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Molten Salt Reactor PROGRAM

Salt and Materials Interaction

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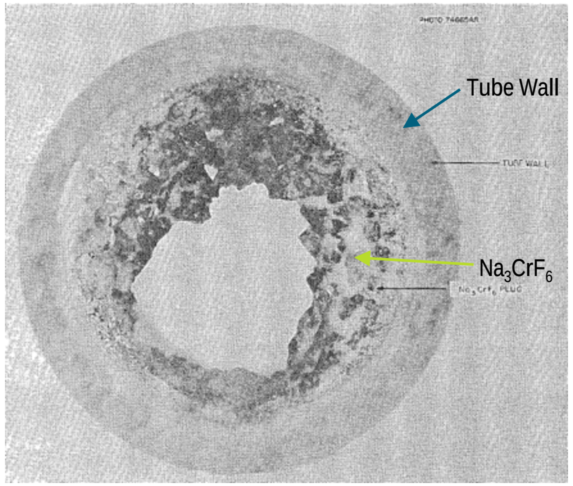
Acknowledgments

- **Funding: DOE Office of Nuclear Energy, Molten Salt Reactor Campaign**
- **ORNL team**
  - Dino Sulejmanovic: salt purification, handling and characterization
  - Rishi Pillai: modelling
  - Evangelia Kiosidou: electrochemistry (new)
  - Adam Willoughby: thermal convection loops
  - Yi-Feng Su and Michael Lance: characterization
  - Jim Keiser, David Holcomb, Lou Qualls: consulting
- **Kairos Power: salt (A. Kruizenga) and feedback (G. Young)**

What are we afraid of?	How do we study it?	How do we understand it?

What are we afraid of?

- Inconsequential: Cr surface depletion
- **Mass transfer**
  - Block flow in channel!

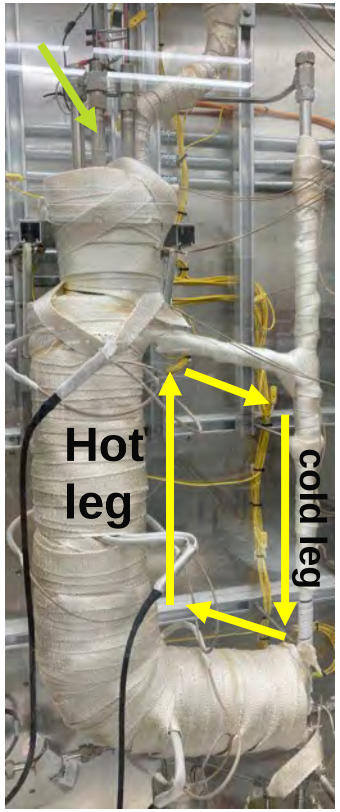


Hastelloy N, $NaBF_4$-NaF-KBF_4
8760 h, TCL 605°-460°C
- J. Koger, Corrosion, 1974

How do we study it?

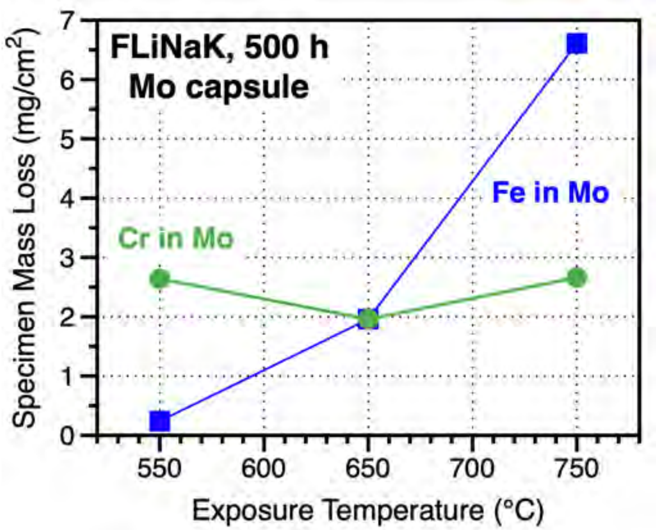
- Flowing salt experiments
  - Forced convection loop
  - Thermal convection loop

2021 ORNL FLiBe TCL



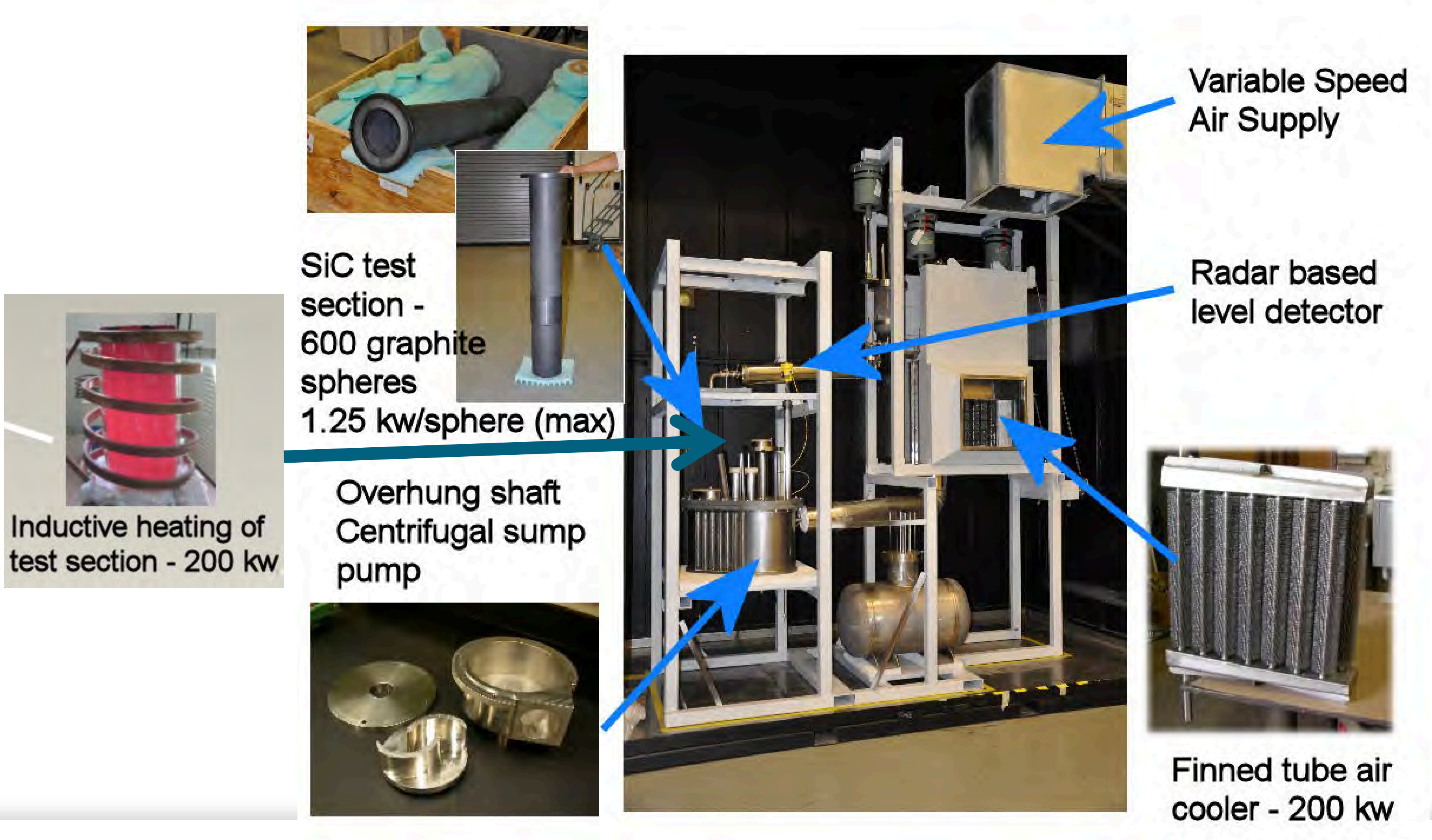
How do we understand it?

- Dissolution experiments
  - Compare Cr and Fe in isothermal salt
  - Experiments in FLiNaK and FLiBe in progress
    - 550°-850°C



ORNL FLiNaK pumped loop: prototypic conditions

But we first learn about corrosion on inexpensive TCLs

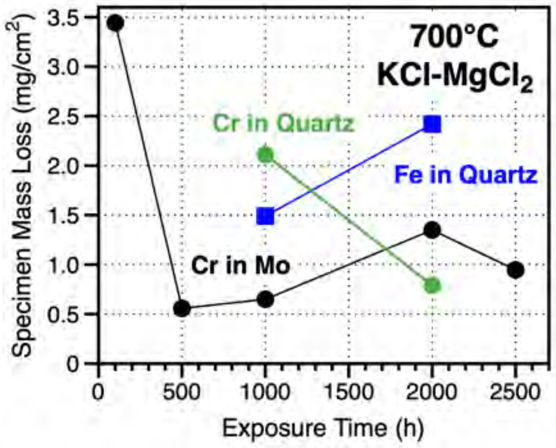


FLiNaK salt leak on 316H TCL

Salt and Materials Interactions: R&D Goals

- **FY22-23 goals**
  - Cr and Fe dissolution experiments in FLiNaK and FLiBe
    - FY22: Three temperatures: 550°, 650°, 750°C + three times (500-2000 h)
    - FY23: One temperature: 850°C + three times (100-2000 h)
  - Continue to develop lifetime model (Pillai et al., JNM 2021, JOM 2023)
- **Future topics**
  - FLiBe/316H interaction: higher temperatures/accident scenario TCLs
    - Feedback from MSR developer
  - Compatibility effect of impurities/additives (salt purity standards)
  - Further development of corrosion sensors/electrochemistry aspects
  - Similar studies for Cl salts
- **FY25 goal**
  - De-risk the transition from 316H to higher performance alloy 709
    - Focus on higher temperature operation and lifetime modeling

Unexpected temperature effect:

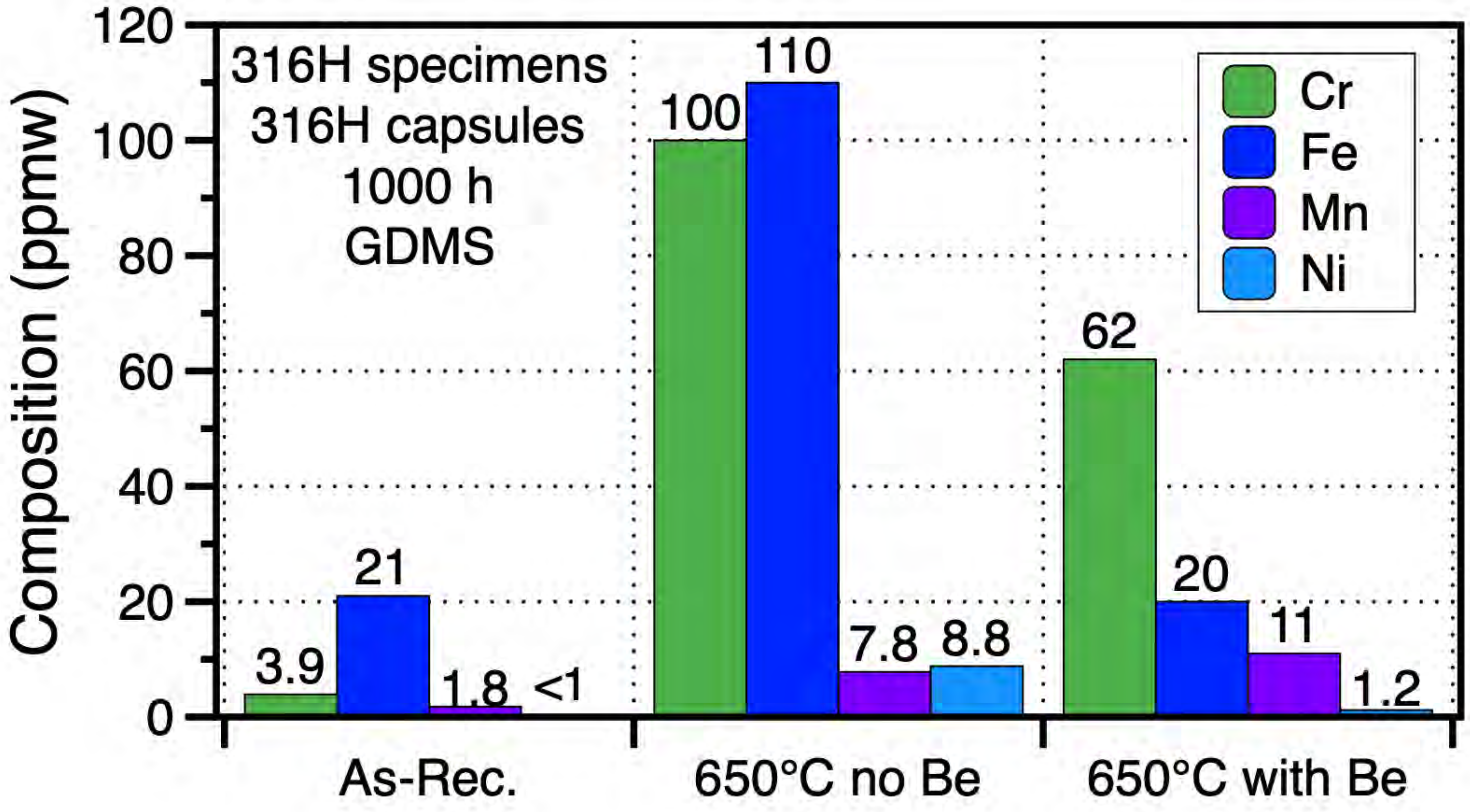


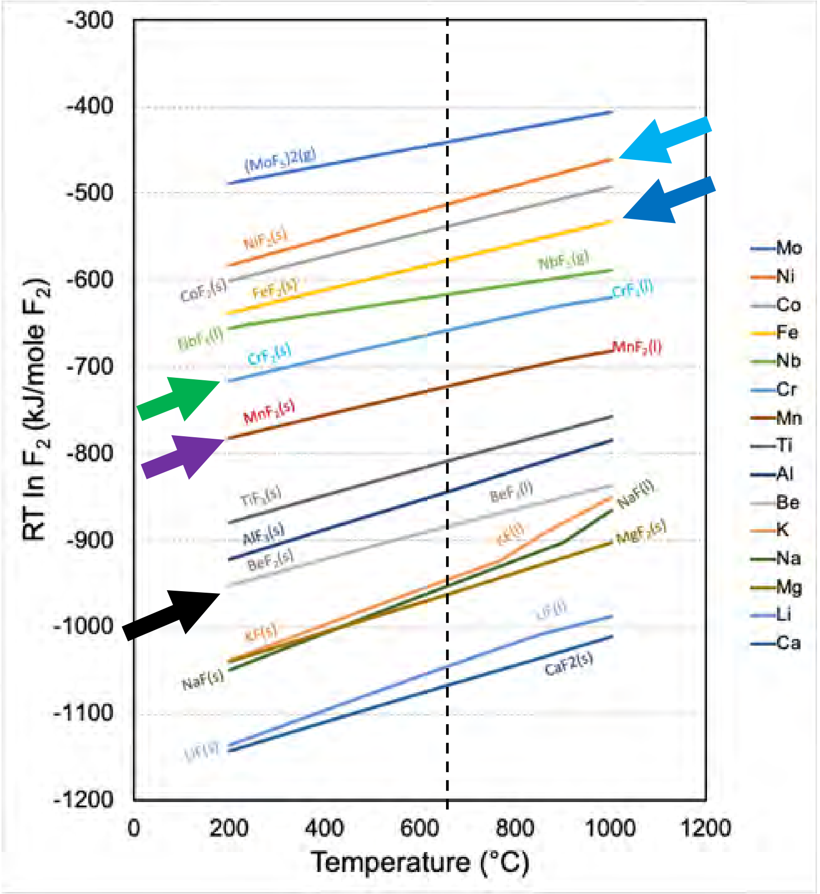
Salt and Materials Interactions: FY22 overview

- **FY22 Carryover M3RD-23OR0603031: Measure Cr/Fe dissolution in FLiNaK and FLiBe**
  - Three temperatures (550°, 650°, 750°C) and three times (500-2000 h)
  - Completed Cr in FLiNaK
  - Repeating Fe in FLiNaK
- **M3RD-23OR0603032: Measure Cr/Fe dissolution at 850°C in two halide salts**
  - Three times (100-2000 h)
  - More complete data set needed for modeling

Both Cr and Fe are dissolving

316H: 68wt.%Fe-16.5Cr-10.4Ni-1.9Mo-1.5Mn-0.3Si-0.4Cu-0.034C



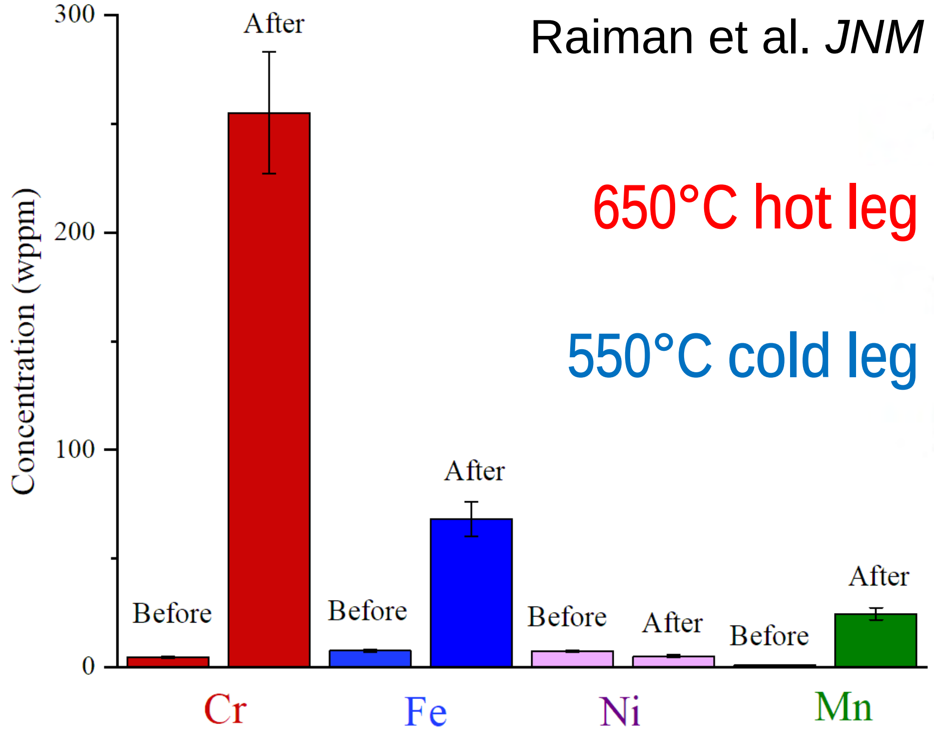


FLiBe: low initial impurities

No Be: Cr and Fe increase ≥90 ppm, Ni increase

With Be: Cr and Mn increase (~45% less mass loss)

ICP-OES of FLiNaK: Increased Fe after 1,000h TCL

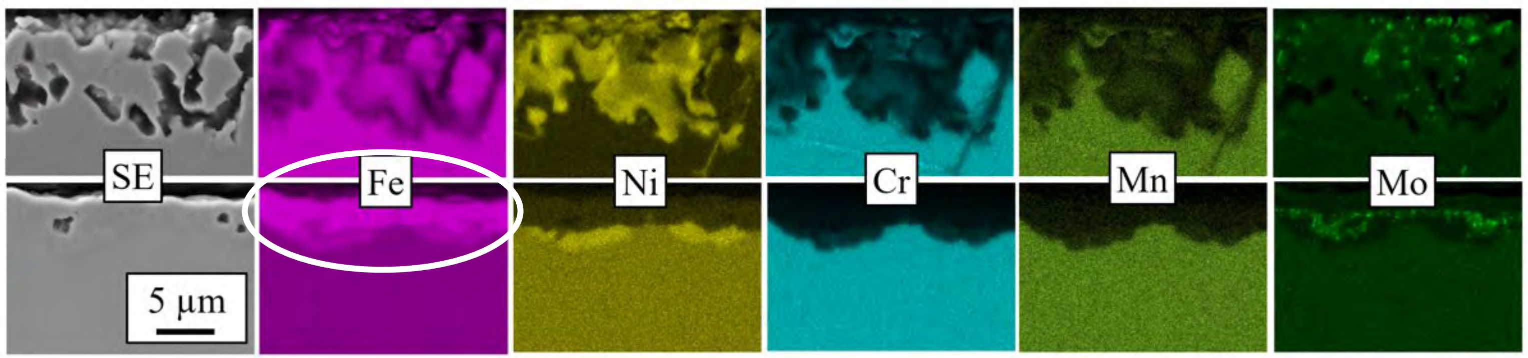


Inductively coupled plasma-optical emission spectroscopy

Raiman et al. *JNM 2022*

650°C hot leg

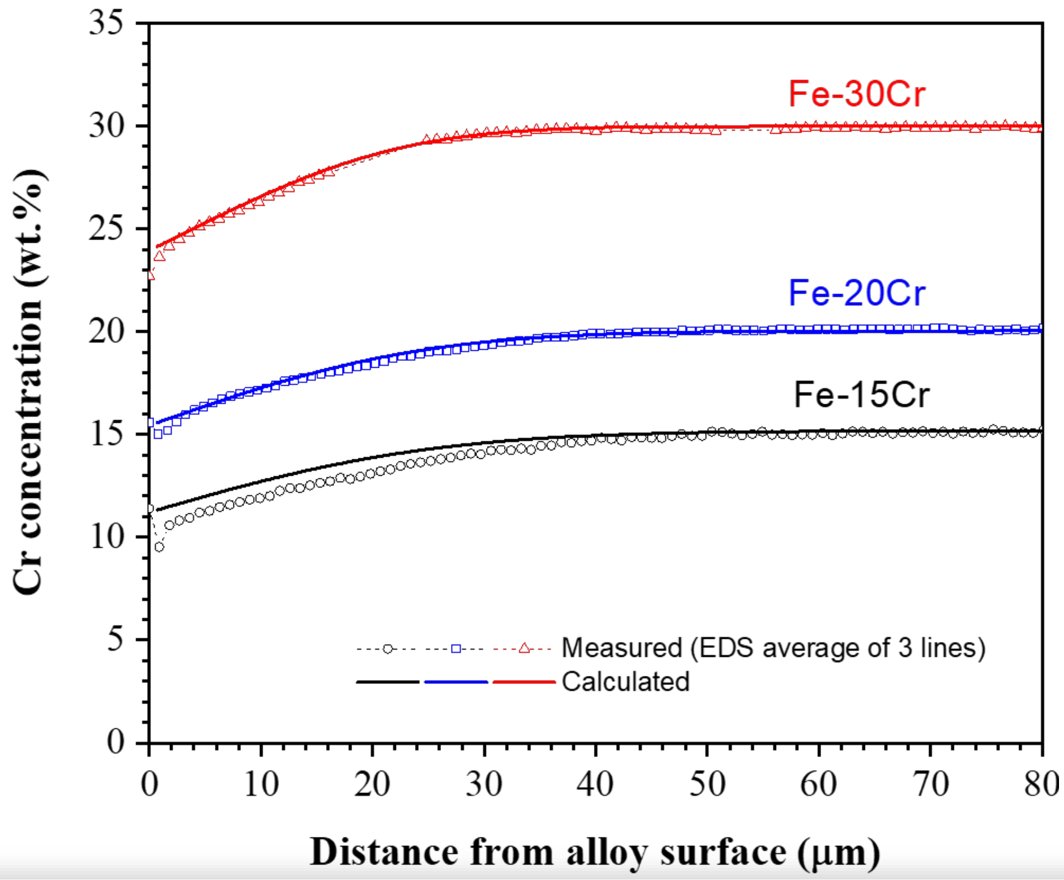
550°C cold leg

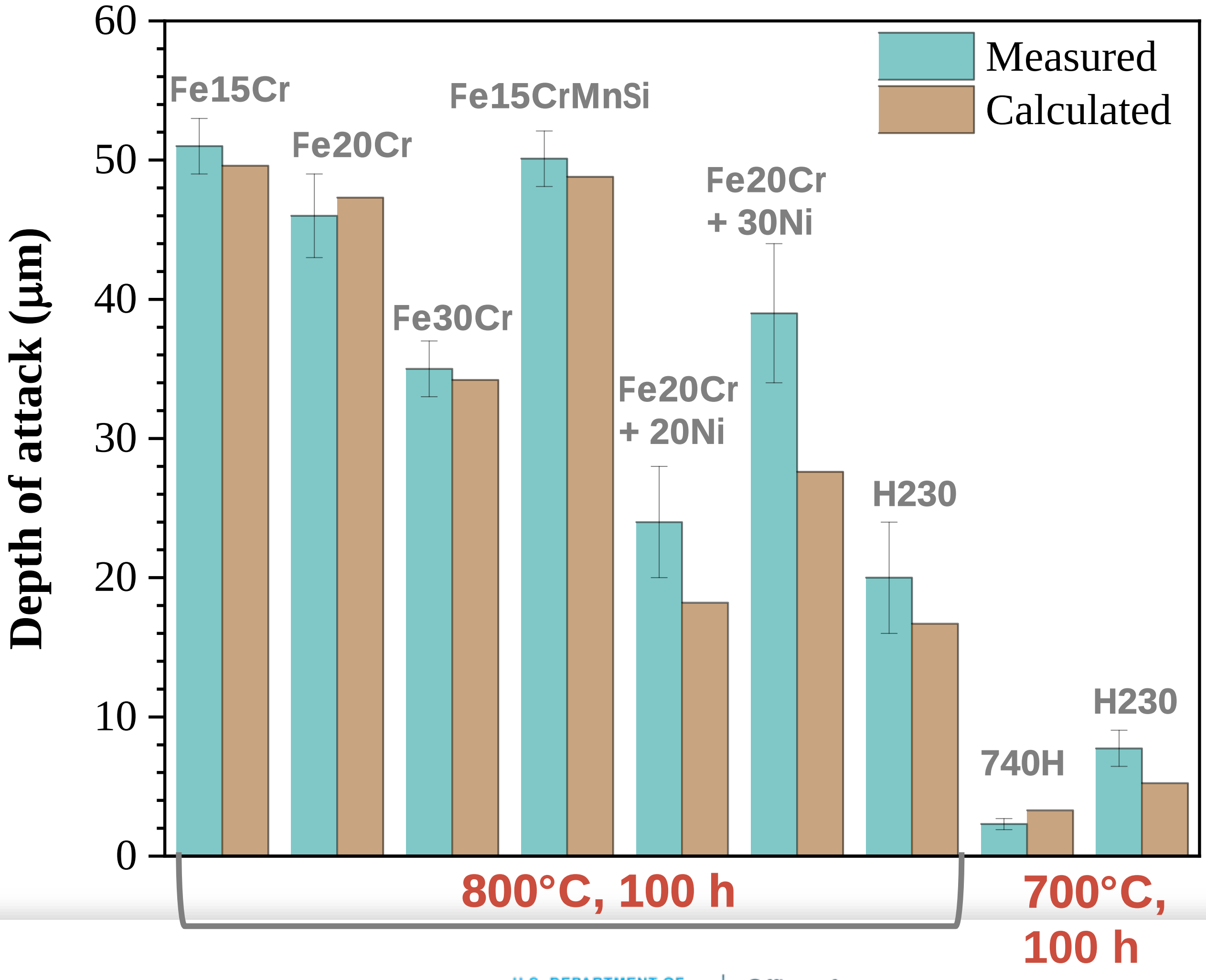


- FLiNaK contained 400ppm Zr
- Higher Cr in salt after TCL
- Evidence of Fe deposition in CL

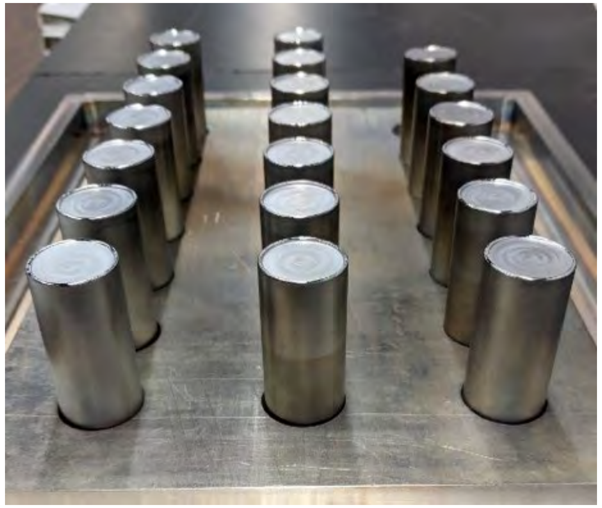
Model can predict Cr depletion and depth of Cl salt attack across different alloy chemistries at different isothermal temperatures: Need dissolution rates for model

KCl:$MgCl_2$, 800°C, 100h





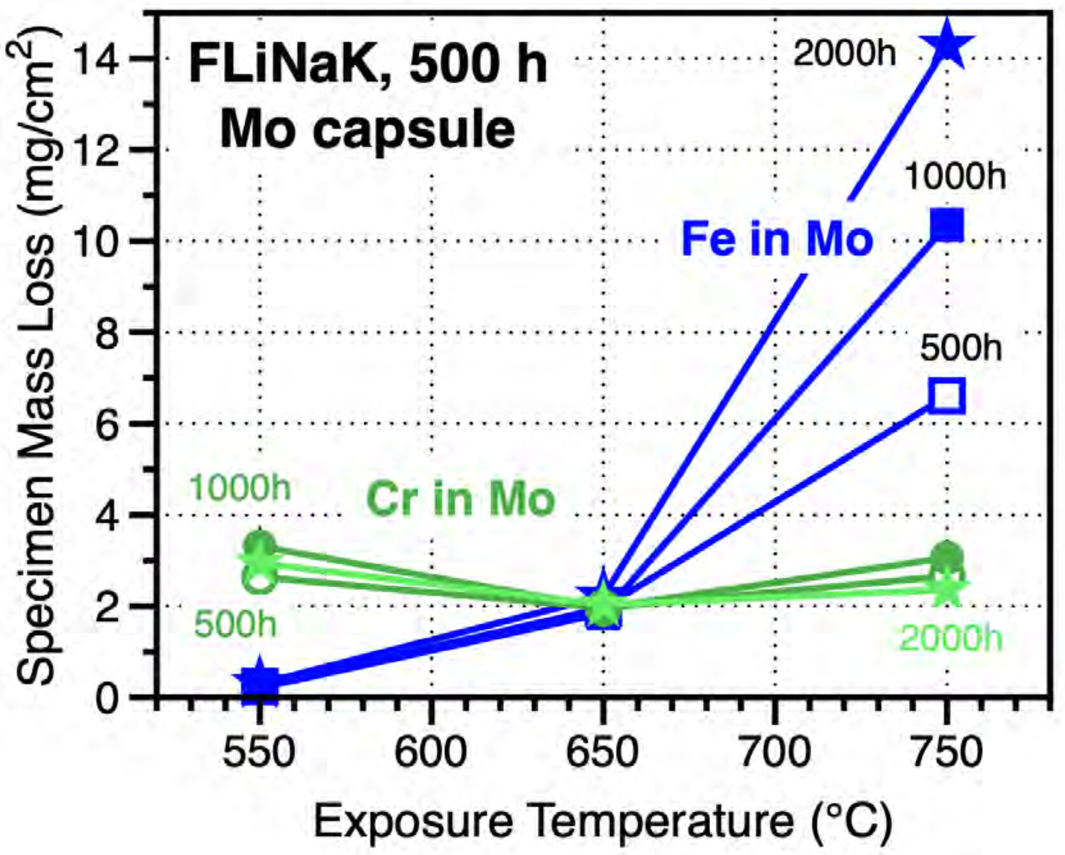
Capsule testing to identify dissolution rates



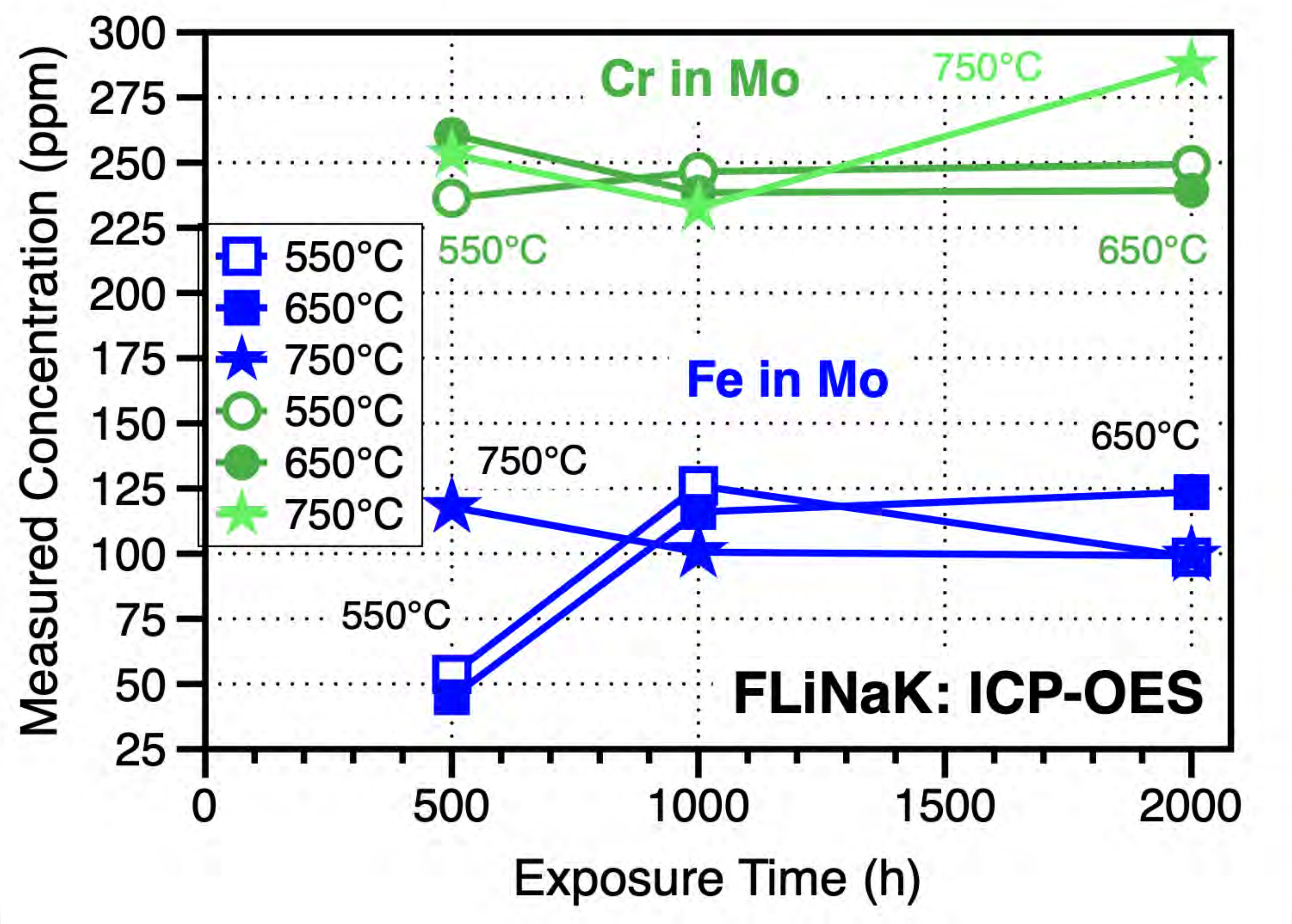
100-2000 h in box furnace
Flip at the end for removal

Dissolution mass losses: 500-2000 h at 550°-750°C

- **Mo capsules**
  - standard ORNL procedure
- **Purified FLiNaK salt (+400ppm Zr)**
- **Little change with longer time except for Fe specimens at 750°C**

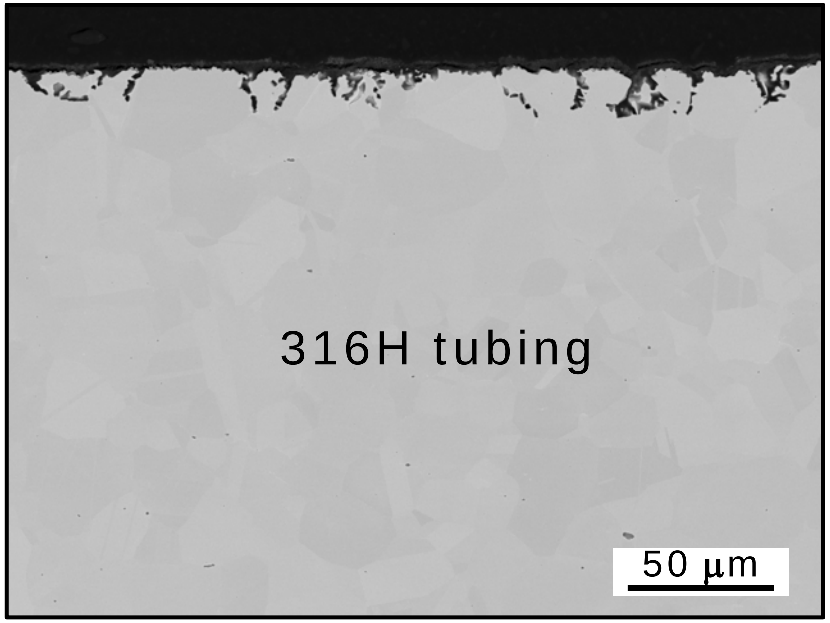


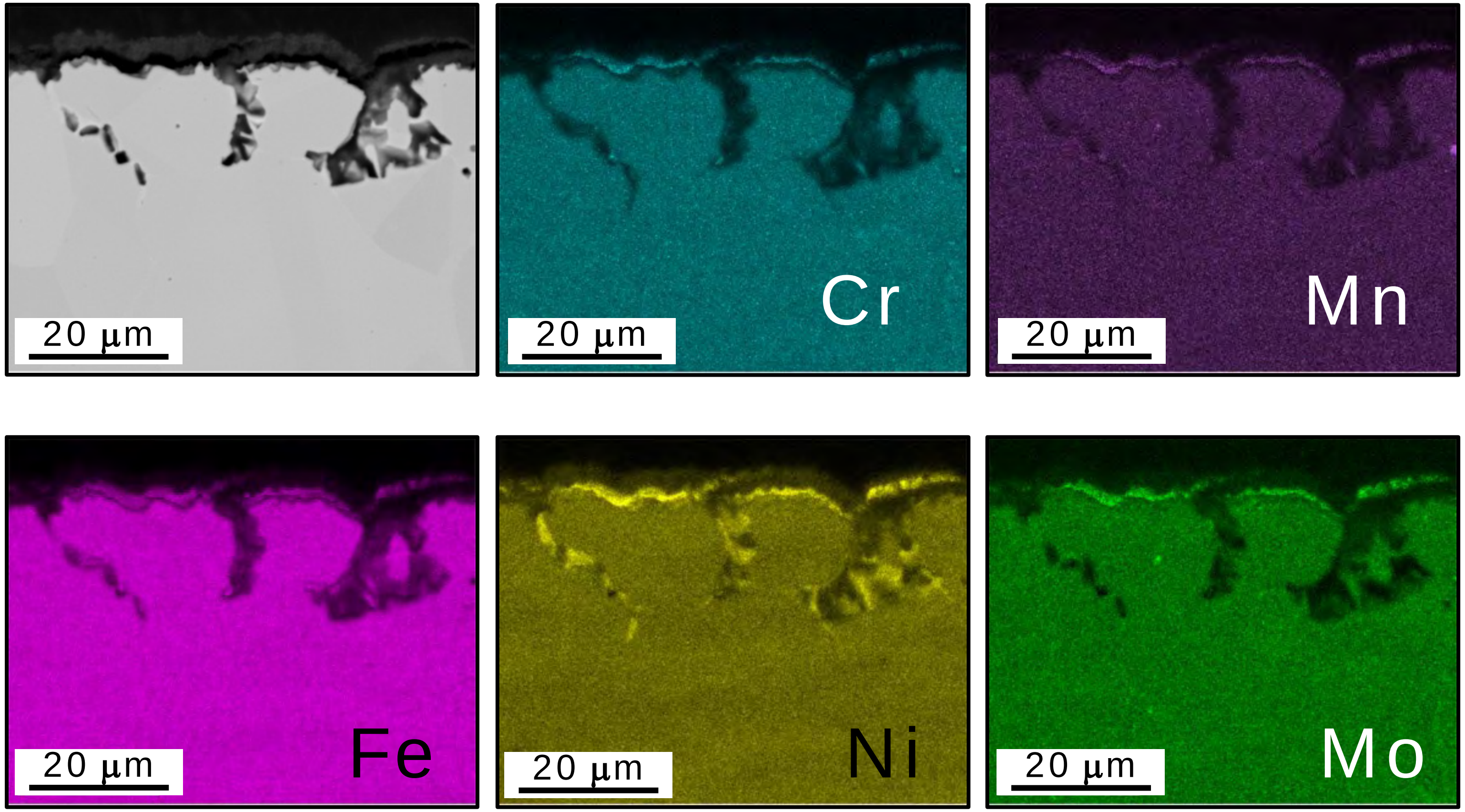
Uh oh: low Fe in salt after 750°C tests
Cr somewhat consistent



- Mo-Fe interaction
  - Capsule effect!
- Need to repeat test with Fe capsule
  - First tests no Fe sample
  - Waiting for ICP-OES analysis of tests at 550°-850°C
  - Next tests will include Fe specimen

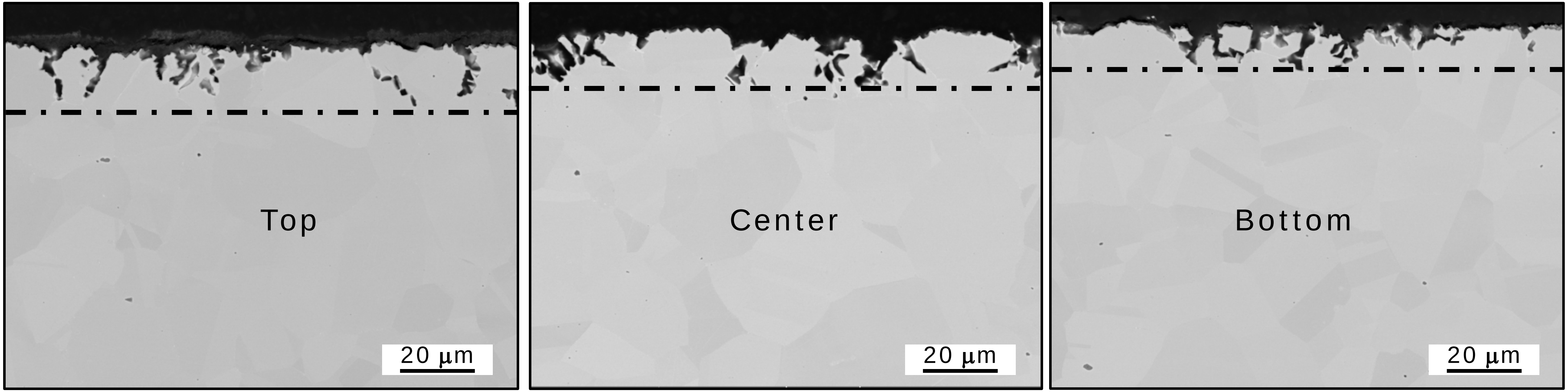
To validate model in flowing salt: Need to characterize FLiNaK/316H loop



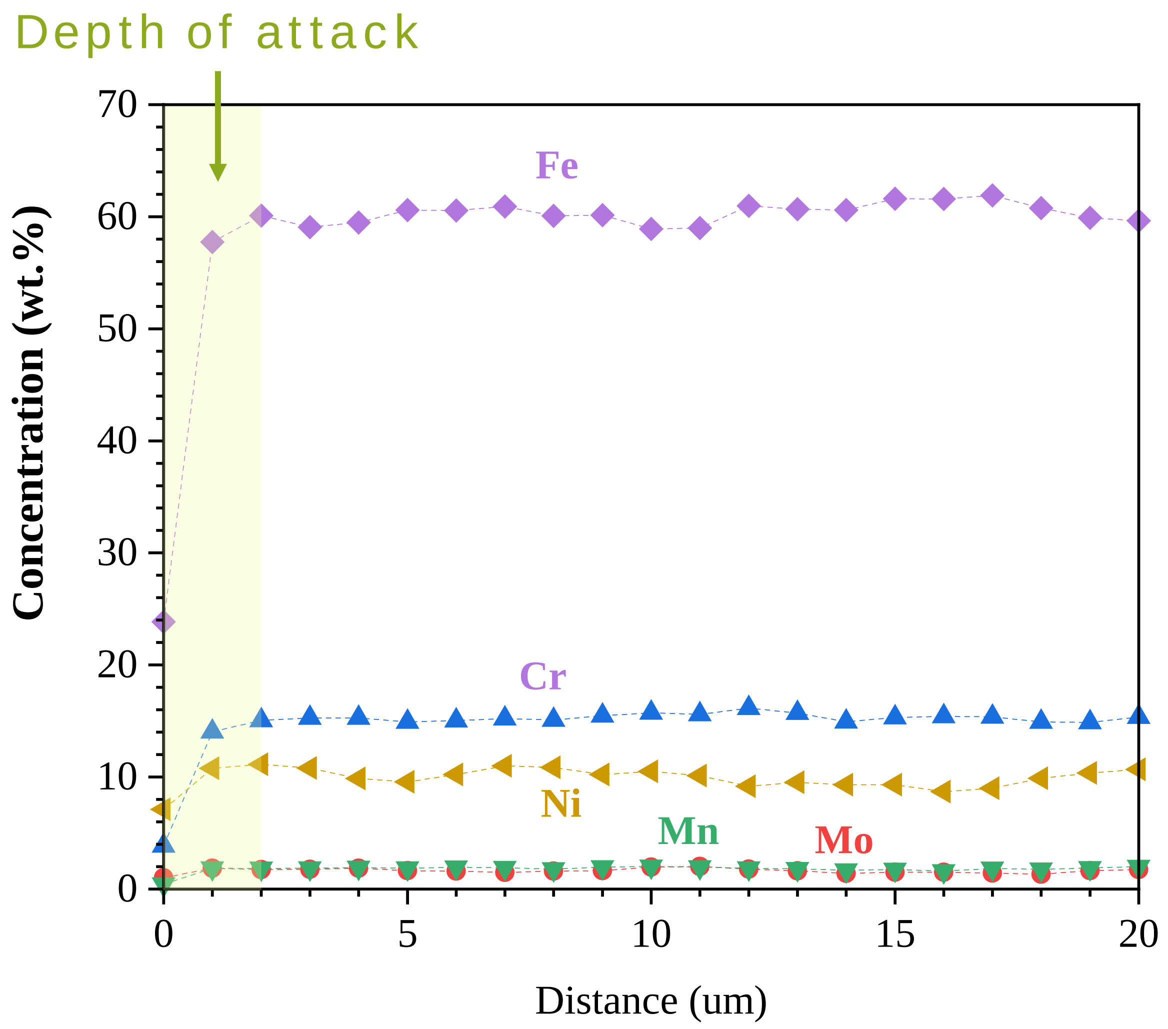


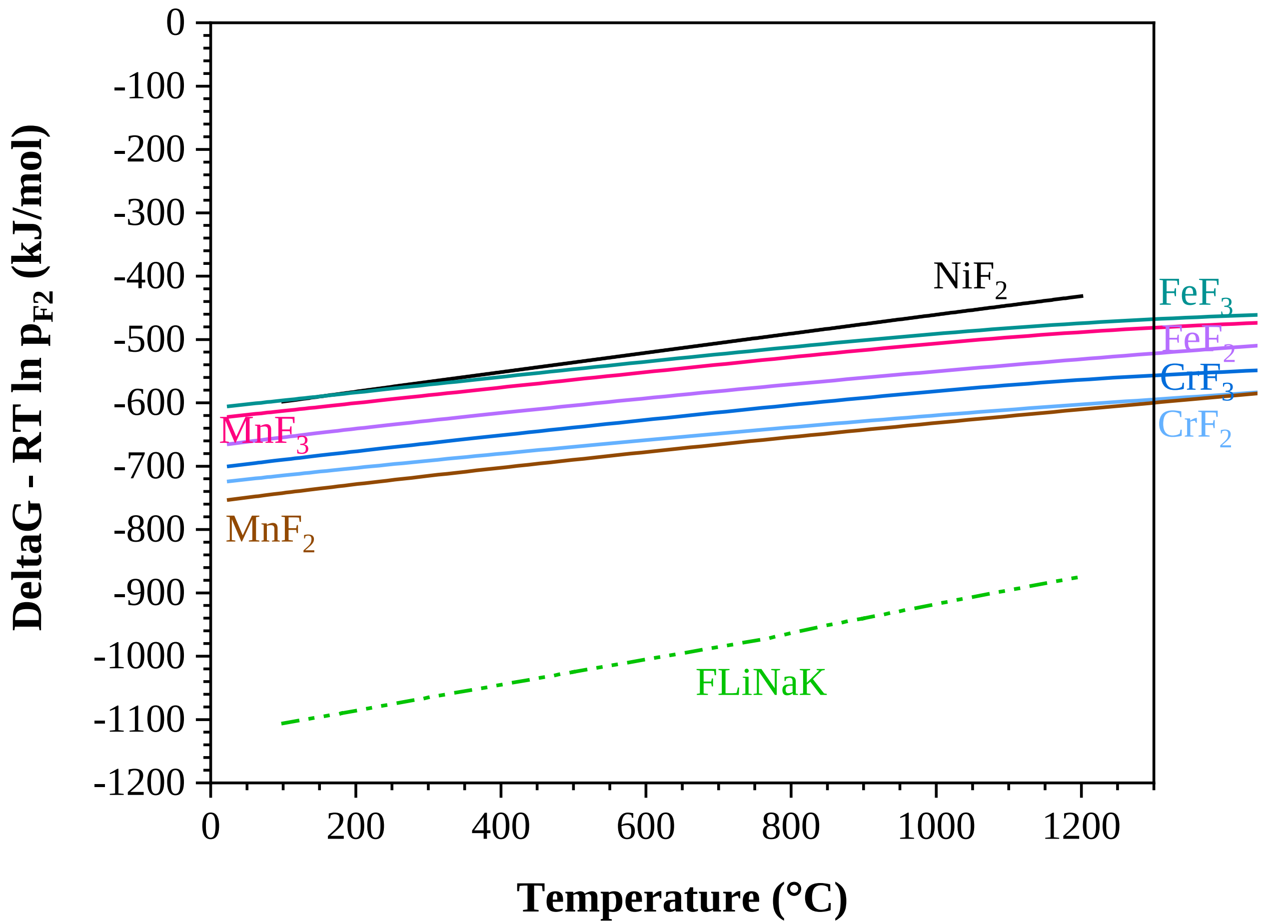
Topmost section of hot leg (~647 °C) shows dissolution of Cr, Mn, Fe and Mo

316H tubing attack was observed to decrease from the top to the bottom of the TCL hot leg



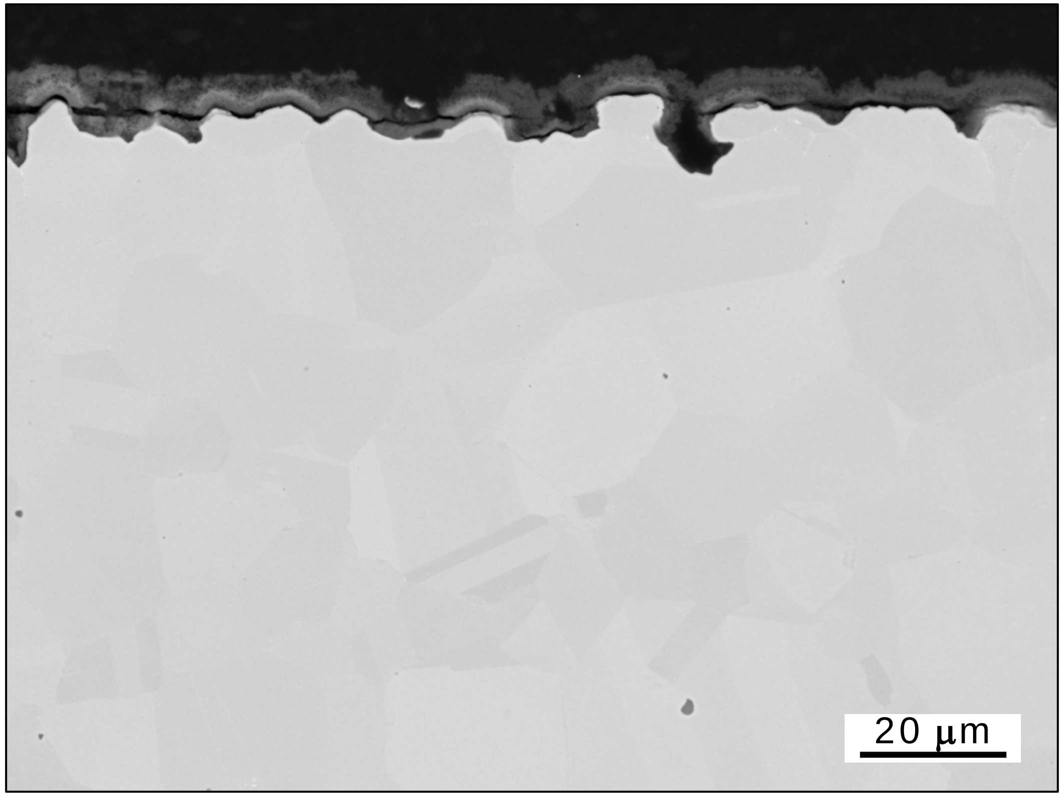
Measured concentration profiles suggest depletion of all key elements

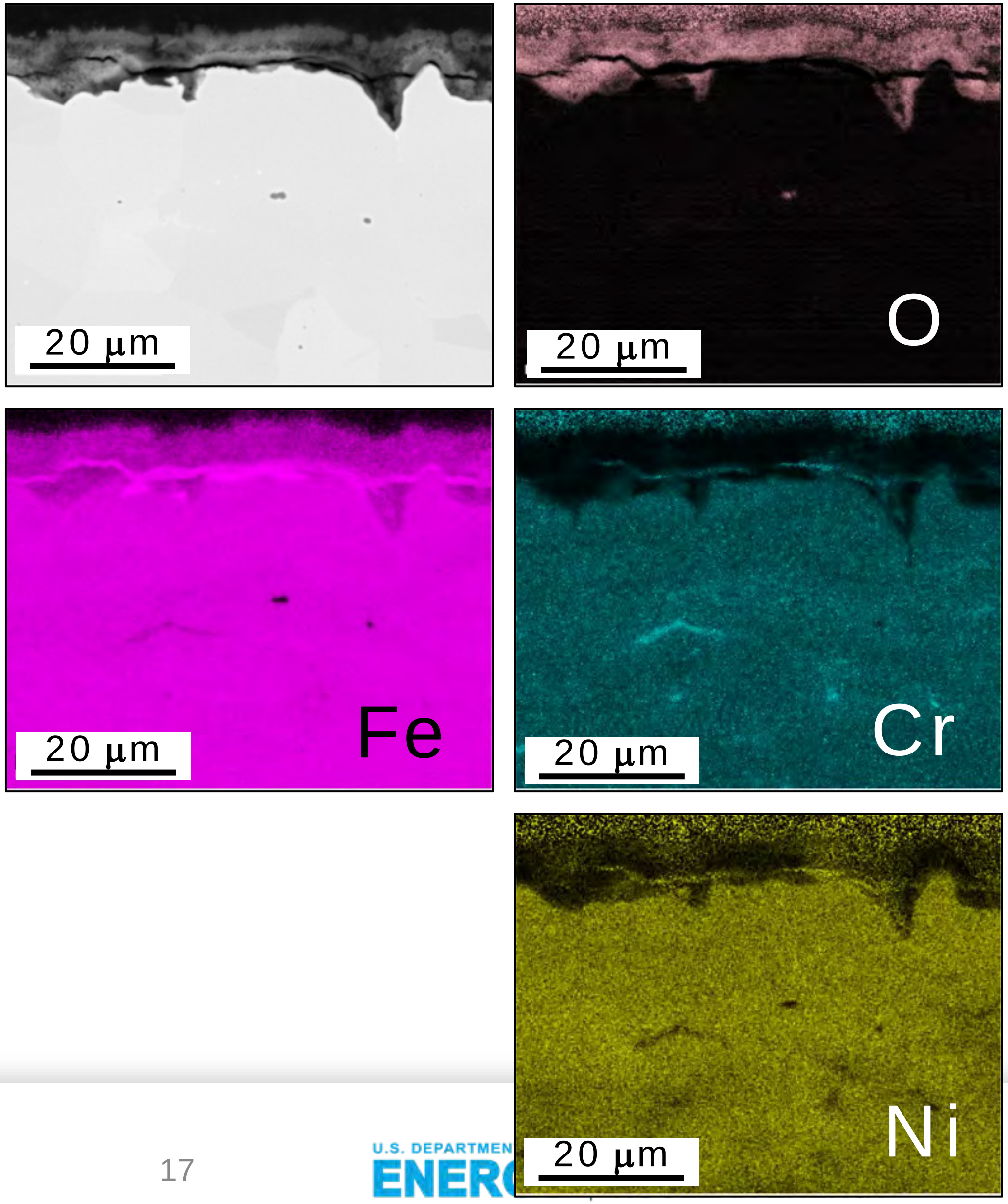




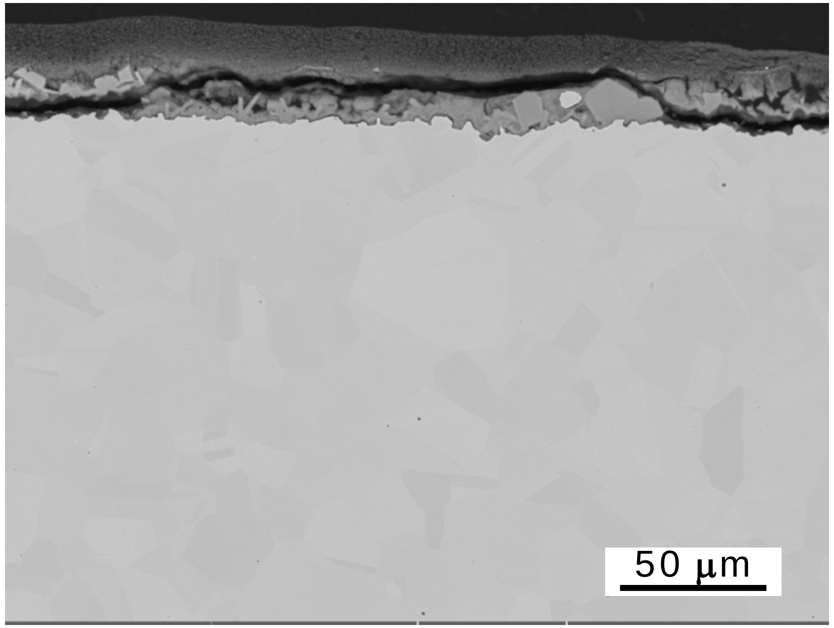
➢ **Observation agrees with thermodynamic stability of fluorides**

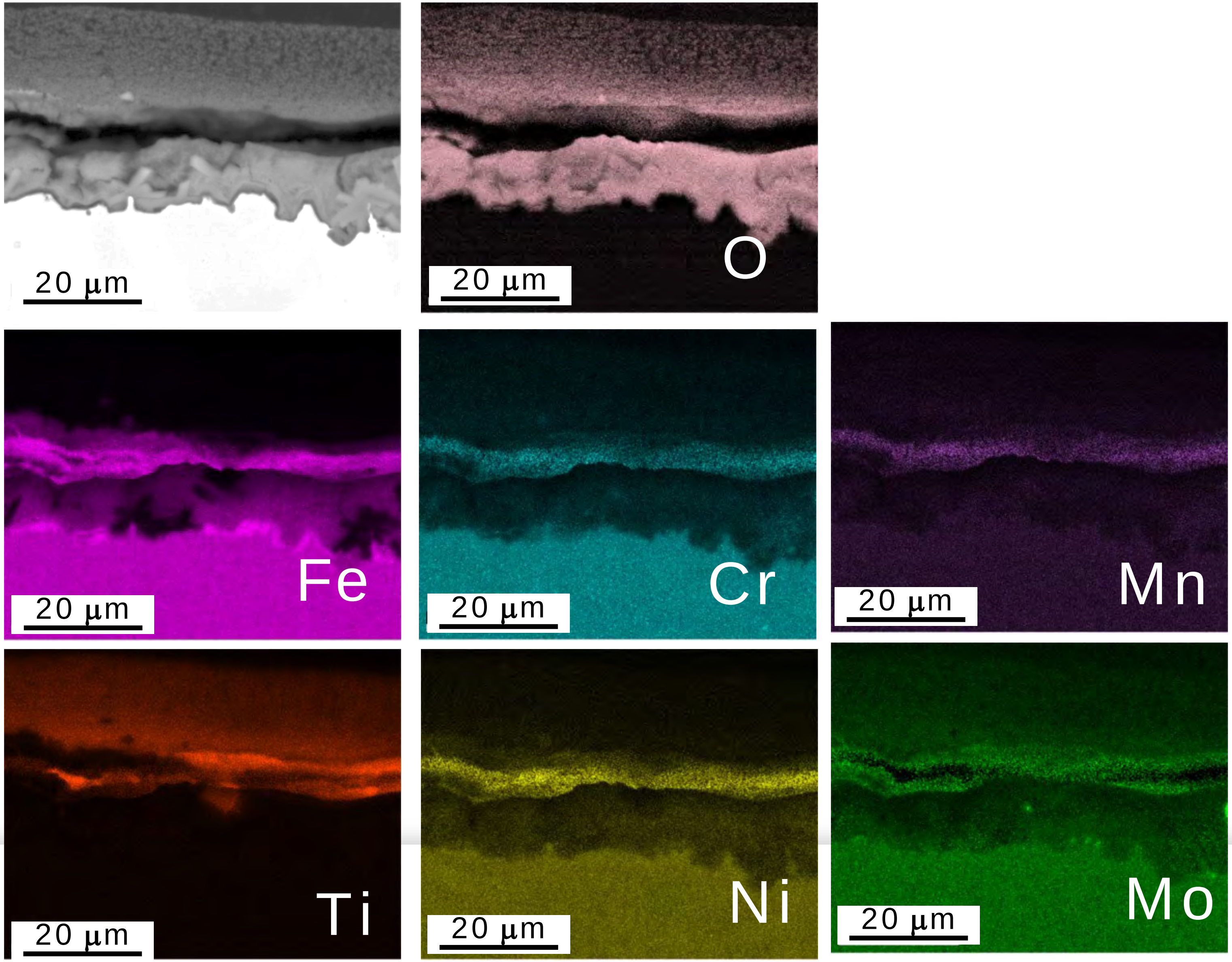
Topmost section of cold leg (~604 °C) shows deposition of Fe-rich oxides





Bottom section of cold leg (~542 °C) shows deposition of Ti-rich oxides (most likely from alloy 600 wire used to hold the specimens)



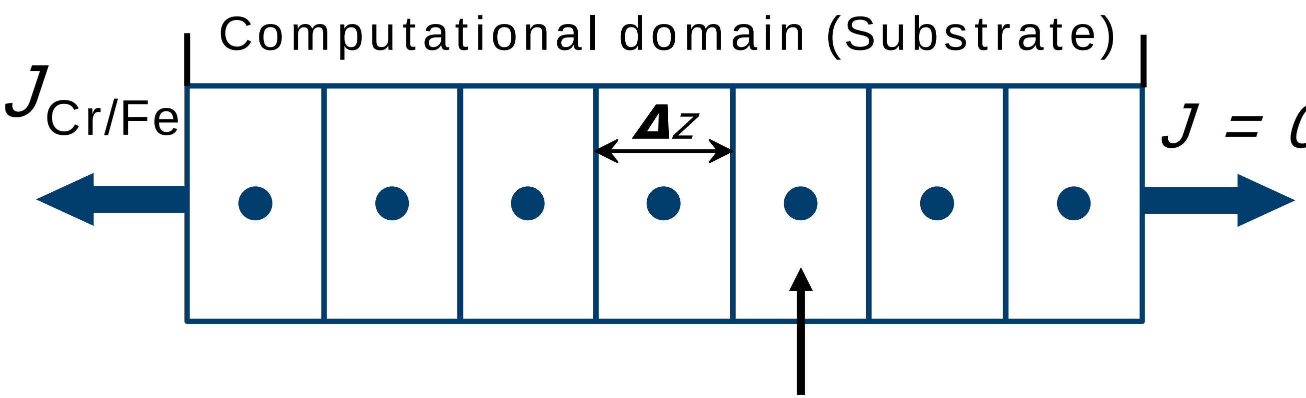


Hypothesis: Corrosion of alloying constituents is primarily governed by their chemical activity (thermodynamics) and mobility (kinetics) in the alloys

Coupled thermodynamic-kinetic modeling approach

$$J_{Cr/Fe,alloy} \propto N_{Cr/Fe,pure} * a_{Cr/Fe,alloy}$$

Dissolution rate of alloying elements in multicomponent alloys is directly proportional to their dissolution rate in pure form and chemical activity in the alloy



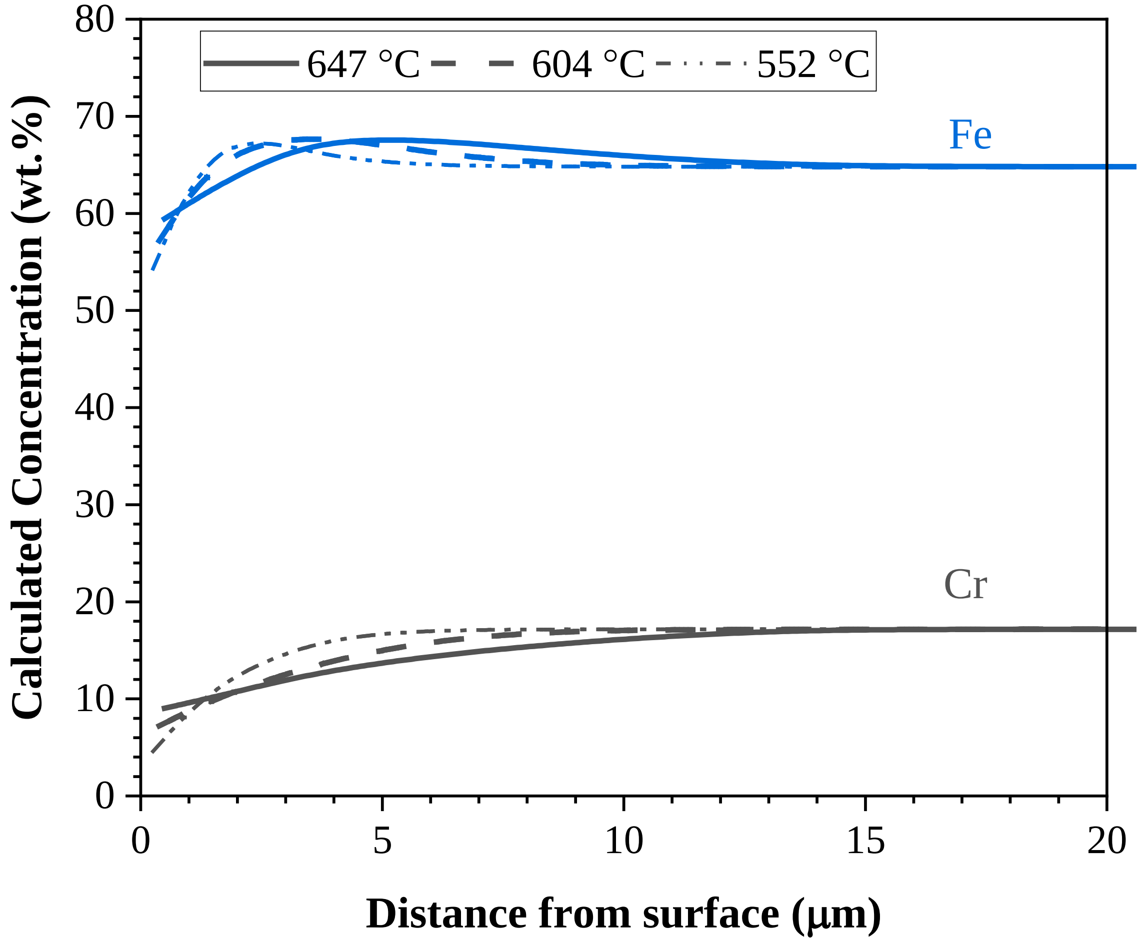
- *Calculation of element fluxes (chemical potential gradients)*
- *Calculation of phase equilibria*

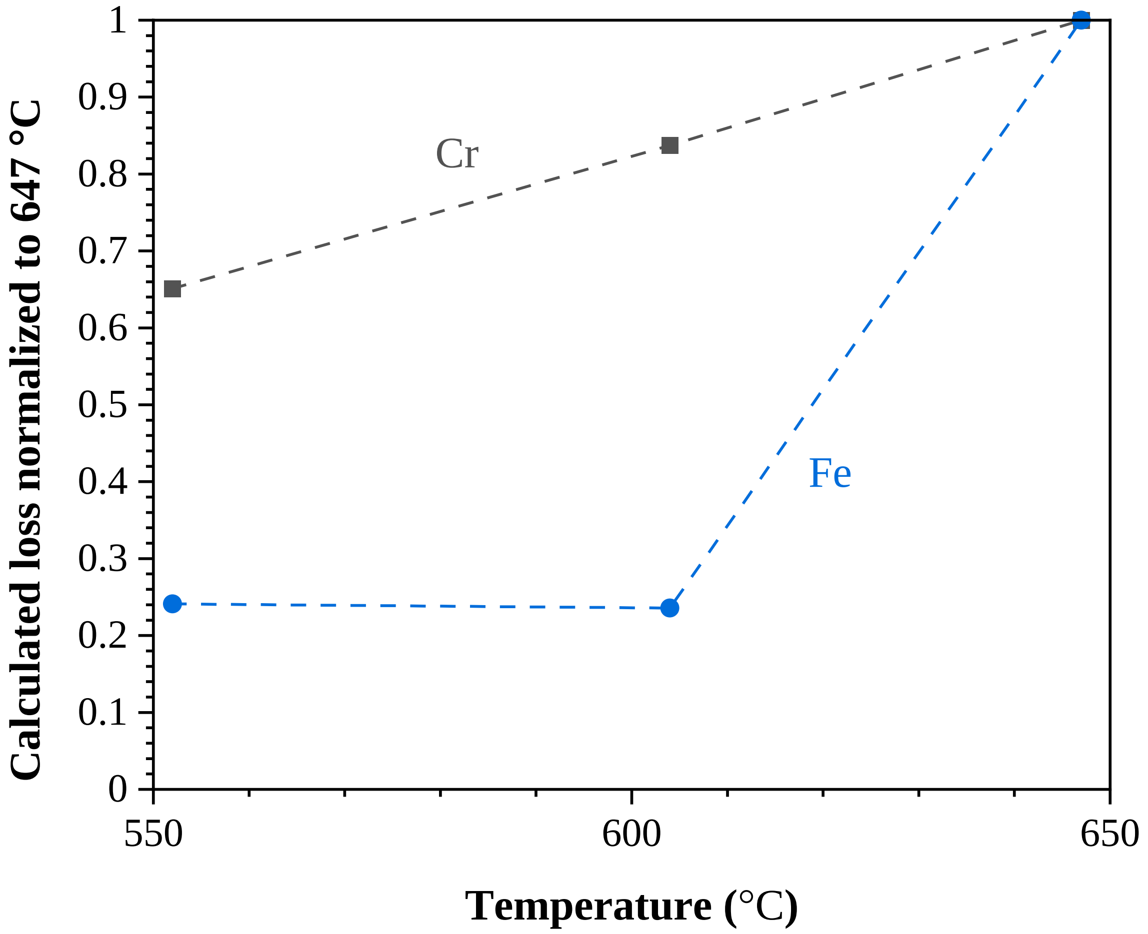
– Calculate diffusion in the alloy

- Using measured Cr and Fe concentrations after exposure of pure Cr and Fe in purified FLiNaK
- Use of independent thermodynamic-kinetic data -TCNI/MOBNI (Thermo-Calc)
- Consideration of relevant elements & phases in commercial high temperature alloys and coating systems
- Mesh adaption accounts for surface recession (predictions for metal loss)
- Thermodynamic calculations on multiple cores

** Pillai et al., JOM, 2023*

Cr loss from 316H is expected to slightly increase with temperature while Fe loss significantly drops at lower temperatures

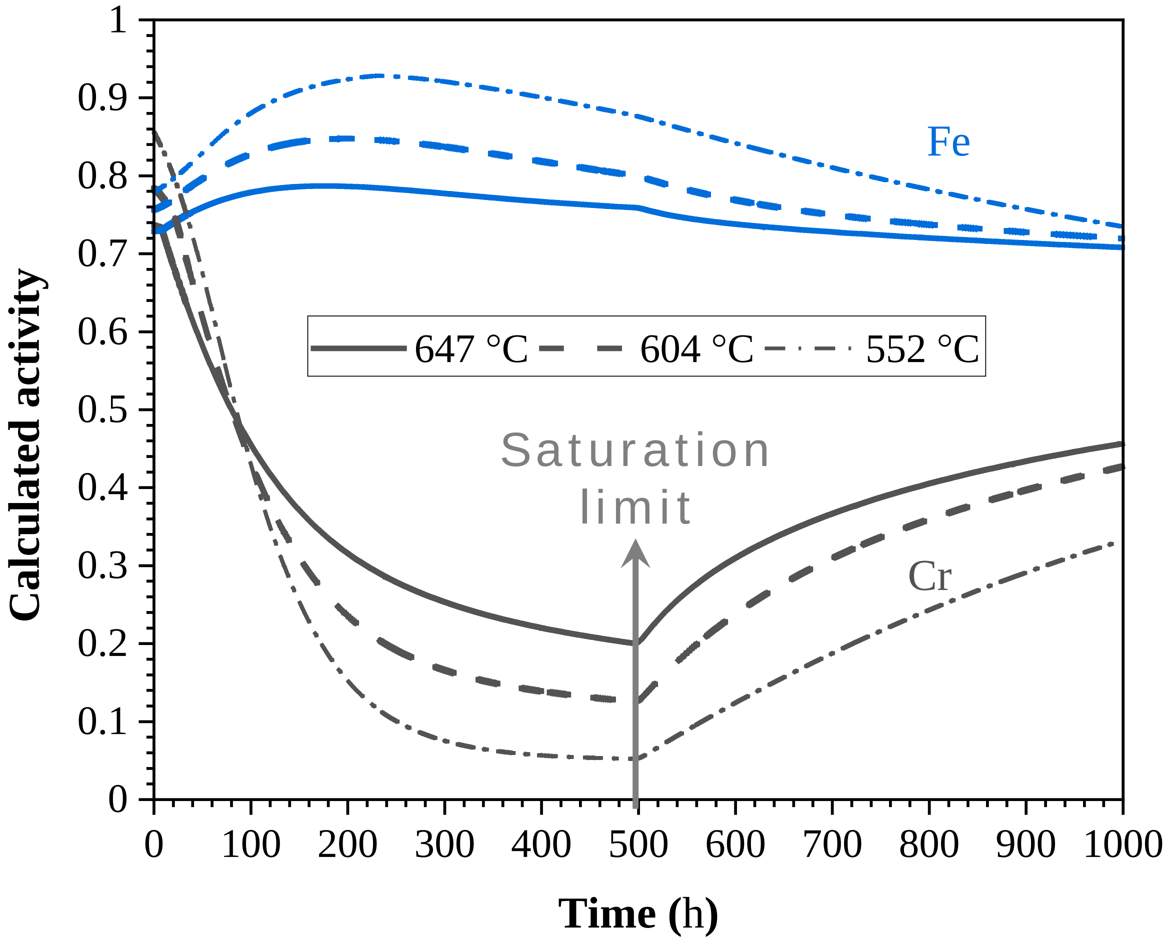




- ➢ **Ongoing tests to acquire precise Fe dissolution rates in FLiNaK**
- ➢ **Results agree well with experimental observations***

** Raiman et al., JNM, 2022*

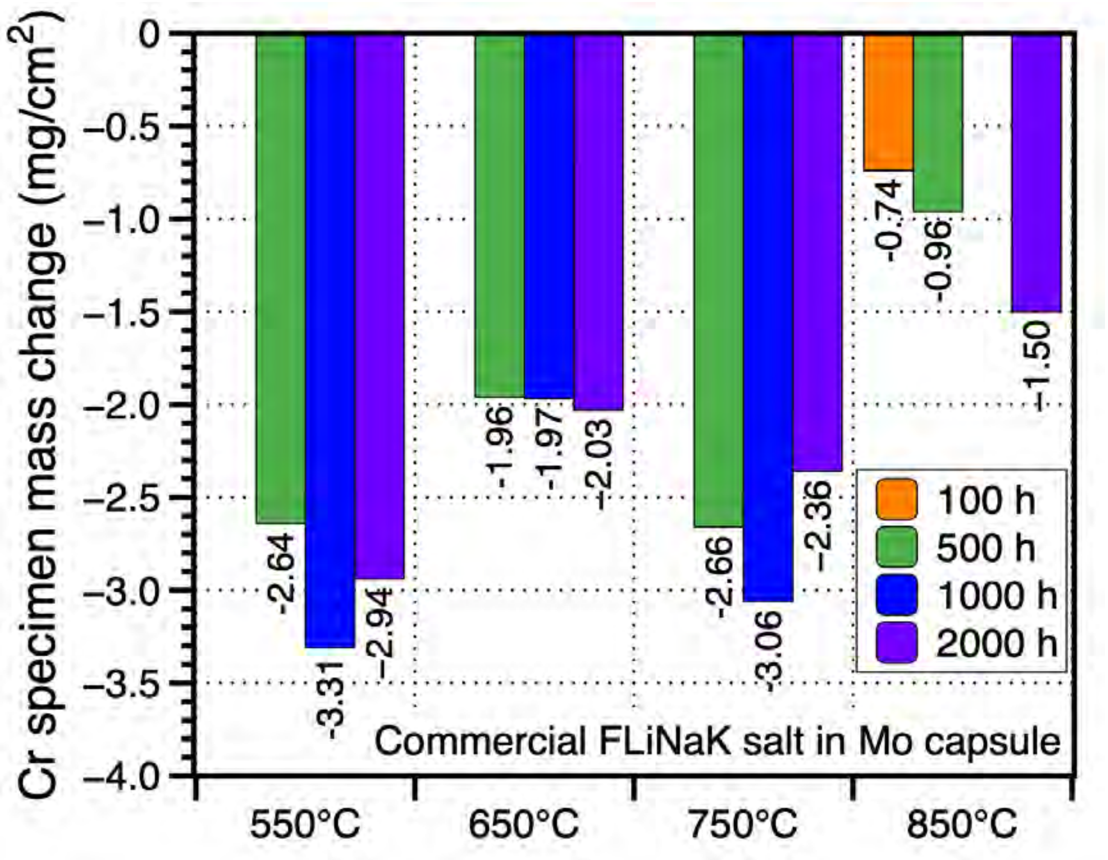
Calculated time evolution of Fe and Cr surface activity shows Fe dissolution begins after Cr is saturated in the salt



- **Saturation limit (~280 ppm) was measured in FLiNaK dissolution tests**
- **Much slower Fe dissolution rate was derived from dissolution tests**

Next: effect of temperature + testing in FLiBe

Initial 850°C results: less Cr loss

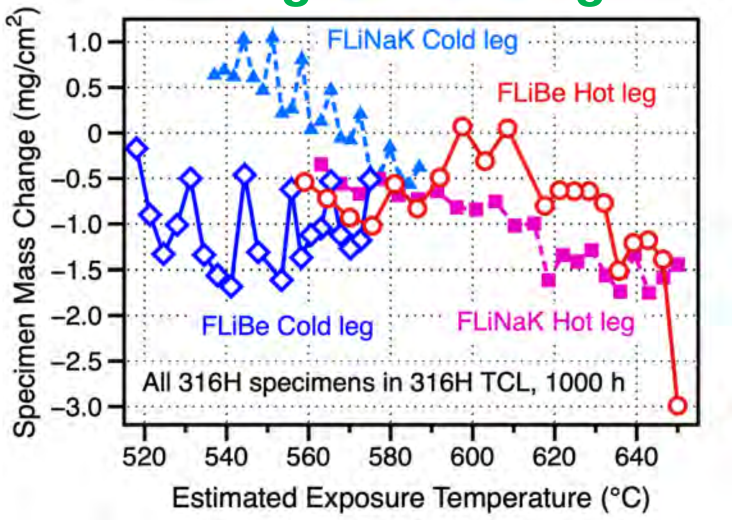


FLiBe salt arrived from Kairos
Capsules being assembled

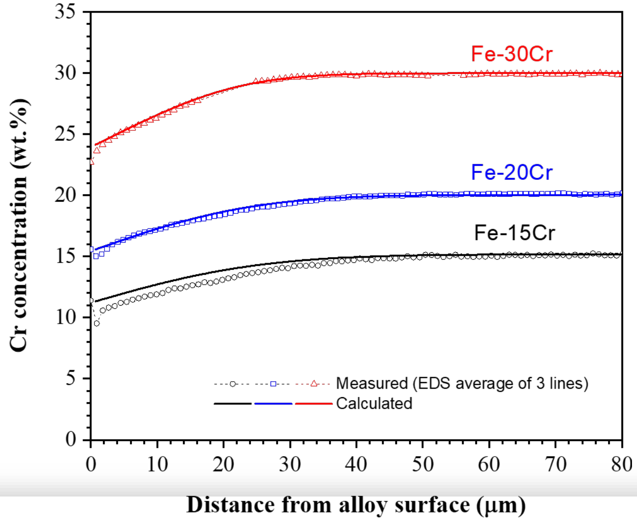
Path forward: modeling dissolution in purified, flowing salt

- **Model used Cr data in Cl salt**
  - Pillai et al., **JNM 2021, Ni-Cr alloys**
  - Pillai et al., **JOM 2023**, Fe-Cr alloys
- **Collecting dissolution data in FLiNaK and FLiBe**
- **Current task to model dissolution and mass transfer in flowing salt**

Mass change in flowing salt



$KCl:MgCl_2$, 800°C, 100h Model vs. data



Gordon Conference on High Temperature Corrosion

- **July 16-21, 2023**
- **Colby Sawyer College**
    - New London, NH
- **You're all invited to attend**
- **For students and early career:**
    - Gordon Research Seminar 7/15-16

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Thank you

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