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# Accelerated Corrosion Testing of Cold Spray Coatings on 304L in Chloride Environments

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PNNL: Kenneth Ross

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**SAND**  
**PNNL-SA-170262**

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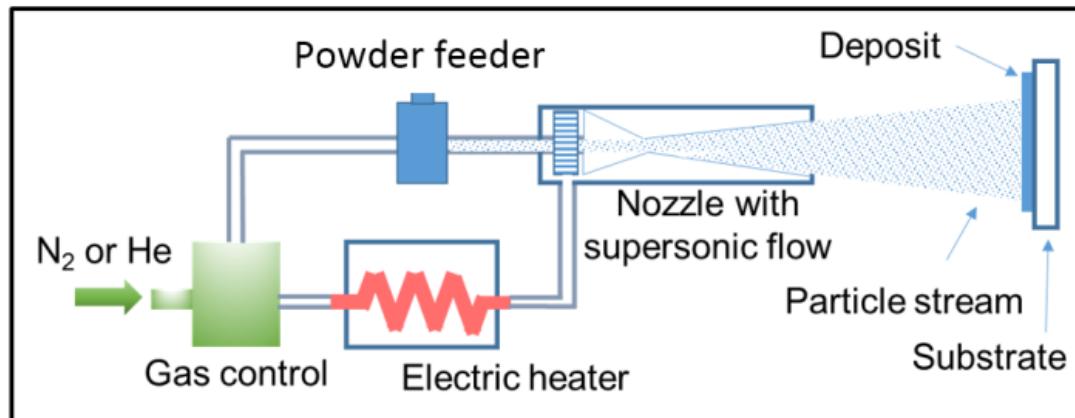


# Cold Spray Technique

## Cold Spray:

Metal particles accelerated by a stream of inert gas into a substrate.

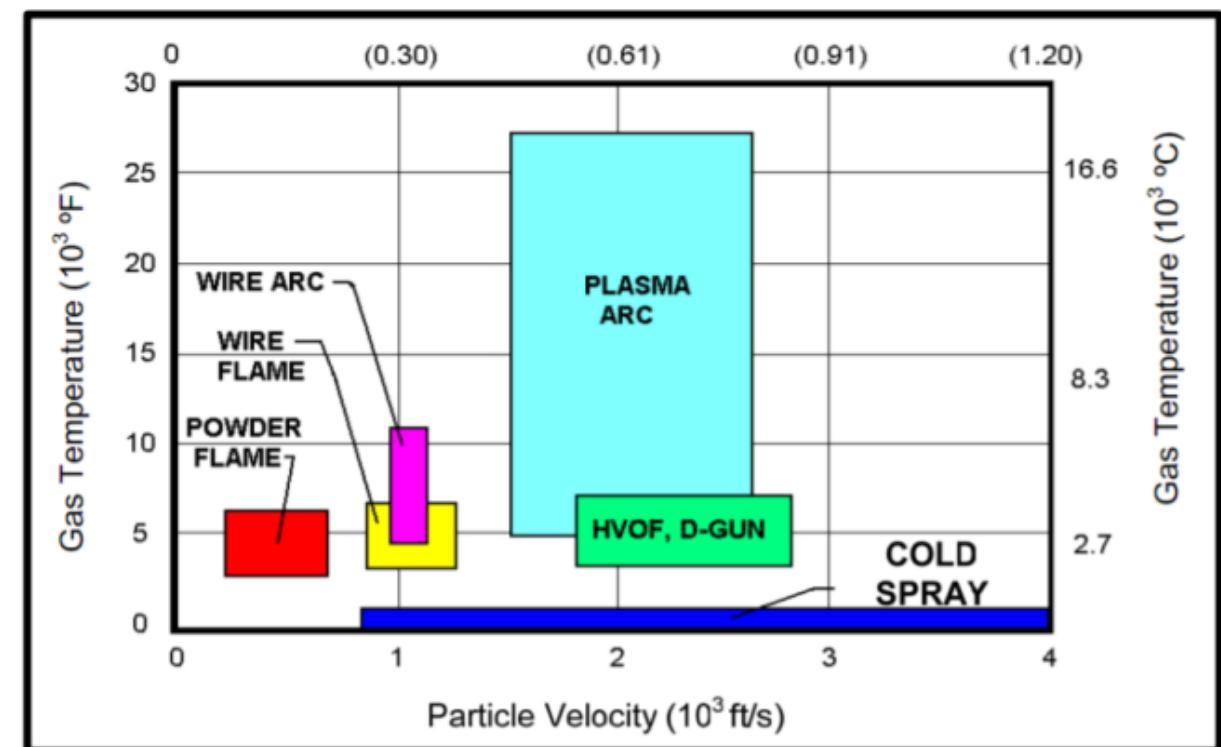
Adhered by plastic deformation (not melting like thermal spray & other additive manufacturing techniques).



High-pressure cold spray process. Image from Ken Ross

lower temperature helps prevent:

- temperature-dependent crystalline transformations
- oxidation
- vaporization



M. Neshastehriz, 2014, Pennsylvania State University

# Cold Spray Applications

## Functions:

- Additive manufacturing light weighting
- Structural repairs
- Temperature protection coating
- Corrosion prevention/repair coating

## Applications:

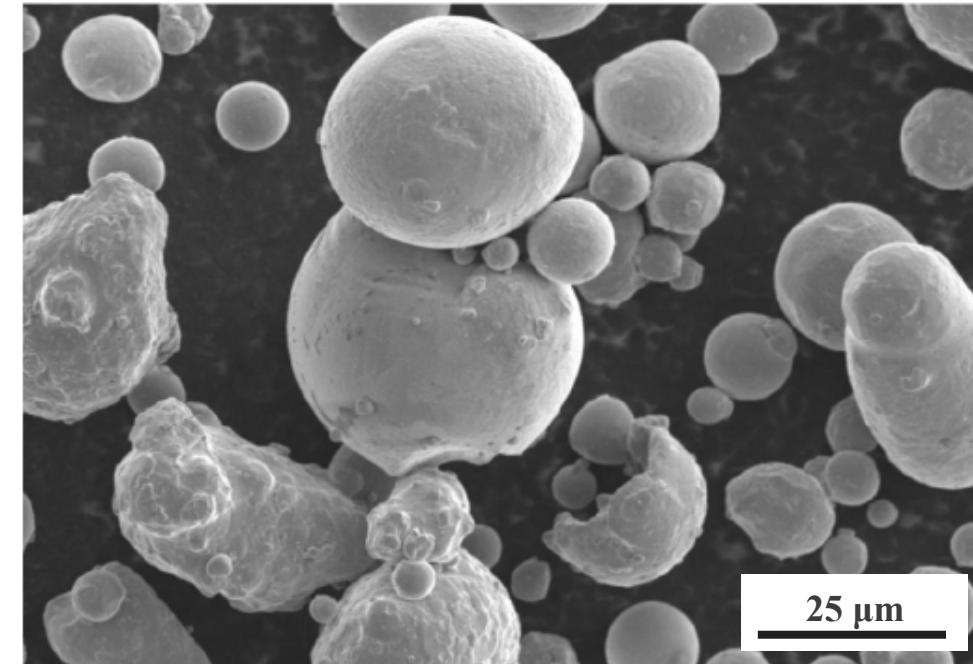
- Medical devices
- Aircraft
- Automobiles
- Nuclear reactors

## Potential Concerns:

1. Whole surface may not be accessible or able to be coated
2. Patch leaves interface between coating and substrate exposed
3. In-situ spraying presents difficulties with surface finishing

Down-selection for corrosion prevention/repair coating on 304L

Characterize cold spray (as-sprayed and polished)  
Characterize pitting stability at patch edge



316L stainless steel powder

## Coatings of Interest:

Inconel 625

Super C

Nickel

316

304

Duplex Stainless Steel

} Start with these

# Cold Spray Samples

304L stainless steel base material

Cold Spray:

- inconel 625, nickel, super C
- nitrogen, helium

Interface:

- blended, masked

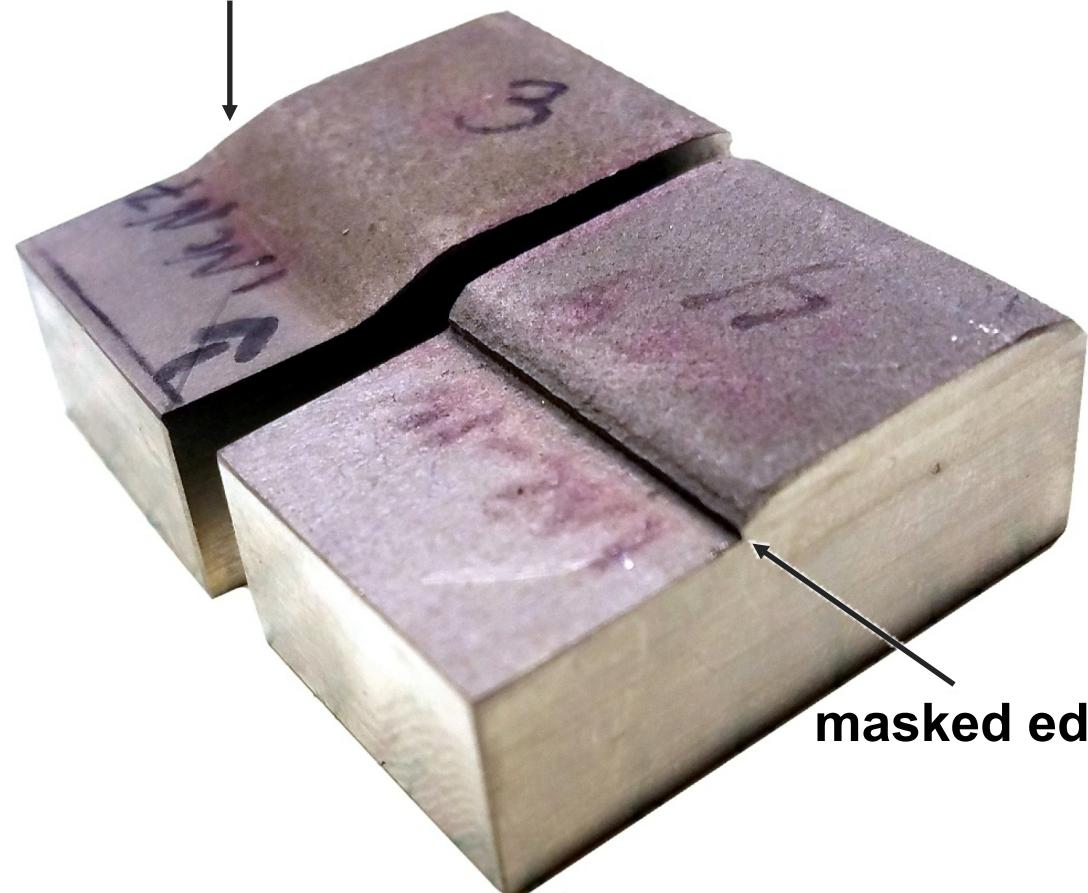
Aqueous, Accelerated Testing:

- electrochemical, ferric chloride

**blended edge**



**masked edge**



# Electrochemical Testing

Assess electrochemical & corrosion properties of the cold spray

Tested 3 spots per sample (compare to substrate)

0.6 M NaCl, platinum counter electrode, Ag/AgCl reference electrode

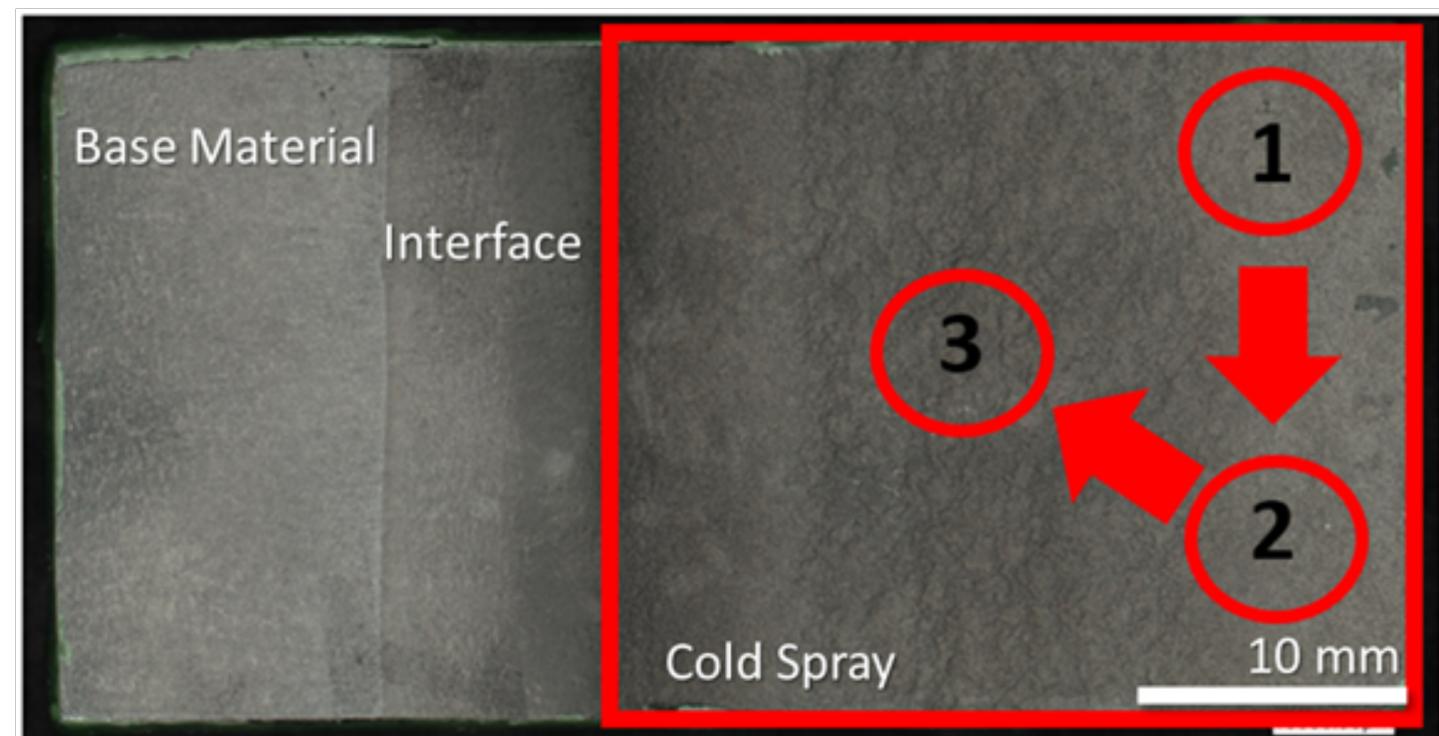
Finishes:

- As-Sprayed
- Polished (600, 1200 grit)

1 hour open circuit potential (OCP)

Potentiodynamic scan, 0.167 mV/s

- Metastable pitting
- Breakdown potential
- Passive Current Density



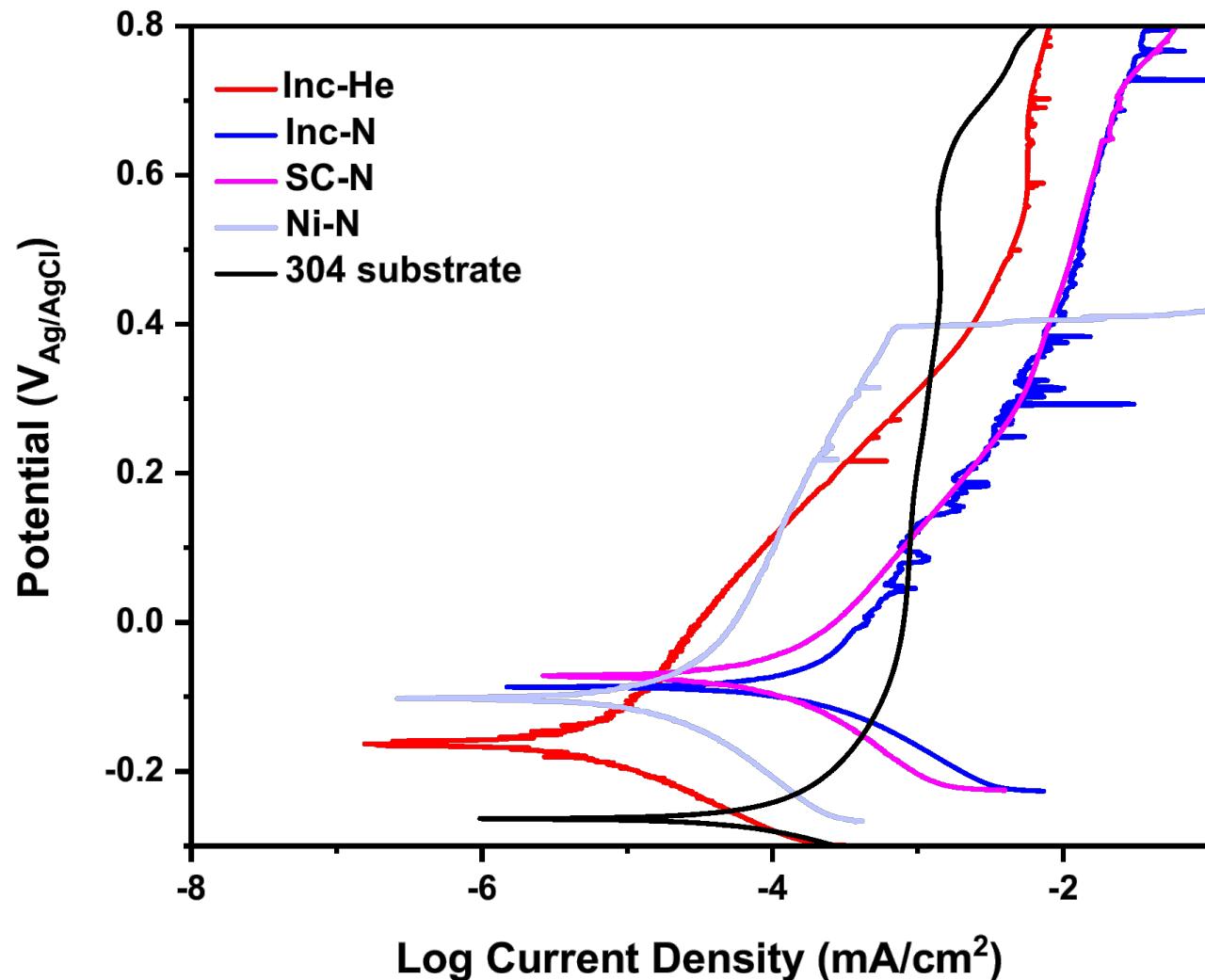
# Potentiodynamic Scans: As Sprayed

- Ni breakdown very early in scan
- Metastable pitting

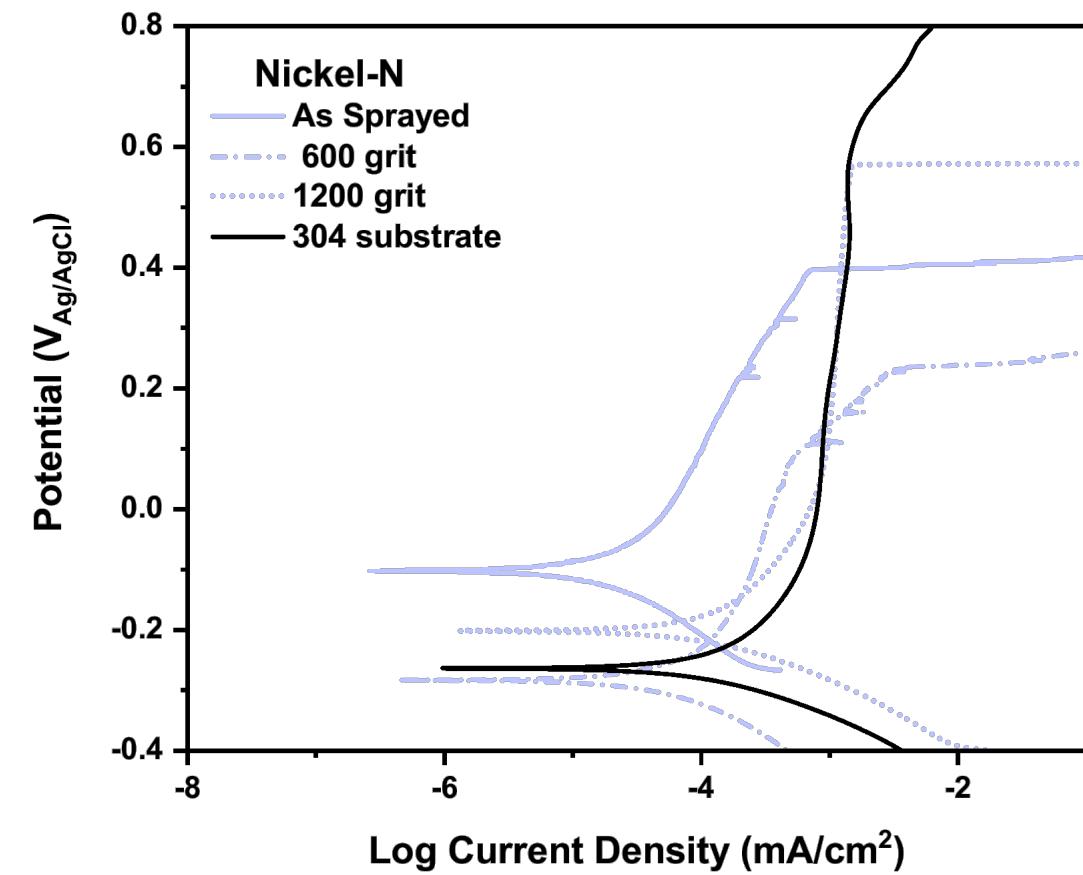
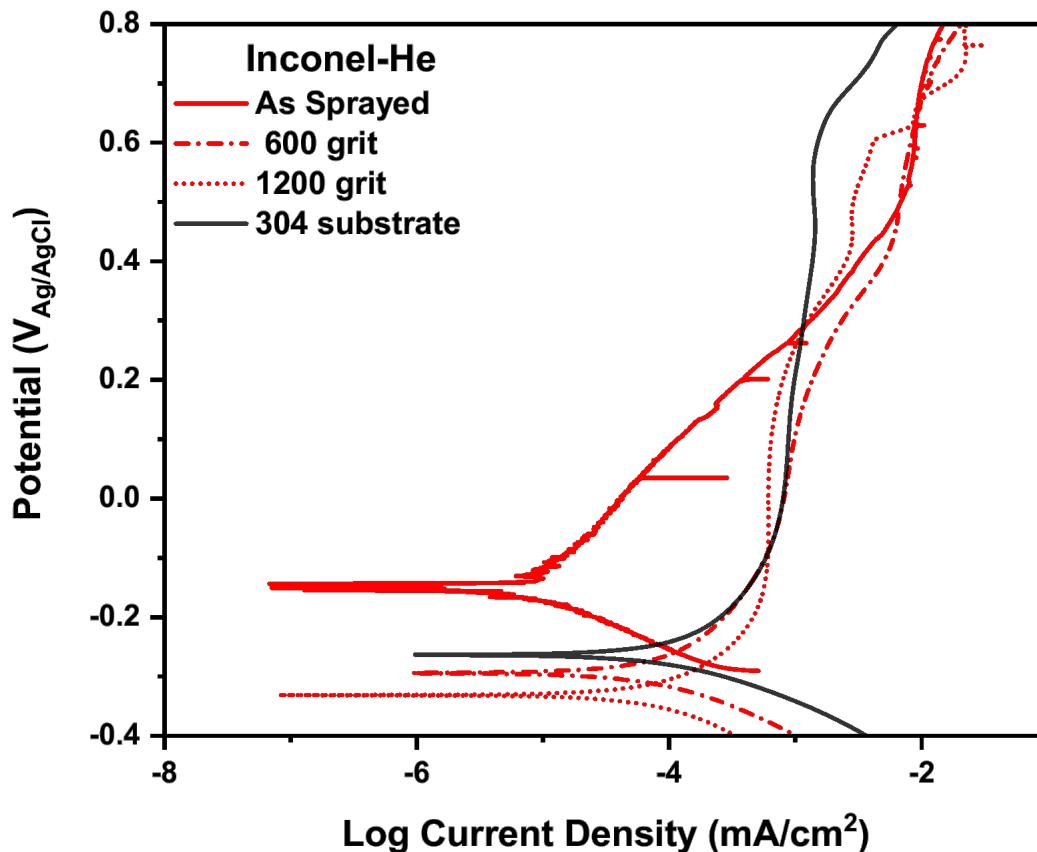
## Passive Current Density

- Lower- Ni & Inc-He
- Higher- SC & Inc-N

Sample	Porosity (%)	$S_a$ ( $\mu\text{m}$ )
SC-N	$5.51 \pm 0.44$	$16.7 \pm 0.5$
Inc-He	$1.21 \pm 0.20$	$15.7 \pm 0.5$
Inc-N	$5.79 \pm 0.18$	$17.2 \pm 0.6$
Ni-N	$3.78 \pm 0.59$	$18.5 \pm 0.6$



# Effects of Polishing



- OCP changes due to polishing (expected)
- Nickel breakdown very early in scan
- Polishing reduces metastable pitting
- Polishing removed native oxide, higher passive current density observed initially

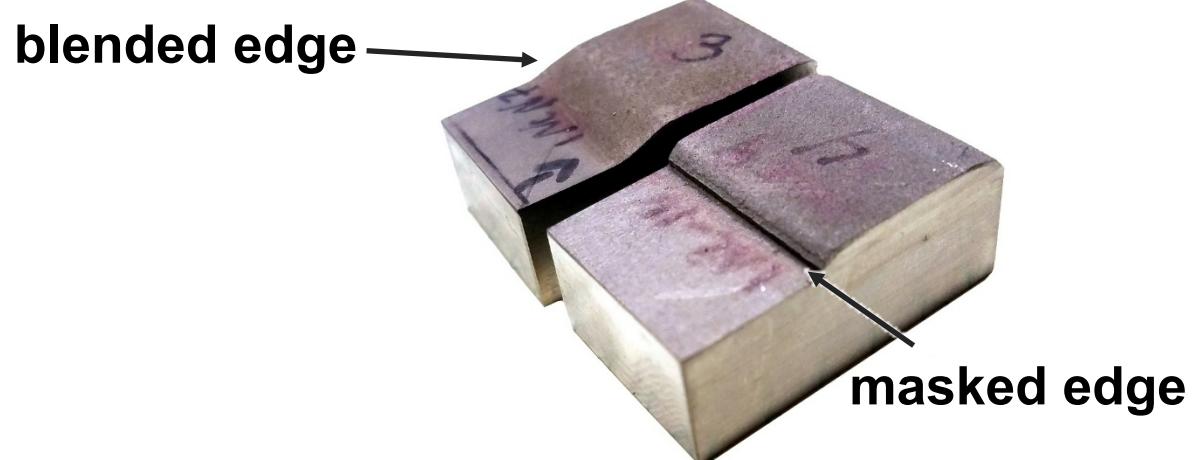
# Ferric Chloride Testing

Samples were coated in Peelseal Green Maskant for full immersion

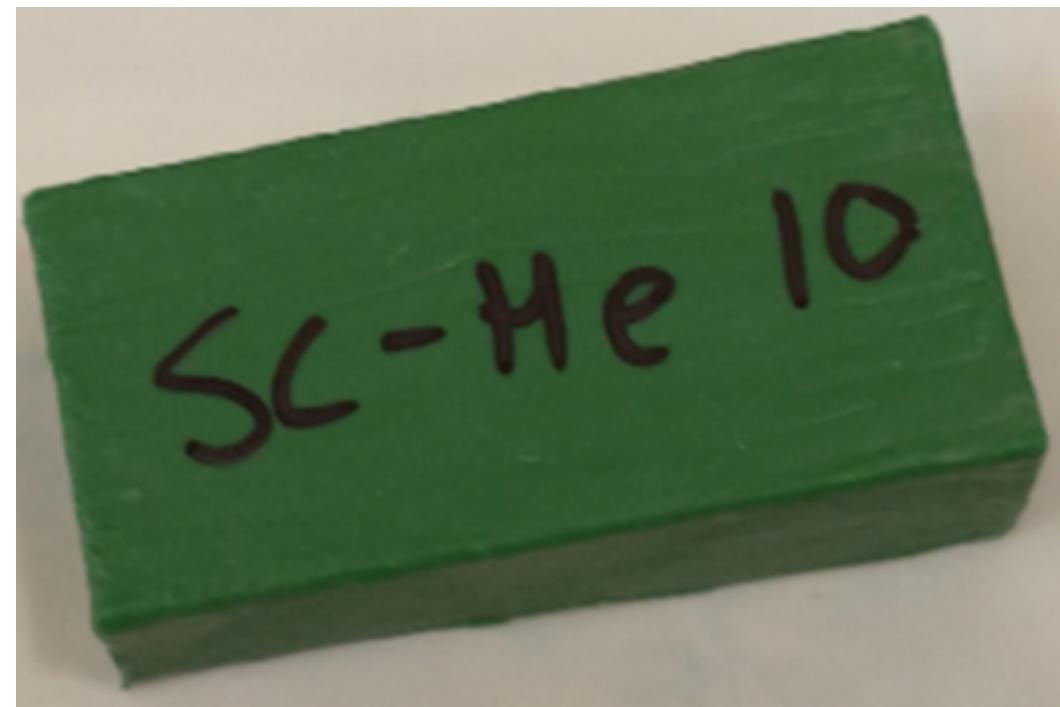
ASTM G48 Method A

6% by mass ferric chloride solution

72 hours at 22 °C



- Accelerated pitting test
- Evaluate potential galvanic couple influences
- Evaluate interface-type impact



# Material Selection

SC-N blended

Interface

Base Material

Cold Spray

10 mm

Inc-N blended

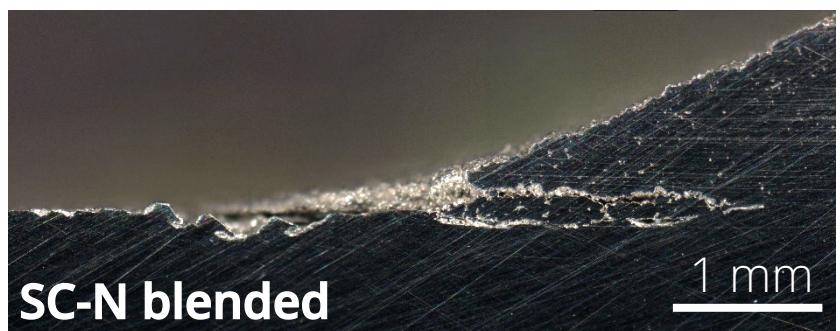
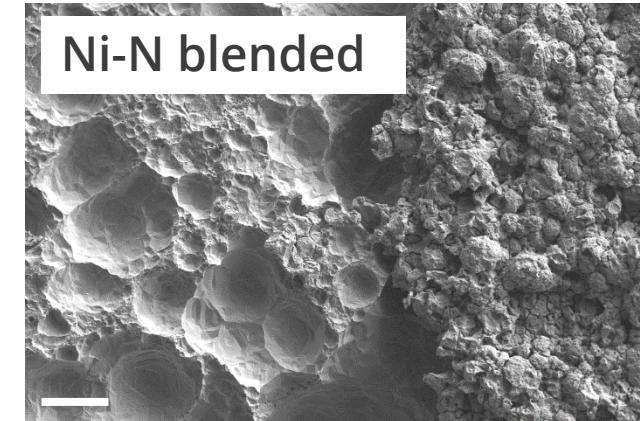
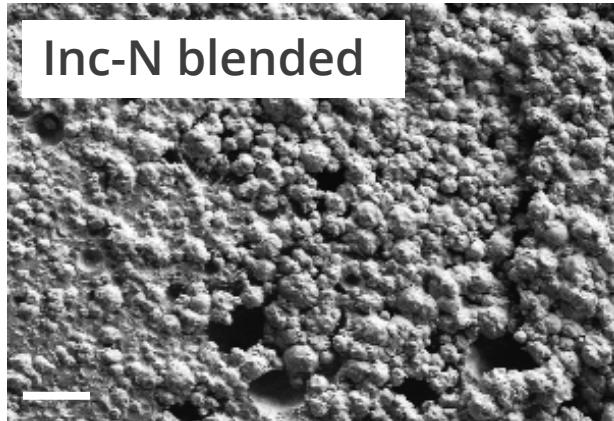
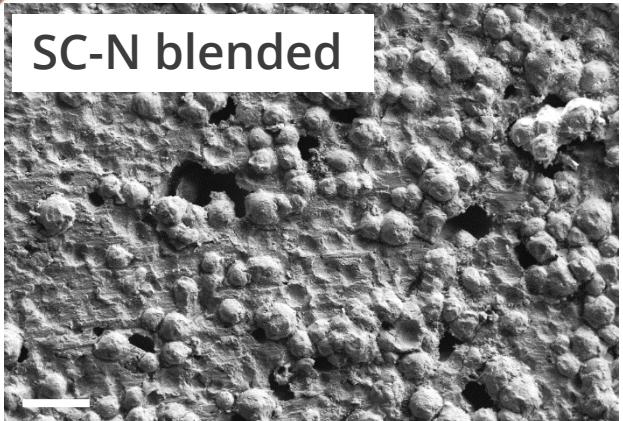
10 mm

Ni-N blended

10 mm

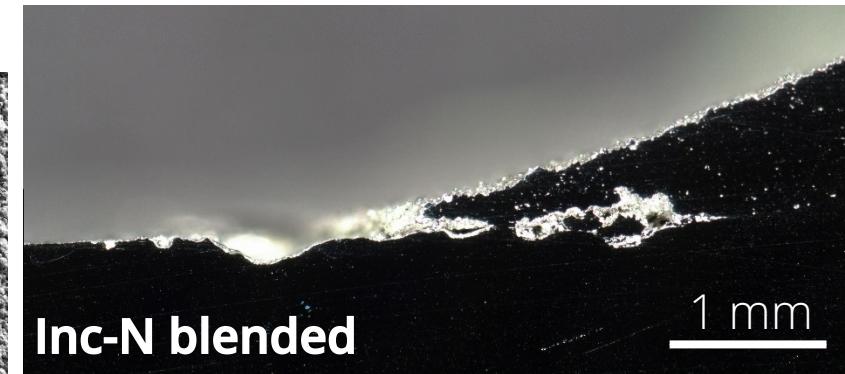
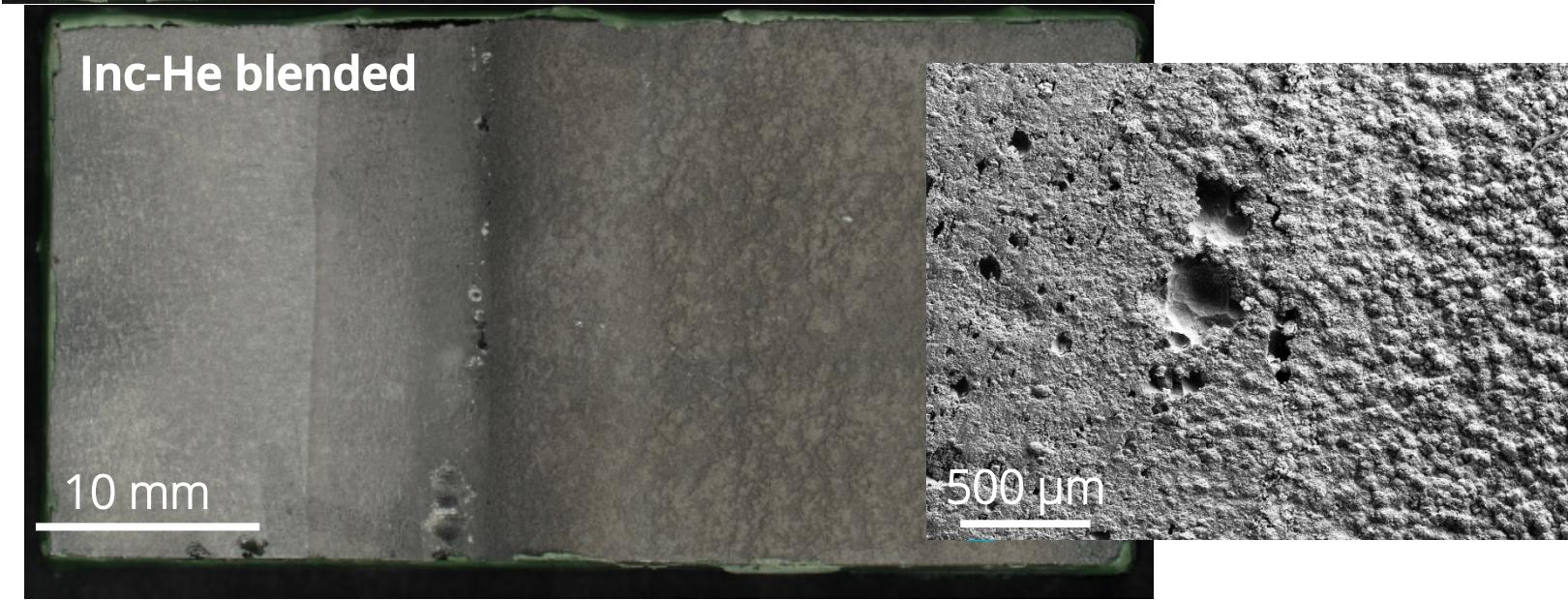
- Ni shows lots of damage in the cold spray & at the interface
- Inc-He damage looks different than Inc-N
- Inc-N & SC-N damage is harder to see

# Material Selection



Sample	Porosity (%)	$S_a$ ( $\mu\text{m}$ )
SC-N	$5.51 \pm 0.44$	$16.7 \pm 0.5$
Inc-N	$5.79 \pm 0.18$	$17.2 \pm 0.6$
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# Gas Selection



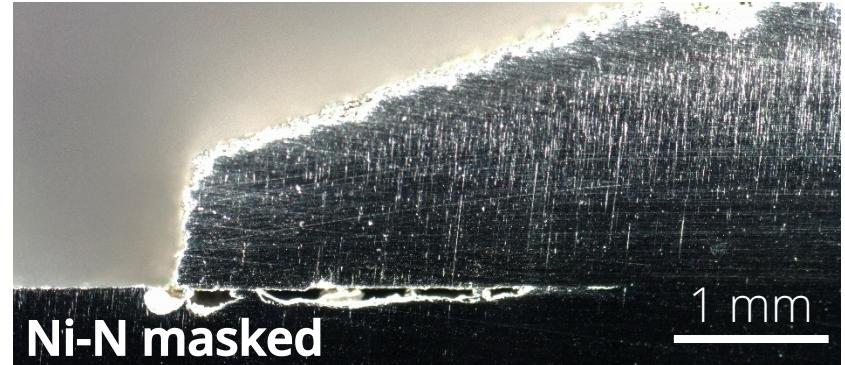
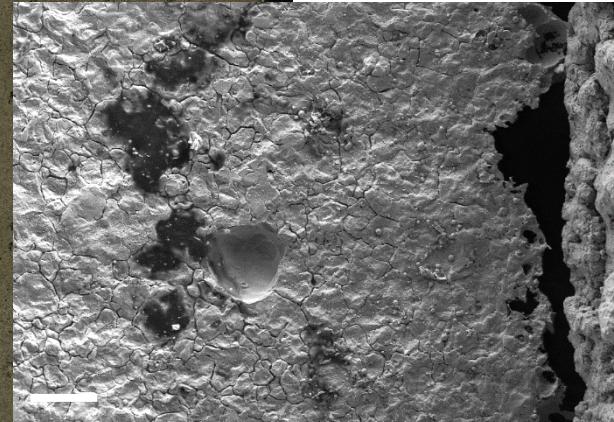
Sample	Porosity (%)
Inc-N	$5.79 \pm 0.18$
Inc-He	$1.21 \pm 0.20$



# Masked vs Blended: Ni-N

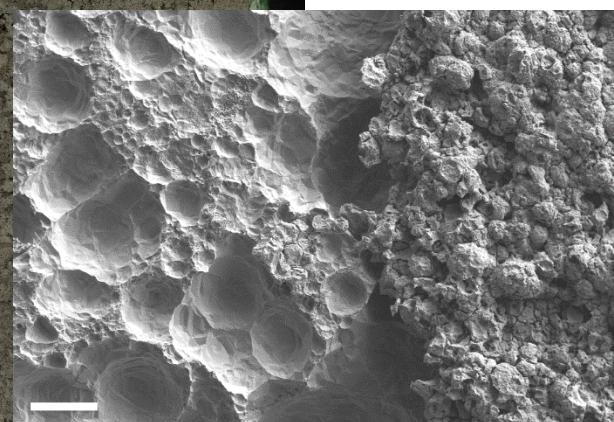
Ni-N masked

10 mm



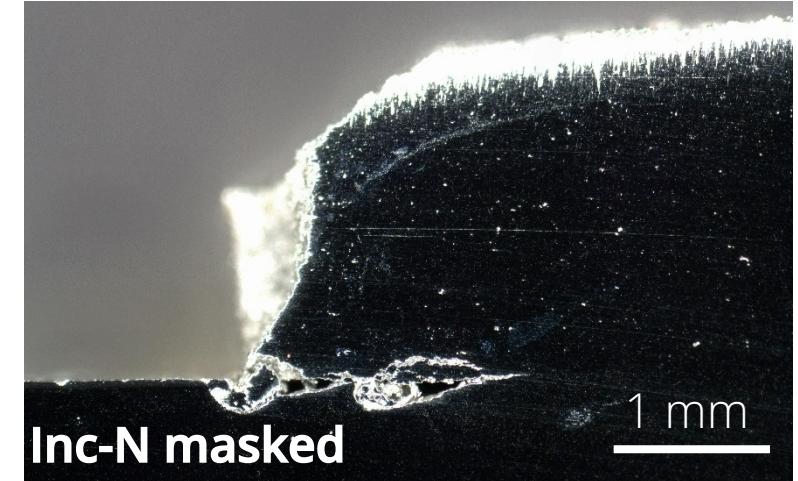
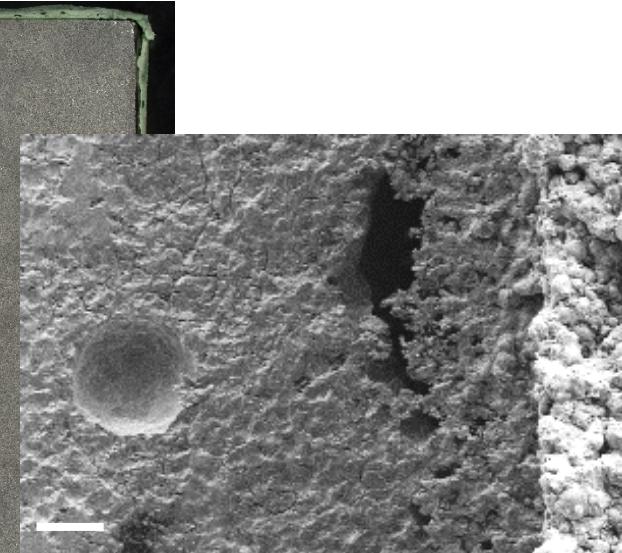
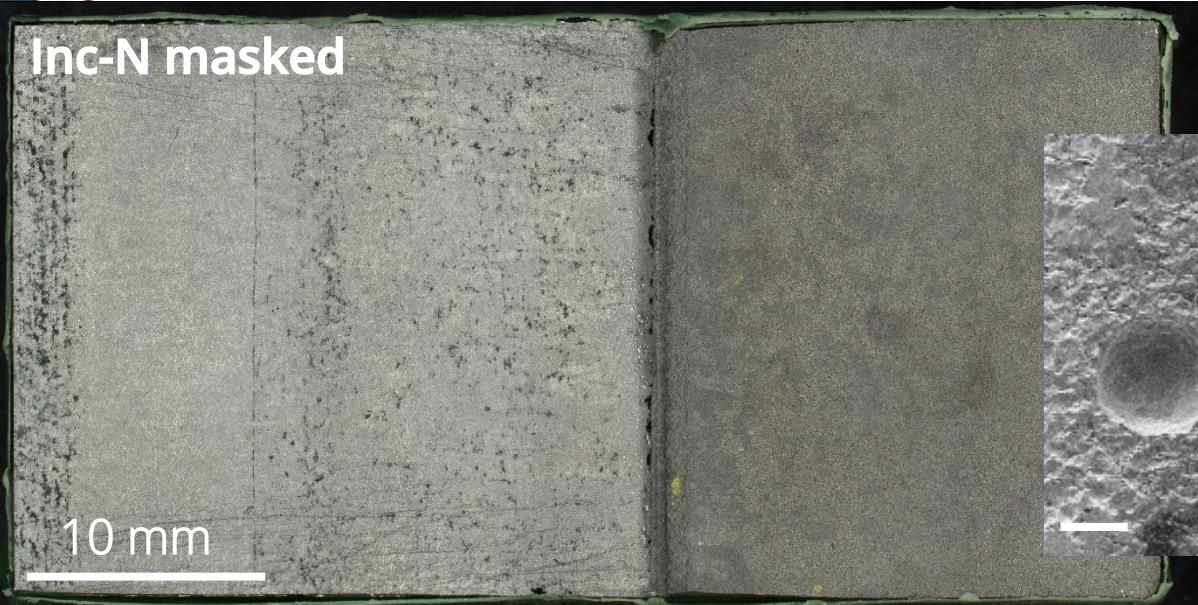
Ni-N blended

10 mm



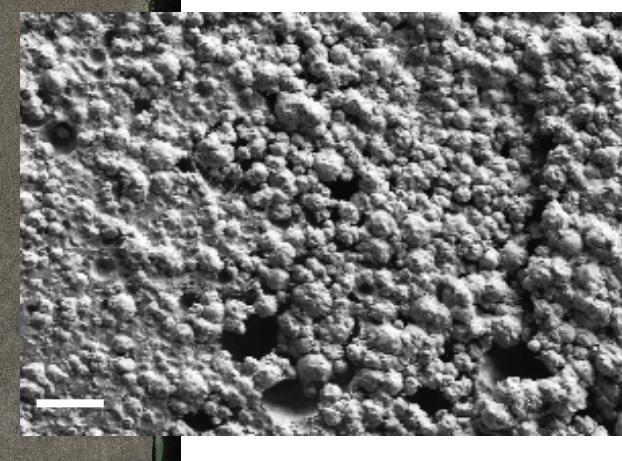
# Masked vs Blended: Inc-N

Inc-N masked



Inc-N masked

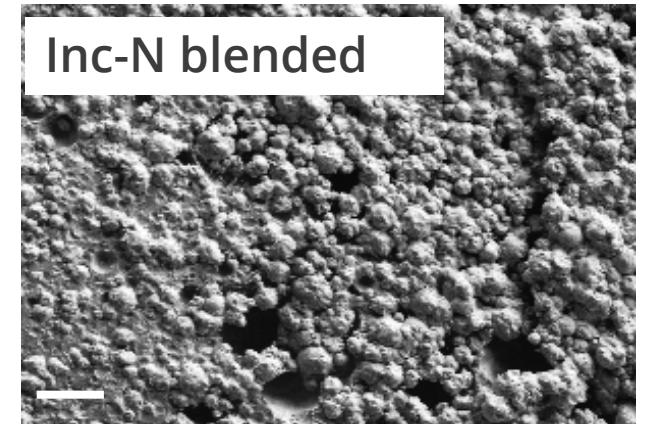
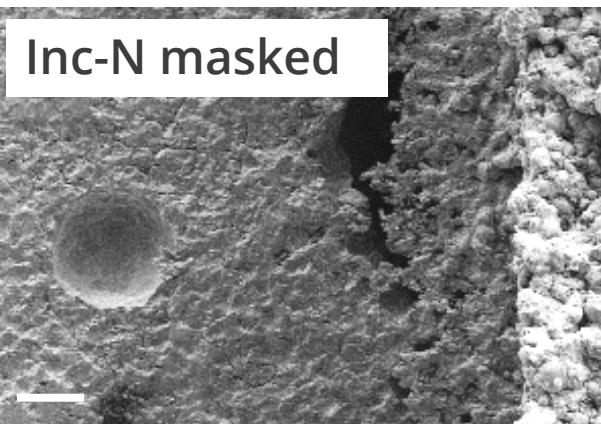
Inc-N blended



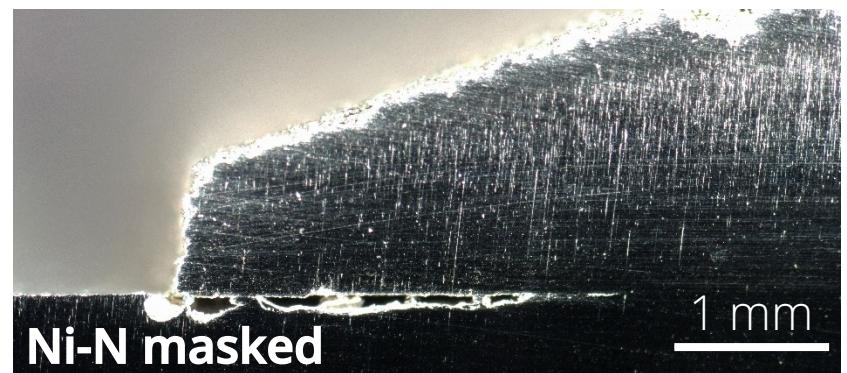
Inc-N blended

# Interface Damage

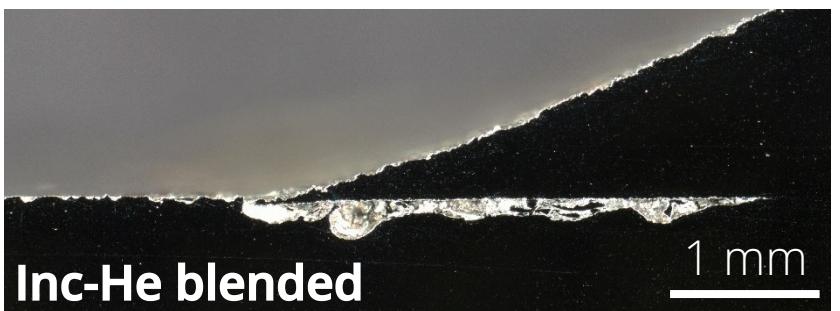
- Mask samples have deep crevicing attack at interface with some pits
  - Distance between crevicing and pits
- Blended samples tend to have less defined edge of attack
  - Surface roughness obscures extent of attack
- Top-down damage  $\neq$  corrosion under the cold spray



100  $\mu\text{m}$  scale bars



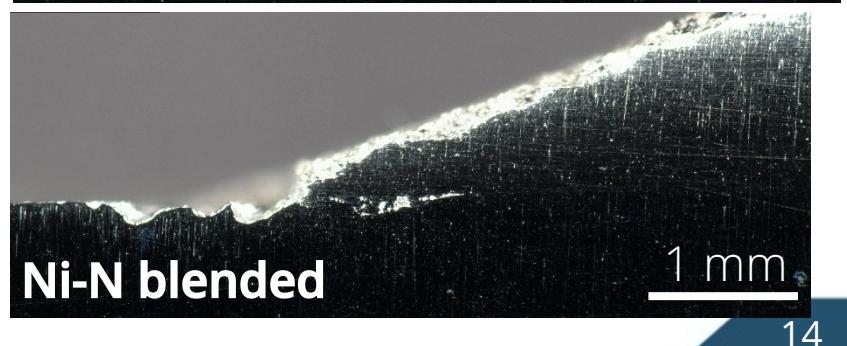
1 mm



1 mm

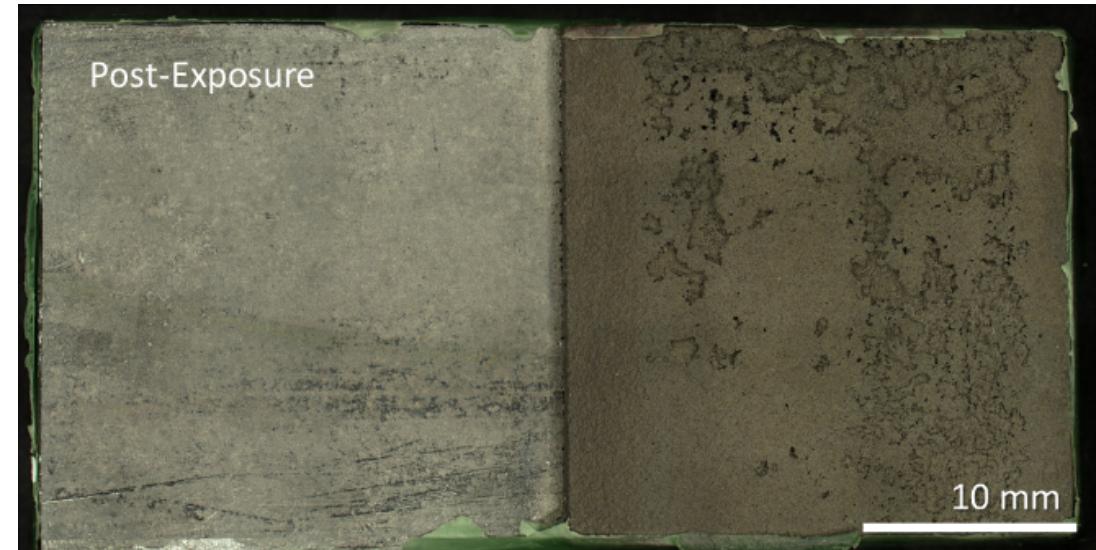
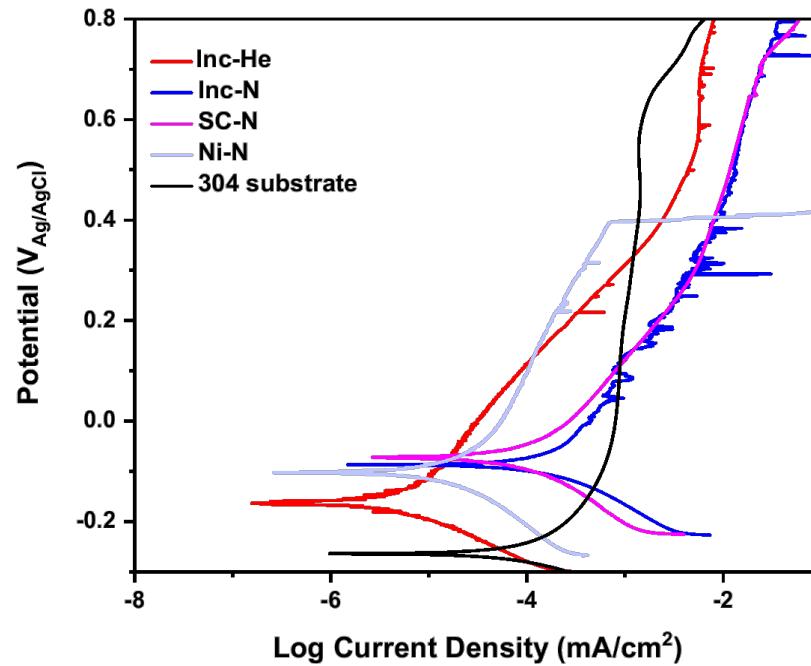


1 mm



1 mm

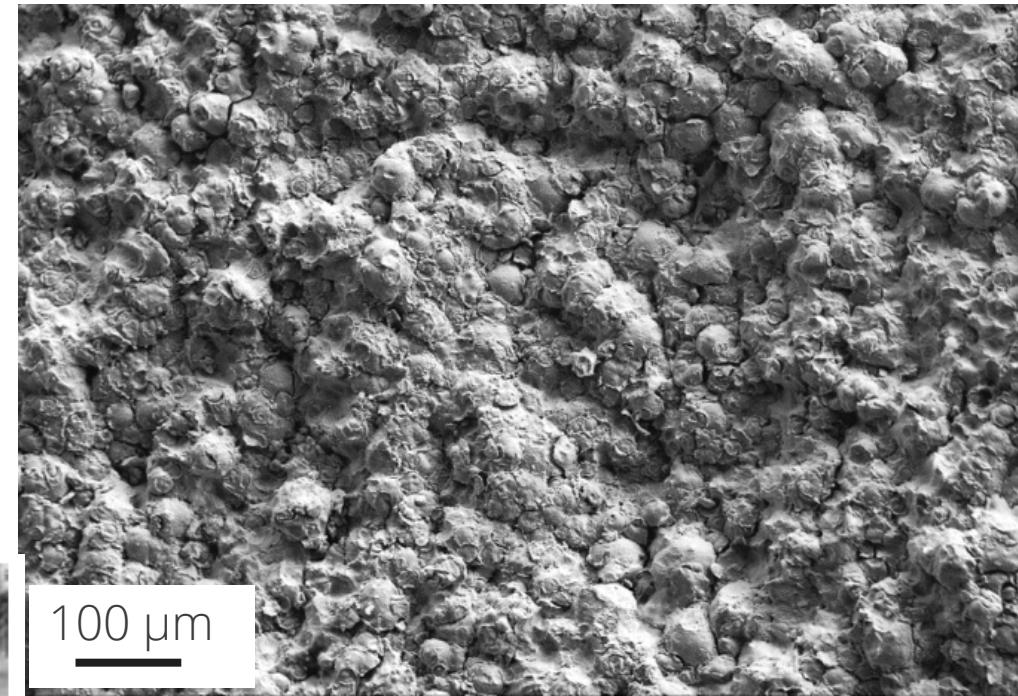
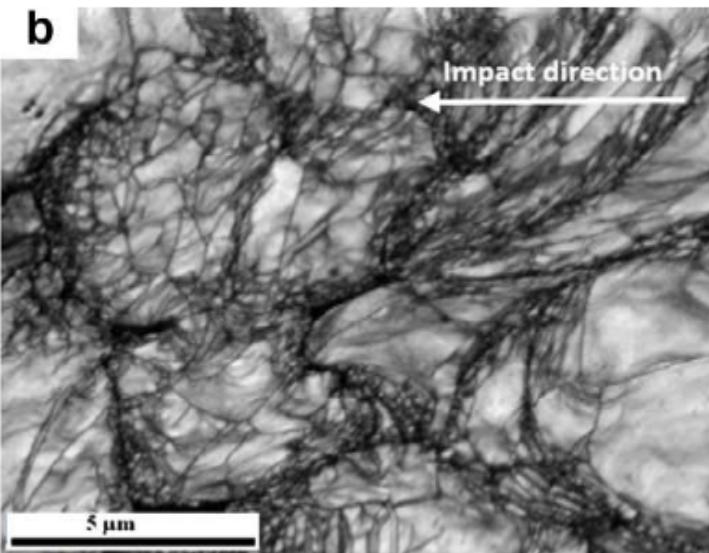
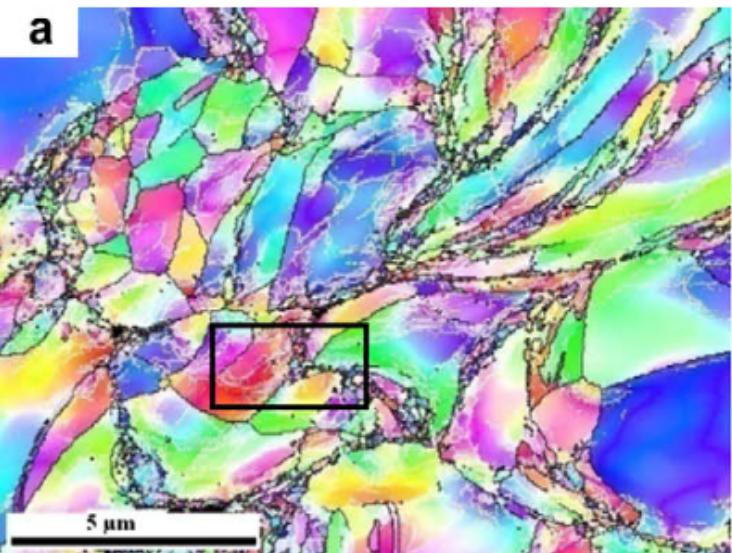
# Conclusions from Accelerated Testing



- Selection of near galvanic series materials is insufficient for corrosion protection in patch scenario in ferric chloride environment
- Material selection is important (interface & independent behavior)
- Processing parameters of the cold spray are important (porosity)
- Surface finish changes cold spray corrosion response (evaluated separate from the substrate)
- The type of interface (blended vs mask) will change where damage occurs

# Future Work

- Expand types of tests (boiling  $MgCl_2$ , salt fog, atmospheric)
- Stainless steel cold spray (304, 316, Duplex)
- Electron backscatter diffraction in cross-section
- Examine residual stress in substrate



Inc-He Cold spray surface  
as-sprayed post ferric chloride testing



# Other Talks

## **Localized Corrosion**

“Atmospheric Corrosion of Laser Powder Bed Fusion 316L Stainless Steel”  
Michael Melia PhD, Monday 9 am

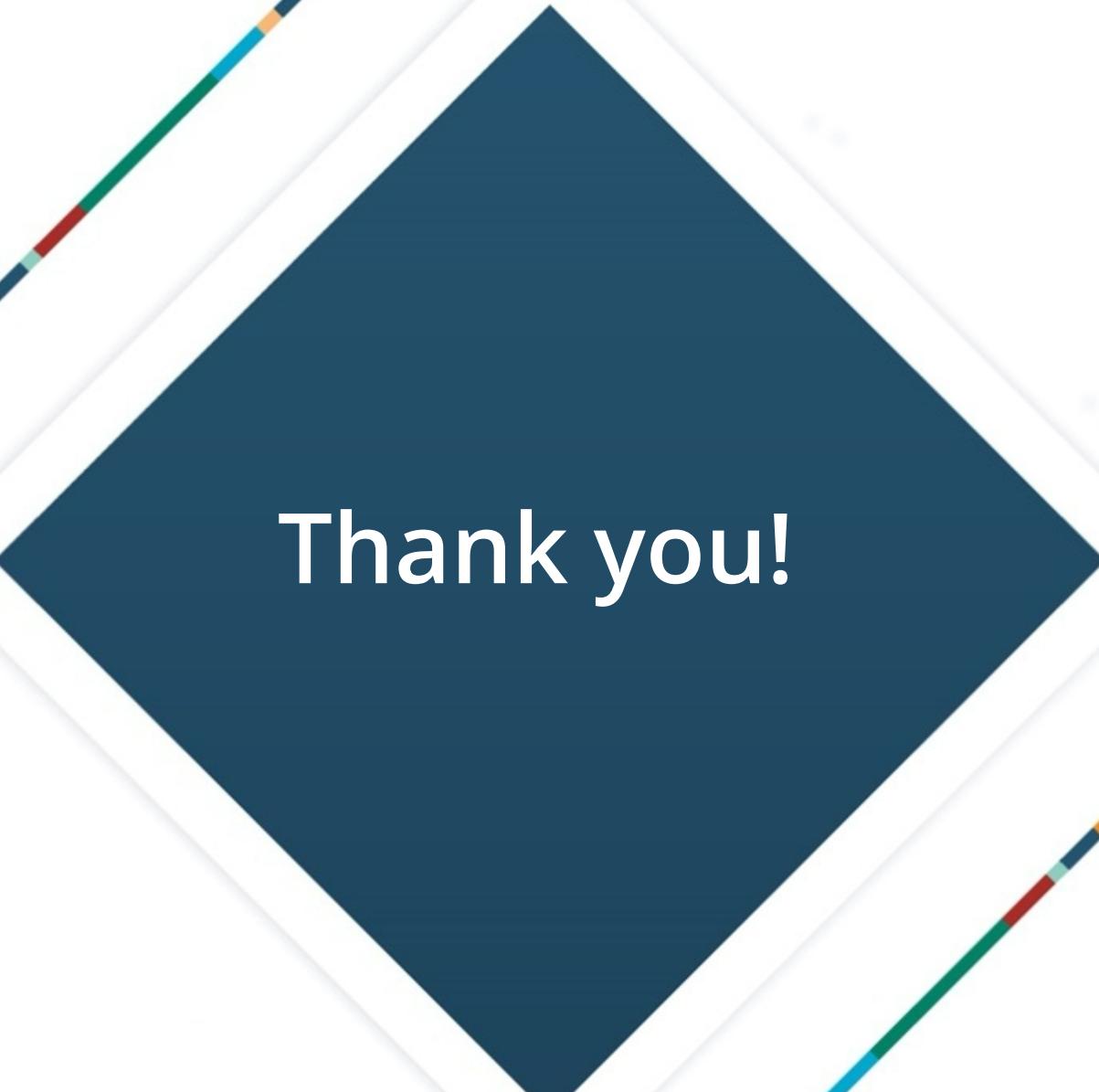
“Environmental Influences on Maximum Pit Sizes for Austenitic Stainless Steels Utilized In Spent Nuclear Fuel Storage” Ryan Katona PhD, Monday 11 am

## **Environmentally Assisted Cracking**

“Stress Corrosion Cracking of Austenitic Stainless Steel in Concentrated Chloride Environments” Ryan Katona PhD, Tuesday 11:25 am

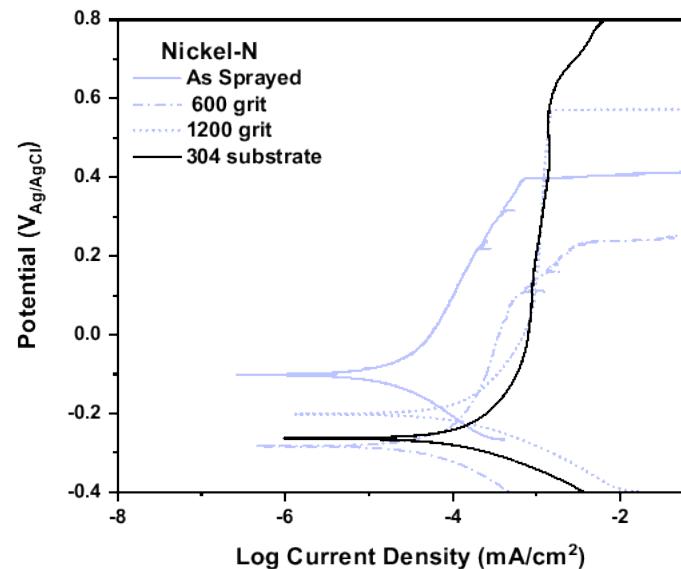
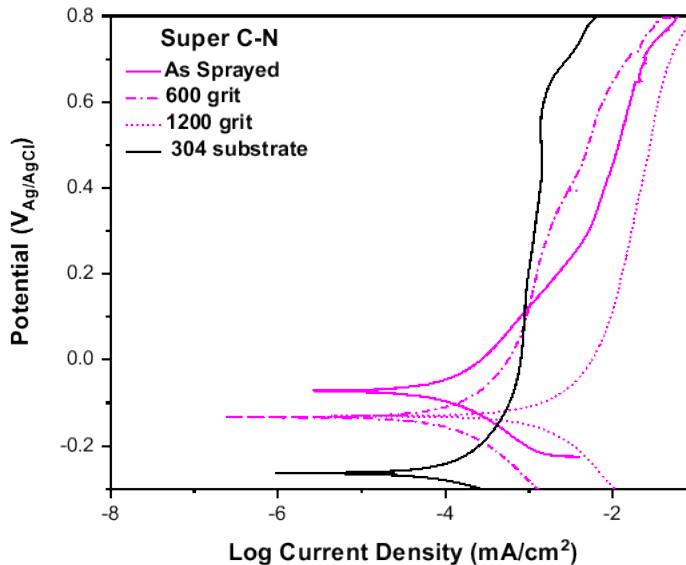
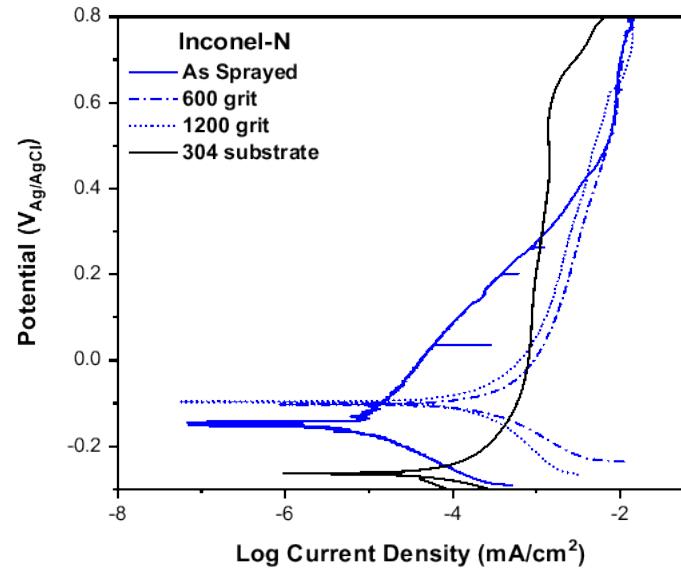
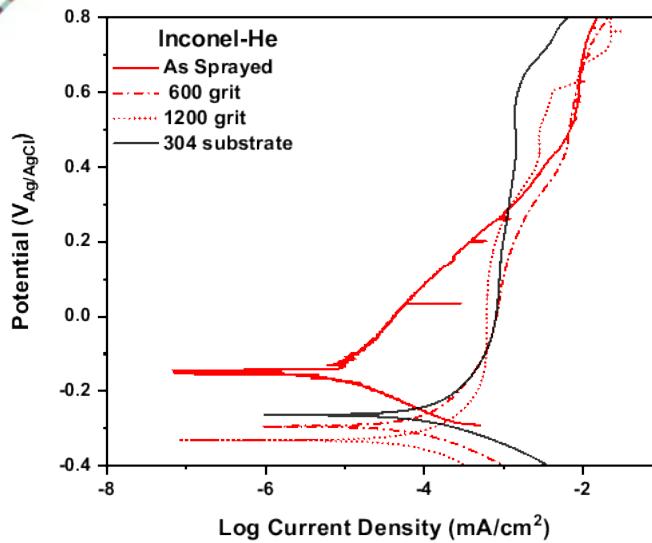
## **Student Poster Session**

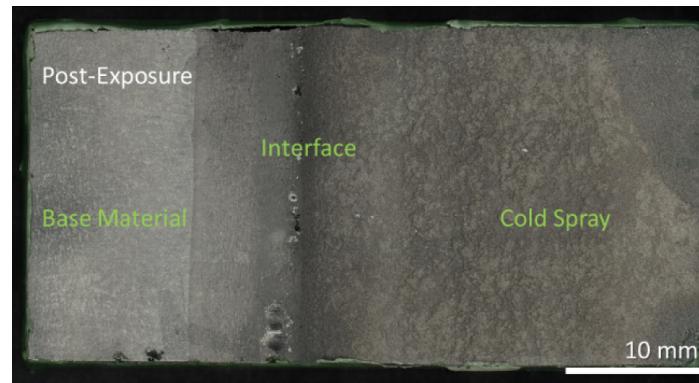
“Long Term, Simulated Marine Diurnal Exposure of Austenitic Stainless Steel at Elevated Temperatures” Timothy Montoya



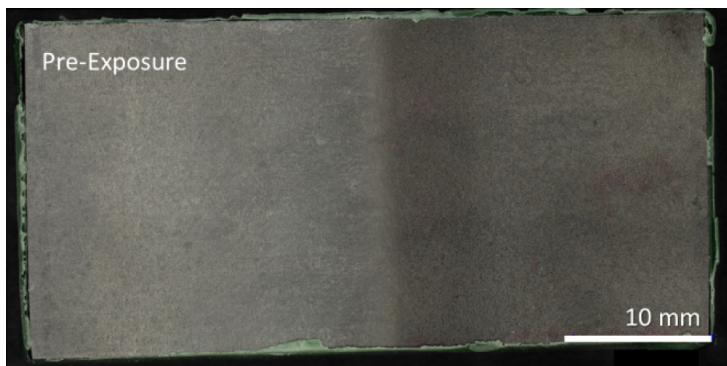
Thank you!

# Effects of Polishing

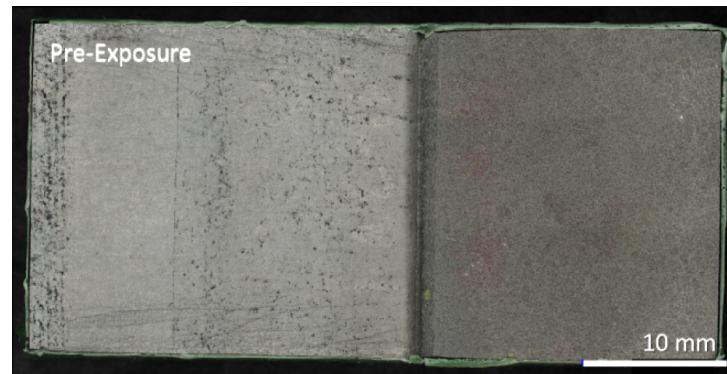




Inc-He blended



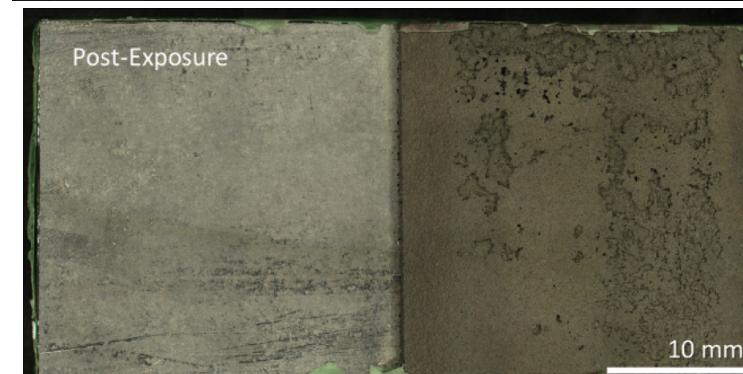
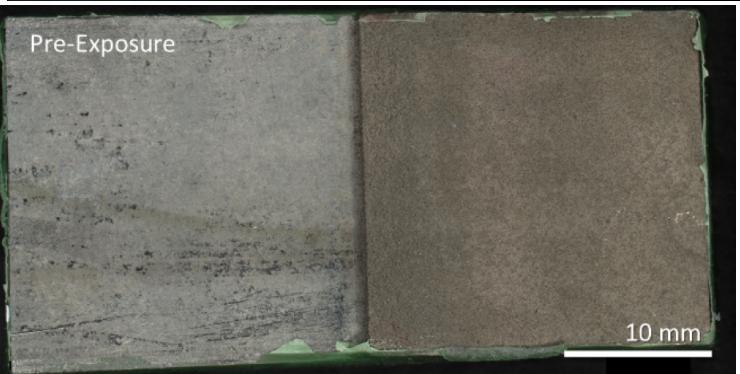
Inc-N blended



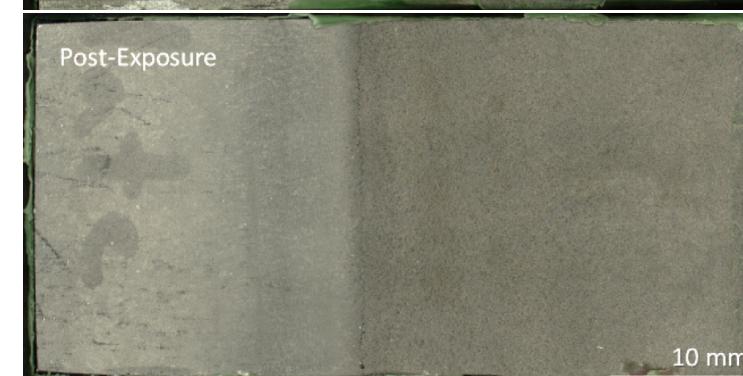
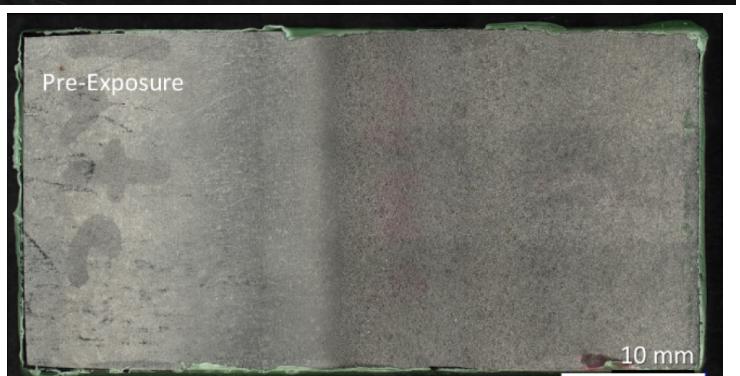
Inc-N masked



N-N blended



N-N masked



Super C-N blended