ARMY RESEARCH LABORATORY

Adhesives: Test Method, Group Assignment, and Categorization Guide for High-Loading-Rate Applications

by Robert Jensen, Daniel DeSchepper, David Flanagan, Wendy Kosik Chaney, Jason Robinette, Gerard Chaney, and Charles Pergantis

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Weapons and Materials Research Directorate, ARL

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[*] Refer to *Document Types and Naming Conventions* (LHC-PM-QA-202.00 rev 1.2, CERN, Geneva Switzerland) for formatting recommendations.

1.0 Purpose

This document provides guidance from within the Adhesives and Interfaces Research Team of the Coatings, Corrosion, & Engineered Polymers Branch (CCEPB) and the U.S. Army Research Laboratory (ARL) for initial performance-based screening evaluation and grouping priority assignments/categorization of candidate adhesives for high-loading-rate applications relevant to U.S. Army needs.

2.0 Scope

This standard process description (SPD) provides a guideline for assessing adhesives in the CCEPB. This SPD is not intended for acquisition acceptance. Rather, adhesives are screened for potential usage over a very broad performance spectrum to provide Army engineers, scientists, and researchers with the direction needed to meet operational priorities in a timely manner. Any deviation from the process details must be justified, fully documented, and submitted to the governing authority.

3.0 Policy

This SPD applies to all personnel developing, fabricating, testing, characterizing, and evaluating adhesives for bonding applications within the CCEPB.

4.0 Responsibilities

The Branch Chief, CCEPB, Adhesives Team leader, and principal investigator(s) will ensure all personnel working on adhesives development, fabrication, testing, characterization, and evaluation have full knowledge and/or proper training of this process, including equipment usage/operation test procedures and safety issues/factors before commencing relevant work within the CCEPB.

5.0 Requirements

All researchers performing adhesives development and evaluation work in the CCEPB must follow the procedures described within this SPD.

6.0 Terms and Definitions

For the purposes of this document, the following terms and definitions apply.

Database: As stated within ISO/IEC 2382-17:1999, a database shall be "a collection of data organized according to a conceptual structure describing the characteristics of these data and relationships among their corresponding entities, supporting one or more application areas."[1]

Digital Archive: The term "digital archive" shall be referred to as an electronic repository of digital data and metadata "to preserve the authenticity, reliability, integrity, and usability of such records" as stated in ISO 13008:2012(E).[2]

Group: The term "group," when referring to adhesives, shall be defined as a collection of adhesives meeting designated property requirements; the term is based upon recognized testing standards. Group requirements are intended to show correlation against response measured in nonstandard Army testing configurations. For example, the groups defined in this SPD define property regions from bonded single-lap-joint tensile tests with performance in bonded armor configurations tested against ballistic threats. Grouping assignments/categorizations of adhesives are intended to be amendable as Army needs change or are further refined. The grouping assignment/categorization is independent of the adhesive's chemical, processing, application, and property data and is based exclusively on single-lap-joint tensile data and performance. Multiple grouping assignments/categorizations for a given adhesive are possible under this convention. Therefore, an adhesive could potentially be assigned simultaneous high- and low-priority groups in differing SPDs, depending on the chemical, processing, application, or property data used in the specific correlation. For example, adhesive requirements for armor and munitions applications typically weigh importance to differing properties. Grouping requirements/categorizations are specified using conventionally accepted testing standards, which are elaborated upon further within this SPD. The intention is to facilitate communication of U.S. Army–specific property requirements for research and development to industry, as well as academia.

Material Pedigree: The term "material pedigree" shall refer to the documentation used to ensure the traceability of an adhesive and its constitutive formulation ingredients to the original manufacturer.[3]

Metadata: Per ISO/IEC 2382-17:1999, the term "metadata" pertains to "data elements, possibly including their data descriptions, and data about data ownership, access paths, access rights and data volatility."[1]

Tier: The term "tier" shall be considered a layer of testing protocols used to progressively characterize adhesive response. Each successive tier level requires an increasing investment commitment because of increasing experimental and analysis complexity.

7.0 Records

Per Army Regulation (AR) 25-400-2, all records (including data and associated metadata) are assumed to "have value beyond the business process, such as for historical, lessons learned, or research purposes; these are generally long-term records." Records will be kept in physical laboratory notebooks and digitally archived for permanent retention, consistent with AR 25-400-2 code "TP" (i.e., Transfer Permanent).[4]

8.0 Method Used to Characterize and Evaluate Adhesives

The method to characterize and evaluate an adhesive is a tiered system approach, whereby the adhesive is characterized and evaluated through standardized tests following a progression of increasing experimental difficulty. The first tier testing represents an initial screening level for the candidate adhesive, which must be passed to warrant further investment in the progressively more involved higher-tier screening levels. Advancement through the testing tiers is entrusted to the discretion of the researcher(s). The testing standards referenced in each tier are used as guidelines and should be followed when applicable. Deviations from the testing standards must be fully noted and accepted by the governing authority.

8.1 Adhesive Groups

Adhesives will be assigned and categorized according to the following groups based on single-lap-joint tensile performance at room temperature (RT) under dry conditioning per sample preparation and testing procedures specified in ASTM D1002-10 (Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension

Loading [Metal-to-Metal]).[5] Samples are to be stored in a desiccator cabinet or sealed in moisture-resistant sample bags immediately following sample preparation, with continued moisture-free storage until testing. Displacement at failure is not specified in ASTM D1002-10 but is required for grouping assignment/categorizing in this SPD and shall be defined as the following:

Group I: Group I adhesives shall be categorized as having a maximum strength greater than or equal to 10.0 MPa (1450 psi) and a minimum complete failure displacement limit greater than 3.81 mm (0.15 in); (e.g., $d_{failure}$ > 3.81 mm [0.15 in] and S_{max} ≥ 10.0 MPa [1450 psi]).

Group II: Group II adhesives shall be categorized as also having a maximum strength greater than or equal to 10.0 MPa (1450 psi), yet their complete failure displacement limits fall between the range of greater than or equal to 1.60 mm (0.063 in) and less than or equal to 3.81 mm (0.15 in); (e.g., 1.60 mm [0.063 in] ≤ $d_{failure}$ ≤ 3.81 mm [0.15 in] and S_{max} ≥ 10.0 MPa [1450 psi]).

Group III: Group III adhesives shall be categorized with a maximum strength greater than or equal to 10.0 MPa (1450 psi) but producing complete failures at a displacement less than 1.60 mm (0.063 in); (e.g., $d_{failure}$ < 1.60 mm [0.063 in], S_{max} ≥ 10.0 MPa [1450 psi]).

Group IV: Group IV adhesives shall be categorized as having a maximum strength less than 10.0 MPa (1450 psi) regardless of the amount of displacement at complete failure.

Adhesive grouping rejoins associated with their maximum strength versus displacement at complete failure is represented in Figure 1.

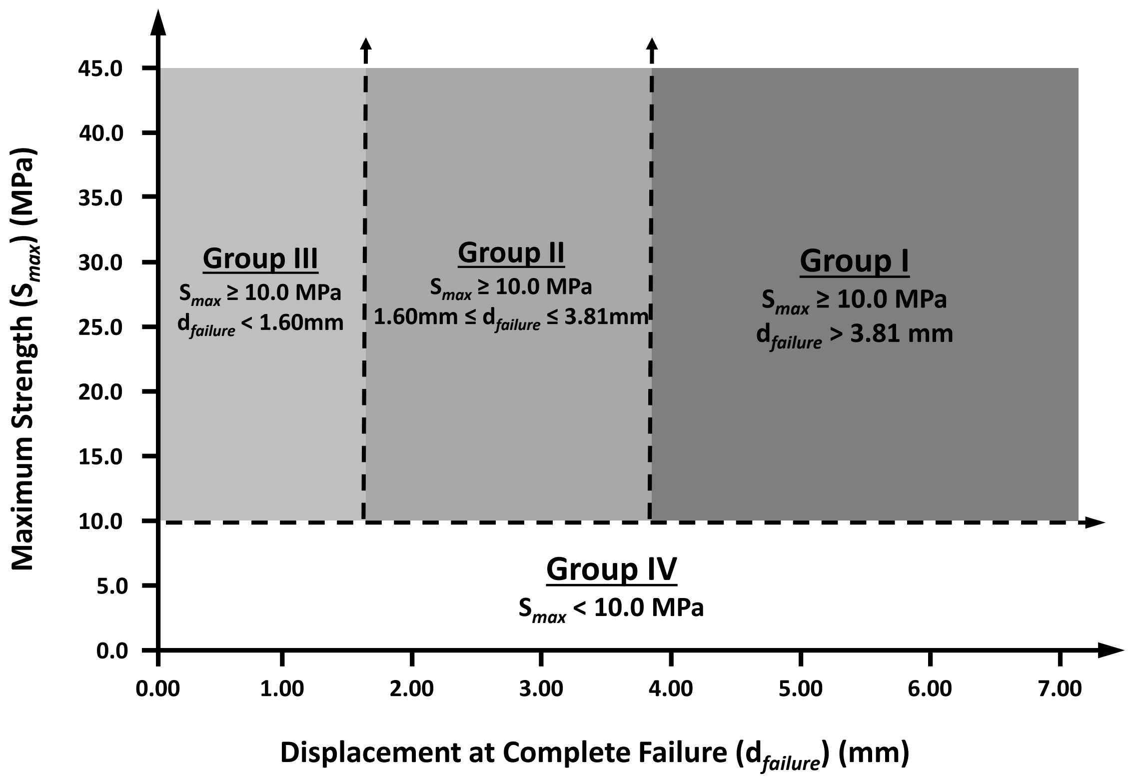

Figure 1. Adhesive groups based upon S_{max} and $d_{failure}$ single-lap-joint performance at RT (dry conditioning).

8.2 First Tier

The first tier of tests involves collecting the material's pedigree and determining adhesive properties via the standard single-lap-joint tensile test conducted at RT per ASTM D1002-10. Adhesives tested under first tier screening shall be assigned an initial grouping number associated with their performance (i.e., Group I, Group II, Group III, or Group IV).

8.2.1 Material Pedigree

The purpose of documenting the pedigree of the adhesive material is to gather and identify the original manufacturer's technical data sheet (TDS) and material safety data sheet (MSDS) information as references for the subsequent digitally archived experimental data. "Tagging" the TDS and MSDS to the adhesive allows for future tracking through public domain searches in the event that the original manufacturer is no longer viable or existent. In addition, material pedigree documentation is also required for adhesive formulations originating from academia due to student graduation. Furthermore, material pedigree data will also be documented and digitally archived for all ARL-developed adhesive formulations.

8.2.2 Single-Lap-Joint Test at Room Temperature (Dry Conditioning)

The single-lap-joint test specimen per ASTM D1002-10 is a convenient geometry for screening adhesive performance. An illustration of a typical test specimen is shown in Figure 2. The distribution of stress is nonuniform,[6] and fundamental constitutive adhesive properties are difficult to derive. However, the overwhelming experimental simplicity, with respect to both fabrication and testing, heavily favors the single-lap-joint geometry as an initial screening configuration.[7] Likewise, this joint geometry has also been studied extensively by academia, with numerous peer-reviewed literature results and modeling strategies available for comparison.[8] The single-lap-joint is also a favored industry standard.

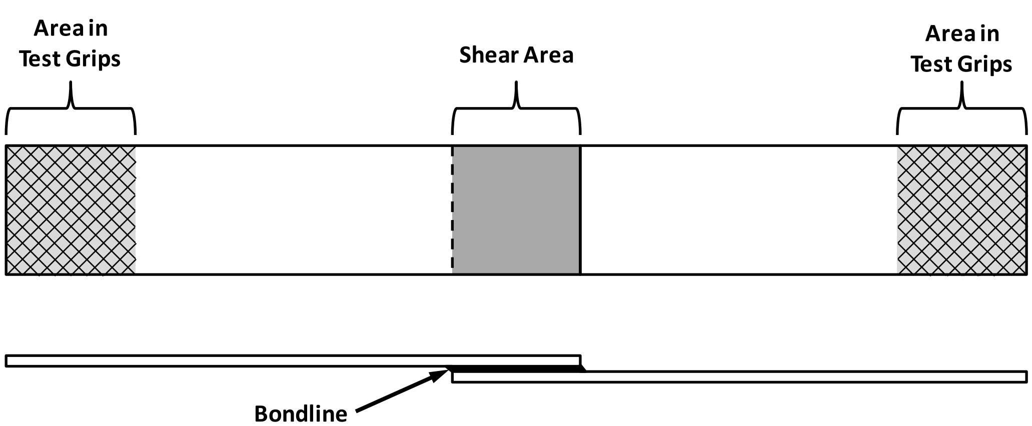

Figure 2. Illustration of the adhesively bonded single-lap-joint test specimen configuration (refer to ASTM D1002-10 for dimensions).

The specimen is placed within a mechanical testing machine and held into place at its ends by mechanical grips. Tensile load is applied until the joint fails. The maximum tensile strength (S_{max}) of the adhesive joint is defined as the maximum load (P_{max}) per shear area. Displacement at failure ($d_{failure}$) is taken directly from the crosshead displacement of the testing machine. A typical load versus displacement curve showing P_{max} and $d_{failure}$ is illustrated graphically in Figure 3.

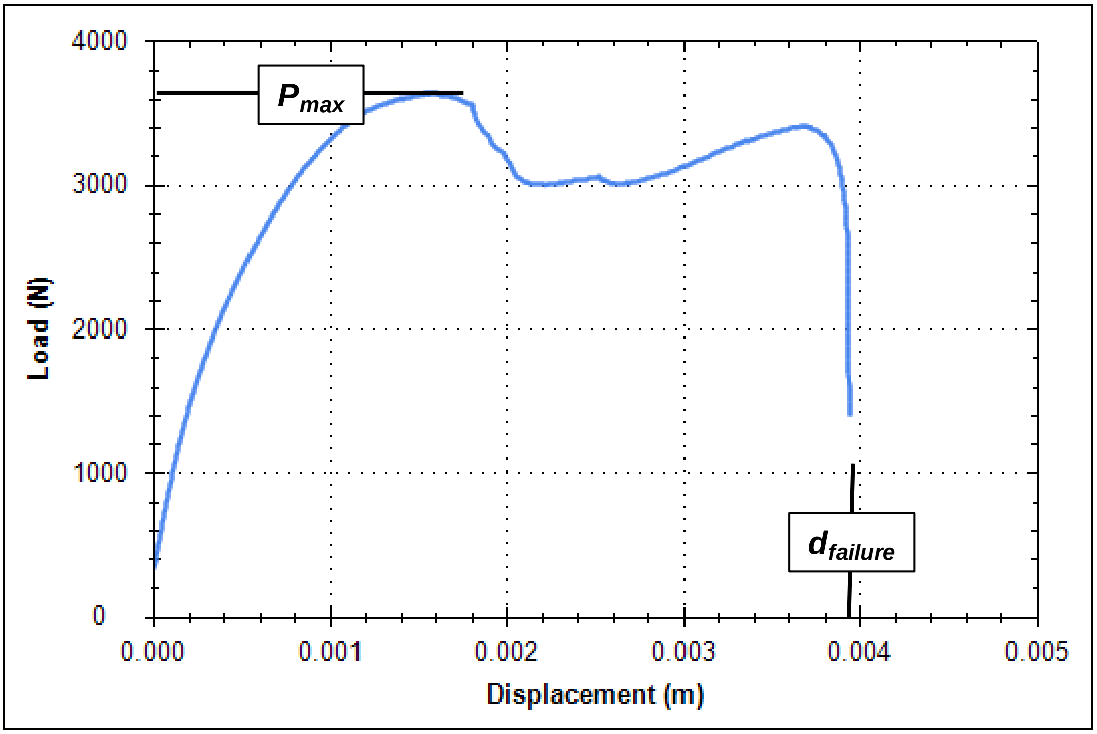

Figure 3. Graphical representation of P_{max} and $d_{failure}$ for a single-lap-joint tensile load vs. displacement curve.

8.3 Second Tier

The second tier testing comprises fabricating additional single-lap-joint specimens to satisfy additional testing requirements under two different environmental loading conditions, which shall consist of hot and hot/wet conditioning environments. It is at the discretion of the researcher(s) if they care to fabricate all test specimens and generate data for the first and second tiers simultaneously. In some instances this may be acceptable, but others may require a more step-wise approach because of availability of resources, funding, and manpower. To be accepted for third tier testing, adhesives must pass second tier part one and part two requirements.

8.3.1 Single-Lap-Joint Testing After Hot/Wet Conditioning

This first part of the second tier test requires submersion of lap-joint specimens (i.e., nonambient moisture conditioning) in a water immersion tank for fourteen (14) days at a constant temperature of 63 °C, +/–3 °C (145 °F, +/–5 °F). At the completion of the conditioning, specimens will be pat-dried and tested no later than thirty (30) minutes after being removed from the water immersion tank. As in the first tier, single-lap-joint tensile test data will be obtained via a mechanical testing machine at RT and at ambient air conditions as per ASTM D1002-10. Test duration and conditioning temperature is based upon considerations referenced in MIL-STD-810G, Laboratory Test Method 507.5, Humidity.[9] Experimental technique considerations can be found in ASTM D5229/D5229M-12 (Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials),[10] ASTM D1151-00 (2013) (Standard Practice for Effect of Moisture and Temperature on Adhesive Bonds),[11] and ASTM E865-12 (Standard Specification for Structural Film Adhesives for Honeycomb Sandwich Panels).[12]

8.3.1.1 Requirements

Acceptance to the next tier of testing requires that second tier, part one conditioned adhesives retain a minimum of 75% of their initial dry maximum strength measured in first tier testing, section 8.2.2. It is important to note that group assignment/categorization is entirely based upon the initial S_{max} and $d_{failure}$ at RT at dry conditioning. Displacement at failure following hot/wet conditioning will be digitally archived but will not alter the first tier group determination. Likewise, if S_{max} (RT, dry) for a given Group I, II, or III adhesive is at or just above 10.0 MPa (1450 psi) then measurement of the 75% strength retention following hot/wet conditioning will not reassign the adhesive to Group IV.

8.3.2 Single-Lap-Joint Testing at Elevated Temperature

Second tier, part two, tests shall progress by testing fabricated samples to an elevated temperature condition of 71 °C, +/–3 °C (160 °F, +/–5 °F) using an in situ heated test chamber on the mechanical testing frame. Prior to loading specimens, the test chamber shall be heated for at least forty five (45) minutes to ensure the chamber air and mechanical components of the mechanical testing machine, such as grips, are at temperature and are stable, as measured by a thermocouple probe/sensor. Once a sample is loaded within the heated chamber, but prior to tensile testing, it shall be held in situ for at least ten (10) minutes until it reaches temperature equilibrium and is stable as measured by a thermocouple probe/sensor. The temperature of the test is based upon considerations referenced in MIL-STD-810G, Laboratory Test Method 501.5, High Temperature.[13] Experimental technique considerations can be found in ASTM D1151-00(2013) (Standard Practice for Effect of Moisture and Temperature on Adhesive Bonds)[11] and ASTM E865-12 (Standard Specification for Structural Film Adhesives for Honeycomb Sandwich Panels).[12]

8.3.2.1 Requirements

Acceptance to the next tier of testing also requires that second tier, part two conditioned adhesives retain a minimum of 75% of their dry maximum strength as measured in section 8.2.2. As in section 8.3.1.1, group assignments/categorizations are based upon the initial S_{max} and $d_{failure}$ at RT and dry conditioning results. Displacement at failure at elevated temperature will be digitally archived but will not alter the first tier group determination. Likewise, if S_{max} (RT, dry) for a given Group I, II, or III adhesive is at or just above 10.0 MPa (1450 psi), then measurement of the 75% strength retention at elevated temperature will not reassign the adhesive to Group IV.

Adhesives that pass both second tier part one and part two requirements may be considered for additional ARL testing. Test data and metadata for adhesives that do not pass both second tier part one and part two requirements will still be retained in a digital archive to (1) allow for data to be matched against alternative SPD requirements (both public releasable and restricted distribution), (2) enable sharing of nonproprietary commercial and academic data to other DoD organizations with either similar or differing adhesive requirements, and (3) enable a feedback mechanism with commercial and academic formulators, specifically in determining where the requirements specified by ARL-ADHES-QA-001.02 rev 1.0 are difficult to meet. Such “lessons learned” could have potential value in addressing future research focus.

Testing progression protocols beyond the second tier are intended for ARL administrative/ operational use only.

9.0 References

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13. Department of Defense Test Method Standard, “Environmental Engineering Considerations and Laboratory Tests.” MIL-STD-810G, Laboratory Test Method 501.5, High Temperature, 31 October 2008, superseding MIL-STD-810F 1 January 2000.

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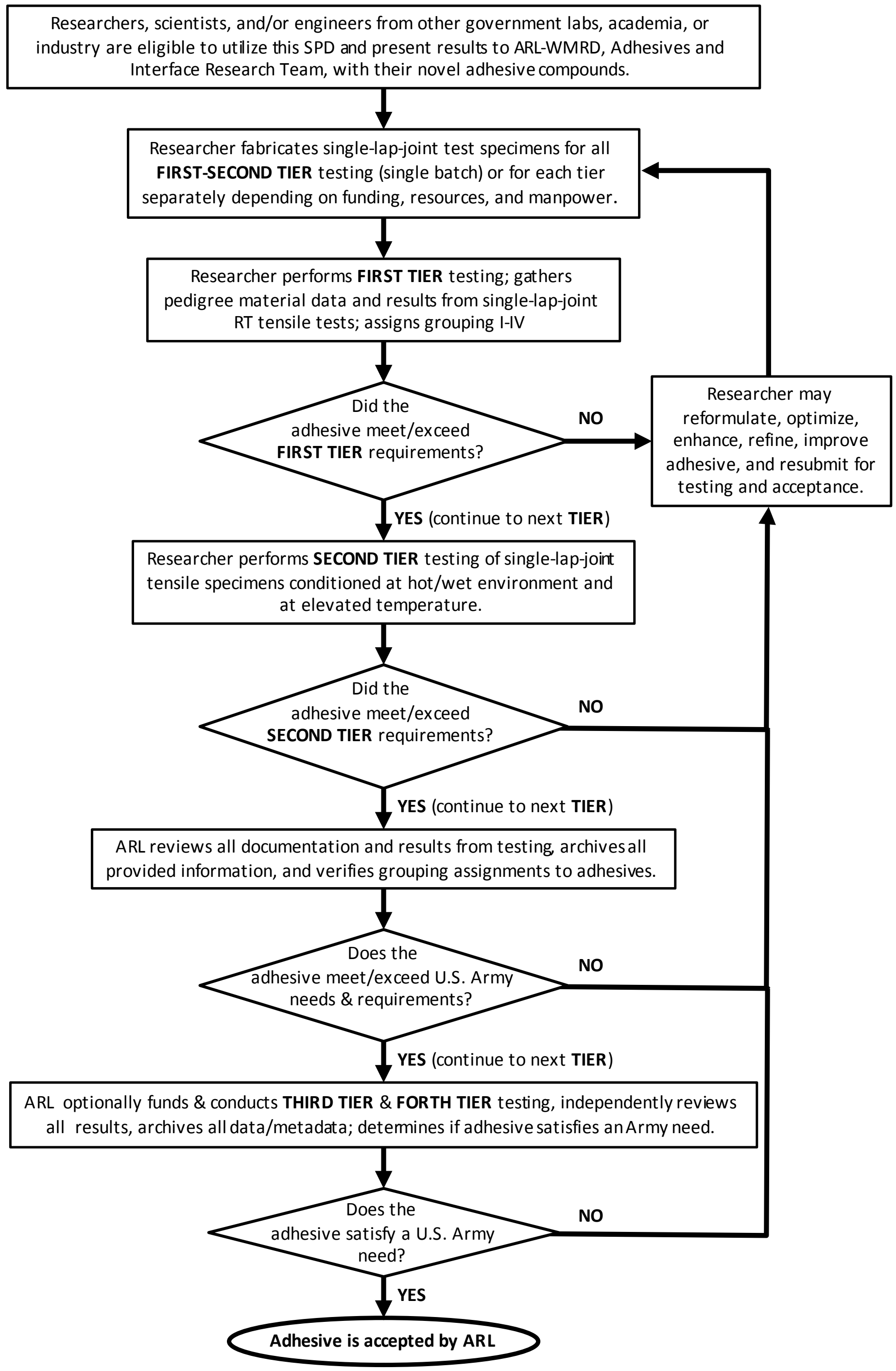
Appendix. Tier Progression Flow Chart



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14. ABSTRACT

This standard process description (SPD) provides the instructions needed in qualifying candidate adhesives for the screening tests specified in ARL-ADHES-QA-001.00, Sections 8.2 and 8.3. The single-lap-joint testing configuration relies upon a relatively small bond area between adherends, which can lead to high standard deviations in measured strengths. Geometrical misalignment and variations in bondline thickness are potential sources of inaccurate property measurements. Poor surface treatment of the adherends prior to bonding will increase the likelihood failure of testing following hot/wet conditioning as specified in ARL-ADHES-QA-001.00, Section 8.3. This SPD illustrates the techniques for the preparation and testing of adhesively bonded single lap joints. Supplemental drawings are illustrated for coupon and tooling fixture machining. Checklists are also provided to assist in documenting experimental metadata during fabrication, testing, and adhesive bondline thickness measurement.

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1.0	07/06/2011		Originally reviewed by Coatings, Corrosion, and Engineered Polymers Branch (CCEPB) ISO 9001:2008 working group and approved by CCEPB Chief as "Branch Ops SPD #L1028/35/40/44-01 Preparation, Testing, and Analysis of Adhesively Bonded Aluminum Single-Lap-Joints". Document not OPSEC approved for release.
2.1	4/6/2016		Updated per guidance by Coatings, Corrosion, and Engineered Polymers Branch (CCEPB) ISO 9001:2008 working group. Major revisions prepared by Adhesives and Interfaces Research Team. Sample preparation and testing techniques modified. Technically reviewed by 2 ARL reviewers external to the Adhesives and Interfaces Research Team. Reviewed by Chief (CCEPB). OPSEC approved for release to DoD components only. Simultaneously released as ARL-SR-0355.
2.2	4/6/2016		Technically reviewed by 2 ARL reviewers external to the Adhesives and Interfaces Research Team. Reviewed by Chief, CCEPB. OPSEC approved for public release; distribution unlimited. Operational procedures specific to ARL are omitted. Simultaneously released as ARL-SR-0356.

Authorization Signatures

Originator: ______________________ Date: 4/6/16

CCEPB Quality Assurance Coordinator ______________________ Date: 4/6/16

Approval: ______________________ Date: 4/6/16

*Refer to *Document Types and Naming Conventions* (LHC-PM-QA-202.00 rev 1.2, CERN, Geneva, Switzerland) for formatting recommendations.

1.0 Purpose

This document provides guidance from within the Adhesives and Interfaces Research Team of the Coatings, Corrosion, and Engineered Polymers Branch (CCEPB) and the US Army Research Laboratory (ARL) operating procedures for the preparation, testing, and evaluation of adhesively bonded single lap joints.

2.0 Scope

This standard process description (SPD) provides the instructions needed in qualifying candidate adhesives for the screening tests specified in ARL-ADHES-QA-001.00 rev 1.0, Subsections 8.2 and 8.3. The single-lap-joint testing configuration relies upon a relatively small bond area between adherends, which can lead to high standard deviation in measured strength. Geometrical misalignment and variation in bondline thickness are potential sources of inaccurate property measurements. Poor surface treatment of the adherends prior to bonding will increase the likelihood of failure in testing following hot/wet conditioning as specified in ARL-ADHES-QA-001.00, Subsection 8.3. This SPD illustrates the techniques for the preparation and testing of adhesively bonded single lap joints. Supplemental drawings are illustrated for the machining of coupons and fixture tooling. Checklists are also provided to assist in documenting experimental metadata during fabrication, testing, and adhesive bondline thickness measurement.

3.0 Policy

This SPD applies to all personnel developing, fabricating, testing, characterizing, and evaluating adhesives for bonding applications within the CCEPB.

4.0 Responsibilities

The Branch Chief, CCEPB, Adhesives Team Leader, and Principal Investigator(s) will ensure that all personnel working on adhesives development, fabrication, testing, characterization, and evaluation have and maintain full knowledge and/or proper training of this process, including equipment usage/operation, test procedures, and safety issues/factors before commencing relevant work within the CCEPB.

5.0 Requirements

All researchers performing adhesives development and evaluation work in the CCEPB must follow the procedures described within this SPD.

6.0 Terms and Definitions

For the purposes of this document, the following terms and definitions apply:

Adherend: As stated within ASTM D907-12a, an adherend is “a body held to another body by an adhesive.”[1] Specifically for this SPD, the bodies are 2 rectangular sheets of 2024 T3 aluminum alloy.

Adhesion Promoter: As stated within ASTM D907-12a, an adhesion promoter is “a substance used to improve bonding of the adhesive to the substrate.”[1]

APS – 3-aminopropyltrimethoxylsilane (Chemical Abstracts Service [CAS] registry number 13822-56-5): An amine functionalized silane coupling agent.

Bondline: As stated within ASTM D907-12a, bondline shall be "the layer of adhesive which attaches the two adherends."[1]

Coupling Agent: As stated within ASTM D907-12a, "A substance having functional groups that are capable of reacting with the surfaces of two different substances, thereby chemically bridging them."[1]

Database: As stated within ISO/IEC 2382-17:1999, a database shall be "a collection of data organized according to a conceptual structure describing the characteristics of these data and relationships among their corresponding entities, supporting one or more application areas."[2]

Digital Archive: The term "digital archive" shall be referred to as an electronic repository of digital data and metadata "to preserve the authenticity, reliability, integrity, and usability of such records" as stated in ISO 13008:2012(E).[3]

GPS – 3-glycidyloxypropyltrimethoxylsilane (CAS registry number 2530-83-8): An epoxy functionalized silane coupling agent.

Lap Joint: As stated within ASTM D907-12a, a lap joint shall be "a joint made by placing one adherend partly over another and bonding together the overlapped portions." [1]

Material Pedigree: The term "material pedigree" shall refer to the documentation used to ensure the traceability of an adhesive and its constitutive formulation ingredients to the original manufacturer.[4]

Metadata: As stated within ISO/IEC 2382-17:1999, the term "metadata" pertains to "data elements, possibly including their data descriptions, and data about data ownership, access paths, access rights and data volatility."[2]

MPS – 3-methacryloxypropyltrimethoxysilane [CAS registry number 2530-85-0]: A methacrylate functionalized silane coupling agent.

Surface Preparation: As stated within ASTM D907-12a, surface preparation shall pertain to "physical or chemical treatments, or both, applied to adherends to render suitable for bonding."[1]

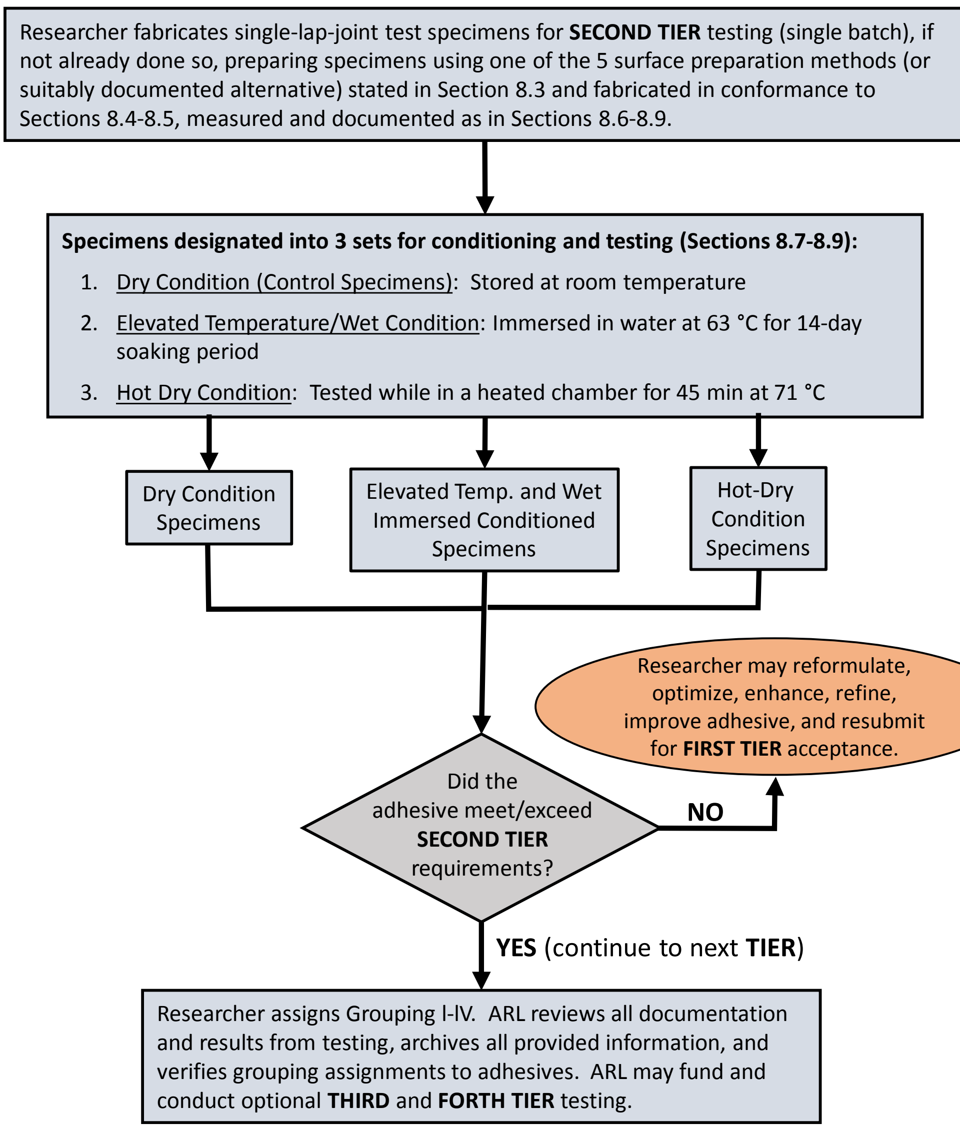
Trustworthiness: In the context of this SPD, trustworthiness relates to "judgments of reliability, dependability and competence"[5,6] of the experimental sample preparation and testing.

7.0 Records

Per Army Regulation (AR) 25-400-2, all records (including data and associated metadata) shall be considered to "have value beyond the business process, such as for historical, lessons learned, or research purposes; these are generally long-term records."[7] Records will be kept in physical laboratory notebooks and digitally archived for permanent retention, consistent with AR 25-400-2 code "TP" (i.e., Transfer Permanent).[7]

8.0 Preparation of Lap-Joint Coupon Panels and Testing of Single-Lap-Joint Test Specimens

The flow chart below illustrates the procedure to prepare and fabricate lap-joint coupon test specimens, as well as to test, record, and analyze data used by ARL to determine if the adhesive is acceptable for further consideration for higher-tier testing. Under this process, specimens must be tested in 3 environmental conditions—dry-room temperature, hot/wet, and dry-elevated temperature—with the data obtained, analyzed, and categorized by grouping prior to ARL's review.



8.1 Safety Precheck

Prior to the preparation or testing of single-lap-joint coupons or test specimens, a safety precheck shall be performed. The safety precheck shall focus on the following actions, and compliance shall be documented. If any safety issues arise, they must be resolved and corrected before proceeding.

- Address all protective clothing, protective equipment, and ventilation requirements.[8–13]
- Inspect all equipment to be used to identify and correct any damage or malfunction that could hinder operation or create a potential hazard.[14]
- Address all chemical hazards and ensure all material safety data sheets (MSDSs) for the chemicals to be used in the aluminum surface preparation and adhesives are readily available.[15]
- Address start-up for all equipment needed for the fabrication and testing processes. Refer to applicable internal operating procedures when needed to operate laboratory equipment.[16]

8.2 Lap-Joint Coupon Panel Dimensions and Thickness Measurements

The complete drawing with dimensions and tolerances of the aluminum lap-joint coupon panel shall conform to ASTM D1002-10, "Fig. 3 Optional Panel for Acceptance Tests Only"[17] (refer to Section 10.0, Appendix A, Test Panel Dimensions). A drawing of the panel geometry is shown in Fig. 1, having a recommended aluminum plate thickness of 1.62 ± 0.125 mm (0.064 ± 0.005 inch) with an overlap length of 12.7 ± 0.25 mm (0.5 ± 0.01 inch). Aluminum alloy 2024, T3 temper, is the recommended grade for testing this panel. (The panel has been modified with the inclusion of alignment holes for use in the tooling fixture.)

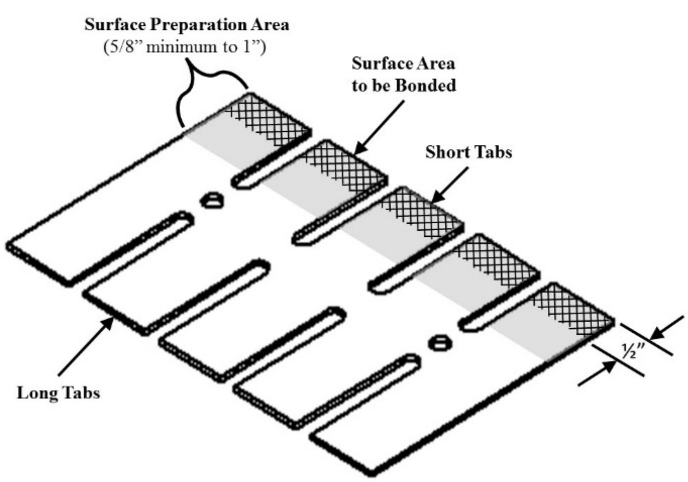


Fig. 1 Test panel design configuration used for single-lap-joint preparation and testing in accordance to ASTM D1002-10[17]

8.3 Surface Preparation

The first step in the fabrication of single-lap-joint test specimens is the preparation of the surfaces to be bonded. The surface preparation methods stated are appropriate for a wide range of adhesives, which are most commonly used by ARL. ARL also acknowledges that the outlined protocols may not be suitable for optimal bond performance for all adhesives. The surface preparation technique used, either recommended or alternative, shall be fully documented in a laboratory notebook with sufficient detail to serve as trustworthy database metadata. There are 4 distinct laboratory-scale surface preparation methods ARL performs for an aluminum substrate: Basic Untreated, Acetone Solvent Wipe, Grit Blasting, and Silane Coupling Agent. A general description of the procedures used in each method is listed here, followed by a more detailed description.

Basic Untreated Surface Preparation

- Basic cleaning of the surface with a clean, dry lint-free cloth

Acetone Solvent Wipe Surface Preparation

- Includes Basic Untreated method
- Uses acetone as a solvent to wipe clean the surface

Grit-Blasting Surface Preparation

- Includes Basic Untreated method
- Includes Acetone Solvent Wipe method
- Uses grit blasting to pretreat the surface before cleaning

Silane Coupling Agent Surface Preparation

- Includes Basic Untreated method
- Includes Acetone Solvent Wipe method
- Includes Grit-Blasting method
- Employs either GPS, MPS, or APS as a coupling agent following the other preparation

8.3.1 Basic Untreated Surface Preparation

This method is the most basic surface preparation technique and is used within all other procedures as a first step. Remove loose debris from the fabricated aluminum lap joint test panel using a clean and dry cloth or paper towel. Single-lap-joint testing results obtained from untreated aluminum panels are useful for troubleshooting suspect chemical and etch surface treatments by providing an estimate of the lowest bond strength for the adhesive. The chemical or etch surface treatment process needs to be examined if it provides similar or lower bond strength values as those obtained from untreated surfaces.

8.3.2 Acetone Solvent Wipe Surface Preparation

This method includes Subsection 8.3.1 preparation and also involves wiping the bonding surfaces of the lap-joint coupon panel tabs to be adhered with acetone (CAS #67-64-1) with a soft, lint-free cloth and allowing it to air dry, as shown in Fig. 2. This method of surface preparation is recommended for very rapid screening tests, such as studying/investigating process optimization (i.e., mixing, application, and cure) of an adhesive with unfamiliar handling characteristics.

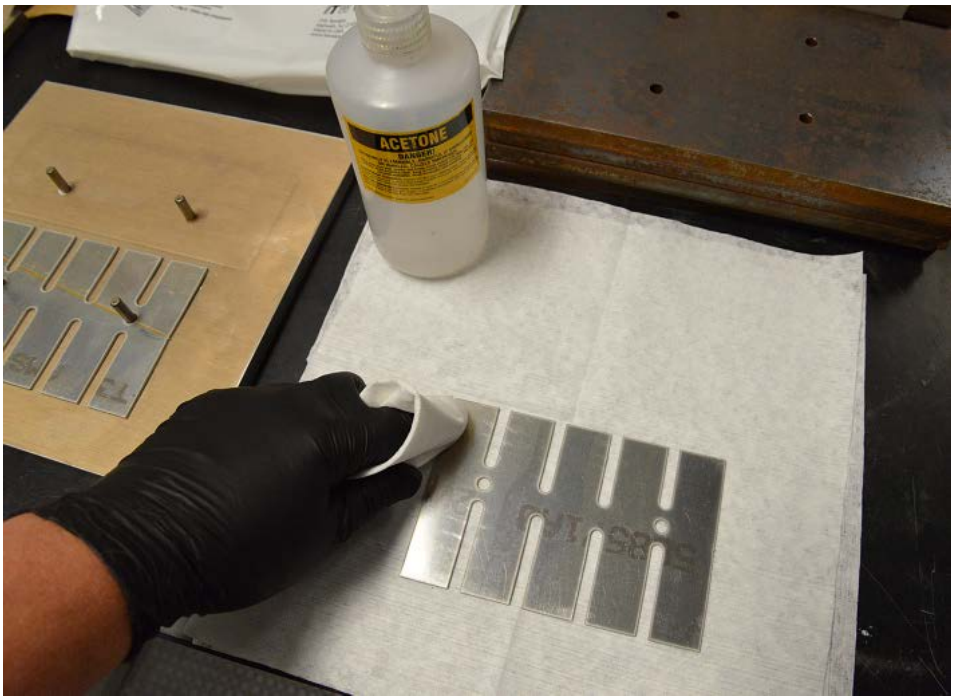
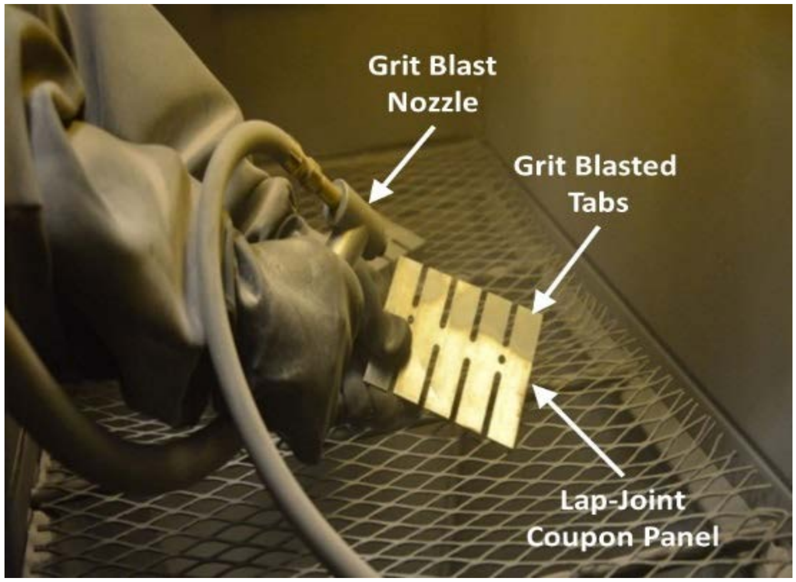


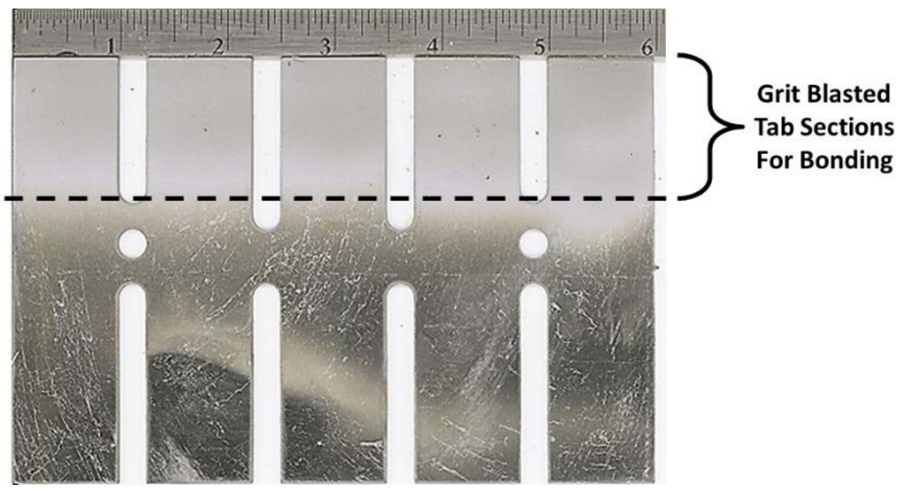
Fig. 2 Surface treating the lap-joint coupon panel using the acetone solvent wipe surface preparation method

8.3.3 Grit Blast Surface Preparation

This method includes Subsections 8.3.1 and 8.3.2 preparation. After the lap-joint coupon panel is cleaned of dirt and debris and is solvent wiped, the bonding surface is then ready and submitted for the grit-blasting process. Virgin 54- to 60-grit aluminum oxide abrasive blasting media shall be used for the grit-blasting process.[18] It is critical that unused aluminum oxide is used at all times during this procedure. Grit-blasting cabinets are typical commodity laboratory equipment that can have multiple users, which can result in significant cross-contamination of the grit-blasting medium. The media also has a finite life in the blasting process. This critical application requires fresh, sharp-edged particles of a specific size. Grit-blasting cabinets using compressed air shall be fitted with a moisture separator. The grit-blasting cabinet shall be equipped with a foot-operated blast gun capable of at least 12 cubic feet per minute (CFM) and a 100-CFM dust collector. Grit blasting is to be repeated until the bonding surfaces of each short tab of the lap-joint coupon panel are visually uniform in color and appearance. For aluminum lap-joint panels the surface appears gray and dull as shown in Fig. 3. Further guidance can be found in TT-C-490F and SSPC-SP 10/NACE No. 2.[19,20] Heavy surface oxidation may be removed prior to grit blasting using an abrasion pad (Scotch-Brite 048011-65530 A VFN, or equivalent), as shown in Fig. 4.



(a)



(b)

Fig. 3 Image showing the grit-blasting surface preparation method applied to the bonding tab surfaces of a lap-joint coupon panel inside the grit-blasting cabinet and note the typical nozzle stand-off distance (a) and an image of the finished grit blasted coupon panel (discolored tab sections due to abrasion) (b)

Fig. 4 Heavy surface oxidation may be removed prior to grit blasting using an abrasion pad (Scotch-Brite 048011-65530 A VFN, or equivalent)

Upon removal from the grit-blasting cabinet, make sure the abraded portions of the lap-joint panel's tabs are only in direct contact with either other grit-blasted panel tabs or the air. The lap-joint coupon panel is further cleaned using a high-volume gas spray, such as a pressurized stream of nitrogen gas, to remove loose dust and grit blast debris particles on the surface, as shown in Fig. 5. ARL typically uses bottled or in-house nitrogen gas to avoid surface contamination at approximately 690 kPa (100 psi) outlet pressure, directed from a 6.35-mm (0.25-inch) inner diameter plastic tubing to concentrate the flow (orange tubing shown in Fig. 5). Never use house-compressed air, since it can contain too many contaminants, such as dirt, water, and oil. It is very important when using this method that panels be bonded within 4 h after gas spray cleaning to minimize exposure to ubiquitous airborne contaminants and moisture degradation.

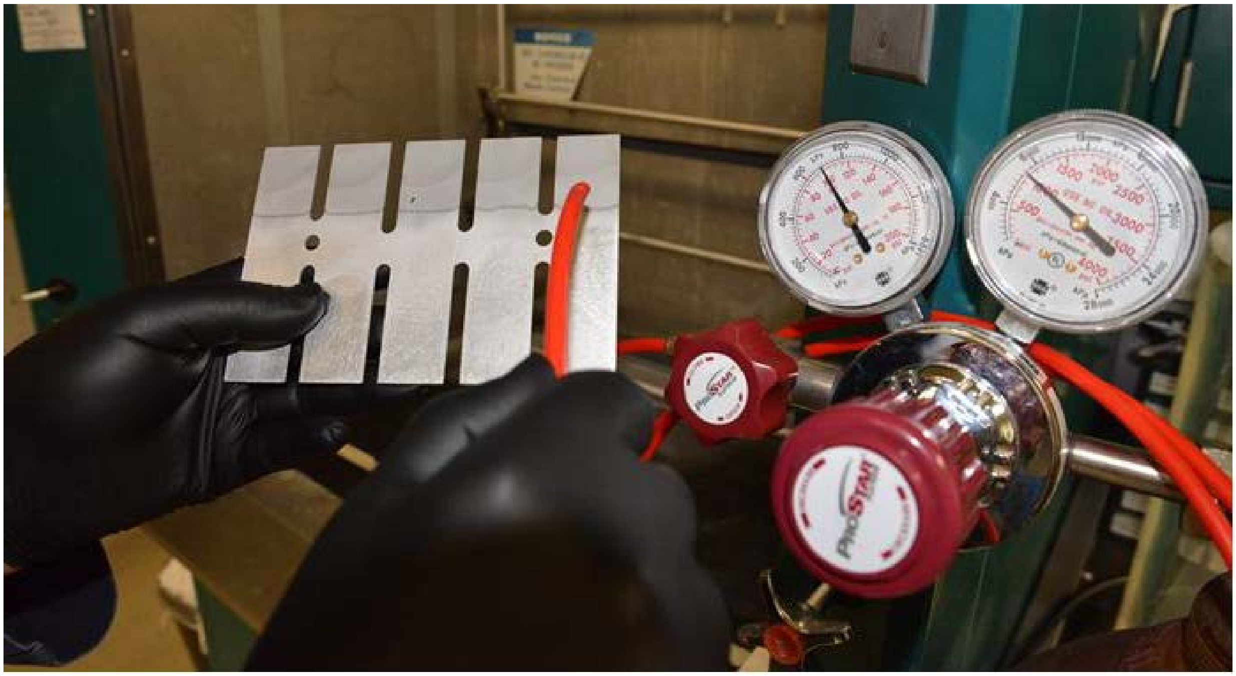


Fig. 5 View showing the typical gas spray cleaning process used to remove residual surface grit medium after the grit-blasting process from tab bonding surfaces

8.3.4 Silane Coupling Agent Pretreatment Preparation

The preparation for all silane coupling agent pretreatment methods (e.g., GPS, MPS, and APS) includes first performing the steps outlined in Subsections 8.3.1, 8.3.2, and 8.3.3: basic surface cleaning, solvent swiping, and grit blasting the lap-joint coupon panel tabs, respectively.

8.3.4.1 GPS Silane Coupling Agent (3-glycidyloxypropyltrimethoxylsilane) Solution Preparation

The following steps outline the preparation process for GPS silane coupling agent solution:

8.3.4.1.1 Mix 4 L by volume of 90:10 ethyl alcohol and water (e.g., 3.6 L of ethyl alcohol [64-17-5] and 0.4 L of deionized [DI] water). Use a large graduated cylinder and a glass funnel to measure and pour the components, making sure to clean the glassware thoroughly before and after use by scrubbing and/or brushing with soap and water, then rinsing with DI water. Gently shake the solution after the components are all added.

8.3.4.1.2 Next, to the 90:10 alcohol mixture, add with a clean glass pipette enough acetic acid [64-19-7] to adjust the pH to 4.5 and label the container "90:10, EtOH:H2O, with Acetic Acid, pH = 4.5, For GPS."

8.3.4.1.3 Transfer 200–300 mL of the acidic EtOH:H2O mixture to a clean glass flask and add 1% by weight GPS silane coupling agent material to form the solution. Stir moderately on a magnetic stir plate using a clean stir bar for 30–60 min. Use of a clock or stopwatch is recommended.

8.3.4.1.4 Transfer the mixed solution into a glass container large enough to coat the tabs of the coupon panel. Waste generation of about 250 mL of excess solution is common for each use. Waste can be labeled as "____ mL 90:10 EtOH:H2O with 1 weight % GPS silane."

8.3.4.1.5 The silane coupling agent solution must be applied to the single-lap-joint coupons within 4 h of initial mixing. Mixed silane coupling agent solutions are to be considered expired after 4 h.

8.3.4.2 GPS Silane Coupling Agent Application

After the grit-blasting process (Subsection 8.3.3), the abraded lap-joint coupon panel tabs shall be treated with the GPS silane coupling agent solution bath, prepared in Subsection 8.3.4.1. A thin chemical dip coating shall be applied to the tabs by immersing them for 90 s within the bath, as shown in Fig. 6. Once removed from the bath, immediately remove any excess GPS silane coupling agent solution using a high-volume nitrogen gas spray, using approximately 690 kPa (100 psi) outlet pressure directed from a 6.35-mm (0.25-inch) inner diameter plastic tubing, as shown in Fig. 7. Caution must be taken to avoid rewetting the tabs once dry. Using a low angle approach, start the nitrogen gas spray at one end of the panel tabs, remove the excess from beneath the aluminum substrate (the back side opposite of the bonding surface); this method will reduce splatters and the inadvertent redeposition of GPS silane solution back onto the bonding surface. It is crucial to not touch the treated tabs (specifically, the bonding area), even with gloves, after treatment is completed. Bake the treated lap-joint coupon panel for 1 h at 100 °C, +/–3 °C (212 °F, +/–5 °F) in a closed static (nonconvection) oven. (The GPS coupling agent shall be applied within 4 h after the grit-blasting process.) Store the treated lap-joint coupon panel in an inert atmosphere until it is ready for bonding. The bonding process shall begin no later than 4 h after treatment.

Fig. 6 Immersion of grit-blasted primed aluminum single-lap-joint coupon panel tabs submerged in the GPS silane coupling agent solution bath for 90 s by the clock

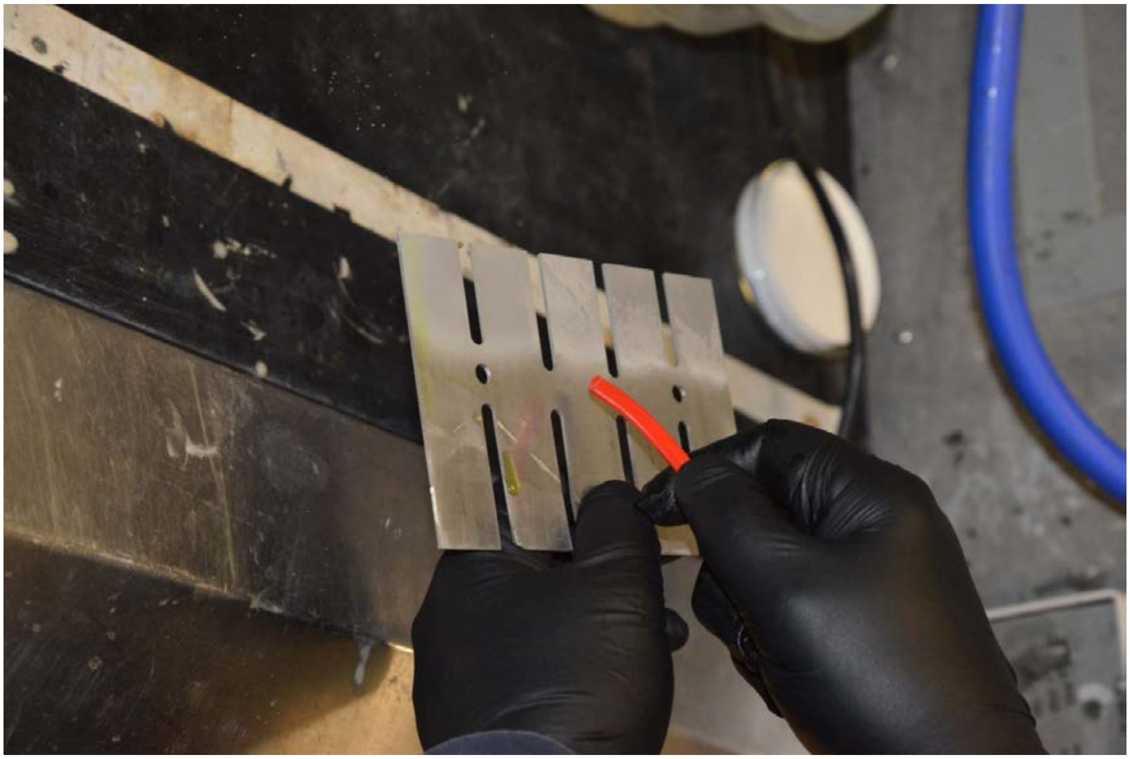


Fig. 7 The removal of excess GPS silane coupling agent solution from the treated lap-joint tabs using the directed nitrogen gas method

8.3.5 MPS Silane Coupling Agent (3-methacryloxypropyltrimethoxysilane) Pretreatment Preparation

Follow the surface treatment procedures outlined in Subsections 8.3.4 and 8.3.4.1 and substitute the GPS with MPS.

8.3.6 APS Silane Coupling Agent (3-aminopropyltrimethoxylsilane) Pretreatment Preparation

Follow the surface treatment procedures outlined in Subsections 8.3.4 and 8.3.4.1 and substitute the GPS with APS but eliminate the acetic acid pH adjustment step. APS will self-hydrolyze its methoxysilane groups into hydroxyl groups and does not require pH adjustment.

8.4 Adhesive Mixing

It is beyond the scope of this SPD to provide universal instructions for the mixing and handling of the adhesive component. As a starting point, the operator should refer to the manufacturer's technical data sheet (TDS) and MSDS for preliminary guidance. Operator safety is the first and most important consideration in preparing single lap joints due to the potential for chemical exposure. Once any safety concerns are addressed, the key factors in fabricating a reliable single lap joint are ensuring a complete cure, ensuring consistent bond thickness, and minimizing voids. Therefore, the operator should first prepare (if possible) a bulk sample of the adhesive in the safety of a ventilated fume hood to gauge an experimental understanding of the handling characteristics. The operator should note the adhesive's viscosity, pot life, and if normal atmospheric conditions (oxygen or moisture) inhibit the cure. If the adhesive does not gel at room temperature, or additional postcuring is required, the operator should observe for out-gassing and large exothermic reactions at elevated temperature. The operator will note any possible handling and cure behavior of the adhesive that will lead to incomplete cure, inconsistent bond thickness, or voiding (i.e., formation of voids and/or porosity) within the single lap joints.

The operator will document in a laboratory notebook with sufficient detail to serve as trustworthy database metadata the adhesive preparation procedure, the method of application of the adhesive to the single-lap-joint samples, cure cycle, mixing to applied pressure time, pressure during cure, temperature at sample preparation, and the ambient temperature and humidity during sample preparation.

8.5 Single-Lap-Joint Coupon Panel Assembly and Fabrication

The single-lap-joint coupon panel tooling fixture provides joint overlap and alignment control when used with the test panels described in Subsection 8.2. An illustration indicating the tooling's stacking assembly to fabricate single-lap-joint coupon panels is depicted in Fig. 8. The actual tooling fixture is shown in Fig. 9, with the stacking sequence to fabricate a single-lap-joint coupon panel illustrated in Figs. 10–19. Complete engineering drawings, with dimensions for tooling, are provided in Appendix B.

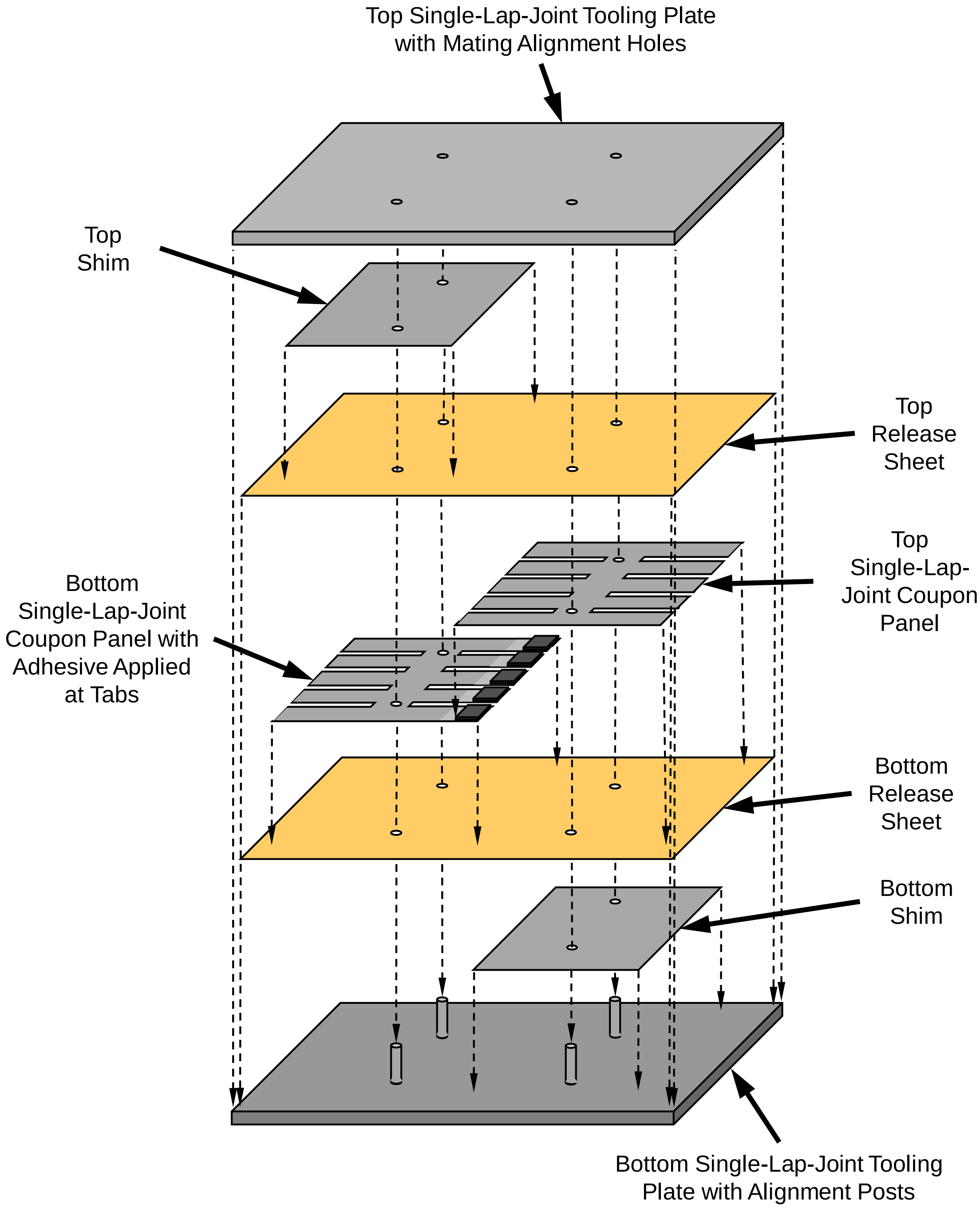


Fig. 8 The tooling stacking assembly for the fabrication of a single-lap-joint coupon panel

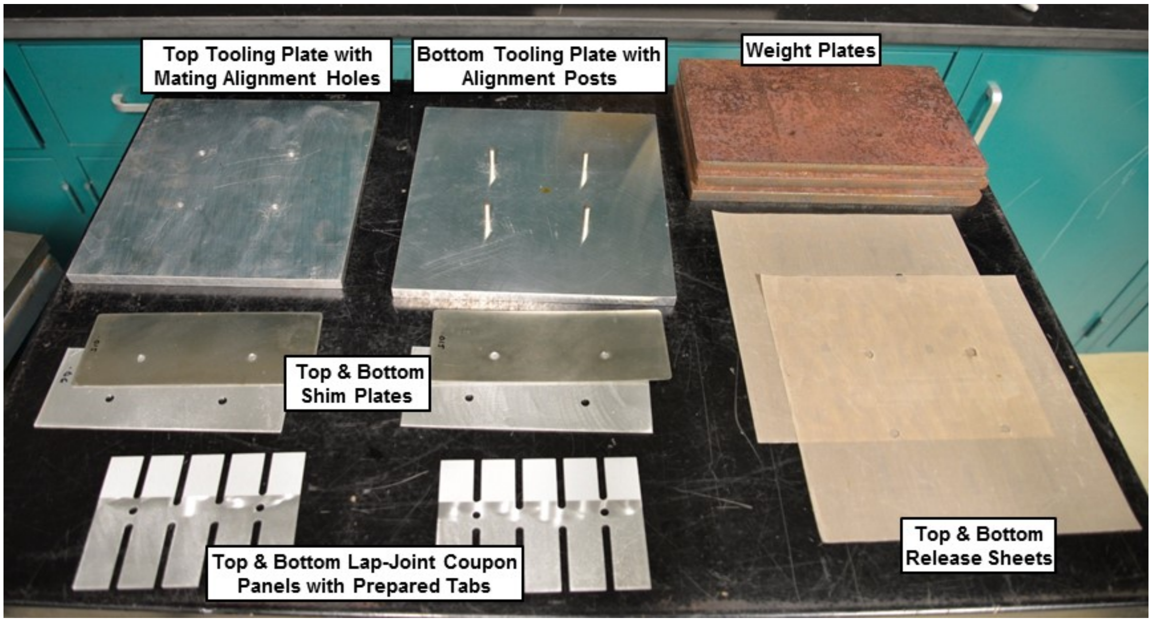


Fig. 9 Single-lap-joint tooling fixture components, including release sheets to prevent bonding of the tooling fixture and shim plates to control bondline thickness

8.5.1 Single-Lap-Joint Coupon Panel Assembly and Fabrication Procedure

8.5.1.1 Step 1

Make sure the work area to assemble the lap-joint tooling fixture, as well as the surfaces of each tooling fixture component, is clean, dry, and free of any dirt and debris. Start by placing the bottom tooling plate with alignment posts on a sturdy flat, level surface. Inspect the alignment posts to ensure they are not bent, loose, or damaged. Figure 10 shows the bottom lap-joint tooling plate for the base of the assembly.

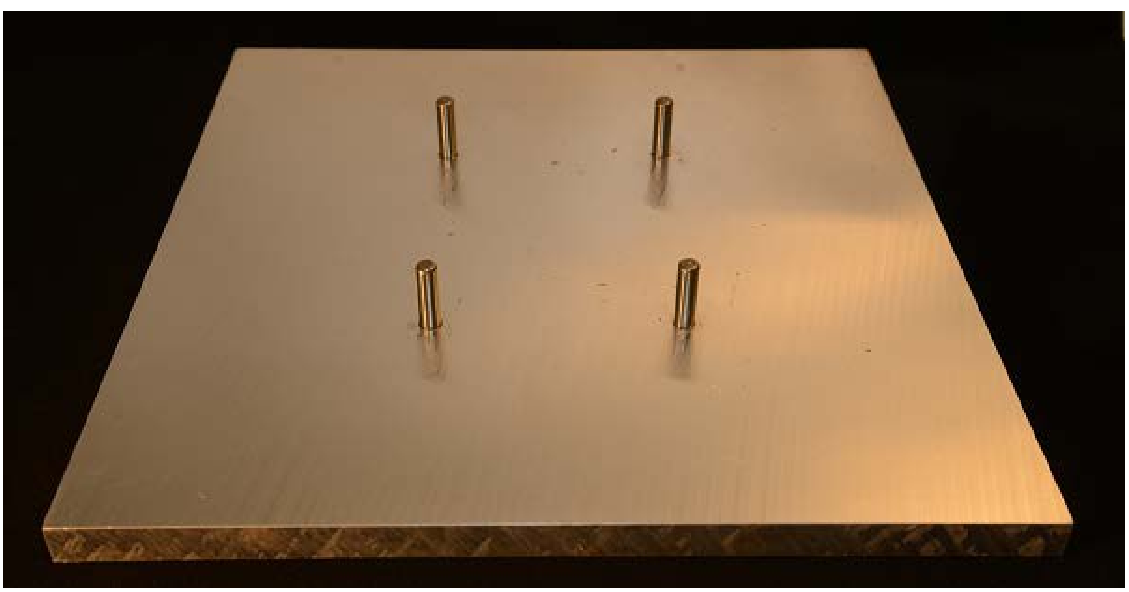


Fig. 10 Single-lap-joint tooling fixture stacking sequence: bottom tooling fixture plate with alignment posts

8.5.1.2 Step 2

Place the bottom shim plate(s) for the required controlled bondline thickness as needed onto the bottom tooling fixture plate, along one side, as the next level in the stacking sequence. Shims should slide freely over posts and lay flat as shown in Fig. 11.

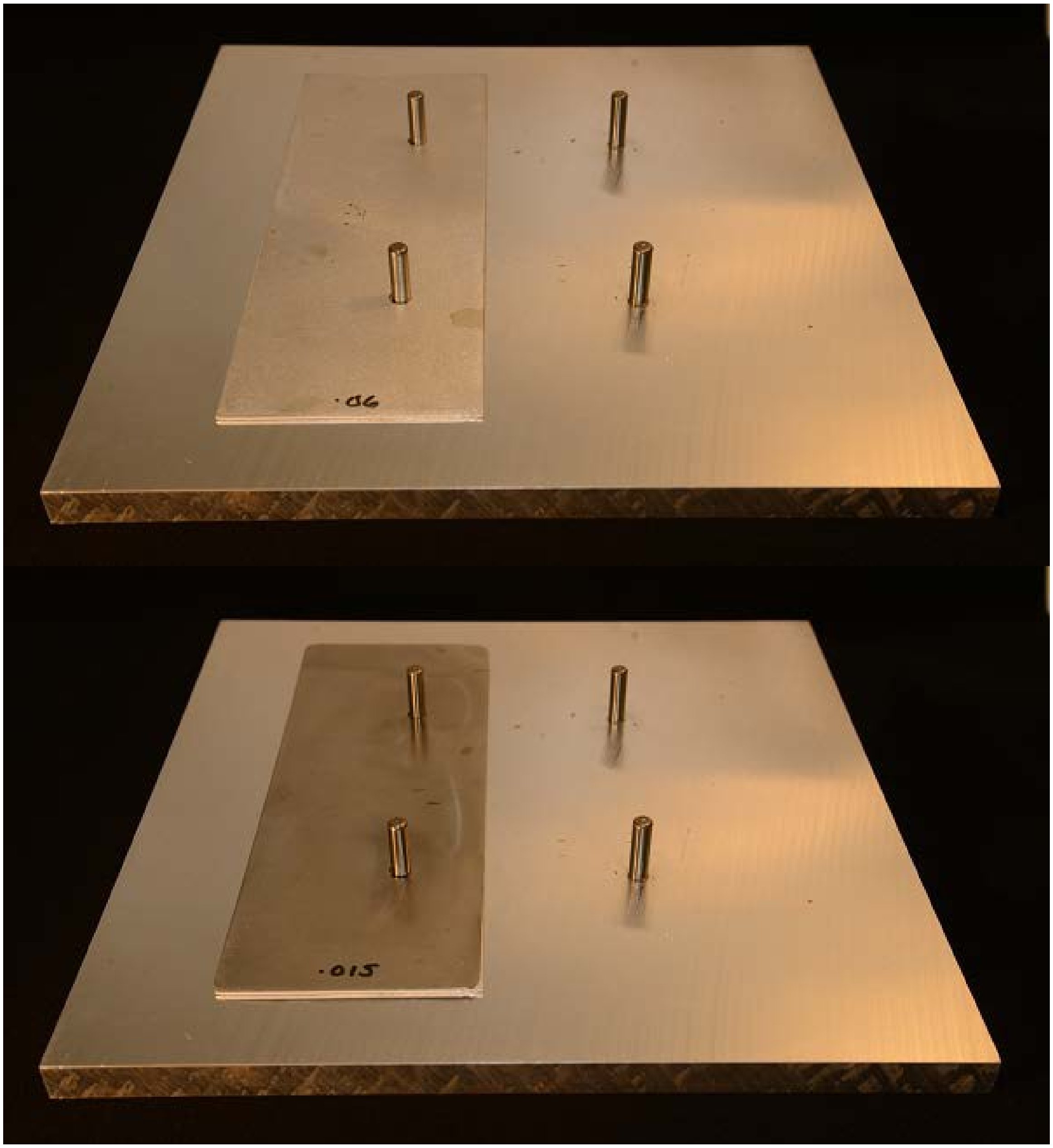


Fig. 11 Shim plates placed on top of the bottom tooling fixture plate. Note the thicker shim plate (top) compensates for the single-lap-joint panel thickness, and the thinner, secondary shim plate (bottom) sets the bondline thickness

8.5.1.3 Step 3

Place the bottom release sheet as shown in Fig. 12. It is highly recommended to use release sheets to ease in dismantling the tooling fixture and removing the lap-joint panel specimen from the tooling due to adhesive overflow.

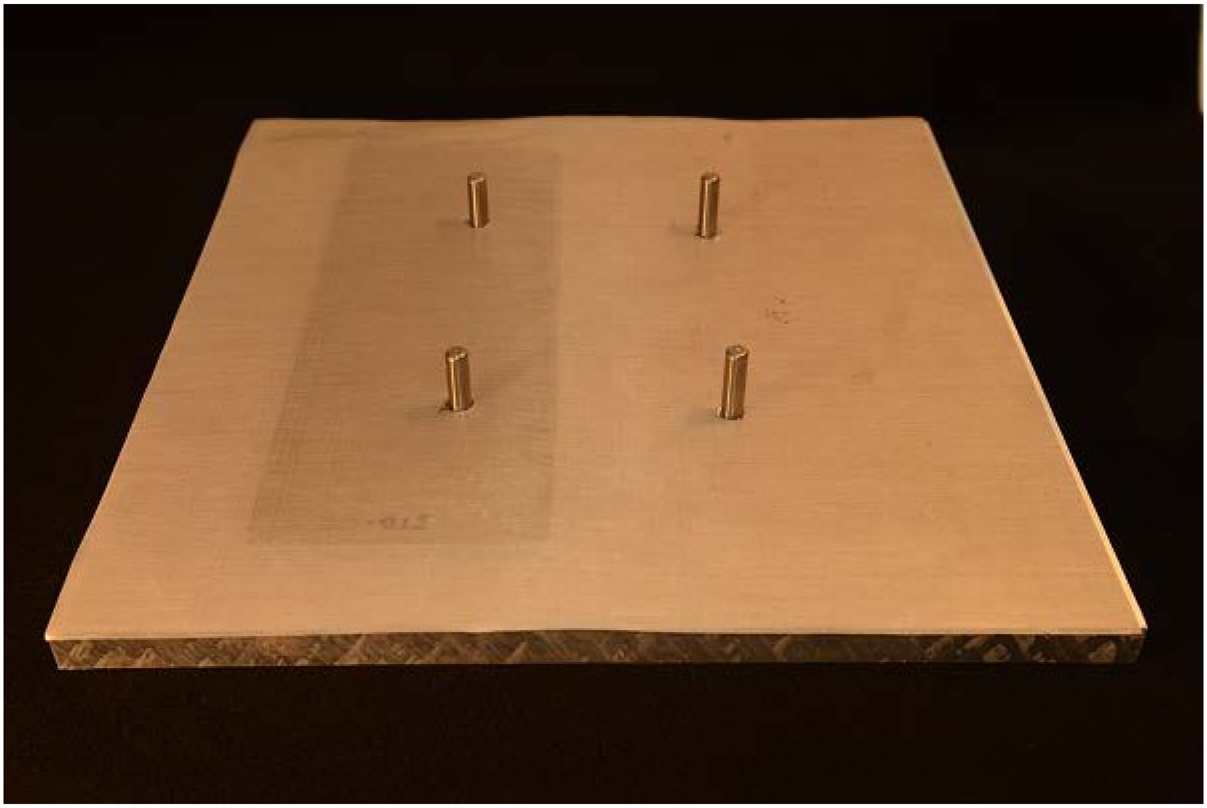


Fig. 12 Placement of the bottom release sheet layer

8.5.1.4 Step 4

Place the bottom lap-joint coupon panel opposite to the bottom shim on top of the release sheet, tabs facing inward, with the prepared tab surfaces facing upward as shown in Fig. 13. The lap-joint panel should slide freely over alignment posts with little lateral movement.

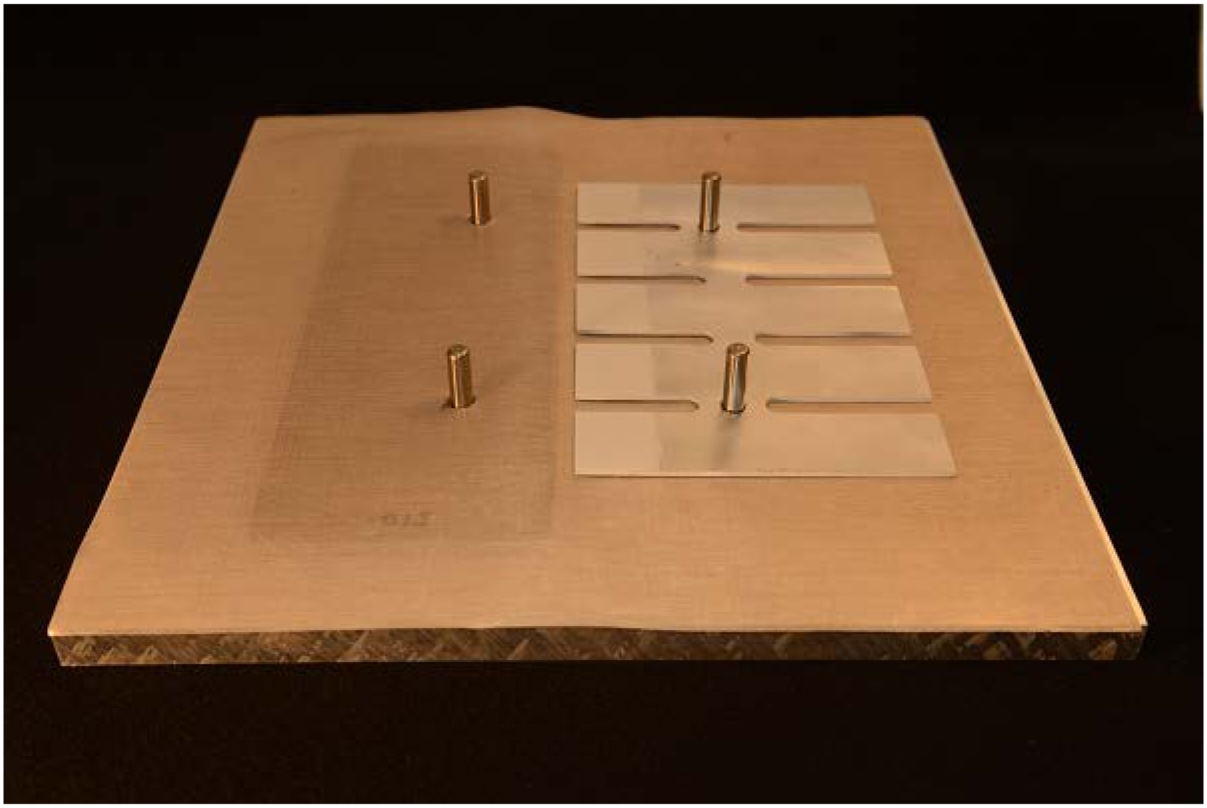


Fig. 13 Placement of bottom prepared single-lap-joint panel

8.5.1.5 Step 5

Apply the adhesive to a sufficient length on the prepared tab surfaces of the bottom single-lap-joint coupon so that the adhesive will cover a space approximately 6 mm (0.25 inch) longer than the full prepared 12.7-mm (0.50-inch) bond overlap area, as shown in Fig. 14.

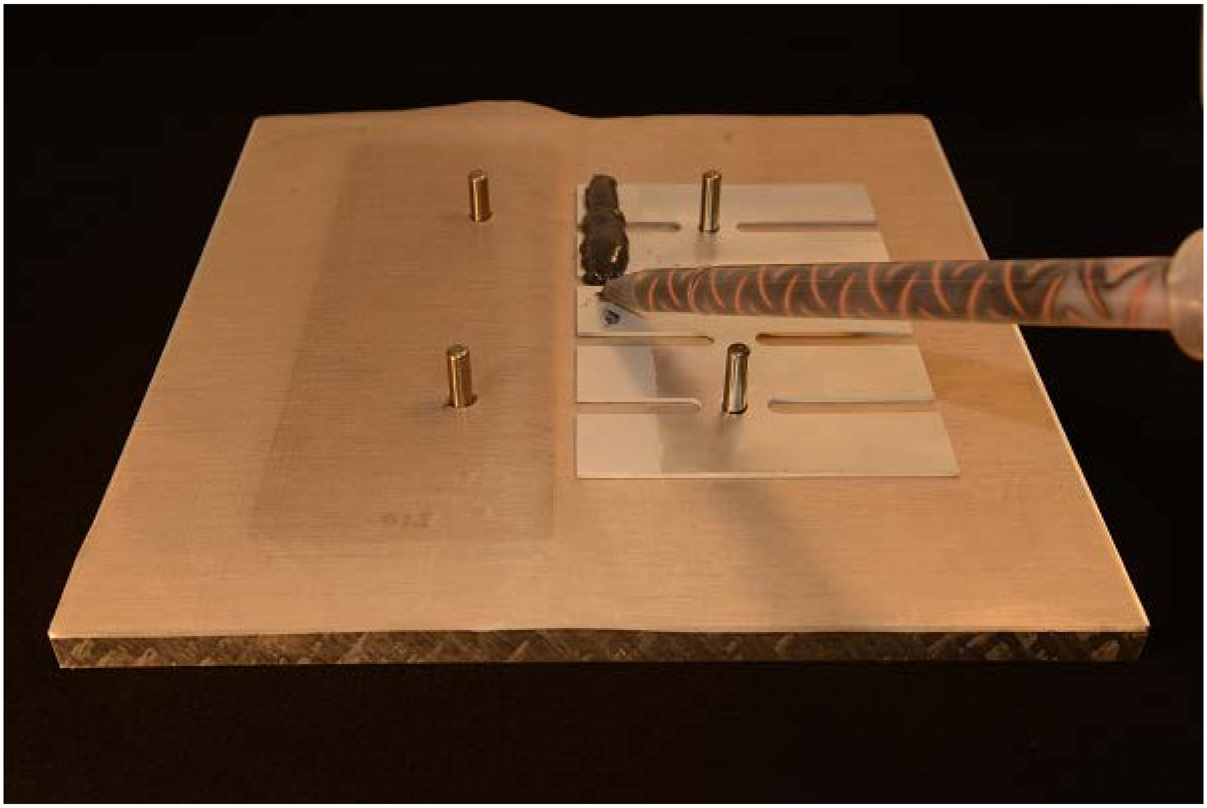


Fig. 14 Application of adhesive to prepared tabs onto bottom lap-joint coupon panel

8.5.1.6 Step 6

Place the top lap-joint coupon panel onto the adhesive opposite the bottom coupon panel with its prepared tabs facing downward as shown in Fig. 15. The bonded panels will be held rigidly in place by the alignment posts during curing. In addition, the length of the overlap will be controlled, and shims will provide a fixed bondline thickness.

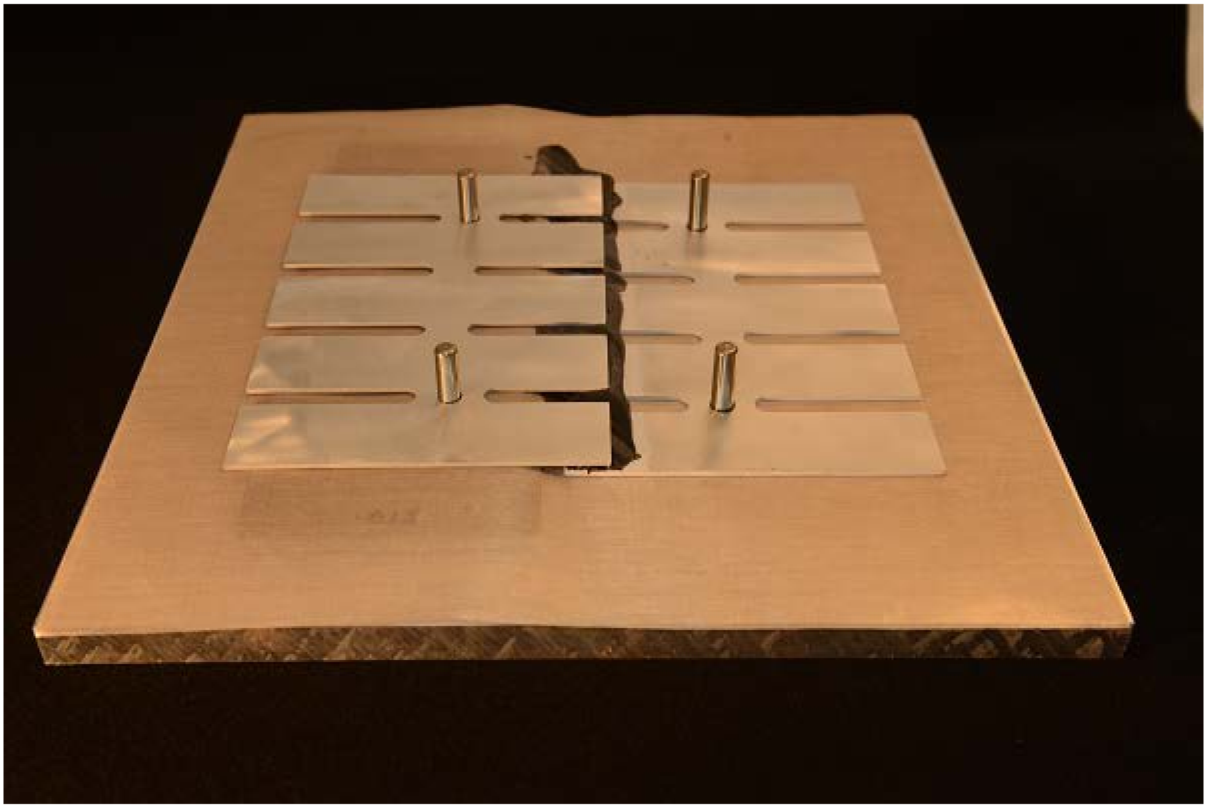


Fig. 15 Placement of top lap-joint panel

8.5.1.7 Step 7

Place the top release sheet over the lap-joint coupon panels as shown in Fig. 16. Release sheets provide a layer of protection for the other tooling components from the adhesive while being processed.

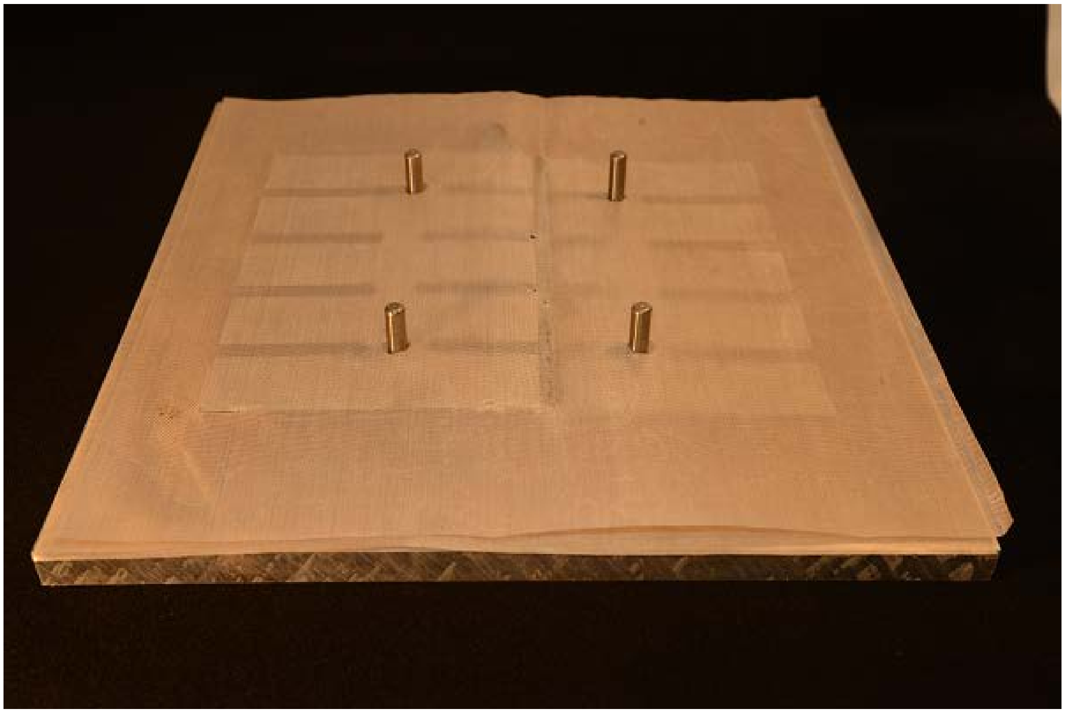


Fig. 16 Placement of top release sheet

8.5.1.8 Step 8

Place the top shim(s) over the right side of the alignment posts as shown in Fig. 17. The single-lap-joint tooling fixture configuration provides thickness and overlap controls through the use of the shims and alignment posts, respectively. In the example shown, the shims will produce an approximately 0.762-mm (0.030-inch) bondline thickness. A bondline thickness of 0.762 mm (0.030 inch) is a recommended starting point, but this may not be optimal for all adhesives.[21–23] Bondline shims are also available in 0.127 mm (0.005 inch), 0.381 mm (0.015 inch), and 1.14 mm (0.045 inch), with additional thicknesses available by acquiring the needed shim stock.

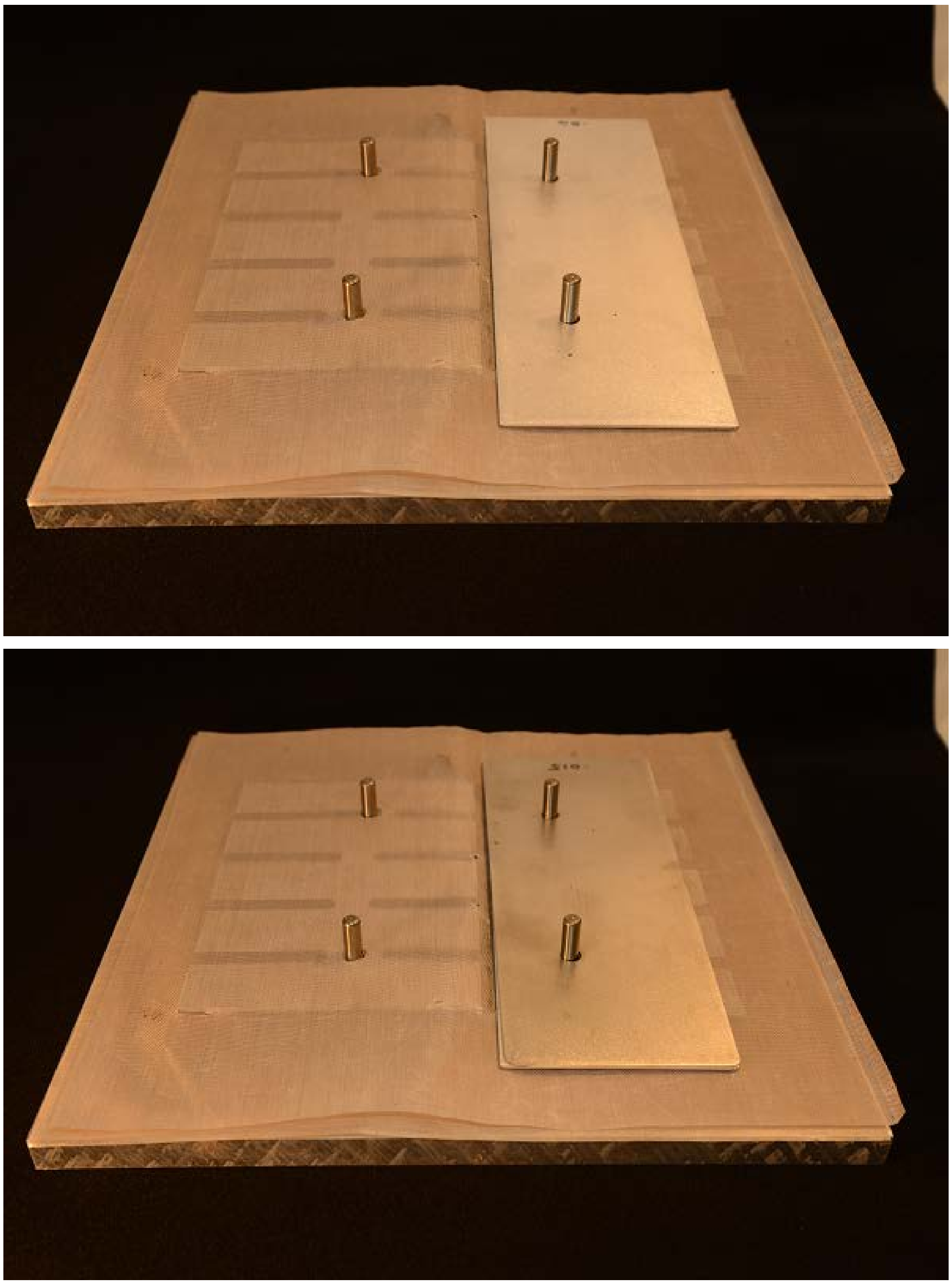


Fig. 17 Placement of the top shim plate over the top release sheet, aligned with the lap-joint coupon panel. (The shim plates match the coupon and bondline thickness to evenly offset the top single-lap-joint panel.)

8.5.1.9 Step 9

Place the top tooling fixture plate with the alignment holes to complete the tooling package as shown in Fig. 18.

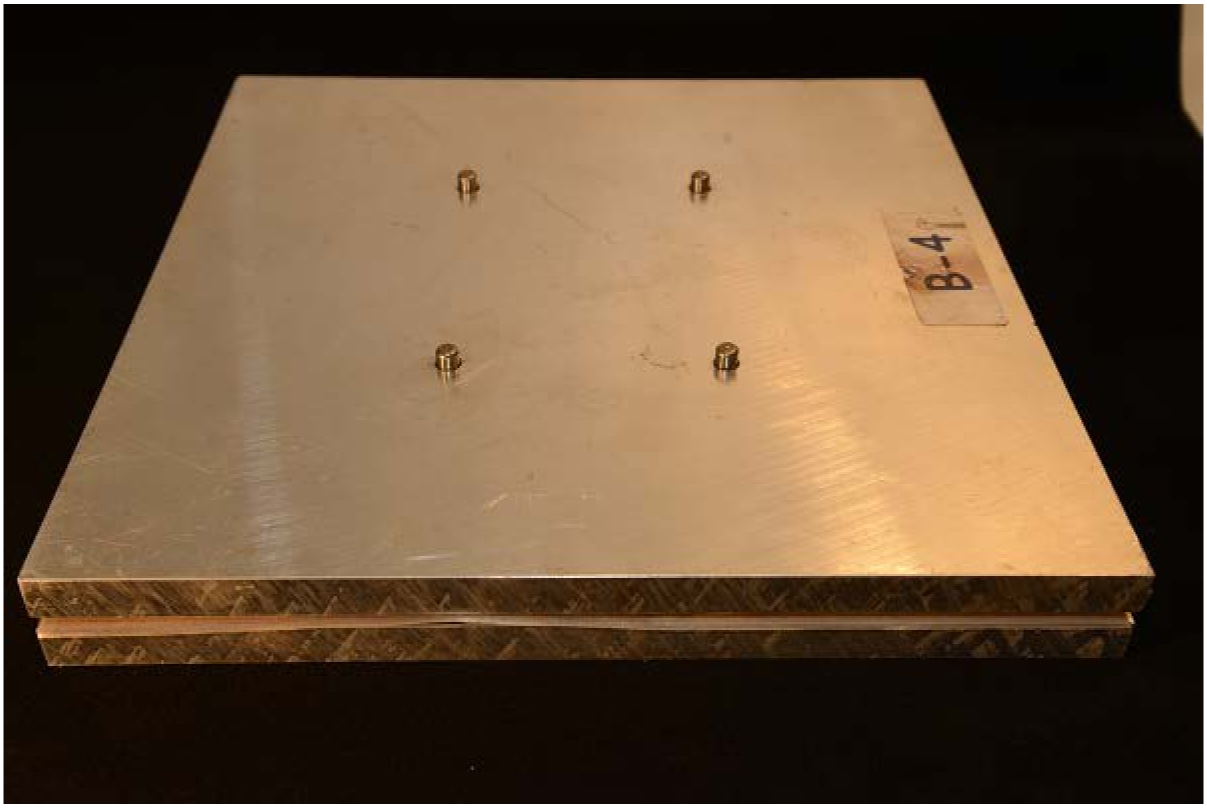
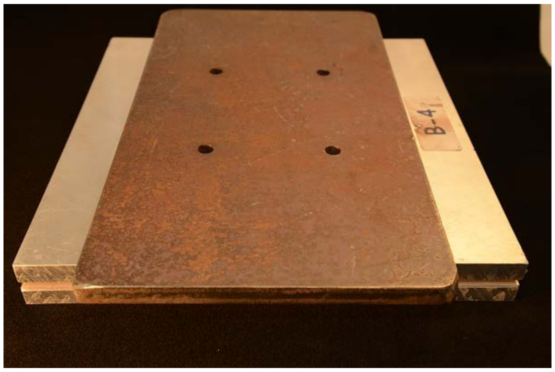


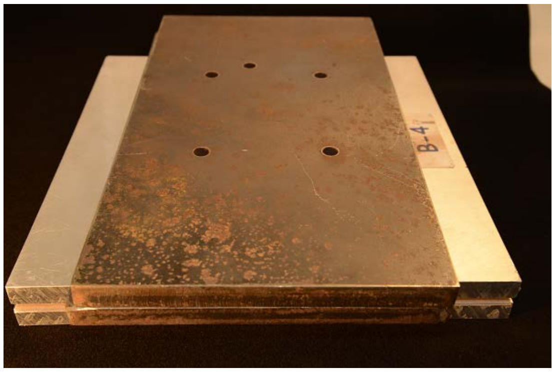
Fig. 18 Completed lap-joint panel tooling package

8.5.1.10 Step 10

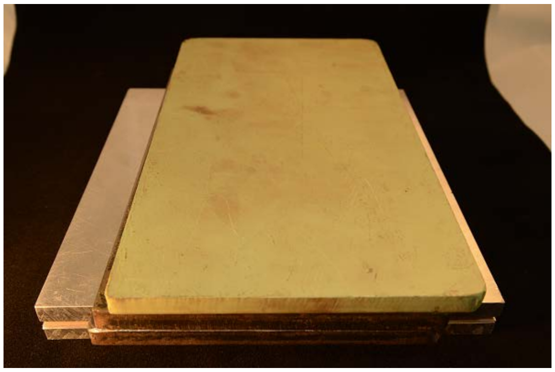
For simplicity ARL incorporates the stacking weighted plate method of applying pressure (~7.8 KPa) to the single lap joints during bonding as shown in Fig. 19. Three steel plates (5.4 kg each) are stacked on top of the single-lap-joint tooling fixture during cure. (The first stacking plate contains alignment holes that also mate with the alignment posts of the bottom tooling plate.) To obtain the needed pressure, 3 stacking plates are used. Using this method, the pressure can also be adjusted as necessary to optimize for specific adhesives.



a. Single-stack weighted plate setup



b. Double-stack weighted plate setup



c. Triple-stack weighted plate setup

Fig. 19 Single-lap-joint tooling fixture weighted plate stacking method with additional weighted plates for increased bonding pressure

8.5.1.11 Step 11

If the adhesive is a room temperature cure material, no further processing assistance is required. Conversely, if heat is required for the curing, the tooling can be placed in an oven. The tooling fixture can also be vacuum-bagged and heated in an oven, vacuum-bagged for autoclave processing, or simply processed in a hydraulic press where higher bonding pressure is required (the last 2 methods would not require any plate stacking). Furthermore, the tooling fixture can also be vacuum-bagged and backfilled with a nitrogen gas purge for adhesives that are moisture or oxygen sensitive. Figure 20 shows the tooling fixture placed within a typical convection oven, with unobstructed circulated airflow around the tooling package. Thermal lag can be compensated for by referencing the cure schedule to a thermocouple measurement taken from between the tooling fixture plates. Alternative methods can also be used during the curing process as dictated by the adhesive.

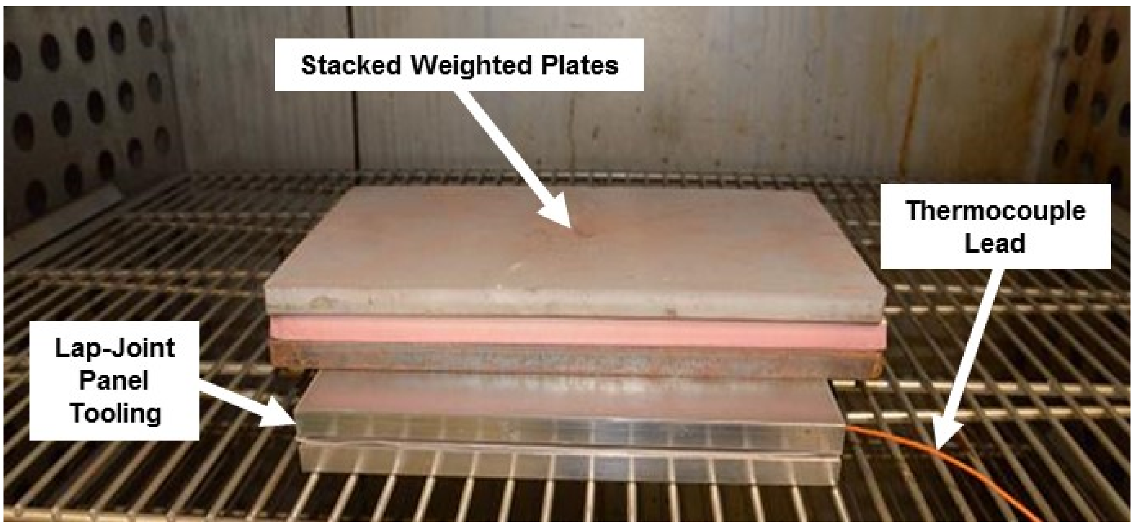


Fig. 20 Representative example of the plate-loaded single-lap-joint tooling fixture undergoing oven cure at elevated temperature

8.5.1.12 Step 12

Remove the cured and bonded single-lap-joint coupon panel from the tooling fixture as shown in Fig. 21. Precautions should be taken to avoid unnecessary stresses in bending the panel in cases where the alignment holes are binding to the locating pins of the tooling fixture. Excessive force may degrade the bond strength of the joint.

Separate each individual single-lap-joint coupon specimen from the bonded panel as shown in Fig. 22. It is normal to observe adhesive side overflow of the bonded single-lap-joint panel and the formation of adhesive fillets. For added stability during the cutting process, cardboard shim stock can be placed under the single-lap-joint coupon panel to keep it flat, steady, and firm, and will reduce chatter and vibration when passing through the blade. Separate each coupon specimen using a band saw to rough-cut between each specimen and remove the excess overflow adhesive from the sides of the joints. The saw blade used for this demonstration contained 19 mm (0.75 inch) × 0.90 mm (0.035 inch) × 0.3–0.5/teeth per mm (8–12/teeth per inch), which is sufficient to produce a clean cut.

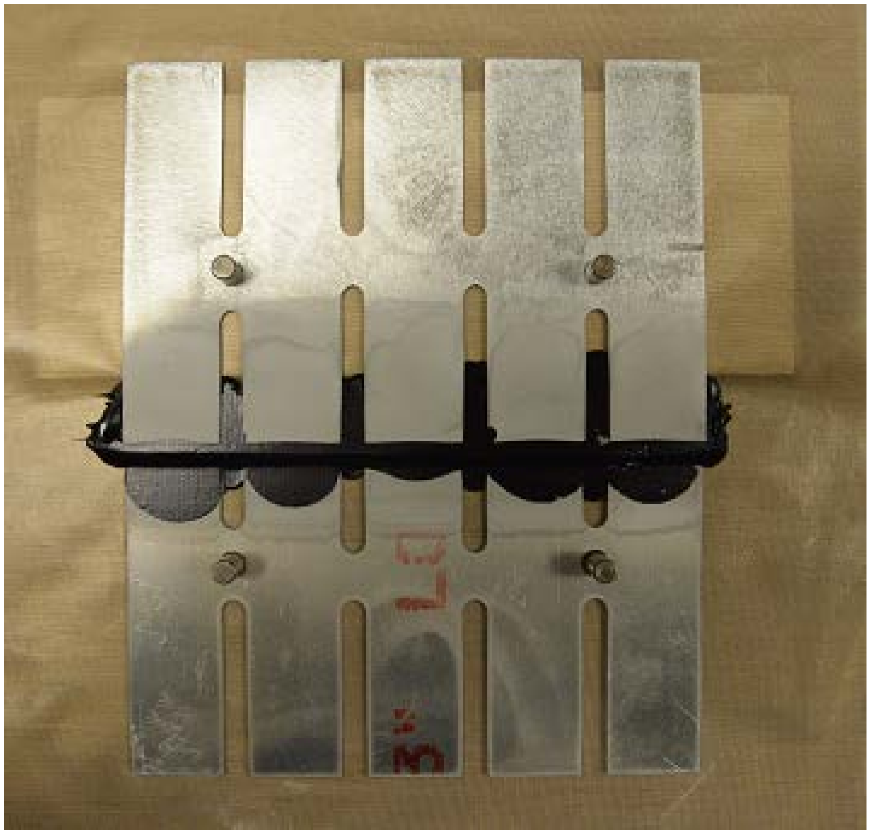


Fig. 21 Representative example of fully bonded single-lap-joint coupon panel upon completion of cure in the tooling fixture

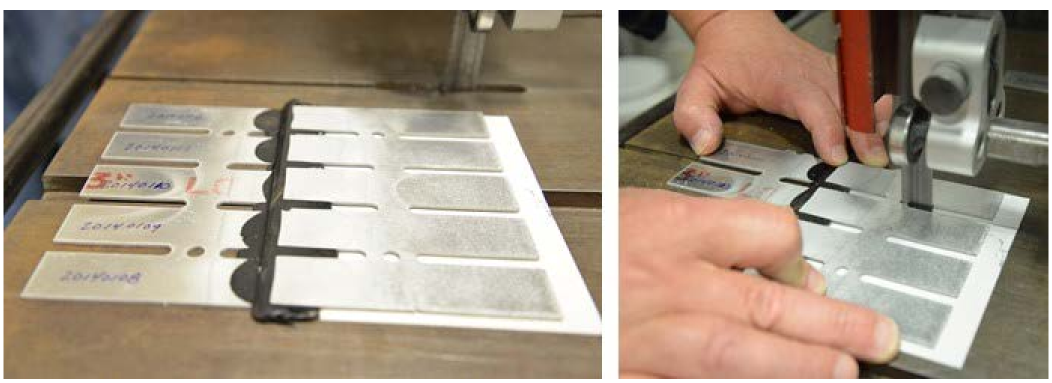


Fig. 22 "Intact" bonded single-lap-joint panel showing adhesive fillets, side overflow, and the cardboard shim (left) and each individual single-lap-joint coupon specimen being separated from the panel using a band saw (right)

Finally, each lap-joint coupon specimen is finished into a test specimen in accordance with ASTM D1002[17] as shown in Fig. 23. Any sharp edges remaining on the aluminum adherends after cutting with the band saw are removed using a belt sander, which produces smooth edges and reduces any cutting injury risk to laboratory personnel during further handling and testing. Remove all excess adhesive from the sides of the joint. This facilitates an easier visual inspection for detecting possible excessive voids within the bondline and ensures an accurate bondline thickness measurement.

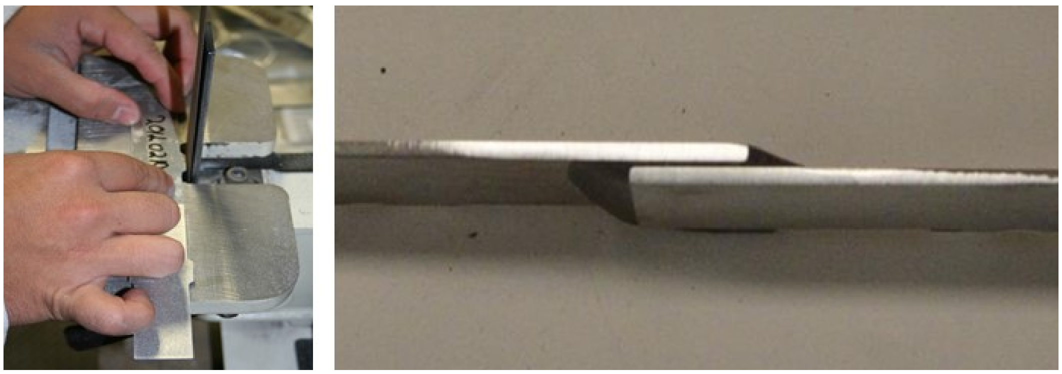


Fig. 23 Finishing preparation of a single-lap-joint coupon test specimen using a belt sander (left) and a side view of the test specimen after removing the excess adhesive (right)

8.6 Bondline Thickness Measurements

Once individual single-lap-joint test specimens have been fabricated, the adhesive bondline thicknesses need to be recorded for each specimen. It is important when taking thickness measurements of the single-lap-joint test specimen that a calibrated micrometer, such as the one shown in Fig. 24, is used.

Fig. 24 Perform the single-lap-joint bondline thickness measurements using a calibrated micrometer

Three measurements shall be taken along the width of the lap-joint test specimen at specified locations, and then averaged, as indicated in Fig. 25. Measurements of the overall thickness include the thickness of adherend A and B over their prepared substrate surfaces of the lap joint itself at location C. Bondline thickness can be simply calculated by subtracting the adherend A and B measurements from the overall thickness measurement. Averages shall be determined and recorded. Furthermore, the operator will document in a laboratory notebook with sufficient detail to serve as trustworthy database metadata the method of obtaining the average thickness of the adhesive layer. The following Table is an example of an appropriate table that one can use to record, calculate, and submit as metadata.

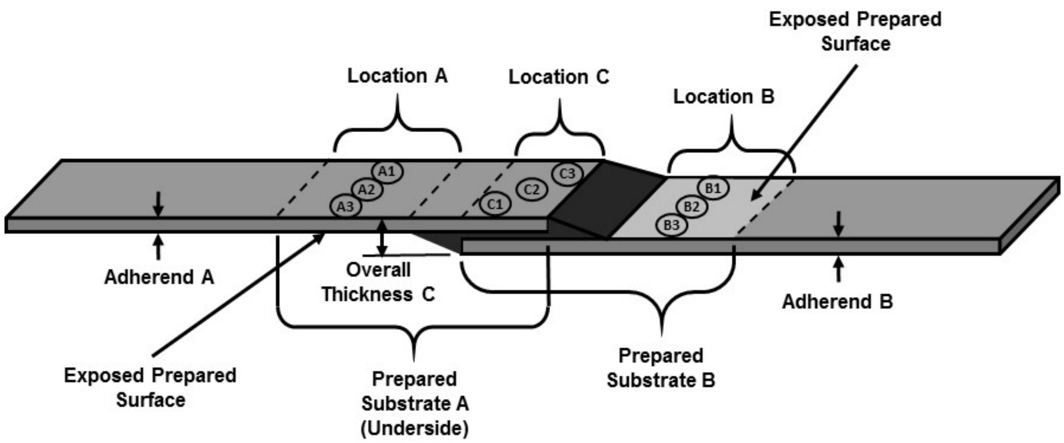


Fig. 25 Illustration of the single-lap-joint bondline thickness measurement points

Table. Representative single-lap-joint bondline thickness measurements and calculations

Sample ID:					
Section	Bondline thickness units (circle) – mm or in				Comments
	Adherend A (A)	Adherend B (B)	Adherend A & B Plus Bondline (BL) (C)	Bondline Calculation (BL=C-(A+B))	
Location 1	(A1 Value) 0.063	(B1 Value) 0.0627	(C1 Value) 0.1545	(BL1 Value) 0.0288	
Location 2	(A2 Value) 0.0627	(B2 Value) 0.0625	(C2 Value) 0.1552	(BL2 Value) 0.03	
Location 3	(A3 Value) 0.0625	(B3 Value) 0.0624	(C3 Value) 0.1555	(BL3 Value) 0.0306	
Average	(Avg. of A) 0.0627	(Avg. of B) 0.0625	(Avg. of C) 0.1551	**(Avg. of BL)** 0.0298	

8.7 Environmental Conditioning of Specimens

It is essential that test specimens be environmentally conditioned and then mechanically tested to assess the adhesive's performance and that ARL determine the acceptance of the adhesive for higher-tier testing outlined in ARL-ADHES-QA-001.00. It is necessary to condition specimens in accordance to this standard.

Test specimens shall be designated into the following 3 sets:

- Room Temperature–Dry Conditioning
- Hot/Wet Conditioning
- Elevated Temperature–Dry Conditioning

Prior to mechanical testing, samples shall be kept under dry conditions at ambient temperatures until tested and then stored in sealed plastic bags, dry desiccant containers, nitrogen purged containers, or similar. Even samples to be tested following hot/wet conditioning (i.e., high temperature/water immersion) will still be stored in dry conditions prior to initiating the immersion cycle. The operator will document the sample conditioning and all relevant test information in a laboratory notebook with sufficient detail to serve as trustworthy database metadata.

A sufficient number of lap-joint test specimens (a minimum of 5) within a set shall be mechanically tested, as per Subsection 8.8, to determine the average tensile breaking strength of that set, under each condition. Specimens producing extremely low values from the test set average may be omitted and an additional sample tested. Anomalies could be due to flaws within the bondline, inadequate surface preparation, or possibly an issue with the testing apparatus.

8.7.1 Room Temperature–Dry Conditioning

Room temperature–dry conditioning samples will be considered baseline values for the adhesive. Samples shall be taken from the sealed dry container and tested within 30 min.

8.7.2 Elevated Temperature/Water Immersion (Hot/Wet) Conditioning

Samples conditioned at elevated temperature and 100% submersed in water (hot/wet conditioning) shall be placed within a water immersion tank (circulating or noncirculating), preferably using DI or distilled water, maintaining a constant water temperature of 63 °C, +/–3 °C (145 °F, +/–5 °F), for a duration of 14 days.[24] Duration and temperature of the conditioning are based upon considerations referenced in MIL-STD-810G, Laboratory Test Method 507.5, Humidity.[25]

As an alternate method, if an immersion tank is not available, individual lap-joint test specimens may be placed within a large test tube, with a screw top, to fully accept the length of the specimen, filled with water and cap securely tightened. Test tubes may then be placed in a rack, sitting upward and placed into a static or convection oven set at 63 °C, +/–3 °C (145 °F, +/–5 °F), as shown in Fig. 26. In both test methods it is highly recommended that a calibrated digital thermometer will be continually used to monitor the water temperature of the immersion tank or the air temperature within the oven as a verification.

After the 14-day duration, samples shall be removed from the bath, patted dry, and allowed to cool at room temperature to be handled. Samples *must* be mechanically tested no later than 30 min after removal from the water immersion tank.

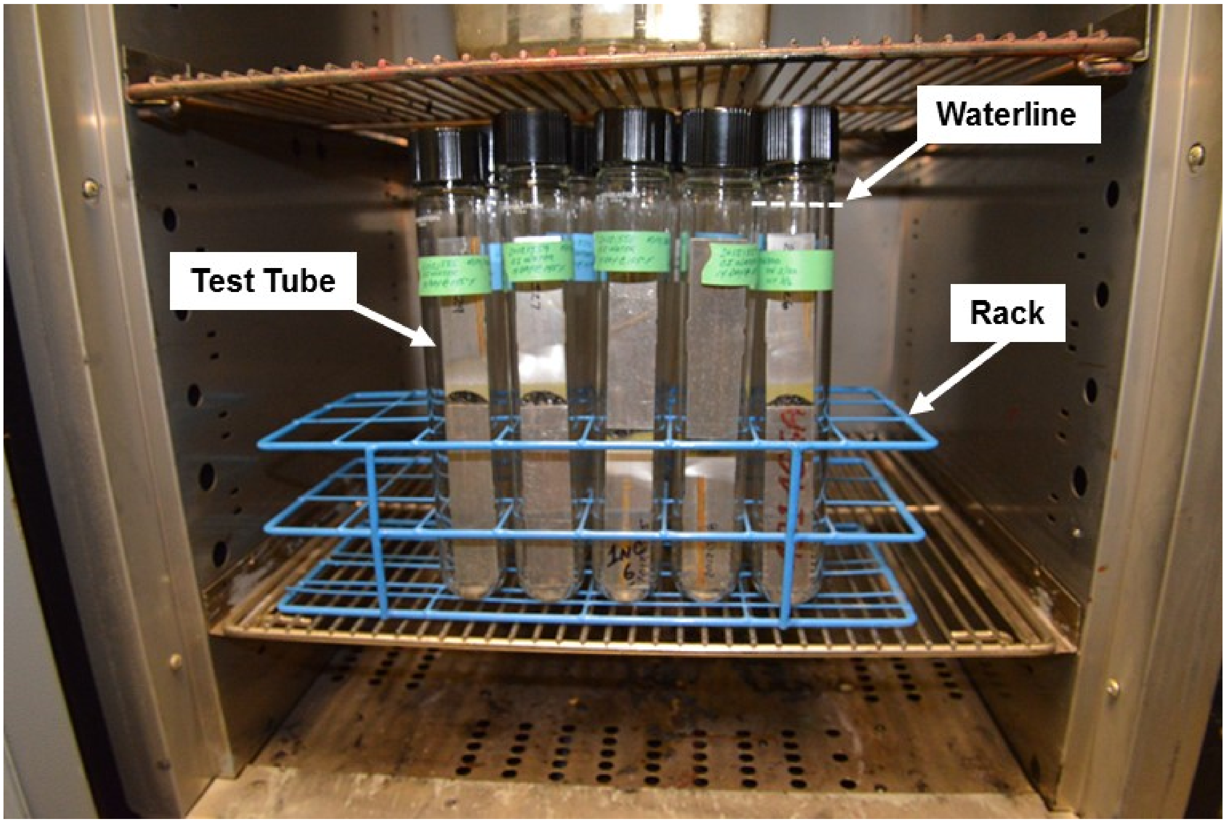


Fig. 26 Alternate elevated temperature/water immersion conditioning test for lap-joint test specimens using the test tubes and convection oven method

8.7.3 Elevated Temperature–Dry Conditioning

The temperature of the test is based upon considerations referenced in MIL-STD-810G, Laboratory Test Method 501.5, High Temperature.[26] A representative example of high-temperature testing is shown in Fig. 27. Dry-elevated temperature conditioning consists of a specialized heated test chamber that surrounds the test specimen and the grips of mechanical test apparatuses yet still allows mechanical testing to be performed. The left-hand image of Fig. 27 shows the specialized test chamber with its door open and a single lap joint installed, ready to be conditioned for mechanical testing. The right-hand image shows the door in the closed position, allowing the test chamber air to equilibrate to the proper elevated temperature for testing.

Prior to placing a test specimen in the chamber, the test chamber air shall be set to an elevated and stabilized temperature of 71 °C, +/–3 °C (160 °F, +/–5 °F), and held for at least 45 min as measured by an adjacent thermocouple (TC) within the chamber. A single-lap-joint test specimen shall then be placed inside and the chamber door closed. The test chamber shall then be allowed to reach a stable 71 °C, +/–3 °C (160 °F, +/–5 °F), for dwell time of no less than 10 min prior to testing. This will allow the test chamber and test specimen to reach steady-state equilibrium.

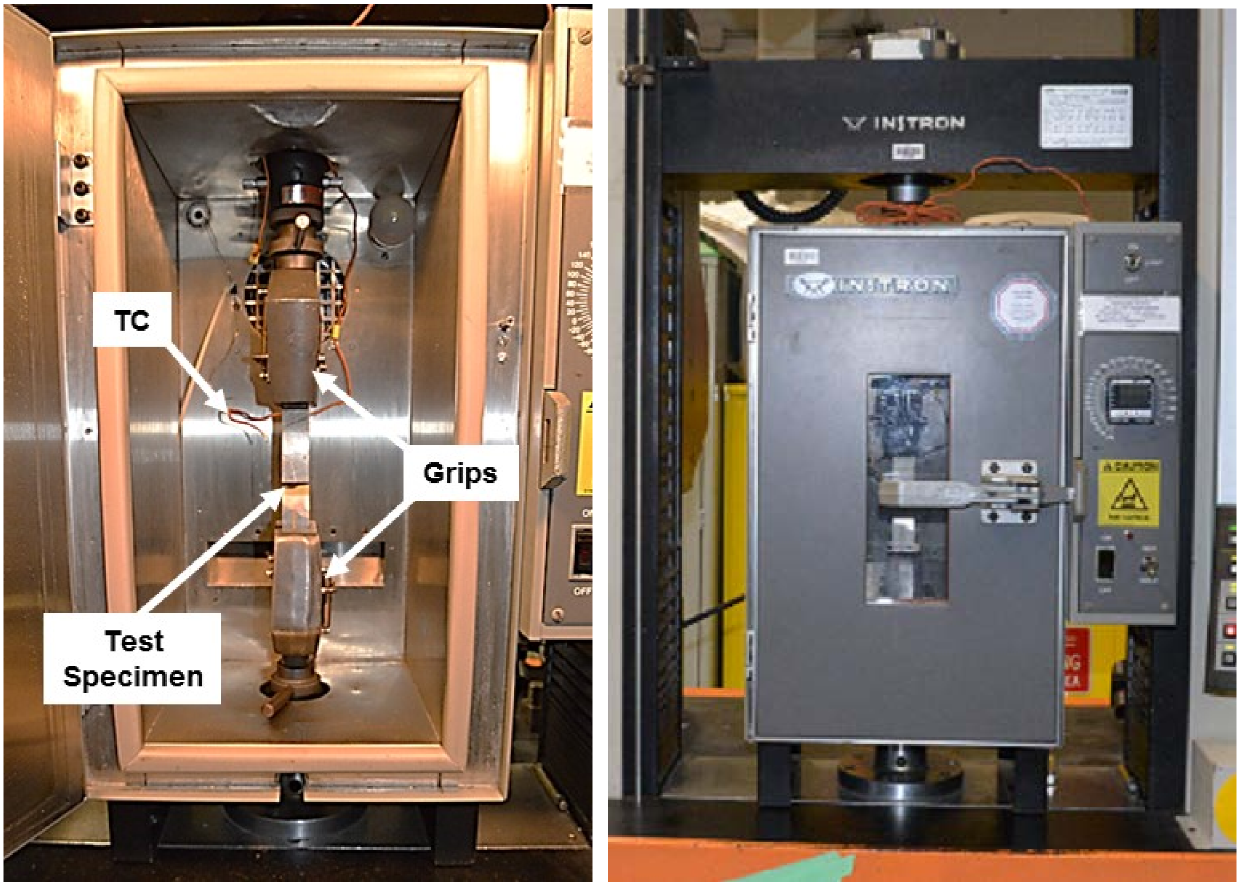


Fig. 27 Samples undergoing elevated temperature testing at 71 °C, +/–3 °C (160 °F, +/–5 °F), with the chamber door opened (left) and closed (right)

8.8 Mechanical Testing

The test procedure for the tensile breaking strength of the single lap joint is performed according to Paragraphs 9.1 and 9.2 of ASTM D1002-10.[17] An instrumented mechanical testing frame shall be used, with an adequate load cell selected to ensure that the breaking load of the single lap joint shall fall between 15% and 85% of its full-scale capacity. A typical mechanical test frame and load cell is shown in Fig. 28. The crosshead speed shall be 0.0212 mm/s (1.27 mm/min, 0.05 inch/min) and will be equipped with a suitable pair of self-aligning grips to hold both ends of the single lap joint. The jaws of the grips shall engage the outer 25.4 mm (1 inch) of each end of the single lap joint. The mechanical testing frame shall conform to Paragraph 5.1 of ASTM D1002-10. An example of an acceptable test setup is shown in Fig. 29.

The operator will document the test information, test conditions, and test frame in a laboratory notebook with sufficient detail to serve as trustworthy database metadata.

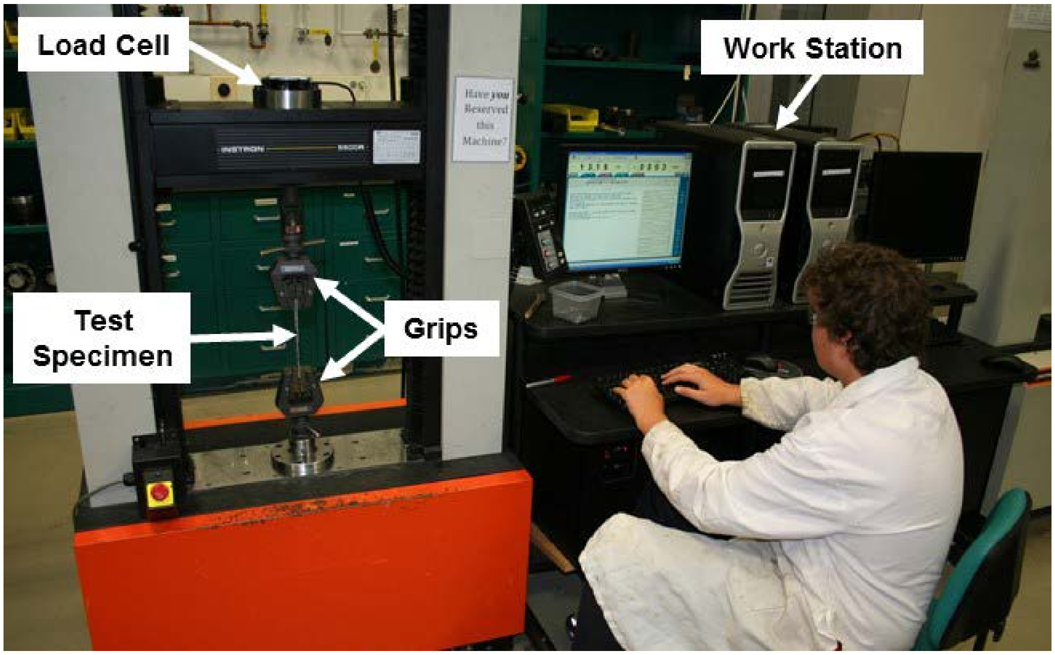


Fig. 28 An instrumented mechanical testing frame with a load cell and work station is used to measure the load vs. displacement response of the single lap joint until breaking failure

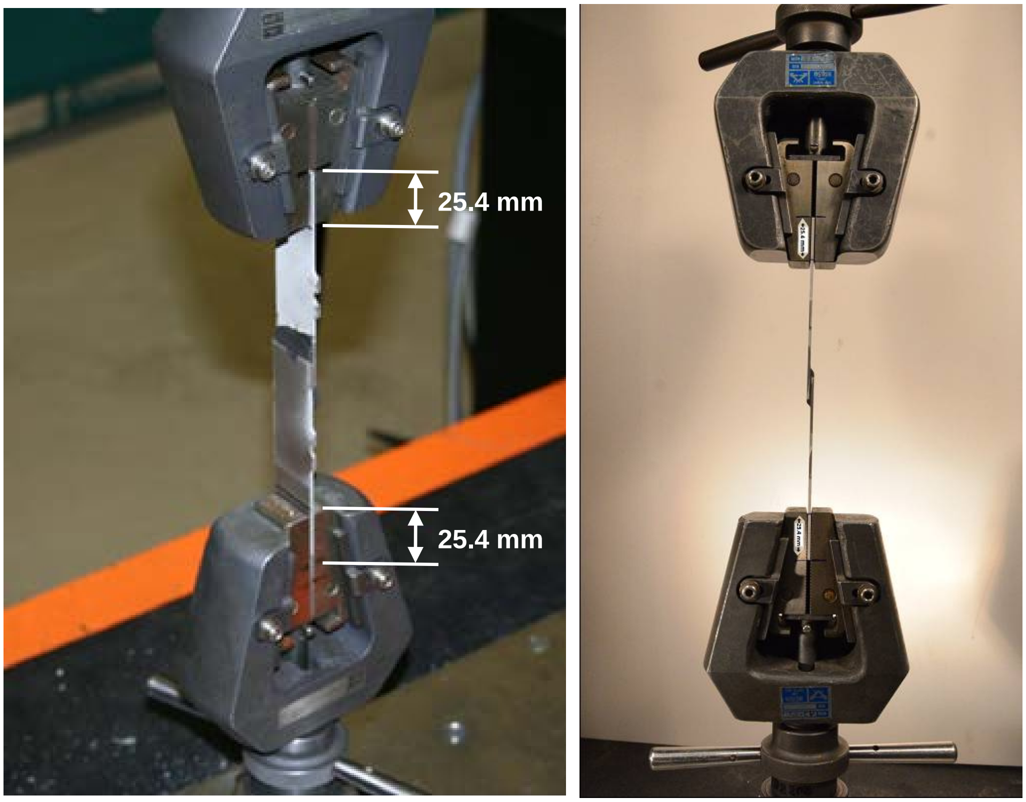


Fig. 29 Images showing the jaws of the grips engaging a single-lap-joint specimen at each end, secured in the center of the grips and parallel to the direction of tensile force to be applied as per ASTM D1002-10, Paragraph 5.1

8.9 Data Collection

All the raw experimental mechanical test data for each lap-joint specimen shall be saved in a comma-separated values (CSV) file format. Both the load versus displacement and load versus time data are required for subsequent analysis and are graphically shown in Figs. 30 and 31, respectively. Any unit of measure is acceptable provided it is indicated in the data files.

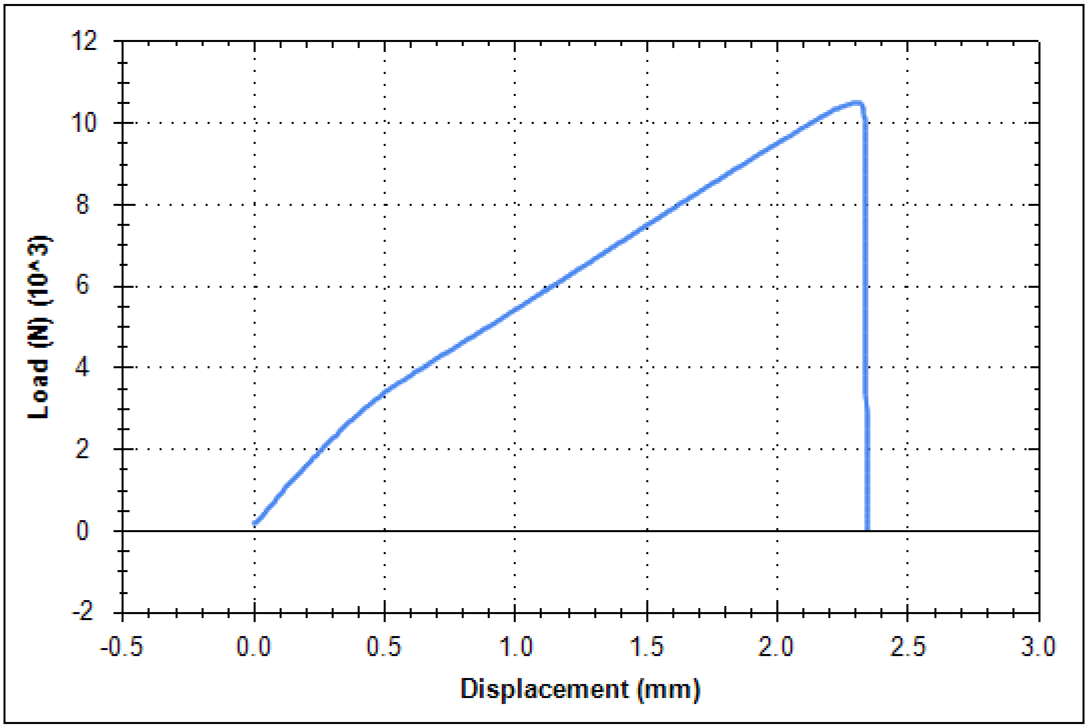


Fig. 30 Representative example of raw load vs. displacement plot

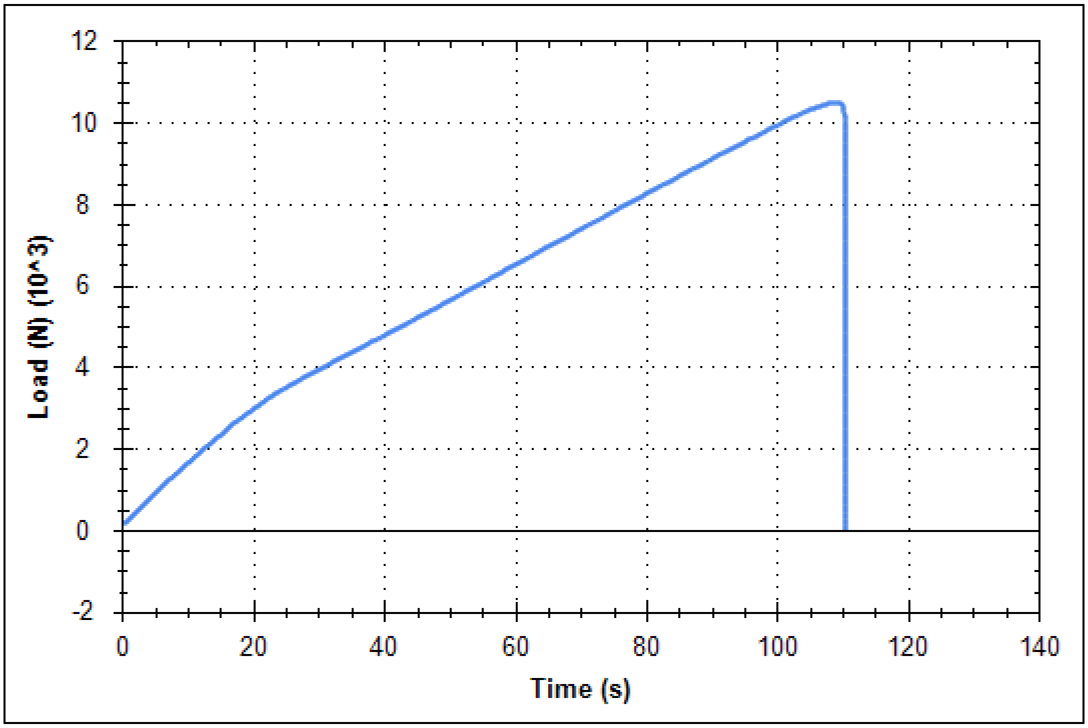


Fig. 31 Representative example of raw load vs. time plot

The failed test coupons will be manually labeled with appropriate sample identification (ID) information to embed this information within the image, which preserves data integrity by eliminating file-naming errors. Images will be 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.[27] Test coupons will minimally be visually assigned either an adhesive, cohesive, or mixed-mode of failure. More complex mode-of-failure descriptions can be found in ASTM D1002-10 and ASTM D5573-99.[28] A representative scanned failure surfaces showing sample ID information embedded within the image is shown in Fig. 32.

Fig. 32 Representative scanned failure surfaces showing sample ID information embedded within the image (ruler is included for quantitative dimensional scaling)

8.9.1 Data Assembly and Retention

The following information will be collected and retained:

- Adhesive TDS and safety data sheet
- Individual load versus displacement results exported in CSV format
- Electronically scanned Single-Lap-Joint Fabrication Checklist (Appendix C)
- Electronically scanned Single-Lap-Joint Mechanical Testing Checklist (Appendix D)
- Electronically scanned Bondline Thickness Measurements sheets (Appendix E)
- Scanned TIFF formatted failure surfaces

9.0 References and Notes

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11. APG Regulation 385-4, Chapter 12, Paragraph 12–4 Eye Protection. Aberdeen Proving Ground (MD): Department of the Army (US); 1991 Aug 2.

12. ANSI/ISEA Z87.1-2010. American national standard for occupational and educational personal eye and face protection devices.

13. US Army environmental hygiene agency occupational vision guide.

14. M&MSD SOP no. 6.4, Safety, emergencies and housekeeping.

15. APGR 200-60. Environmental quality – hazardous waste management.

16. Army Research Laboratory Internal Operating Procedures: L1032, L1035, & L1040-001 an IOP for scale & balance operations; L1035, L1036, & L1040-003 an IOP for fume hood operations; L1028-013 grit blast cabinet; L1044-008 safe operations for blue m ovens; L1086-006 blue m humidity cabinet; L1035, L1036, 1040-003 fume hood operation; L1032, L1035, L1038, & L1040-001 scale & balance operation; L1040/44-003 digital hot plate with stirrer; L1044-008 blue m ovens operation.

17. ASTM D1002-10. Standard test method for apparent shear strength of single-lap-joint adhesively bonded metal specimens by tension loading (metal-to-metal). West Conshohocken (PA): ASTM International; 2010. DOI: 10.1520/D1002-10.

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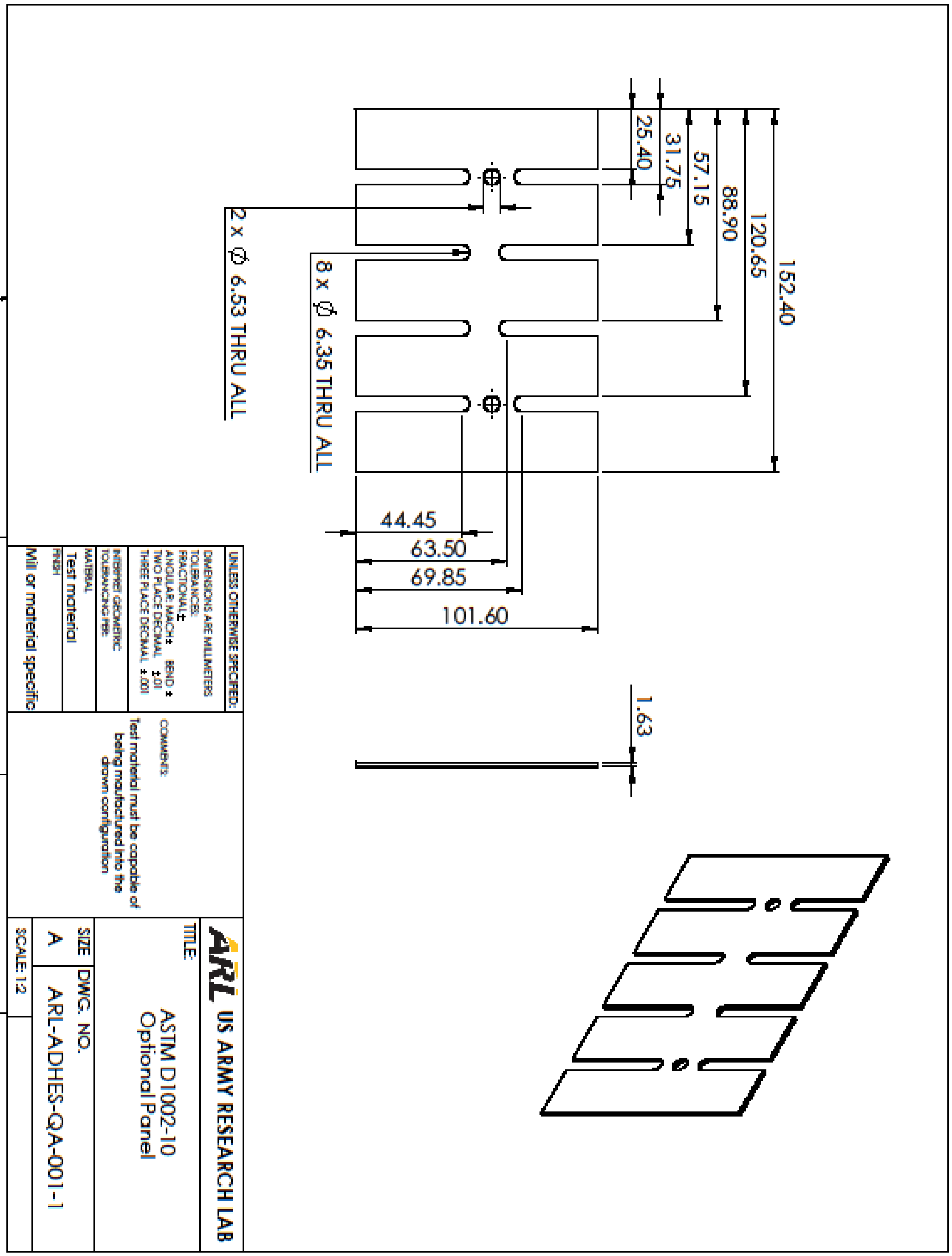
10.0 Appendix A. Test Panel Dimensions

For viewing ease and use with computer-aided design software, supplemental test panel and tooling fixture drawings (as PDFs and DXFs) are attached to this PDF.



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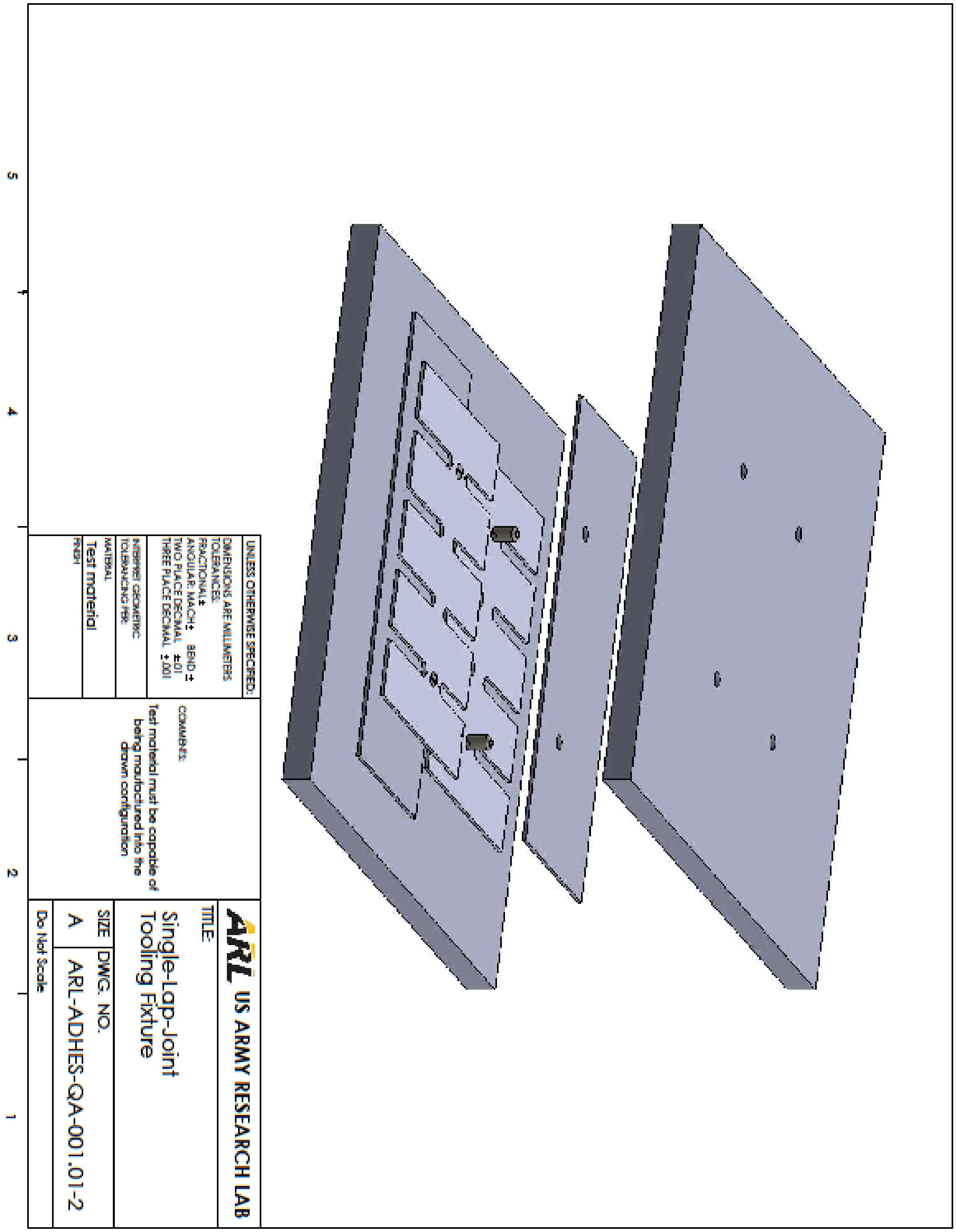
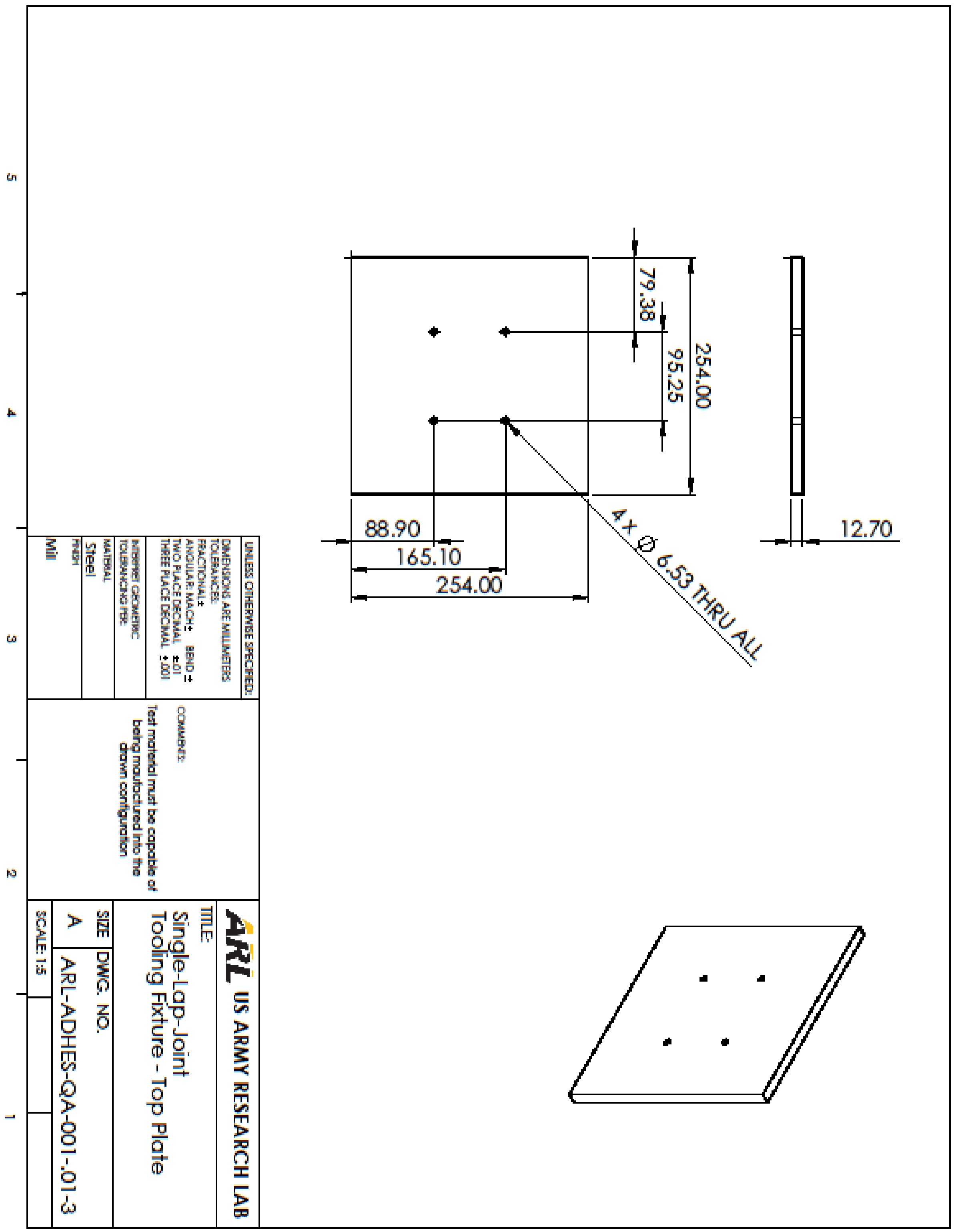
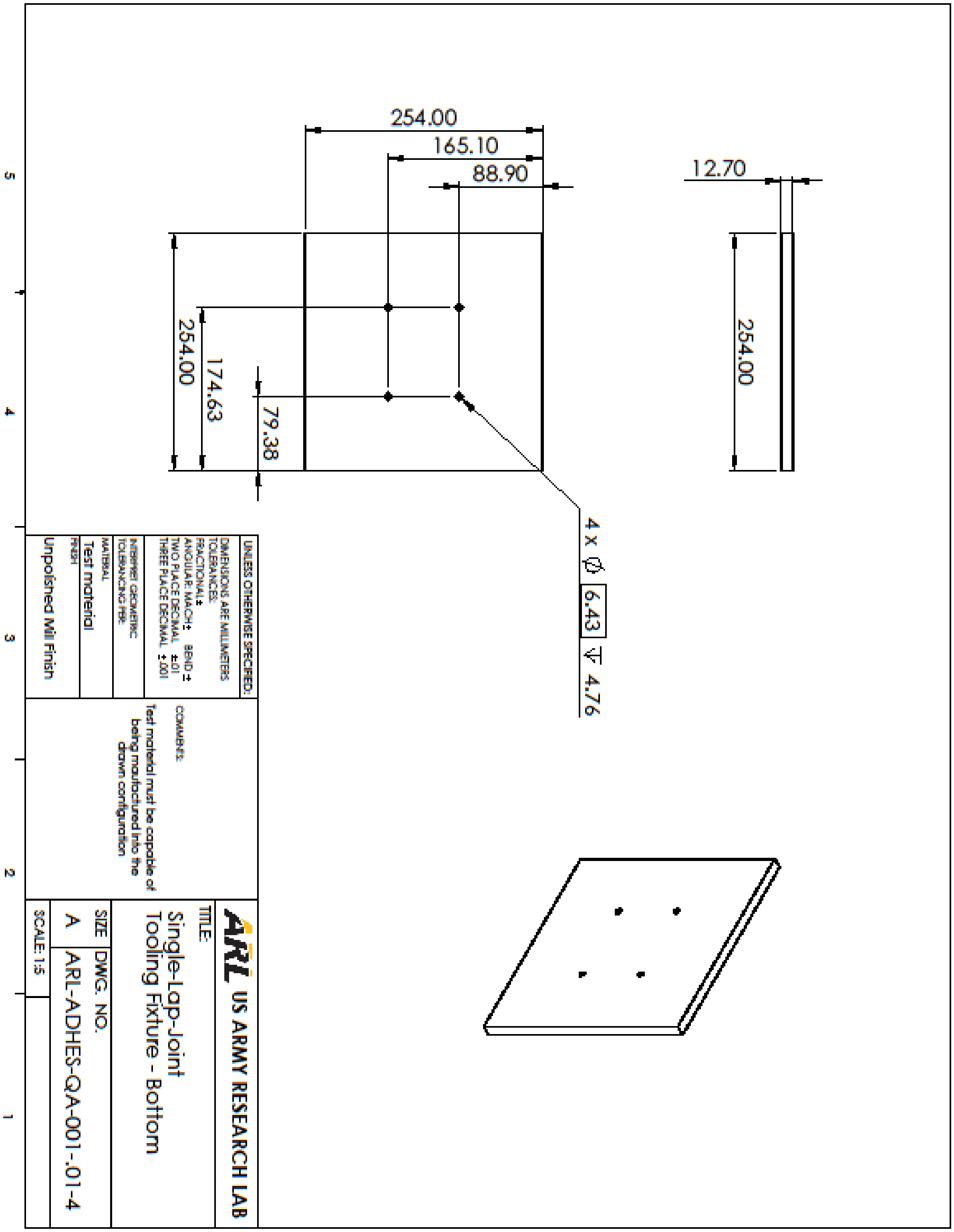
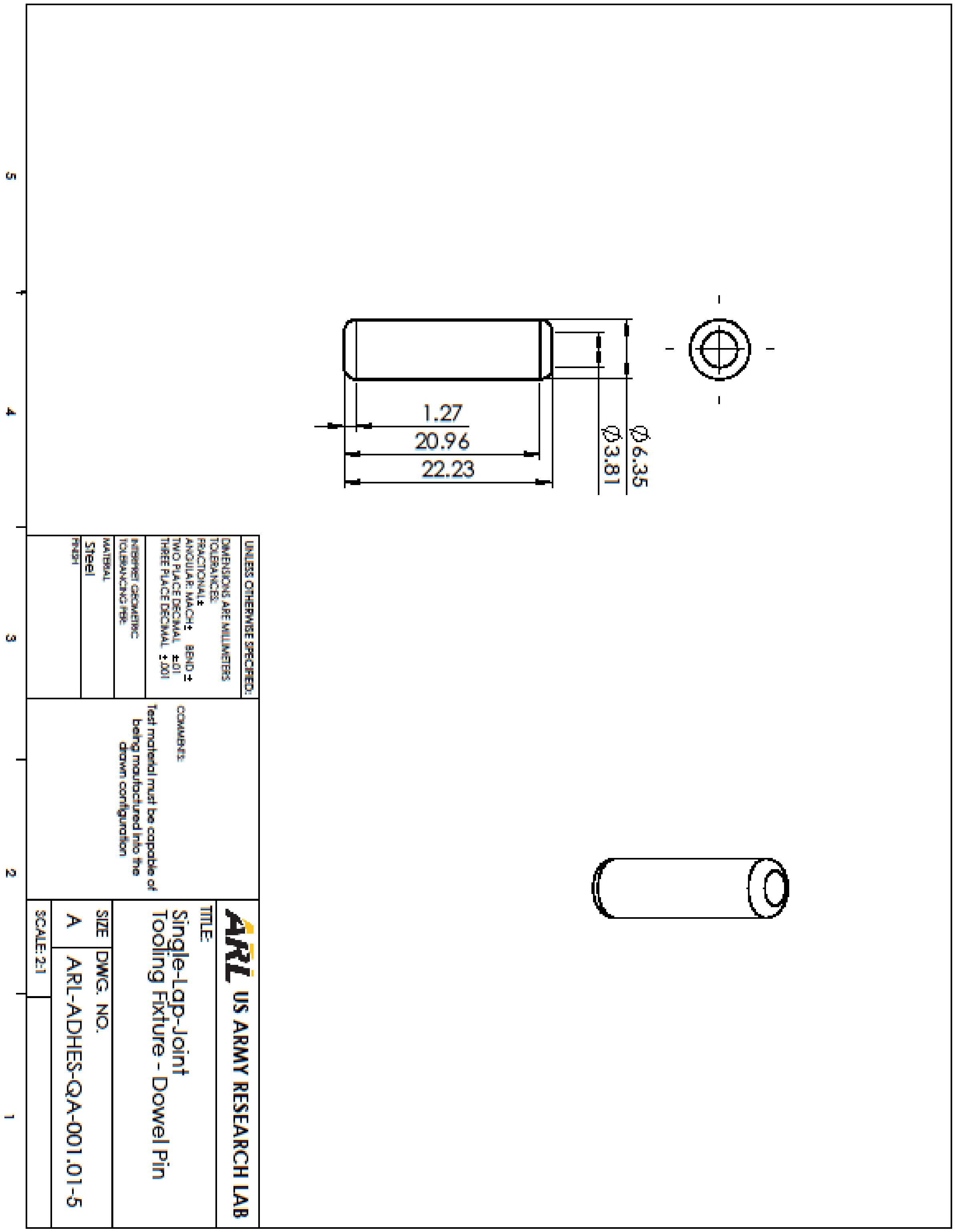
11.0 Appendix B. Tooling Fixture Dimensions




79.38
95.25
254.00
88.90
165.10
254.00
4x Ø 6.53 THRU ALL
12.70
UNLESS OTHERWISE SPECIFIED:
DIMENSIONS ARE MILLIMETERS
TOLERANCES:
FRACTIONAL ±
ANGULAR: MACH ± BEND ±
TWO PLACE DECIMAL ±.01
THREE PLACE DECIMAL ±.001
INTERPRET GEOMETRIC TOLERANCING PER:
MATERIAL
Steel
FINISH
Mill
COMMENTS:
Test material must be capable of being manufactured into the drawn configuration
ARL US ARMY RESEARCH LAB
TITLE:
Single-Lap-Joint Tooling Fixture - Top Plate
SIZE
A
DWG. NO.
ARL-ADHES-QA-001-.01-3
SCALE: 1:5



Approved for public release; distribution is unlimited.


1.27
20.96
22.23
Ø6.35
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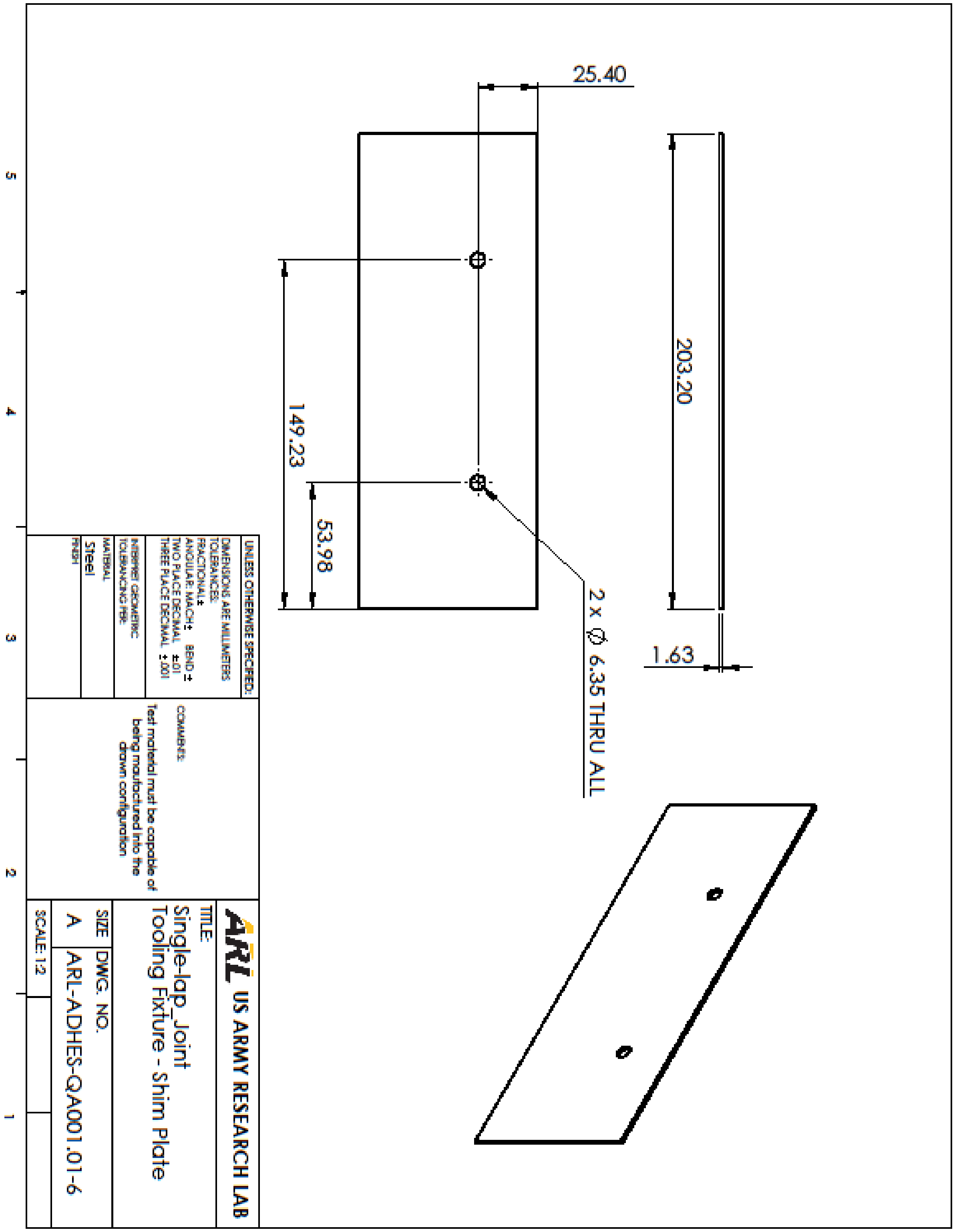
UNLESS OTHERWISE SPECIFIED:
DIMENSIONS ARE MILLIMETERS
TOLERANCES:
FRACTIONAL±
ANGULAR: MACH± BEND ±
TWO PLACE DECIMAL ±.01
THREE PLACE DECIMAL ±.001
INTERPRET GEOMETRIC TOLERANCING PER:
MATERIAL
Steel
FINISH
COMMENTS:
Test material must be capable of being maufactured into the drawn configuration
ARL US ARMY RESEARCH LAB
TITLE:
Single-Lap-Joint Tooling Fixture - Dowel Pin
SIZE
A
DWG. NO.
ARL-ADHES-QA-001.01-5
SCALE: 2:1


25.40
203.20
149.23
53.98
2 x ∅ 6.35 THRU ALL
1.63
UNLESS OTHERWISE SPECIFIED:
DIMENSIONS ARE MILLIMETERS
TOLERANCES:
FRACTIONAL ±
ANGULAR: MACH ± BEND ±
TWO PLACE DECIMAL ±.01
THREE PLACE DECIMAL ±.001
INTERPRET GEOMETRIC TOLERANCING PER:
MATERIAL
Steel
FINISH
COMMENTS:
Test material must be capable of being manufactured into the drawn configuration
ARL US ARMY RESEARCH LAB
TITLE:
Single-lap_Joint
Tooling Fixture - Shim Plate
SIZE
A
DWG. NO.
ARL-ADHES-QA001.01-6
SCALE: 1:2
1
2
3
4
5

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12.0 Appendix C. Single-Lap-Joint Fabrication Checklist

Sample ID's:____________, ____________,____________,____________,____________

Date:________________ Temperature:________________ Humidity______________

Testing facility and operator:____

Adhesive, type, and form:____

____

Lot ID, manufacturer's code, expiration date:___________________________________

____

Method of cleaning and preparing bonding surface:_____________________________

____

____

____

____

Adhesive preparation, application, and bonding conditions:

____

____

____

____

____

Tooling fixture and bonding pressure notes: ___________________________________

____

____

Adhesive mixing to applied pressure time:______________________________________

Cure cycle:____

____

____

Oven used (model and serial number):____

Conditioning procedure:____

____

____

Comments:____

____

____

____

____

____

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13.0 Appendix D. Single-Lap-Joint Mechanical Testing Checklist

Sample ID's:_____________, _____________,_____________,_____________,____________

Test date:_______________ Test temperature:______________ Test humidity____________

Testing facility and operator:____

Test frame model and serial number:____

Test frame calibration date:____

Load cell model and serial number:____

Load cell calibration date:____

Crosshead speed: 1.3 mm/min (0.05 in/min), list other________________________________

Nature of failure and other comments:

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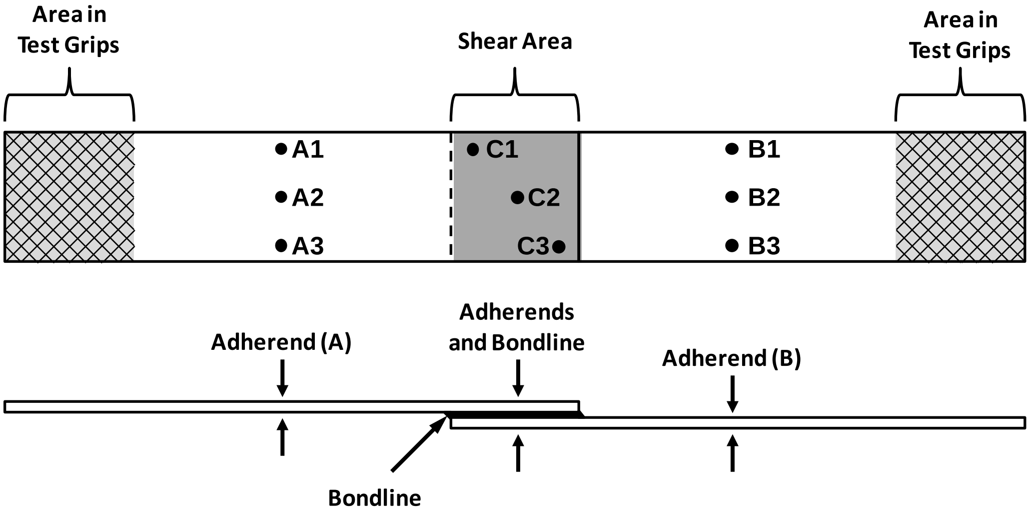
14.0 Appendix E. Bondline Thickness Measurements



Aluminum grade:___________________ Mfg. nominal thickness:______________(mm – in)

Micrometer model, serial number, calibration date __________________________________

____

Sample ID:					
Section	Bondline thickness units (circle) – mm or in				Comments
	Adherend A (A)	Adherend B (B)	Adherend A & B Plus Bondline (BL) (C)	Bondline Calculation (BL=C-(A+B))	
Location 1	(A1 Value)	(B1 Value)	(C1 Value)	(BL1 Value)	
Location 2	(A2 Value)	(B2 Value)	(C2 Value)	(BL2 Value)	
Location 3	(A3 Value)	(B3 Value)	(C3 Value)	(BL3 Value)	
Average	(Avg. of A)	(Avg. of B)	(Avg. of C)	(Avg. of BL)	

Sample ID:					
Section	Bondline thickness units (circle) – mm or in				Comments
	Adherend A (A)	Adherend B (B)	Adherend A & B Plus Bondline (BL) (C)	Bondline Calculation (BL=C-(A+B))	
Location 1	(A1 Value)	(B1 Value)	(C1 Value)	(BL1 Value)	
Location 2	(A2 Value)	(B2 Value)	(C2 Value)	(BL2 Value)	
Location 3	(A3 Value)	(B3 Value)	(C3 Value)	(BL3 Value)	
Average	(Avg. of A)	(Avg. of B)	(Avg. of C)	(Avg. of BL)	

Sample ID:					
Section	Bondline thickness units (circle) – mm or in				Comments
	Adherend A (A)	Adherend B (B)	Adherend A & B Plus Bondline (BL) (C)	Bondline Calculation (BL=C-(A+B))	
Location 1	(A1 Value)	(B1 Value)	(C1 Value)	(BL1 Value)	
Location 2	(A2 Value)	(B2 Value)	(C2 Value)	(BL2 Value)	
Location 3	(A3 Value)	(B3 Value)	(C3 Value)	(BL3 Value)	
Average	(Avg. of A)	(Avg. of B)	(Avg. of C)	(Avg. of BL)	

Sample ID:					
Section	Bondline thickness units (circle) – mm or in				Comments
	Adherend A (A)	Adherend B (B)	Adherend A & B Plus Bondline (BL) (C)	Bondline Calculation (BL=C-(A+B))	
Location 1	(A1 Value)	(B1 Value)	(C1 Value)	(BL1 Value)	
Location 2	(A2 Value)	(B2 Value)	(C2 Value)	(BL2 Value)	
Location 3	(A3 Value)	(B3 Value)	(C3 Value)	(BL3 Value)	
Average	(Avg. of A)	(Avg. of B)	(Avg. of C)	(Avg. of BL)	

Sample ID:					
Section	Bondline thickness units (circle) – mm or in				Comments
	Adherend A (A)	Adherend B (B)	Adherend A & B Plus Bondline (BL) (C)	Bondline Calculation (BL=C-(A+B))	
Location 1	(A1 Value)	(B1 Value)	(C1 Value)	(BL1 Value)	
Location 2	(A2 Value)	(B2 Value)	(C2 Value)	(BL2 Value)	
Location 3	(A3 Value)	(B3 Value)	(C3 Value)	(BL3 Value)	
Average	(Avg. of A)	(Avg. of B)	(Avg. of C)	(Avg. of BL)	

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