ARMY RESEARCH LABORATORY

Support and Development of Workflow Protocols for High Throughput Single-Lap-Joint Testing - Experimental

by David Flanagan, Daniel DeSchepper, Edmond Elburn, and Robert Jensen

ARL-RP-425 **April 2013**

A reprint from the *Proceedings of the 36th Annual Meeting of the Adhesion Society, Inc.*, Daytona Beach, FL, 3 March 2013.

NOTICES

Disclaimers

Army Research Laboratory
Aberdeen Proving Ground, MD 21005-5069

ARL-RP-425 **April 2013**

Support and Development of Workflow Protocols for High Throughput Single-Lap-Joint Testing - Experimental

David Flanagan, Daniel DeSchepper, Edmond Elburn, and Robert Jensen
Weapons and Materials Research Directorate, ARL

A reprint from the *Proceedings of the 36th Annual Meeting of the Adhesion Society, Inc.*,
Daytona Beach, FL, 3 March 2013.

REPORT DOCUMENTATION PAGE	*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE *(DD-MM-YYYY)*
April 2013

2. REPORT TYPE
Reprint

3. DATES COVERED (From - To)
1 May 2012–1 March 2013

4. TITLE AND SUBTITLE
Support and Development of Workflow Protocols for High Throughput Single-Lap-Joint Testing – Experimental

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
David Flanagan, Daniel DeSchepper, Edmond Elburn, and Robert Jensen

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
U.S. Army Research Laboratory
ATTN: RDRL-WMM-C
Aberdeen Proving Ground, MD 21005-5069

8. PERFORMING ORGANIZATION REPORT NUMBER
ARL-RP-425

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES
A reprint from the *Proceedings of the 36th Annual Meeting of the Adhesion Society, Inc.*, Daytona Beach, FL, 3 March 2013.

14. ABSTRACT
The absence of documented properties for damage tolerant adhesives is a limiting factor in the research and development of improved bonding for Army applications. Research efforts are ongoing to study the effects of aluminum coupon thickness, bond line thickness, surface preparation, and presence of an overflow fillet for a high strength epoxy and ductile methacylate adhesive. A unique feature of this study was the use of untrained GEMS (Gains in the Education of Mathematics and Science) high school and middle school students to fabricate the samples to meet the large sampling set requirements for statistical analysis. Sample fabrication was limited to approximately 75 minutes to cover both the educational and experimental laboratory aspects of the GEMS program. The GEMS students were assigned to 4 teams consisting of 12 members, with approximately a 10 minute intermission break between teams. The workflow protocol needed for successful single-lap-joint fabrication required a high degree of efficiency to accommodate the scheduling and educational bounds of the GEMS program. ARL technicians performed extensive tooling and adhesive handling development prior to the arrival of the GEMS students. This protocol development was successful as the students were able to fabricate test samples with fairly tight tolerances. Further workflow protocols were developed to facilitate post-curing of the adhesive for rigorous mechanical testing by the GEMS students within 2 days of initial fabrication. The lessons learned for experimental workflow protocols will be reported with the anticipation of expanding adhesive joint test configurations as part of the GEMS program.

15. SUBJECT TERMS
single lap joint, adhesion, aluminum, epoxy, methacrylate

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			David Flanagan
					19b. TELEPHONE NUMBER (*Include area code*)
Unclassified	Unclassified	Unclassified	UU	8	410-306-0691

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

Support and Development of Workflow Protocols for High Throughput Single-Lap-Joint Testing - Experimental

David Flanagan, Daniel Deschepper, Edmond Elburn, and Robert Jensen
Weapons and Materials Research Directorate
Army Research Laboratory
Aberdeen Proving Ground, MD, 21005, USA
david.p.flanagan6.civ@mail.mil

Introduction

The absence of documented properties for damage tolerant adhesives is a limiting factor in the research and development of improved bonding for Army applications. Research efforts are ongoing to study the effects of aluminum coupon thickness, bond line thickness, surface preparation, and presence of an overflow fillet for a high strength epoxy and ductile methacylate adhesive. A unique feature of this study was the use of untrained GEMS (Gains in the Education of Mathematics and Science) high school and middle school students to fabricate the samples to meet the large sampling set requirements for statistical analysis. Sample fabrication was limited to approximately 75 minutes to cover both the educational and experimental laboratory aspects of the GEMS program. The GEMS students were assigned to 4 teams consisting of 12 members, with approximately a 10 minute intermission break between teams. The workflow protocol needed for successful single-lap-joint fabrication required a high degree of efficiency to accommodate the scheduling and educational bounds of the GEMS program. ARL technicians performed extensive tooling and adhesive handling development prior to the arrival of the GEMS students. This protocol development was successful as the students were able to fabricate test samples with fairly tight tolerances. Further workflow protocols were developed to facilitate post-curing of the adhesive for rigorous mechanical testing by the GEMS students within 2 days of initial fabrication. The lessons learned for experimental workflow protocols will be reported with the anticipation of expanding adhesive joint test configurations as part of the GEMS program.

Experimental

The two adhesives tested in this experiment were SG300, a methacrylate based adhesive produced by SCIGRIP Americas, and CEP100, a two-part epoxy formulated by Air Products and Chemicals Inc. Table 1 shows the basic information of each adhesive.

Table 1. Chemistry, mix ratio, and cure cycle conditions for the SG300 and CEP100 adhesives.

	SG300	**CEP100**
Chemistry	Methacrylate	Epoxy with cycloaliphatic amine curative
Ratio	10:1 (resin to activator)	18 parts curative to 100 parts resin
Cure Cycle	24 hours @ room temperature, 3 hours @ 60°C	24 hours room temperature, 2 hours 60° C, 3 hours @ 150°C

The methodology behind the experimental research was set up in a three part procedure: fabrication of samples, mechanical testing, and digital capture of specimen information and test results for subsequent analysis. ARL technicians completed extensive testing and evaluation of dedicated control samples prior to the arrival of the GEMS students for method development and to validate the efficiency of the bonding procedures.

The single-lap-joints were bonded from “five finger” machine cut 2024 T3 aluminum templates. The aluminum templates were held in position for bonding using machined tooling plates measuring 25.4 cm x 25.4 cm with four alignment screws placed closer to the center to ensure proper bonding dimensional tolerances. The SG300 and CEP100 adhesives being tested were applied to the coupons using different applications procedures, but a GEMS student completed the fabrication in both procedures.

When using the SG300 adhesive, the students were first given instructions on cleaning the bonding surface of the coupons. Initially, acetone was used to clean the bonded area. During the second cycle of GEMS students, a grit-blast/silane coupling agent treatment was completed on the samples to determine how surface preparation affected adhesion, as shown in Figure 1.

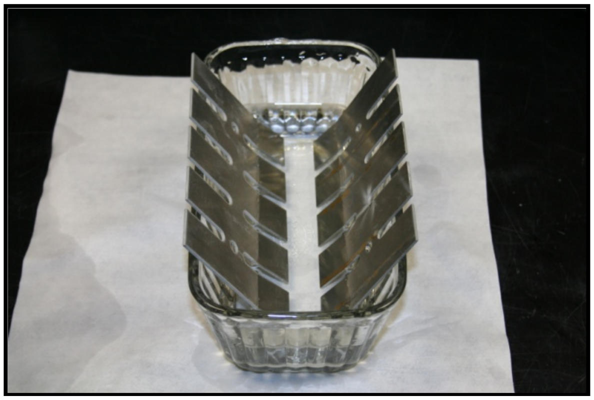

Figure 1. Single-lap-joint coupons were either cleaned with acetone (not shown) or chemically treated with a silane coupling agent (shown).

Each individual specimen in the coupon received a Materials Selection and Analysis Tool (MSAT) generated unique sample identification number for future organization. Non-stick release tape was placed along the bonding surface on the coupons to easily remove fillets, or overflow adhesive from the coupons. Next, two aluminum shims, one the thickness of the coupon and one the target bond line thickness, were placed over two of the alignment screws. The shims allowed for controlled bond thicknesses of 0.127, 0.381, 0.762, and 1.143 mm. A sheet of polytetrafluorethylene (PTFE) release film was placed over the shims, and the first coupon was placed over the other two alignment screws, opposite the shims. The SG300 was given to the students in a weighing tray using a pneumatic gun that mixes the components of the adhesive in the correct 10:1 resin to activator ratio. The adhesive was stirred for a minute by each student using a wooden spatula before being placed on the bonding surface. The students were instructed to completely cover the bonding surface with adhesive and to apply more than what they thought would be sufficient. The second coupon was placed face down over the first coupon without pressure applied to the bonding surfaces. The PTFE film, shims, and tooling lid were placed on the coupons, along with 13.6 kg of steel plates prior to curing.

For the two-part epoxy CEP100, the same method for surface treating the aluminum coupons for prior to bonding was used, but a more complex method for mixing and applying the adhesive was implemented. Approximately 30 grams of the resin was placed in a small beaker and placed to the side. The amount of curative needed was calculated using the ratio given: 18 parts curative to 100 parts resin. The appropriate amount of curative was measured using an analytic balance and placed in a separate beaker. The two components were mixed for one minute, followed by placing the epoxy in a vacuum for 6 minutes to remove any air present in the system, as well as allow the reaction between the two chemicals occur. Each student received approximately 4 ml of the epoxy for applying to the coupon surface. The students applied the epoxy using a syringe, as shown in Figure 2, after a 25 minute waiting period to allow for a slight viscosity build prior to application on the joint.

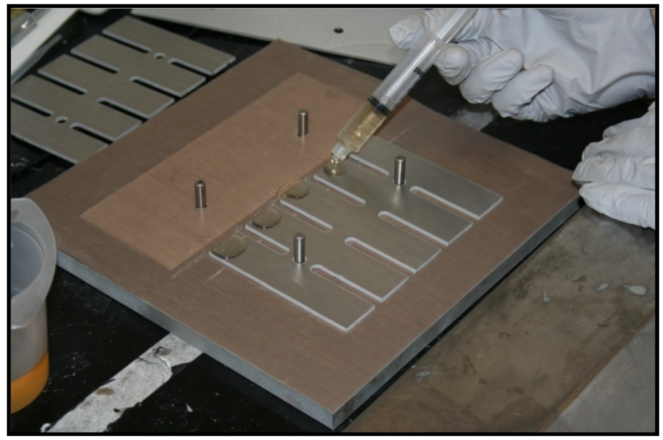

Figure 2. CEP100 being applied to the bonding surface using a syringe.

Each adhesive had a specific cure cycle as shown in Table 1. Figure 3 shows one of the ovens used to complete the curing cycles loaded with actual GEMS samples. Once the coupons had cooled overnight, they were removed from their fixtures and cut into sets of five individual samples. Samples with release tape applied had their fillets removed and all the samples were cleaned using acetone. Thicknesses measurements were completed to determine the bond line thickness for each individual sample, which was used to provide an accuracy rating compared to the target thickness. The thicknesses were placed in a spreadsheet and uploaded to the MSAT database with the corresponding unique specimen ID.

Figure 3. Oven post-curing of single-lap-joints in the bonding tool fixtures.

Results and Discussion

The GEMS students were able to successfully fabricate 960 single-lap-joint samples for testing. A cumulative total of 123 students were exposed to a brief 10 minute laboratory introduction presentation of adhesion and adhesives. The grade break-out was as follows: 5 sixth graders, 23 seventh graders, 36 eighth graders, 30 ninth graders, 17 tenth graders, 11 eleventh graders, and 1 twelfth grader. Each of the 123 students was afforded their own individual single-lap-joint tooling fixture and set of aluminum substrate coupons for bonding. The experimental aspects of the single-lap-joint design of experiments consisted of systematically varying the adhesive (SG300 versus CEP100), coupon thickness (1.143, 1.524, and 2.286 in), bond thickness (0.127, 0.381, 0.762, and 1.143 mm), and surface treatment (acetone wipe versus silane coupling agent treatment). The samples were then tested mechanically (to be discussed by Deschepper et al.[1]). Preliminary observations during the mechanical testing phase of the experiments revealed that the students prepared reasonably consistent samples. However, ARL technician consistency appears to have been higher for the samples with larger bond thicknesses.

Conclusions

The GEMS program provided an excellent opportunity to both teach fundamental adhesion concepts and obtain relevant testing results. 123 students fabricated 960 single-lap-joints using adhesives with possible relevance to Army applications. Specific feedback comments from the students regarding the adhesion portion of their GEMS experience at ARL include the following:

- This was really cool because it was a real life application.
- It was cool, but I wish I could see the results.
- I will remember the smell of adhesion forever.
- Smelly, but cool.
- Could have more seats
- We should have chairs.
- Great scientists and good experiments.
- Seats would have been nice.
- Good.
- Nicely explained.
- The person could have explained this better.
- Awesome.
- Was a fun, hands-on activity.
- It was fun putting that glue on the coupons and seeing how it came out.
- Very fun and advanced.
- Very fun, both presenters were very friendly & fun.
- It was fun.
- Informative, good.
- Adhesive had a very strong smell.
- Interesting.
- Pretty fun.
- Cool.
- It was interesting.
- Fun experiment.
- Probably the most interesting and fun.
- Really cool.
- Interesting to work on our own adhesive.
- Had fun making coupons.
- Ok, but could be for a shorter time.
- It was okay liked making adhesive.
- Different in a good way – enjoyed session.
- It was fun but, if it moved a little faster it would have been better.
- A lot of time standing around.
- Great instructors; not much to the lab.
- Liked how our data will be used by the Army.
- Nice people.
- Awesome.
- The process was interesting.
- Cool facts about armor and designs – nice lab
- Love the fact we're producing real and useful data
- Very cool, loved the hands on.
- I did not enjoy standing so long.
- A lot of standing – kind of loud in the lab – otherwise good.
- A lot of fun, learned a lot.
- There was a lot of waiting.
- I liked how our data was actually useful in their job.
- It was fun and organized.
- Awesome.
- I found it cool in learning how to do it.
- New – mixing glue.
- This was fun and enjoyable.
- It was interesting.
- Great time.
- Something to do while waiting?
- I liked that what we were doing was real work.

As can be implied from the grade levels and comments the GEMS students covered a broad range with respect to level of skill and interest (to be fair, the students were pushed for full work days between a variety of experiments, so fatigue was an issue during the afternoon sessions). But, the data was collected with a very high level of pedigree and integrity and should provide a rich source for further analysis.

Acknowledgements

This research was supported in part by materials funding and an appointment to the US Army Education Outreach - Gains in the Education of Math & Science Program at the U.S. Army Research Laboratory administered by the George Washington University. Mr. Elburn was funded through the Science and Engineering Apprentice Program (SEAP) also administered by the George Washington University. The authors also thank Air Products and Chemicals, Inc., for supplying the CEP100 under cooperative agreement W911NF-08-2-0062.

References

1. D. Deschepper, D. Flanagan, E. Elburn, R. Jensen, B. Henrie, and P. Wimberley, *Proceedings of the 36th Annual Meeting of the Adhesion Society, Inc., Daytona Beach Fl, March 3, 2013.*

NO. OF COPIES	ORGANIZATION
1 (PDF)	DEFENSE TECHNICAL INFORMATION CTR DTIC OCA 8725 JOHN J KINGMAN RD STE 0944 FORT BELVOIR VA 22060-6218
1 (PDF)	DIRECTOR US ARMY RESEARCH LAB RDRL CIO LL 2800 POWDER MILL RD ADELPHI MD 20783-1197
1 (PDF)	GOVT PRINTG OFC A MALHOTRA 732 N CAPITOL ST NW WASHINGTON DC 20401
1 (PDF)	RDRL WMM C D FLANAGAN