

# Selective Atmospheric Plasma Coating Removal (APCR) for Composite Substrates

WP20-1174

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<b>14. ABSTRACT</b> The technical objectives of this project are listed below: <ul style="list-style-type: none"> <li>• Material Properties Characterization of composite substrates after APCR and comparison to conventional coating removal methods to detect possible damage</li> <li>• Determination of efficient and effective operating parameters for APCR on carbon fiber substrates</li> <li>• Characterization of re-coating adhesion after the stripping of coatings using APCR</li> </ul>					
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# Project Team

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- **Diane Buhrmaster**, Principal Investigator, Air Force Research Laboratory
- **Levi Snowden**, Co-performer, Atmospheric Plasma Solutions, Inc.
- **Thaddeus Williams**, Co-performer, Air Force Research Laboratory
- **Xiao-Ying Yu**, Co-performer, Pacific Northwest National Laboratory

# Background

- Original statement of need:

SERDP WPSOON-20-C1 for “Development of Advanced Coatings”

- ◆ The ability to selectively strip or apply coatings
- ◆ Scalable, less complex application, curing, or stripping techniques or methods

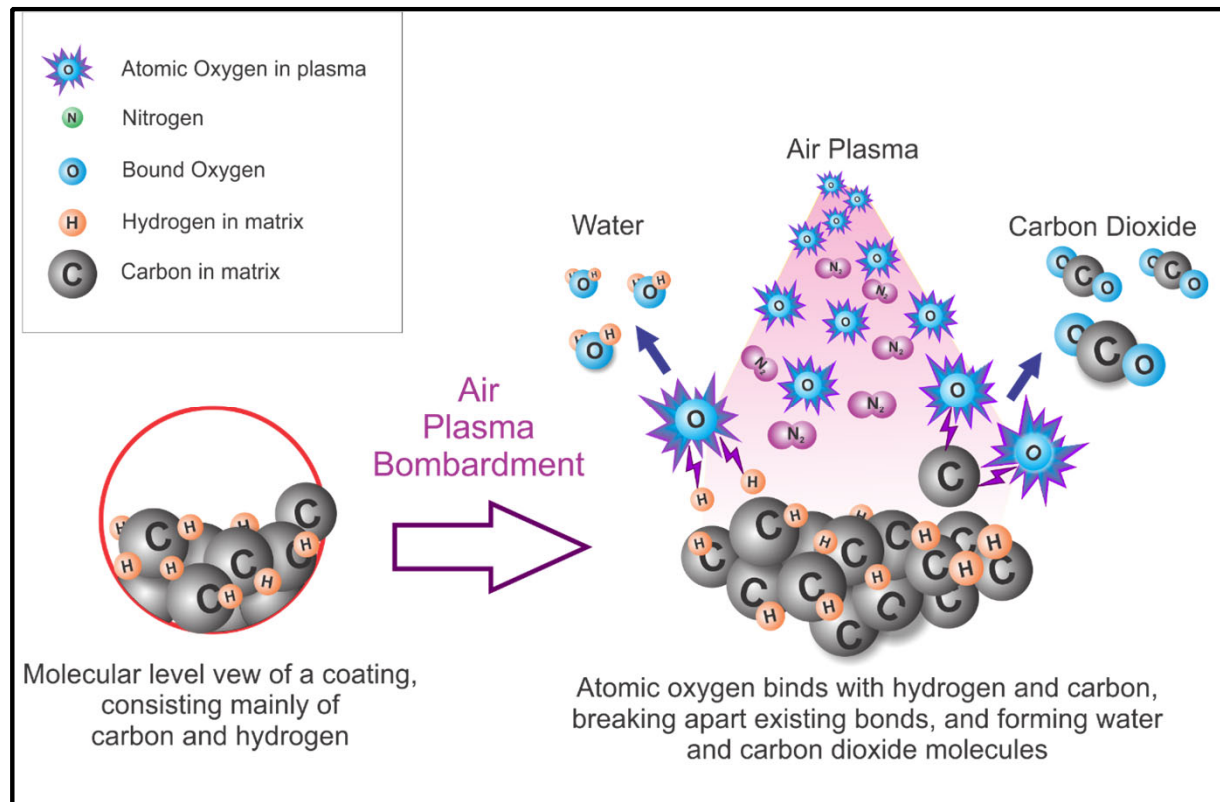
# Background

- Current coating removal methods involve toxic chemicals, excessive blasting media, labor, and energy, leading to environmental and health risks.
- Current solutions include costly waste disposal, and volatile components remain a challenge given exposure risks to personnel and civilians.



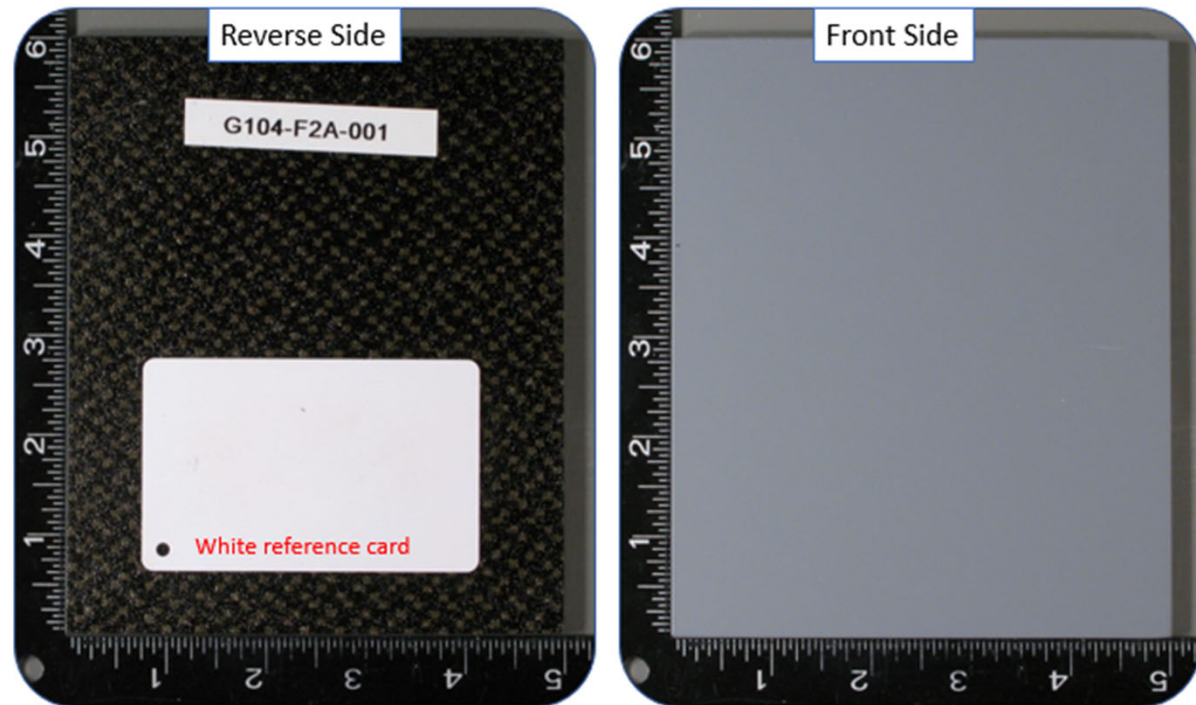
# Background

- APCR utilizes air plasma, disassociating diatomic oxygen molecules to highly reactive atomic oxygen, which bonds with coating elements, forming benign compounds like carbon dioxide and water vapor.



# Background

Carbon fiber panels used for this project

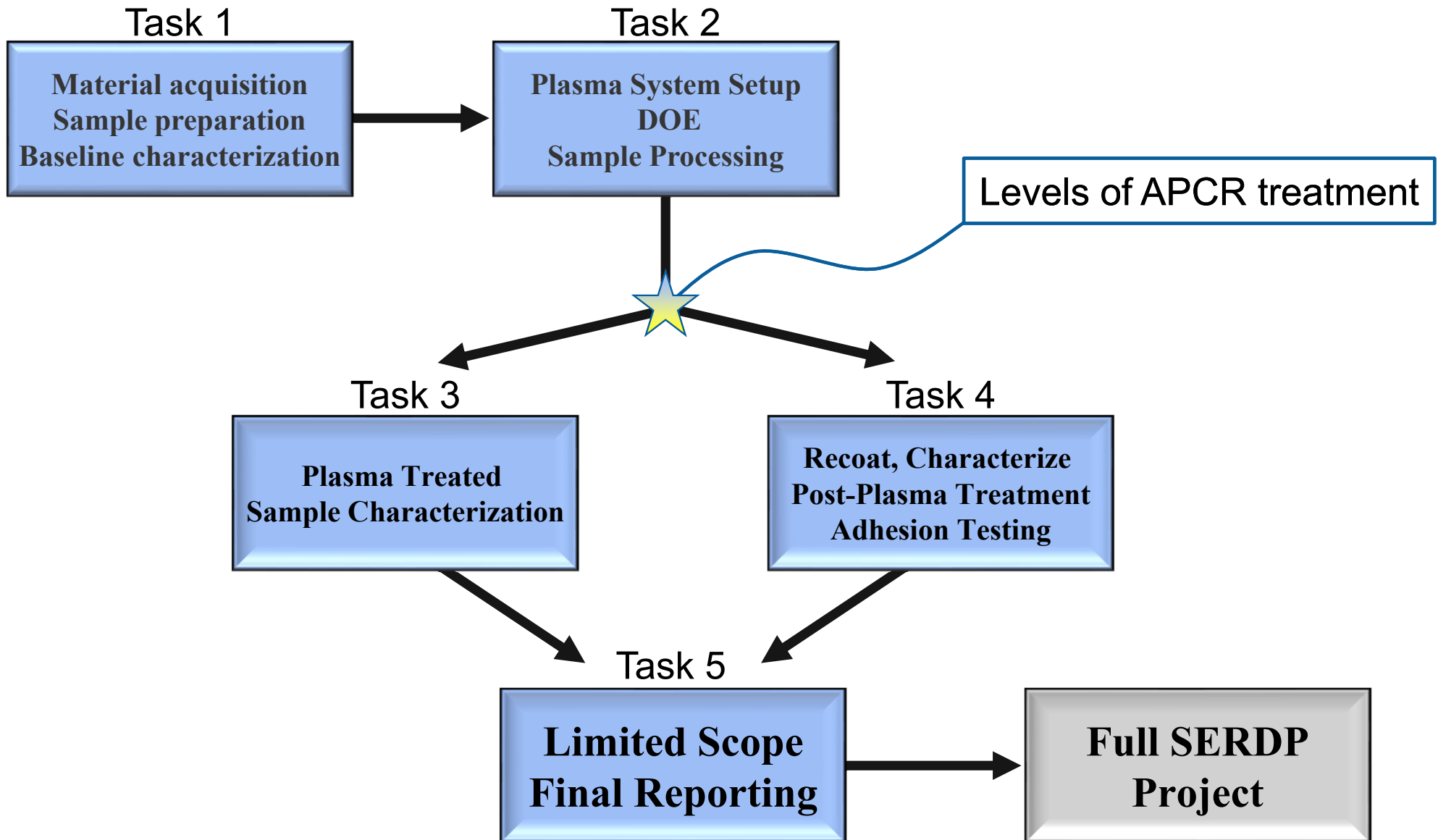


- IM7/977-3 graphite/epoxy cross-ply continuous fiber composite panels
- 8 plies in a satin fiber weave with fibers oriented in 0° and 90° directions
- 5” x 6” x 0.172”
- 0.6-0.9mils MIL-PRF-23377 Type 1, Class N primer
- 1.7-2.3mils MIL-PRF-85285 Type I, Class H topcoat

# Technical Objectives

- Material Properties Characterization of composite substrates after APCR and comparison to conventional coating removal methods to detect possible damage
- Determination of efficient and effective operating parameters for APCR on carbon fiber substrates
- Characterization of re-coating adhesion after the stripping of coatings using APCR

# Technical Approach

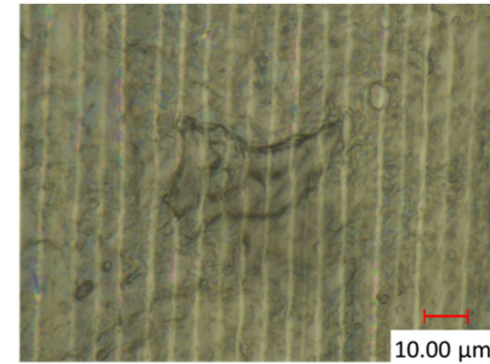


# Results

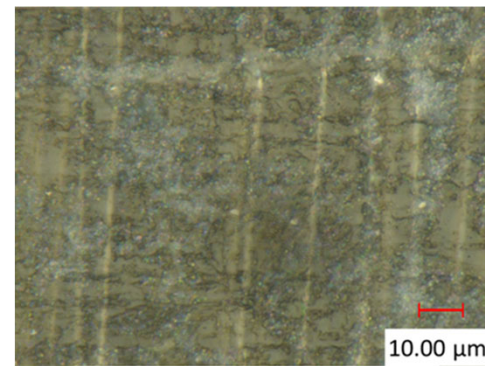
Photographs and 3D maps of the surface were taken with a digital confocal microscope.

No difference in the average peak-to-valley distance was seen between the bare control and plasma processed samples.

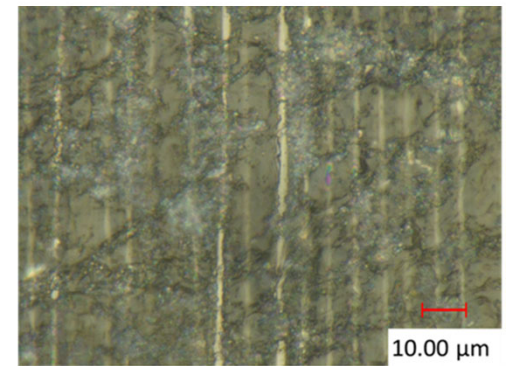
Sanded specimens showed larger peak-to-valley distance than the other specimens.



A) Bare-Control, 3000x Optical Zoom



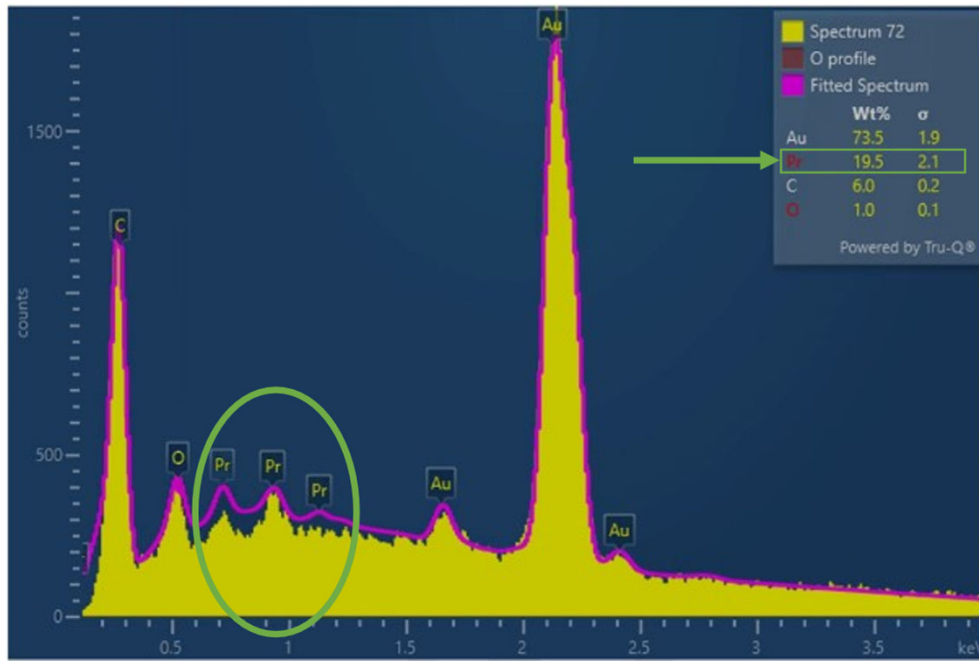
B) Plasma 2, 3000x Optical Zoom



C) Sanded, 3000x Optical Zoom

Specimen	Treatment	Average Max Peak-Valley Difference (micron)
Bare	No treatment; control	9.58
5	Sanded	14.57
9	Plasma 1	8.48
13	Plasma 2	6.19
17	Plasma 3	8.90

# Results

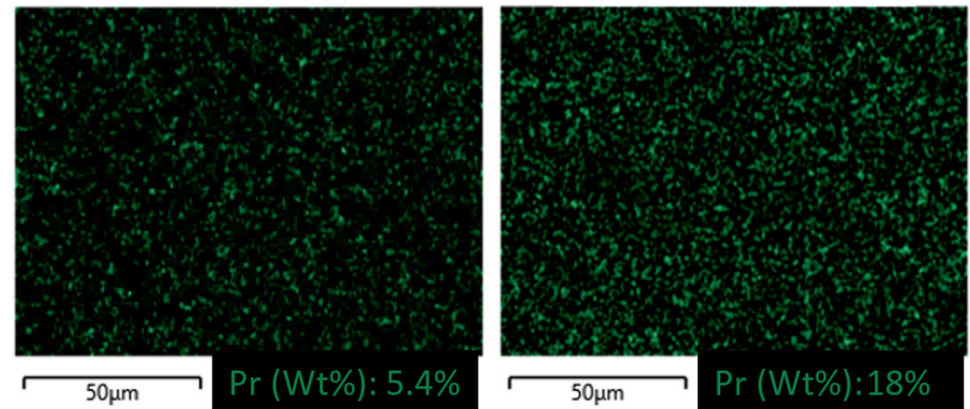


SEM/EDS scans of the surfaces indicated that both sanding and plasma coating removal can be controlled enough to leave a layer of primer.

Praseodymium (Pr) was used as a marker for remaining primer as it is characteristic of the studied primer, but not the studied topcoat or substrate.

Sanded, Pr M series

Plasma 2, Pr M series

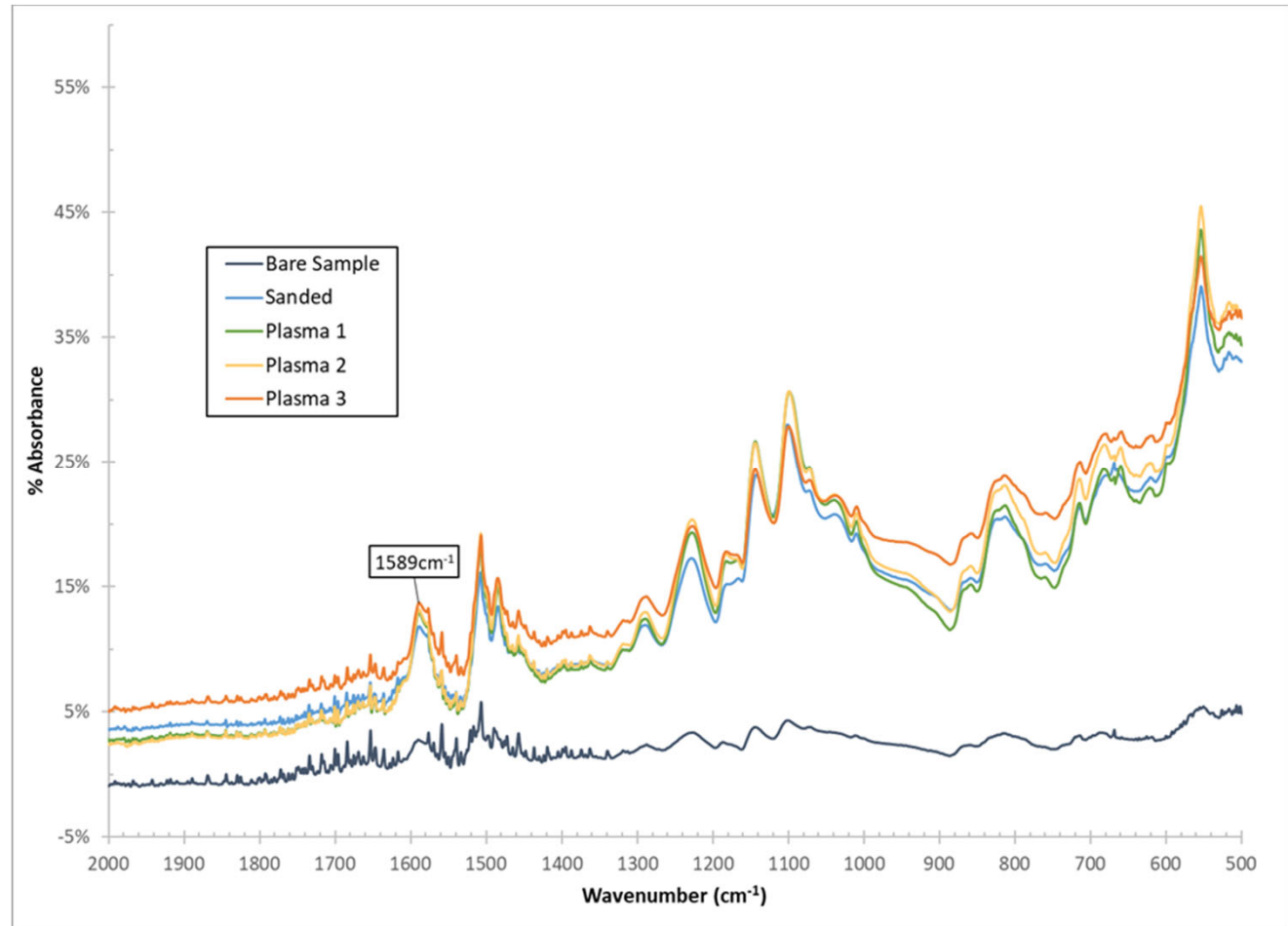


Pr present after both coating removal methods

# Results

FTIR spectra taken of the surface showed small increases in absorption in both the sanding and plasma treated specimens.

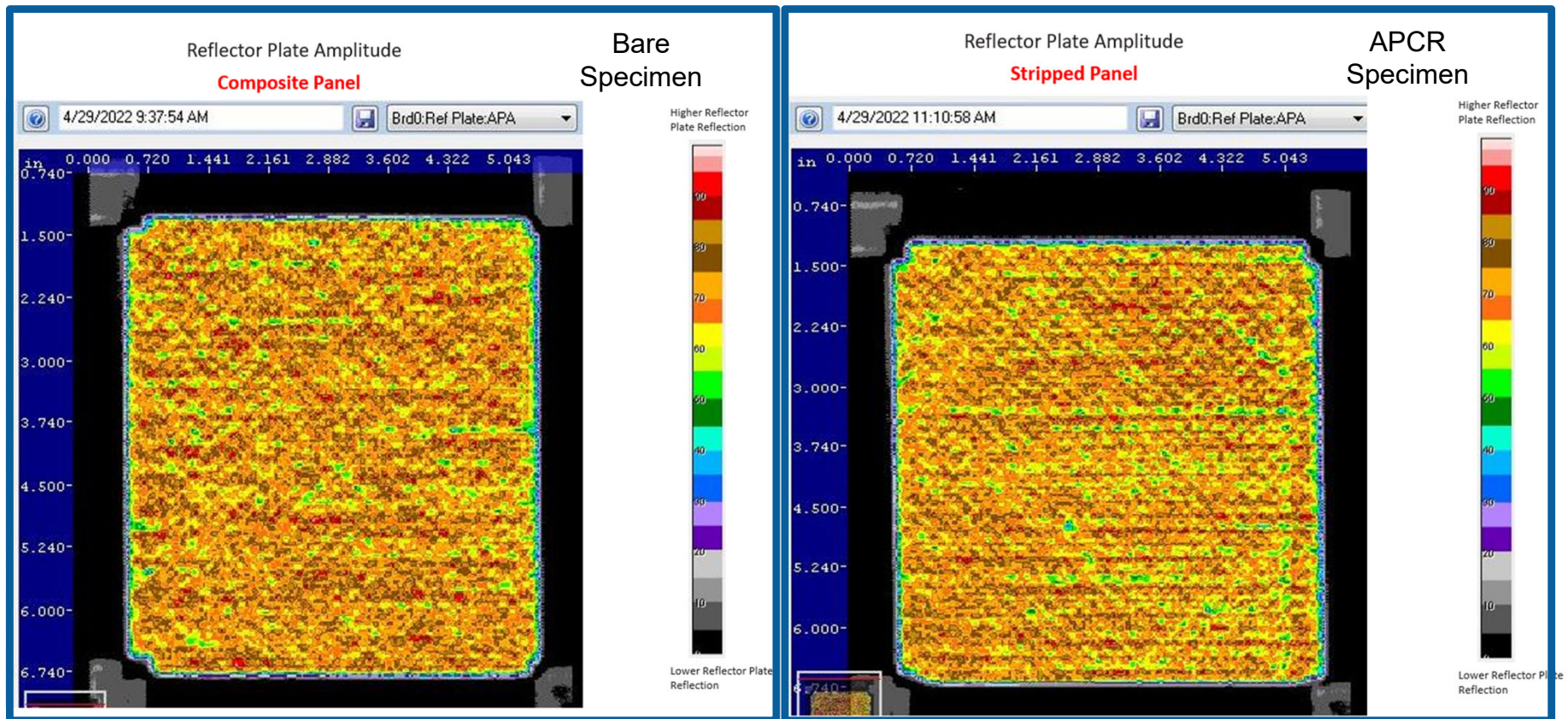
Absorption changes indicative of epoxy thermal degradation in the range of  $1600\text{cm}^{-1}$  to  $1750\text{cm}^{-1}$  was **not** present in APCR samples.



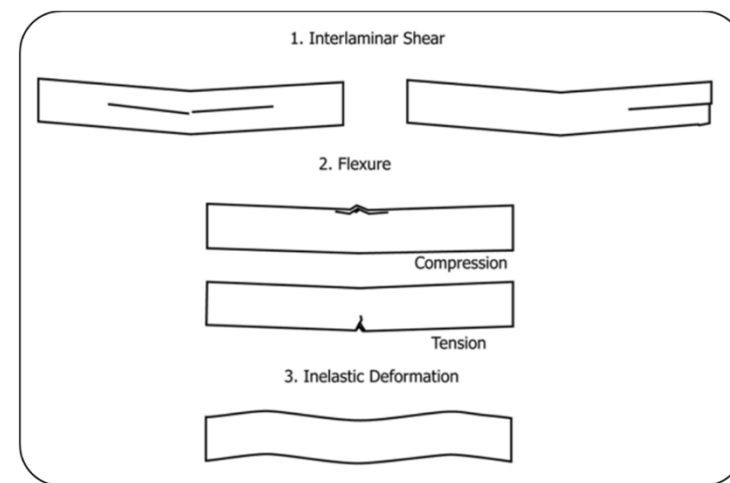
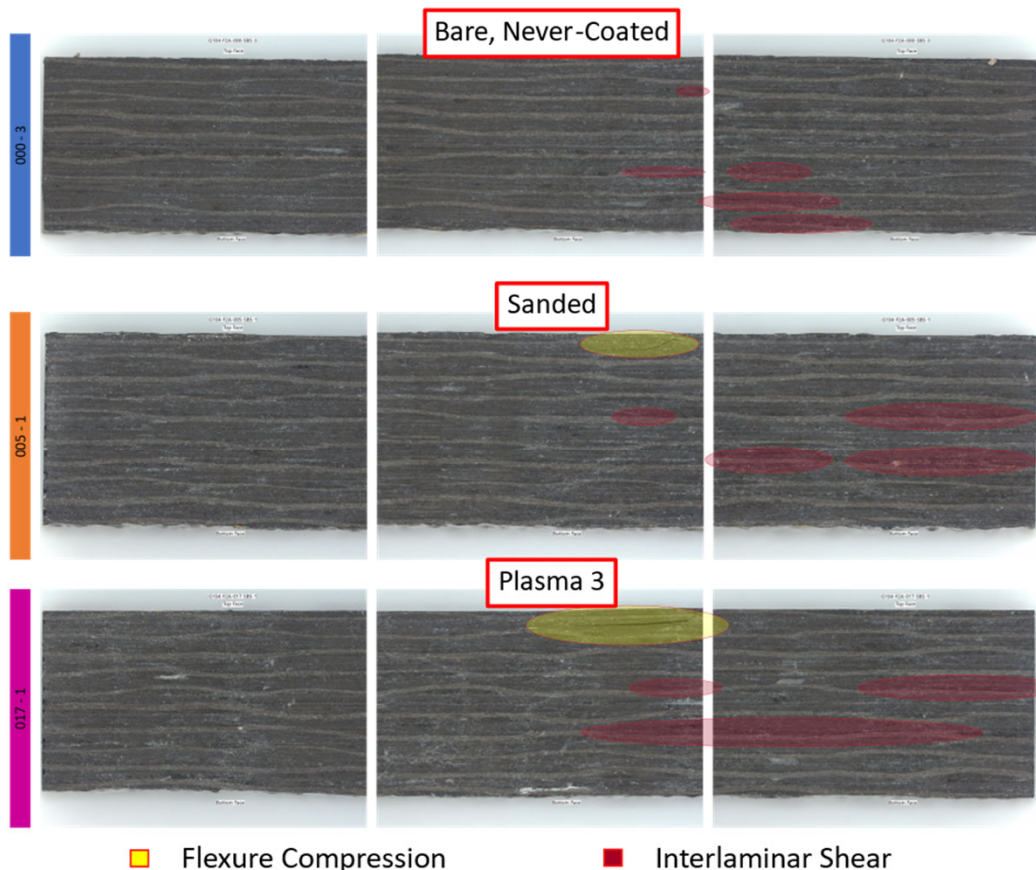
# Results

C-Scan ultrasonic imaging showed no voids present in any of the specimens.

No effect on the bulk material indicates the APCR and sanding processes were superficial.



# Results



Coating Removal Technique	Avg SBS Strength (Psi)	Std. Dev	Failure Mode
Bare	11988	189	Interlaminar Shear
Sanded	11913	148	Interlaminar Shear & Flexure Compression
Plasma 1	11731	194	Interlaminar Shear & Flexure Compression/Tension
Plasma 2	12026	598	Interlaminar Shear
Plasma 3	11821	343	Interlaminar Shear & Flexure Compression

SBS Testing resulted in the strength values not changing from the control to APCR samples.

Interlaminar shear is the predominant failure mode for SBS testing and proved to be the dominant failure mode in all the tested samples.

# Results

DSC Testing was used to measure glass transition temperature (T<sub>g</sub>) of the composite's resin to look for signs of damage.

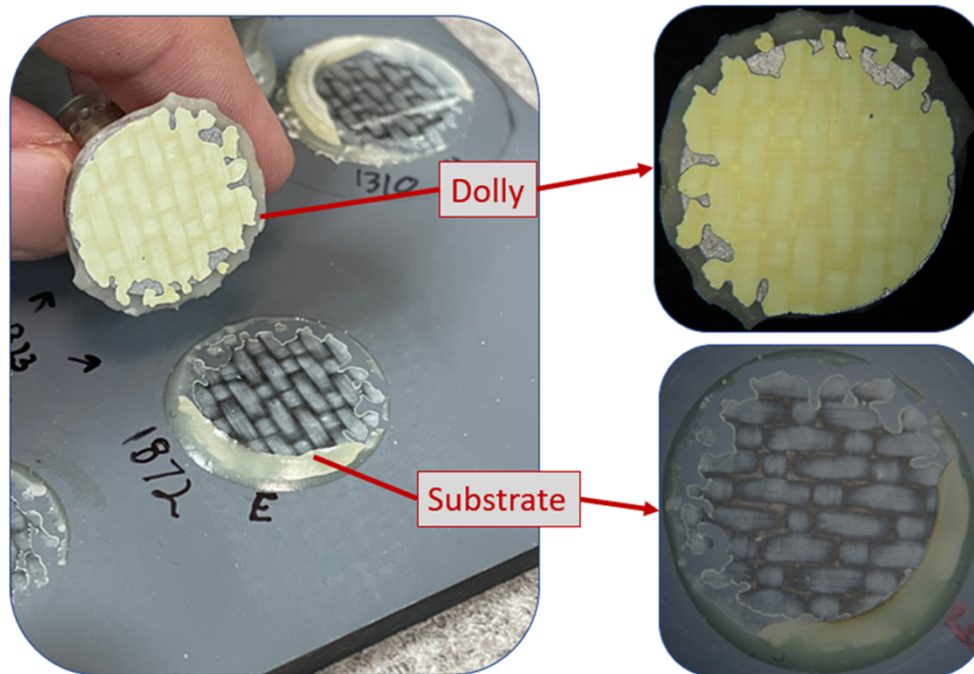
**No change was seen from the controls to APCR specimens.**

Some difficulty in detection of the transitions was experienced in the testing, indicating different specimen geometry or a different test like DMA would be better suited to study material changes.

Coating Removal Technique	Average Glass Transition [°C]		
	Onset of T <sub>g</sub>	End of T <sub>g</sub>	Midpoint of T <sub>g</sub>
Bare	207 ± 12	217 ± 3	212 ± 5
Sanded	204 ± 10	212 ± 7	208 ± 8
Plasma 1	207 ± 8	213 ± 8	210 ± 7
Plasma 2	203 ± 8	214 ± 5	209 ± 3
Plasma 3	204 ± 6	215 ± 10	209 ± 7

# Results

Dolly pull adhesion testing (ASTM D4541) indicated plasma treated specimens had a slight deficit in adhesion strength, counter to the hypothesis that plasma treatment would increase adhesion strength.

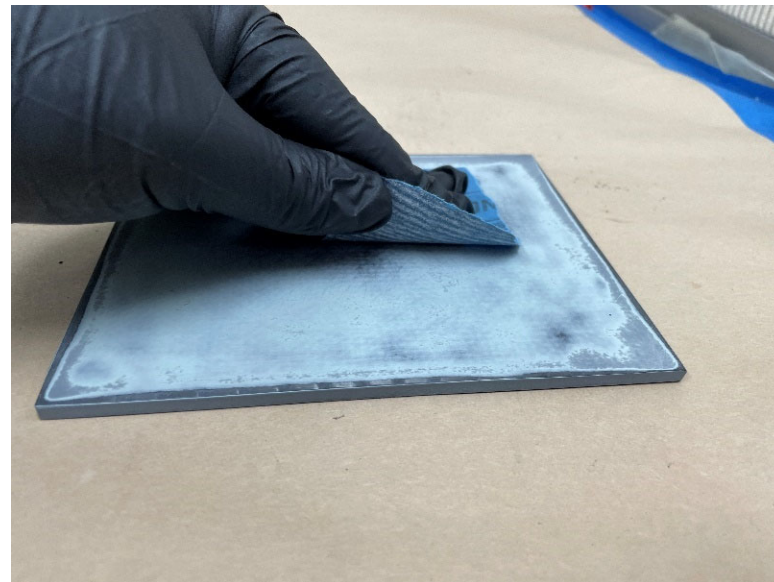
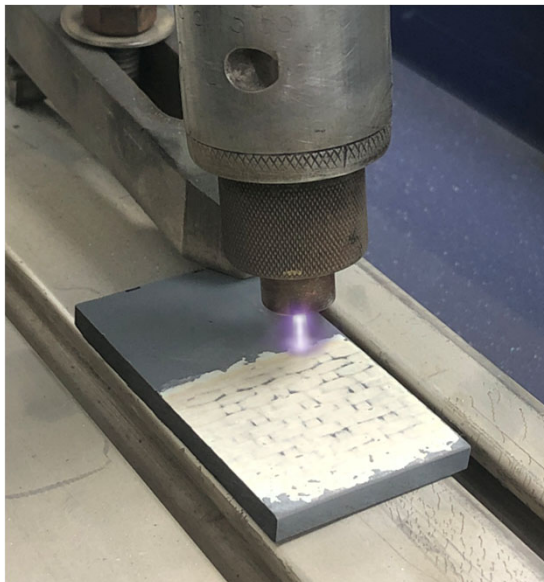


The plasma surface activation effect on primer adhesion to the composite surface is uncertain, potentially influenced by the Pre-Kote process during re-coating, an interaction with the plasma treated primer, or the MEK wipe prior to Pre-Kote.

# Results

A communication error in the recoating process led to the team performing the originally specified coating preparation process that involves solvent wiping with MEK, abrasive surface roughening and a chemical treatment prior to recoating.

These coating preparation steps modified the surface and likely removed any positive or negative effects of APCR on adhesion. Future work will remove the pre-coating process to determine the possibility of removing the solvent wipe and chemical treatment altogether, which could potentially eliminate a major source of VOC emission which is currently present in the coating process.



## Next Steps

- Test modified recoating procedure, removing any post-plasma treatment, to determine the extent of adhesion promotion after APCR.
- Perform more aggressive plasma treatments to determine appropriate process limits for APCR on composites.
- Complete further materials testing with refined and more sensitive test procedures or sample geometries such as Dynamic Mechanical Analysis (DMA) and thinner ply composite panels

# Technology Transfer

- Transition to full SERDP project
- Navy APFIT Program: Deliver and train 83 APCR systems across public & private US shipyards on steel surface ships, carriers, and subs
- Continue building relationships with Navy, Air Force, Army, Marines, and Coast Guard stakeholders who perform and manage corrosion control and asset maintenance activities
- AMPP SP21523 Standard supporting commercial acceptance integration of the process
- Northrup Grumman, Lockheed, Airbus, Spirit Aerospace, Boeing, Tier 1 Automotive Suppliers

## Key Points

- With appropriate process controls, APCR can effectively remove tough aerospace coatings on carbon fiber substrates without damaging the underlying composite
- A rapid, plasma pre-treatment, using an APCR system may be used to eliminate multiple solvent wiping steps during the surface preparation stage of the coating process (requires further verification)
- DoD new technology approval processes require significant testing and evaluation whenever encountering a new command, platform, and/or material type
- Multiple high value use cases have been identified in both commercial and DoD applications.

# Future Research

- Expand study to other composite materials and resin chemistries to determine if certain composites are more or less susceptible to damage from APCR.
- Leverage existing automation methods and equipment to support APCR on composites
- Explore using a rapid plasma surface treatment to eliminate the use of the current mandatory MEK solvent wiping, and additional liquid chemical surface conditioning steps used in the surface preparation stages of coating procedures.

# Publications

- SERDP Poster Sessions
- At least one conference presentation expected in FY 2024 to present findings
- Socialization of the Limited Scope Project with interested DoD parties

## Supporting Material

- SERDP WP-1762 “Atmospheric Plasma Depainting”
- AF SBIRs: FA8650-08-M-5606, FA8650-09-C-5607
- AF8650-15-M-5041 : Replacement of multi-step SolGel process with single step plasma treatment for Titanium adhesive bonding
- Navy SBIRs: N00014-09-M-0161, N00014-C-10-0322 (coating removal on DH36 steel)
- N68335-20-C-0384 ( Fatigue testing on HY80)
- AMPP Standard SP21523 “Non-thermal Plasma Surface Preparation of Metals”
- NAVSEA TWH Approval on HY80 on Surface ships, Carriers, and Submarines (currently limited to midship sections, full approval letter expected in Q1-2024)

# WP20-C1-1174 : Selective Atmospheric Plasma Coating Removal (APCR) for Composite Substrates

**Performers:** Atmospheric Plasma Solutions, Inc. (APS) and Air Force Research Laboratory (AFRL)

## Technology Focus

- *Atmospheric Plasma Coating Removal (APCR) of coatings on carbon fiber composites used in aircraft*

## Research Objectives

- *Validate the APCR process on composite substrates, against a traditional coating removal procedure, and demonstrate that no damage was caused to the resin matrix in the process.*
- *Characterize the reapplication of coatings to plasma treated composite substrates.*

## Project Progress and Results

- *APCR samples showed no signs of damage or change to the mechanical or chemical properties of the composite substrate*
- *Coating adhesion testing found inconclusive due to error in processing*

## Technology Transition

- *Transition to a full scope SERDP project*
- *Engage NAVAIR, Air Force, and industry stakeholders*

