

Laser-Interference Surface Preparation for Enhanced Coating Adhesion and Adhesive Joining of Multi-Materials

Project Number WP-2743

**Adrian Sabau
Computer Science and Engineering Division
Oak Ridge National Laboratory**

**End-of-Project Presentation
November 5, 2021**



REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.
PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE (DD-MM-YYYY) 05/11/2021		2. REPORT TYPE SERDP Project Outbrief		3. DATES COVERED (From - To)	
4. TITLE AND SUBTITLE Laser-Interference Surface Preparation for Enhanced Coating Adhesion and Adhesive Joining of Multi-Materials				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Adrian Sabau Harry Meyer				5d. PROJECT NUMBER WP-2743	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Oak Ridge National Laboratory 1 Bethel Valley Road, bldg 4508, MS 6083 Oak Ridge, TN 37831				8. PERFORMING ORGANIZATION REPORT NUMBER WP-2743	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Strategic Environmental Research and Development Program 4800 Mark Center Drive, Suite 16F16 Alexandria, VA 22350-3605				10. SPONSOR/MONITOR'S ACRONYM(S) SERDP	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) WP-2743	
12. DISTRIBUTION/AVAILABILITY STATEMENT DISTRIBUTION STATEMENT A. Approved for public release: distribution unlimited.					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT Understand and demonstrate the use of a one-step process at ambient conditions based on laser-interference structuring techniques to replace the chemical conversion coating steps prior to the application of: (a) coating systems and/or (b) adhesive bonding for aluminum (Al) and titanium (Ti) aircraft components.					
15. SUBJECT TERMS Laser-Interference Surface Preparation, Enhanced Coating Adhesion, Adhesive Joining, Multi-Materials					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UNCLASS	18. NUMBER OF PAGES 39	19a. NAME OF RESPONSIBLE PERSON Adrian Sabau
a. REPORT UNCLASS	b. ABSTRACT UNCLASS	c. THIS PAGE UNCLASS			19b. TELEPHONE NUMBER (Include area code) 865-241-5145

Project Team at ORNL and Steering Committee

<i>Team Member</i>	<i>Discipline/Role</i>
Dr. Adrian Sabau	Materials Scientist (Photonic processing, Laser-material interaction) - PI
Dr. Harry Meyer III	Physical Chemist (XPS)
Dr. Leonard Donovan	Electron Microscopy
Dr. Jianlin Li	Materials Scientist (Wetting, Surface energy)
Dr. Claus Daniel	Materials Scientist (Laser-material interaction)
Dr. Don Erdman	Mechanical testing engineer (shear lap)
Dr. Jiheon Jun	Materials Scientist (Corrosion science)
Mike Stephens	Coating (equipment, application, testing)
Rick Osterman and Stan Bean	Steering committee, M&P Solution, LLC. (coating application and joining for aerospace applications)
Tom Naguy	Deputy Technical Director, AF Material Command, Wright Patterson AFB (coating application and joining for AF)
M. Casey Jones	Materials Engineer, Wright Patterson AFB

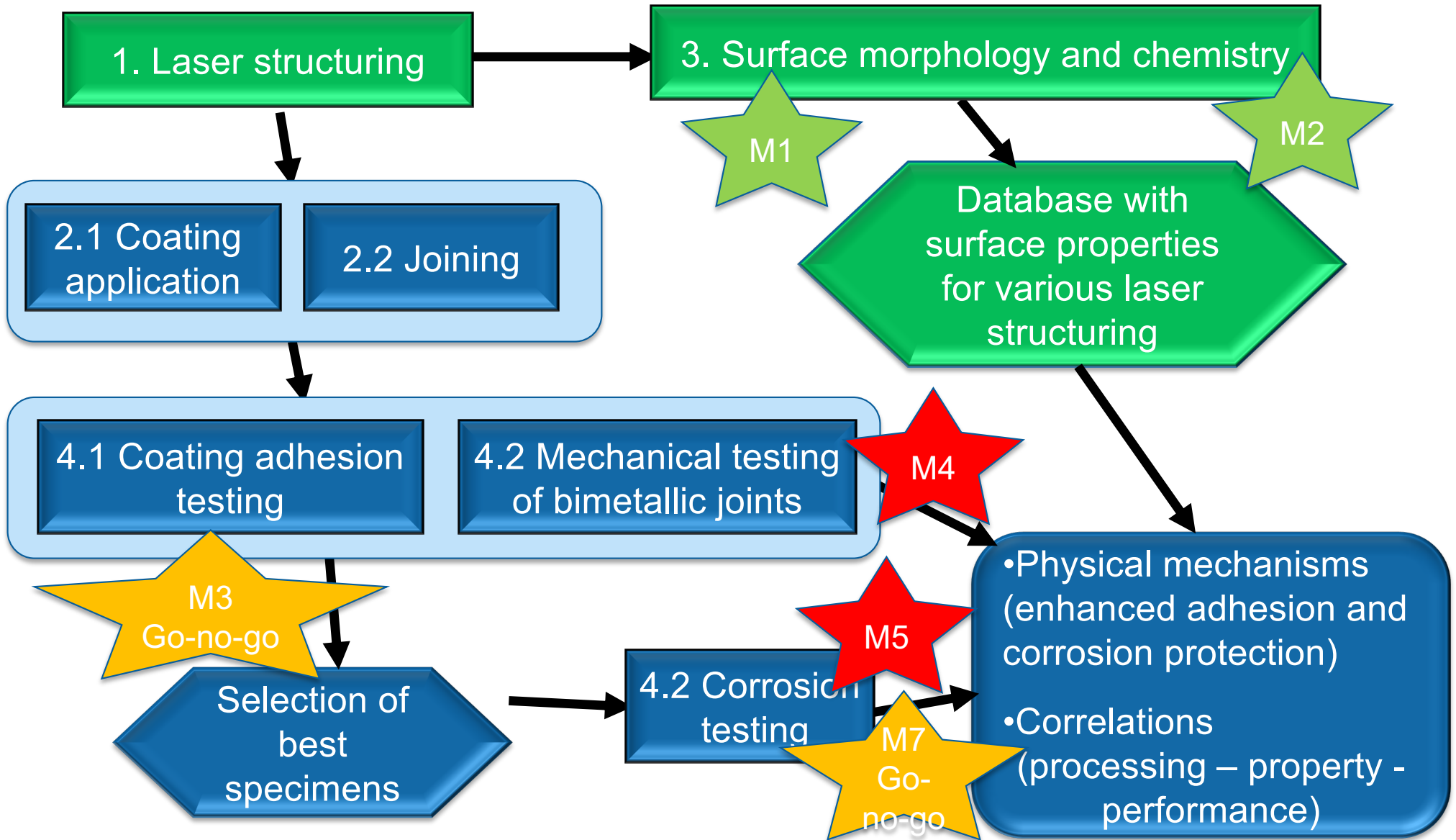
Background

- Corrosion protection of DoD's tens of thousands of aircraft, ground vehicles, and ships is accomplished using multi-step, complex painting/coating processes requiring cadmium, volatile organic compounds, and chromates
 - ◆ DoD cost for corrosion protection is larger than \$20 billion/year.
 - ◆ Chromate solutions (hexavalent chrome - type I) are highly toxic.
- **Challenge:** New non-chemical surface preparation would drastically reduce environmental impact, risks, and costs.
- SERDP WPSO-17-03: "*Surface morphology modification by non-chemical methods to enhance coating adhesion and mechanical bonding of metal surfaces.*"
- Project initiated: Febr. 2017

Technical Objective

- Understand and demonstrate the use of a one-step process at ambient conditions based on laser-interference structuring techniques to replace the chemical conversion coating steps prior to the application of: (a) coating systems and/or (b) adhesive bonding for aluminum (Al) and titanium (Ti) aircraft components.

Technical Approach



Results were very promising

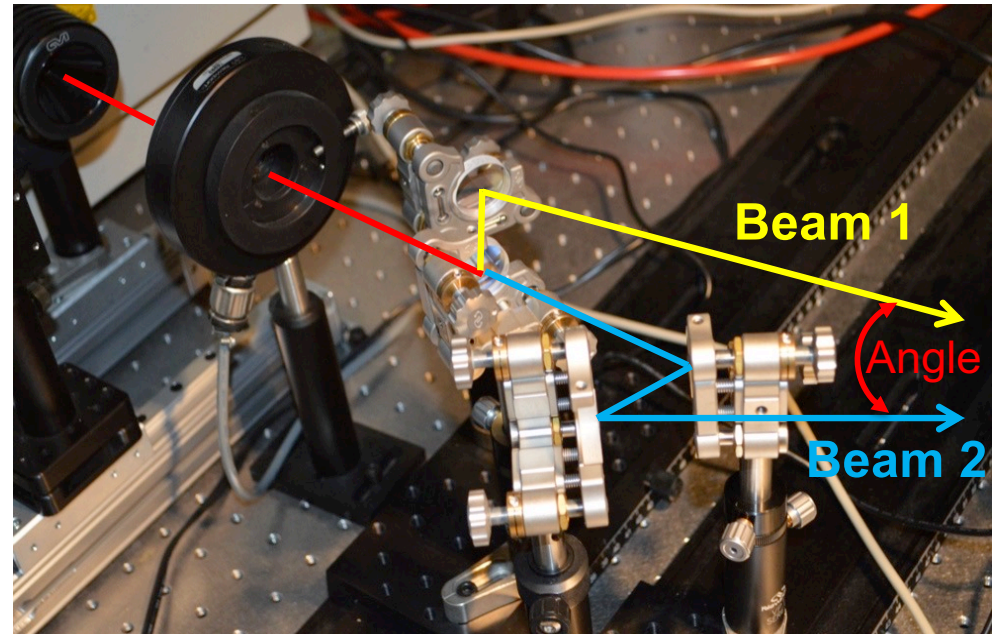
Milestone	Description	Date passed
3 Go-no-go	Attain 80% or better coating adhesion properties with laser-structured surfaces of Al than the baseline specimens	8/18
4	Attain 80% or better single-lap shear with laser-structured surfaces of Al than the baseline specimens	11/18
5	Attain 80% or better corrosion properties than the baseline specimens for 2x2in Al using ASTM B 117 for 500h	12/18
7 Go-no-go	Attain 80% or better corrosion protection than the baseline specimens for Al using ASTM B 117 at 2000h	3/20

Laser-interference structured (LIS) specimens:

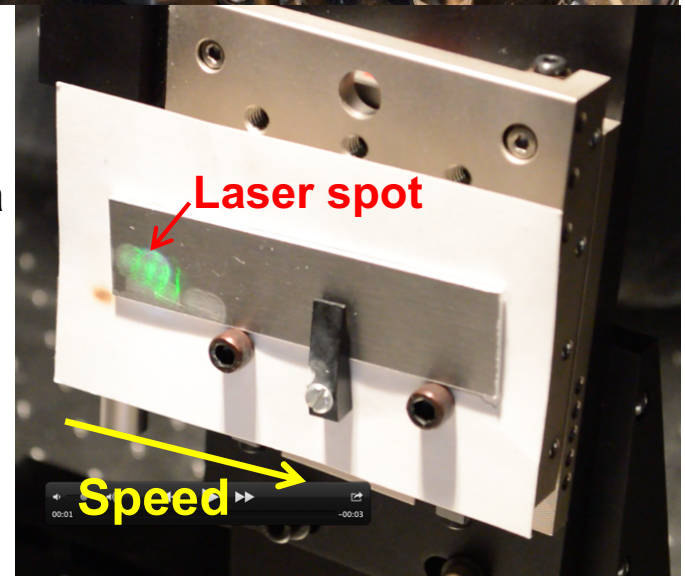
- Met the performance requirements for the coating adhesion
- Performed well in corrosion testing

Technical Approach - Laser Setup involves two or more beams originating from same laser

- Q-switched Nd:YAG laser with an harmonic generator enabling the selection of sharp wavelengths (1064, 532, 355, or 266nm)
- Pulse duration 10ns, frequency 10Hz
- Main variables related to laser-structuring:
 - ◆ Wavelength
 - ◆ Beam angle
 - ◆ Spot size
 - ◆ Rastering speed (or number of shots)



Sample mounted on a translational stage controlled by LabView

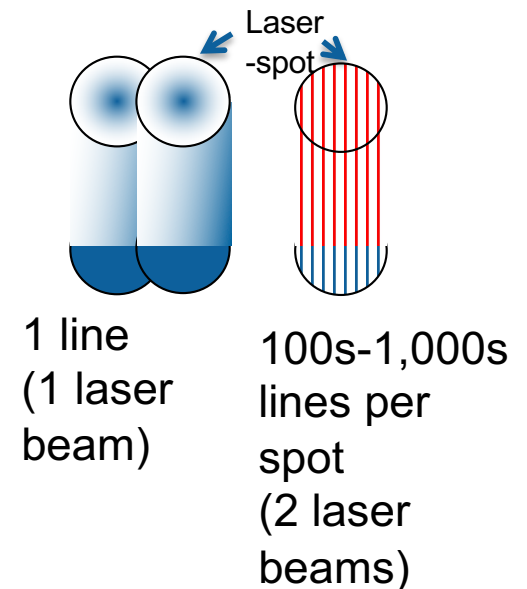
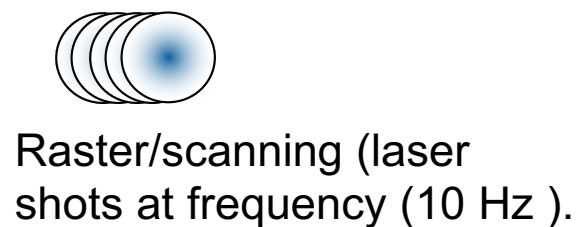
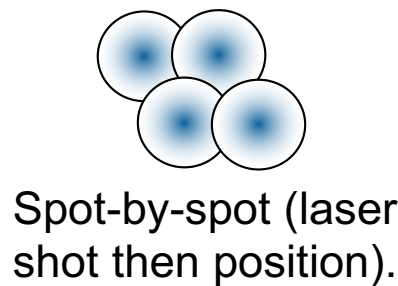
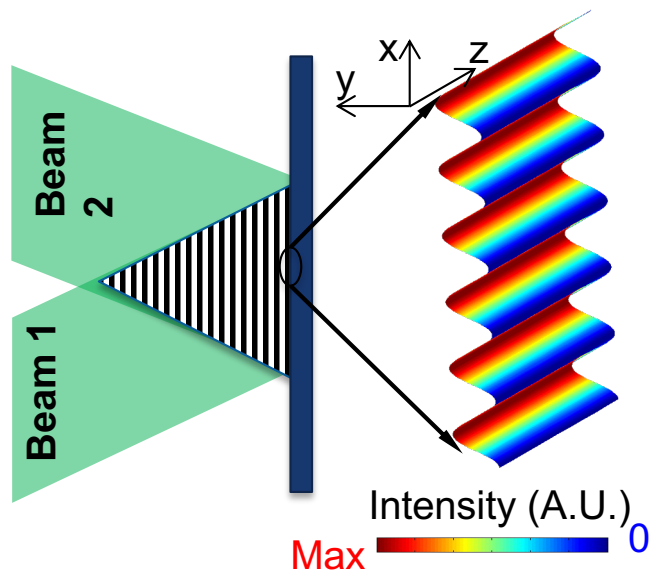


Nd:YAG = neodymium-doped yttrium aluminum garnet; $\text{Nd:Y}_3\text{Al}_5\text{O}_{12}$)

Paradigm Shift of Laser-interference

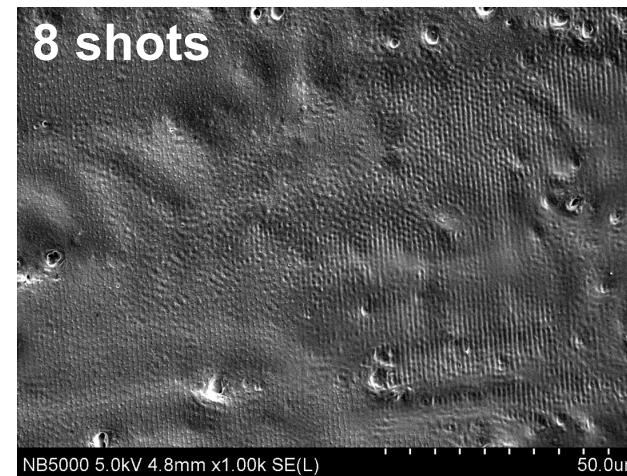
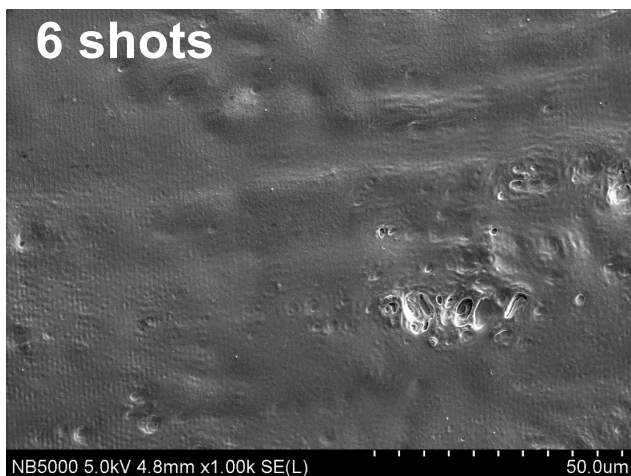
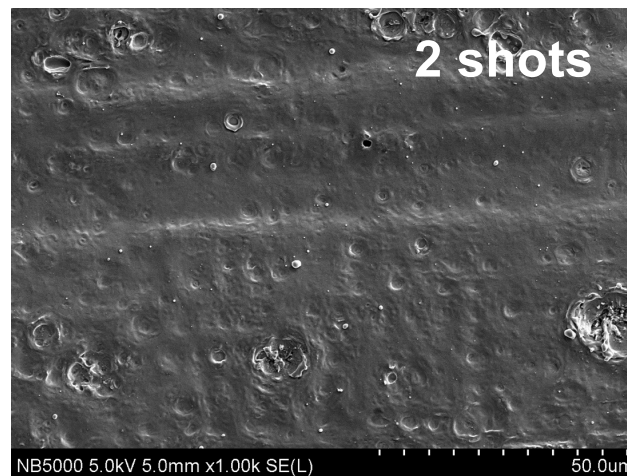
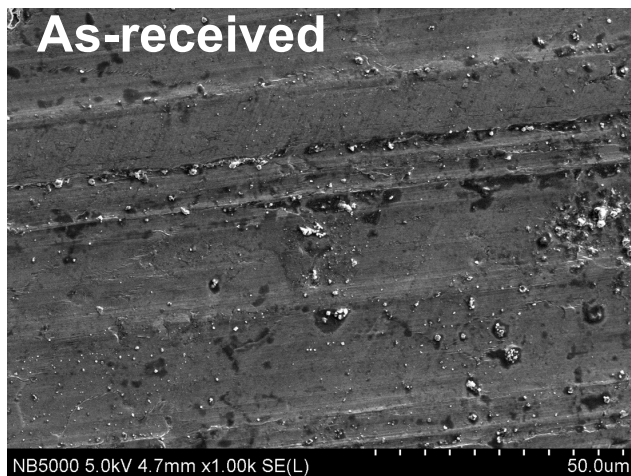
More Structuring at the Same Productivity

Method	Traditional (1 beam laser)	New Laser-Interference (2 or more beams)
Structure	1 dimple per spot	300 to 15,000 mini dimples (pits) per spot
Sizes	80-500 μm diameter 10 μm depth	0.4-20 μm diameter 200 nm – 3 μm depth



Through wave-interference, alternating regions of high-power (**constructive**) and low-power (**destructive**) are created on the specimen surface.

Laser-interference technique is “smoothing-out” all the sharp features from the as-received rolling surface, minimizing surface defects – *beneficial effect on fatigue*

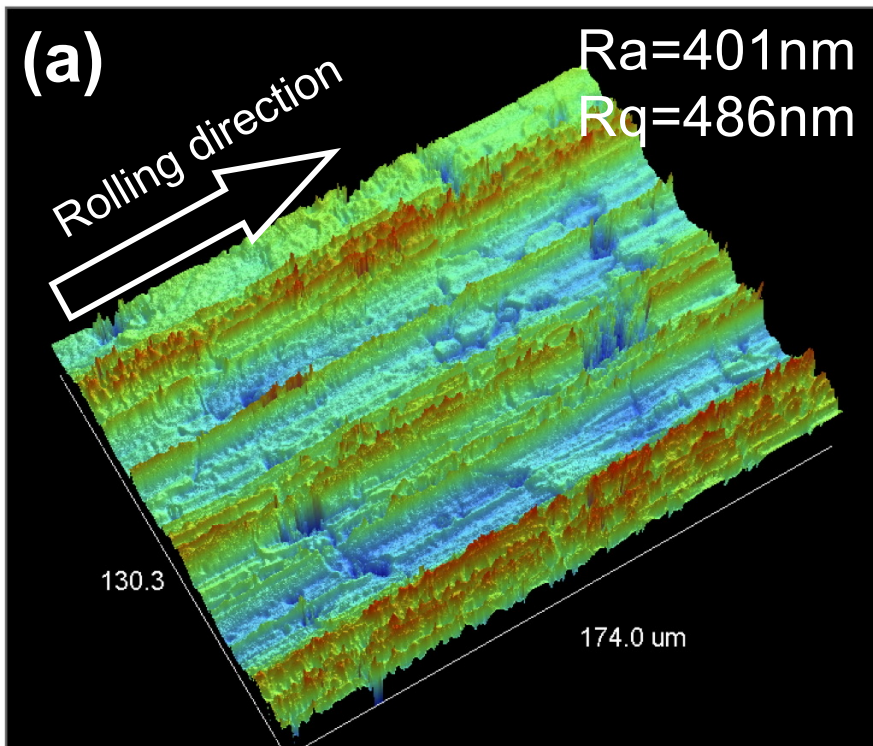


Field Emission Scanning Electron Microscopy (FESEM) images.

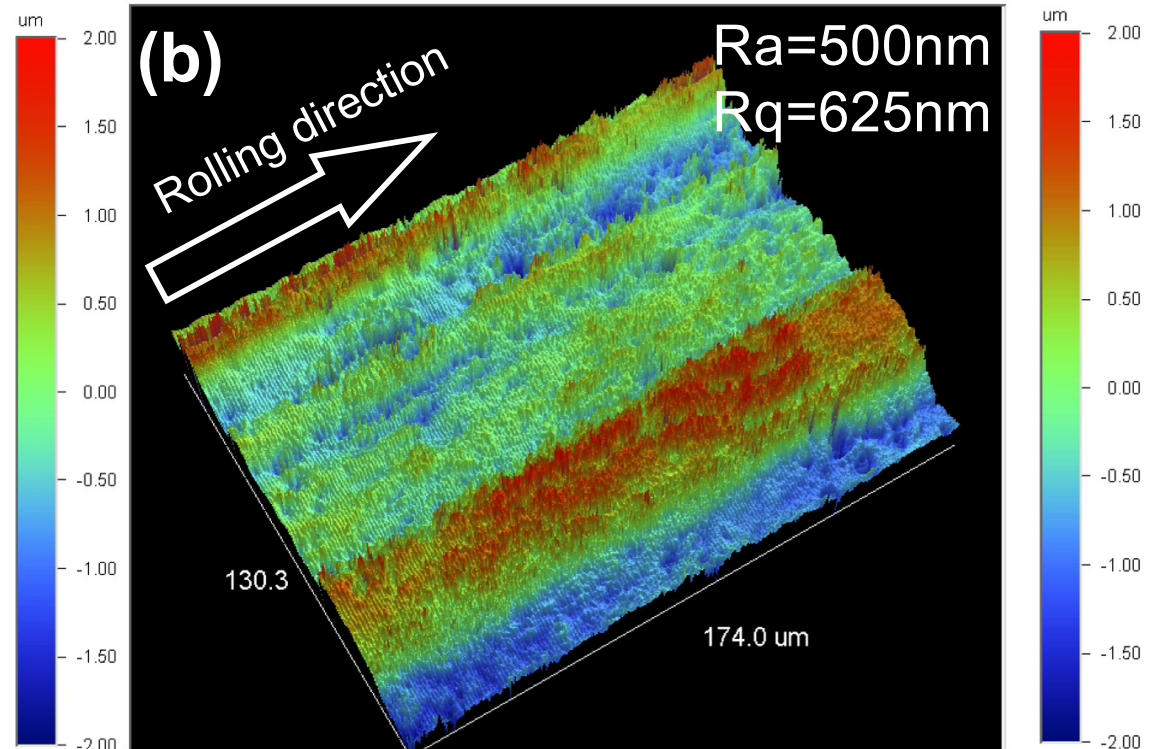
Note: micro-cracking of Al alloy surfaces are seen around melt cavities produced with “traditional one-beam” laser surface structuring.

Results: More Surface Area with Laser-structuring

As-Received vs. Laser-Interference Structured Surfaces



As-received, unprocessed



Striations normal to rolling direction were produced by laser-interference structuring

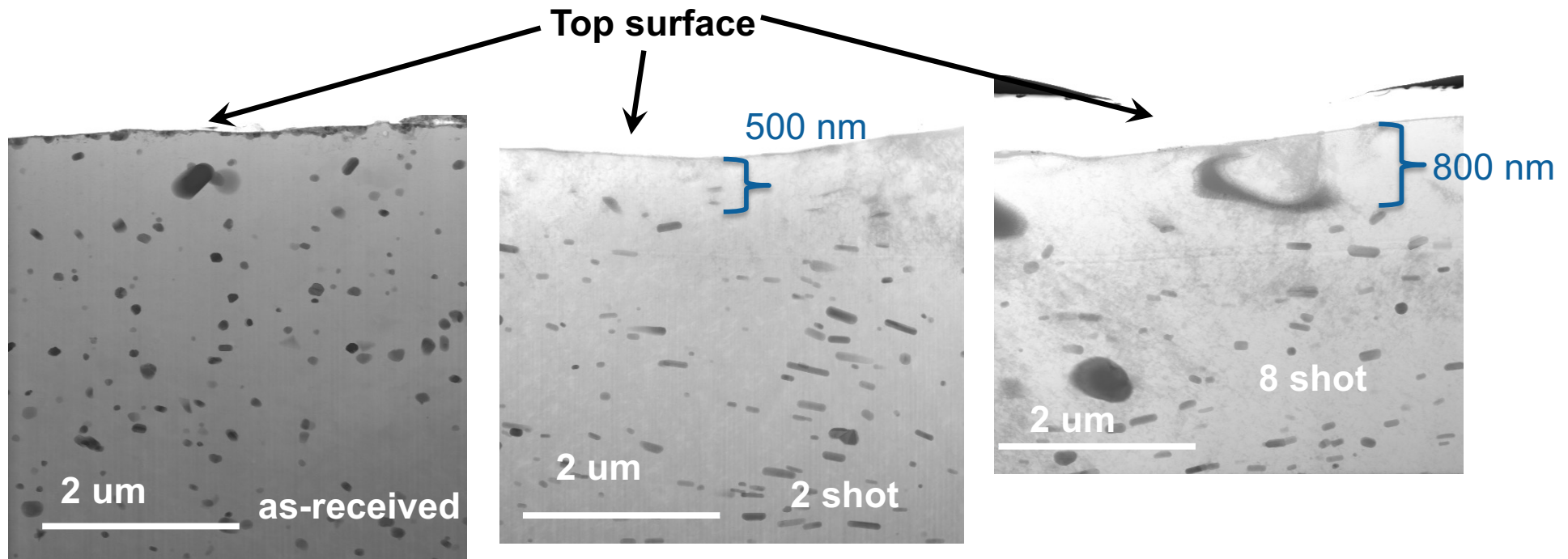
Optical surface profile

Ra = arithmetical mean roughness

Rq = root mean square average roughness

4 shots/spot ($\lambda=355$ nm,
 pulse fluence of 1.2 J/cm²)

Results for Milestone 2: Dissolution of Al-Cu-Mn precipitates occurred near the surface for the laser-interference specimens



Al-2024-T3 baseline material – as-received specimen (without any laser processing)

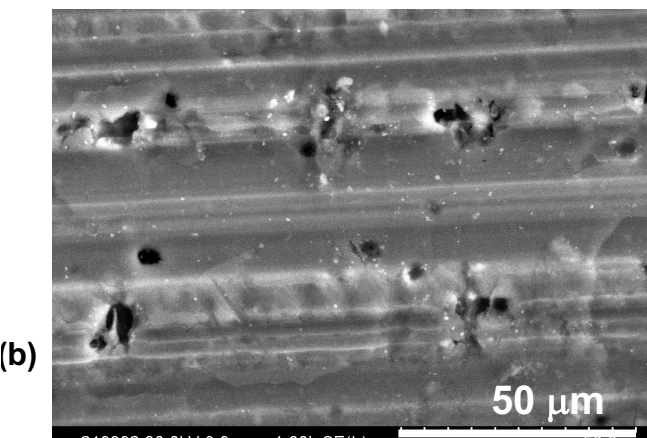
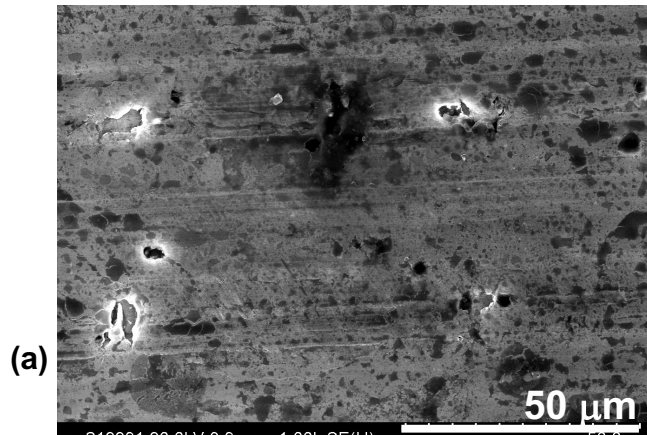
Laser processed specimens with:

2 shots/spot

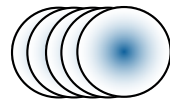
8 shots/spot

Precipitate dissolution near the surface (500-800 nm) is expected to enhance the corrosion protection of Al-2024-T3.

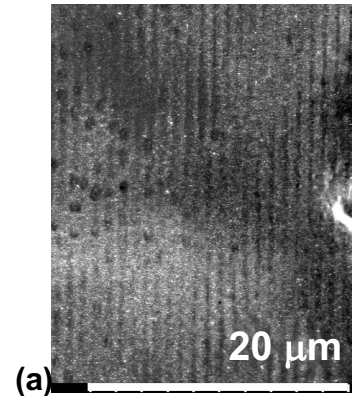
Surface structuring by raster/scanning was attained using laser-interference



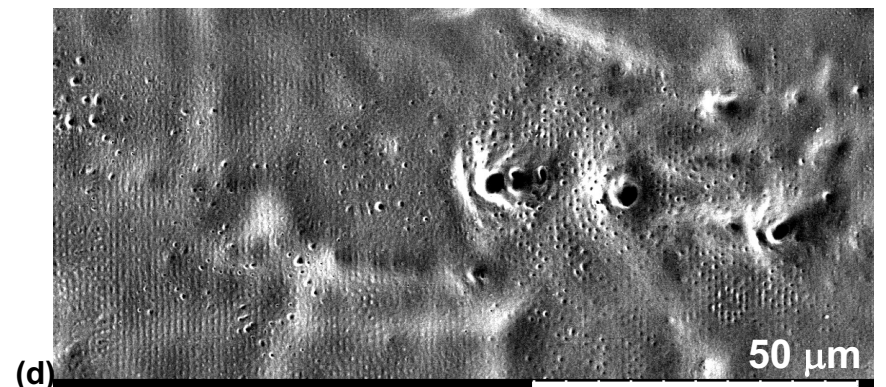
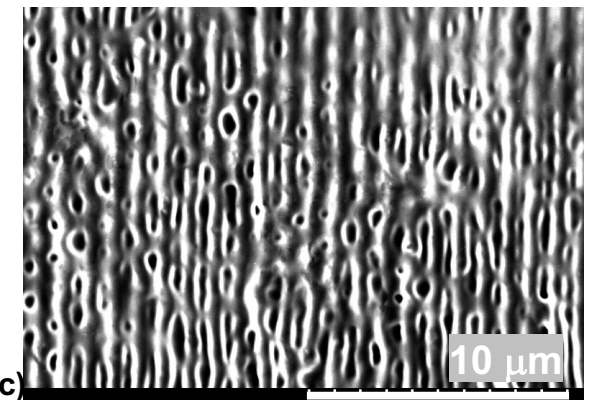
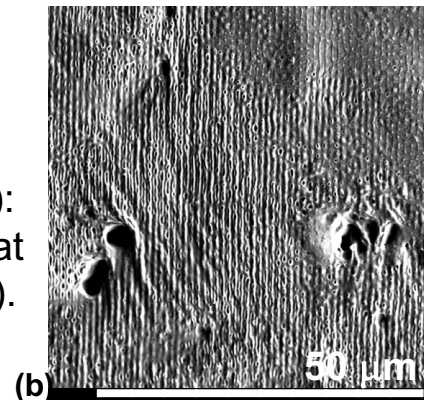
Top view FESEM images for as-received (i.e., without any laser structuring). Same area: (a) in-lens SE detection, and (b) lower SE detector.



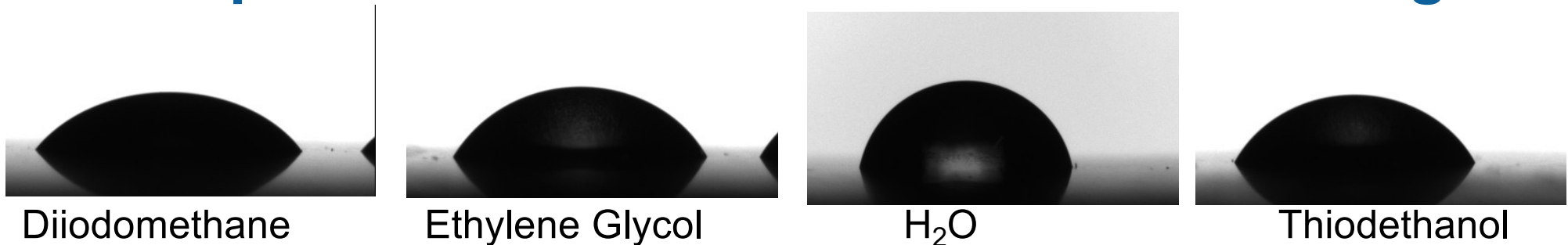
Raster (scanning): laser fires shots at frequency (10 Hz).



Top view FESEM images for the laser-structuring at raster speeds of: (a) 2 mm/s, (b, c) 4 mm/s, and (d) 6 mm/s



Results contact angle measurements indicate that laser-structured specimens were compatible with chemical conversion coating

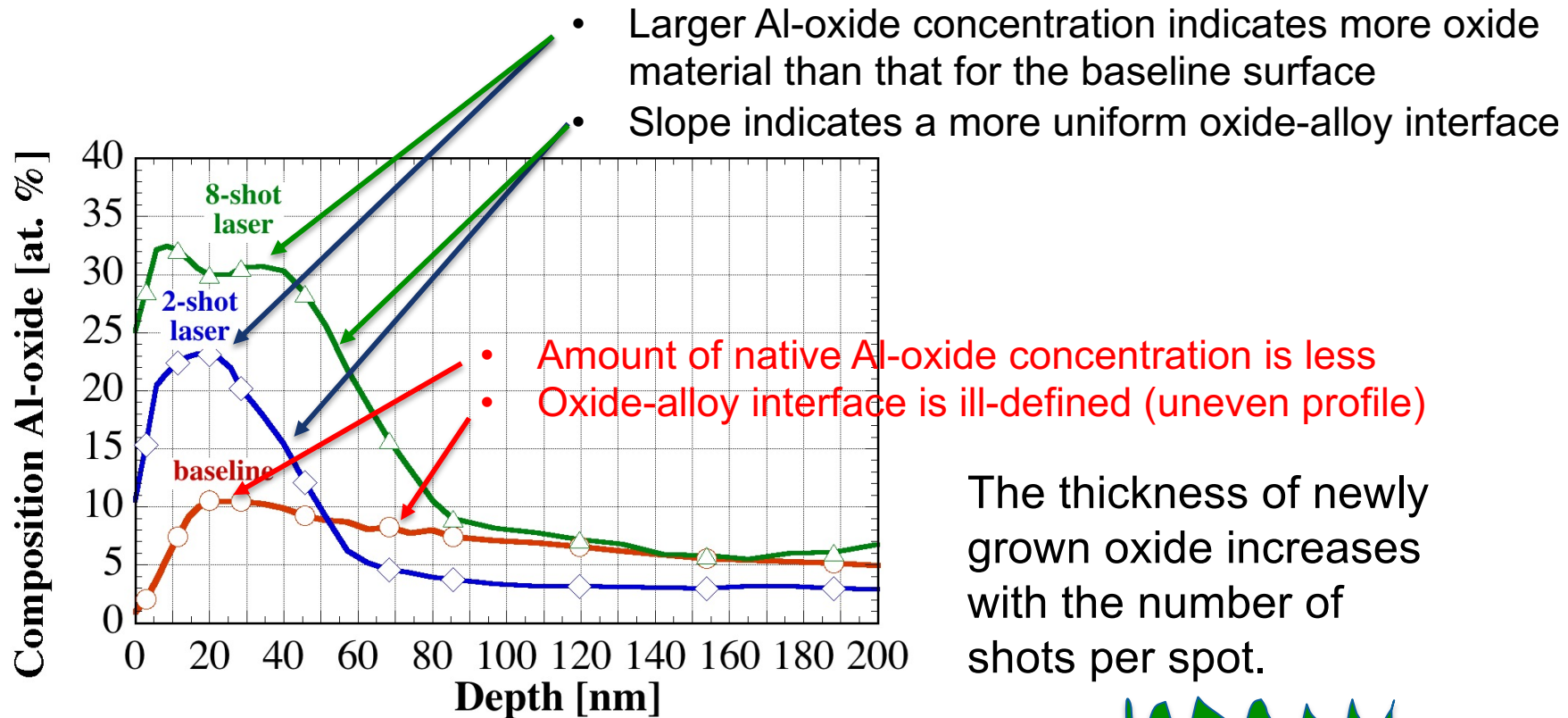


Pictures used for contact angle measurements of liquid droplets on Al2024-T3 specimens laser-interference processed with 8mm/s raster speed.

Liquids	Laser A1-6		Laser A1-8		CCC	SAA		
	Mean (°)	Std. Dev. (°)	Mean (°)	Std. Dev. (°)	Mean (°)	Std. Dev. (°)	Mean (°)	Std. Dev. (°)
Diiodomethane	48.84	2.31	43.58	1.40	54.96	0.75	9.40	0.90
Ethylene Glycol	73.56	0.74	72.36	0.52	53.90	1.46	-	-
Thiodethanol	68.60	1.22	67.14	0.44	67.86	1.04	20.4	0.98
Water	96.92	1.17	97.16	1.83	90.10	2.50	27.25	12.24

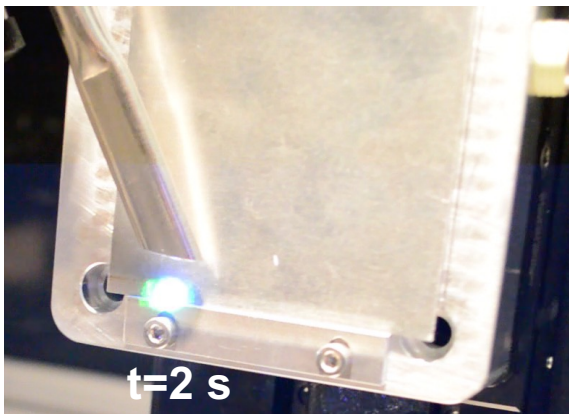
Two baseline specimens, which were provided by M&P Solutions, were considered:
 (a) Chemical Conversion Coating (CCC), MIL-DTL-5541 type I with Cr(VI), and
 (b) Sulfuric Acid Anodization (SAA).

Following laser processing, a new oxide grows: The amount of newly grown Al-oxide is larger and more uniformly distributed than that for the baseline specimen

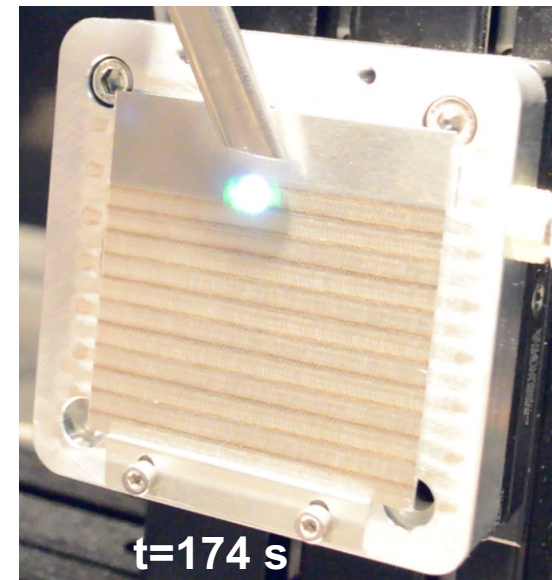
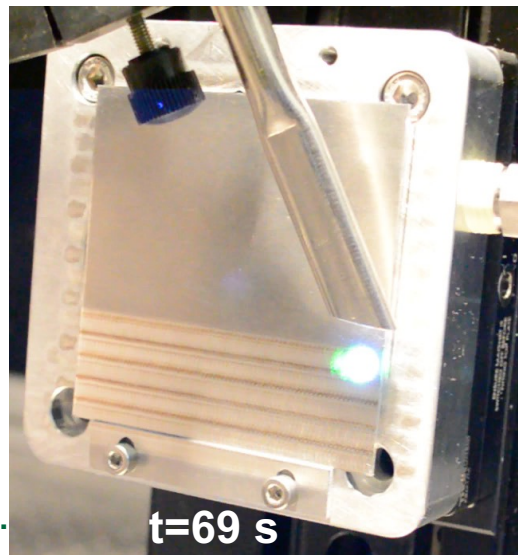


Non-chemical or very chemical-light (wiping), laser-interference surface preparation procedure for coating-only

Specimen Type	Surface Preparation	Storage	Prior to coating
Control (Baseline)	<ul style="list-style-type: none"> • Chemical Conversion Coating (CCC), MIL-DTL-5541 type I with Cr(VI), or • Sulfuric acid anodized (PAA) 	Plastic cases	Alcohol wipe
Laser-structured	Laser-interference structuring	Plastic cases	None
Laser-structured	Laser-interference structuring	Plastic cases	Acetone wipe



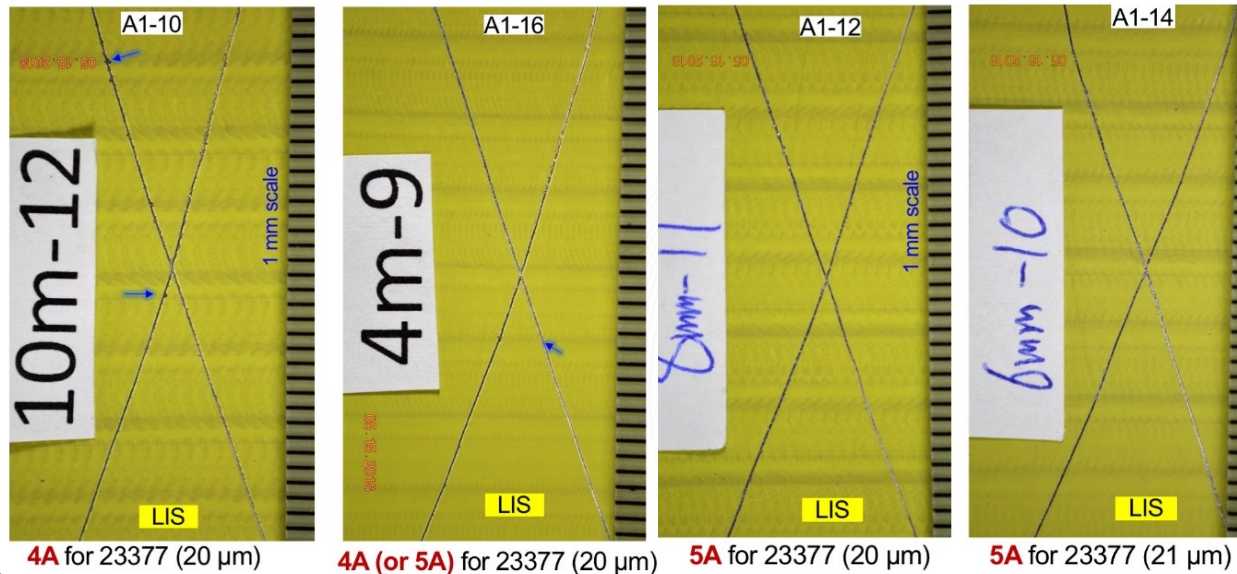
Translational stages can accommodate large specimens.



An existent paint booth with explosion-proof exhaust was setup at ORNL for spraying Air Force and Navy coatings

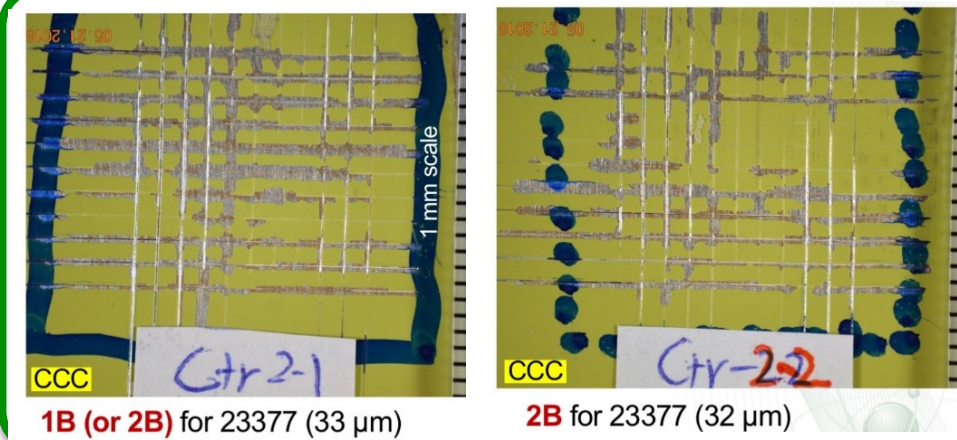


Go-no-go milestone was successfully passed



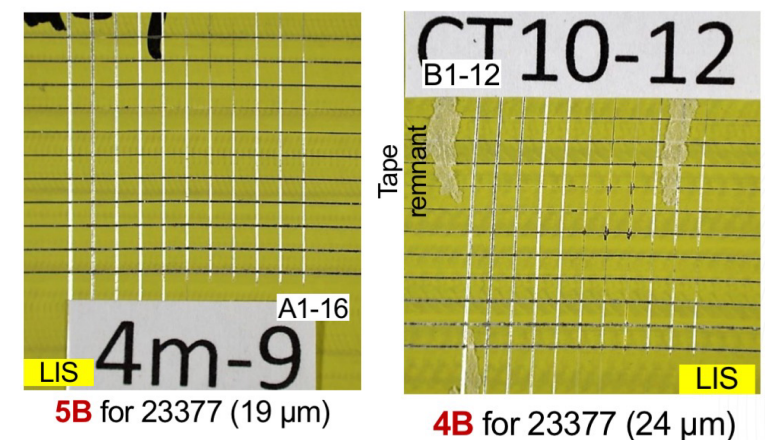
Pictures after X-cut tests of four coated **laser-interference structured** Al2024-T3 panels indicate excellent coating adhesion.

MIL-PRF-23377, Type I, Class C2 (solvent-based; contains SrCrO₄)



1B (or 2B) for 23377 (33 μm)

2B for 23377 (32 μm)



5B for 23377 (19 μm)

4B for 23377 (24 μm)

Laser processed specimens met the performance requirements in the coating adhesion specifications by having a higher or identical ranking for coating adhesion specimens than those prepared with **current state-of-the-art chemical conversion coating (CCC)** or **sulfuric acid anodizing (SAA)**.

Very good coating adhesion was obtained even after 74 days since laser structuring without any additional cleaning

ID	Laser speed [mm/s]	Open time [day]	Avg. thickness [um]	X-cut scale
A1-10	10	49	20	4A
A1-12	8	49	20	5A
A1-14	6	49	19.7	5A
A1-38	6	70	18	5A
A1-38	6	70	18	5A
A1-40	6	70	21	5A
A1-40	6	70	21	4A
A1-16	4	49	20.7	4A/5A
A1-46	4	70	20	4A
A1-46	4	70	20	5A
A1-48	4	70	17	5A
A1-48	4	70	17	5A

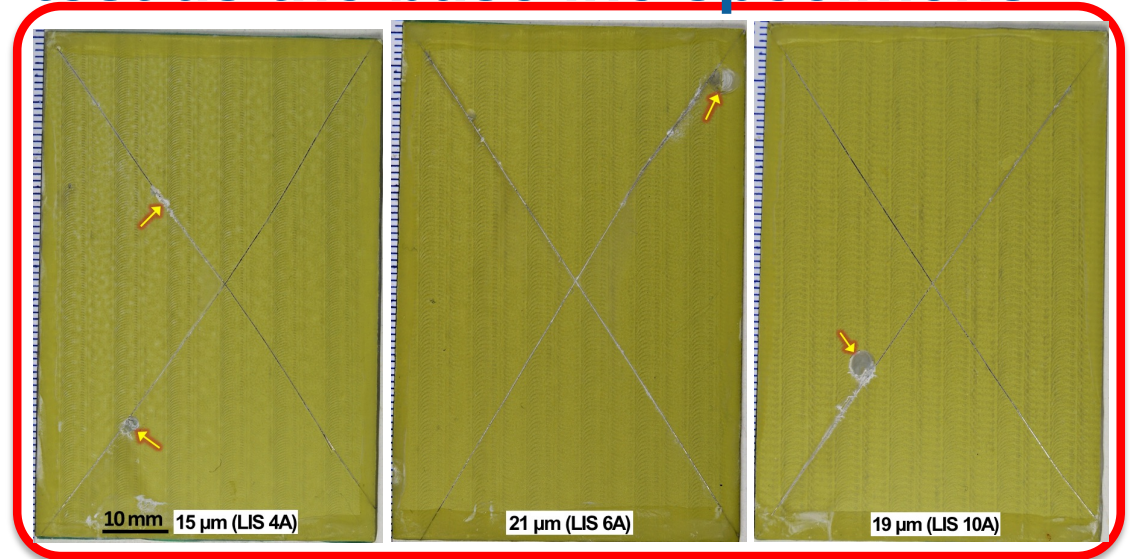
ID	Laser speed [mm/s]	Open time [day]	Avg. thickness [um]	Cross-hatch scale
CT10-7	10	74	26	4B
B1-12	10	26	23.5	4B
A1-12	8	49	20	4B
A1-14	6	49	19.7	4B
A1-39	6	70	22	3B
A1-39	6	70	22	3B
A1-39	6	70	22	2B
A1-16	4	49	20.7	5B

Surface prep	MIL-23377 primer		MIL-85582 primer	
	X-cut	Cross-hatch	X-cut	Cross-hatch
LIS 4	4.7 (5)	3.7 (3)	4.7 (6)	5 (5)
LIS 6	4.8 (5)	3 (4)	4.5 (8)	5 (3)
LIS 8	5 (1)	4 (1)	4.9 (8)	5 (4)
LIS 10	4 (1)	-	5 (4)	5 (4)
CCC	1.5 (2)	2.2 (3)	-	3.5 (4)
SAA	-	4 (2)	-	5 (4)

MIL-PRF-23377, Type I, Class C2 Max rating = 5

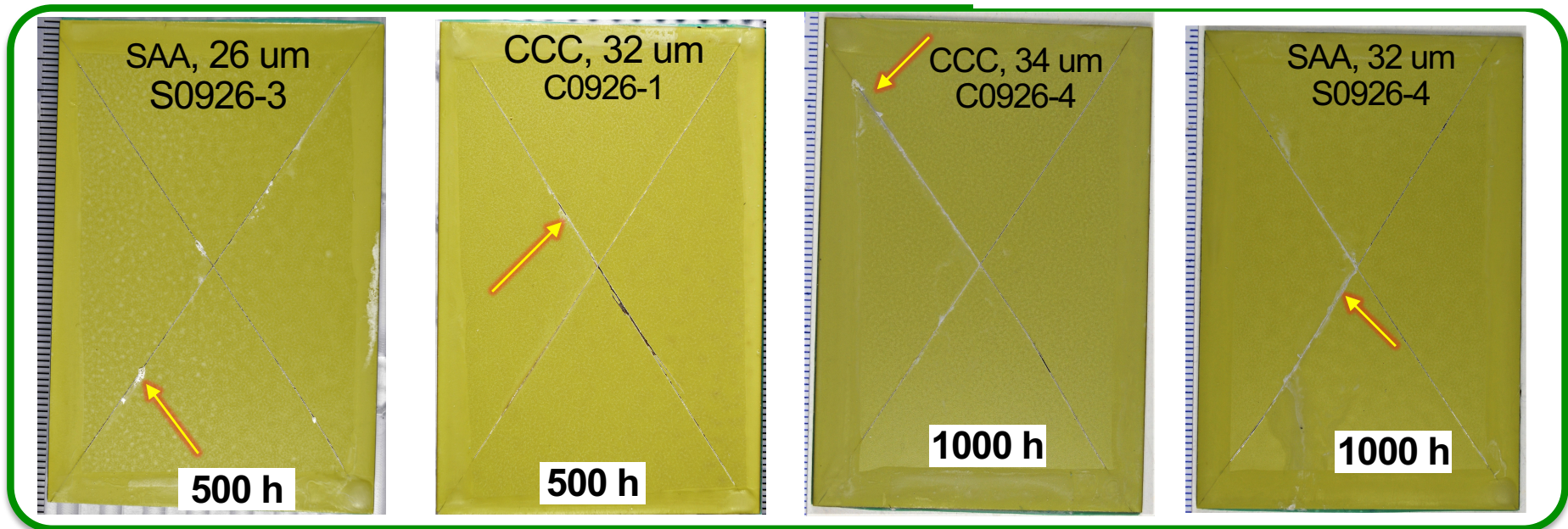
Results for Milestone 5: Attain similar corrosion damage after salt-spray ASTM B 117 test as the baseline specimens

Pictures of selected LIS test panels with the most corrosion damage after 1000 h exposure for conditions. LIS fluence $F_1 = 1.782 \text{ J/cm}^2$ with no additional wiping after LIS.



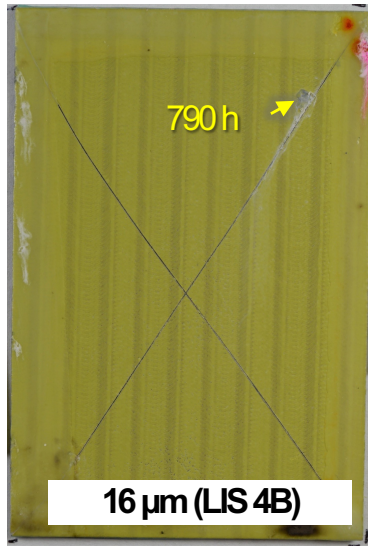
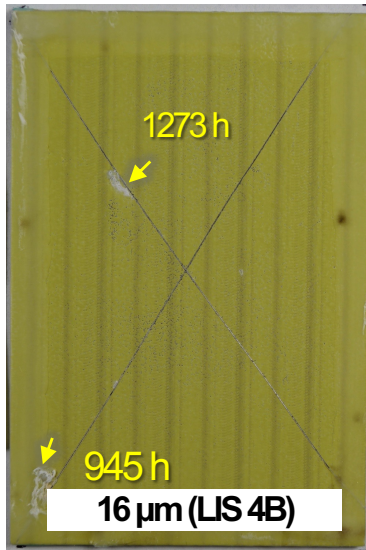
Control specimens: Pictures of coated panels with CCC and SAA pre-treatments indicating minor corrosion damage after 500 h or 1000 h exposures.

MIL-PRF-23377, Type I, Class C2



Minor corrosion damage was observed on LIS specimens coated with MIL-PRF-23377 primer

MIL-PRF-23377, Type I, Class C2



The most corrosion
 damage of LIS test panels **after 2063 h** exposure ASTM B117.

- About 50% of corrosion damage was very minor (showing less than 1 mm creepage/blister).
- Some coated panels developed more pronounced corrosion damage (with max. 5 mm creepage and/or blister).

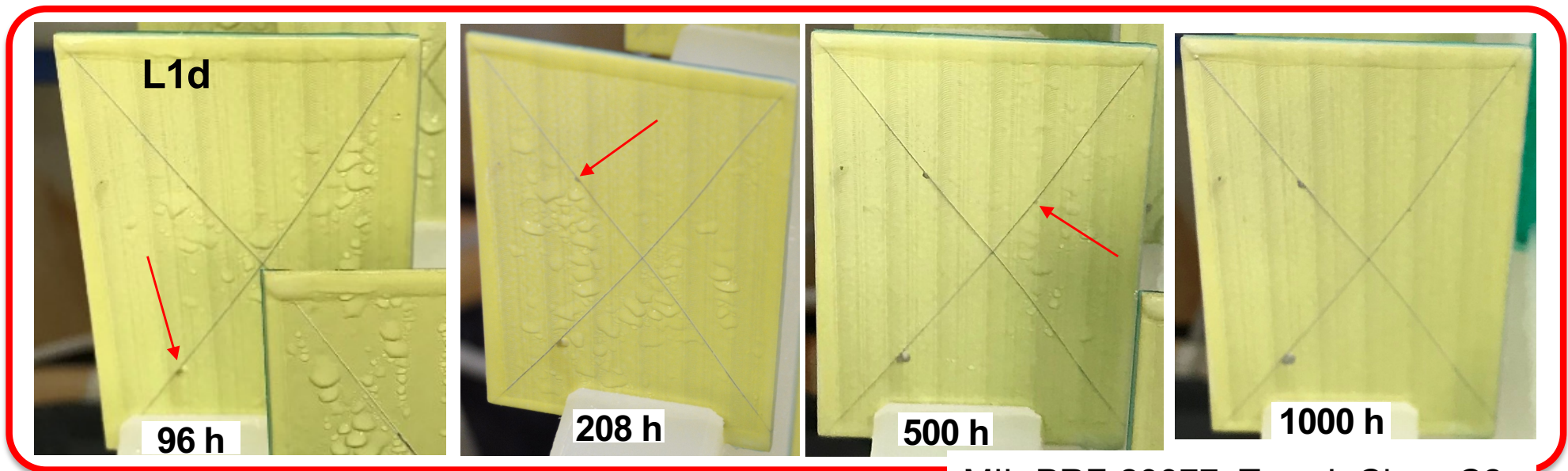


The least corrosion (none)
 damage of LIS test panels **after 2063 h** exposure ASTM B117.

Conditions: laser fluence of $F_1 = 1.238 \text{ J/cm}^2$ and acetone wiped right after LIS.

The ASTM D1654 creepage ratings (which do not consider blisters) were at least 9 (out of 10) for all primer coated panels.

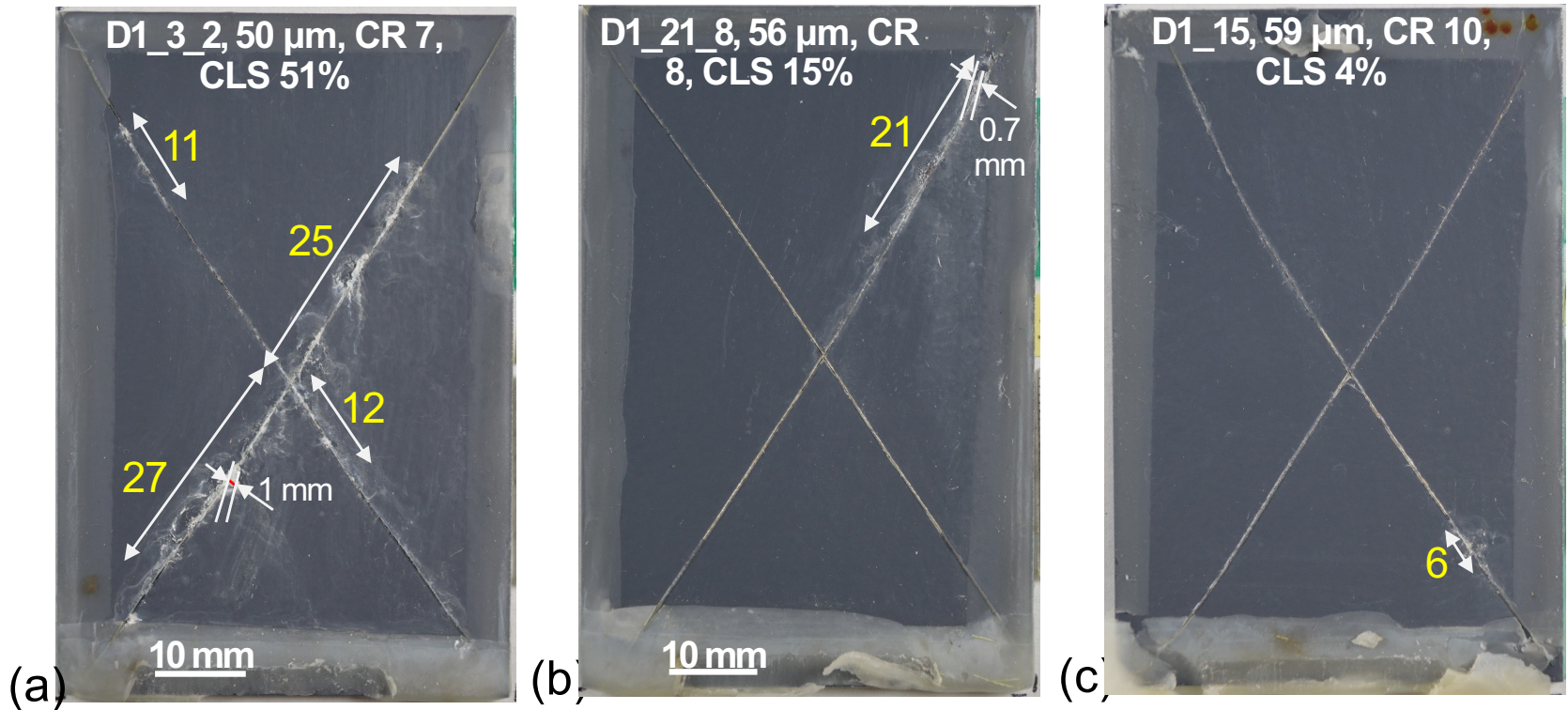
Minor, *stable*, blister corrosion damage were observed for some laser-structured specimens with 23377-I-C2 primer after salt-spray test (ASTM B117)



MIL-PRF-23377, Type I, Class C2

- Laser-structured specimens do not show corrosion damage along scribe lines,
 - Both the CCC specimens and SAA specimens showed corrosion along scribe lines at 500 h.
- Laser-structured specimens show minor blister damage;
 - Some of the blisters appear after 96h of salt spray exposure
 - However, blisters were stable, i.e., did not grow past their initial appearance

As expected, top-coated specimens shows more corrosion damage than for the primed-only specimens



Steps	Control (Baseline)		Laser-interference		
	CCC	SAA	LIS_T1	LIS_T2	LIS_T3
Chemical	chemical conversion coating	Sulfuric acid anodized	None	None	None
Laser (Fluence)	N/A	N/A	1.78 J/cm ²	1.24 J/cm ²	2.78 J/cm ²
Solvent wipe	Alcohol wipe	Alcohol wipe	Acetone wipe	Acetone wipe	None
*Storage	Yes	Yes	Yes	Yes	Yes
Prekote	None	None	Yes	Yes	Yes

Light-chemical, laser-interference surface preparation procedures for joining

Control (Baseline) joining specimens reported in the AFRL reports
(e.g., AFRL-ML-WP-TR-2002-4131)

Specimen Type	Surface preparation of baseline specimens (literature)
PAA	Phosphorous Acid Anodized (PAA)
Grit blast	<ol style="list-style-type: none"> 1. Surface cleaned with acetone and wiped with lint-free wipes 2. Surface abraded with 3M Scotch-Brite nylon pads until shiny surface 3. Surface blasted with 50mm aluminum-oxide grit 4. Surface blown with compressed nitrogen to remove as much residual grit from the surface as possible

This project

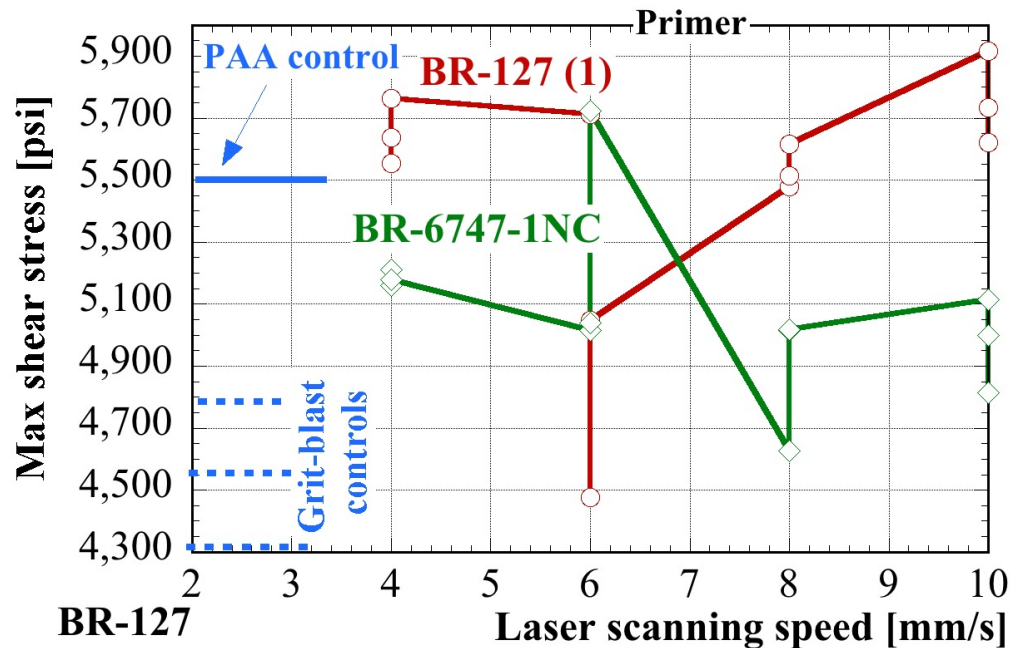
Specimen	Surface preparation at ORNL
Laser-structured	<ol style="list-style-type: none"> 1. Surface cleaned with acetone and <i>wiped</i> with lint-free wipes 2. Laser-interference structuring (LIS) 3. Surface cleaned with acetone and <i>wiped</i> with lint-free wipes (right after LIS)

Surface covered with sol-gel or bond primer after 10 to 120 min after LIS.

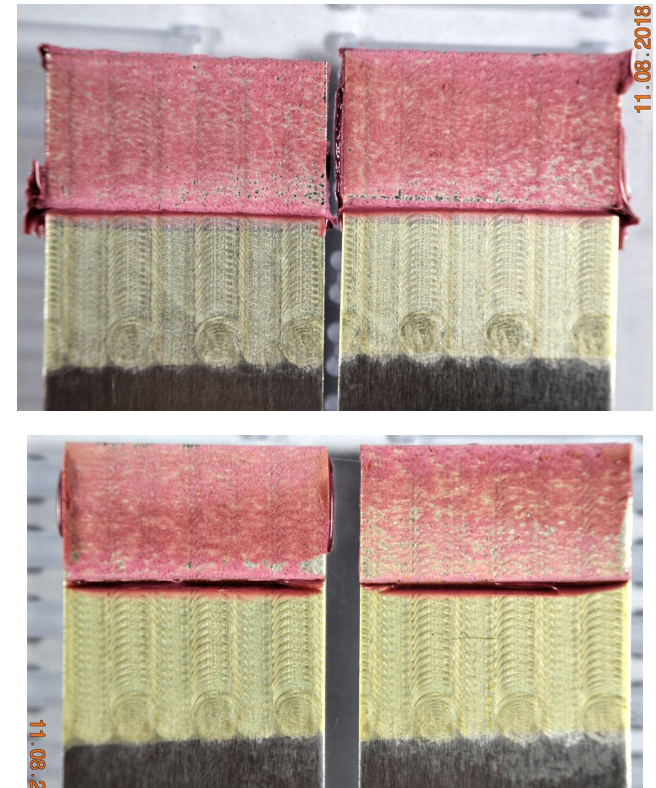
Example of timeline for joining – bonding same day for sol-gel & BR-6747-1NC

Start Time [h]	Duration [h]	Activity	Responsible
		Warm-up of BR-6747-1NC. Take out from fridge	Zach/Jay
0	1.5	Laser-structuring	Mike L.
0.75	0.75	Transport ½ lot of specimens to spray booth Acetone clean Al specimens in spray booth	Mike L. Zach/Mike S.
1	-	Mix sol-gel AC-130	Zach/Mike S.
1.5	-	Transport ½ lot of specimens to spray booth Acetone clean Al specimens in spray booth	Mike L. Zach/Mike S.
1.5	-	Brush apply sol-gel AC-130	Zach/Mike S.
2.5	0.5 to 1	Spray paint bond primer Start air-drying for 0.5 to 1 h	Zach/Mike S.
		Warm-up of film adhesive. Take out from “oF” fridge	Mike S./Jay
3-3.5	1.5	Oven cure at 250°F (+/- 10°F) or 121°C (+/- 6°C) for 1 h (extra 30 min for specimen heating)	Mike S./Jay
4-4.5	-	Transport specimens in plastic cases from oven to the joining lab	Mike L.
4-4.5	1	Adhesive bonding and vacuum bagging of bonded specimens	Dustin/Dave
5-5.5	0.5	Transport vacuum bagged of bonded specimens to autoclave	Dustin/Dave
6 -9	3	Autoclave cycle	Dustin

Results for Milestone 4: Shear-strength of single-lap of laser-interference structured (LIS) joints is larger for BR-127 joints than for the BR 6747-1NC joints



All joint failures were cohesive



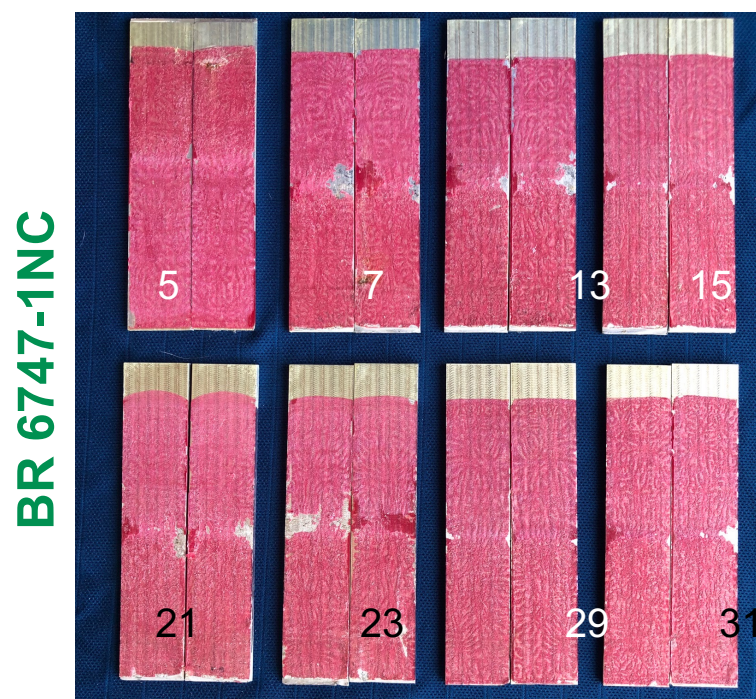
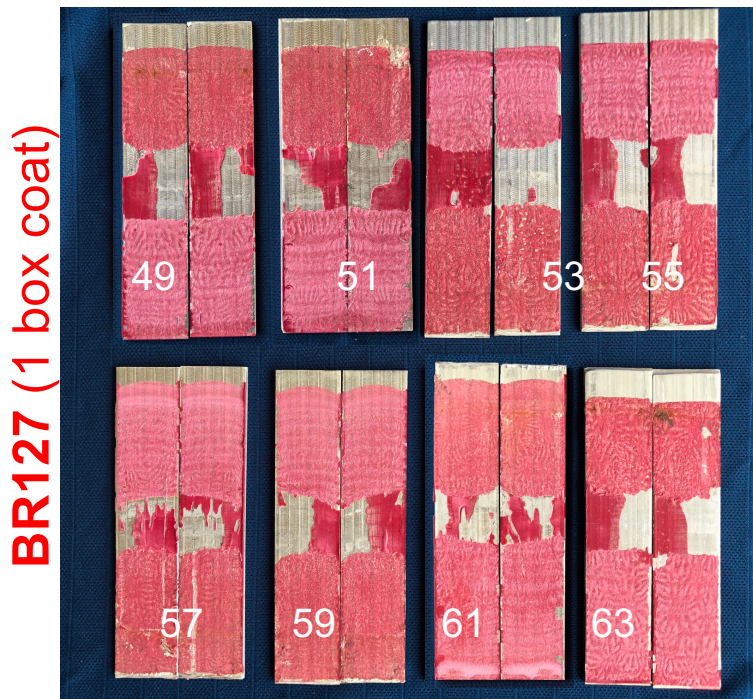
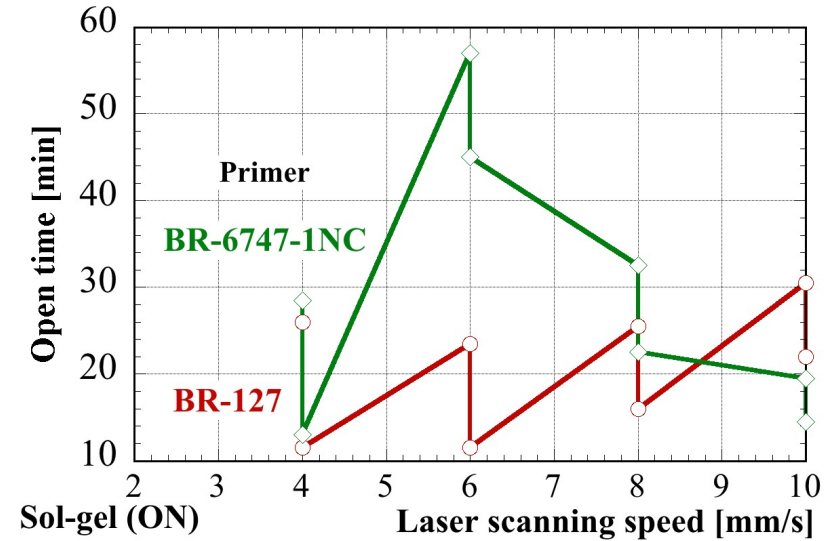
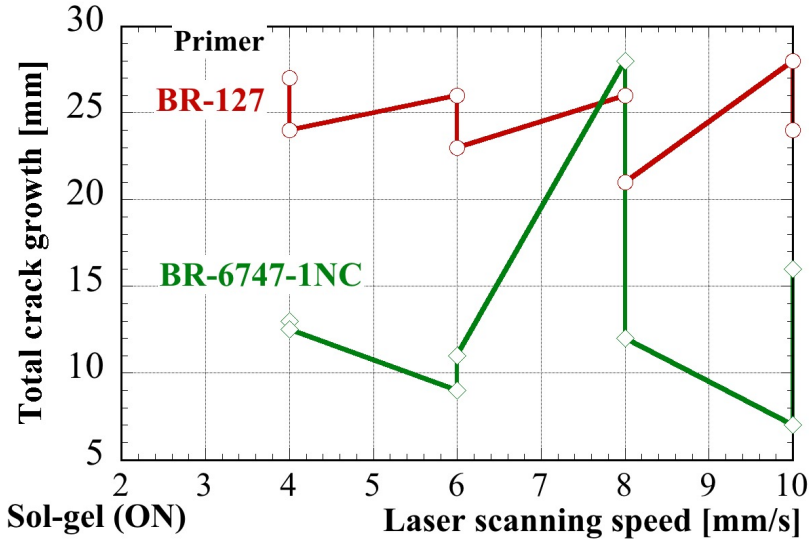
Adhesive system:

- AC-130,
- BR 6747-1NC, versus BR127 (1 box coat),
- AF163-2M

laser fluence of 1.78 J/cm²

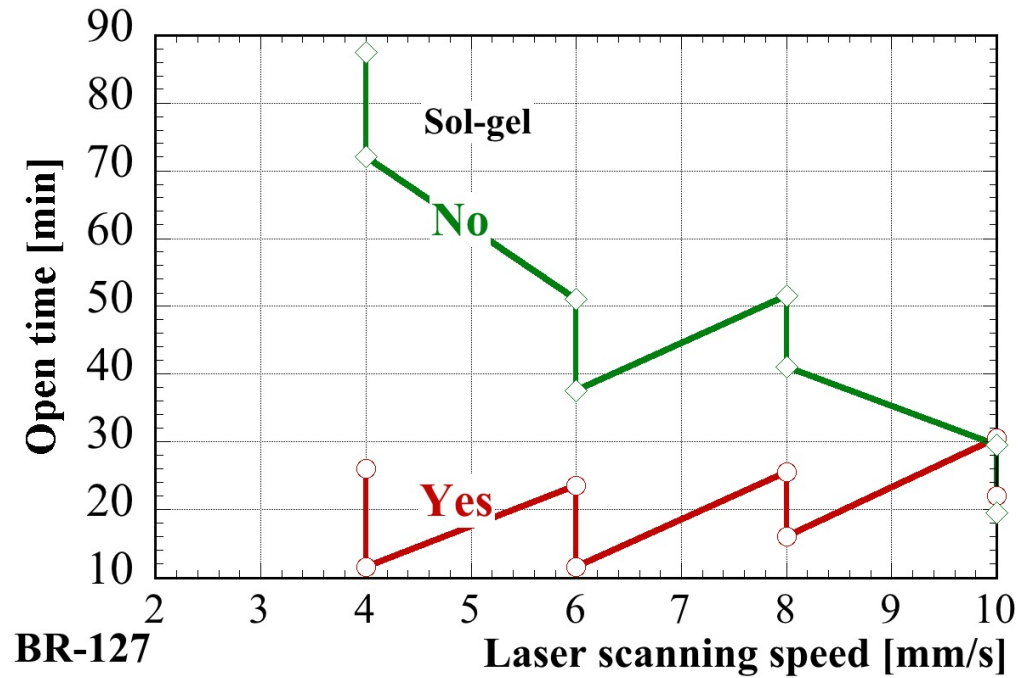
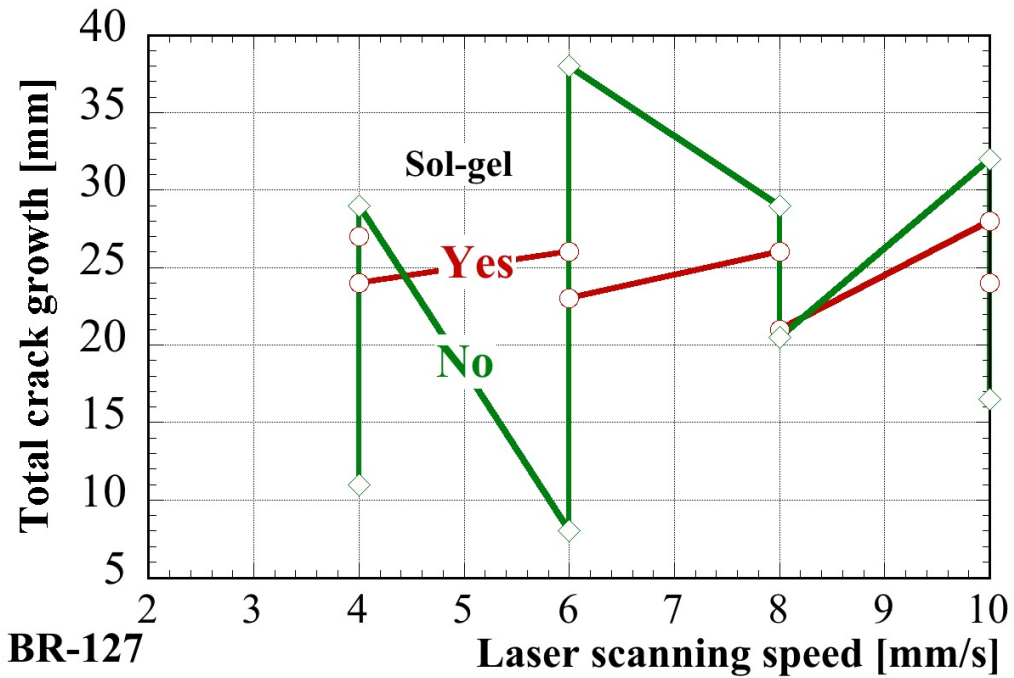
- PAA control and Grit-blast control data from 2004 Air Force report.
- Bond Primer effect on shear-strength of single-lap joints.

Results for Milestone 4 (extra): 28-day wedge-crack tests were conducted to assess the effect of bond primer



- Laser-interference structured BR 6747-1NC joints passed the wedge-crack test.
- BR-127 joints failed the wedge-crack test.

28-day wedge-crack tests were conducted to assess the effect Sol-Gel effect for BR-127



- The crack growth is more consistent with sol-gel application.
- However, both batches with and without sol-gel *failed the wedge crack tests* as the cohesive area failure was less than 95% (not shown).

Period accomplishments in the context of the project's overall technical objectives

Coating systems	Primer	Top-coat	Coating adhesion	Corrosion performance
MIL-PRF-23377, Type I, Class C2	Yes	None	Excellent	Very good
MIL-PRF-85582, Type I, Class N	Yes	None	Excellent	Poor (blisters)
MIL-PRF-23377, Type I, Class C2	Yes	Yes	-	Good
MIL-PRF-85582, Type I, Class N	Yes	Yes	-	-

joining system = sol-gel(AC-130) + bond primer + film adhesive (AF163-2M)

AF163-2M Joining systems	SrCrO ₄	Shear-lap	Wedge crack
Sol-gel + BR127	Yes	Best	Fail
BR127	Yes	ok	Fail
Sol-gel + BR 6747-1NC	No	ok	Pass
BR 6747-1NC	No	Best	Fail

Laser (1.78 J/cm²)
 benefits for Al-Al joining:
 1) Shear-lap only:
 Eliminate one step in the process and keep the chromate
 2) Eliminate chromate completely and use two-step process

PY2 effort was for a laser fluence of 1.78 J/cm²

Next Steps

- 0.1 FTE funding available for writing 2-3 journal papers
 - ◆ Surface and subsurface microstructure for raster mode
 - ◆ Joining performance
 - ◆ Surface energies
- Seek ESTCP/SERDP funding
 - ◆ Eliminate blister corrosion defects
 - ◆ Explore larger periodicities (e.g., 2, 3, 5, 9 μm)
 - ◆ Explore new more powerful lasers,
 - ◆ Develop a laser screening app for laser use evaluation

Technology Transfer Activities

Webinars

- T. Naguy and A.S. Sabau, "Laser De-Paint and Surface Preparation Mechanism Technologies" SERDP & ESTCP Webinar Series, October 19, 2017, <https://www.serdp-estcp.org/Tools-and-Training/Webinar-Series/10-19-2017>

Presentations at key conferences:

1. Adrian S. Sabau and Jiheon Jun, (invited) Coating adhesion for a nano-structuring laser-interference surface treatment, Int. Conf. on Processing & Manuf. of Advanced Mater. - Processing, Fabrication, Properties, Applications; THERMEC 2021, Virtual, May 10-14, 2021.
2. A.S. Sabau, H.M. Meyer III, D.N., Leonard, Surface Chemistry and Subsurface Microstructure after Spot-by-spot laser-interference Processing of Al 2024-T3, Presentation to the Advances in Surface Engineering II Symposium, 2020 TMS Annual Meeting & Exhibition, Febr. 23-17, 2020 San Diego, CA.
3. A.S. Sabau and J. Jun, Enhanced coating adhesion through a nano-structuring laser-interference surface treatment, Presentation to the Substrate Protection for Corrosion Prevention Symposium, Materials Science & Tech. 2019, Sept. 29-Oct. 3, Portland, OR.
4. A.S. Sabau, J. Jun, Z. Burns and M. Stephens, Corrosion Resistance of Laser-Interference Structured Aluminum alloy 2024 Coated with MIL-PRF-85582 Primer, DoD-Allied Nations Tech. Corrosion Conf.: Corr. Prevention and Control, Oklahoma City, OK, Aug. 11-15, 2019.
5. J. Jun, A.S. Sabau, Z Burns and M. Stephens, Corrosion Performance of MIL PRF-23377 Primer on Laser-Interference Structured Aluminum alloy 2024, DoD-Allied Nations Tech. Corrosion Conf.: Corr. Prevention and Control, Oklahoma City, OK, Aug. 11-15, 2019.

Technology Transfer Activities

Presentations at key conferences

6. A.S. Sabau, H.M. Meyer III, C. Daniel, Surface chemistry after spot-by-spot laser-interference processing of AA 5128 alloy, Presentation to Advances in Surface Engineering Symp., 2019 TMS Annual Meeting & Ex., March 10-14, 2019, San Antonio, Texas
7. A.S. Sabau, H.M. Meyer III, D.N. Leonard, and J. Chen, (invited) Sub-Surface Microstructure of Aluminum Alloys after Laser-Interference Structuring, Int. Conf. on Processing & Manuf. of Advanced Mater. - Processing, Fabrication, Properties, Applications; THERMEC 2018, Paris, France, July 9-13, 2018.
8. A.S. Sabau, J. Li, S. Yangping, H.M. Meyer III, J. Chen, Wetting behavior of Al 2024 alloy surfaces after spot-by-spot laser-interference processing, Presentation to the Advances in Surface Engineering Symposium, Materials Science & Technology 2018, Columbus, OH.

Key points

Laser-interference structured specimens met the performance requirements for coating adhesion and joining

- Laser-interference enables a major paradigm shift in surface preparation:
“More structuring at the same productivity”
 - ◆ This study investigated only for small periodicities (1.7 μm) with shallow structure depths
- Laser-interference structured specimens met the performance requirements for the coating adhesion
- Acetone wiping after laser treatment increases corrosion protection
- Patent granted in 2021 for the laser technology, 6 journal publications
- Al-Al joining:
 - ◆ The maximum shear-stress for 12 specimens (bond primer BR-127) exceeded that of the PAA controls; All joint failures were cohesive
 - ◆ Laser surface preparation will eliminate the chromate completely from the bond primer (Sol-gel + BR 6747-1NC)

Transition Plan

- The following interim results would be useful to an end-user right now:
 - Industry-accepted materials for coating and joining were used in the project
 - Surface characterization data: SEM/STEM, EDS maps, XPS, profilometry, surface energy
 - Coating adhesion test data
 - Corrosion test data
 - Laser-structuring parameters conditions
- Transition to final field use can be accomplished through Service, ESTCP funding, or commercialization with a private industry partner after demonstration/Validation to an end-user:
 - Air Force has started a world-wide demonstration, deploying 10-12 laser systems for corrosion removal
- Air Force, Army, and Navy were identified as user communities in this research ensuring user community “buy in” of the final product:
 - Steering committee includes subject matter experts on coating and joining application for Air Force applications
- There are NO regulatory issues associated with transitioning the technology

BACKUP SLIDES

Publications

- Provide a list of all publications, patents, awards, etc. resulting from this work.

Publications

Journal

1. A.S. Sabau, J. Li, H. Meyer III, C. Daniel, Laser-Interference Surface Preparation for Enhanced Coating Adhesion, **U.S. Patent** No. 11,065,719 07/20/2021.
2. J. Jun, A.S. Sabau, and M.S. Stephens, Corrosion Behavior of Laser Interference Structured Al 2024 with Chromate-containing Epoxy Primer Coatings, *CORROSION*, 2021, Vol. 77 (5): pp. 577-590. doi: <https://doi.org/10.5006/3717>.
3. Chen, J., Sabau, A.S., H.M. Meyer III, and D.N., Leonard, Surface and subsurface characterization of laser-interference structured Ti6Al4V, *Applied Surface Science*, Vol. 555, 149576, doi: 10.1016/j.apsusc.2021.149576, 2021.
4. A.S. Sabau, H.M. Meyer III, and D.N., Leonard, Laser-interference Pulse Number Dependence of Surface Chemistry and Sub-surface Microstructure of AA2024-T3 Alloy, *Optics and Laser Tech.*, Vol. 131, Nov. 2020, doi:10.1016/j.optlastec.2020.106457
5. A.S. Sabau, J. Jun, D. McClurg, Coating adhesion of a chromate-containing epoxy primer on Al2024-T3 surface processed by laser-interference, *Int. J. of Adhesion and Adhesives*, Vol. 102, 2020: p. 102641. doi:10.1016/j.ijadhadh.2020.102641
6. H.M. Meyer III, A.S. Sabau, and C. Daniel, Surface chemistry and composition-induced variation of laser interference-based surface treatment of Al alloys, *Applied Surface Science*, September 2019, Vol. 489, pp. 893-904.

Conference

1. A.S. Sabau, J. Jun, Z. Burns and M. Stephens, Corrosion Resistance of Laser-Interference Structured Aluminum alloy 2024 Coated with MIL-PRF-85582 Primer, DoD-Allied Nations Tech. Corrosion Conf.: Corr. Prevention and Control, Oklahoma City, OK, Aug. 11-15, 2019.
2. J. Jun, A.S. Sabau, Z Burns and M. Stephens, Corrosion Performance of MIL PRF-23377 Primer on Laser-Interference Structured Aluminum alloy 2024, DoD-Allied Nations Tech. Corrosion Conf.: Corr. Prevention and Control, Oklahoma City, OK, Aug. 11-15, 2019.

Planned Journal Publications

1. A.S. Sabau, J. Jun, Surface energies of Al 2024 alloy surfaces after spot-by-spot laser-interference, To be submitted Journal of applied physics, *March 2022*.
2. Adrian Sabau, Jianlin Li, Sheng Yangping, and Harry M. Meyer III, Surface energies of Al 2024 alloy surfaces after spot-by-spot laser-interference, To be submitted Journal of applied physics, *March 2022*.

WP-2743: Laser-Interference Surface Preparation for Enhanced Coating Adhesion and Adhesive Joining of Multi-Materials

Performers: Oak Ridge National Laboratory

Technology Focus

- Developing and transitioning new non-chemical processes for surface preparation are sought to *drastically reduce* the environmental impact, risks, and costs in manufacture/maintenance of DoD weapons systems

Research Objectives

- Research objective:** demonstrate a one-step surface preparation laser-based technique at ambient conditions to reduce and/or replace the chemical conversion coating,
- Criteria for success:** achieve 80% or better coating adhesion, shear lap strength (or other mechanical testing for joining)

Project Accomplishments

- laser-interference structured (LIS) specimens met the performance requirements for the coating adhesion
- Minor corrosion damage was observed on LIS specimens coated with MIL-PRF-23377, Type I, Class C2 primer
- The maximum shear-stress for 12 specimens (bond primer BR-127) exceeded that of the PAA controls; All joint failures were cohesive

Technology Transition

- Work closely with a steering committee of coating/joining experts for Air Force applications

