Compatibility assessments for fusion applications: Sn, Li, Pb-Li and FLiBe

Bruce Pint, Marie Romedenne and Dino Sulejmanovic

Corrosion Science and Technology Group
Materials Science and Technology Division
Oak Ridge National Laboratory

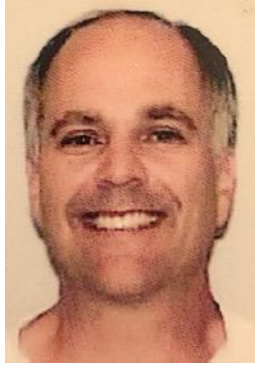
ISFNT, September 2023

Acknowledgments

- Funding
  - U.S. Dept. of Energy, Office of Fusion Energy Sciences (**Kessel**, Humrickhouse, Smolentsev)
  - U.S.-Japan FRONTIER project (Sn/ODS FeCrAl, Prof. M. Kondo, task co-leader)
  - ORNL Laboratory Directed Research & Development SEED funding (FLiBe)
- ORNL team
  - Adam Willoughby, Mike Stephens, Brandon Johnston, Jiheon Jun: LM experimental work
  - Shane Hawkins, Kelsey Hedrick: tensile testing
  - Characterization: Tracie Lowe, Victoria Cox, Ercan Cakmak, Yi-Feng Su (TEM)
- T. Nozawa, QST, Japan: F82H plate
- Mentors: Jack DeVan, Jim DiStefano, Peter Tortorelli, Steve Pawel

DeVan 1929-2000

DiStefano 1935-2013



2013 APMT TCL design

ORNL compatibility research has several current tasks

- **US DOE FESS LM PFC project (2020-2025)**
  - Investigating liquid metal **embrittlement of F82H (Fe-8Cr-2W) in Li**
- **US-Japan FRONTIER emphasis on Sn (2019-2024)**
  - Pre-oxidized FeCrAl (ODS, APMT): Sn thermal convection loop (2021)
  - HFIR irradiation pre-oxidized FeCrAl in Sn at 400°C (0.8 dpa in 2022)
- **US DOE Blanket & Fuel cycle project (2019-2024)**
  - ORNL Pb-Li project ended 2019 (4 monometallic APMT (FeCrAlMo) loops)
  - More fusion relevant materials in **flowing Pb-Li** (APMT tubing)
    - TCL #5: SiC, ODS FeCrAl (700°C peak, completed April 2020)
    - TCL #6: SiC, Al-coated RAFM (650°C peak, completed in September 2021)
    - **TCL #7 : SiC, Al-coated RAFM (650°C peak, 2000 h operation completed Sept. 2023)**
- **ORNL SEED: explore steel-$Be_{12}Ti$ interaction in FLiBe (2023)**
  - Initial static capsule testing in FLiBe at 550°-750°C in September 2023

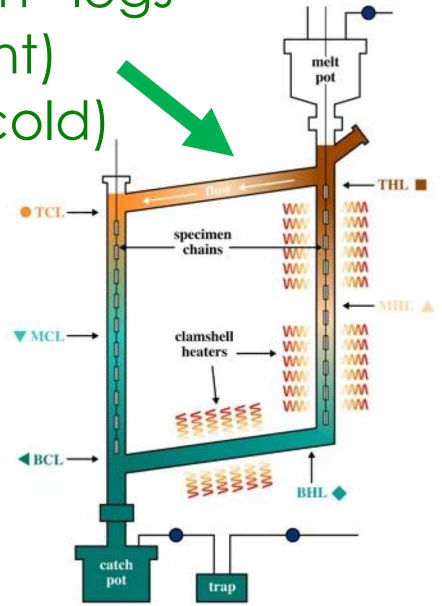
Liquid physical properties and compatibility TRL

Property	Li	Pb-Li	Sn	FLiBe
Melting Temp. (°C)	**181**	235	232	**459**
Density (g/cm^3)	**0.5**	**9.9**	6.5	2.0
Viscosity (N•s/m^2)	0.0006	**1.4**	0.002	0.07
Heat capacity (J/kg•K)	**4170**	190	248	2414
Thermal Conductivity (W/m•K)	**65**	25	33	**1.1**
Electrical Conductivity (μΩ•cm)	25	1	48	0.4
Compatibility TRL	**high**	**highest**	**low**	**lower**
Thoughts	MHD mitigation, No SiC, No Ni	Radiation? Magnetic field?	Corrosive	??

All concepts are far from where they need to be for designing DEMO/FPP

How do we assess and quantify LM compatibility?

- Thermodynamics
  - First screening tool (assessments published, but data is not always available)
- Static capsule/crucible (screening test only)
  - Isothermal test, first experimental step
  - Prefer inert material and welded capsule to prevent impurity ingress
  - **Dissolution rate changes with time**: key ratio of liquid/metal surface
  - **No assessment of mass transfer**
- Flowing thermal convection loop (TCL)
  - Flowing liquid metal by heating one side of "harp" with specimen chain in "legs"
  - Relatively slow flow and ~100°C temperature variation (design dependent)
  - **Captures solubility change in liquid**: dissolution (hot) and precipitation (cold)
    - Dissimilar material interactions between specimens and loop material
- Flowing forced convection or pumped loop
  - Most realistic conditions for flow
  - Historically, similar qualitative corrosion results as TCL at 10+X cost
  - Necessary progression for other aspects of LM blanket development
  - **Need results ASAP, including with magnets and radiation**



Liquid metal plasma facing components? Li vs. Sn

Li has high vapor pressure

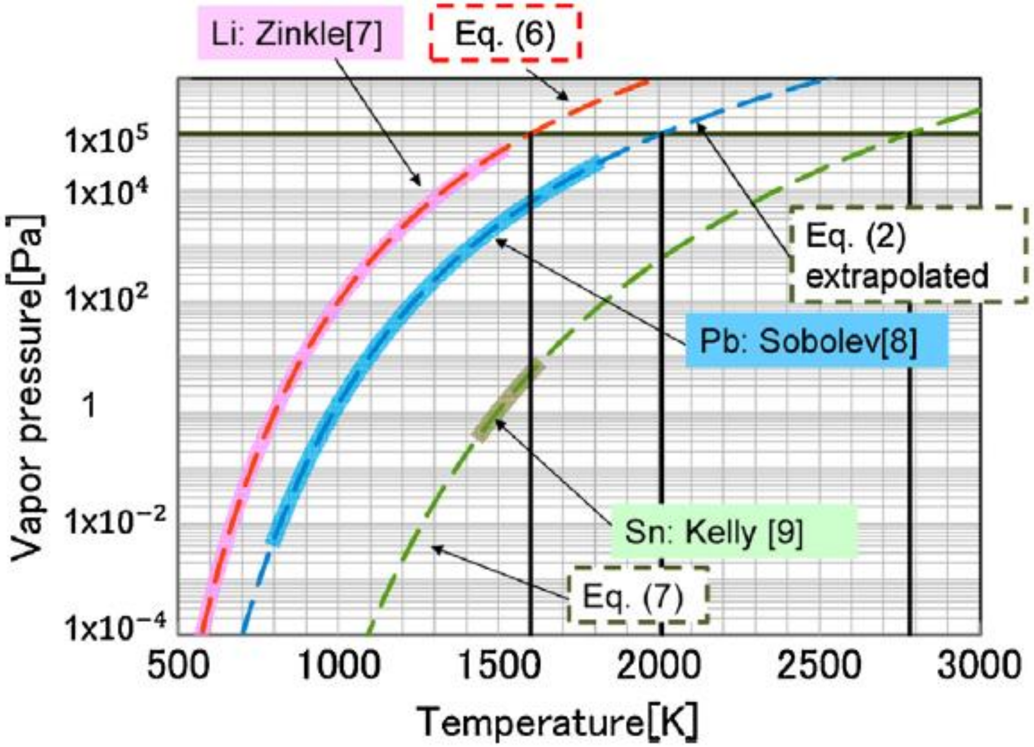
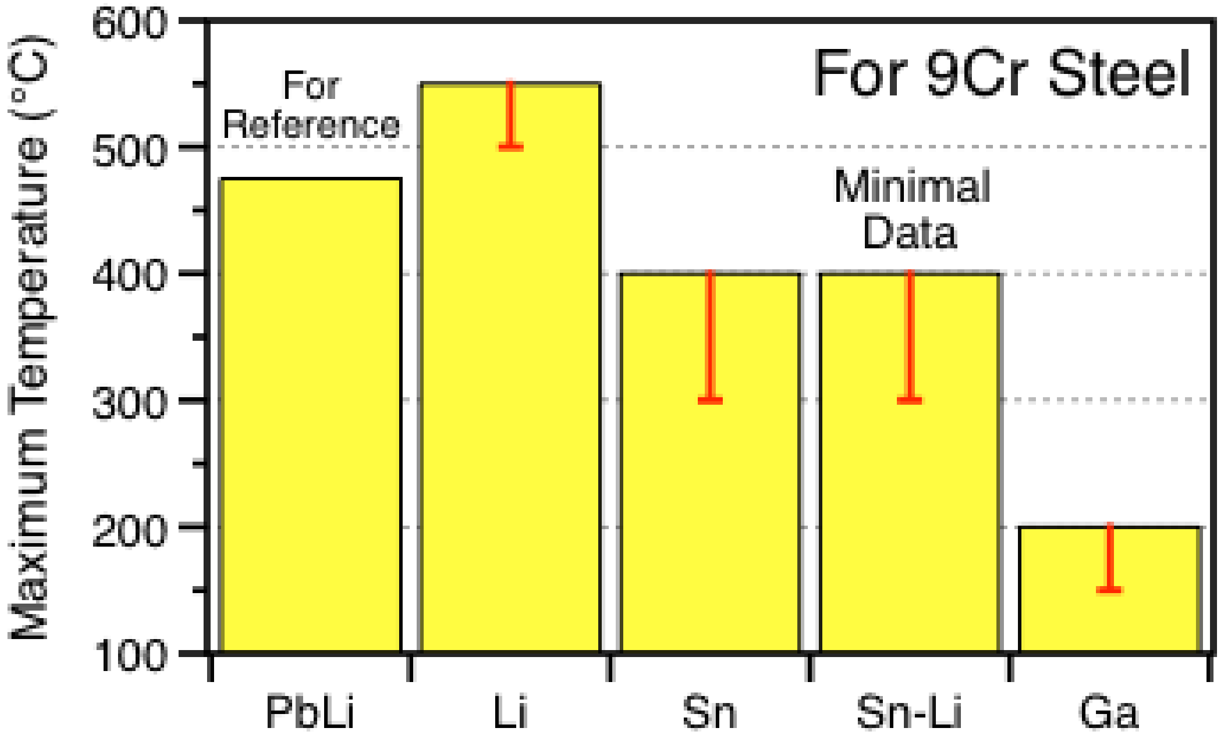


Fig. 1. Vapor pressure of Li, Pb, and Sn given in [7–9] and from Eqs. (2), (6) and (7) for extrapolation.

From Kondo et al.

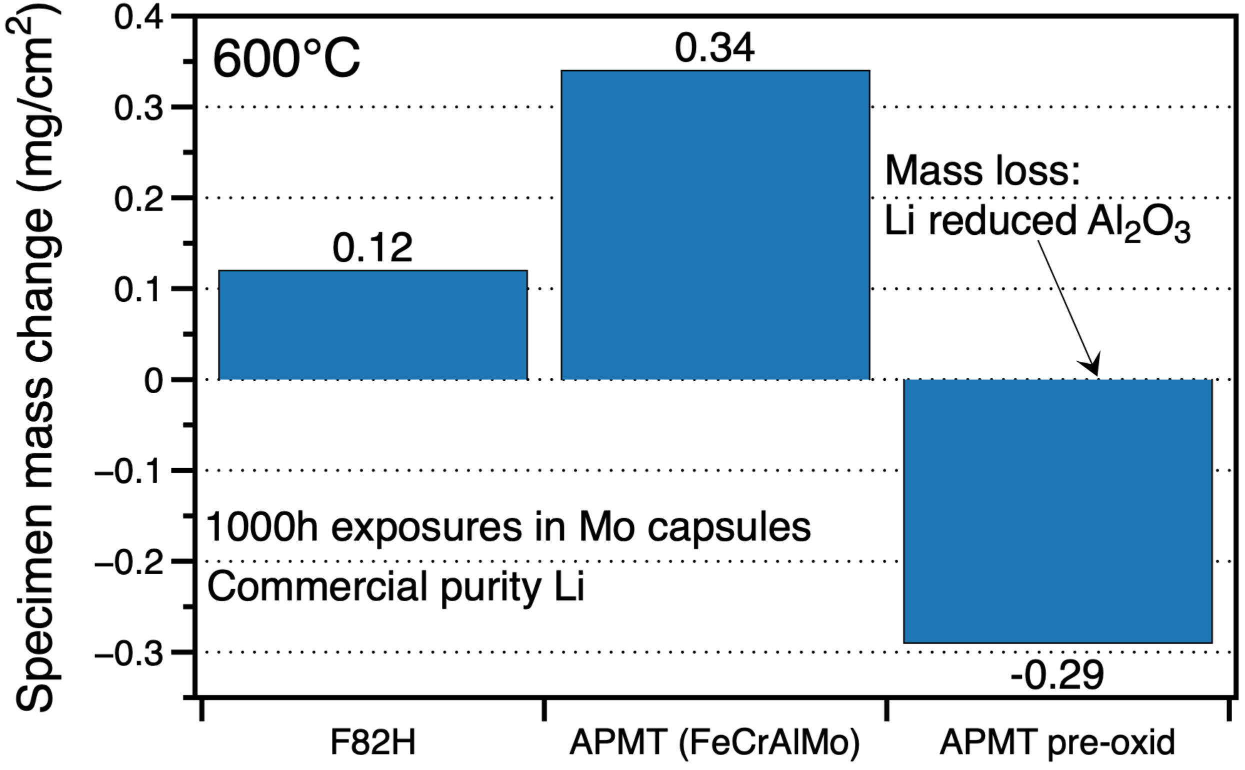
But better Li-RAFM compatibility



#1 Is Li liquid metal embrittlement a concern?
#2 can Sn be compatible?

Li capsule testing: minimal mass changes at 600°C/1000 h

Mass change after 1000h in Mo capsule



F82H: Fe-8Cr-2W
APMT: Fe-20Cr-5Al-3Mo+Y,Zr,Hf,Ti,O
Preox = pre-oxidation for 2h at 1000°C

Static capsule testing:

Thermodynamics:
$6Li+Al_2O_3 \rightarrow 3Li_2O+2Al$

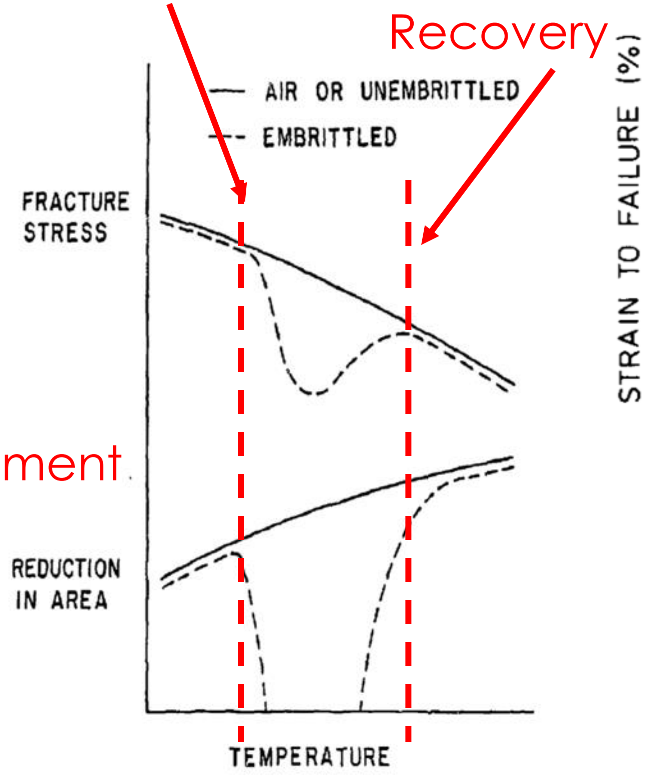
F82H: OK with Li at 600°C

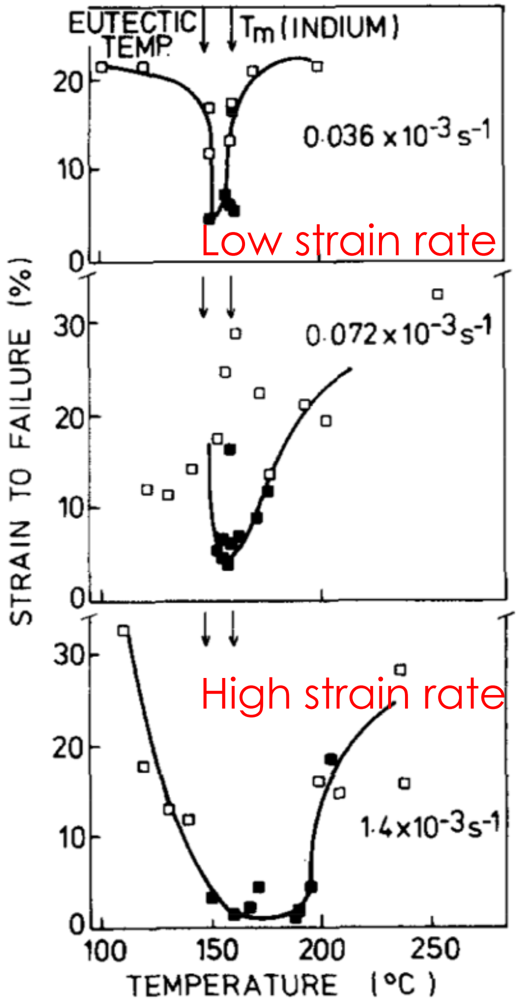
OAK RIDGE National Laboratory

Li Liquid Metal Embrittlement (LME) is still a concern

- Temperature near Li T_m=181°C
- **Traditional differentiation between long-term degradation and short-term LME fracture**
- Role of wetting?
  - Wetting needed for corrosion
- BUT wetting not needed if:
  - Native oxide fractures
- **Is F82H susceptible to Li LME?**

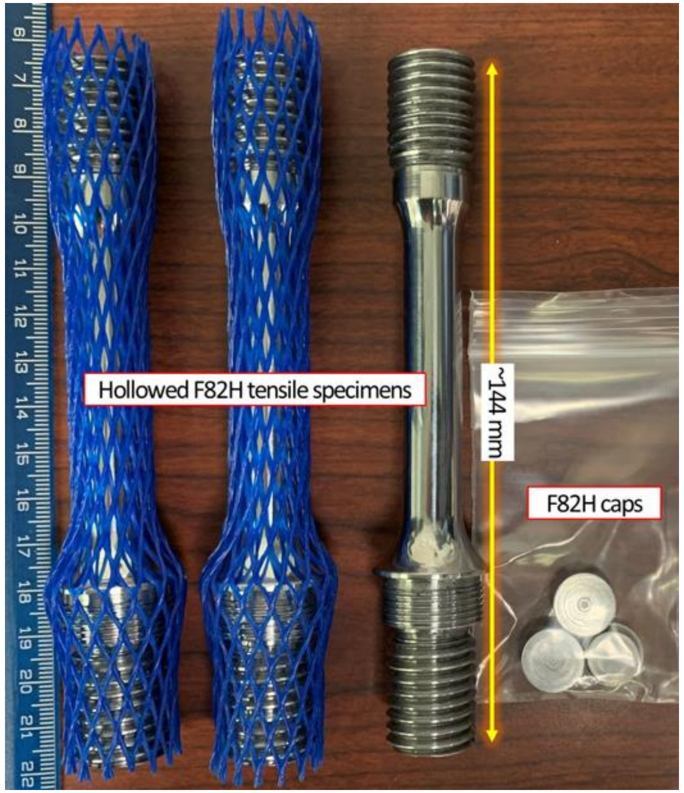
Reduction in ductility or stress required to fracture

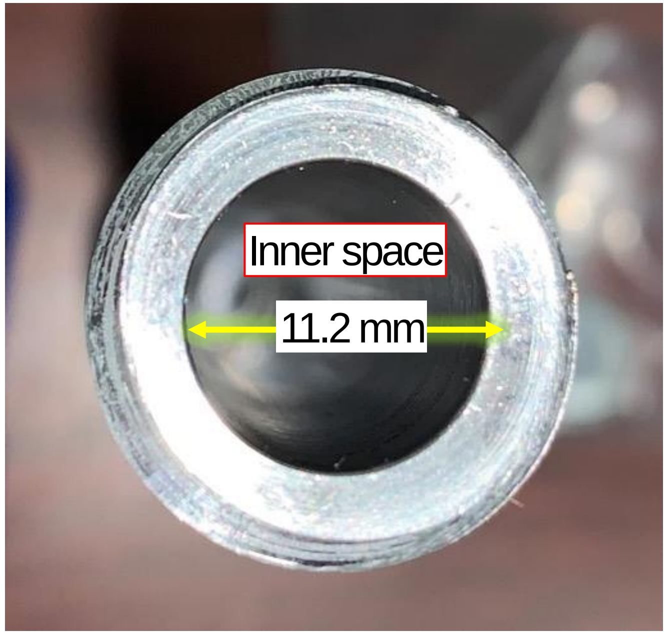




Input from C. Kessel, ORNL

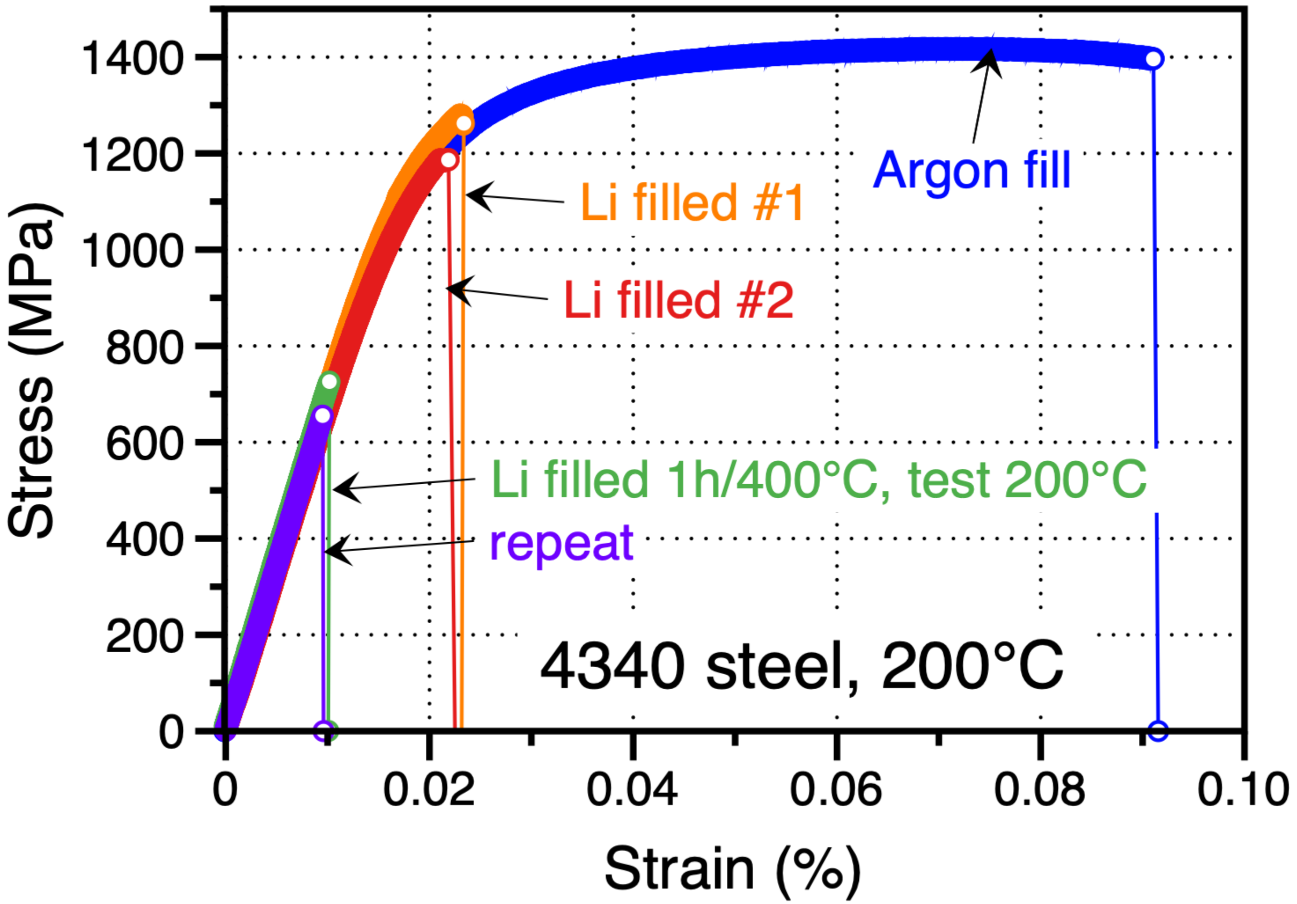
Hollow specimens were machined for safe LME testing





- F82H: Fe-8Cr-2W plate from Japan QST
- 4340 steel (1.8%Ni,0.8%Cr,0.25%Mo,0.4%C)
- 200°C tensile test per ASTM E21
  - Near Li melting point (181°C)
  - Strain rate 0.005/min
  - Controlled Ar environment (no extensometer)

4340 steel: demonstrated hollow specimen methodology

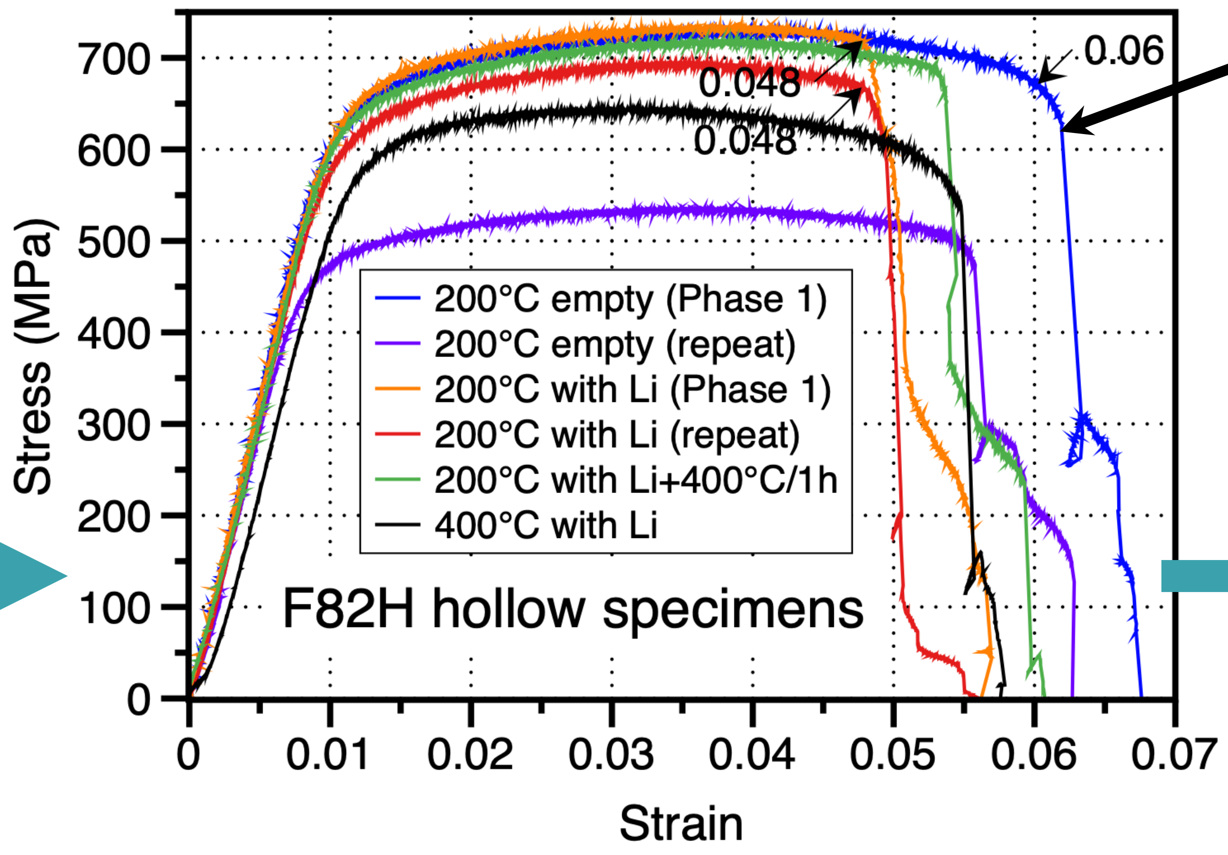


- All 200°C tensile tests, 0.005/mm strain rate
- Plus 400°C/1h anneal for wetting
- Reproducible results

Hollow F82H tensile specimens: no indication of Li embrittlement

Tensile test at 200°C or 400°C: 0.005/min strain rate per ASTM E21

Li-filled F82H: Fe-8Cr-2W tensile specimens

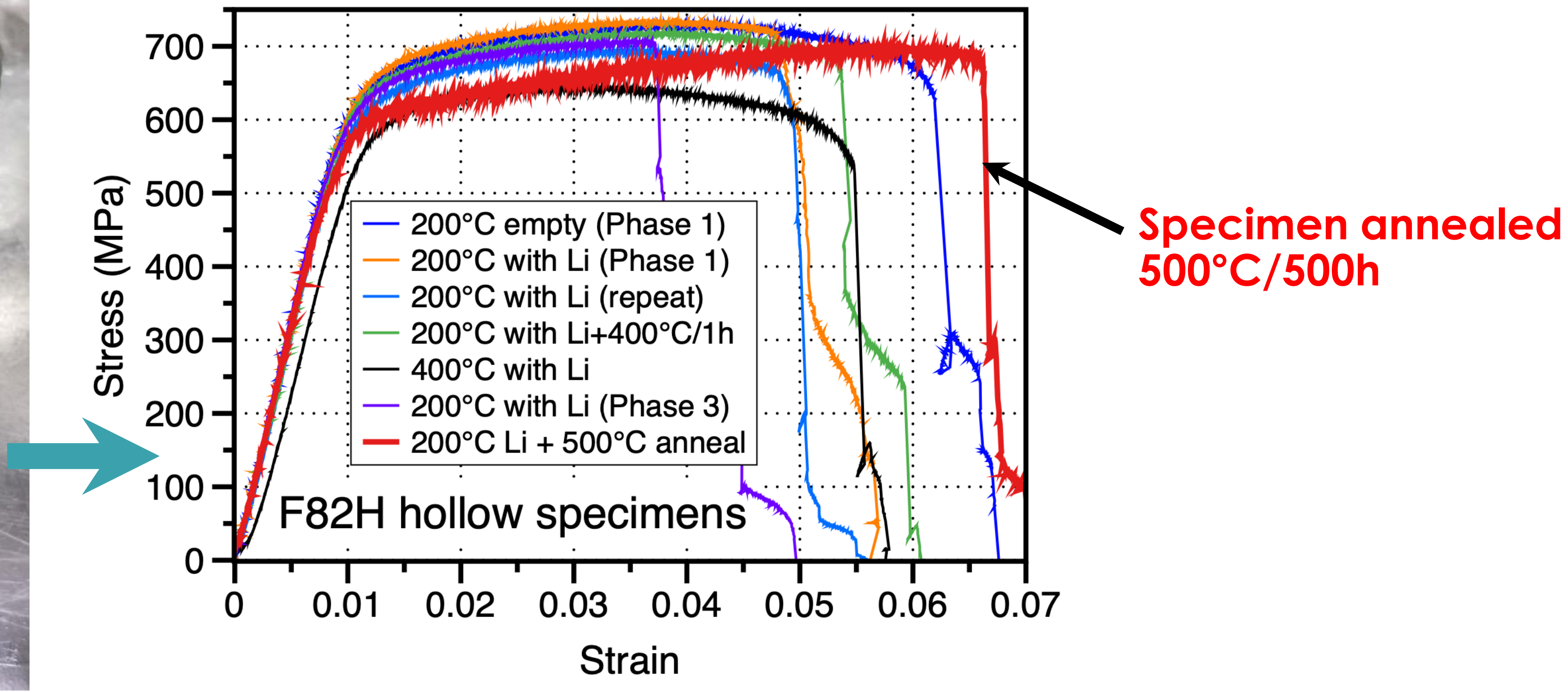


One empty specimen (no Li)

Hollow F82H tensile specimens: no indication of Li embrittlement

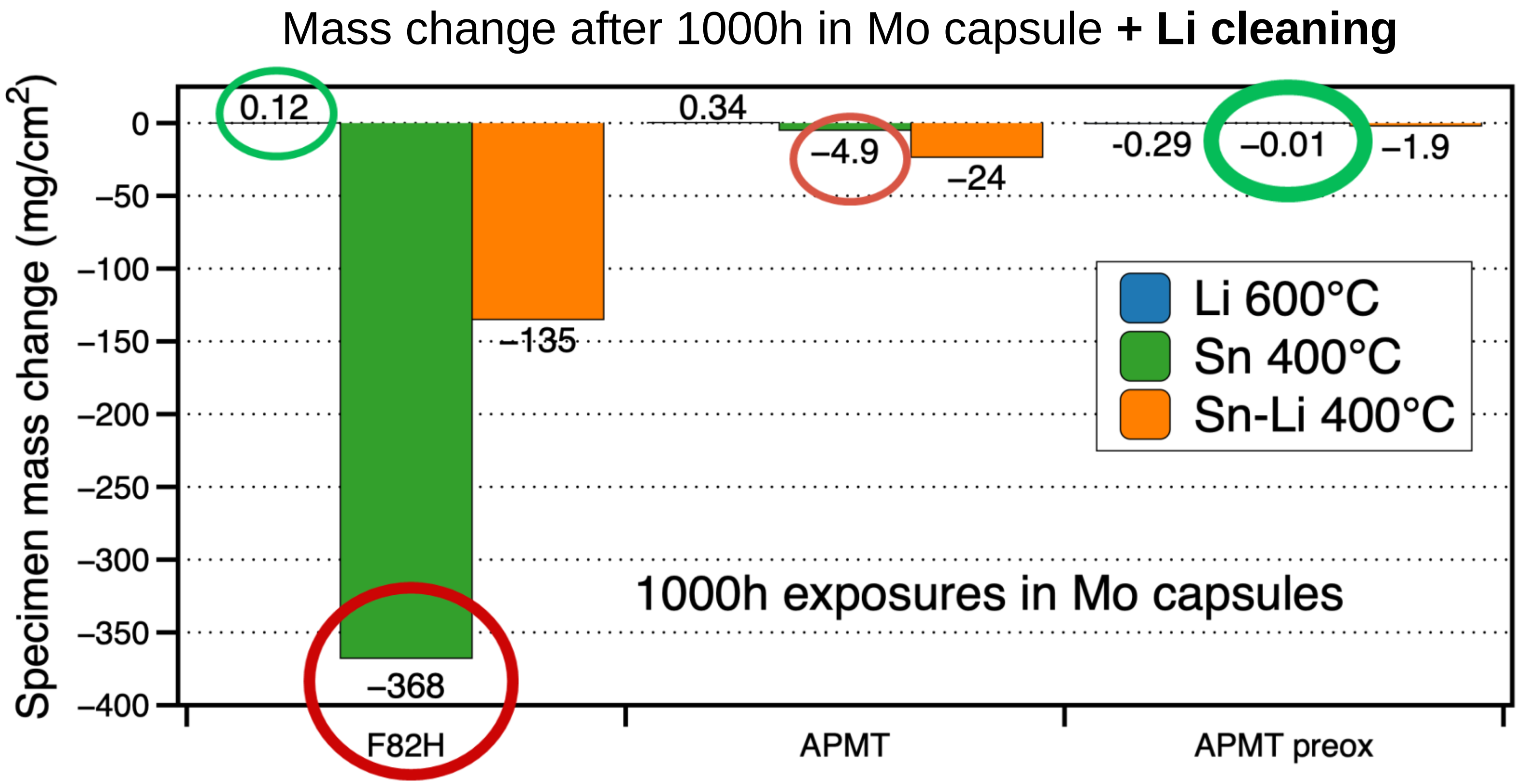
Tensile test at 200°C or 400°C: 0.005/min strain rate per ASTM E21

Li-filled F82H: Fe-8Cr-2W tensile specimens



Manuscript nearly complete:
Romedenne et al. for submission

Sn: bad for F82H at 400°C, good for pre-oxidized FeCrAl



F82H: Fe-8Cr-2W
APMT: Fe-20Cr-5Al-3Mo+Y,Zr,Hf,Ti,O
Preox = 2h/1000°C

Static capsule testing:

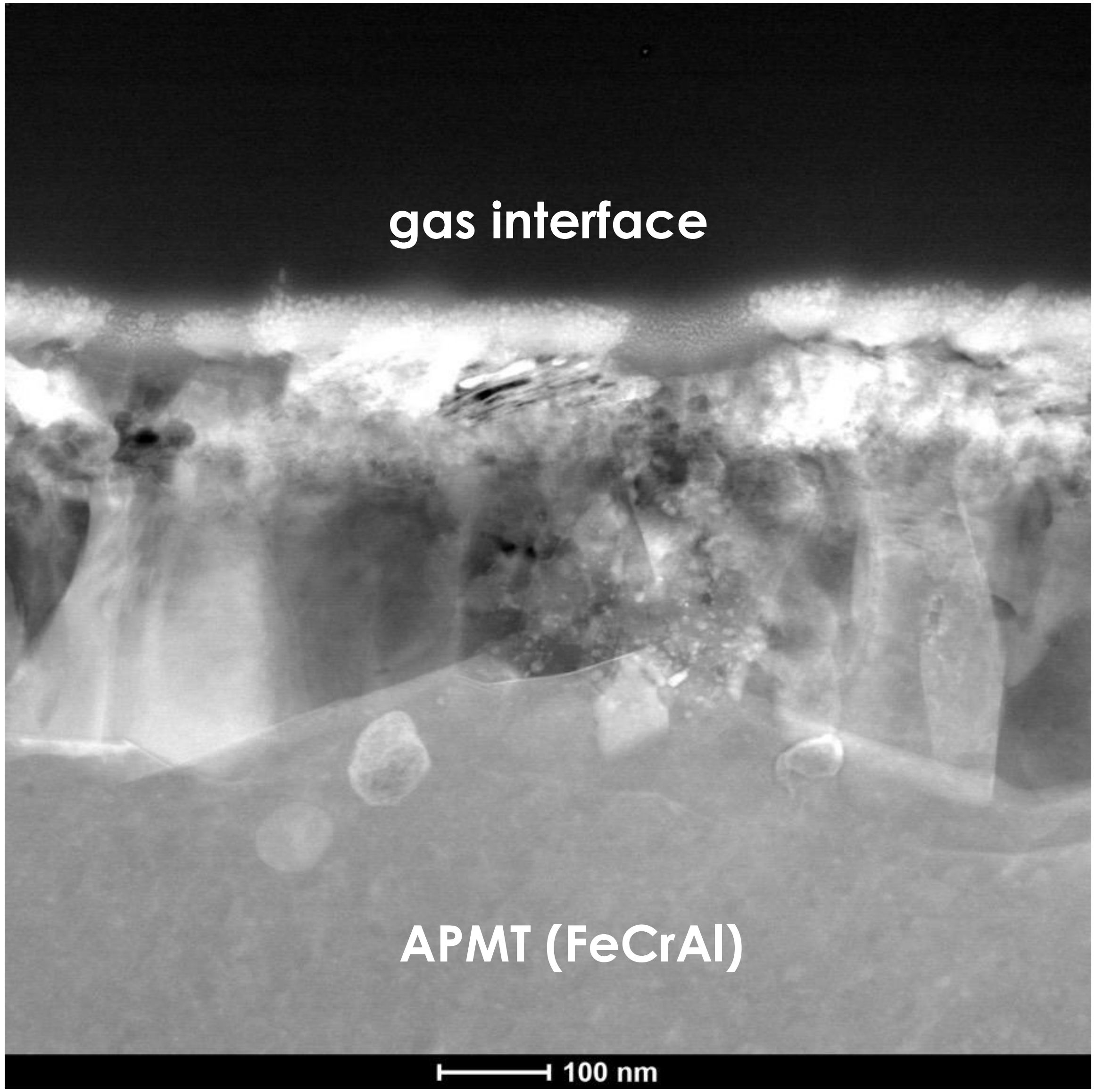
#1 F82H: not compatible with Sn

#2 Sn-Li mass loss for all: no further work

#3 Need flowing test for pre-ox FeCrAl in Sn

Thin α-Al_2O_3 layer formed on APMT after 2 h at 1000°C in air

~400 nm thick

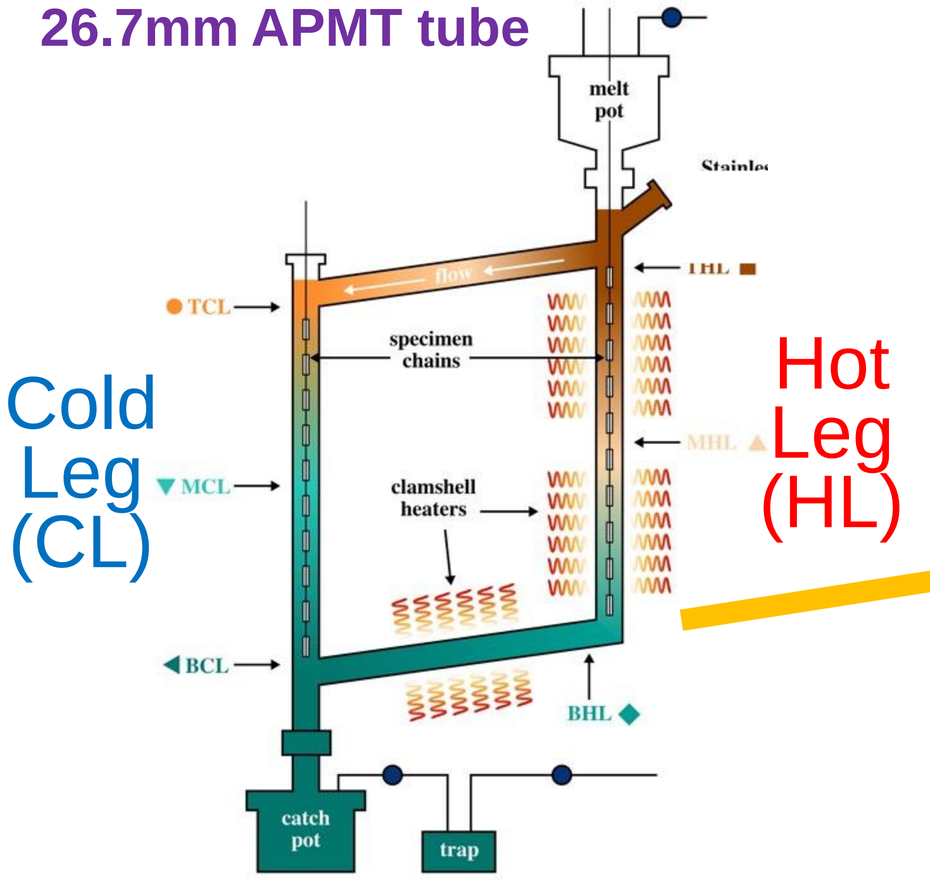


Equiaxed grains
Mixed Fe, Cr, Al oxides

Columnar α-Al_2O_3 grains

TCL: 2 specimen chains in the hot and cold legs of loop

0.75m tall
0.5m wide
26.7mm APMT tube



Three materials exposed:

APMT: Fe-20Cr-5Al-3Mo+Y,Zr,Hf,Ti,O
ORNL ODS: Fe-10Cr-6Al-0.2Y-0.3Zr
Japan ODS: Fe-12Cr-6Al-0.4Y-0.4Zr-0.5Ti

All pre-oxidized 2h/1000°C to form α-Al_2O_3 surface layer (0.5µm thick)

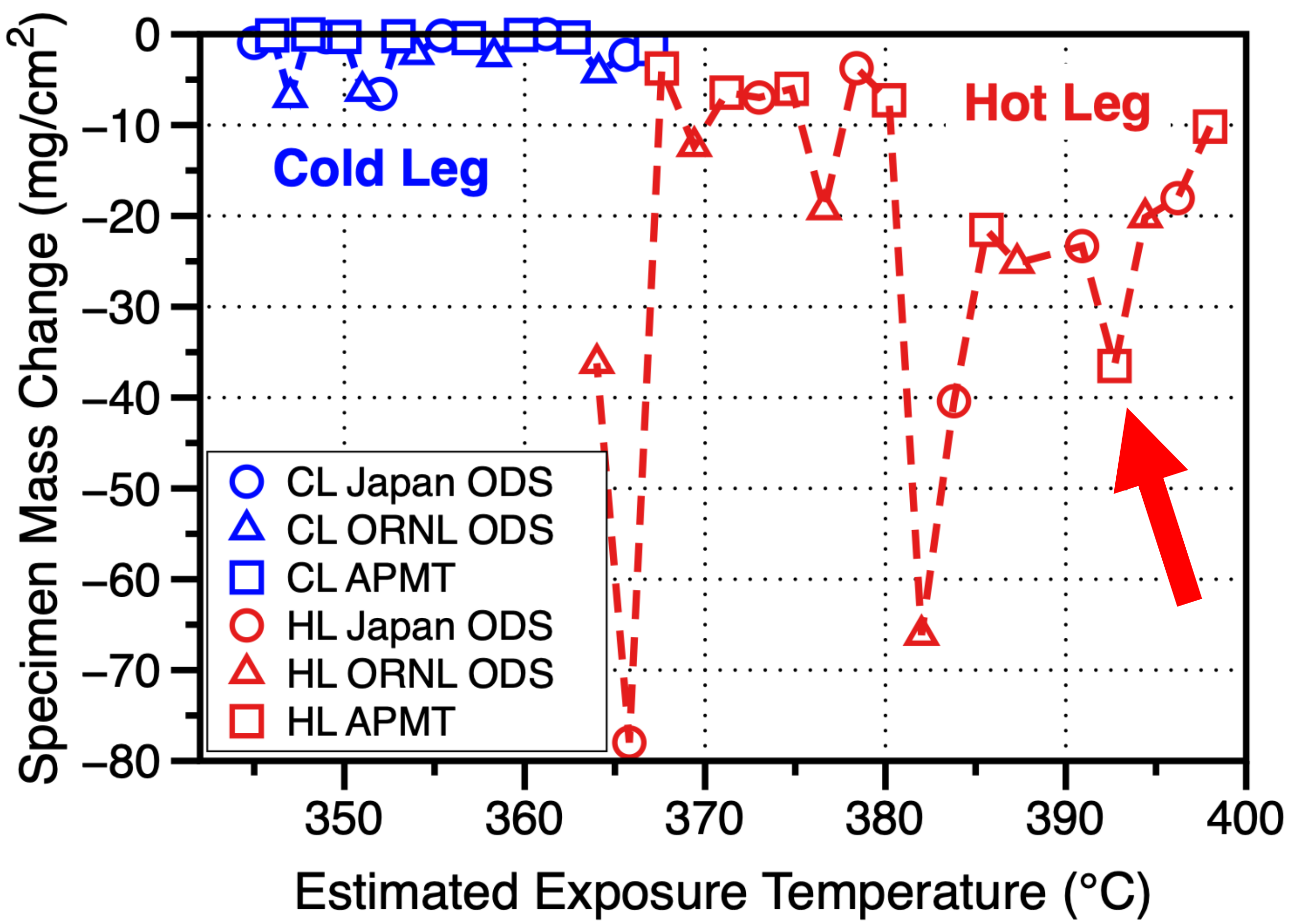
Peak temperature: 400°C

Temperature gradient: 55°C

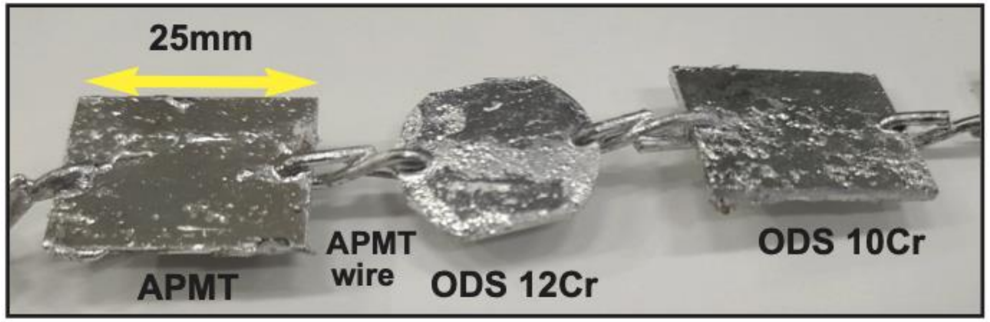
Velocity: ~1 cm/s

OAK RIDGE National Laboratory

Mass change data from Sn loop (1000 h/350°-400°C)



Specimens had Sn remaining on surface after removal
- Removed by dipping in Li at 250°C

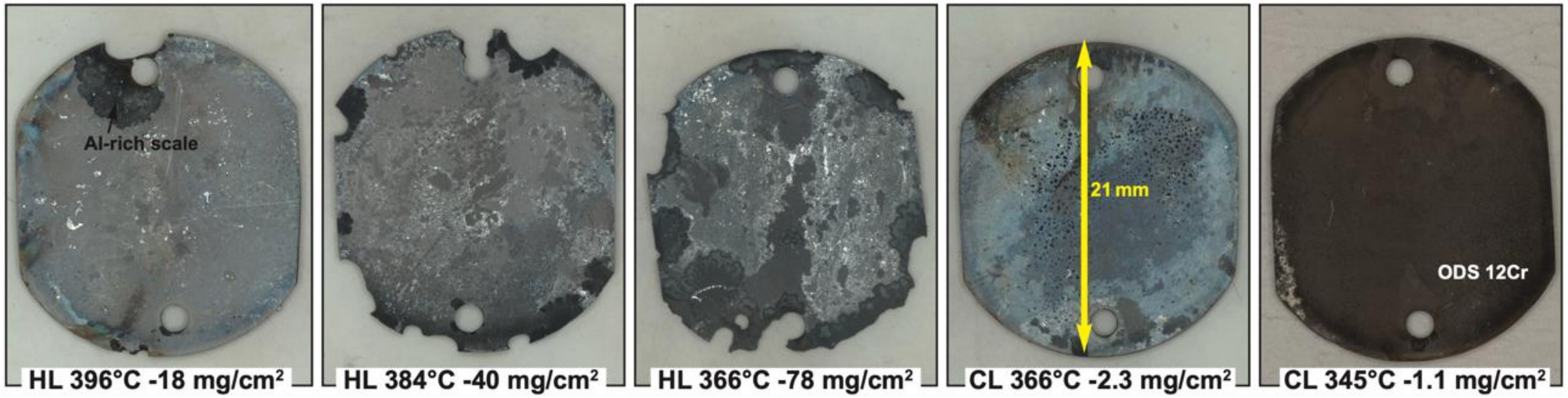


Coupons: 19 x 25 x 1 mm

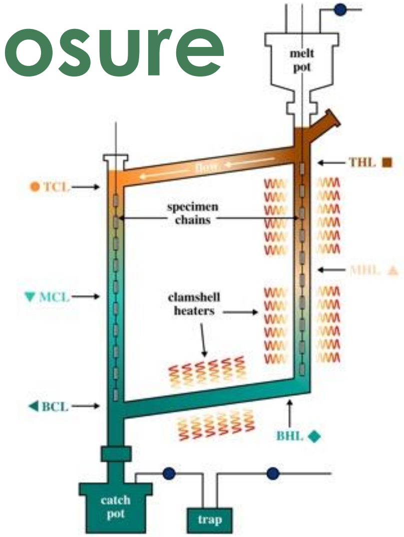
APMT: Fe-20Cr-5Al-3Mo+Y,Zr,Hf,Ti,O
Japan ODS: Fe-12Cr-6Al+Zr,Y
ORNL ODS: Fe-10Cr-6Al+Zr,Y

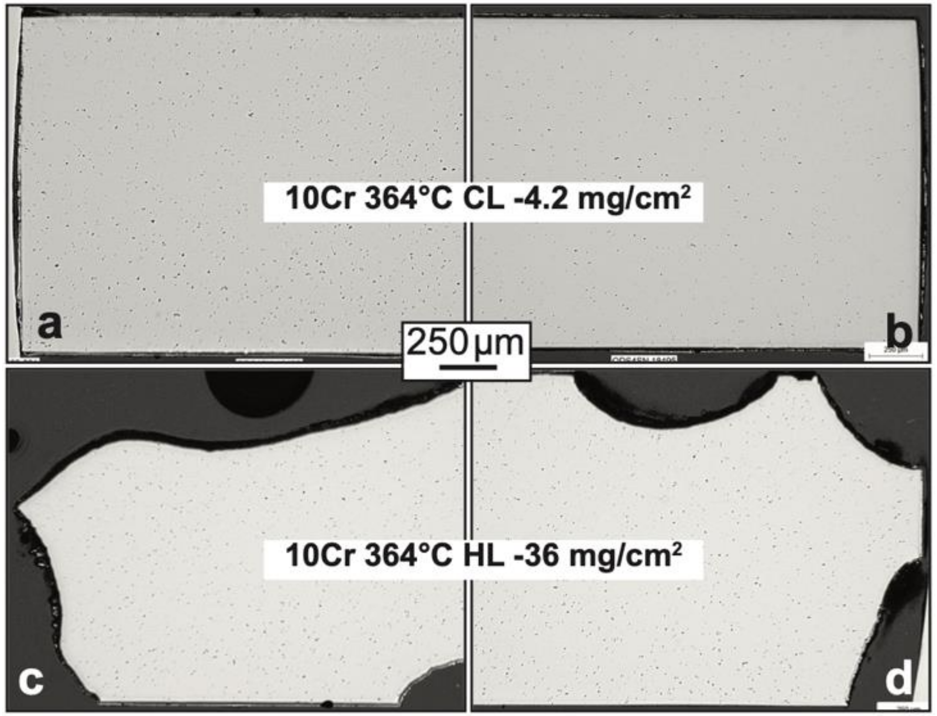
Large mass losses in hot leg for all alloys

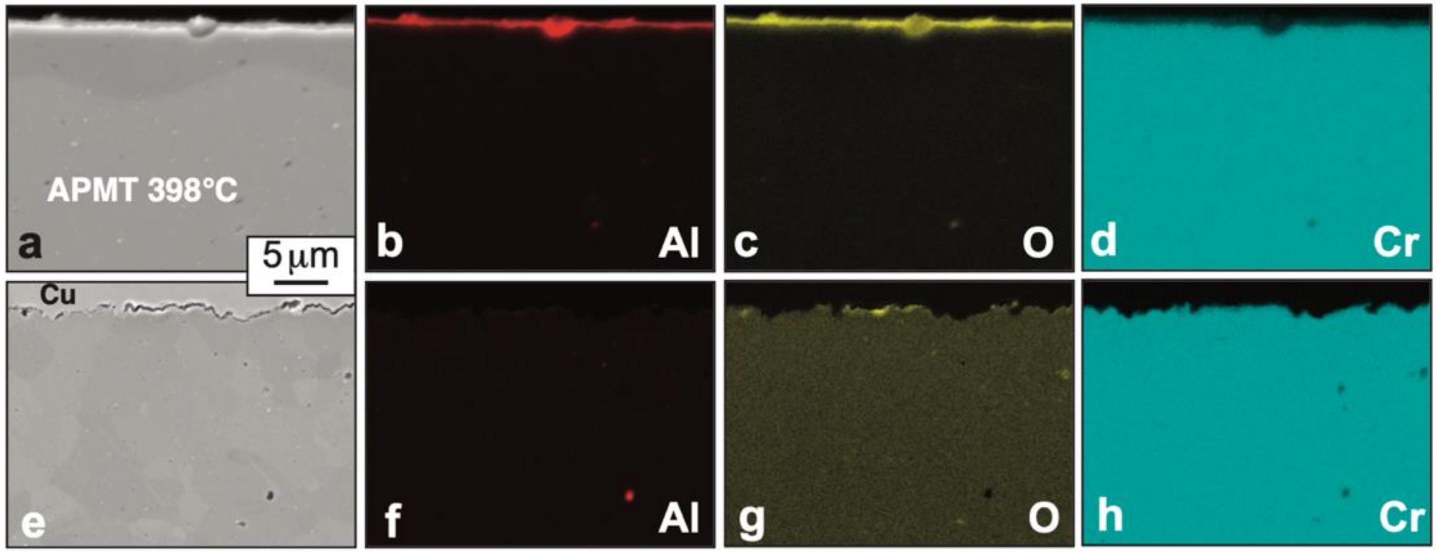
Evidence of pitting on the specimens after Sn exposure



ODS 12Cr
1mm thick







- It appears that breakdowns in the alumina scale lead to dissolution

Oak Ridge National Laboratory

PbLi: expanding on base program foundation

APMT TCL

US Base program (started in 2013)

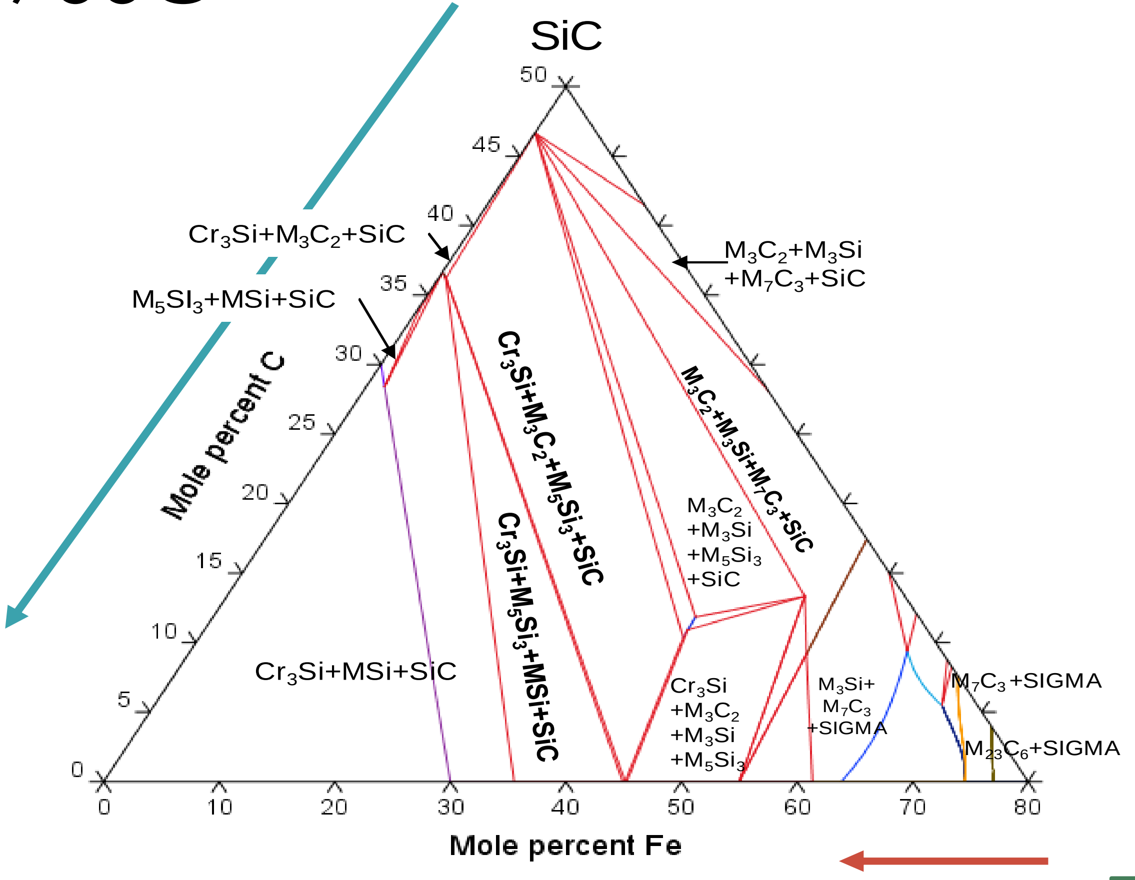
- 3rd Pb-Li loop: 650°C peak, APMT monometallic
  - Small mass changes for APMT (Jun et al. JNM 2020)
- 4th Pb-Li loop: 700°C peak, APMT monometallic
  - Significant degradation at >675°C (Pint et al. FST 2021)

BFC Task 4

- 5th Pb-Li loop: 700°C peak, APMT tubing + CVD SiC/ODS FeCrAl
  - Fantastic dissimilar material interaction (Pint et al. FED 2021)
- 6th Pb-Li loop: 650°C peak, APMT tubing + SiC/aluminized F82H
  - Completed September 2021 (Romedenne JNM 2023)
- 7th Pb-Li loop: 650°C peak, APMT tubing + SiC/aluminized F82H
  - Completed September 2023 (2000 h, study reaction kinetics)

Thermodynamic modeling of Fe-Si-C-Cr

Fe-Si-C + 20 at% Cr
700C



Along SiC-C axis: corresponds to SiC-deposit interface
SiC rich region
Most probable phases
- Cr_3Si
- M_3C_2 (M=Cr,Fe)
- MSi
- SiC

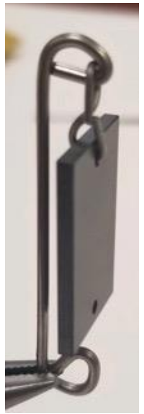
Along Fe,Cr-SiC interface close to surface
Fe,Cr rich region
Most probable phases
- $M_{23}C_6$
- M_7C_3
- M_3Si
- M_5Si_3
- Cr_3Si

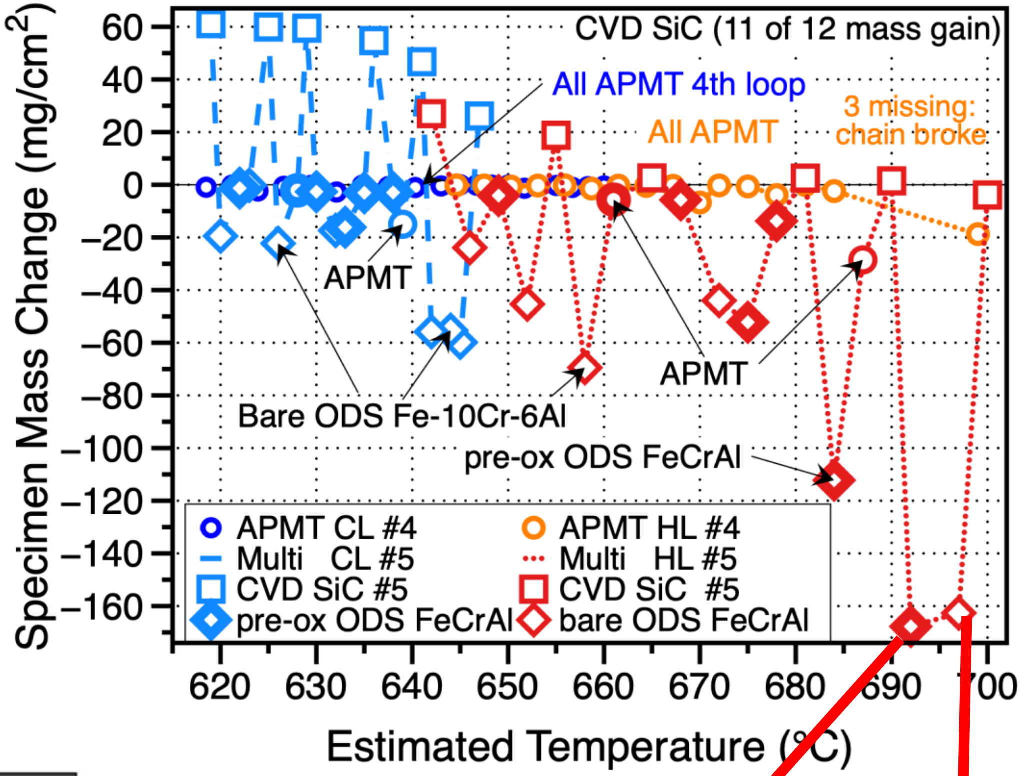
Next: agreement with XRD results!

Pint, Jun and Romedenne, Fusion Engineering Design, 2021

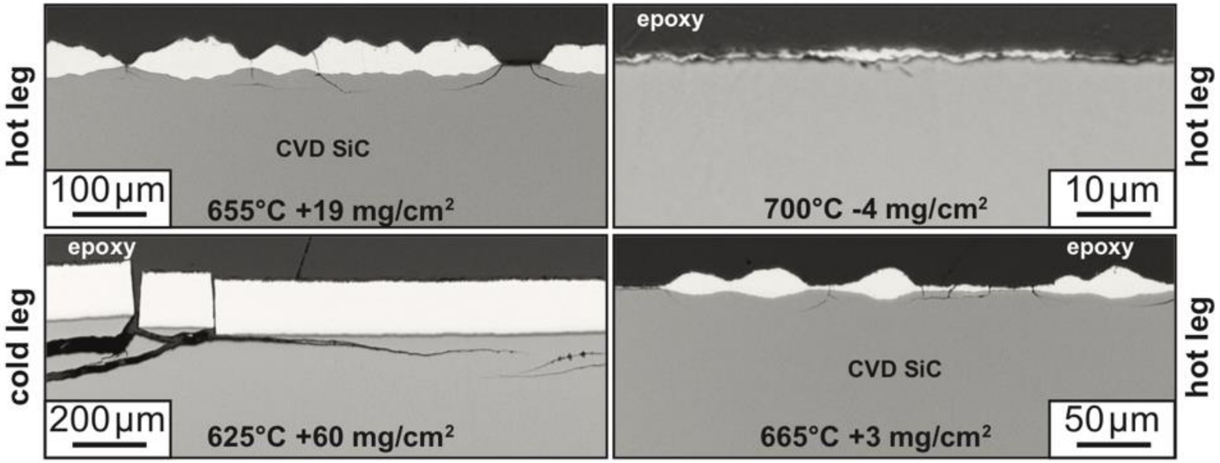
5th PbLi loop: 'Dissimilar material interaction' between SiC & ODS FeCrAl

- High mass changes in Pb-Li
  - CVD SiC gained mass in cold leg
    - Non-uniform (Fe,Cr) carbides + silicides
    - Reaction with Fe and Cr in Pb-Li
  - Large FeCrAl mass losses
    - Acceleration: Fe/Cr removed from Pb
    - Mistake to not-pre-oxidize all specimens
- Conclusion: 700°C is too high!

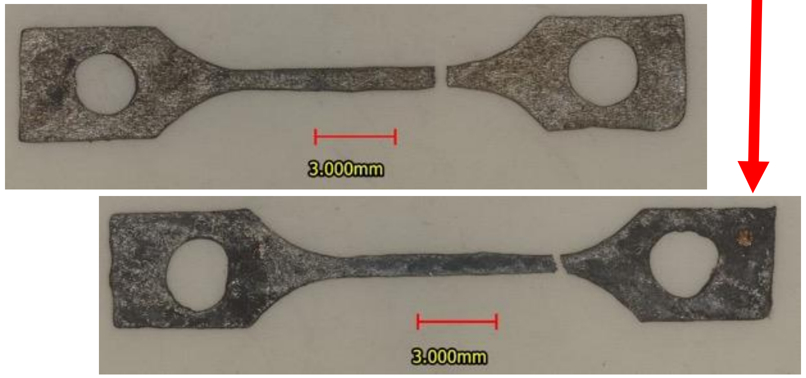




Cross-sections of SiC coupons:



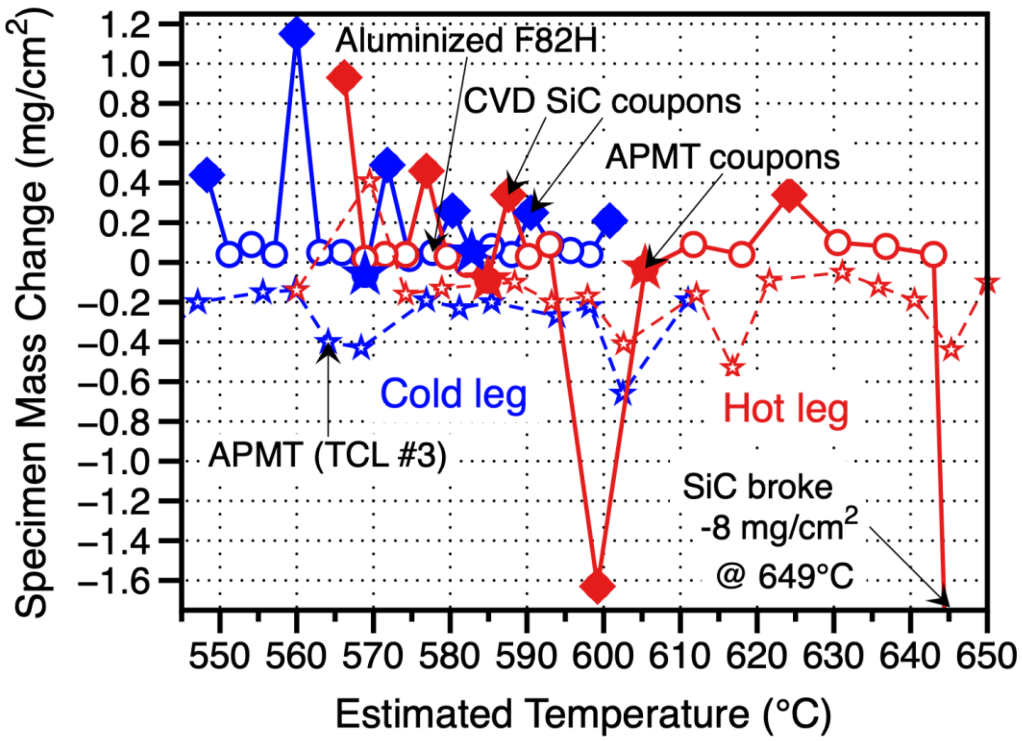
Pre-oxidized ODS FeCrAl



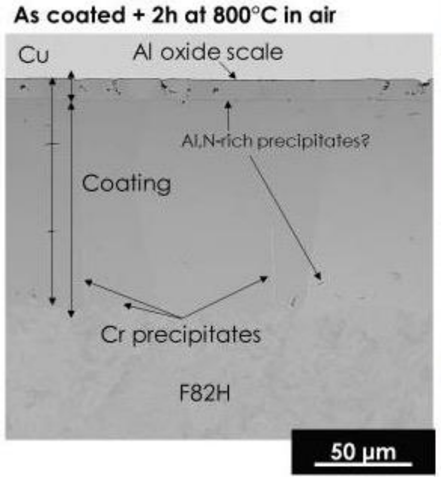
Bare ODS Fe-10Cr-6Al

#6 650°C: significant reduction in SiC reaction at lower T

- Aluminized F82H
  - All pre-oxidized specimens
  - Mostly small mass gains
- CVD SiC
  - One broke (-8 mg/cm^2)
  - 2nd may have chipped
  - **10 of 12 mass gain**
    - **Suggests some reaction**
- APMT coupons
  - 3 of 4 small mass losses like prior monometallic loop #3

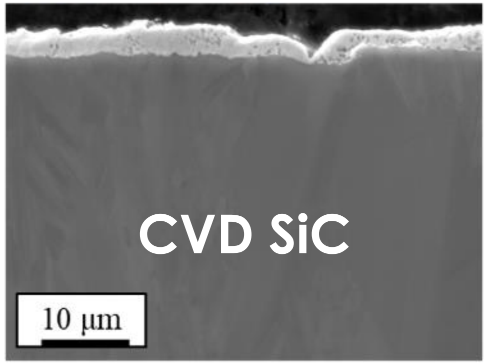


Broken CVD SiC coupon

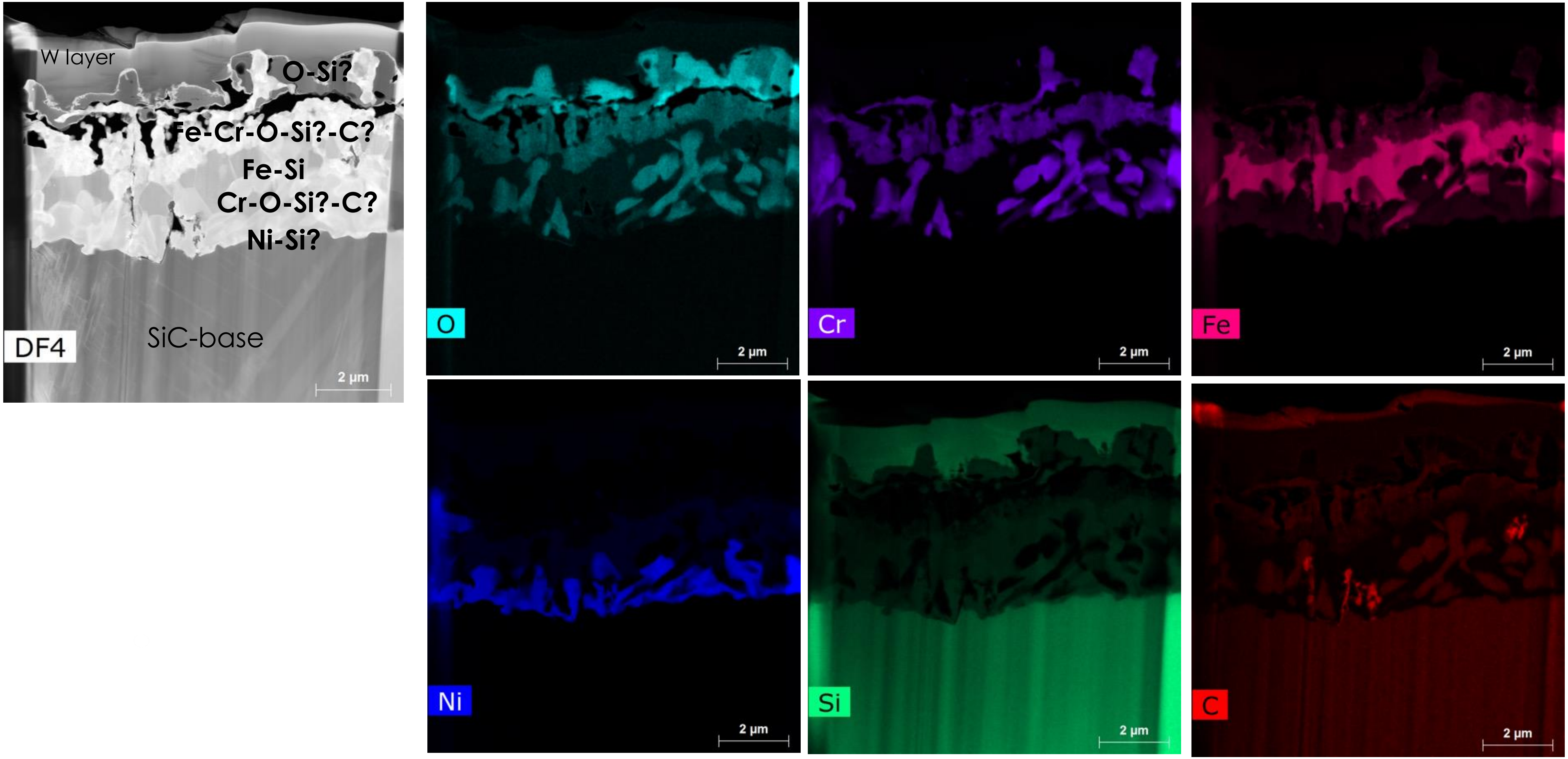


Pack aluminized F82H

556 °C - CL



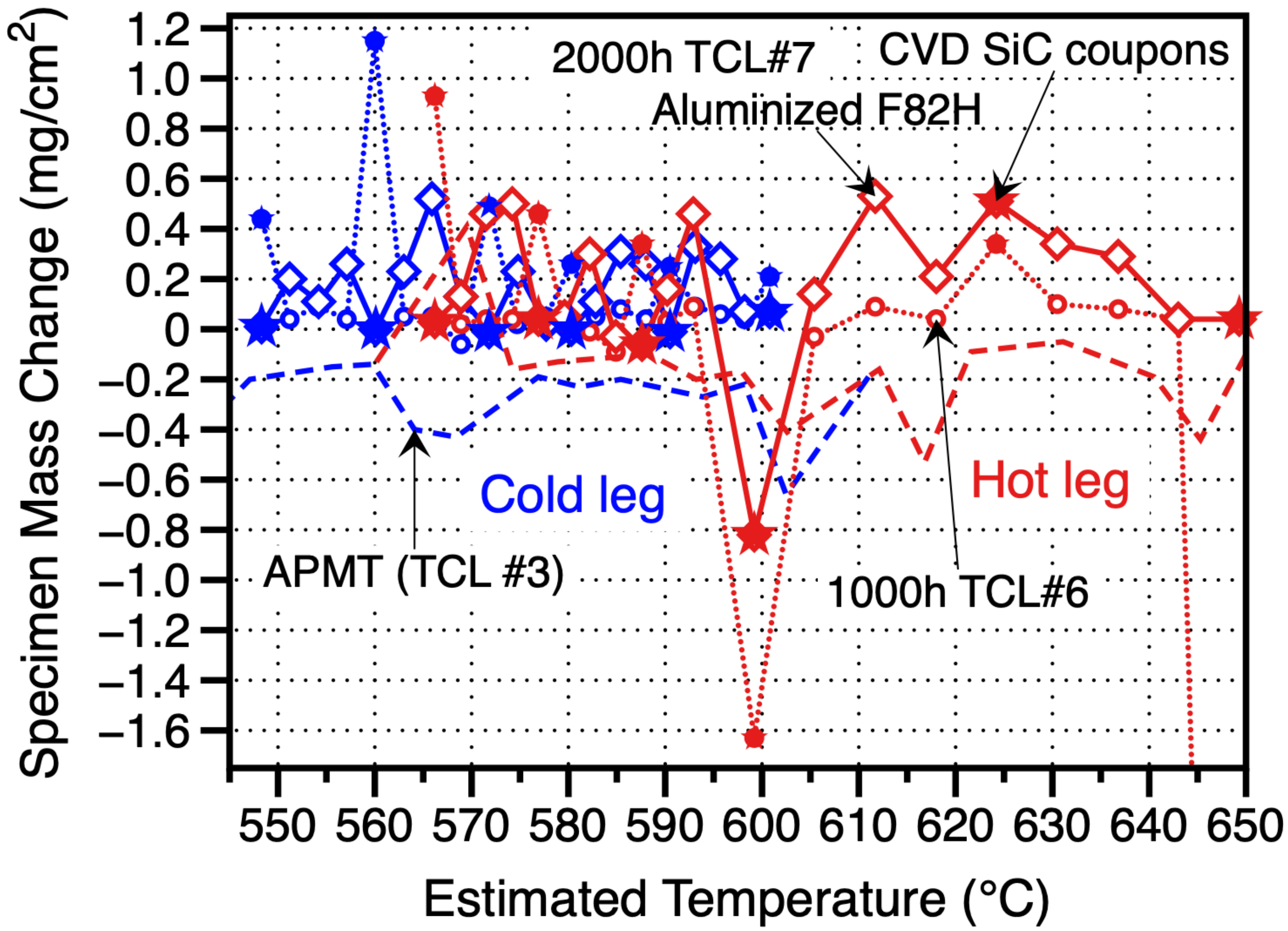
STEM/EDS: maps of reaction product at 566°C (HL)

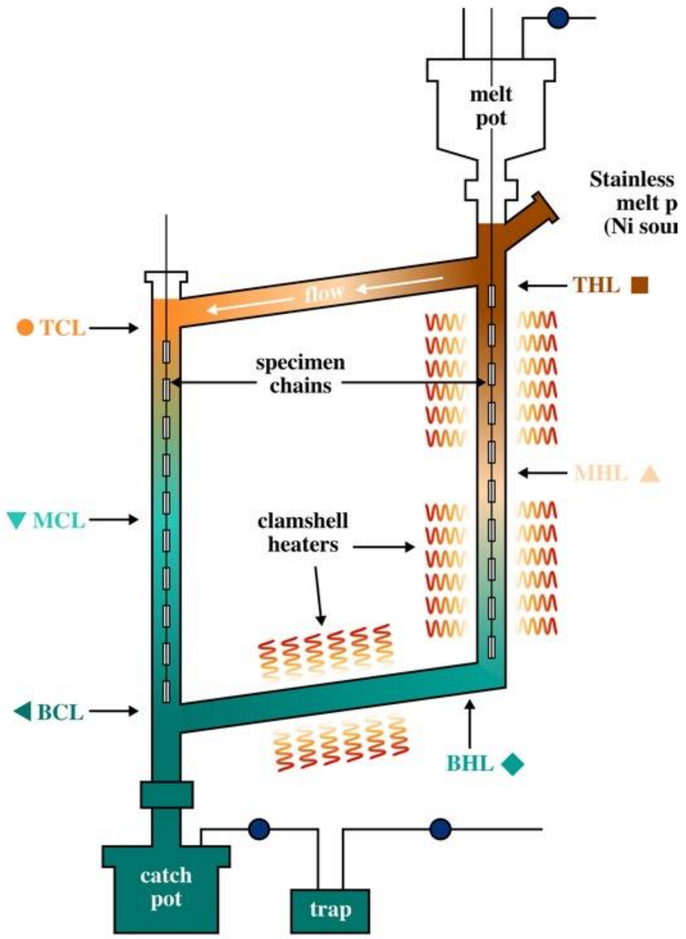


- Complex reaction products, SiC reacting with metals in PbLi: Fe, Cr and Ni?
- Difficult to identify phases

OAK RIDGE National Laboratory

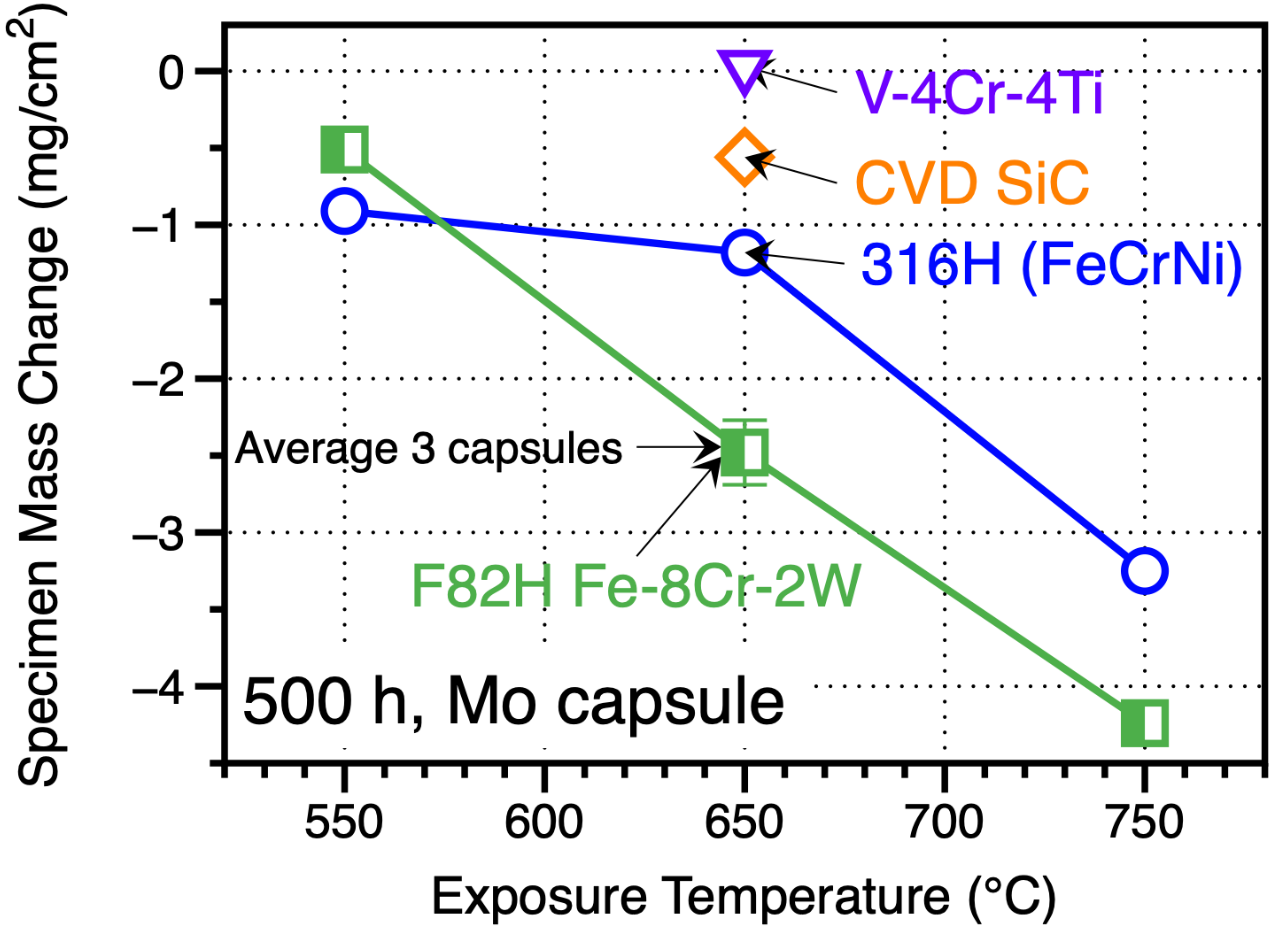
#7 650°C: just completed 2000 h TCL experiment, similar mass change as 1000 h for aluminized F82H and CVD SiC





Characterization in progress for presentation at ICFRM-21

Very recent FLiBe results (static commercial FLiBe)



316 outer capsule

FLiBe salt

specimen

Mo inner capsule

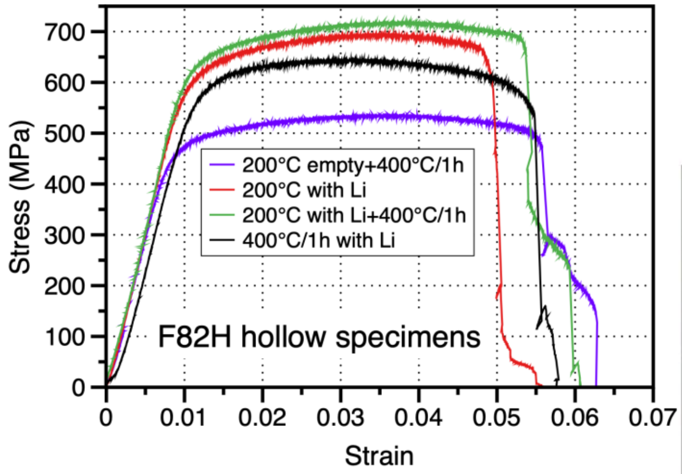
Characterization in progress for presentation at ICFRM-21

Four different liquid compatibility tasks in progress at ORNL

OAK RIDGE National Laboratory

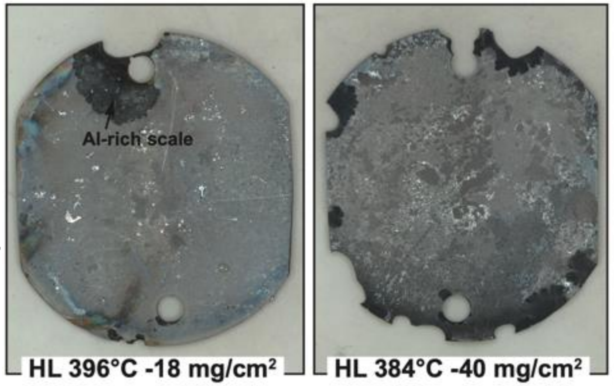
Plasma Facing Comp. (Li)

- Verified LME in hollow specimens with 4340 steel specimen
- No significant LME observed for F82H
  - 200°C tensile/400°C wetting
  - 500°C/500 h anneal



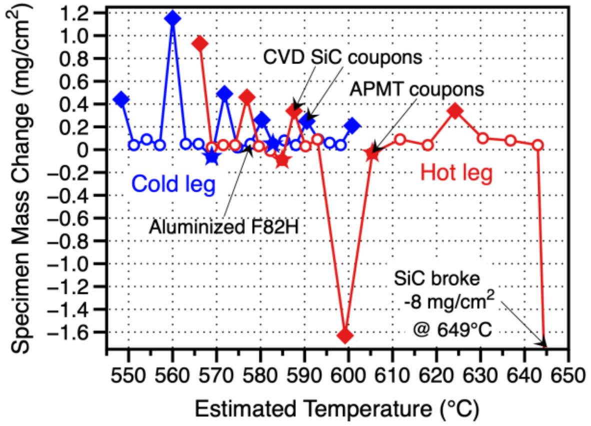
Sn: FRONTIER Task 3

- Flowing Sn loop showed attack
  - Massive FeCrAl dissolution unlike static tests
  - High hot leg loss
    - Al_2O_3 not protective
  - **FeCrAl/Sn not viable**
- Complete HFIR irradiation PIE



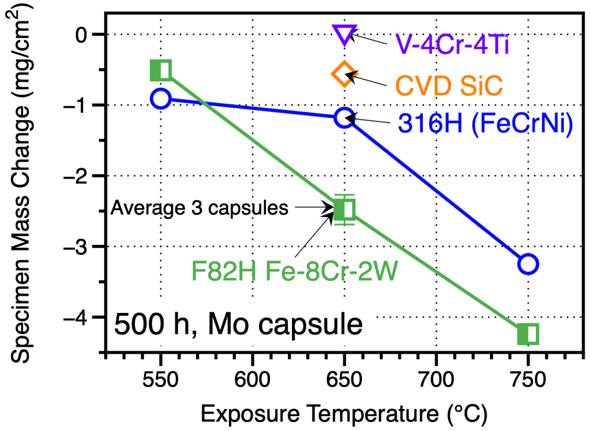
Blanket (PbLi)

- PbLi loop #5: >675°C massive SiC-FeCrAl interaction
- PbLi loop #6: reduced interaction CVD SiC-aluminize F82H: 650°C
- PbLi loop #7 done:
  - 2000 h, 650°C to study reaction kinetics of SiC in PbLi

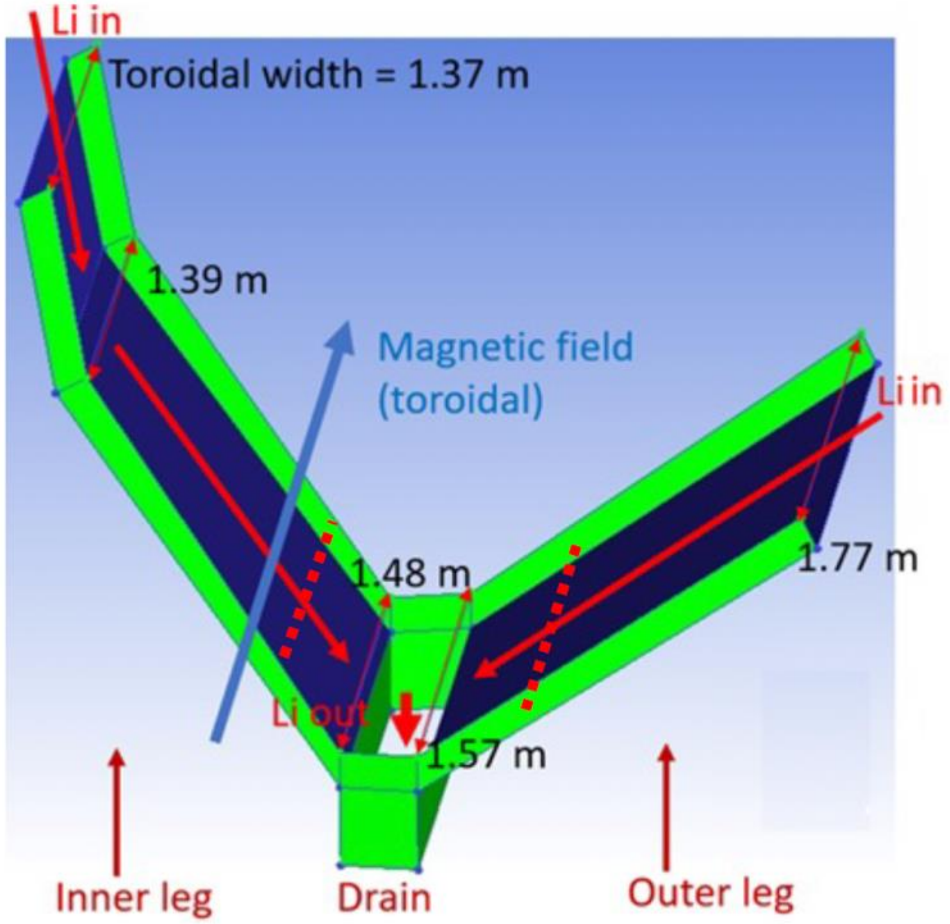


Blanket (FLiBe)

- Linited FLiBe data
- Initial



Questions?



Li in
Toroidal width = 1.37 m
1.39 m
Magnetic field
(toroidal)
Li in
1.48 m
1.77 m
Li out
1.57 m
Inner leg
Drain
Outer leg

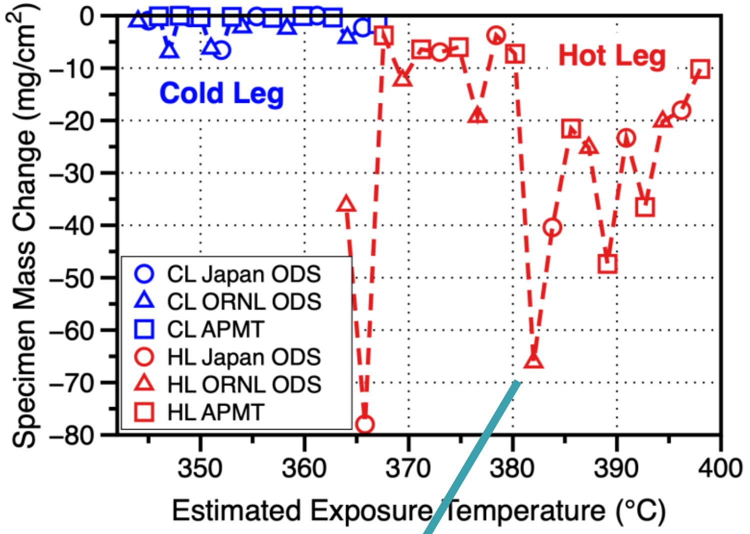
Frontier Task 3: Flowing Sn test at ≤400°C done, HFIR capsules exposed

Objective: Evaluate compatibility of flowing Sn with pre-oxidized FeCrAl alloys at 350°-400°C

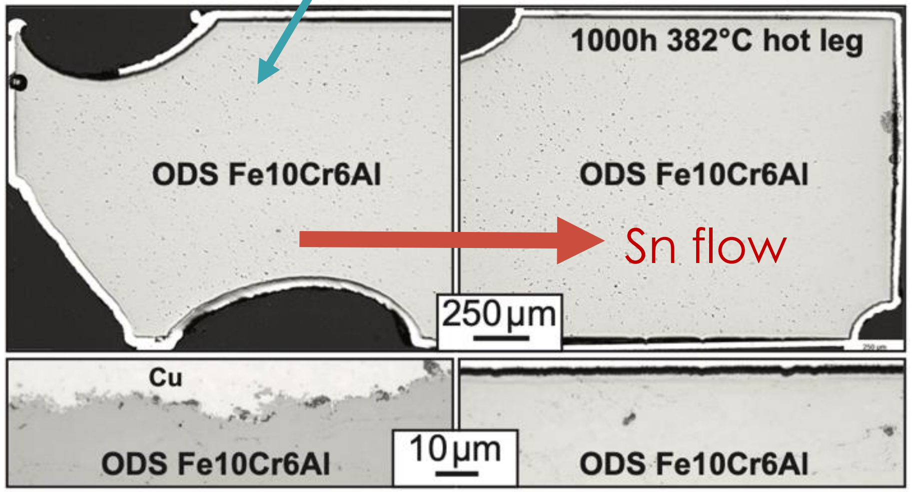
Approach: a thermal convection loop (FeCrAlMo alloy APMT) ran 1000 h with 3 alloy specimens: APMT, Japan ODS(12Cr-16Al) and ORNL ODS (10Cr-6Al); all pre-oxidized for 2h at 1000°C before exposure

Result: Pre-oxidation was not sufficient to protect these alloys in these conditions.

Next: HFIR irradiation without Sn (3mm disks) and with Sn (SSJ tensile specimens): **capsules being built for next HFIR cycle**



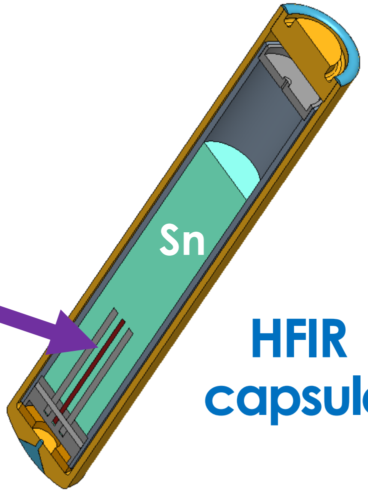
Mass change data from specimens exposed to flowing Sn for 1,000 h with a peak temperature of 400°C



Light microscopy of ODS FeCrAl after exposure: No surface oxide observed in pitted regions

APMT 3 mm disk

ODS SSJ



HFIR capsule