Nuclear Materials and Nuclear Fuel Cycle Center

VIRGINIA TECH

Lessons Learned From Operation of A Forced Convection Chloride Molten Salt Loop

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Our Current Research/Capabilities

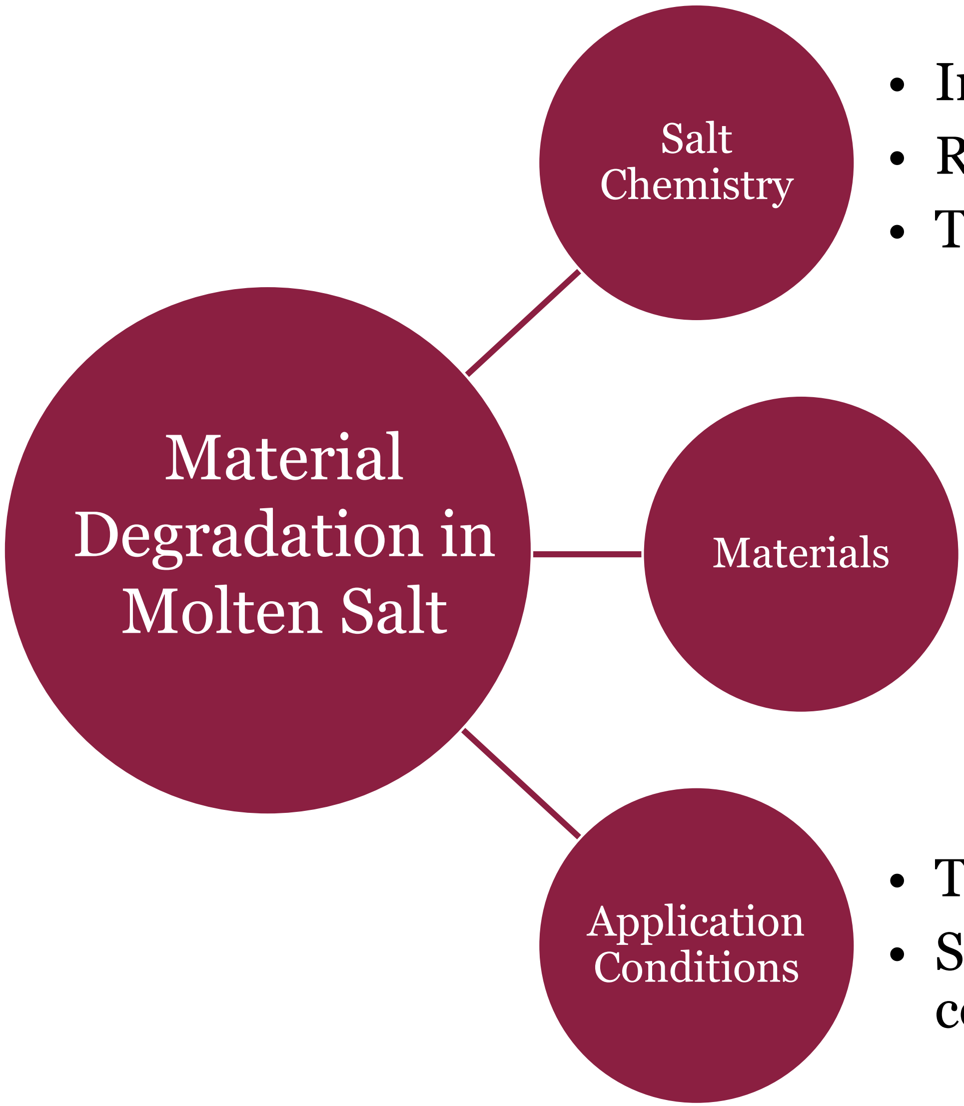
- **On-line salt purification/chemistry**
  - Metal impurity removal
  - Non-metal impurity control/removal
  - Phase diagram/material solubility
- **Salt/alloys interactions (Corrosion)**
  - Redox potential control
  - Thermodynamic data measurements
  - Corrosion kinetic data measurements
  - On-line corrosion measurements (flowing and static tests)
  - Graphite degradation in molten salt
- **Material separation**
  - Oxide reduction
  - Electrochemical separation
  - Liquid/liquid separation
- **Salt Impurity measurement**
  - Metal impurities
  - O/H impurities
  - S/C impurities
- **Model development**
  - Corrosion model in molten salt loop
  - Thermodynamic model for fundamental data
  - Electrochemical separation model for salt purification
  - Safeguards model for molten salt systems
- **Measurements of Salt properties**
  - Liquid salt density,
  - thermal conductivity, heat capacity, latent heat
  - Materials solubility
  - Melting/freezing temperature,
  - Phase diagram
  - Transport properties, ion diffusion coefficient
  - Corrosion kinetics
  - Vapor pressure, vaporization rate
  - Liquid salt viscosity (**method not validated)**

Chloride Molten Salt Advantages and Challenges

Chloride molten salt is the promoted heat transfer fluid (HTF) and thermal energy storage (TES) salt in nuclear and concentrating solar power plants (CSP).

Advantages

- ✓ Lower melting point
- ✓ Higher boiling point
- ✓ Lower vapor pressure
- ✓ Not flammable
- ✓ **Low cost**
- ✓ **Non-toxic**
- ✓ **Good high-temperature thermal stability**



Salt Chemistry:
- Impurities
- Redox potential
- Thermodynamic properties

Materials:
- Compositions
- Fabrication process
- Manufacturing process

Application Conditions:
- Temperatures
- Static or dynamic flow condition

Problem Statements

Histories

- Focused on fluoride salt in MSR
- Focused on nitrate salt in CSP
- Most corrosion work was performed in static conditions
- Materials development on Ni-based alloy
- The flow-induced corrosion tests need to be traced back 1950s-1970s

Others Work

- Different kind of chloride molten salt (composition and ratio)
- Most of the works were focused on static crucible emersion tests
- Commercialized material testing on both Fe and Ni based alloy

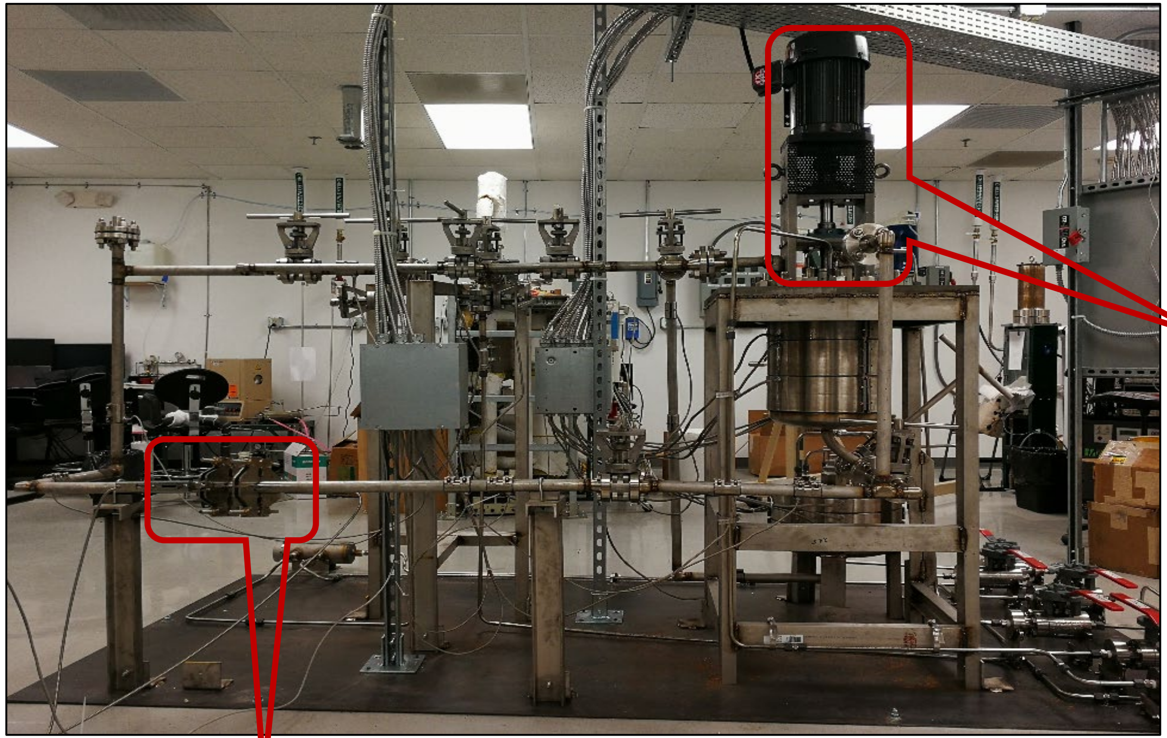
Research Gap

- Scattered of corrosion data in chloride molten salt
- Forced convection chloride molten salt loop design operation experience
- Dearth of flow-induced corrosion data

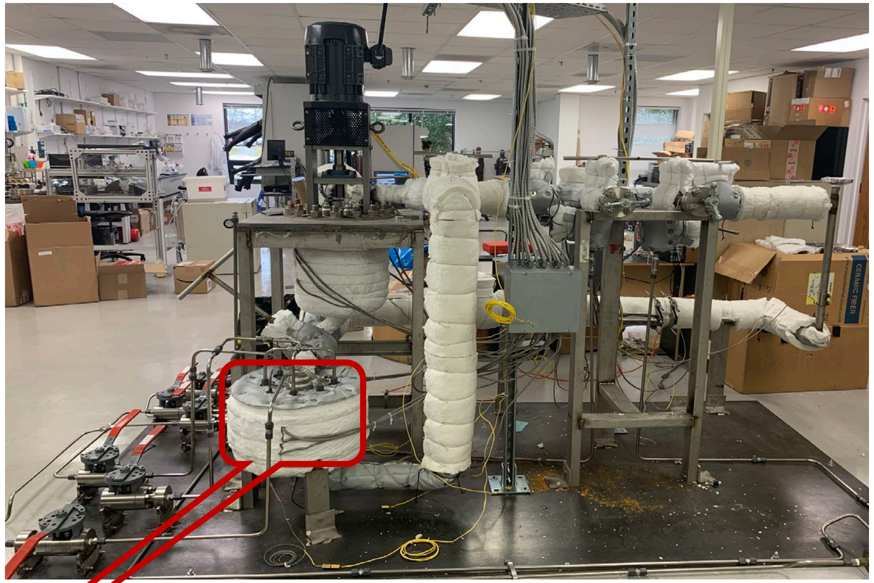
My Work

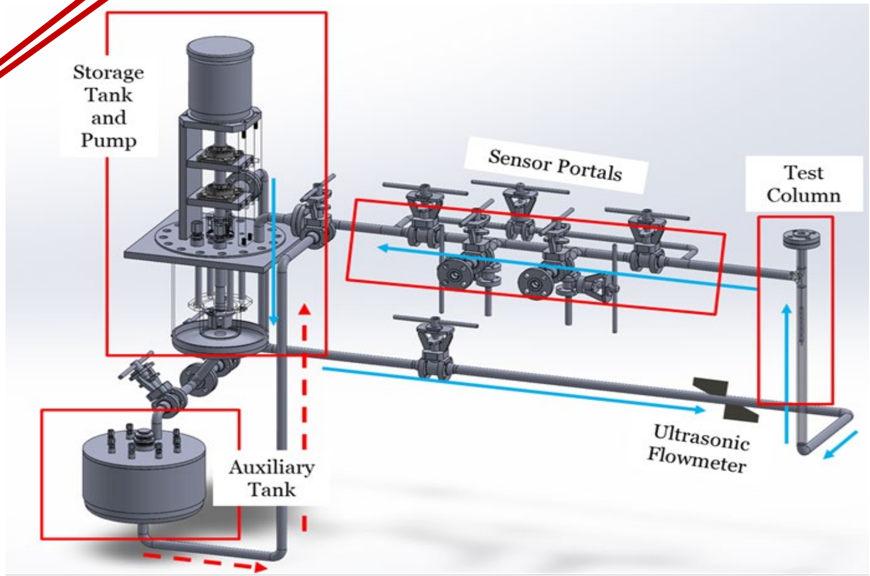
- Leverage understanding of forced flow-induced corrosion.
- Learn and share lessons on chloride salt loop design and operation.
- Two kinds of iron-based alloy A709 and SS316 were tested under flow condition.

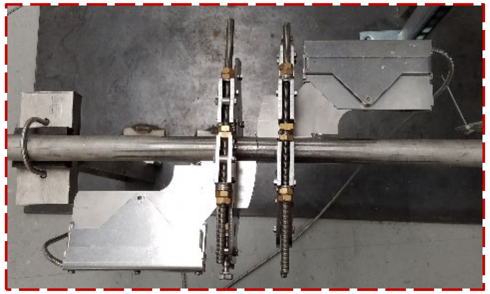
The Forced Convection Chloride corrosion Loop (FCL)



Molten Salt Pump



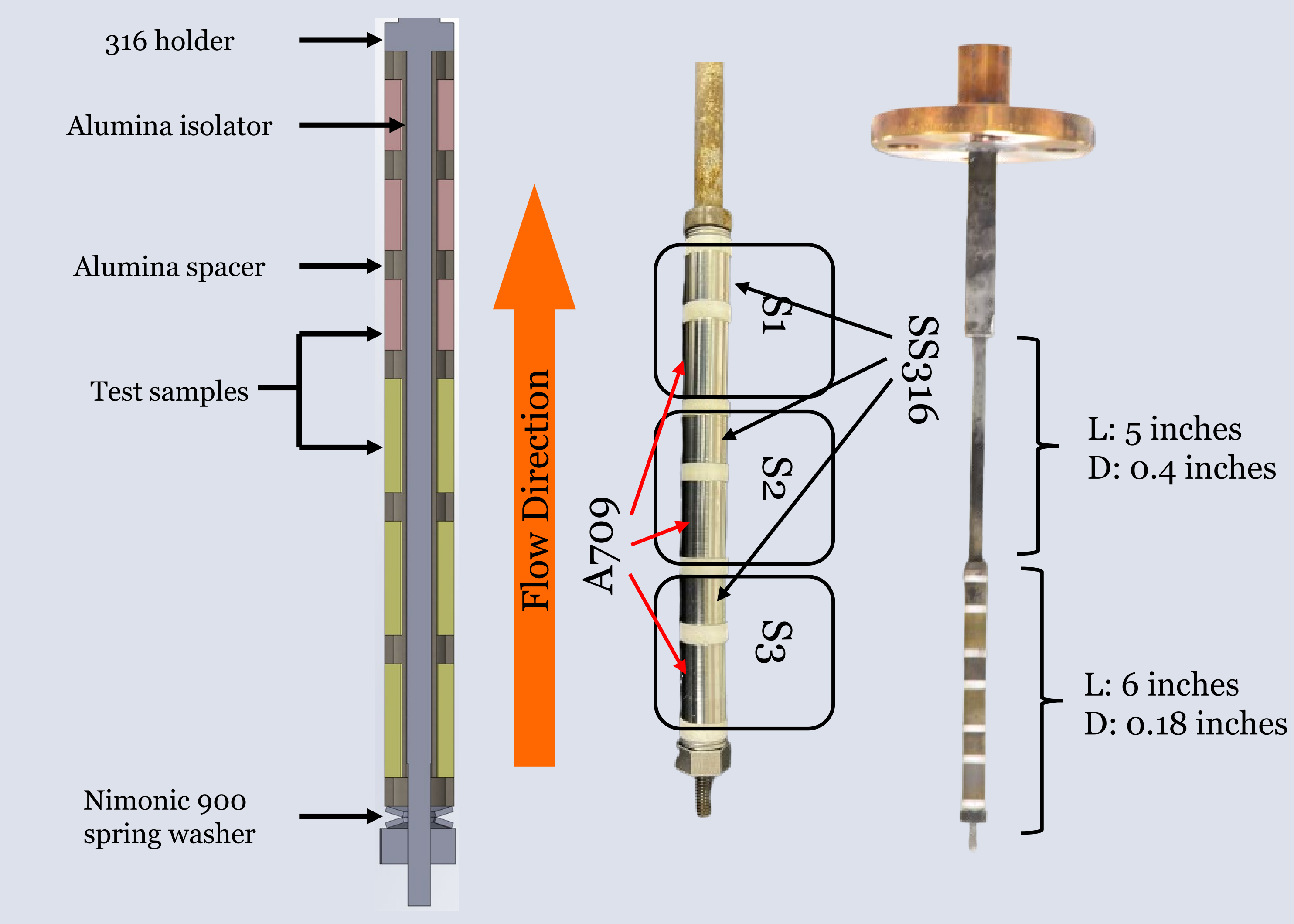




Ultrasonic Flowmeter

Auxiliary Tank

Flow-induced corrosion



wt%	A709	SS316
Cr	19.93	18-20
Ni	24.98	11-14
Fe	Bal.	Bal.
Mn	0.91	1-2.5
Mo	1.51	2-3
Si	0.44	0.3-0.65
Nb	0.2	-
Ti	0.04	-
Cu	-	0.75
N	0.148	-
C	0.066	0.03
P	0.014	0.03
B	0.0045	-
S	-	0.03

MSL Specification and Operation Condition

Capability	Parameters
Salt	$MgCl_2$-KCl-NaCl
Flow rate	0.7 L/s
Design temp.	700 °C
Main heater power	15 kW_{th}
Trace1 heater power	10.8 kW_{th}
Trace 2 heater power	3.6 kW_{th}
Salt volume	30 L (8 gallons)
Main piping	1-inch sch. 160
Pump	3 HP 0-60 Hz
Loop length	8 m

Experimental Condition

Temperature: 650 °C

Salt weight: 40 kg

Volumetric flow rate: 0.45 l/s

Flow rate at main pipe: 1.25 m/s

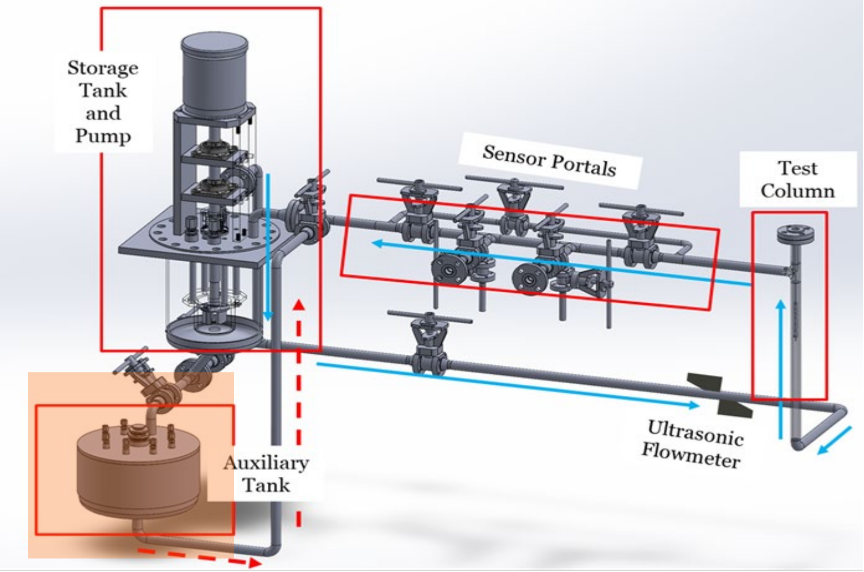
Flow rate at test column: 2 m/s

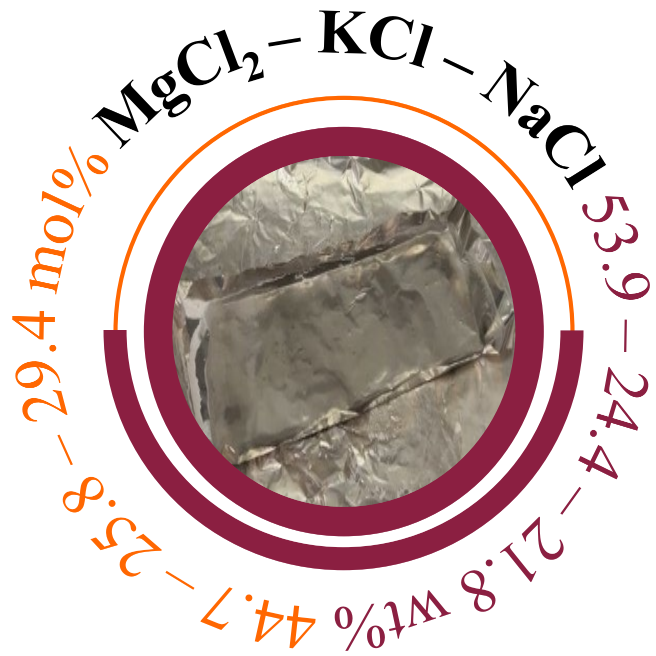
Flow Condition

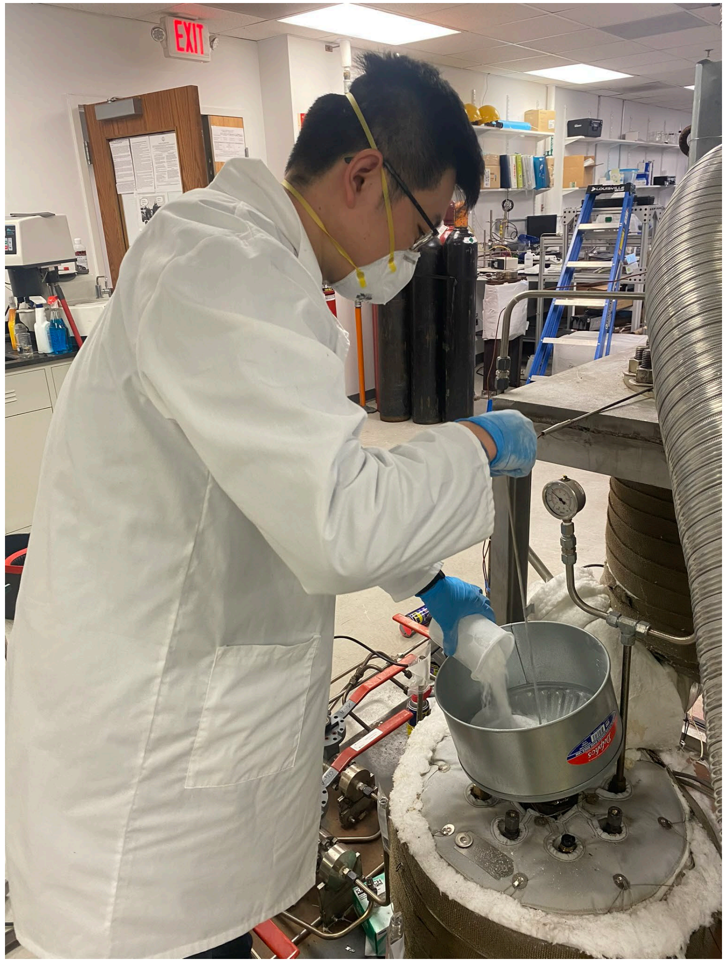
	Meter V m/s	Flow rate L/s	Loop Re	Test column V m/s	Test column Re
FCL-50	0.5	1.68	5657	0.8	3690
FCL-80	1.25	4.21	14892	2	9225
FCL-100	1.25	4.21	14892	2	9225

FCL Operation

Individual powder salts were purchased from Thermo Fisher Scientific with purty of 99%.







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FCL Operation

Loading | **Purification** | Transportation | Circulation | Drainage

Thermal purification with flow

↓

Loading 25+15=40 kg of salt

- 2.5-4.5 LPM flow rate
- Pressure 6.7 Kpa (1psi)
- Heating rate 1 °C/min

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Bubbling for 1 hour at 500 °C

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Heat to 600 °C

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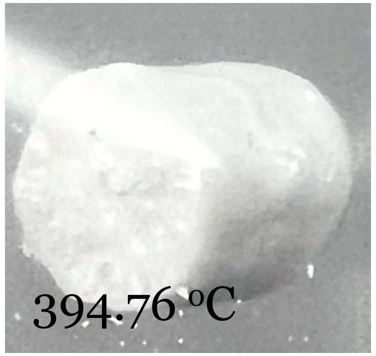
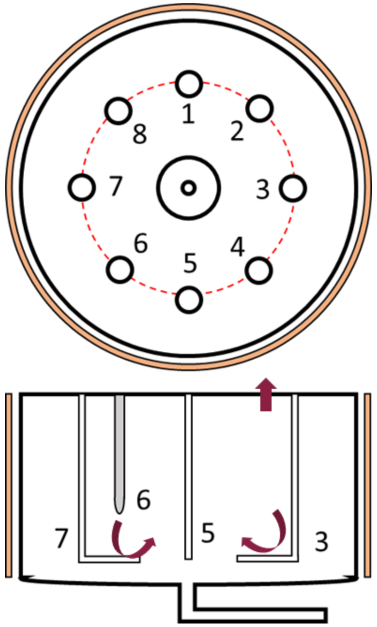
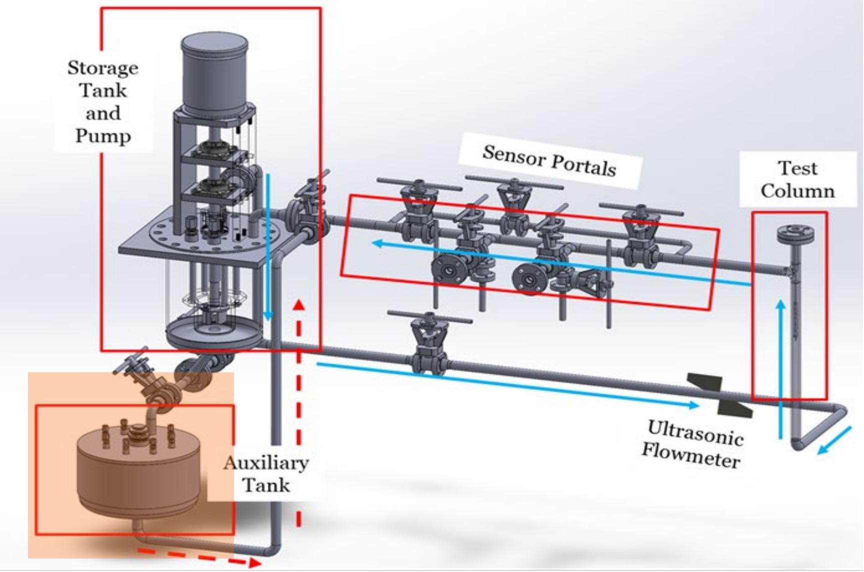
Preheat the transportation line and Sto. to 550 °C





1. Drain line
2. Pressure sensor
3. 5. 7. Purging line
4. Exhaust
6. Thermowell
8. Cover gas



Mole %	Na	Mg	K	Cr ×10-4	Mn ×10-4	Fe ×10-4	Ni	Mo ×10-4
Target	29.5	44.7	25.8	-	-	-	-	-
ICP	28.3	45.0	26.6	2.60	3.27	148	0	6.49

FCL Operation

Preheat the transportation line and Sto. to 550 °C

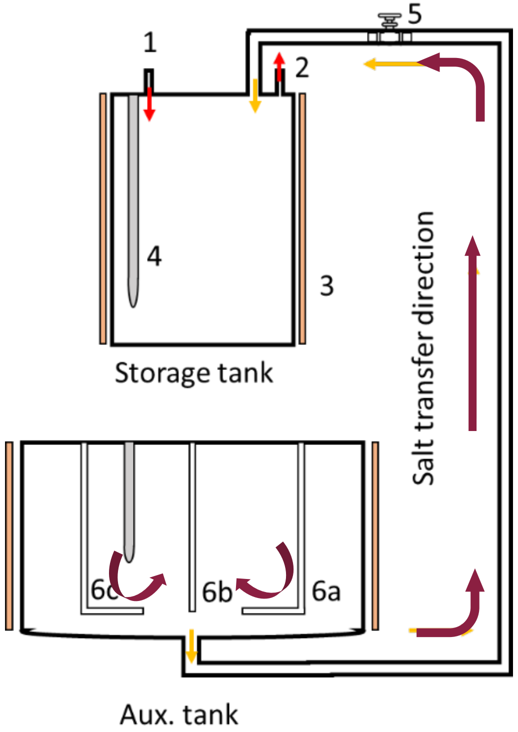
Pressurizing Aux. 1.5-2 psi

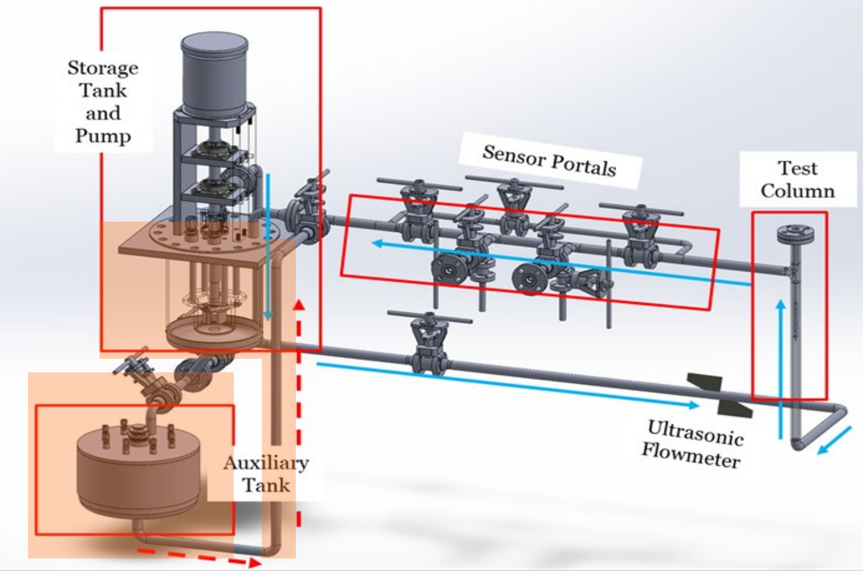
- Open vent line of Sto.
- Open salt Valve #5
- Flow rate at 7.5 LPM

Salt transfer complete at the gas flow suddenly increased to max.

1. Cover gas
2. Exhaust
3. Heater
4. Thermowell

6 a.b.c. purging line

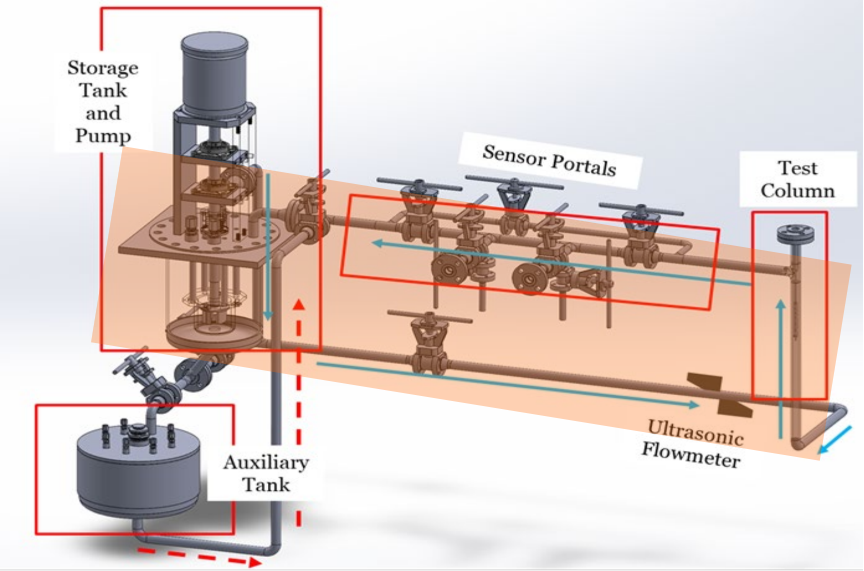


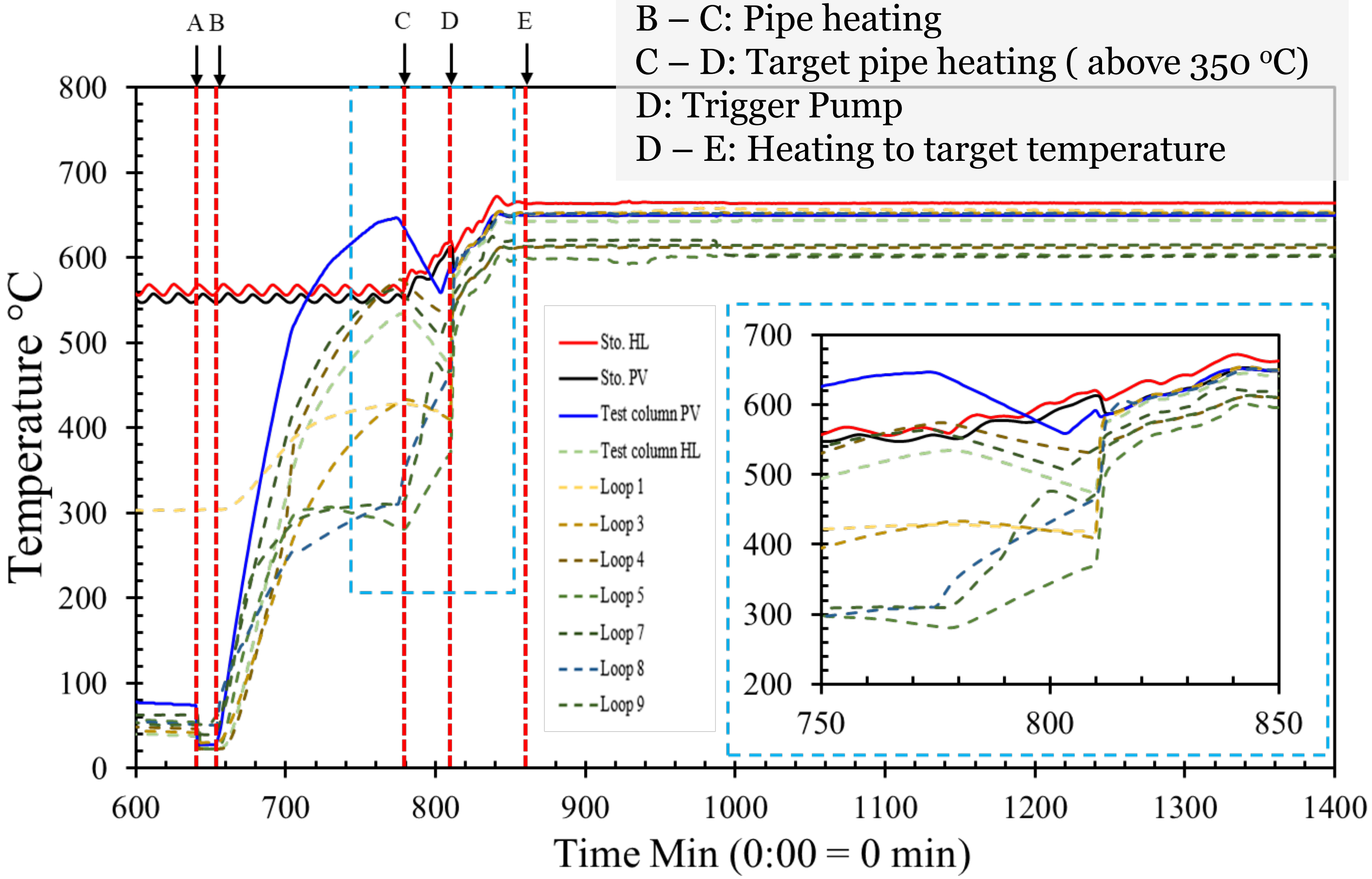


FCL Operation

Temperature

- Heaters could not uniformly warm up the metal pipe
- Flow will uniform the temperature
- Heaters at critical locations are required



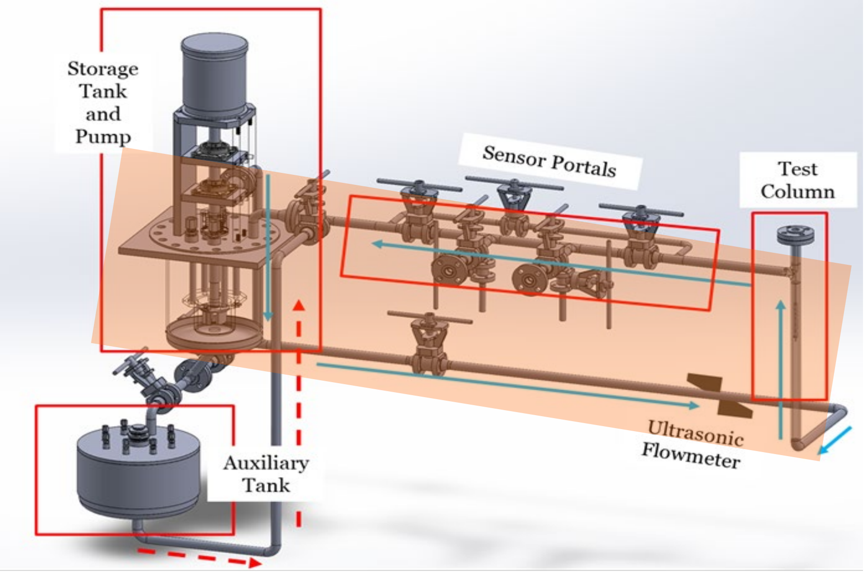


A – B: Switch thermocouples
B – C: Pipe heating
C – D: Target pipe heating (above 350 °C)
D: Trigger Pump
D – E: Heating to target temperature

Lessons From MSL Operation

Vapor Solidification

- Temperature at the top flange is about 350 °C
- Salt circulation in the Sto. tank generates salt vapor
- Cover gas flow is RT before injecting to Sto. tank
- Cover gas flow rate 0.7 L/min through a half-inch tube
- Solidification occurred and block the inlet



Valve leakage

- ORNL loop in 1960s, does not have any valves at the circulation path
- Valve is the most complex part that exposed to the flow
- High-temperature valve leaking is the crucial issue

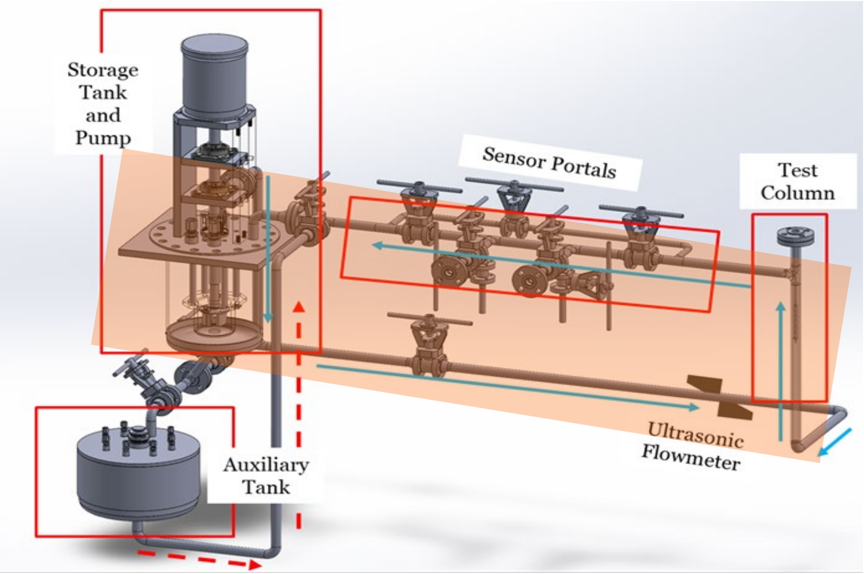
Lessons From MSL Operation

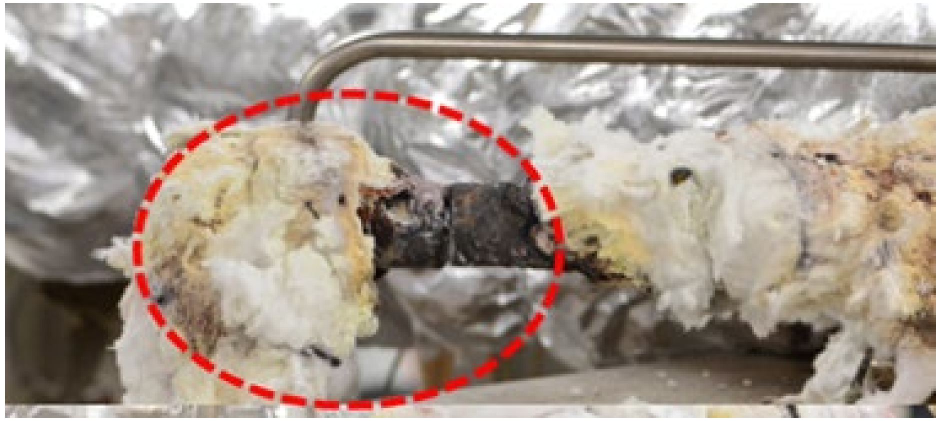
Electrolysis of chlorides

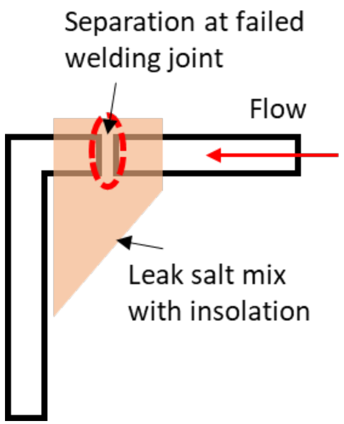
- Leaking salt wets the pipe heaters
- The heater connects to the loop body by the salt
- The heating element is broken, and the current passes loop
- Noxious odor: irritates throat and nostrils

Welding failure accident

- One welding joint is completely broken
- Stress and electrolysis
- Flow meter does not detect any flow rate change
- Salt solidifies around the opening to reduce or prevent the leak







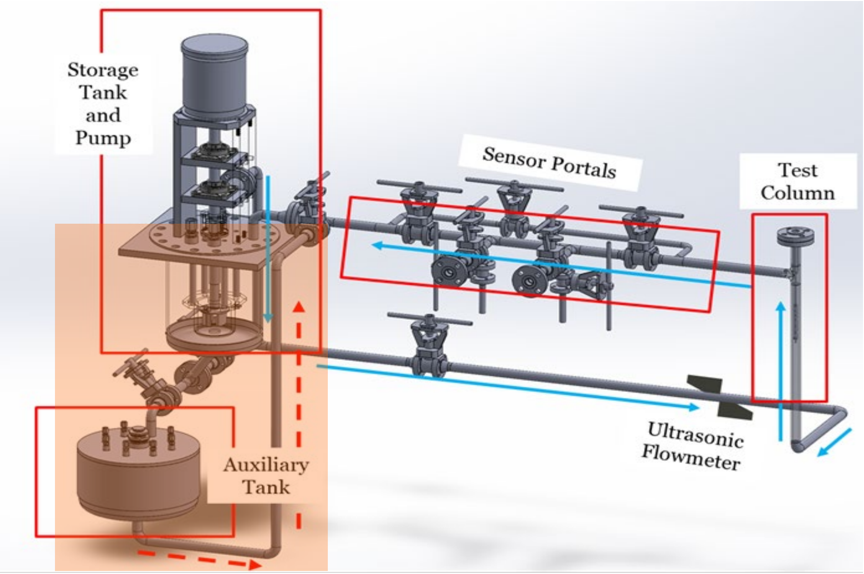
FCL Operation

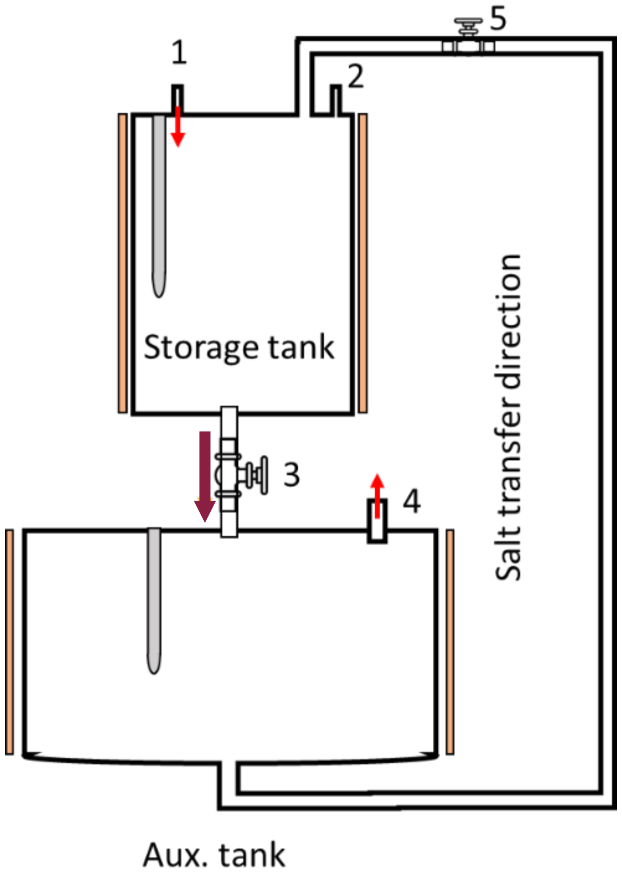
Warm up Aux. tank and drain valve above 550 °C

Vacuum and refill Aux. tank

- Open valve #3
- Open Aux. exhaust line 4

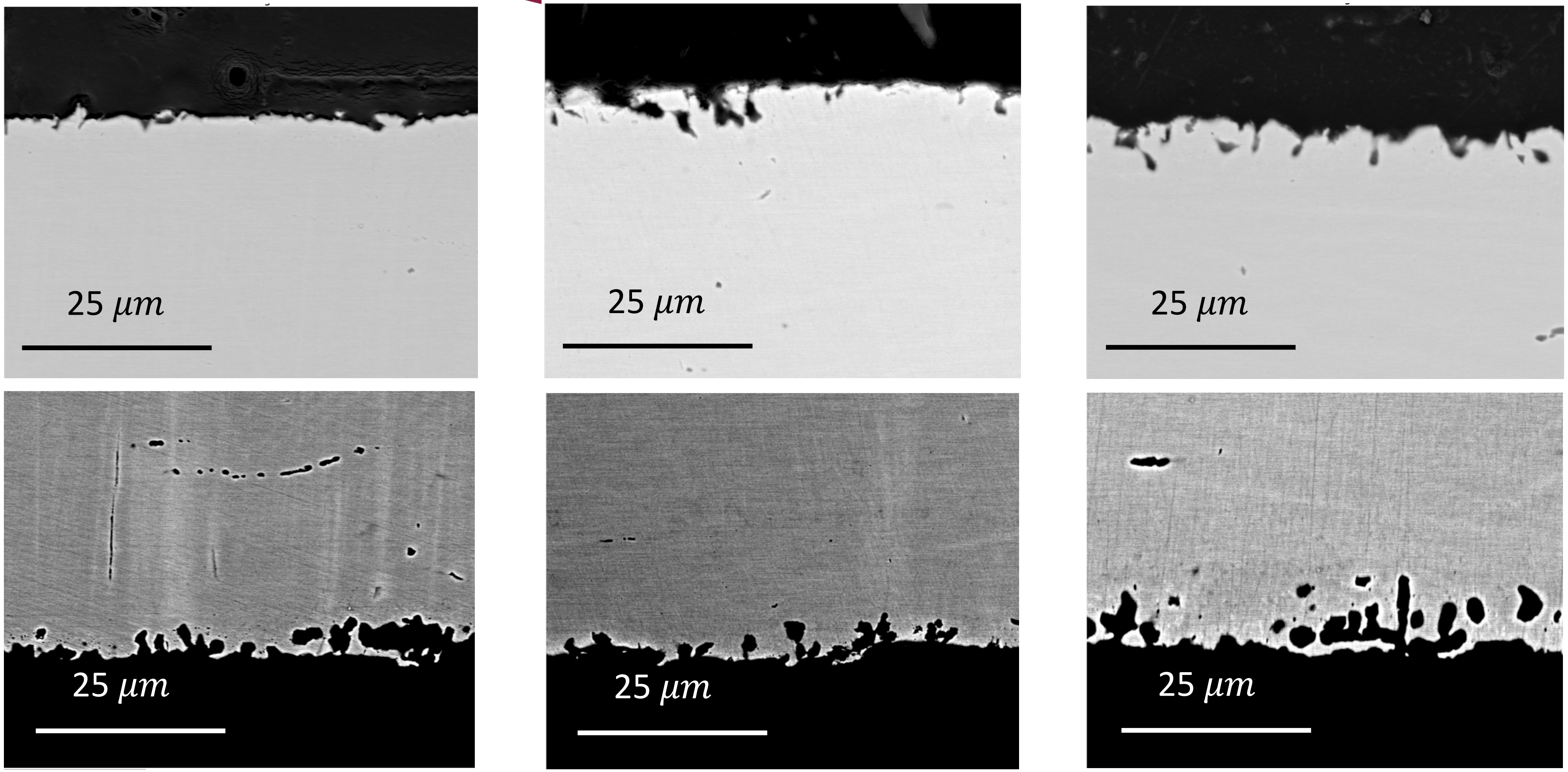
The drainage process is completed once the pressure is in Sto. and Aux. tank are the same

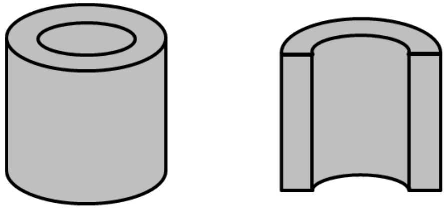




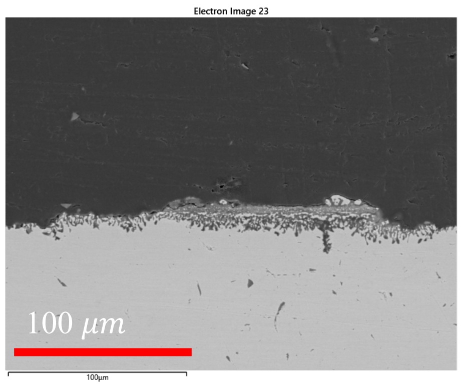
BED images of cross-sections FCL-100-SS316

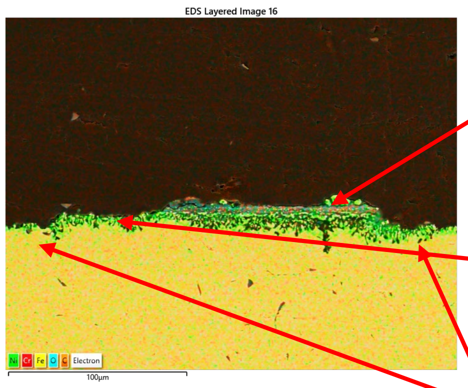
Flow Direction





The evidence of surface removal on FCL-80-SS316

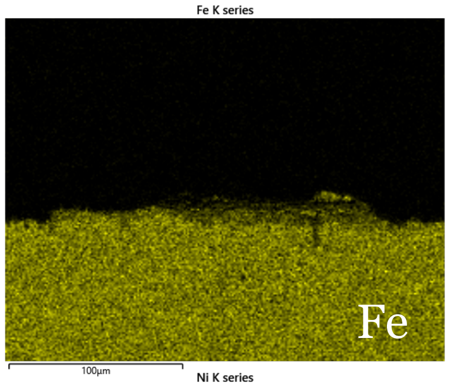


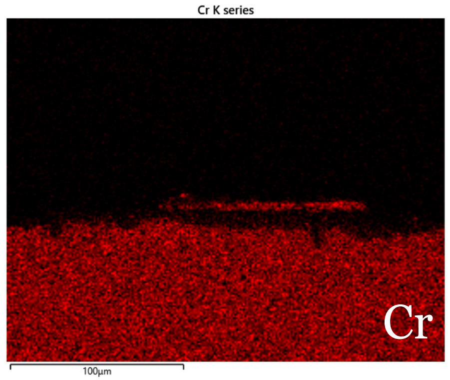


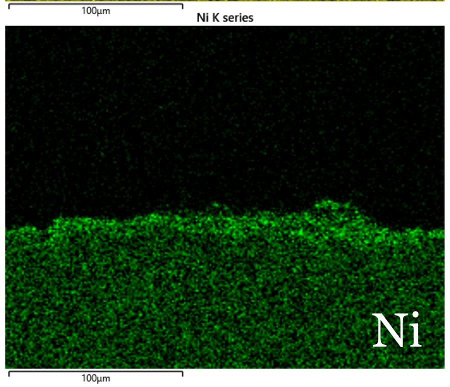
Fully grown corrosion layer

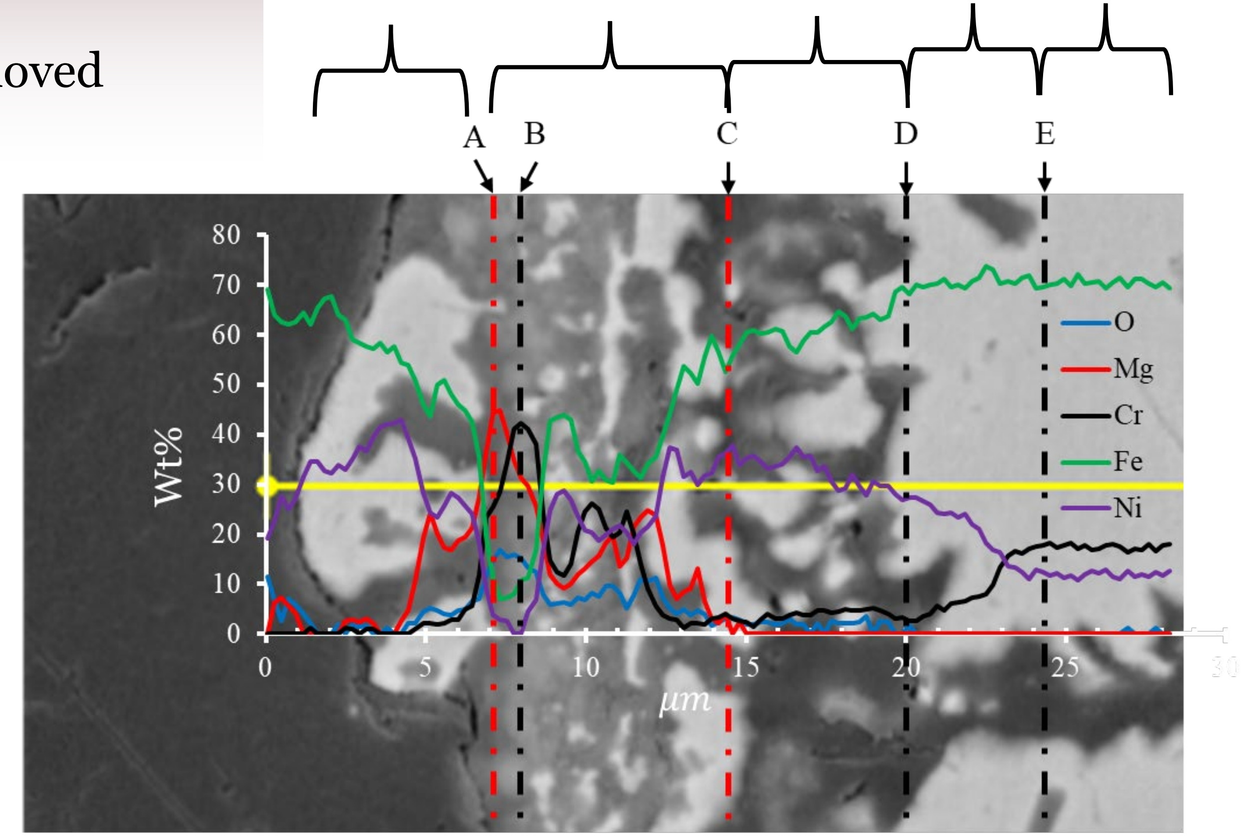
Cr depletion layer growth

Just removed surfaces

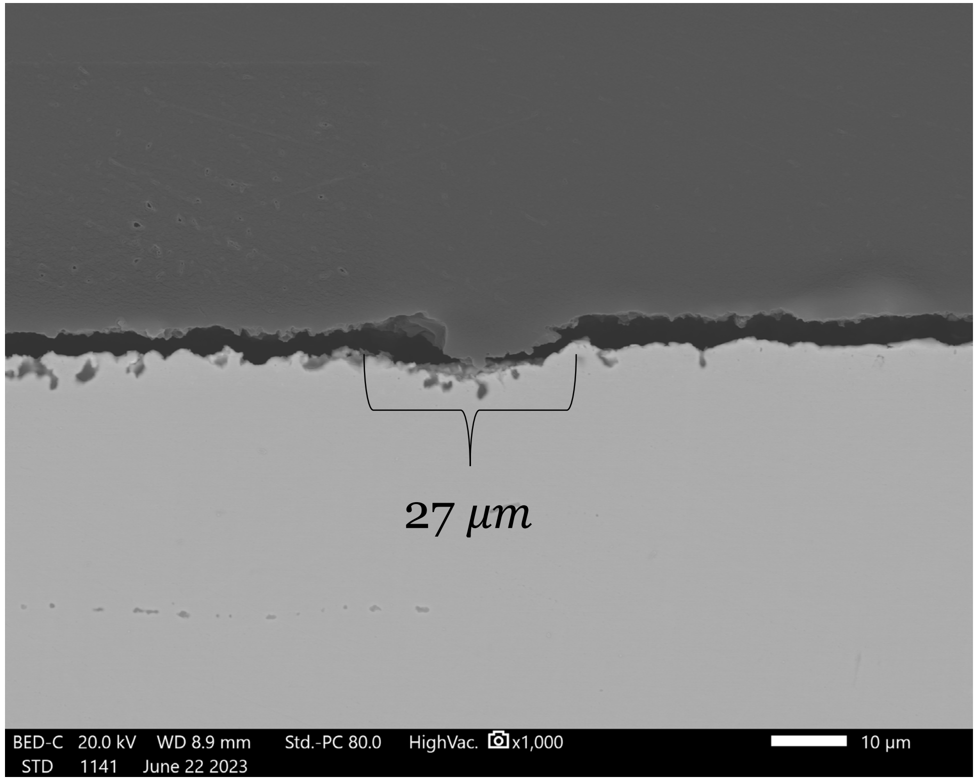


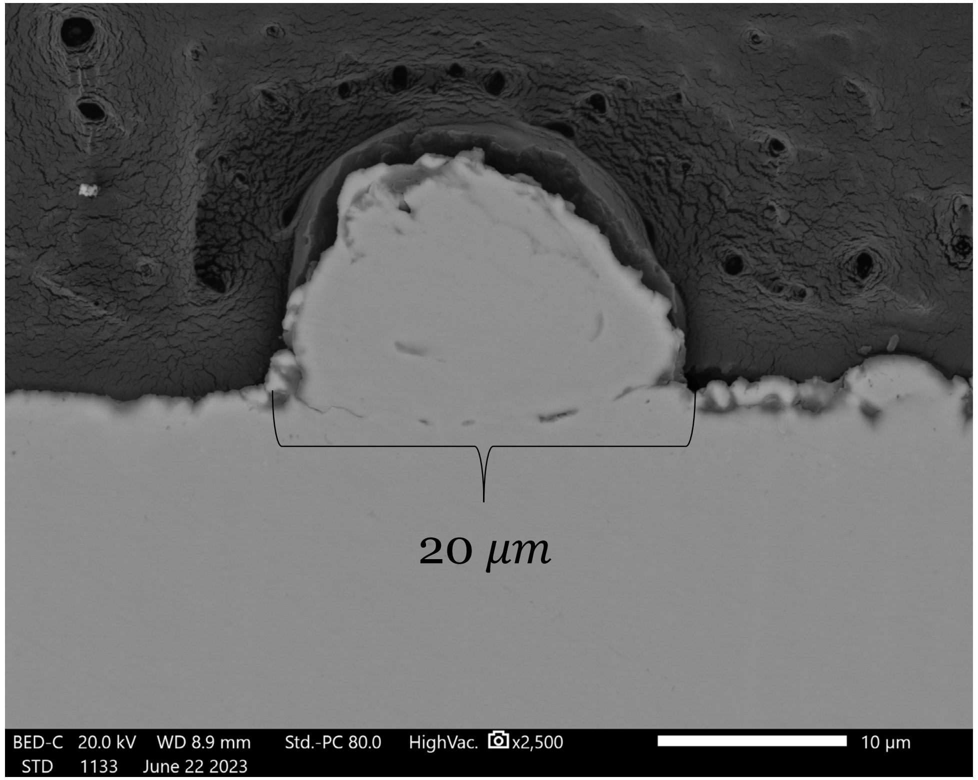






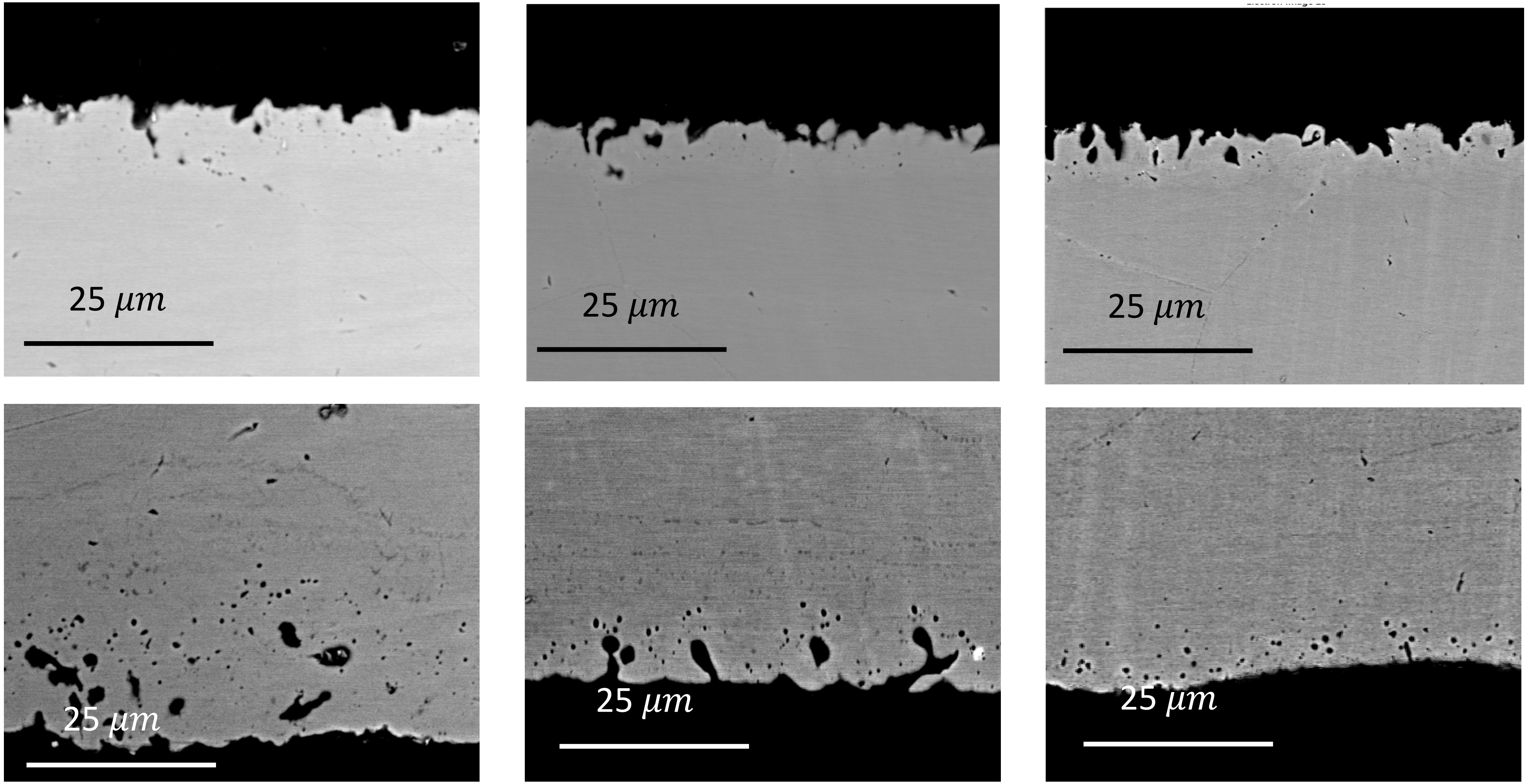
Partial removal evidence FCL-100-SS316-S1

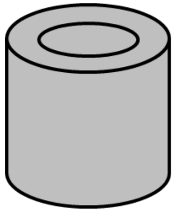




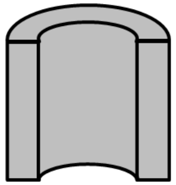
BED images of cross-sections FCL-100-A709

Flow Direction





Original

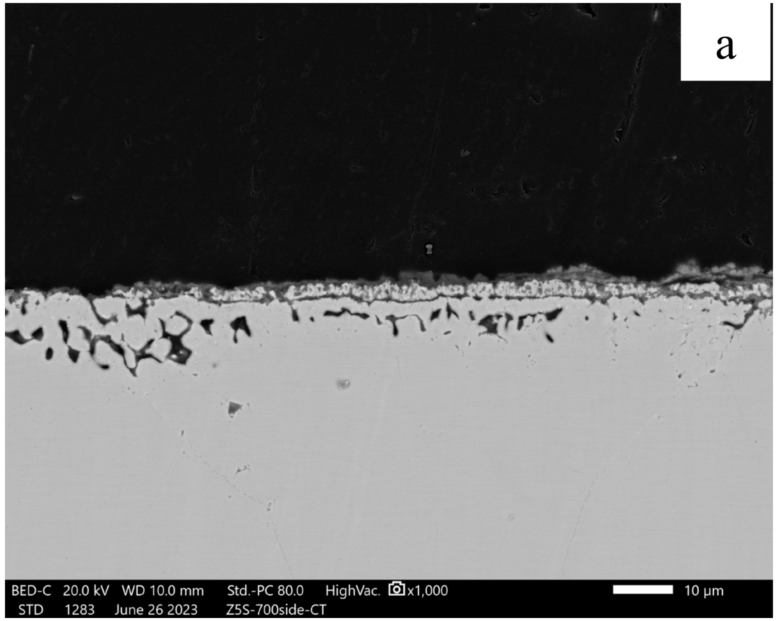


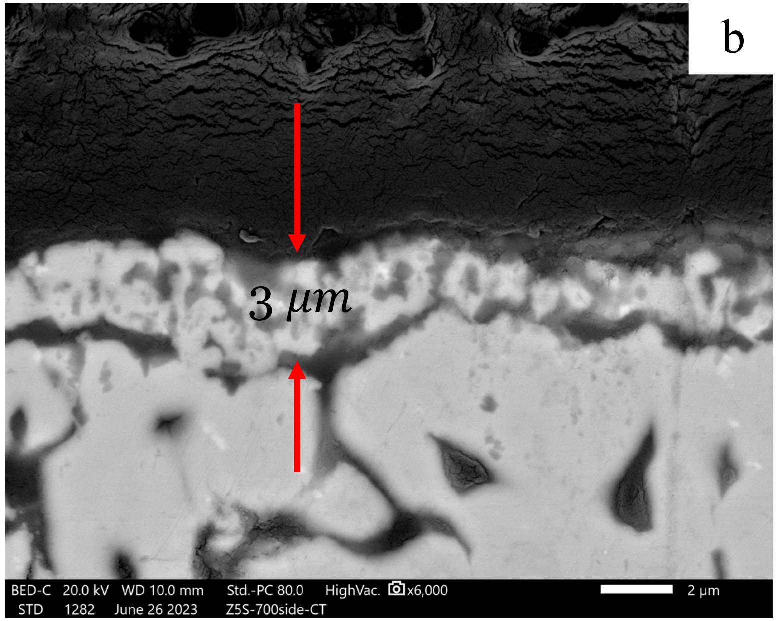
Vertical Cross section

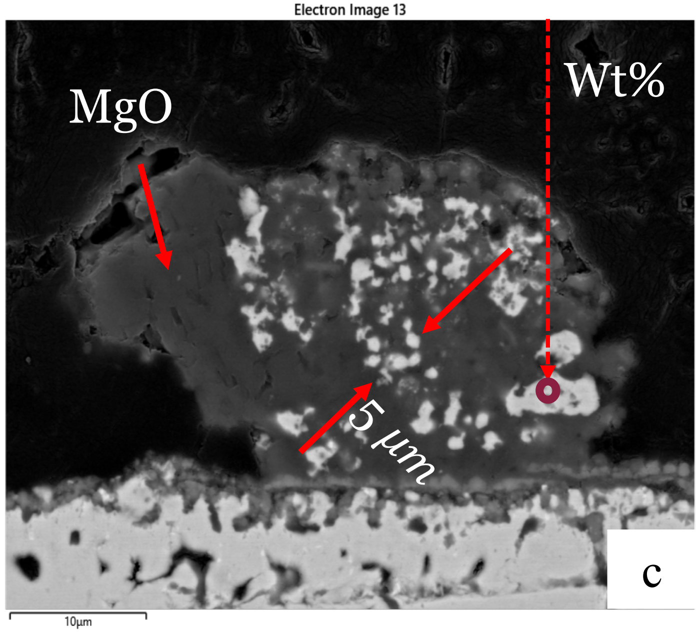
Surface removal evidence FCL-80-A709-S1

Flow Direction

Cr	Fe	Ni
2	50	40

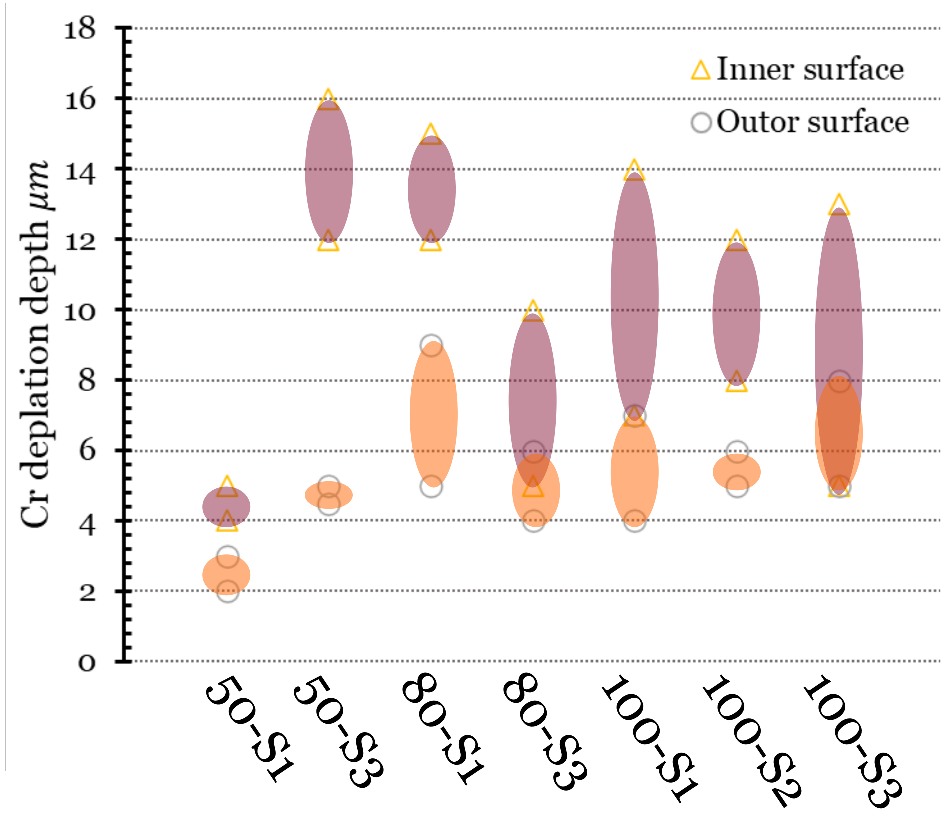




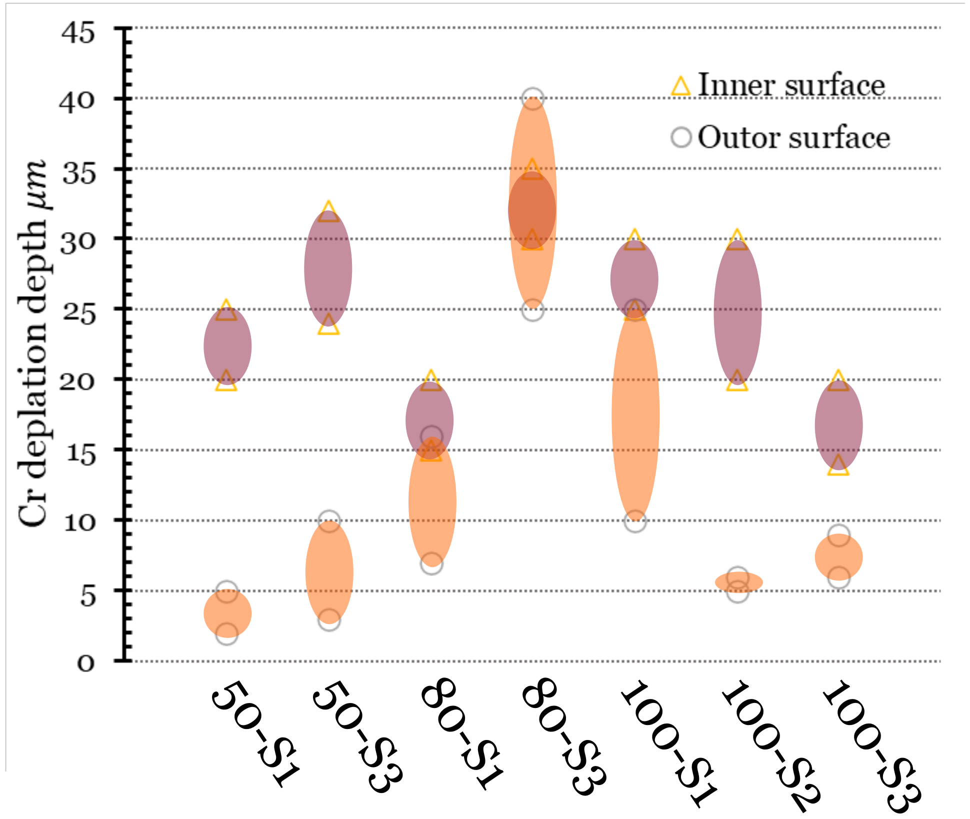


Summary of the Cr depletion depth

SS316



A709

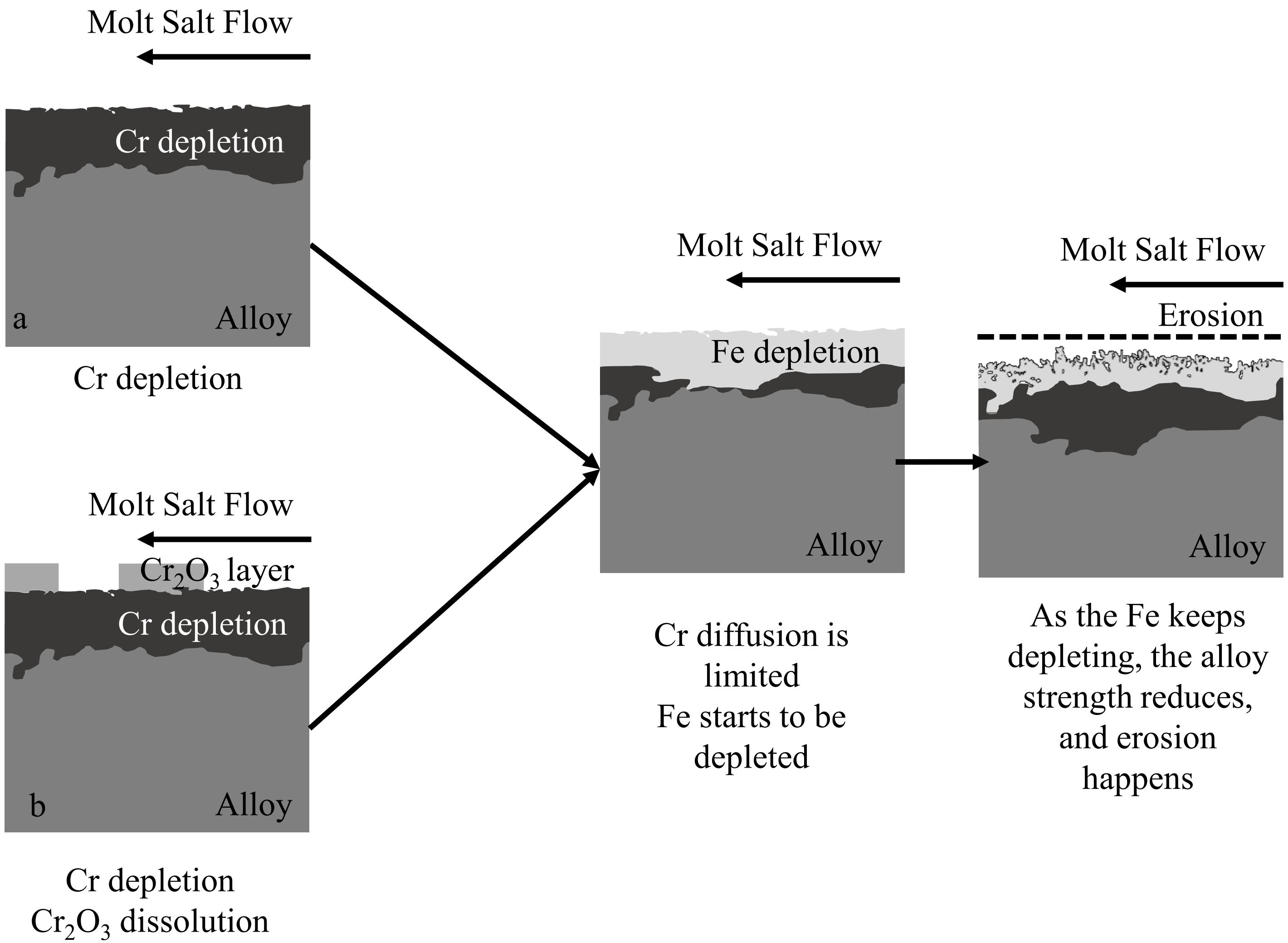


- Inner surface underwent typical static corrosion
- Outer surface underwent the FIC and erosion

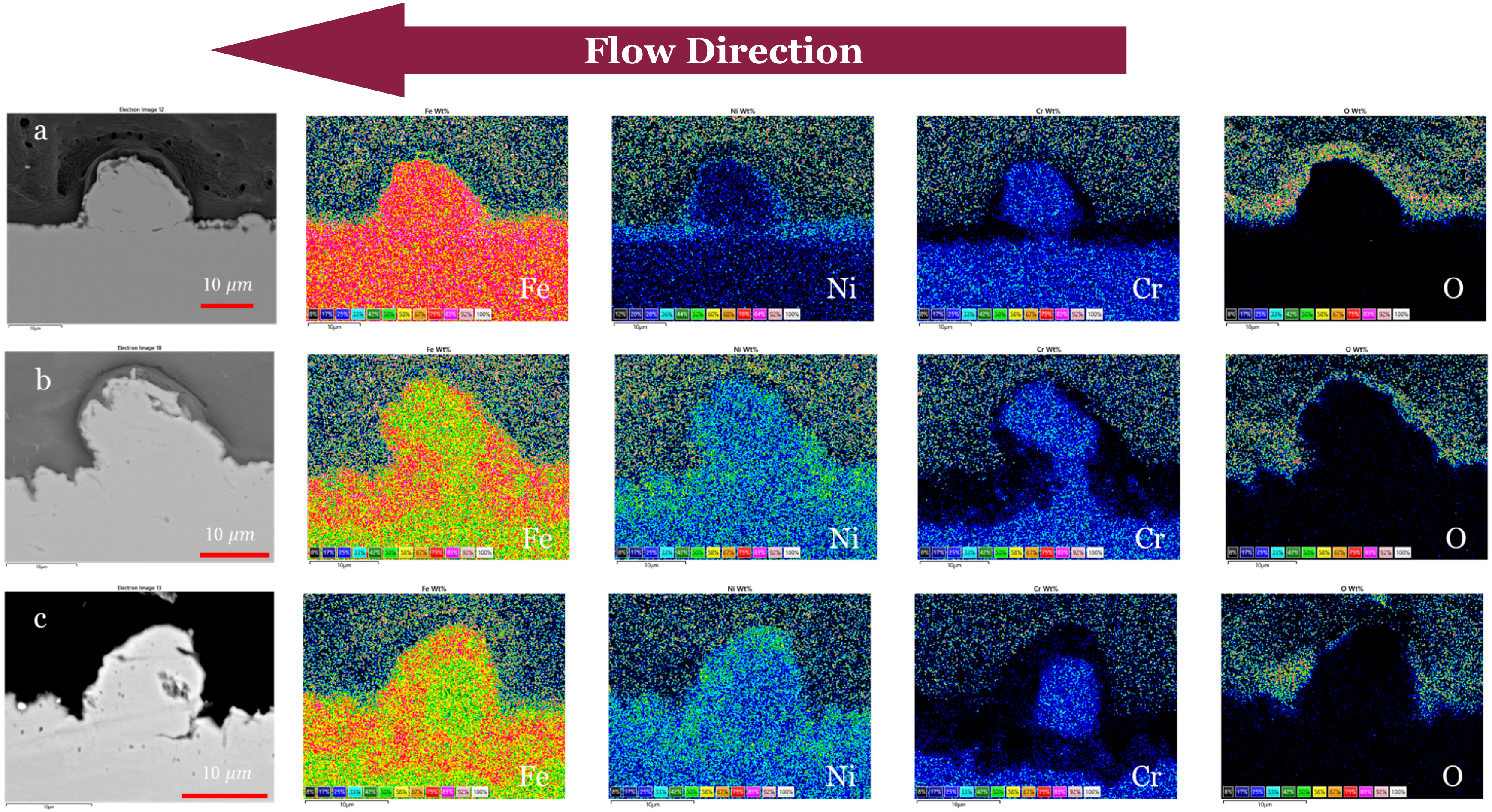
- The Cr depletion layers thickness are $D_{static} > D_{FIC}$

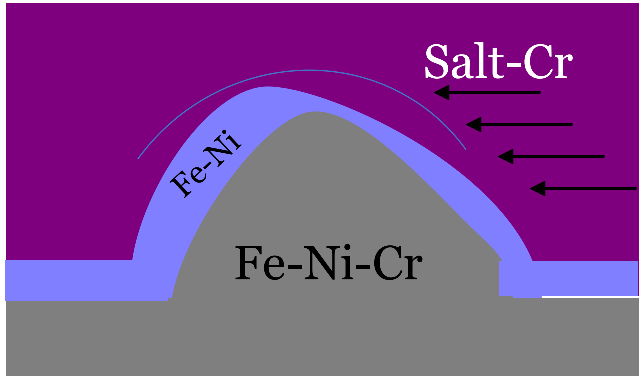
Revealing the FIC mechanism in molten salt

In FIC, the significant mass transfer of both corrosion products and oxidant results in the corrosion reaction occurring on the surface at a much higher rate than under static conditions.



Rib turbulator effect





Conclusions

- Preheating the large loop body is crucial. The quick heat loss can easily block the path.
- The molten salt has the advantage of preventing coolant loss for fuel loss accidents.
- The welding is the best sealing technology in high temperature molten salt application.
- High temperature valve for chloride molten salt needs to be developed.
- Chloride electrolysis incidents are a severe challenge for the testing loop.
- The flow-induced corrosion rate is more significant than static condition tests.
- The flow-induced corrosion process results in server Fe depletion.
- Erosion corrosion happens in the flow-induced condition.

Acknowledgments

U.S. DEPARTMENT OF ENERGY

Office of Science

- Dr. Ting-Leung Sham (Idaho National Laboratory): A709 alloys material
- Dr. Yanli Wang (Oak Ridge National Laboratory) : A709 sample and sample fixture manufacturing

Thank You!

Questions?