Chloride Salt Flow Loop and Corrosion Studies

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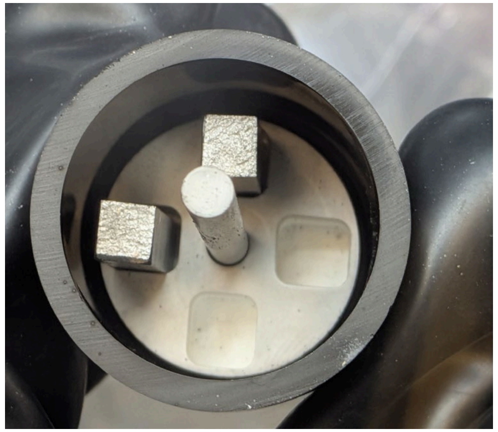
AMMT Molten Salt Corrosion: FY26 Tasks at INL

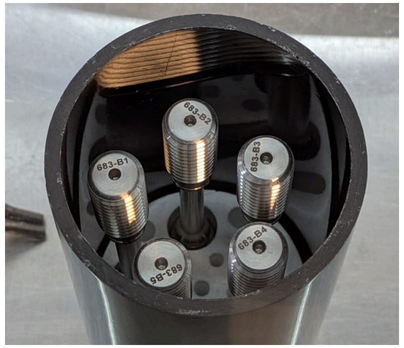
Task 1: Static corrosion of LPBF 316H stainless steel and wrought Alloy 244 in molten chloride salts

Wrought SS316 exposure complete w/ Terrapower and as-purchased salt

1000 hour LPBF 316H static exposure ongoing



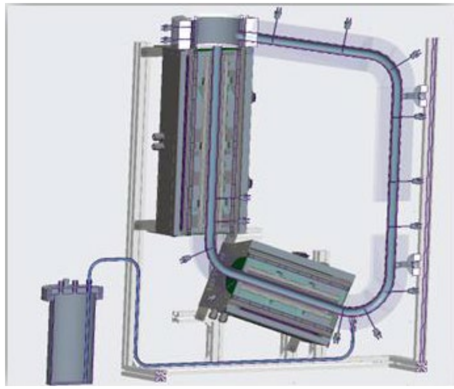
Task 2: Ex situ mechanical testing of LPBF SS316H in NaCl-$MgCl_2$



Wrought SS316 exposure complete

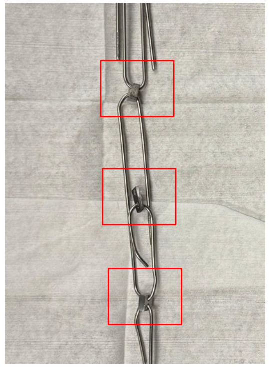
Creep testing of exposed will start soon

Task 3: Corrosion testing of LPBF SS316H and wrought Alloy 244 in flowing LiCl-KCl



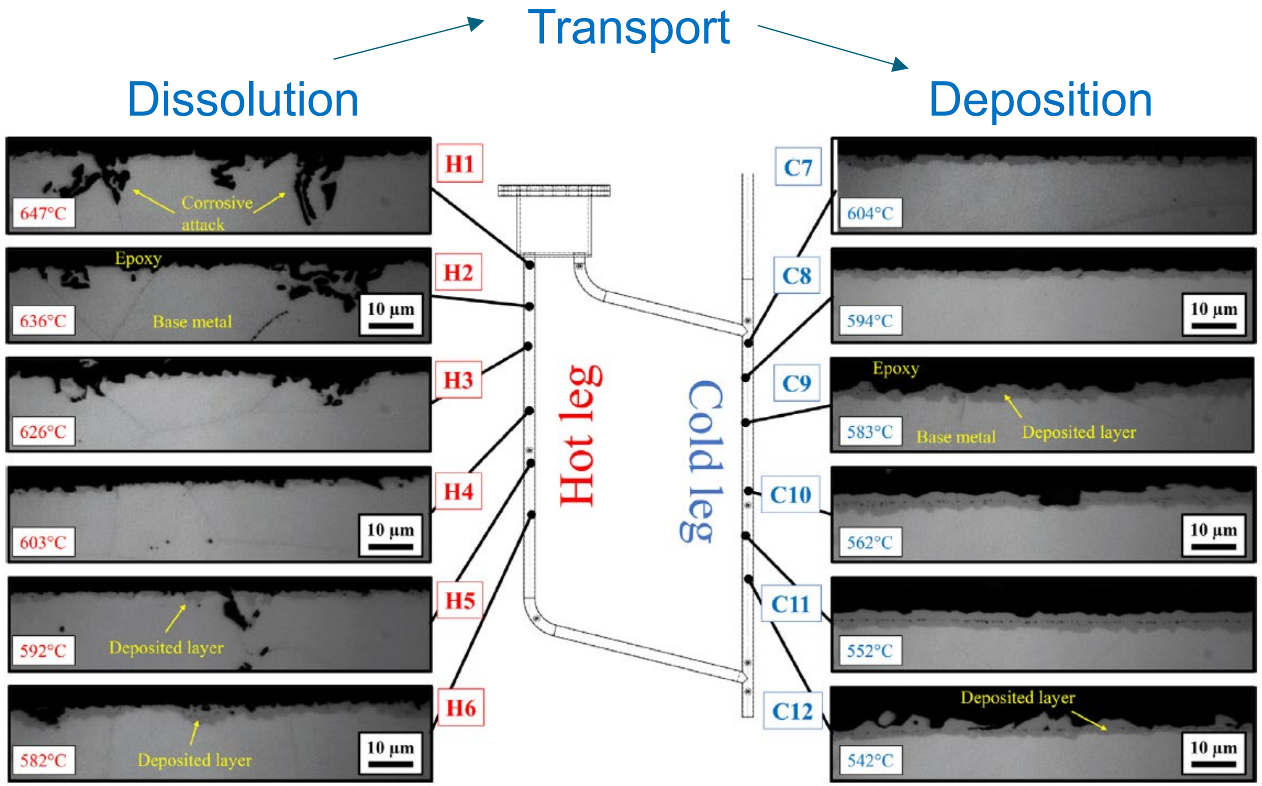
Cross section of INL thermal convection loop

Wrought and LPBF samples in loop sample chain



Why Study Corrosion using Flow Loops?

- Thermal gradients create hot and cold regions coupled by natural circulation
- Flow enables transport of dissolved corrosion products through the loop
- Corrosion process is, therefore, a mass transfer process, instead of only local attack
- Flowing salt tests better represent reactor environments than static capsules

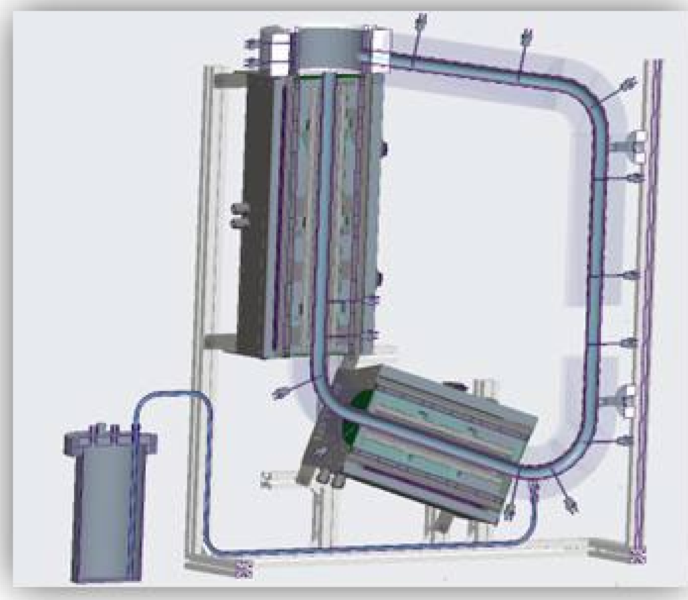


Flow loops capture coupled thermal gradients, transport, and chemistry absent in static exposure

Raiman et al. *Journal of Nuclear Materials.* 2022 (561), 153551. https://doi.org/10.1016/j.jnucmat.2022.153551

Molten Salt Thermal Convection Loop (TCL) at INL

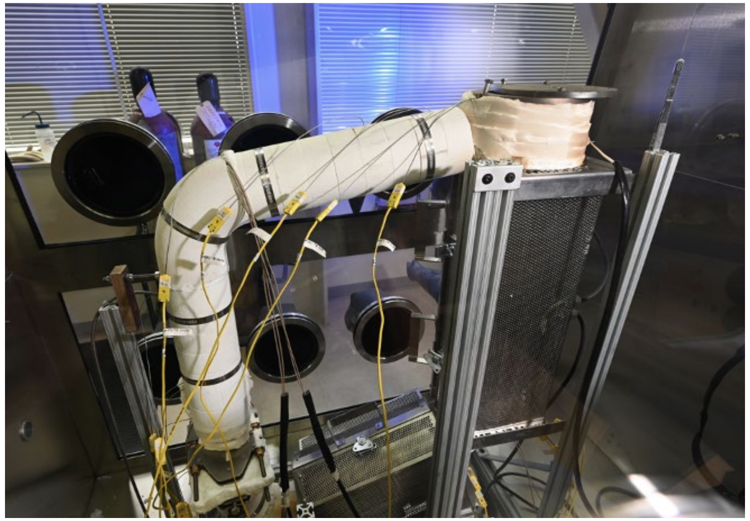
- Inert-atmosphere glovebox operation minimizes air/moisture contamination
- **316L stainless steel** loop (1” ID pipe) charged with **~5 kg eutectic LiCl-KCl**
- Hot-leg temperature: 450–600°C
- Temperature differential across loop: ~100°C
- Total **operating time: 1200 h** across three campaigns
- Fill/drain architecture supports repeated testing and maintenance
- Multiple specimen positions enable temperature-dependent exposure studies

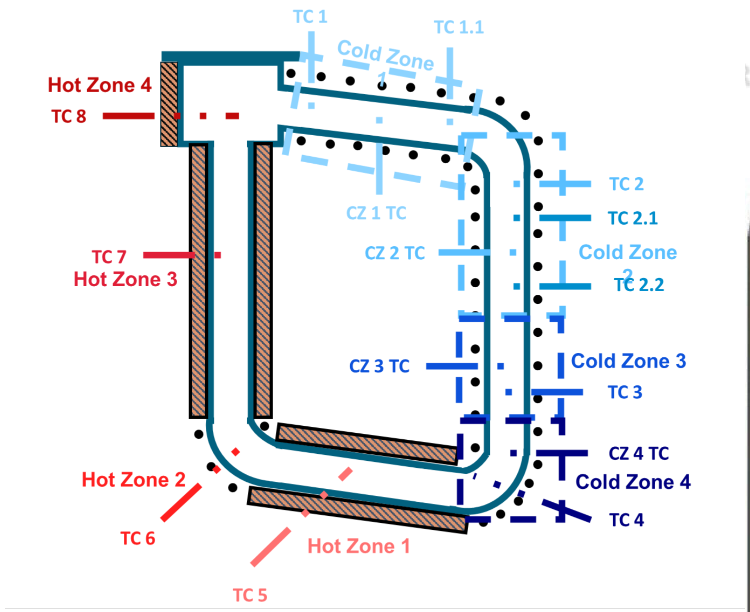


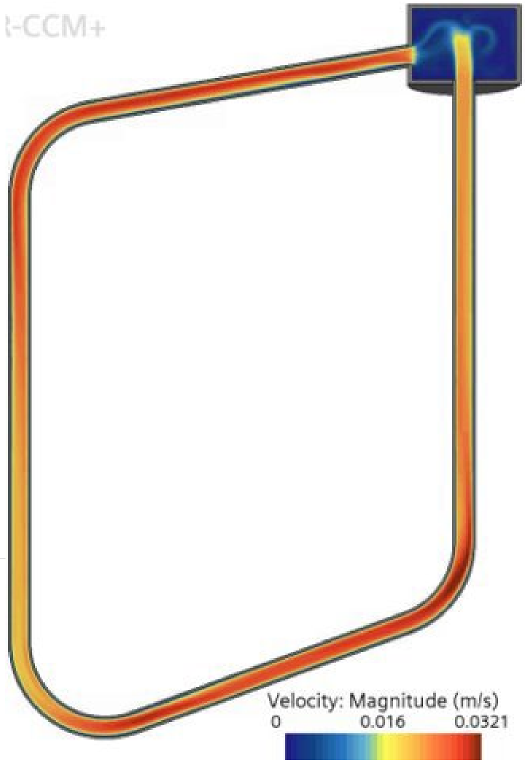
Cross section of INL TCL

TCL enables controlled study of coupled temperature, flow, and corrosion effects

Molten Salt Thermal Convection Loop (TCL) at INL

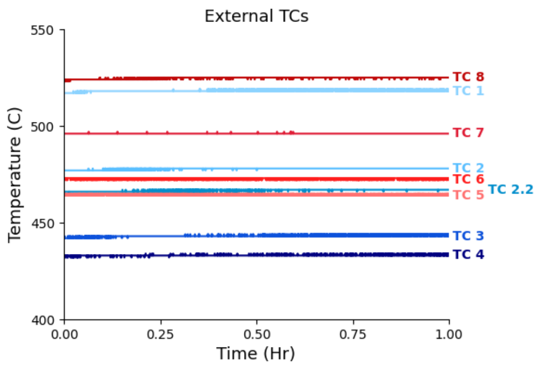


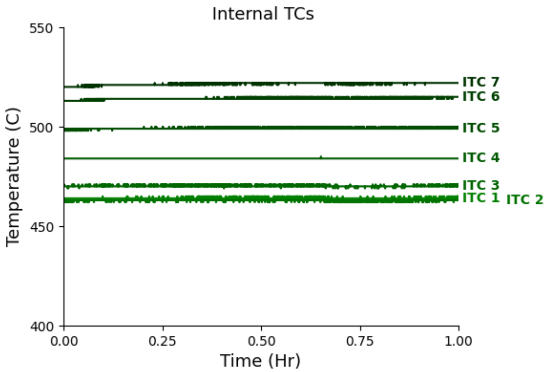




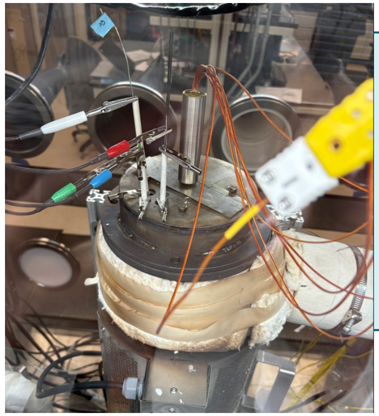
- Steady-state natural circulation CFD simulation (STAR-CCM+)
- Thermophysical properties from literature correlations

Flow Velocity Profile obtained using CFD model





Salt chemistry monitored during 1200 h operation



Electrochemical Sensor implemented during loop operation and corrosion test

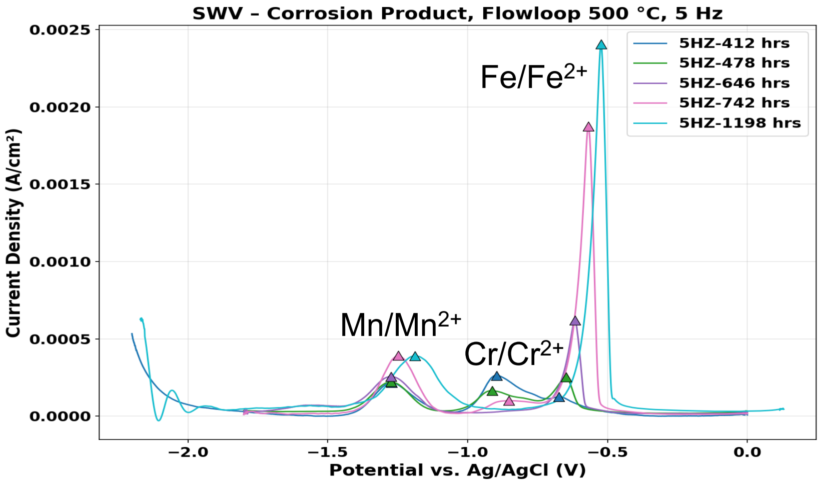
Three-electrode Echem Setup

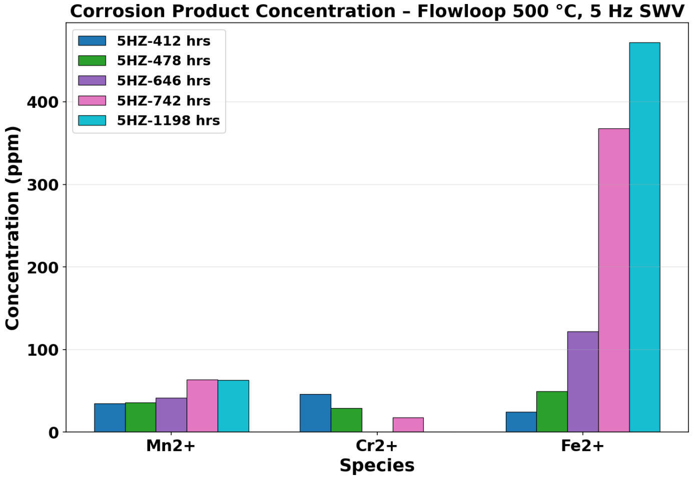
- WE: Tungsten for corrosion products/ Gold for non-metal impurities
- CE: Glassy carbon
- RE: Ag/AgCl

Techniques Probed

- Open circuit potential (OCP)
- Cyclic voltammetry (CV)
- Square wave voltammetry (SWV): more sensitive for identification of trace amount of species

Peak assignments are based on literature-reported potentials and were consistent with the observed peak separation under our test conditions.





- Concentration is calculated using the equation below.

$$I_p = 0.9653nFAC\sqrt{Df}\tanh\left(\frac{\zeta_{sw}}{2}\right)$$

$$i_p = \frac{I_p}{A}, \zeta_{sw} = \frac{nFE_{sw}}{RT}$$

- D of Mn^{2+}, Cr^{2+} and Fe^{2+} are from literature with a value of 2.0 (Hoover et al.), 1.53 (Khalaghi et al.), and 1.4 x 10^{-5} cm^2/s (Inman et al.).

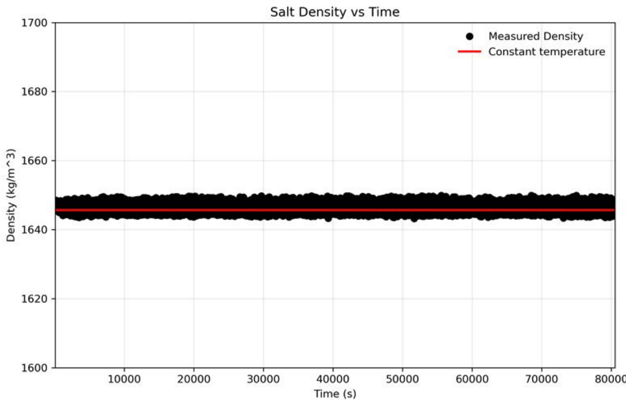
Fe was the dominant detected corrosion product during TCL operation, followed by Mn and Cr

- Horvath et al. https://www.sciencedirect.com/science/article/pii/S0022311517305548
- Lentelme et al. https://www.sciencedirect.com/science/article/pii/002207289280545F
- Inman et al. https://link.springer.com/content/pdf/10.1007/BF00616430?utm_source=scopus&getft_integrator=scopus

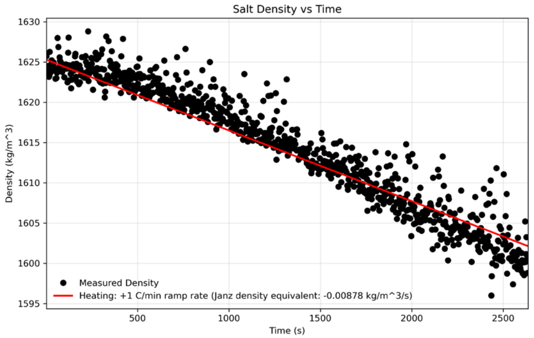
Triple Bubbler Sensor tested under salt flow

Modified triple bubbler installed in the molten salt flow loop

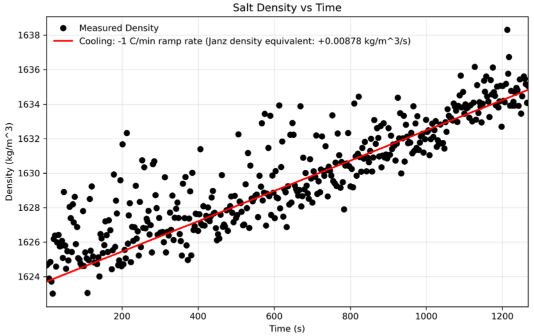
Triple bubbler



Steady-state density of LiCl-KCl



Heating of Salt



Cooling of Salt

Transient density of LiCl-KCl

- Triple-bubbler sensor deployed for in situ density monitoring in molten LiCl-KCl
- Density inferred from differential gas pressure measurements
- **Negligible flow effect** observed under tested laminar-flow conditions
- Stable response with <1% density uncertainty under tested conditions
- Enables **real-time tracking of salt temperature changes** during loop operation.

Triple bubbler data demonstrated stable in-situ density monitoring under TCL operating conditions

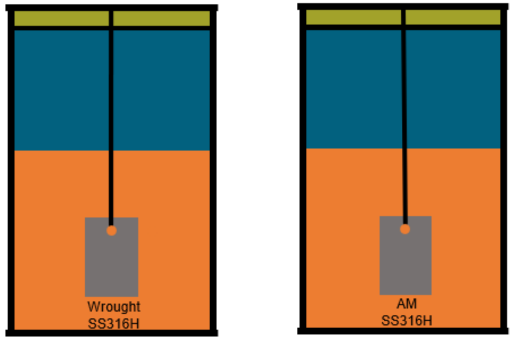
Wrought and AM 316H Specimens in Hot Leg

Corrosion test in flowloop:

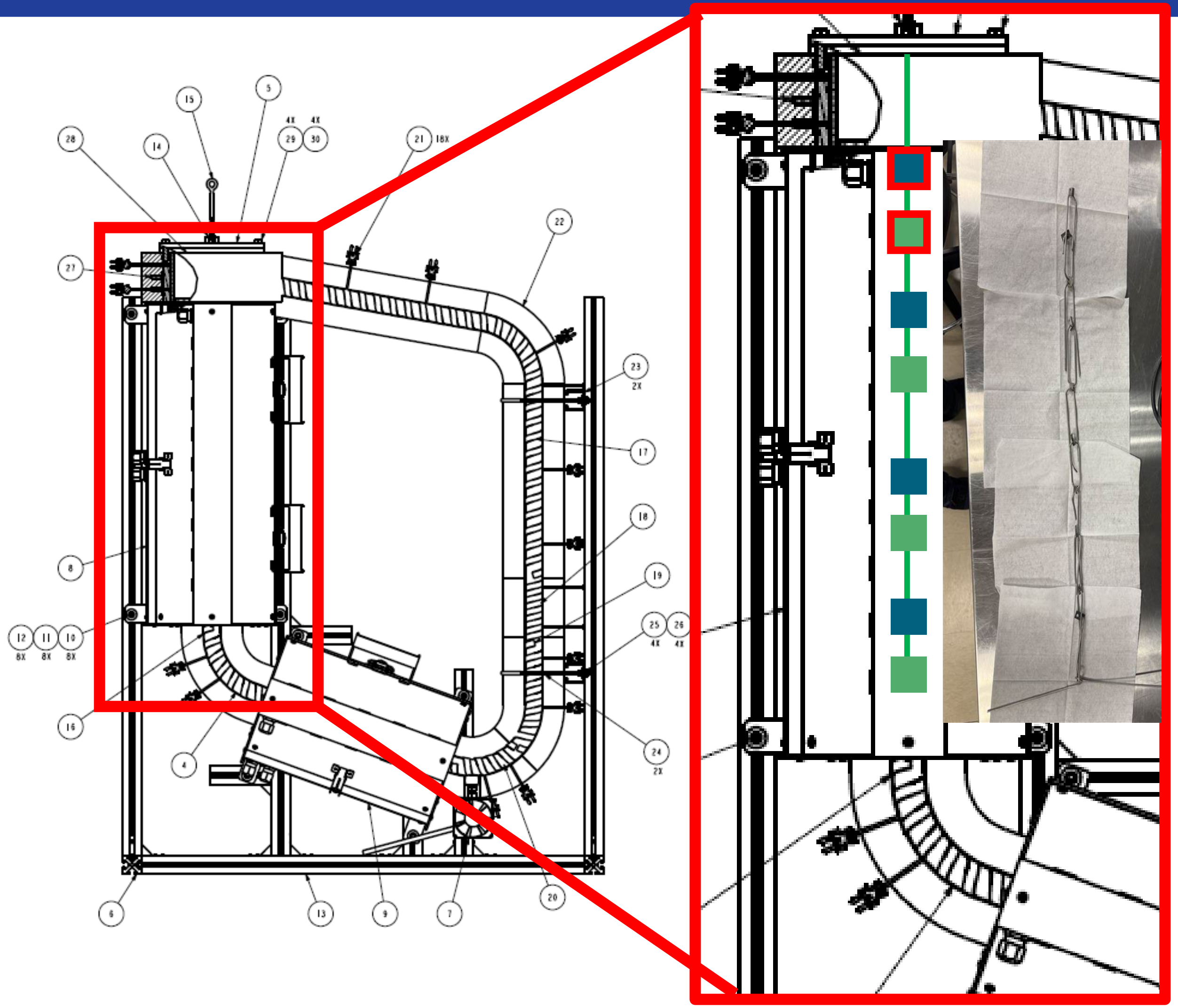
- Wrought and AMSS316H specimens were introduced along different sections of the hot leg
- Samples were exposed to temperatures ranging from 500 ºC to 450 ºC for **250 hours**

Comparative test in static LiCl-KCl salt:

- Wrought and AMSS316H specimens were exposed to static LiCl-KCl salt at 500 ºC for **250 hours**



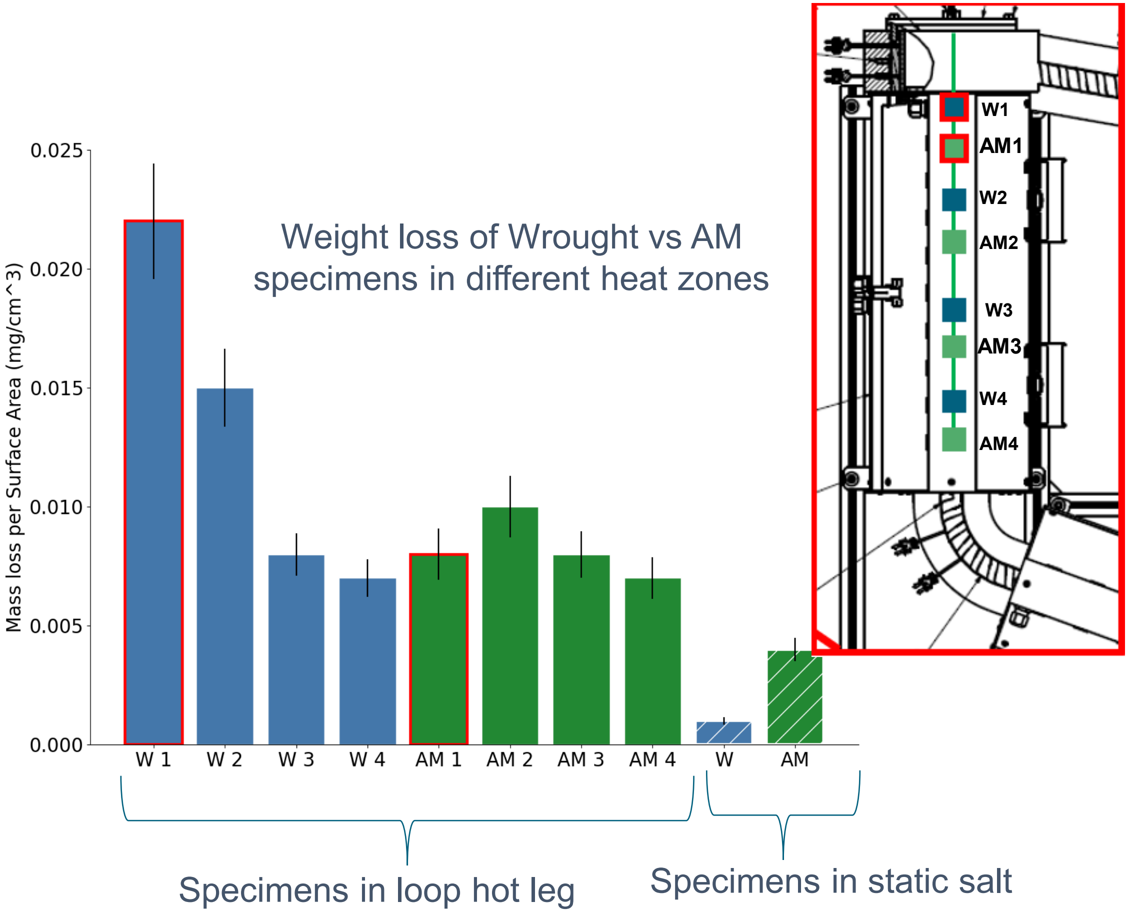
SS316L crucible   SS316L crucible



Flow-driven mass loss

Mass loss from specimens exposed to flowing and static LiCl-KCl salt for 250 hours were compared. We observed:

- Specimens in the hottest section showed higher mass loss compared to the colder sections
- Both materials followed similar temperature-dependent trends, but wrought generally showed higher mass loss than AM
- Samples in the flow loop show higher mass loss than static samples

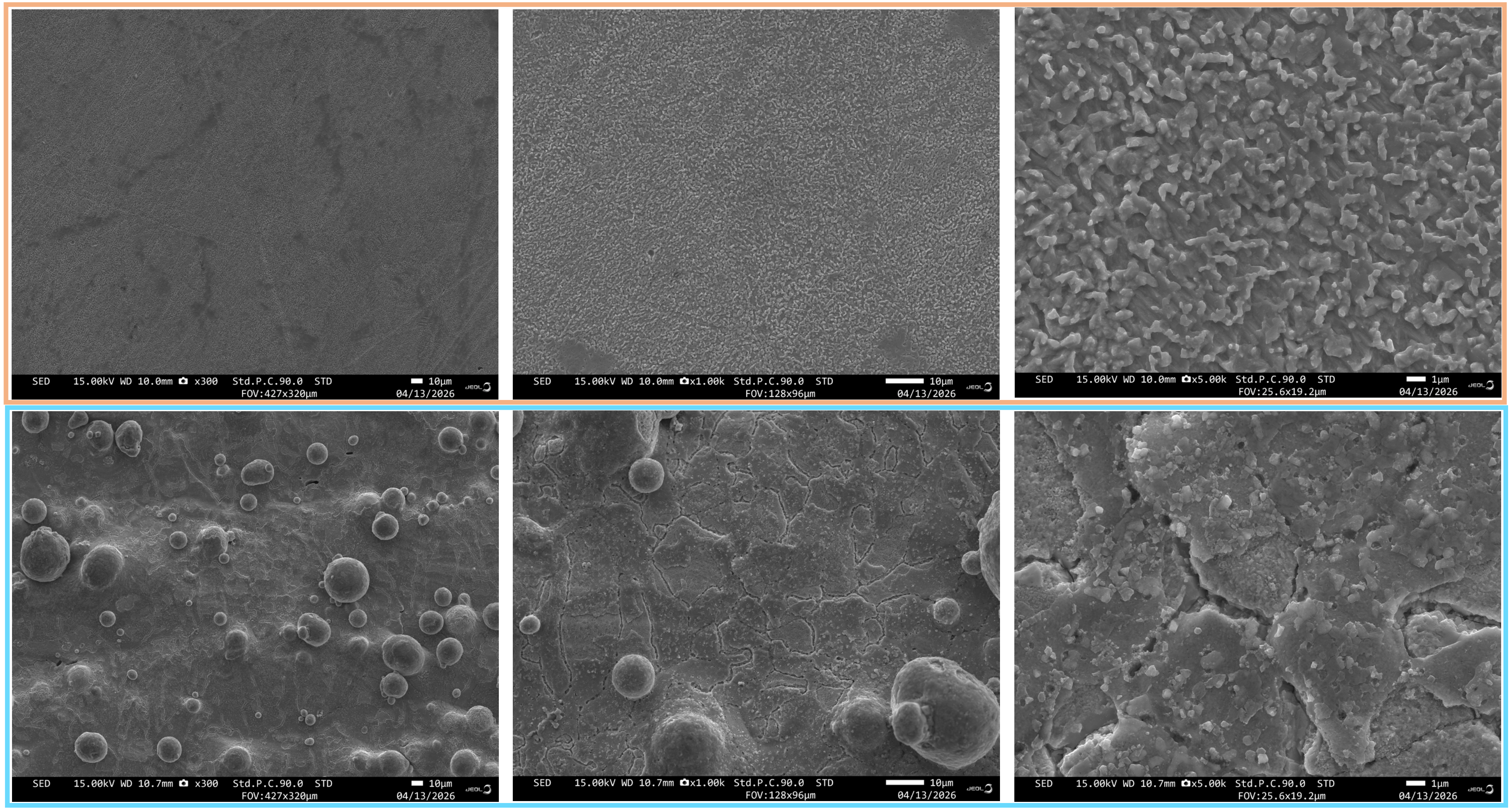


Specimens exposed to flowing salt showed higher net mass loss compared to static exposure under these test conditions

Post-corrosion surface morphology, Flowloop samples: As-deposited vs. machine-cut

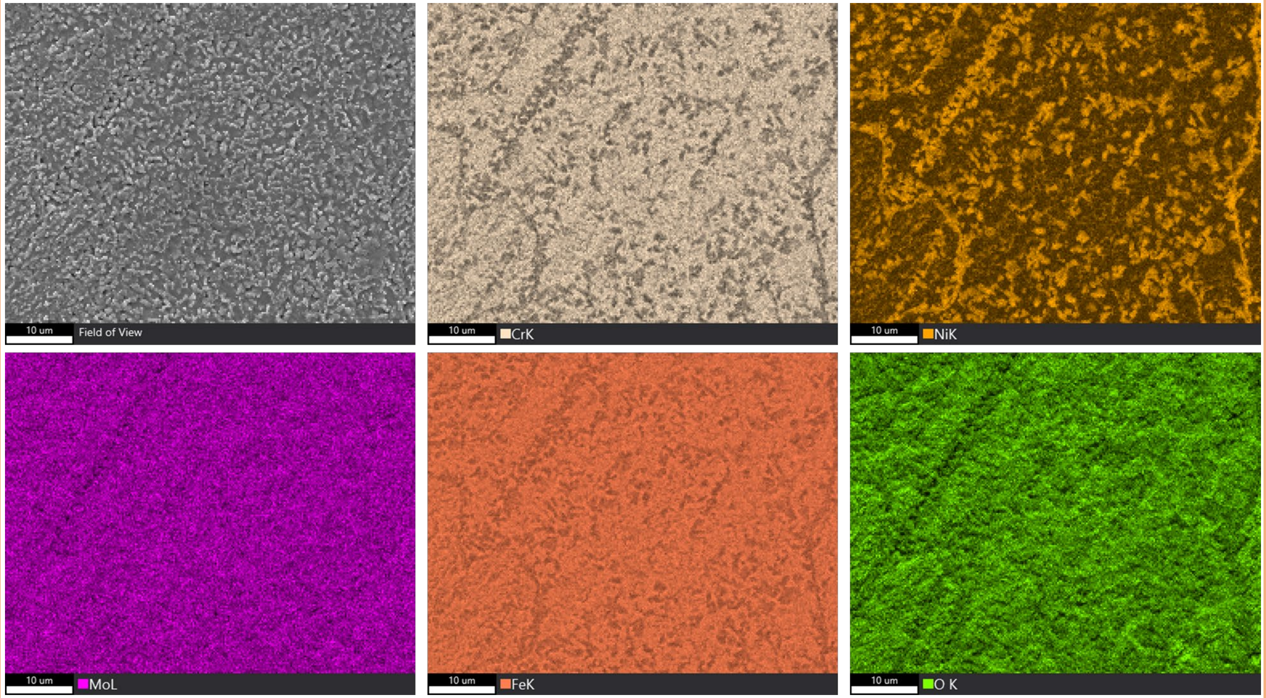
Machine cut Surface

As-Deposited Surface

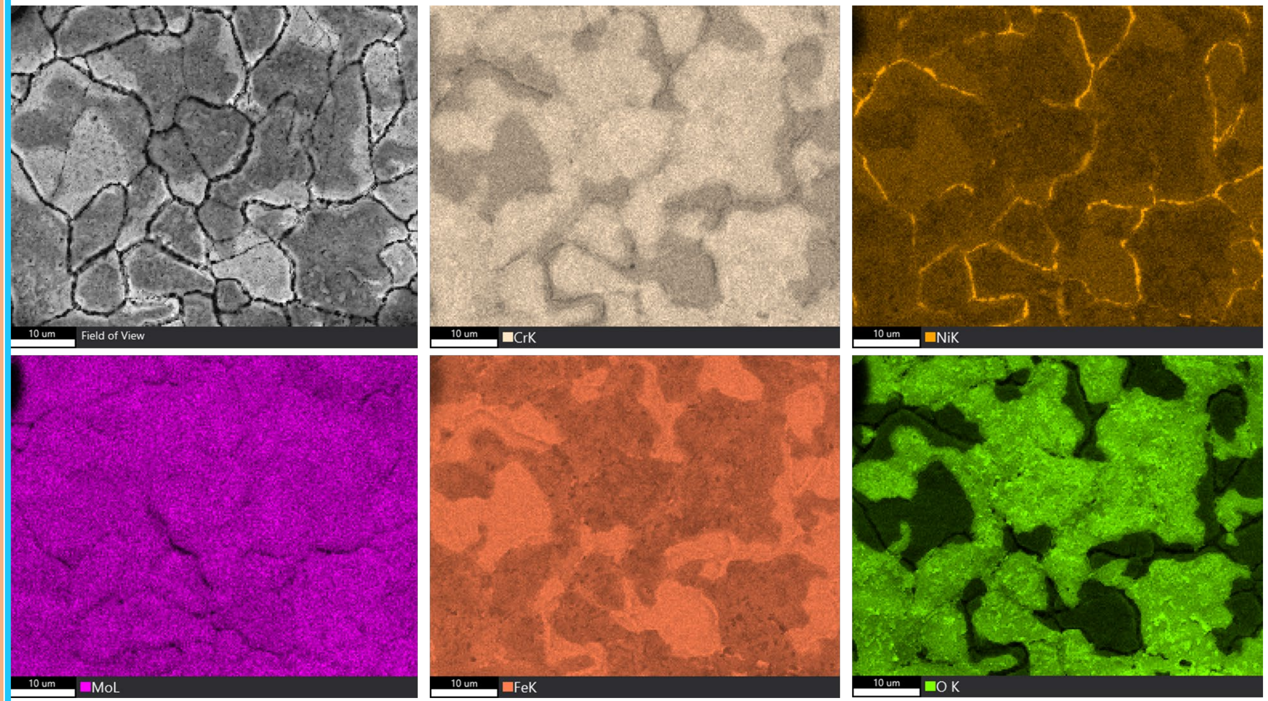


Post-corrosion EDS analysis - elemental redistribution comparison

Machine Cut Surface



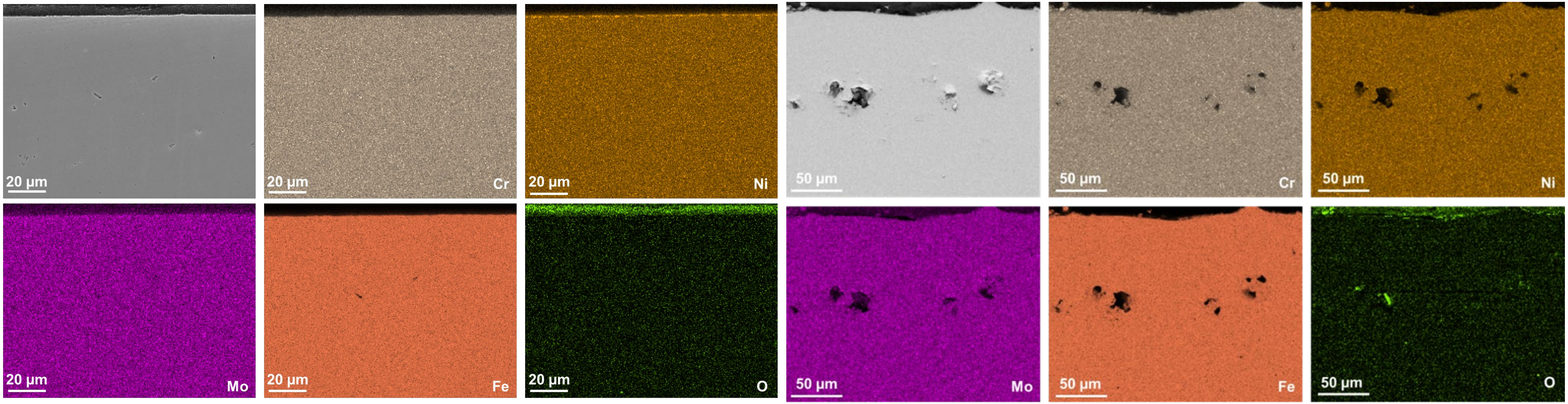
As-Deposited Surface



Elemental redistribution under flow depends strongly on initial surface morphology

Post-corrosion EDS analysis - elemental redistribution comparison

AM - Machine Cut Surface | **AM - As-Deposited Surface**

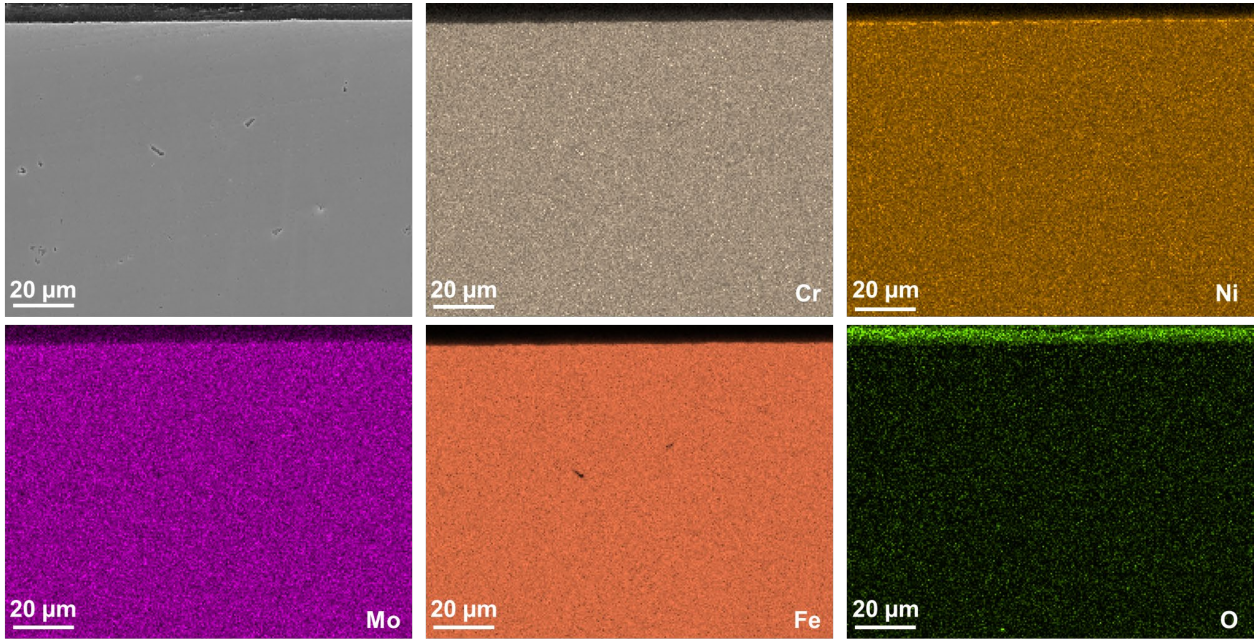


AM SS316H showed largely surface-confined attack regardless of surface finish

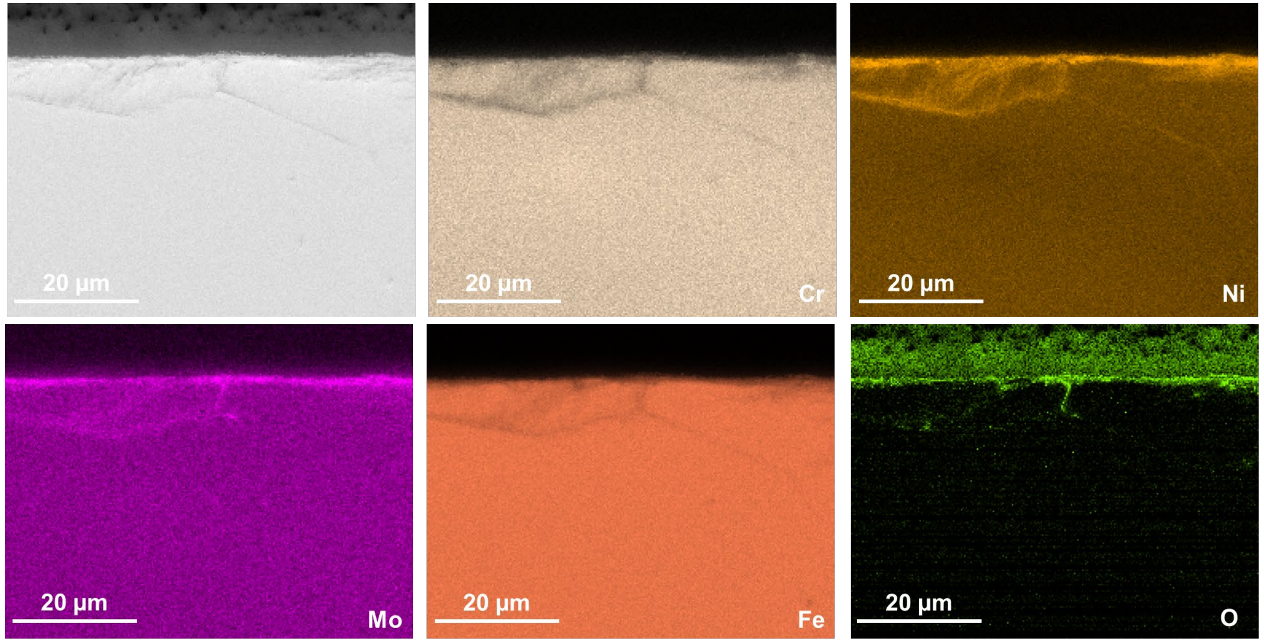
Notably, attack is more uniform in machine cut surface compared to as-deposited surface

Post-corrosion EDS analysis - elemental redistribution comparison

AM - Machine Cut Surface



Wrought – Machine Cut Surface

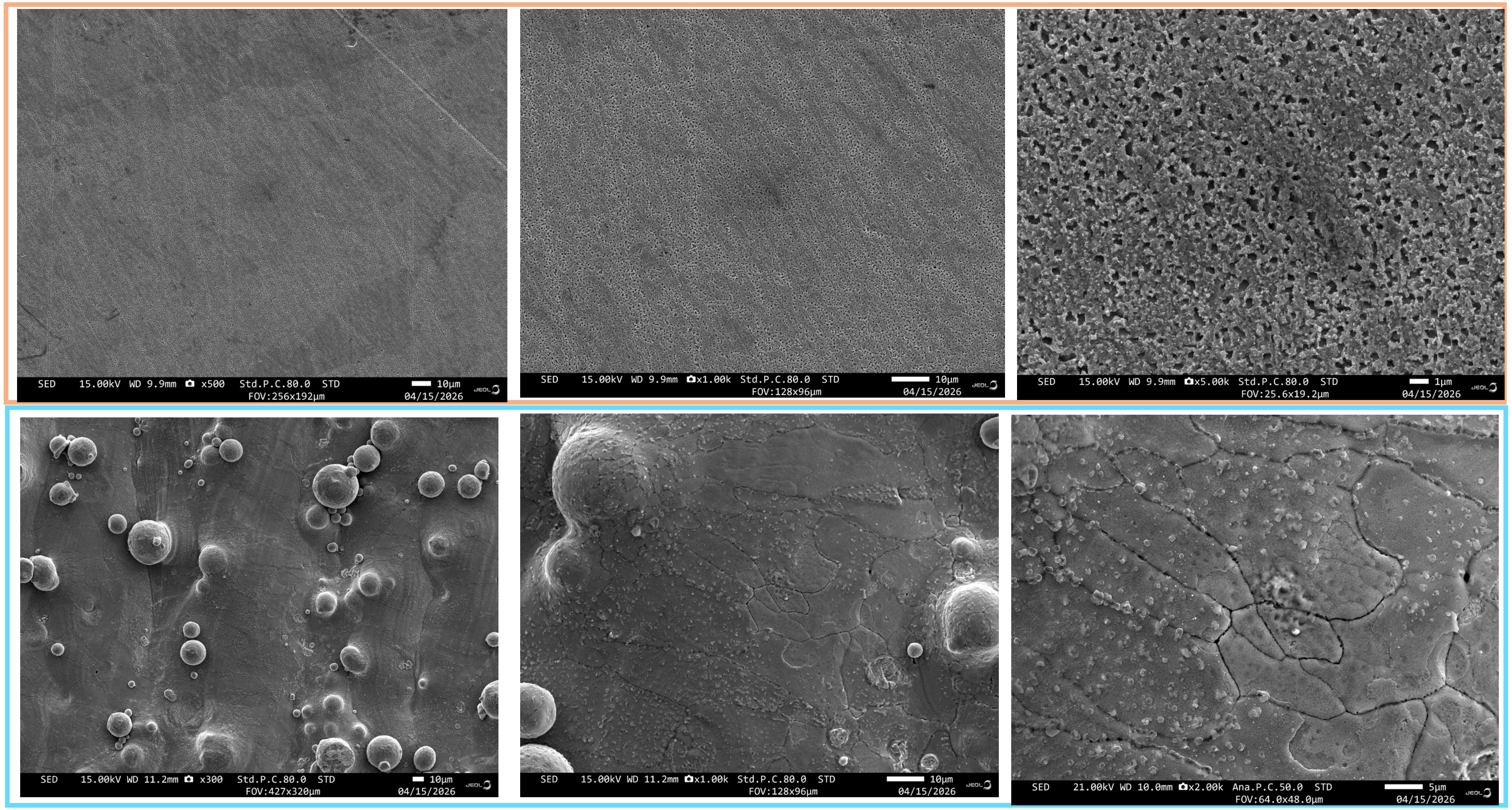


In contrast, notable subsurface intergranular attack was observed in wrought SS316H at grain boundaries intersecting the surface

Post-corrosion surface morphology, Static samples: as-deposited vs. machine-cut

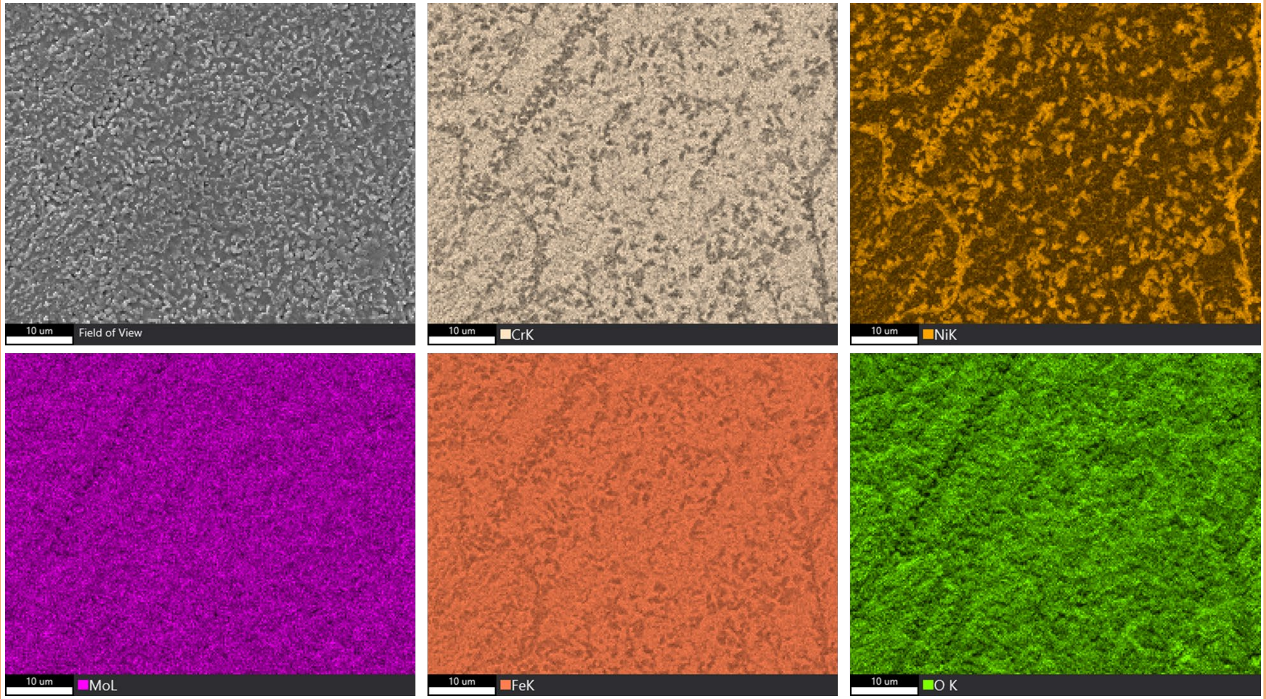
Machine cut Surface

As-Deposited Surface

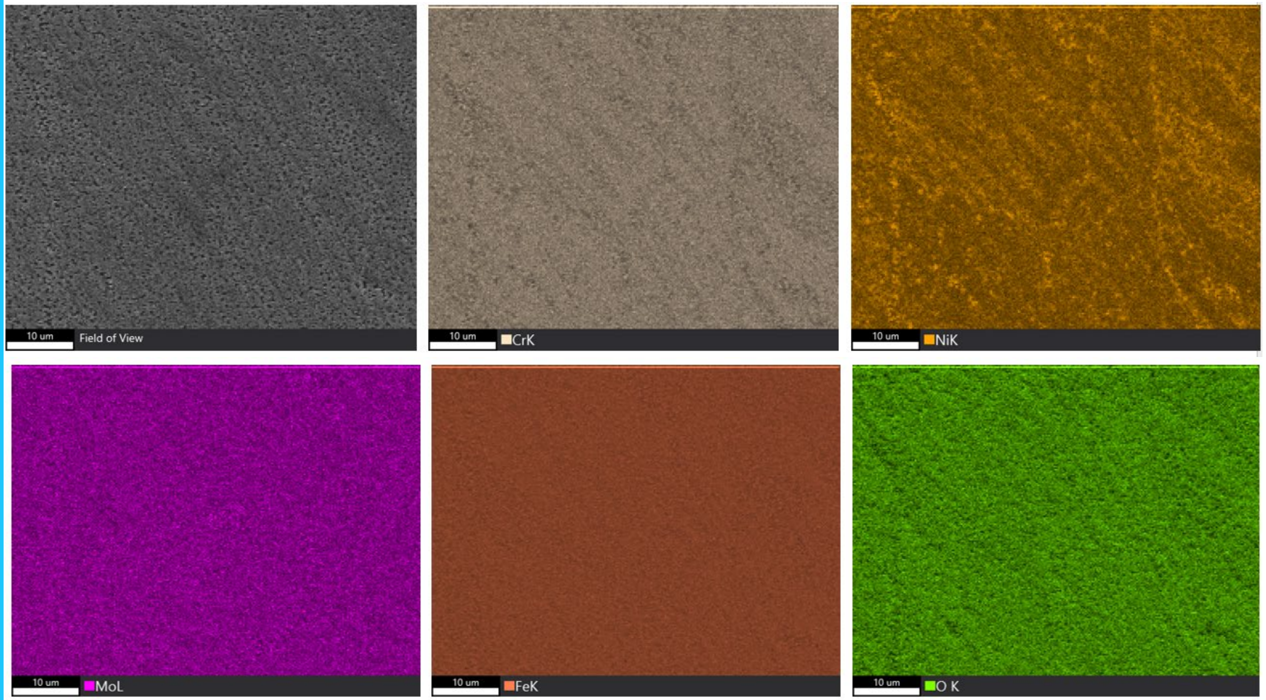


Post-corrosion EDS analysis - elemental redistribution comparison

Machine Cut Surface, Flowloop



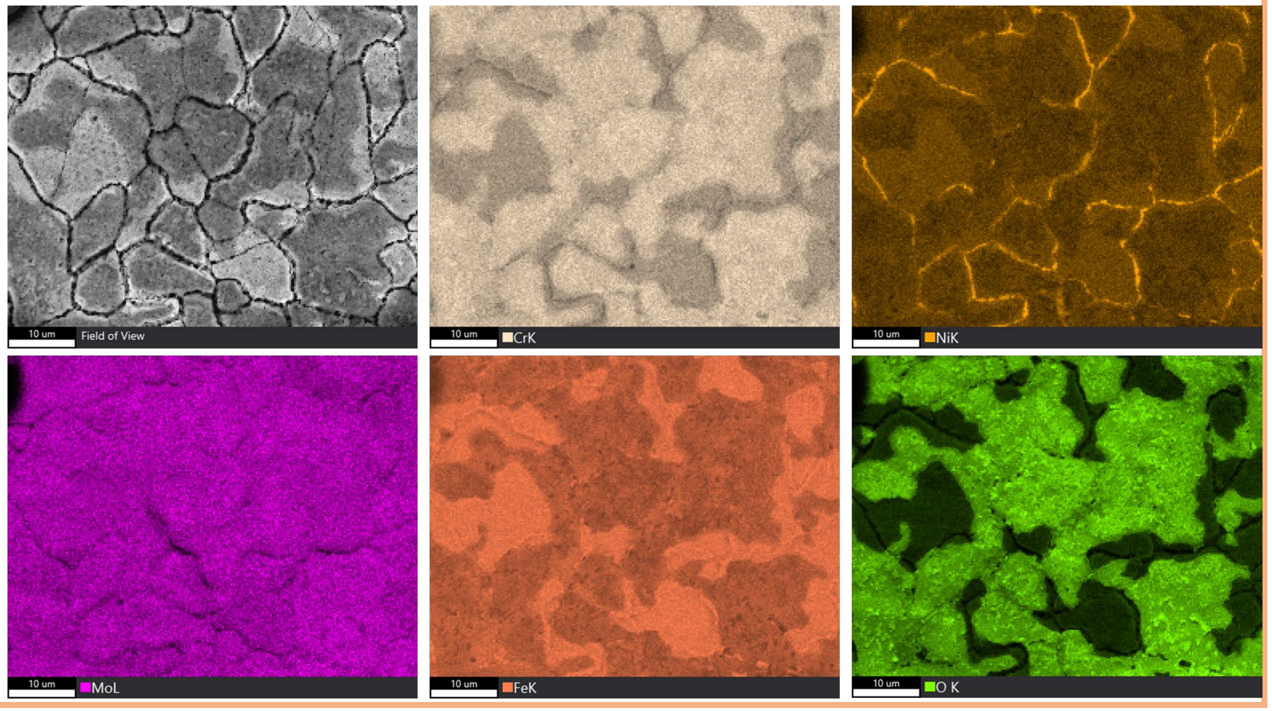
Machine Cut Surface, Static



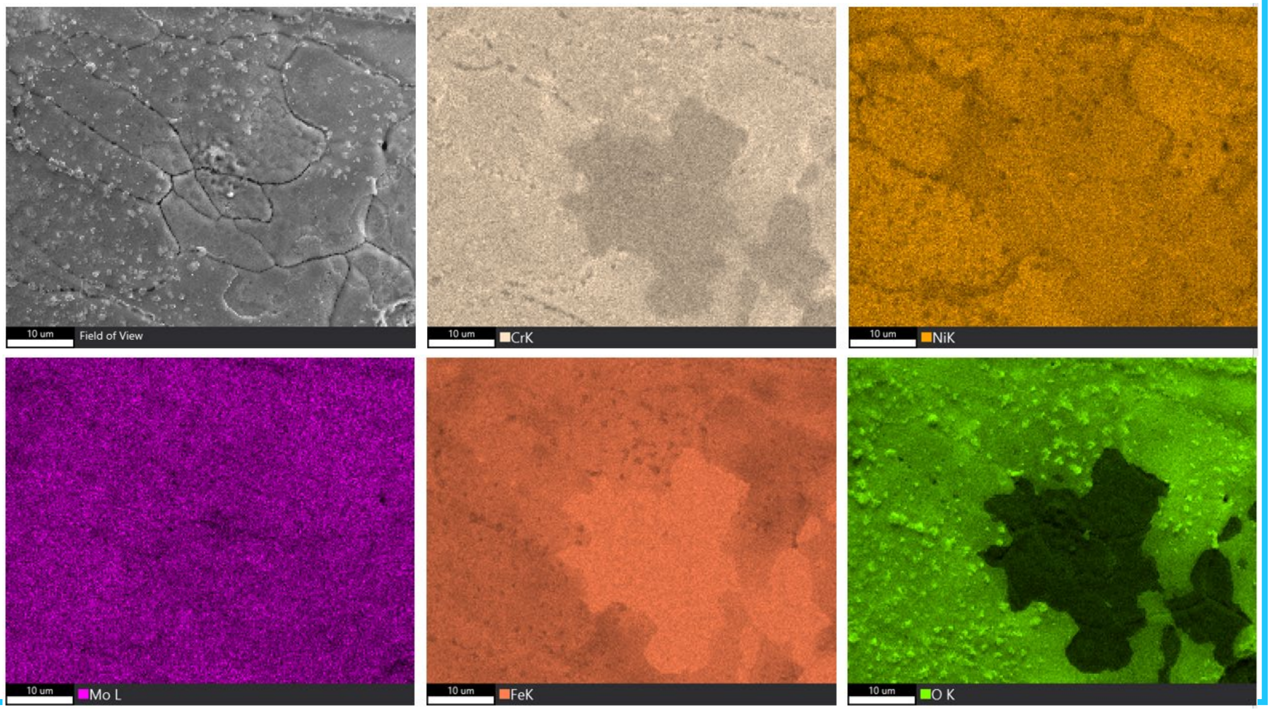
Flow conditions promote more pronounced corrosion attack compared to static salt exposure

Post-corrosion EDS analysis - elemental redistribution comparison

As-Deposited Surface, Flowloop



As-Deposited Surface, Static



Flowloop and static exposures exhibit distinct elemental redistribution patterns, highlighting impact of convective flow on corrosion evolution

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Summary and Next Steps

Summary

- Thermal convection created coupled temperature and mass-transport gradients that influence corrosion evolution
- Flowing LiCl-KCl salt produced greater net mass loss than static exposure under these tested conditions
- AM and wrought SS316H followed different degradation pathways: AM remained more surface-limited, while wrought showed greater subsurface intergranular attack
- For AM material, starting surface condition mattered: machine-cut and as-deposited surfaces showed different morphology and elemental redistribution

Next Steps

- Complete surface characterization of all flowloop specimens to determine how local hot-leg temperature affects corrosion morphology and attack depth
- Conduct 500-hour comparative exposures in flowloop and static salt to evaluate corrosion propagation with time
- Expand material screening to wrought Alloy 244 under both flowing and static LiCl-KCl conditions
- Continue integrating in situ diagnostics to correlate salt chemistry evolution with material degradation

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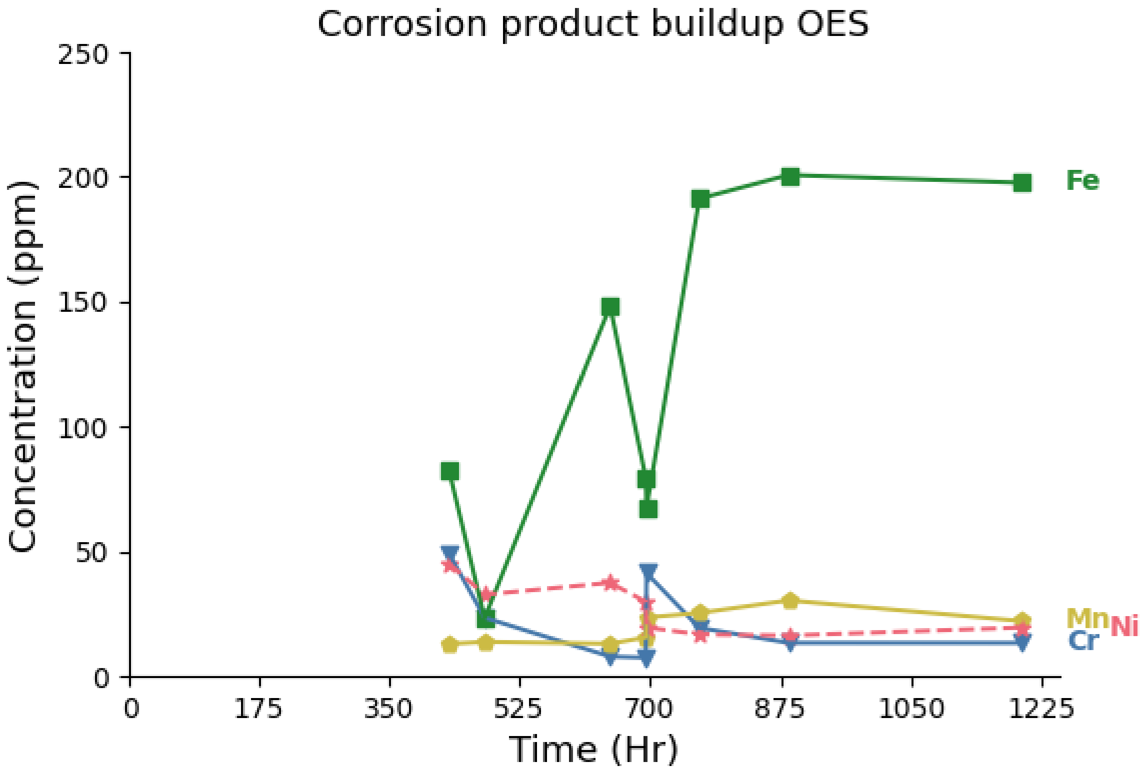
Salt purity monitored during 1200 h operation

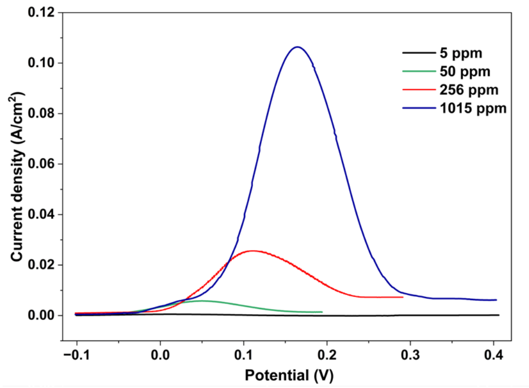
ICP analysis:

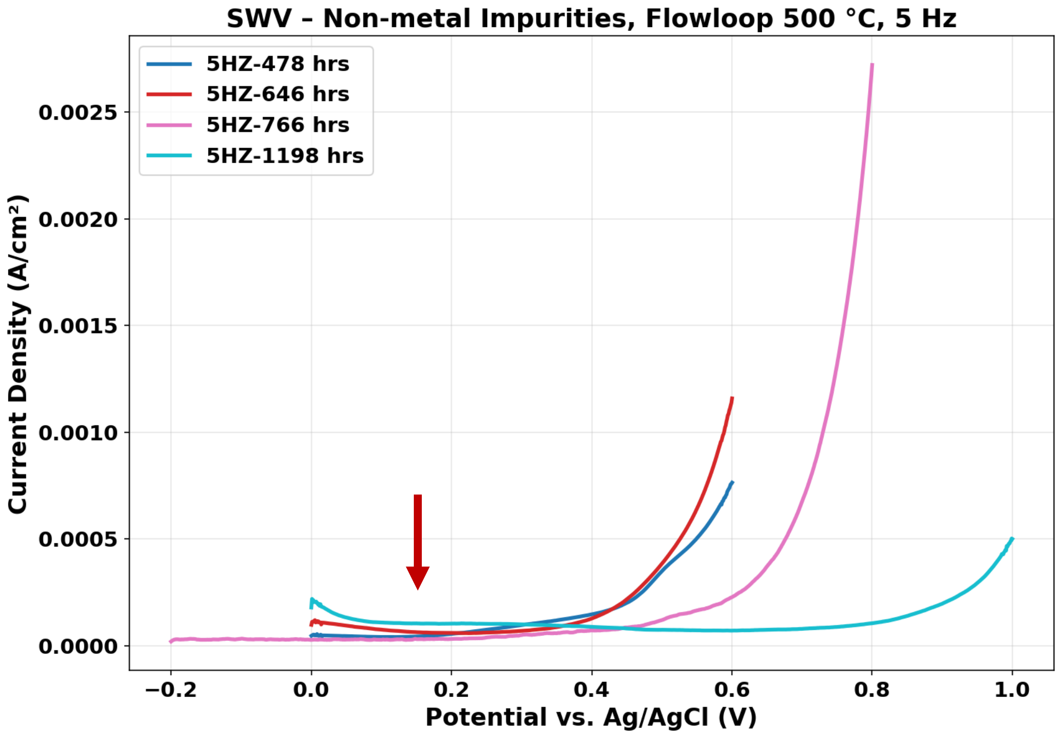


Overall increase in Fe concentration, other than dips at 500 and 700 hours time stamps. Ni is detected with ICP not Echem analysis. Mn trend agrees with Echem data

Salt purity monitored during 1200 h operation

- Oxide impurity was also tracked in flowloop salt throughout the operation using SWV
- Absence of peak around 0.2 V shows the oxygen is below detectability limits, 5ppm (as seen in our previous work)





SWV indicates that oxide concentration is below detectability limits