fixed to 0.762 mm, as shown in Table 5.

Table 5Single-lap-joint experimental parameters where GEMS student samples and ARLtechnician controls overlapped

	Option					
Parameter –	1	2	3	4		
Surface pretreatment	Solvent wipe	Silane coupling agent				
Bondline thickness			0.762 mm (0.030 in)			
Coupon thickness	1.14 mm (0.045 in)	1.52 mm (0.060 in)	2.29 mm (0.090 in)			
Overflow fillet	Yes	No				
Adhesive type	SG300	CEP100				

Figure 17 shows the distributions of maximum strength for CEP100 and SG300 where the GEMS student samples and ARL controls coincided. The distribution plots do not account for surface pretreatment, variations in bondline thickness, coupon thickness, or overflow fillet. As a general observation, the student distributions appear nonuniform for the CEP100 epoxy and are more likely to include very low performers for both adhesives. The low performers in the student strength distribution curves coincide with the finite probability of including samples with either "poor" or "very poor" fillet and adhesive fill quality, which was not experienced for the ARL controls. However, the students also demonstrated the ability to achieve very high strengths when "good" and "very good" samples were produced. In fact, the average single-lap-joint strength for SG300 was slightly greater than the ARL controls, 11.5 MPa versus 10.0 MPa, respectively. The low viscosity of the CEP100 epoxy presented greater processing difficulty for the GEMS students relative to the higher-viscosity SG300 methacrylate adhesive. Summary statistics for the GEMS student and ARL controls are provided in Table 6.



Fig. 17 Distributions of maximum strength (MPa) for CEP100 – ARL controls; CEP100 – GEMS students; SG300 – ARL controls; and SG300 – GEMS students

Summary Statistics	CEP100 - ARL	CEP100 - GEMS	SG300-ARL	SG300-GEMS
Mean	18.4	16.7	10.0	11.5
Std Dev	6.0	7.8	1.7	2.9
Std Err Mean	0.5	0.7	0.2	0.3
Upper 95% Mean	19.4	18.1	10.3	12.0
Lower 95% Mean	17.3	15.2	9.7	11.0
Ν	120	120	120	120
Quantiles				
100.0%	32.5	31.5	12.7	16.1
99.5%	32.5	31.5	12.7	16.1
97.5%	31.7	30.1	12.2	15.7
75.0%	25.6	27.8	11.7	14.4
50.0%	22.1	22.9	11.2	13.3
25.0%	18.7	18.3	10.3	12.0
10.0%	14.0	10.4	9.4	10.7
2.5%	10.2	4.8	7.6	8.3
0.5%	7.3	0.1	7.1	0.8
0.0%	5.3	0.0	0.1	0.0

Table 6Summary statistics (MPa) for the distributions of maximum strength for the GEMSstudent and ARL sample controls

### 4. Conclusions and Future Work

The fabrication of 960 single-lap-joint samples was completed by GEMS high school and middle school students as part of ARL's involvement with STEM youth outreach. ARL technicians prepared an additional 245 controls. Joint inspection and mechanical test results showed that the GEMS students were capable of producing high-quality samples. The resultant high degree of pedigree and integrity for the data and analysis generated is amenable for public disclosure in commonly accessible electronic formats for additional peer review and scrutiny. Future work will involve more rigorous multivariate analysis to probe the concurrent influences of coupon thickness, bondline thickness, surface preparation, presence of an overflow fillet, and adhesive type to the mechanical response of the single lap joint.

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Appendix. Supporting Digital File Archive Index

Tables A-1 through A-4 provide the reader with a reference list and URL links to experimental data and supporting metadata descriptors archived in the National Institute of Standards and Technology (NIST) DSpace repository <u>https://materialsdata.nist.gov/dspace/xmlui/</u>.

Collection	File name <sup>a</sup>	Description
	Results	Size: 589.0 kB Format: MS Excel Strength averages and multivariate analysis results
	Export in SI units	Size: 609.7 kB Format: JMP Formatted categorical and numerical variables, SI consistent units
Analysis Results http://hdl.handle.net/11256/651	Export in US units	Size: 609.7 kB Format: JMP Formatted categorical and numerical variables, US customary units
	Sample controls	Size: 152.3 kB Format: MS Excel ARL technician control and GEMS student sample comparison
	Sample controls	Size: 119.9 kB Format: JMP ARL technician control and GEMS student sample comparison
	DOE and metadata	Size: 2.365 kB Format: MS Excel Test matrix, experimental metadata, and numerical output

Table A-1	Analysis	results
	1	

<sup>a</sup> Abbreviated file name as it appears on NIST site.

Collection	File name <sup>a</sup>	Description
Failure Surface Images http://hdl.handle.net/11256/649	Failure surfaces 1	Size: 391.1 MB Format: ZIP Failure surface scans, samples 20120041 – 20120364, compressed TIFF archive
	Failure surfaces 2	Size: 419.6 MB Format: ZIP Failure surface scans, samples 20120365 – 20120635, compressed TIFF archive
	Failure surfaces 3	Size: 552.9 MB Format: ZIP Failure surface scans, samples 20120636 – 20120935, compressed TIFF archive
	Failure surfaces 4	Size: 545.5 MB Format: ZIP Failure surface scans, samples 20120936 – 20121185, compressed TIFF archive
	Failure surfaces 5	Size: 430.6 MB Format: ZIP Failure surface scans, samples 20121186 – 20130193, compressed TIFF archive
	Failure mode surveys	Size: 8.983 kB Format: PDF Visual mode-of-failure inspection surveys

 Table A-2
 Failure surface images

<sup>a</sup> Abbreviated file name as it appears on NIST site.

Collection	File name <sup>a</sup>	Description
Load vs. Displacement Data http://hdl.handle.net/11256/650	P vs. d	Size: 519.3 MB Format: ZIP Load vs. displacement data, raw and analyzed, all samples, compressed MS Excel archive
	Text file export 1	Size: 2.109 kB Format: Text file Representative mechanical testing summary text file export
	Text file export 2	Size: 28.15 kB Format: MS Excel Representative mechanical testing load, displacement, and time text file export
	Analysis template	Size: 1.801 MB Format: MS Excel Blank single lap joint analysis template
	Analyzed sample 1	Size: 1.966 MB Format: MS Excel Representative single lap joint results, analyzed, with derivative curves
	Analyzed sample 2	Size: 1.805 MB Format: MS Excel Representative single lap joint results, analyzed, without derivative curves

 Table A-3
 Load vs. displacement data

<sup>a</sup> Abbreviated file name as it appears on NIST site.

Collection	File name <sup>a</sup>	Description
Supplementary Data http://hdl.handle.net/11256/652	CEP100 data sheets	Size: 200.0 kB Format: PDF CEP100 technical data sheet and MSDS
	SG300 data sheets	Size: 596.2 kB Format: PDF SG300 technical data sheet and MSDS
	Army adhesive metrics	Size: 561.0 kB Format: PDF ARL-ADHES-QA-001.00 rev 1.0, adhesive performance screening protocols
	Joint bonding process	Size: 2.303 MB Format: PDF ARL-ADHES-QA-001.01 rev 2.2, surface pretreatment and fabrication guidance
	Fab travel sheets	Size: 28.31 MB Format: PDF Handwritten GEMS student travel sheets completed during bonding process
	Bondline thickness	Size: 7.137 MB Format: MS Excel Handwritten and electronic bondline thickness travel sheets
	DMA plots	Size: 1.668 MB Format: MS Excel Dynamic mechanical analysis, SG300 and CEP100, glass transition temperatures
	Student prebriefing	Size: 521.3 kB Format: PDF GEMS student introduction briefing to adhesive bonding for Army applications
	Student evaluations	Size: 198.3 kB Format: PDF GEMS student feedback evaluations of single lap joint bonding and testing
	Calibration certs	Size: 8.610 MB Format: PDF Test equipment calibration certificates
	MAPTIS brochure	Size: 557.8 kB Format: PDF NASA MAPTIS overview description

## Table A-4 Supplementary data

<sup>a</sup> Abbreviated file name as it appears on NIST site.

3-D	3-dimensional
Α	bonded area
ARL	US Army Research Laboratory
ASTM	American Society for Testing and Materials
CSV	comma-separated values
CQL	College Qualified Leaders
DMA	dynamic mechanical analysis
$d_{\text{failure}}$	displacement at complete failure
$d_{\max load}$	displacement at maximum load
DOE	design of experiments
dpi	dots per inch
GEMS	Gains in the Education of Mathematics and Science
ID	sample identification number
MAPTIS	Materials and Processing Technical Information System
MS	Microsoft
MSAT	Materials Selection and Analysis Tool
Ν	number of samples
NASA	National Aeronautics and Space Administration
NIST	National Institute of Standards and Technology
P <sub>max</sub>	maximum load
PDF	portable document format
SCA	silane coupling agent
SEAP	Science and Engineering Apprentice Program
SI	International System of Units
$S_{\max}$	maximum strength

## List of Symbols, Abbreviations, and Acronyms

Std Dev	standard deviation
Std Err Mean	standard error mean
STEM	Science, Technology, Mathematics, and Engineering
SW	solvent wipe
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 LL IMAL HRA MAIL & RECORDS MGMT
- 1 GOVT PRINTG OFC
- (PDF) A MALHOTRA
  - 3 DIR USARL
- (PDF) RDRL WMM C D DESCHEPPER D FLANAGAN R JENSEN