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4 Acronyms

AMPP – The Association for Materials Protection and Performance	NAWCAD – Naval Air Warfare Center Aircraft Division
CFRP – Carbon Fiber Reinforced Polymer	NC – Non-chromate
CGR – Crack Growth Rate	PC – Personal Computer
DAU – Data Acquisition Unit	PI – Principal Investigator
DCB – Double Cantilever Beam Sample	PPG – PPG Industries, Inc.
EAC – Environment Assisted Cracking	Q-FOG CRH – Q-Lab Corporation Cyclic Corrosion Tester
E-coat – Electrophoretic Deposition Coating	RH – Relative Humidity
FEA – Finite Element Analysis	S – Short Transverse
hh:mm – Hours:Minutes	SCC – Stress Corrosion Cracking
Hntz – Hentzen Coatings	SLEAC
L – Longitudinal	T – Long Transverse
LLC – Limited Liability Company	™ – Trademark
L-T – Long Transverse	TM – Test Method
LTD – Limited	UI – User Interface
MDACT – Multivariable dynamic accelerated corrosion test	YS – Yield Strength
n.c. – No Cracking	

5 Summary

The Static Load Environmentally Assisted Cracking (SLEAC™) system and test method are being developed to characterize the capacity of coatings to protect structural alloys from environment assisted cracking (EAC) or stress corrosion cracking (SCC). Previous research by Luna Labs demonstrated that an instrumented load frame could be used to continuously measure cracking *in situ* during atmospheric corrosion tests. The purpose of this project was to advance the technology, refine the test method, and deliver a low rate initial production SLEAC system for evaluation at NAWCAD.

During this two year effort, fracture samples and load frames, without instrumentation, were delivered to NAWCAD for preliminary testing to verify the approach, establish requirements, and provide guidance. For this preliminary testing by NAWCAD, the samples and load frames were evaluated in the recently established multivariate dynamic accelerated corrosion test (MDACT). These NAWCAD results confirmed that the approach was satisfactory for distinguishing coating performance for protection of an EAC susceptible alloy. Also, promising performance was obtained for an aluminum-rich primer.

The NAWCAD and internal test results were used to define performance and design requirements for a fully instrumented, multichannel SLEAC test system. This system was fabricated and iteratively tested to confirm functionality to meet the overall project objective of delivering a multichannel system to NAWCAD for use in their coating evaluations. The internal testing continued to confirm the system utility and trends for the relative performance from best to worst of chromate, aluminum rich, and non-chromate primers were replicated. The test method was also refined and revisions to AMPP TM21449-2021 have been identified.

The project concluded with the delivery of the SLEAC system to NAWCAD. The SLEAC system includes samples, load frame with displacement transducer and load cell, data acquisition, user interface software, and analysis routines. The load and displacement measurements are used with a finite element model for compliance to estimate crack length with time and crack growth rate. Efforts will continue in the Option Period to refine the product and test method and revised the AMPP standard TM21449-2021.

6 Background

SCC is a relevant corrosion mechanism for aircraft structural integrity. Protective primers with inhibitors may promote structural alloy resistance to SCC. The substitution of compliant coatings for hexavalent chrome coatings may present a risk to aircraft structural integrity. Historically, testing of SCC resistance in corrosive atmospheres has not included protective coating systems.

Atmospheric corrosion can produce uniquely aggressive conditions for promoting SCC. Also, galvanic corrosion and crevice corrosion are prevalent on aircraft structures and these conditions

will also affect SCC susceptibility. Coating evaluations for SCC protection should allow for the use of galvanic couples and crevices.

7 Objectives

The overall objective is to establish a measurement system and method that can be used to assess the capacity of an aerospace coating to protect structural alloys from SCC. For this two year project this objective has been achieved by focusing on the goals of:

- Produce a SLEAC system for atmospheric SCC testing
- Demonstrate SLEAC capabilities as a coating performance test

8 Approach

Stress corrosion cracking of a susceptible alloy is due to the combined effects of a corrosive environment and mechanical load. The presence of an occluded cell, or crevice, and galvanic couples may further promote SCC of aerospace aluminum alloys. For naval aviation, aggressive conditions are common in marine environments where high-strength aluminum alloys may be coupled to more noble materials such as stainless steel, titanium, or carbon fiber reinforced composites (CFRP). To mitigate corrosion of structural aluminum alloys, protective finishes and coatings are used. These may include anodization, chemical conversion coatings, pretreatments, primers, and topcoats. The ultimate susceptibility of a structural component is dependent on the service environment, alloy properties, protective coatings, galvanic couples, crevices, part geometry, and loading. The intent of this project was to address the protective properties of coatings using a fracture mechanics sample design and test system that allows for SCC measurement in corrosive atmospheres. The SLEAC requirements and prototype design built upon previous iterations of the test system (Air Force FA8650-19-C-5078 and ONR N00014-20-C-1092). The AMPP standard TM21449-2021, *Continuous Measurements for Determination of Aerospace Coating Protective Properties*, was the basis for the SCC testing and the project results will be used to refine that standard.

8.1 Milestones

- Delivery of load frame and samples to NAWCAD (June 2022)
- Completion of multichannel SLEAC tests system (February 2023)
- Verification of prototype operation (June 2023)
- Completion of coating testing with multichannel SLEAC test system (October 2023)
- Delivery of multichannel SLEAC test system to NAWCAD (8 November 2023)

9 Task Work Summary

The project task work was structured to perform test system design and fabrication in parallel with iterative testing at Luna Labs and NAWCAD to mature the technology and test methodology.

9.1 Materials, Environment, and SLEAC Test System Requirements

The TM21449-2021 standard is the first accepted method for testing aerospace alloys for SCC resistance with coatings, galvanic couples, and crevices in atmospheric corrosion tests. This was accomplished by considering the test objectives and use requirements to inform the sample, load frame, and test system instrumentation design.

Sample

- Coating application
- Instrumentation for crack initiation and growth
- Load mechanism and crack opening mode
- Fracture mechanics analysis
- Crack plane orientation relative to alloy texture
- Fabrication
- Gauge area and stress concentration
- Crevice former
- Galvanic couple

Load frame

- Static loading mechanism
- Instrumentation for crack initiation and growth
- Harsh environment testing

Data acquisition hardware, software, and analysis

- Multichannel
- Harsh environment testing
- Unattended operation
- User interface and test data visualization
- Processing for crack length

9.1.1 SLEAC Test System

The SLEAC test system consists of three primary elements: test station, SLEAC data acquisition unit (DAU), and load frames (Figure 1). The test station includes user software to setup tests and visualize sensor data during a test.

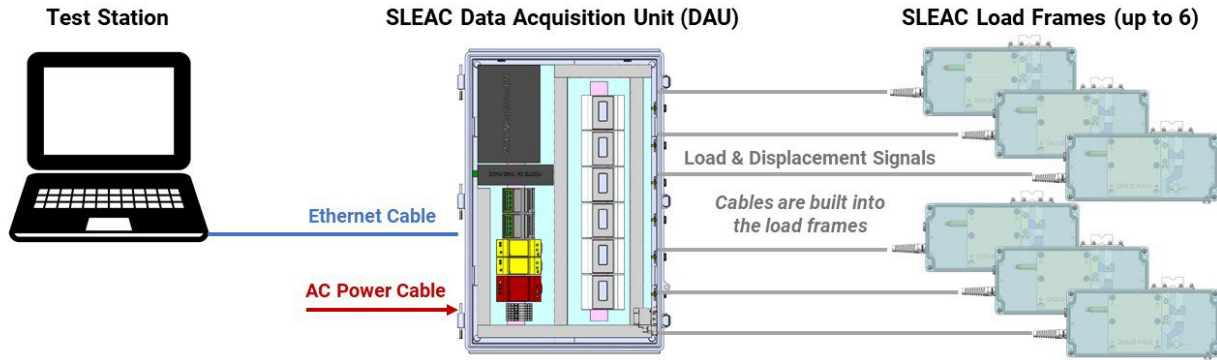


Figure 1. Primary of elements of the SLEAC test system. Multichannel DAU with six load frames.

9.2 Sample

The sample design and loading mode were determined to achieve a number of functionalities. One of the more significant requirements was to allow for coating application. Specifically, line-of-site access is required for spray application of aerospace coatings. To accomplish this, a double cantilever beam sample geometry was selected with a gauge area that is a semicircular (radius of 0.25 inch) edge notch at the end of the sample (Appendix A). The sample is loaded by compressing the beams together resulting in a tensile stress at the base of the semicircular notch gauge area (Figure 2). This loading produces tensile stress normal to the plane of the crack (Mode I crack opening). The overall sample dimensions are 2" x 4.5" x 0.25 inches and allow for a range of crack orientations relative to the aluminum alloy plate texture (Figure 3).

This sample geometry and loading mode improve ease of coating application (Figure 4). Any cleaning or finishing process may be possible on the sample gauge area, including deoxidation, anodization, chemical conversion coatings, and pretreatments. There is no restriction on the coating type, and inorganic or organic coatings such as electroplating, E-coat, powder, or liquid spray may all be possible.

The sample design includes attachment points for the load frame, load actuator, and displacement sensor (Appendix B). The semicircular gauge area can accept a rod to form a crevice and the rod may be any material including fastener alloys for galvanic coupling to the DCB sample.

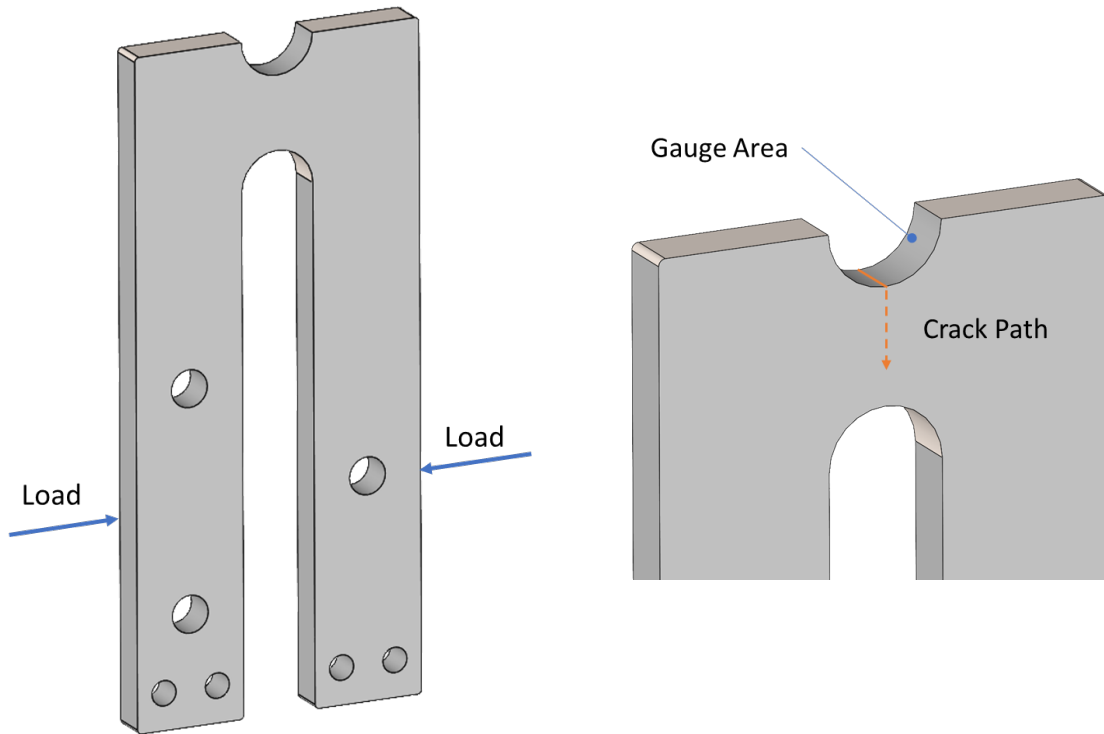


Figure 2. DCB sample with compressive loading (left) to promote Mode I cracking that initiates from the center of the gauge area (right).

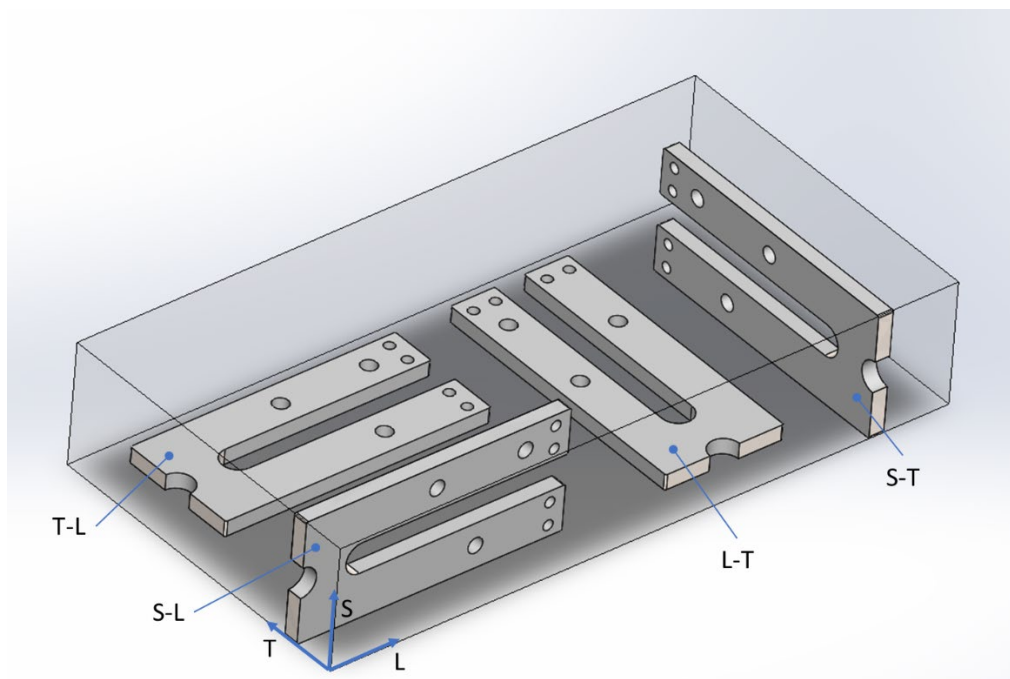


Figure 3. Sample and preferred crack orientations for a wrought alloy plate.

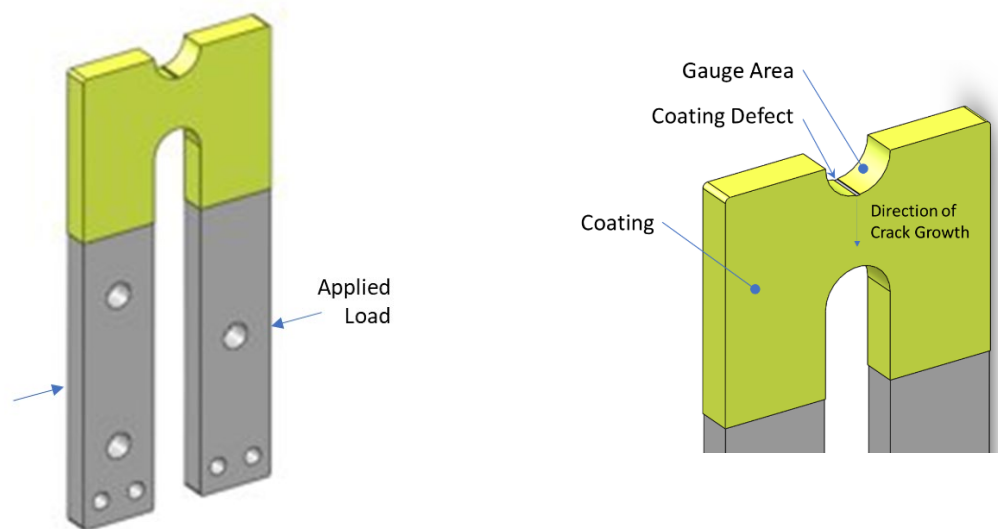


Figure 4. Double cantilever beam sample with coating and coating defect.

9.3 Load Frame

The load frame was designed to apply the required loads to the DCB sample for evaluating SCC of aluminum alloys. The load frame is used to apply a static load to the DCB sample and includes transducers for monitoring load and sample deflection. The complete assembly needs to be robust and hardened for harsh environment testing.

The components of the load frame are the load train with spring actuator, load frame, and transducers (Appendix B). The load frame materials of construction are fiber glass composite structural elements that are connected using stainless steel hardware. The loading is achieved using a spring actuator that is compressed by a controlled amount using a threaded rod and nut. The resultant force and sample deflection are measured using a 1000 N load cell and 3.0 mm displacement transducer.

An enclosure is also used to isolate the load frame from corrosive environments (Figure 5). The enclosure includes cable grips for the transducer wiring, drain plug, and enclosure vent. The DCB sample gauge area is exposed to the environment through an enclosure penetration that is sealed with a gasket at the top of the enclosure (Figure 6).

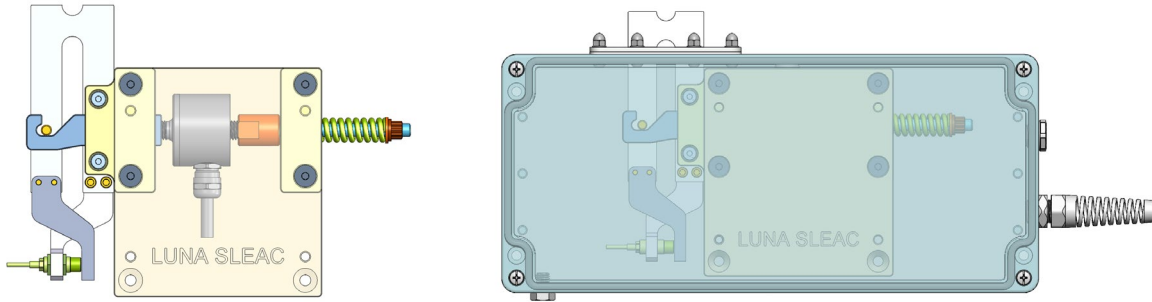


Figure 5. Load frame with DCB sample (left) and load frame with enclosure (right).

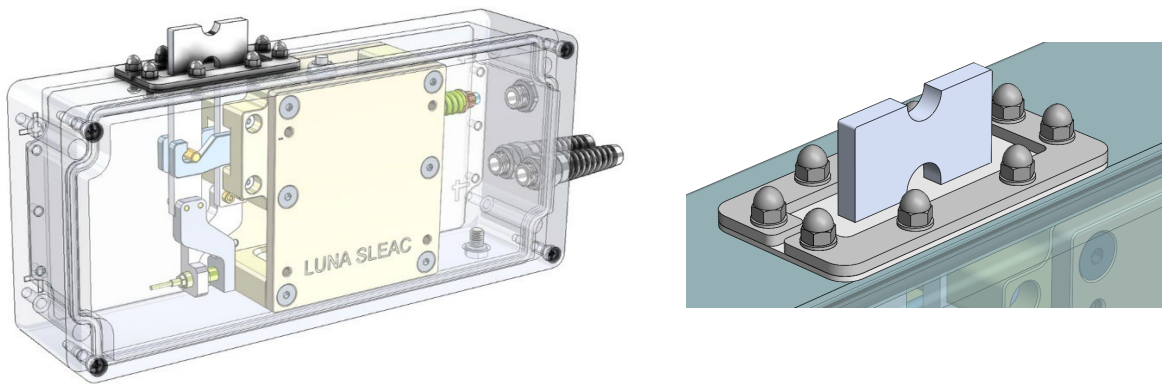


Figure 6. SLEAC load frame enclosure (left) and DCB sample gasket (right).

9.4 DCB Sample and Load Frame Measurements

The load frame, displacement, and load cell are used to apply load and estimate cracking length throughout a test. The estimated crack length is obtained using a compliance calibration curve for the sample geometry and alloy properties. The load frame is capable of applying a load (1000 N) sufficient to achieve 90% of the yield strength (YS) of AA7075-T651 (480 MPa) at the centerline of the gauge area (semicircular edge notch). This is based on a two dimensional finite element analysis (FEA) of an uncracked DCB sample that assumed linear elastic fracture mechanics and plane strain conditions (Figure 7). This analysis is used to determine starting point applied loads to achieve desired percentage of yield strength and select the appropriate load spring to produce the greatest sensitivity to cracking.

- Material: AA7075-T651
- Modulus of Elasticity: 71.7 GPa
- Yield Strength: 480 MPa
- Poisson's Ratio: 0.33
- Density: 2810 kg/m³

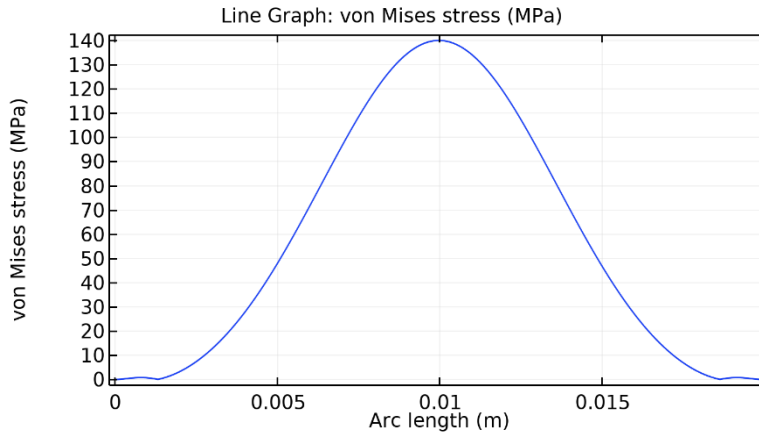


Figure 7. Von Mises stress based on arc length along the gauge area for AA7075-T651 with an applied load of 325 N (30% of YS at gauge area centerline).

A compliance curve, based on the FEA model of an uncracked DCB sample, relates the applied load to beam deflection (Figure 8). Crack growth will alter the sample compliance, therefore, the FEA analysis combined with the load and displacement measurements can be used to estimate crack length as a function of changes in sample compliance (Figure 9). The crack length is linearly interpolated from FEA-simulated crack lengths and the resulting change in compliance.

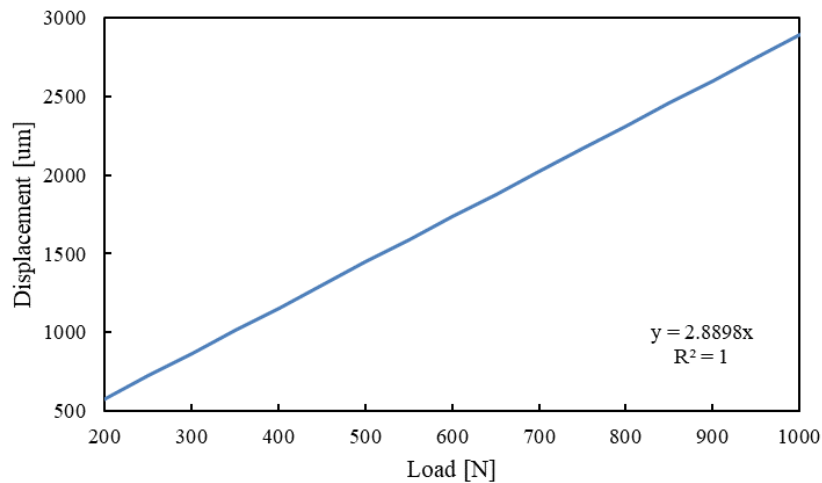


Figure 8. Compliance curve for an uncracked DCB sample (displacement at sensor location).

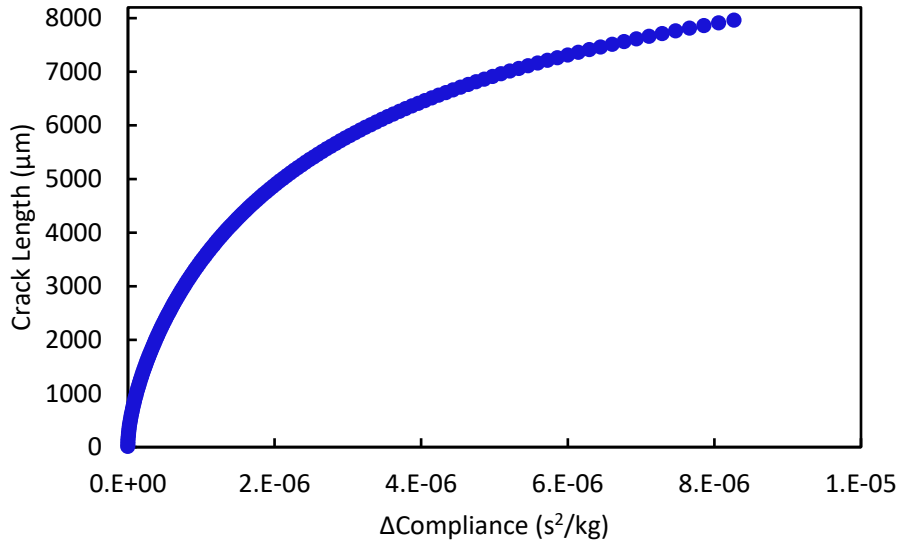


Figure 9. FEA-simulated crack length as a function of change in compliance (displacement at sensor location).

9.5 Data acquisition Hardware, Software, and Analysis

The SLEAC DAU supports up to six SLEAC instrumented load frames (Appendix C). The DAU is line powered and contains power supplies for system component operation. The DAU has an ethernet connection to the test station (PC). The DAU has six pairs of external connectors for the load cells and displacement sensors of each SLEAC load frame. The test station is connected to the DAU through the Moxa NPort 5650-8-DT serial device server that also interfaces with the six load cell channels and displacement sensors.

- Applied Measurements LTD | In-Line Submersible Load Cell | IP68 | Underwater Force Sensor | DDEN
- Kaman Corporation | Non-contact Displacement Measuring System | 9U Standard Probe

The DAU has an enclosure to protect the electronics from environment ingress, but the DAU is not intended for direct exposure in harsh environments. For laboratory testing the DAU can be placed outside the environmental chamber with cabling running from the DAU to the individual load frames that are within the test chamber (Figure 10). The DAU would be located in an unconditioned closed shelter with power when used for outdoor testing.

9.5.1 User Software

A Python user interface (UI) software was developed to monitoring and record data during test setup and execution. The UI is used to monitoring load during initial loading of the sample and continuously monitor, store, and visualize the data during an experiment. The UI supports entering test metadata, selecting number of SLEAC channels being used, identification of load

cells and displacement sensors, setting data rates, and executing commands for start and stop data logging and saving data.



Figure 10. DAU and cyclic corrosion test chamber (left) and SLEAC load frames within chamber (left).

9.5.2 Analysis Software

A Python analysis software was developed to process SLEAC data at the completion of a test. The analysis software is separate from the UI and is still be refined in continued user tests with Luna Labs and NAWCAD.

The analysis software is used to process the load and displacement data to obtain estimates of crack length and crack growth rate as a function of time. The crack length, crack growth rate, and stress intensities are determined from the results of the FEA analysis for the specific alloy being tested. Addition of new alloys will require specific FEA analysis to generate the data libraries used by the analysis software. Currently, all testing and analysis have been done using AA7075-T651. The analysis software includes processing and visualization of load, displacement, change in compliance, crack length, and crack growth rate as a function of time. These analyses also include the calculation of stress intensity and plots of crack growth rate as a function of stress intensity.

9.6 System Environment Resistance

The SLEAC load frame enclosure was designed using a commercial-off-the-shelf (COTS) enclosure for harsh environment use (Figure 5). The SLEAC system was provided to NAWCAD for evaluation in MDACT for 18 days. At the end of the exposure period, the enclosure was inspected for environmental ingress (Figure 11). Minimal ingress was observed on the DCB sample below the gasket, but no other ingress or damage to internal load frame components was observed. Use of a marine sealant applied to the DCB and gasket interface was instituted to minimize solution ingress. It was determined that the enclosure provided sufficient protection from the environment and six additional enclosures were fabricated for testing and delivery to NAWCAD.



Figure 11. DCB sample mounted within the enclosure after 18 days of MDACT exposure.

9.7 System Testing

An accelerated test was performed using the six-channel SLEAC system prior to delivery to NAWCAD.

9.7.1 Alloys and Finishes for SLEAC Testing

Coating systems were selected and 12 DCB samples were coated for testing at Luna Labs (Table 1). Each coated DCB sample had a coating defect centered across the gauge area that was 0.025 in wide (0.635 mm) (Figure 4).

Table 1. Sample and coating combinations for SLEAC testing.

Identification	Reps.	Pretreatment	Primer
Cr	3	Bonderite M-CR 1200S	PPG 02Y040B
PPG-NC	3	Bonderite M-CR T5900	PPG 02GN084
Hntz-NC	3	Bonderite M-CR T5900	Hentzen 16708TEP
Al-rich	3	Bonderite M-CR T5900	PPG GPSP-CA7340

9.7.2 Coated DCB Sample Conditioning

A conditioning step is used for SLEAC testing to provide moisture to the coating and give the inhibitors in the coating an opportunity to migrate and protect the fracture sample substrate at the coating defect.

To condition the samples, filter paper is applied to the gauge area that is dosed with 20 μ L of 2.59 M NaCl (Figure 12). The samples are conditioned in a programmable temperature humidity chamber (Thermotron) set to 90% RH and 40 °C for 72 hours.

At the conclusion of conditioning, the filter paper is removed, and both the filter paper and coating defect inspected. Ideally, both should be imaged and included in test records. The samples are NOT rinsed after conditioning. By not rinsing the sample, any excess inhibitor and salt on the sample gauge surface are present when the sample is loaded at the beginning MDACT cycle.

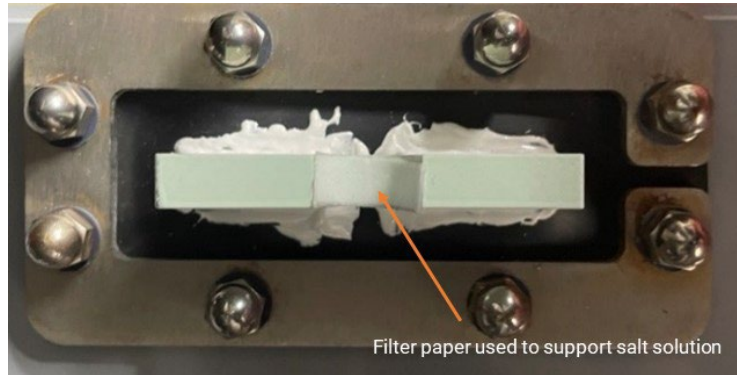


Figure 12. Image of filter paper applied to gauge region during conditioning.

9.7.3 Test Load and Environment

Once the samples are conditioned, they are loaded and exposed to the corrosive test environment.

9.7.3.1 Sample Loading

The samples should be moved to the MDACT and test cycle started as quickly as possible after loading. Given that salt is present on the sample surface after conditioning, cracking may initiate at any time after loading. After conditioning, the samples are loaded to 325 N ($\approx 30\%$ of yield stress) and placed in the cyclic test chamber (Q-FOG CRH) (Figure 10).

9.7.3.2 Test Environment

The SLEAC testing was performed in the MDACT accelerated corrosion test at Luna Labs (Table 2 and Table 3). The MDACT salt solution is pH adjusted to 3, and the method includes fogging and high, medium, and low relative humidity (RH) holds all at a constant temperature of 40 °C.

Table 2. MDACT salt solution.

Test Solution #1 – Modified Sea Salt Solution with Nitrate Ions	
NaCl	11.13 g/L
MgCl ₂ • 6H ₂ O	5.55 g/L
Na ₂ SO ₄	2.00 g/L
NaNO ₃	1.635 g/L
HCl (1N)	pH = 3 (acidified with HCl)

Table 3. MDACT cycle description.

Step	Condition	Duration (hh:mm)
1	Fog	06:00
2	Sub-cycle 2.1 – 2.3 Repeat 11x	(66:00)
2.1	90% RH Hold	02:00
2.2	60% RH Hold	03:00
2.3	40% RH Hold	01:00
3	Fog	06:00
4	Sub-cycle 4.1 – 4.2 Repeat 15x	(90:00)
4.1	90% RH Hold	02:00
4.2	60% RH Hold	03:00
4.3	40% RH Hold	01:00
Return to Step 1		(Total 168)

9.7.4 Results

Testing was done in two sets of six samples to achieve triplicate measurements for the four coating systems. This test matrix demonstrated that the fog cycle can be very aggressive in promoting EAC for coatings with minimal protective properties (Hentzen and PPG non-chromate primers) (Figure 13 and Figure 14). The aluminum-rich primer did not seem to have the same sensitivity to the fog cycle as observed for the other non-chromate primers. Other than the chromate primer, the aluminum-rich primer was better for protecting the AA7075-T651 DCB sample from SCC compared to the other coatings tested, and in only one replicate did the Hentzen 16708TEP match the aluminum-rich performance (Figure 13 and Figure 15).

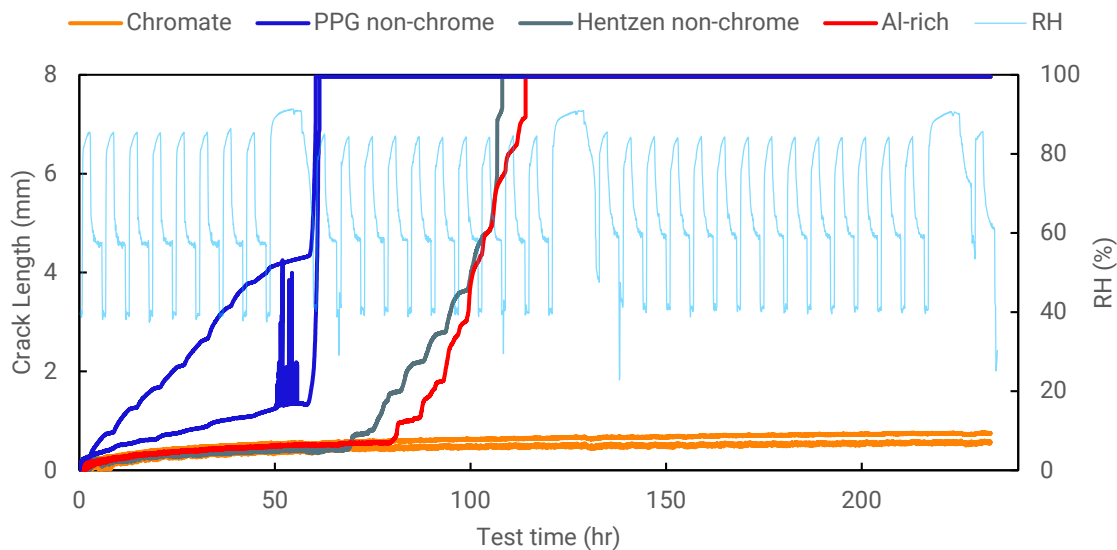


Figure 13. Crack length as a function of time for first test set. Full test period.

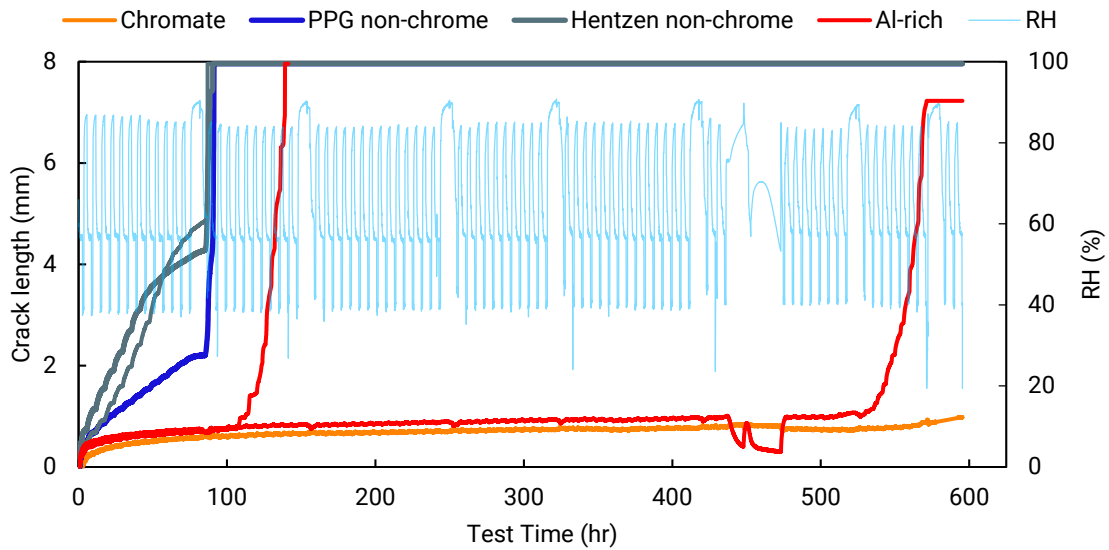


Figure 14. Crack length as a function of time for the second test set. Full test period.

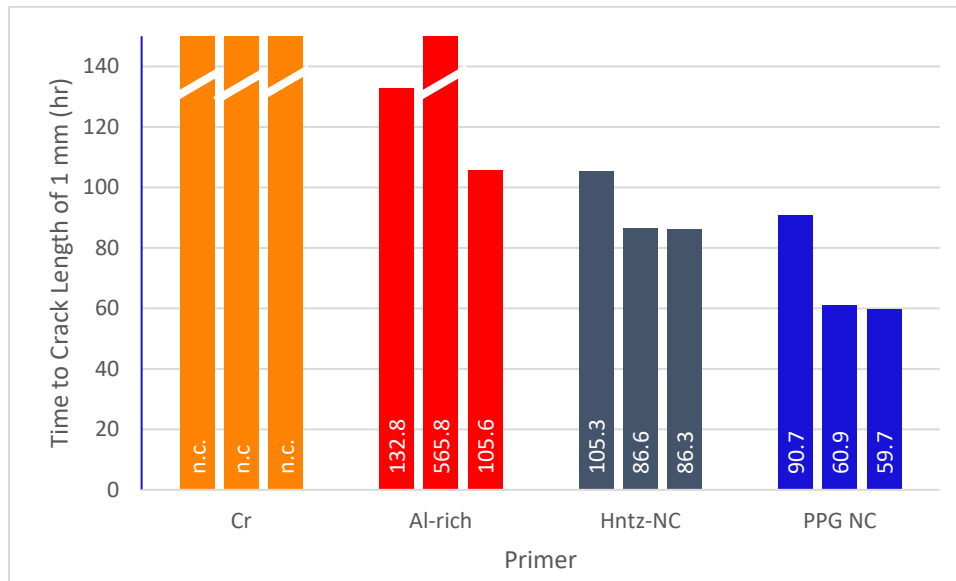


Figure 15. Time to 1 mm of crack length for triplicate samples with each primer (n.c. indicates no cracking).

All six samples were imaged at the conclusion of testing. Based on visual observation, the chromate coated sample did not have obvious corrosion damage or blistering (Figure 16). The other samples had various levels of coating deterioration and corrosion, but more detailed microscopy would be needed to associate coating condition, corrosion, and cracking results.

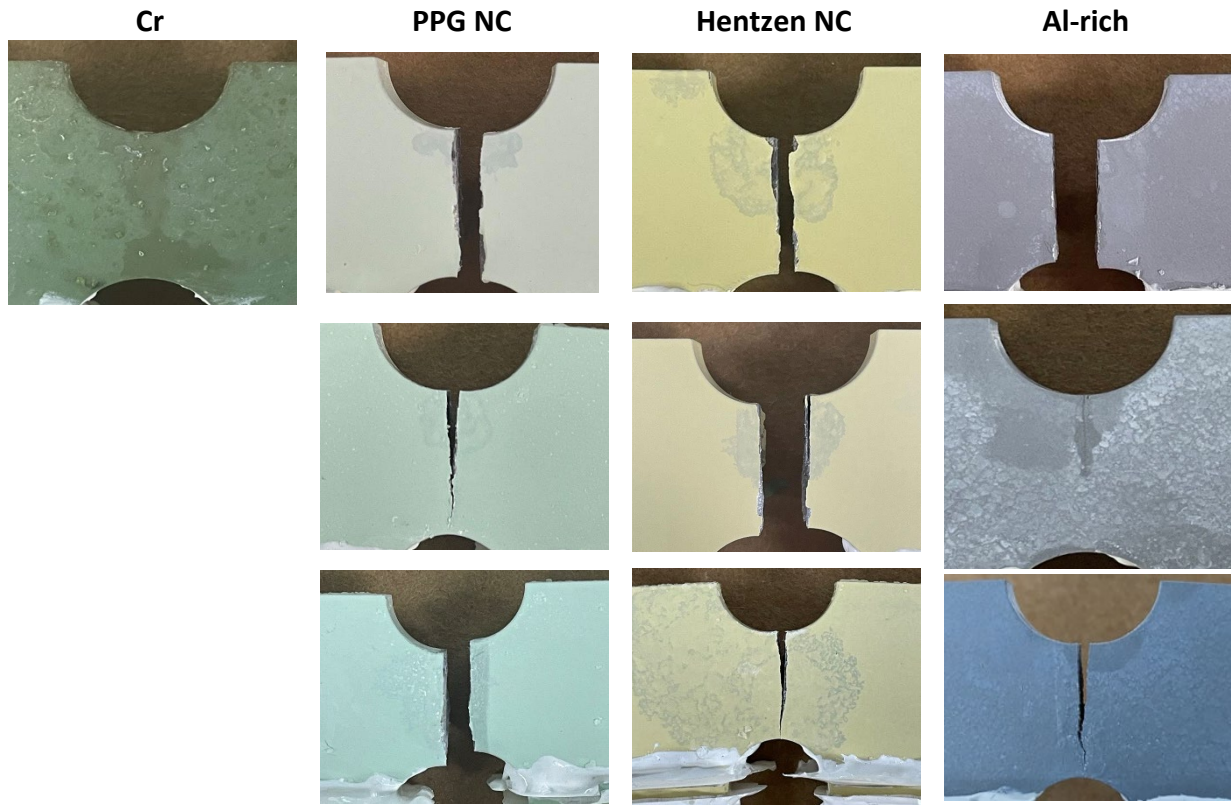


Figure 16. Images of coated DCB samples after MDACT exposure.

9.8 SLEAC System Delivery

After testing was complete, the system was delivered to NAWCAD on 8 November 2023. The system will be used to test coatings and compare results to those obtained in this project. NAWCAD experience will be used to inform test method and system improvements. An operating manual and conditioning protocol were provided to guide system set up and use.

10 Findings and Conclusions

An SCC test system has been completed and used for coating evaluations and making *in situ* measurements of crack length during accelerated corrosion tests.

The conditioning process and MDACT cycle are the starting point for conducting coating performance evaluations and revising TM21449.

11 Plans and Upcoming Events

During the option period, the system will continue to be tested by both Luna Labs and NAWCAD. The system use, test method, and findings will be used to revise TM21449.

12 Transitions and Impacts

The SLEAC test system and EAC coating evaluation method represent significant advancement in coatings testing for performance properties that are relevant to DoD aerospace applications. Coating testing using SLEAC provides quantitative performance data that addresses a significant corrosion mechanism that is relevant to aircraft structural integrity.

The first low rate initial production system was delivered to NAWCAD for coatings testing. The system is available for commercial sale. Validation by NAWCAD, revised standard method, and commercial availability will all support SLEAC system transition and use.

13 Collaborations

Luna Labs has been in close collaboration with Steve Kopitzke, Alex Lilly, and Mike Brindza on the development and use of the test system and methodology. NAWCAD is the intended user for the technology implementation.

14 Personnel

Principal investigator: Fritz Friedersdorf, 1 person-months, Country: USA

Co-PI: Victoria Avance, 2 person-months, Country: USA

Business Contact: Not applicable

Team Members: Nate Brown, 1 person per month, Country: USA

Subs: Not applicable.

15 Students

Not applicable.

16 Technology Transfer

Luna Labs personnel continue to participate in AMPP SC 07 Defense and Aerospace. The AMPP standard TM21449-2021, *Continuous Measurements for Determination of Aerospace Coating Protective Properties* will be updated to be consistent with Navy coating SCC tests. A draft technology transition plan has been completed.

The SLEAC system is available for commercial sale from Luna Labs.

17 Products, Publications, Patents, License Agreements, etc.

Publications resulting from this project:

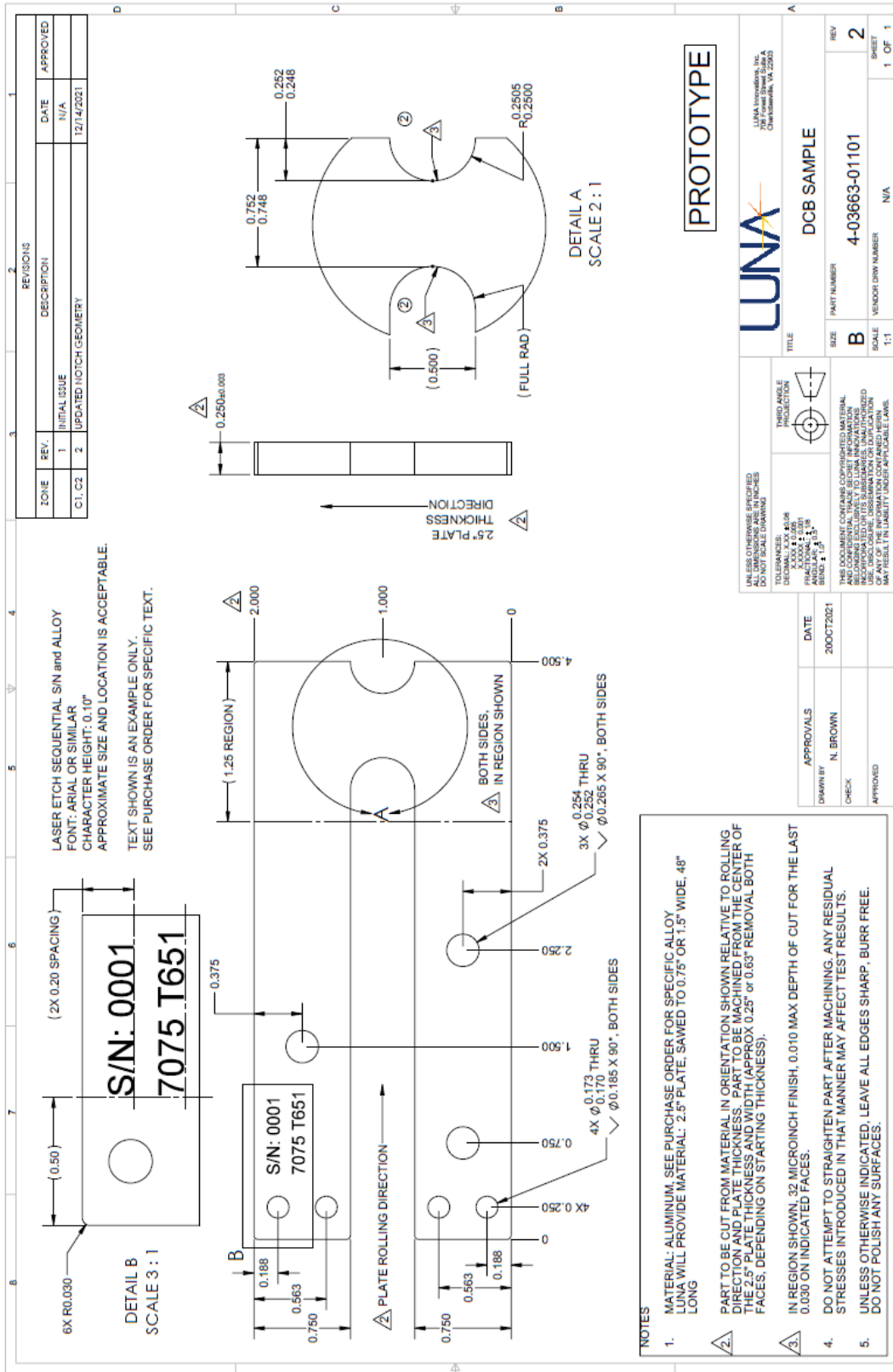
F.J. Friedersdorf and L. Agnew. Evaluating Coating Protection Against Environmentally Assisted Cracking. Presentation at EUROCORR 20223, Brussels Belgium, 27 – 31 Aug 2023.

Other Products: The SLEAC system is available for sale from Luna Labs. The system has been designed and fabricated to be compliant with TM21449 test requirements.

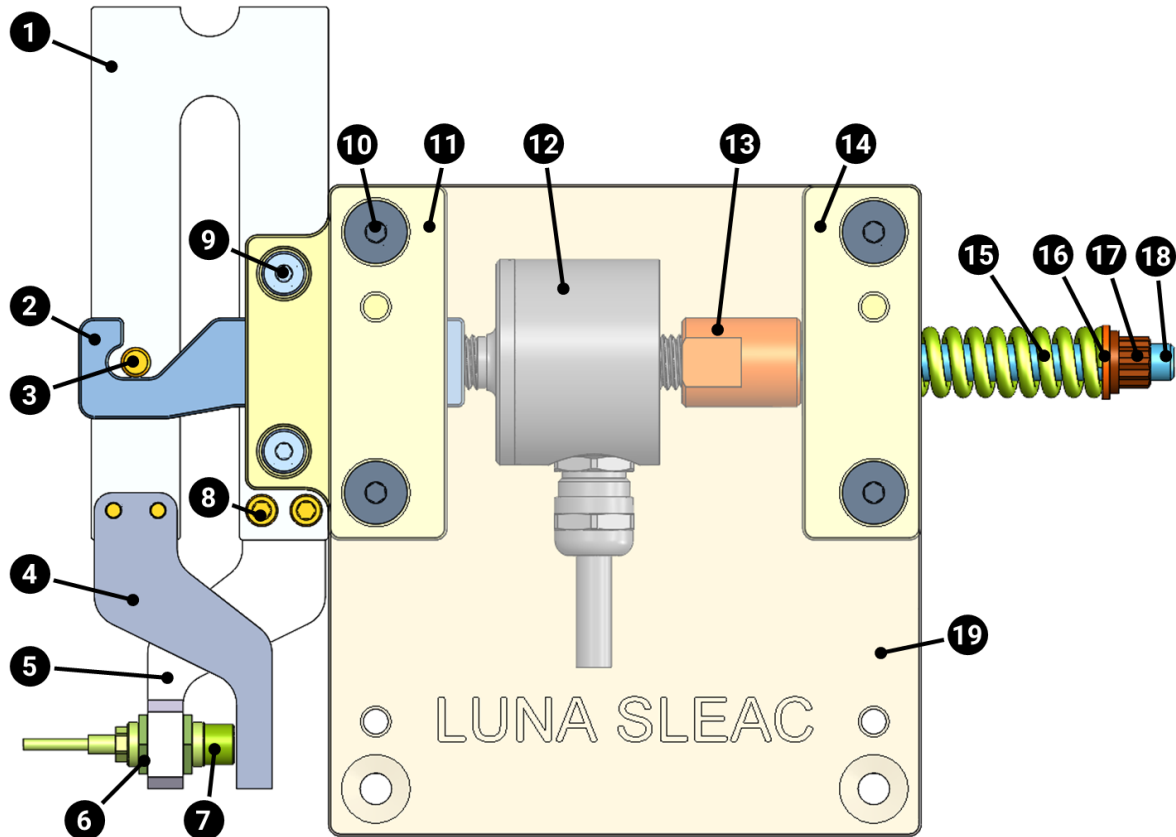
18 Point of Contact in Navy

Name: Alex Lilly
Affiliation: NAWCAD Materials Protection Technology Branch
Contact information: alexander.d.lilly2.civ@us.navy.mil, (301) 342-7436
Date of last contact: 19 December 2023

Appendix A

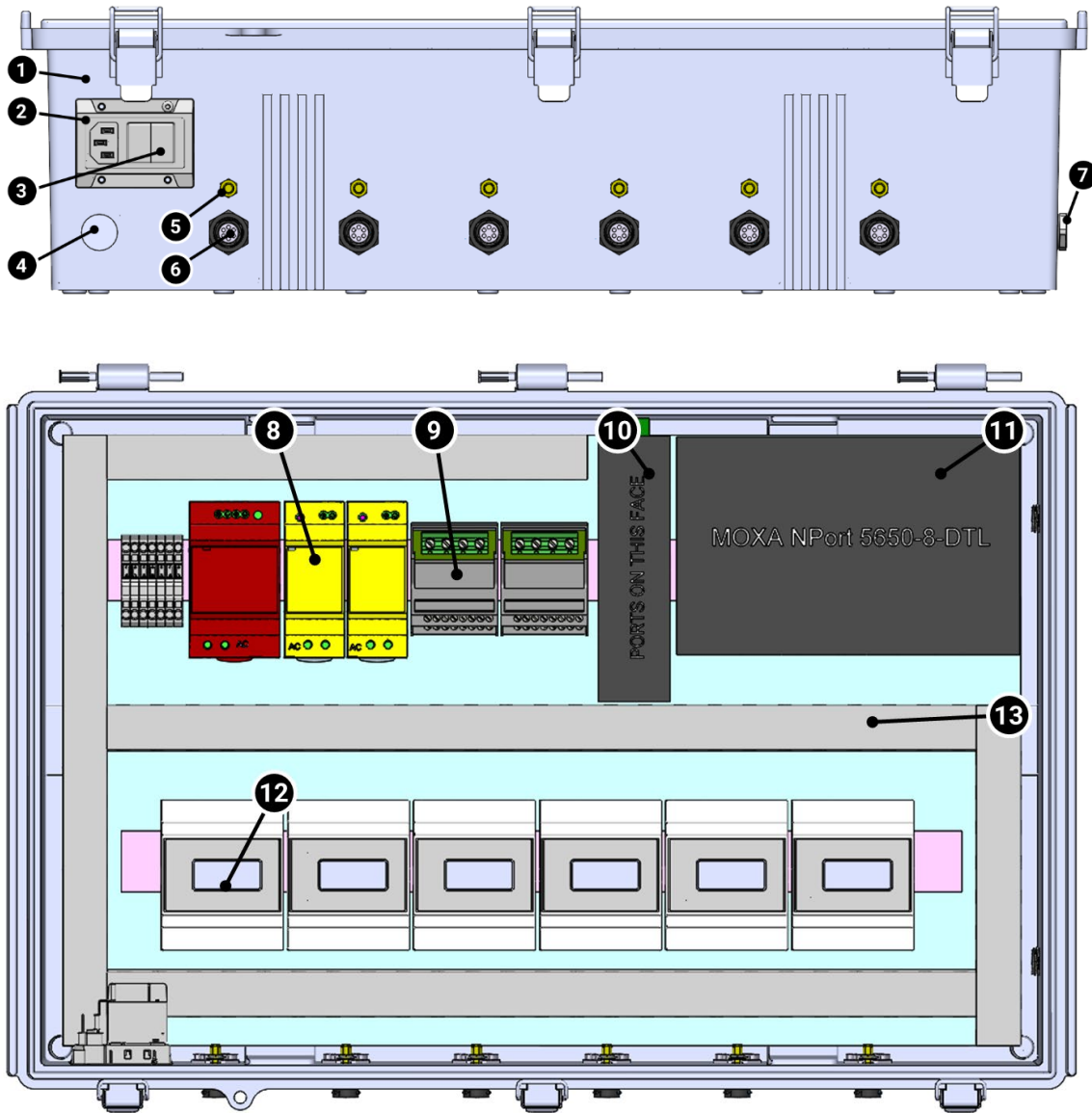


Appendix B



- | | |
|--|--------------------------------|
| 1. Double-Cantilever Beam (DCB) Sample | 14. Load Block |
| 2. Load Clevis | 15. Load Spring |
| 3. Clevis Pin | 16. Load Washer |
| 4. Displacement Sensor Target | 17. Load Nut |
| 5. Displacement Sensor Bracket | 18. Load Rod |
| 6. Displacement Sensor Locknut (2x) | 19. Load Frame Side Panel (2x) |
| 7. Displacement Sensor | |
| 8. Displacement Sensor Target/Bracket Fastener | |
| 9. DCB Clamp Screw (2x) | |
| 10. Side Panel Screw (8x) | |
| 11. DCB Clamp Block | |
| 12. Load Cell | |
| 13. Thread Adapter | |

Appendix C



DAU Enclosure

- | | |
|--|---|
| <ol style="list-style-type: none"> 1. Power Connector 2. Power Switch 3. Ethernet Jack to Test Station (PC) 4. Displacement Sensor Connector (up to 6x) 5. Load Cell Connector (up to 6x) 6. Enclosure Vent 7. Power Supplies | <ol style="list-style-type: none"> 8. Power Distribution Blocks 9. Ethernet Switch 10. Serial to Ethernet Converter (MOXA) 11. Displacement Sensor Instrument (digiVIT) (up to 6x) 12. Wire Duct |
|--|---|