Microstructure and Properties of Hastelloy X Fabricated by Additive Manufacturing

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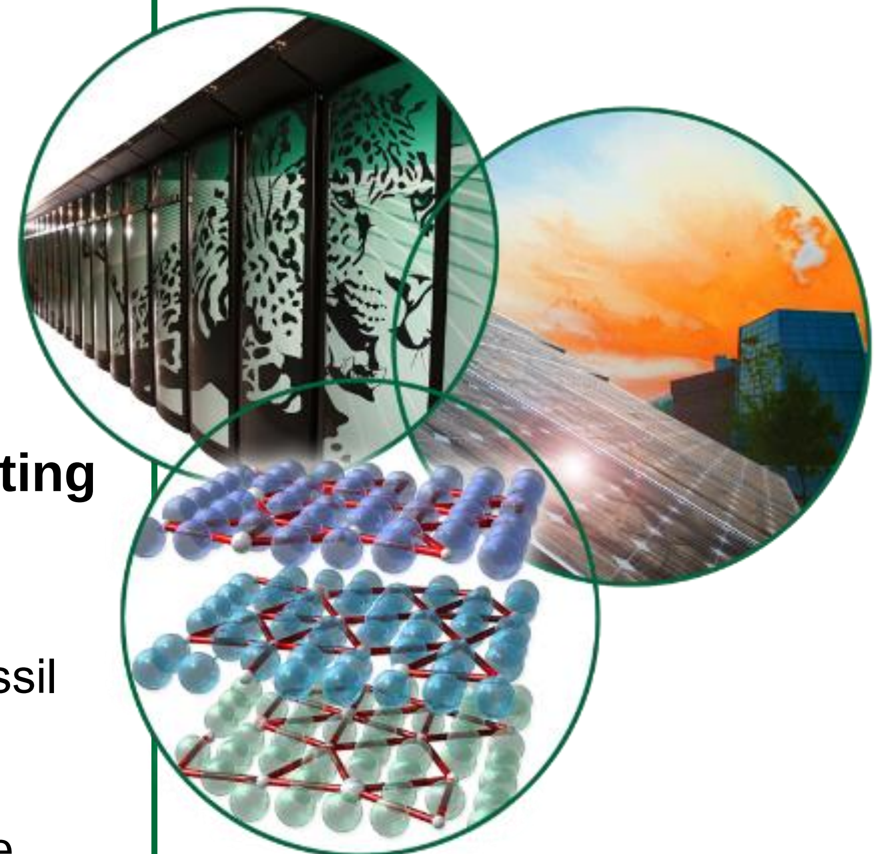
Oak Ridge National Laboratory



2017 University Turbine Systems Research (UTSR) Project Review Meeting

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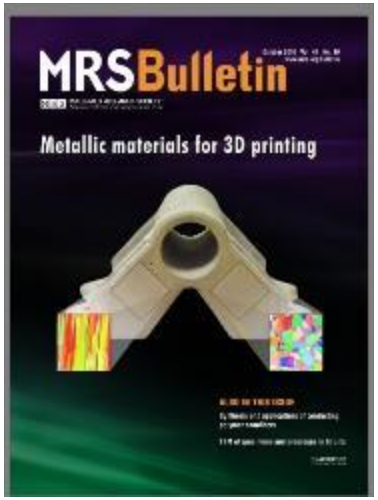
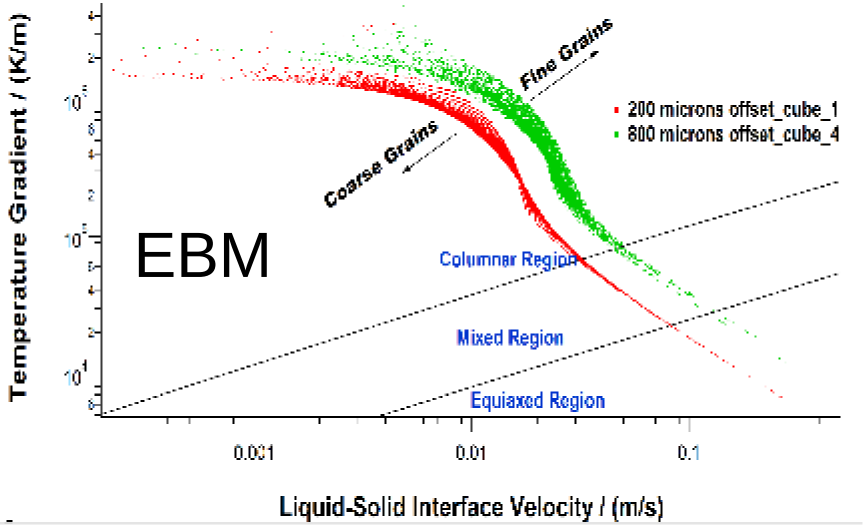
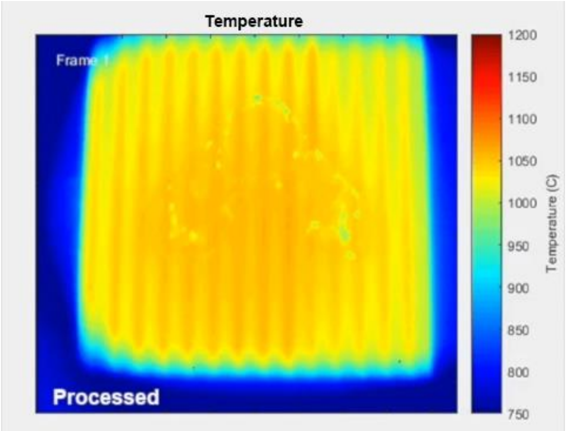
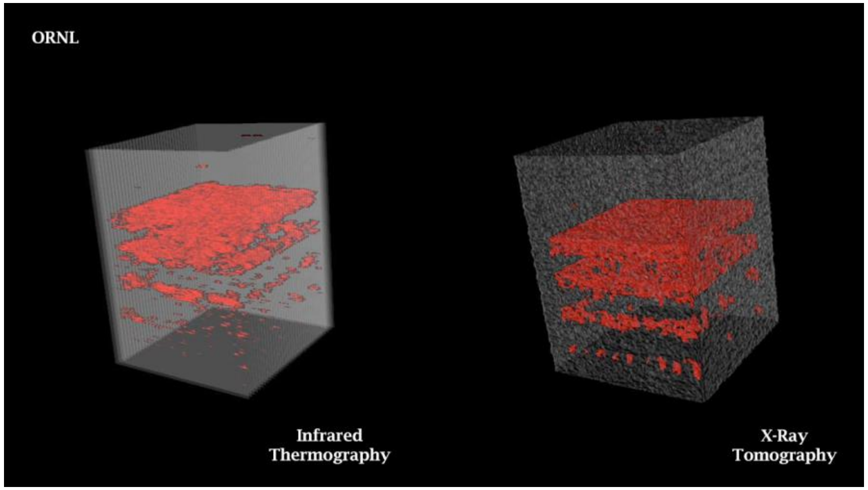
Many Exciting Research Topics on Additive Manufacturing









- Microstructure control
- Modeling at various stages
- In-situ monitoring
- Data Analytics

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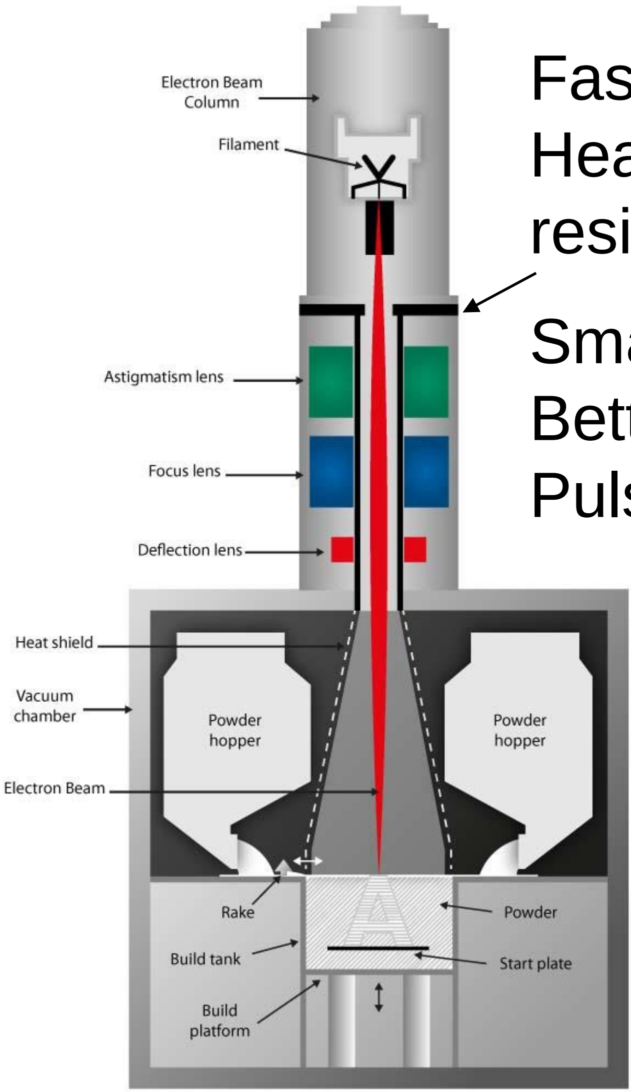
Project Objectives

- Optimize additive manufacturing (AM) fabrication processes for solution strengthened Hastelloy X (HX, Ni-22Cr-19Fe-9Mo) gas turbine components (Fuel injector, Combustor)
- Properties requirement depends on component and application (prototyping, production and repair)
- Compare two AM techniques, electron beam melting (EBM) and selective laser melting (SLM).
- Generate data (Tensile, Fatigue, Creep, Oxidation) relevant for FE applications for Hastelloy (HX, Ni-22Cr-18Fe-9Mo) alloy
- Effect of annealing or HIP'ing on microstructure and mechanical properties
- Effect of different EBM precursor powders on mechanical and oxidation behaviors

HX Made by EBM and SLM

Ebeam (Arcam S12)

Laser (Renishaw AM250)



Faster
Heated bed = lower residual stress

Smaller beam size
Better resolution
Pulsed laser beam

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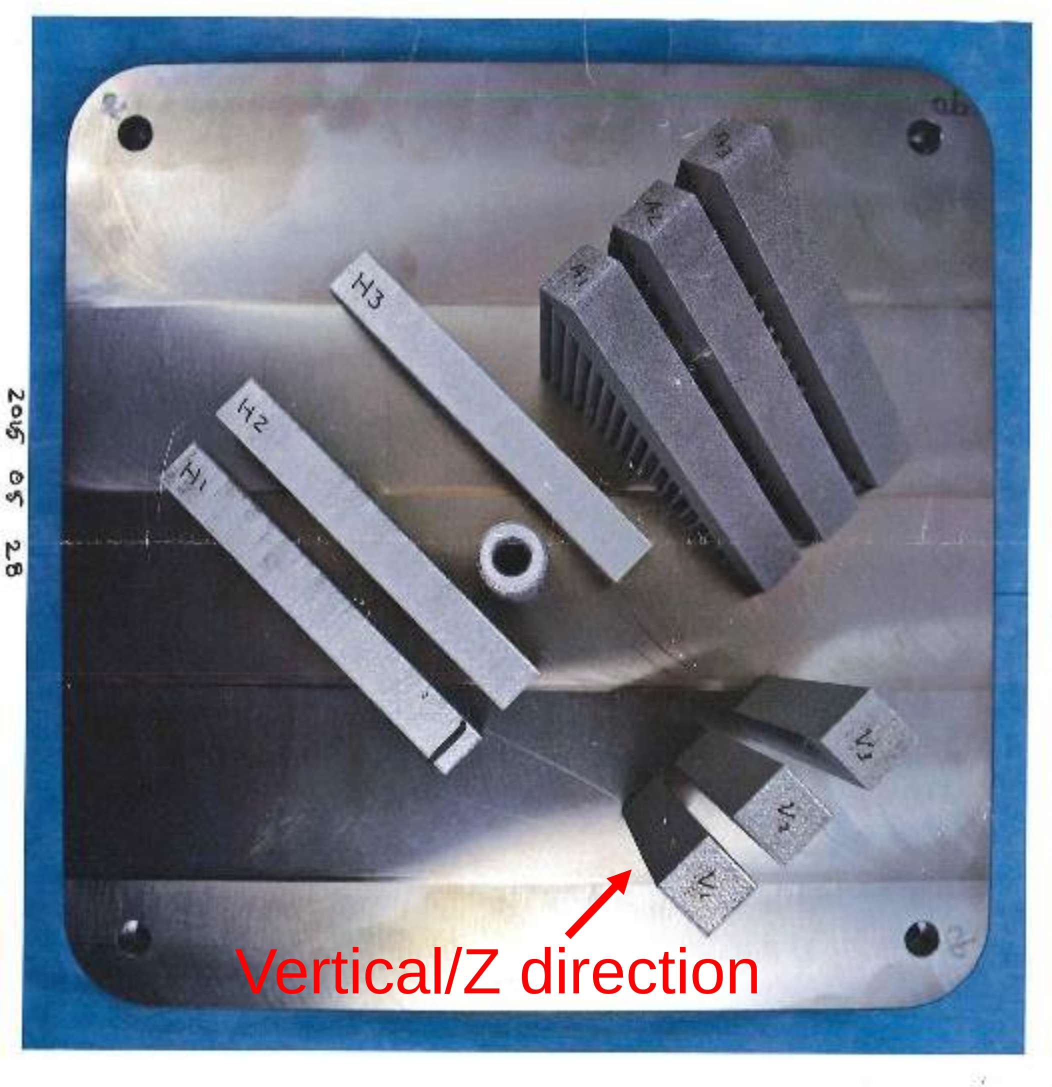
Small SLM HX Cube for Parameter Optimization Based on Microstructure

- Spot time
- Spacing
- Energy

ORNL SLM Builds For Microstructure and Mechanical Characterization



- Previous build fabricated with leftover powder from AMO project
- Characterization of the microstructure and tensile properties in 3 directions
- Presentation on vertical specimens
- Study the effect of annealing and Hip'ing

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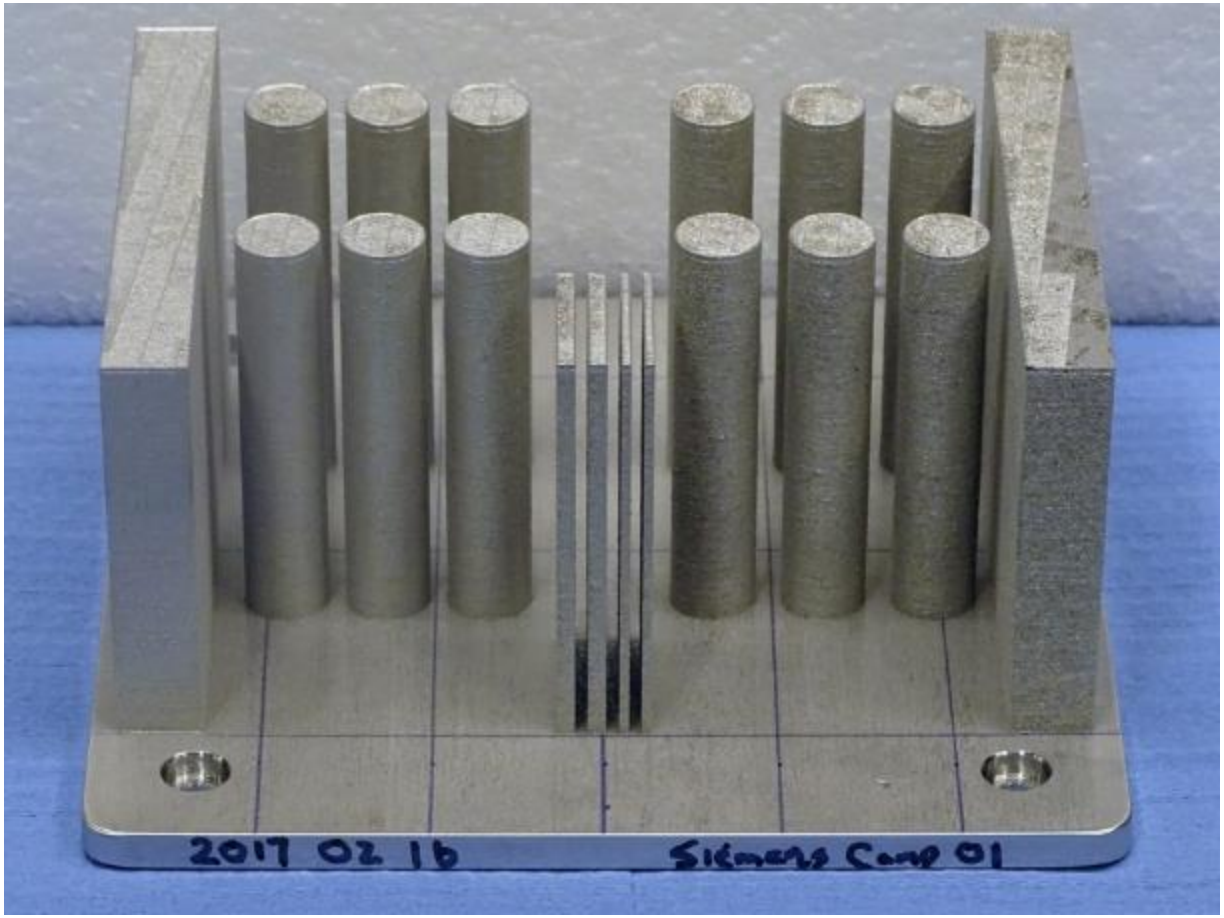
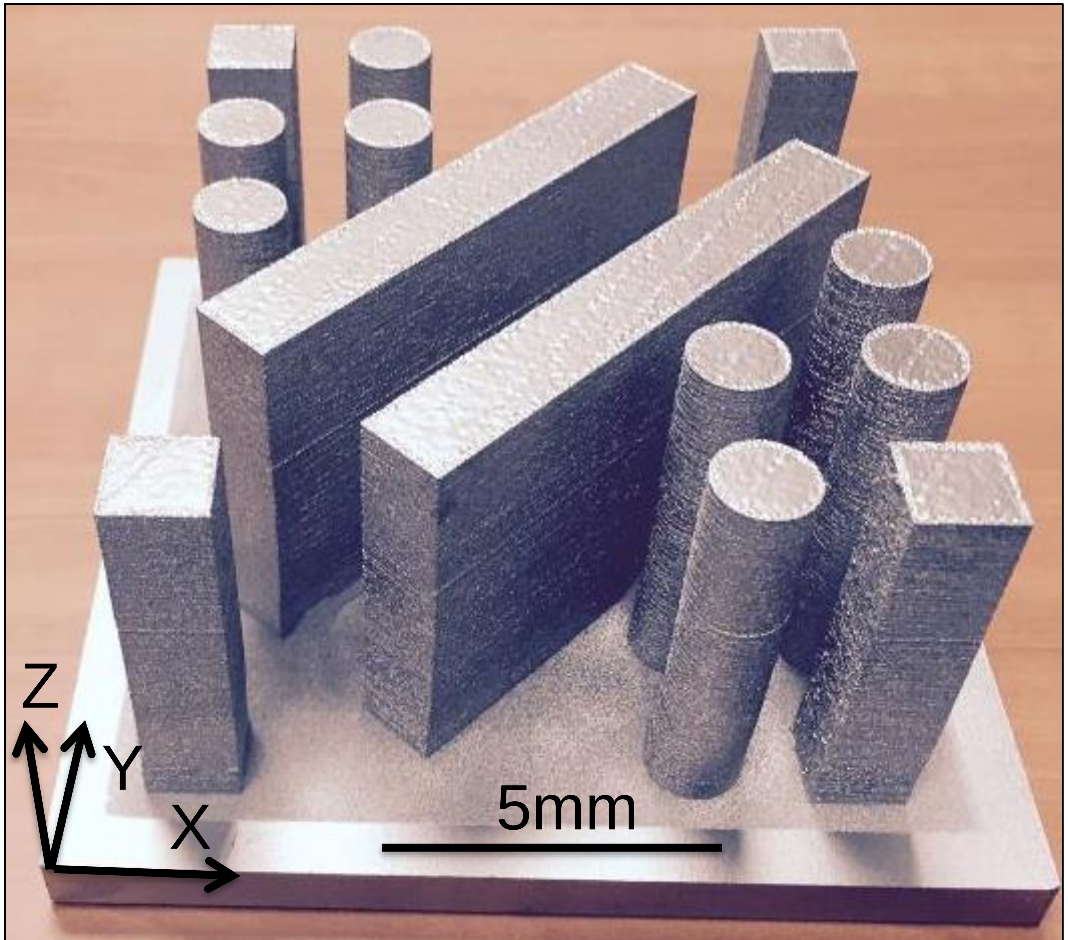
New SLM (SLM-Opt) Build For Extensive HX Characterization



- 35h, ~2000 layers
- 65mm tall
- Rectangular blocks to study properties anisotropy
- Thin wall effect
- Machine available for 2 weeks for process optimization

Fabrication of 20-30 EBM Specimens For Tensile, Creep and Fatigue Testing



- 27h, 1240 layers, ~65mm
- Small builds first to optimize parameters based on 718 & HX previous work
- Similar build parameters + pre-heat temperature
- Study the effect of post annealing and Hip'ing

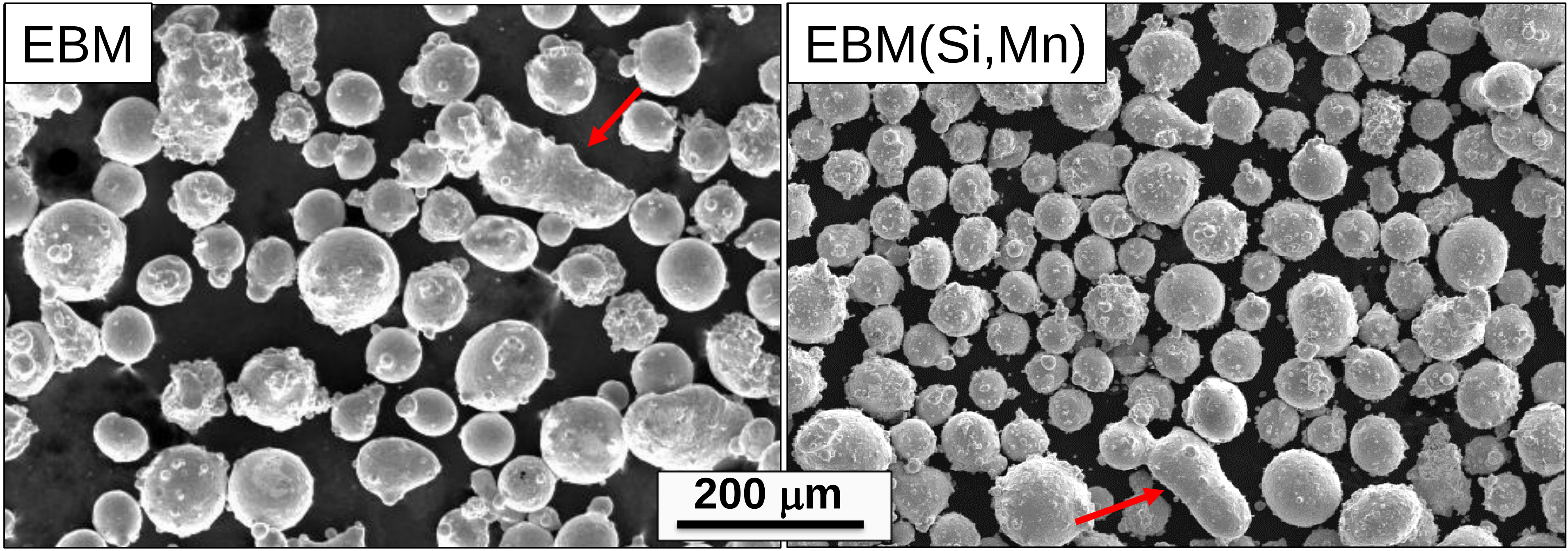
Similar Composition For EBM, SLM and Wrought HX Except for Si &Mn

Solution Strengthened Hastelloy X (Ni-22Cr-19Fe-9Mo)

	Ni	Cr	Fe	Mo	Co	Mn	Si	W	C
EBM Powder	Bal.	21.76	18.43	8.91	1.51	0.07	0.08	0.6	0.08
EBM Alloy	Bal.	21.38	18.55	9.05	1.55	0.01	0.05	0.64	0.078
EBM(Si,Mn) Pow.	Bal.	21.7	18.7	9	1.56	0.93	0.86	0.66	0.06
EBM(Si,Mn) Alloy	Bal.	21.43	18.87	9	1.56	0.67	0.71	0.65	0.048
SLM Powder	Bal.	21.47	18.83	8.96	1.51	0.01	0.16	0.63	0.07
SLM-Opt Powder	Bal.	21.72	18.51	8.87	1.51	0.01	0.06	0.6	0.08
Wrought	Bal.	22.06	17.86	9.53	1.8	0.65	0.31	0.6	0.067

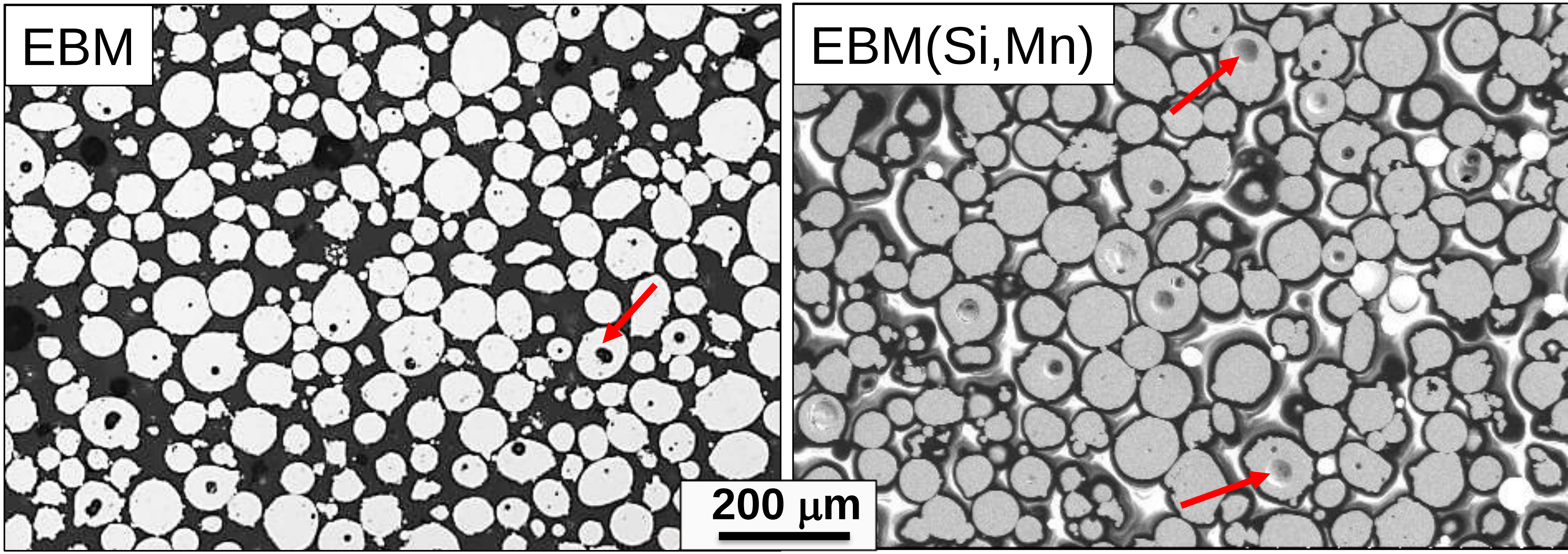
- Alloy composition consistent with EBM powder composition
- High concentration of Mn and Si in EBM(Si,Mn) and wrought HX. Specification: Mn and Si <1%

EBM/EBM(Si,Mn) Powder Morphology Typical of Gas Atomized Powder

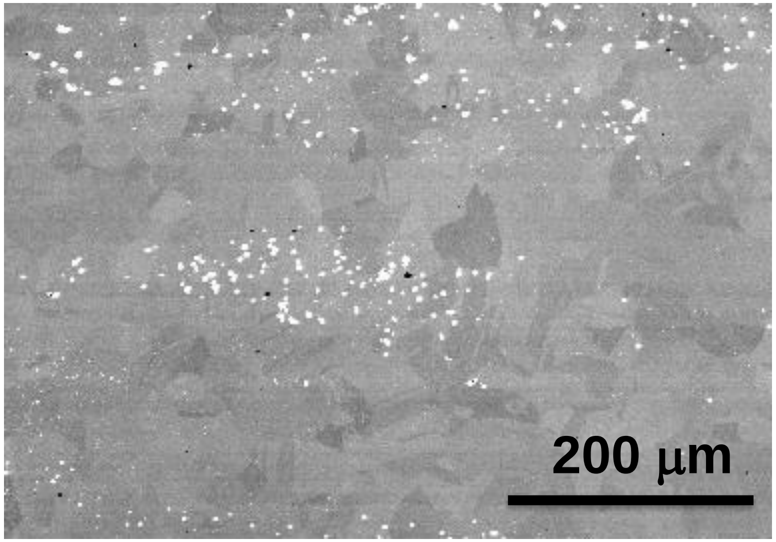


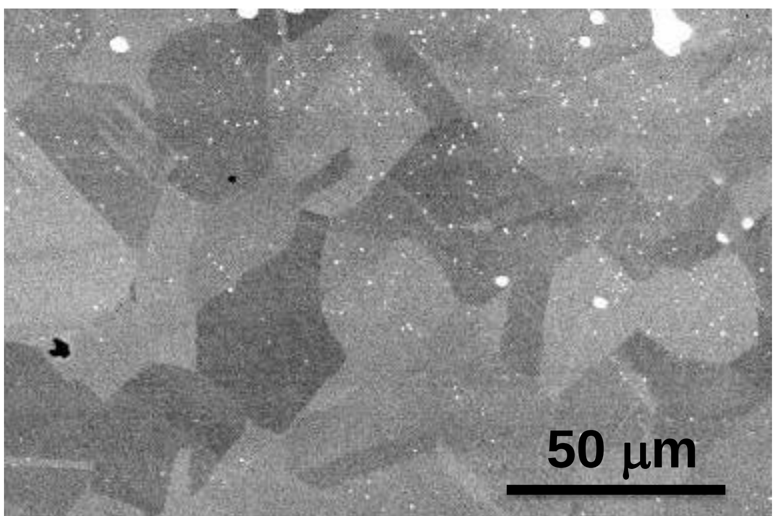
- Most powder particles contain large numbers of satellite particles
- Irregularly shaped particles

Powder Defect: Larger Voids for the EBM powder

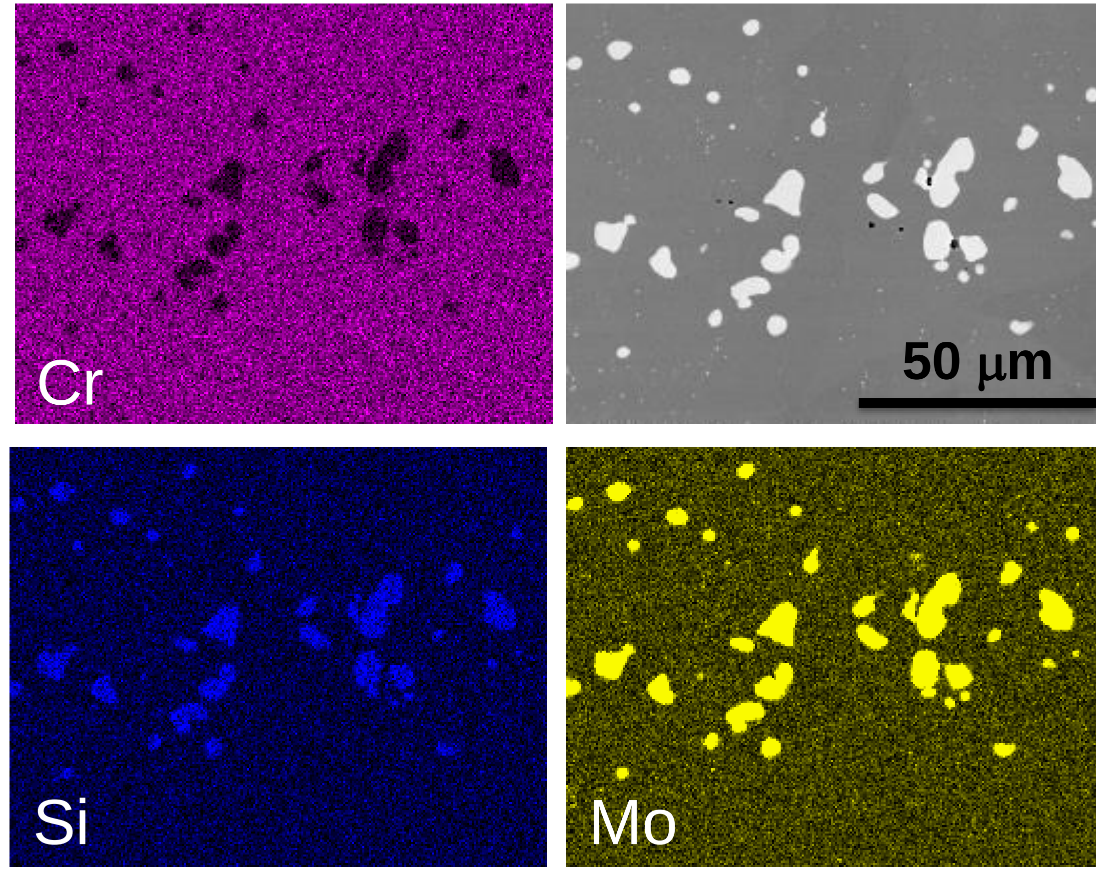


Wrought HX: Random Distribution of (Mo,Si) Precipitates

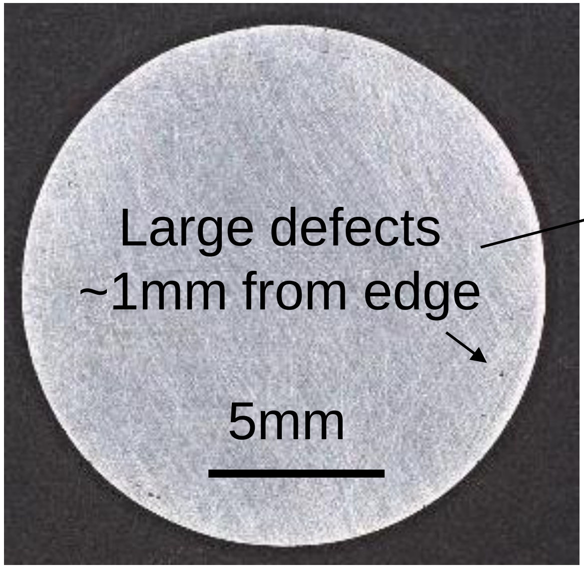


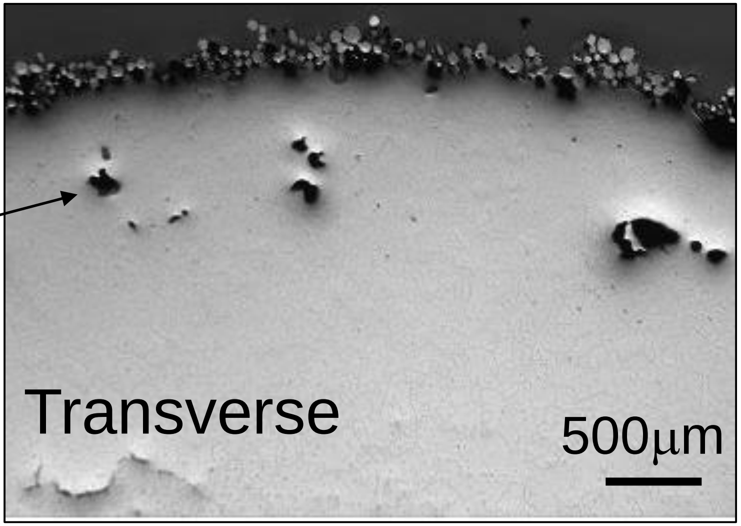


~50μm equiaxed grains

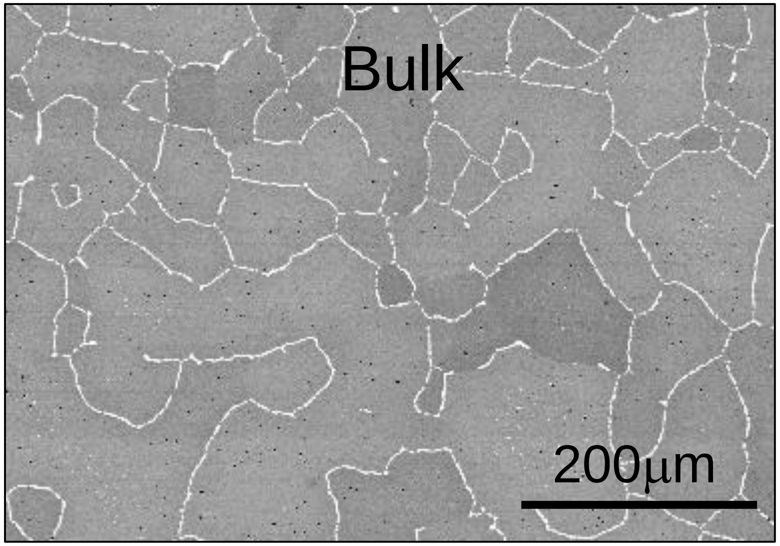


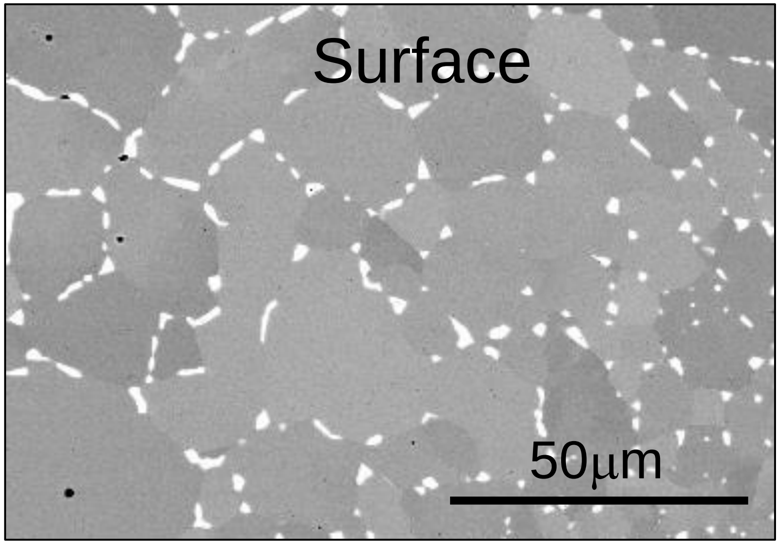
EBM: Larger Grains at the Center vs Surface





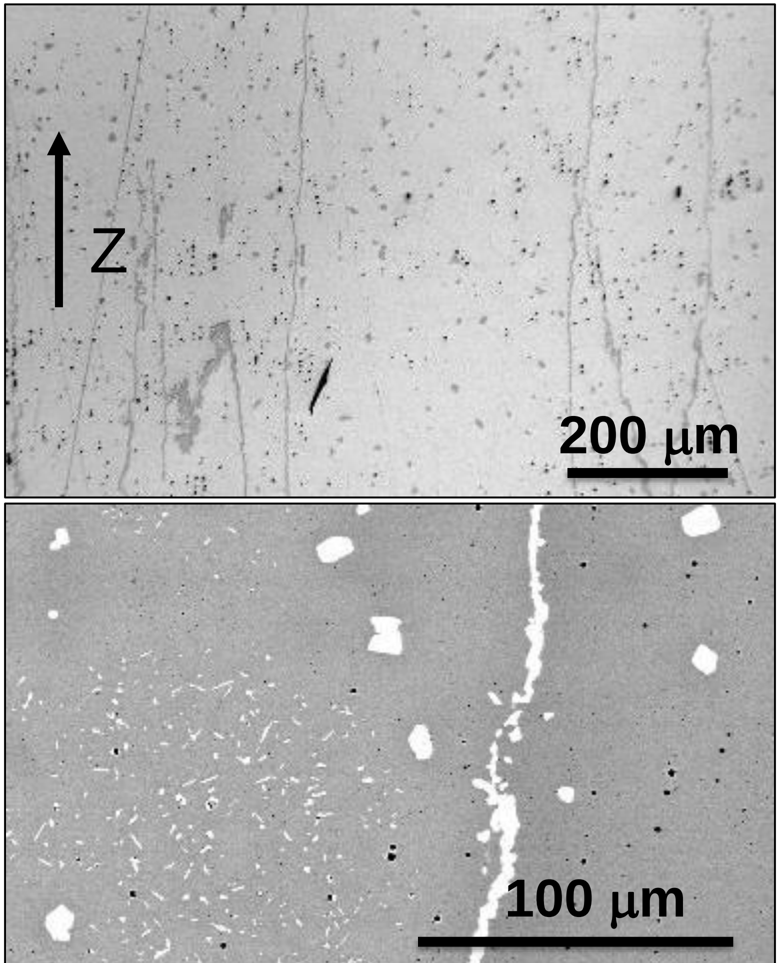
Fabrication of contour first



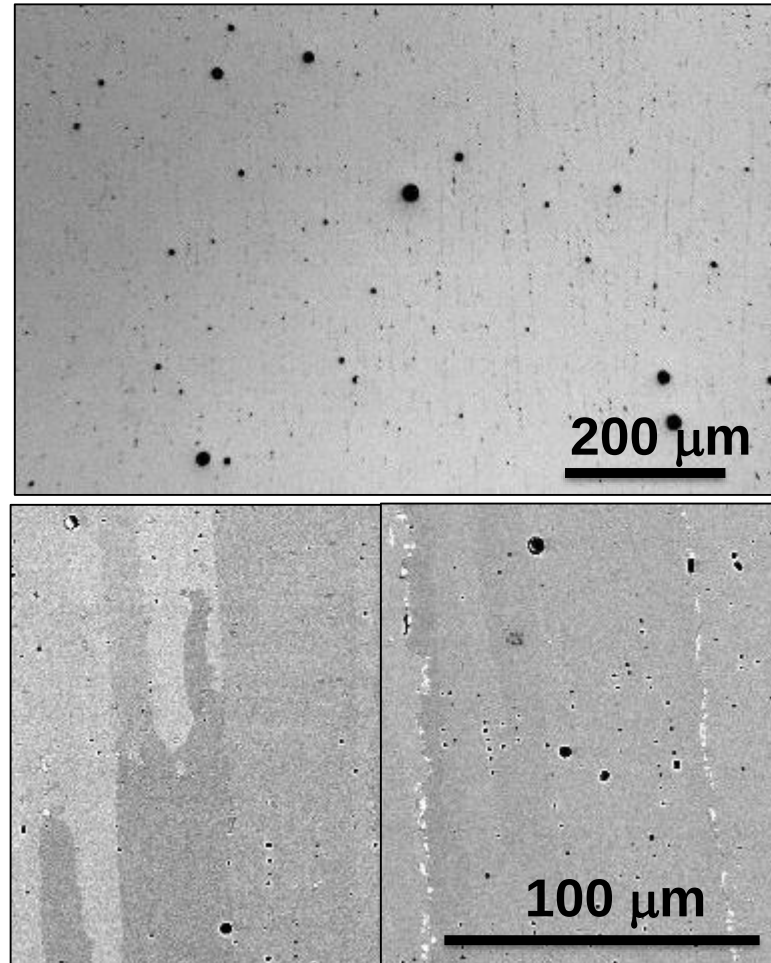


Elongated Grain Along Z Direction More Precipitates for EB(Si,Mn)

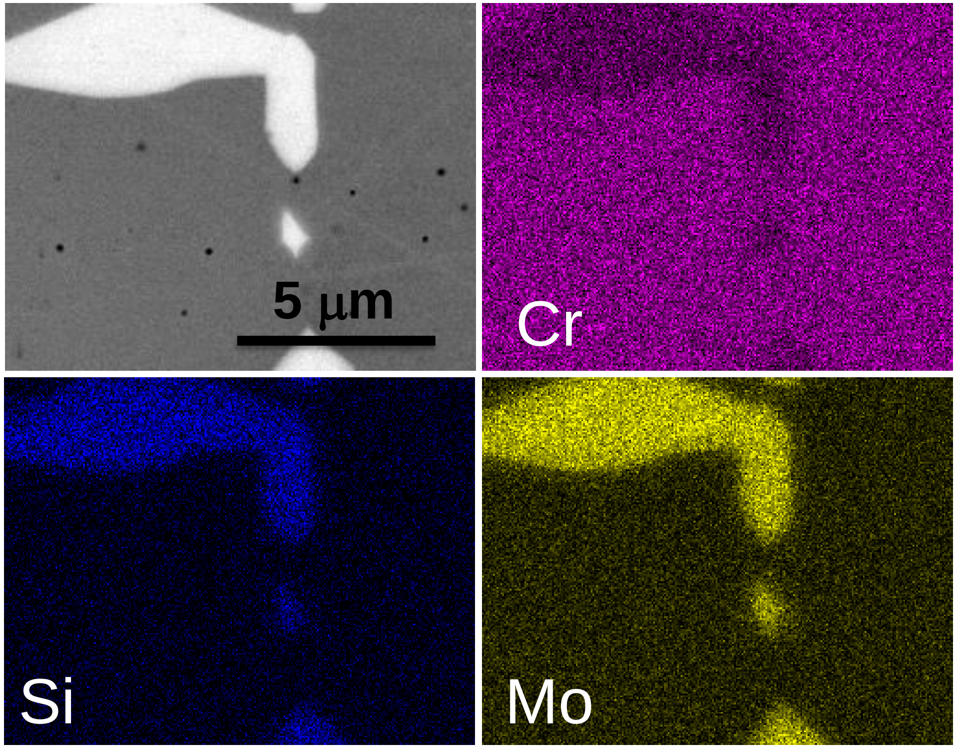
EBM(Si,Mn) = Larger grains

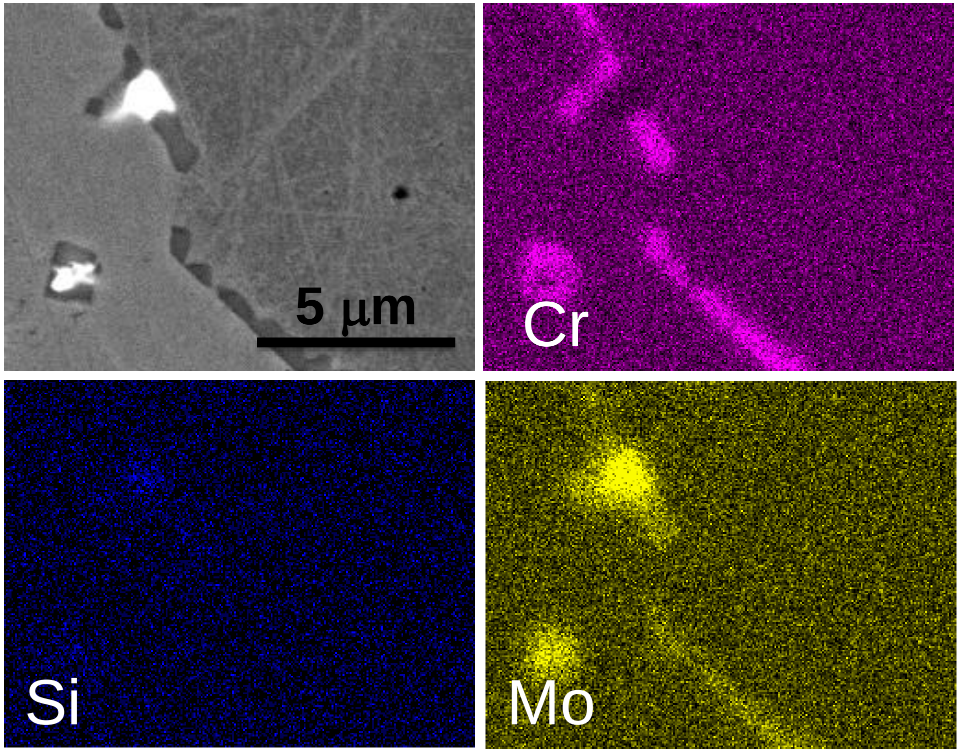


EBM = Larger voids

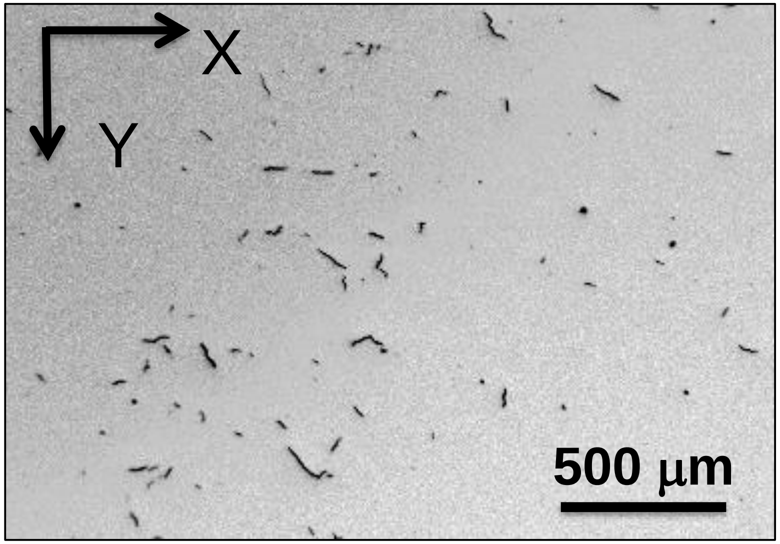


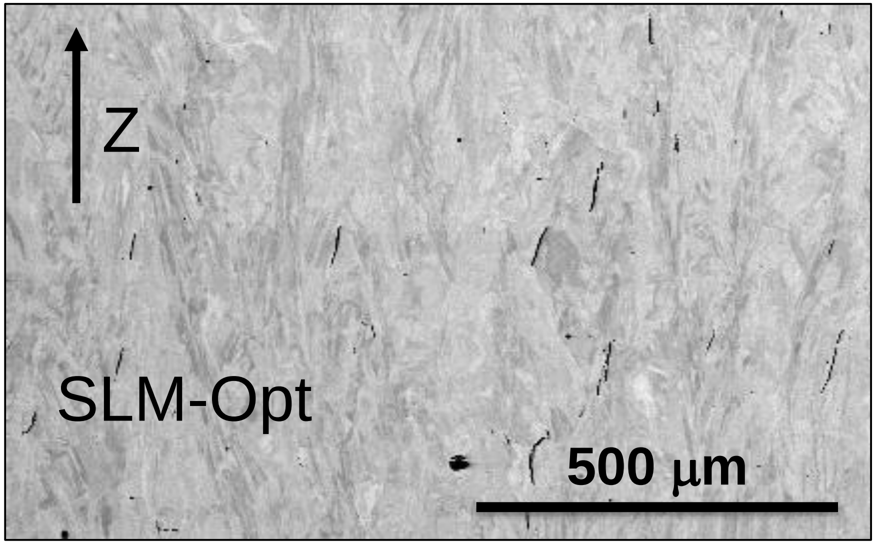
EBM(Si,Mn): (Mo,Si)-rich Carbides
EBM: (Mo,Cr)-rich Carbides at GB

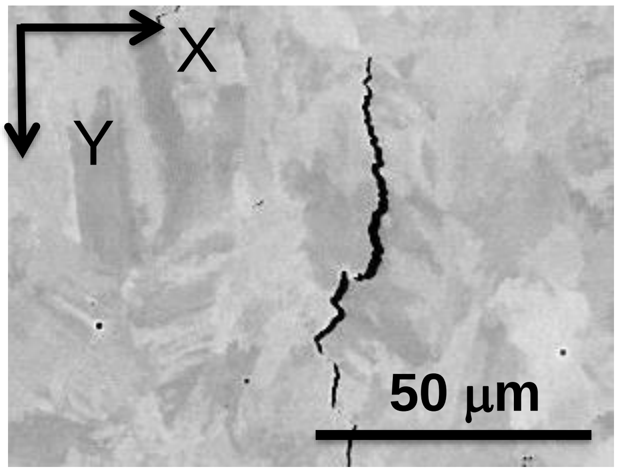


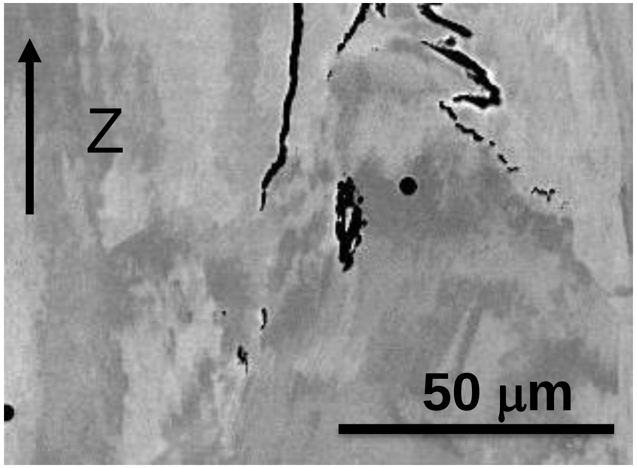


SLM-Opt: Small Grains, No Precipitate Hot Tearing Cracks



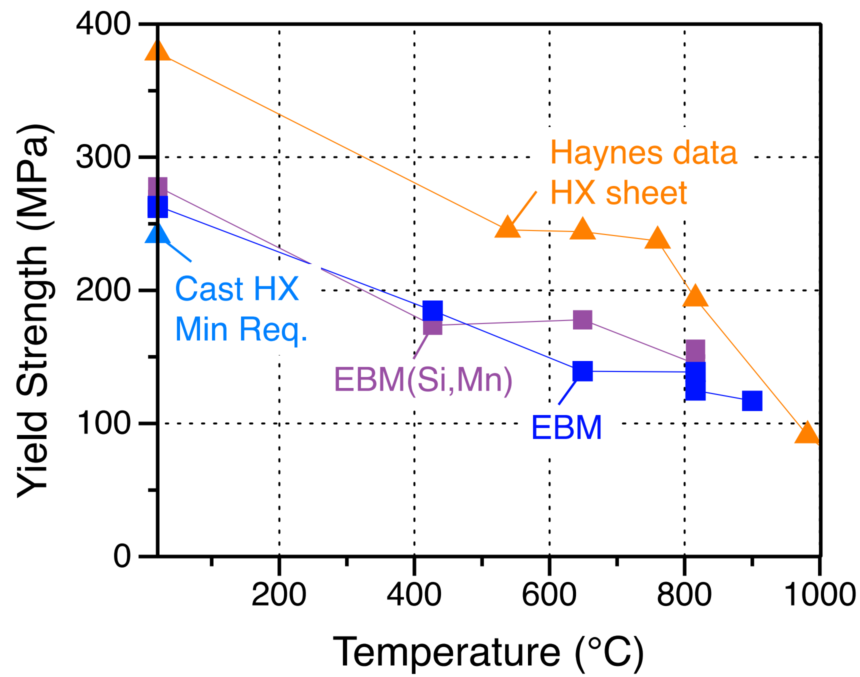




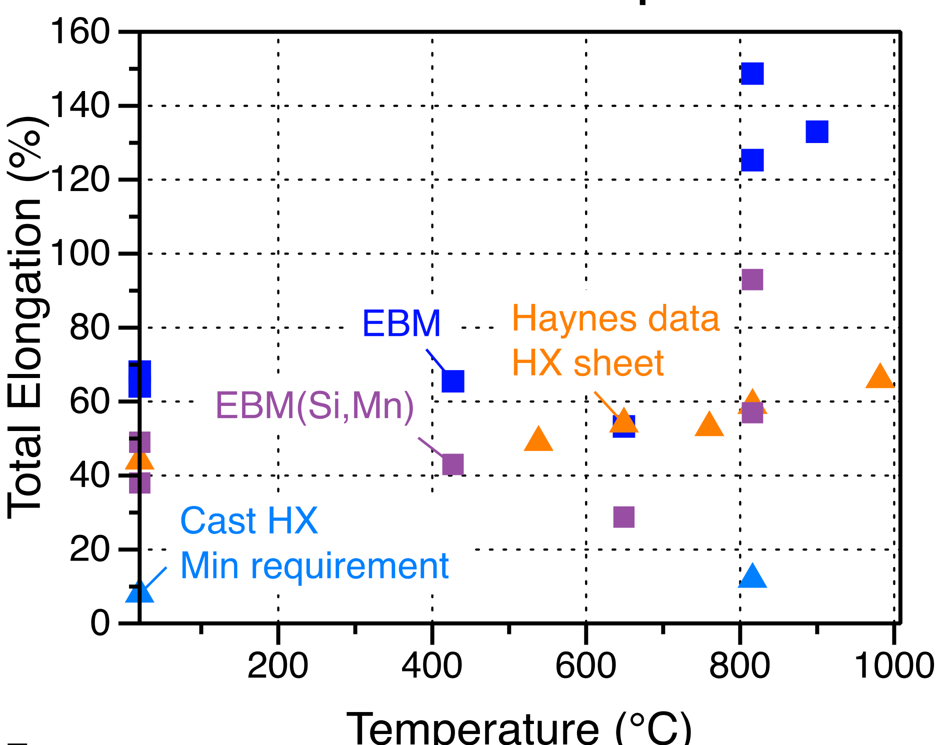


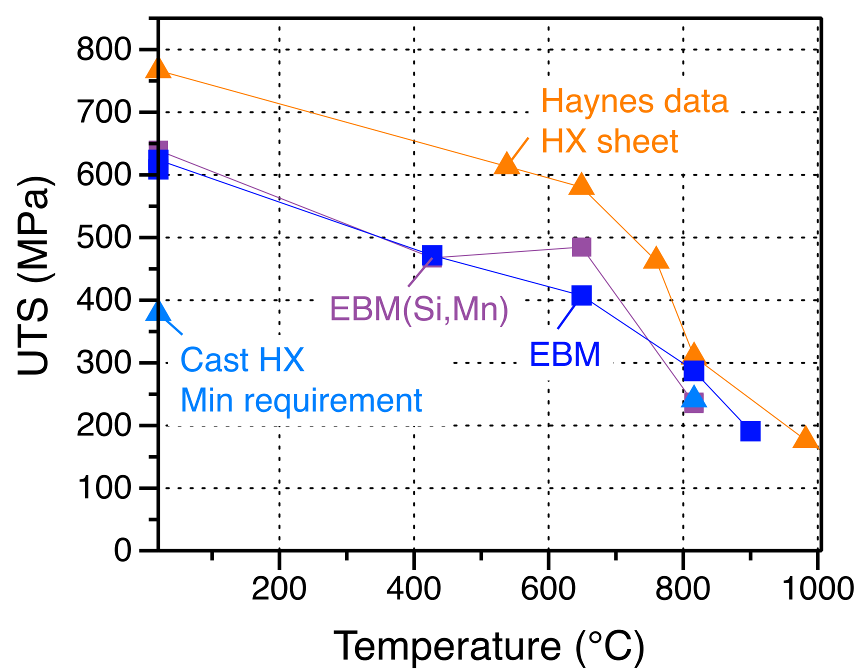
Lower defect density for SLM-Opt
Consistent with literature
Defect could likely be eliminated

Both EBM & EBM(Si,Mn) HX Alloys Exhibit Good Ductility But Lower Strength < 800°C

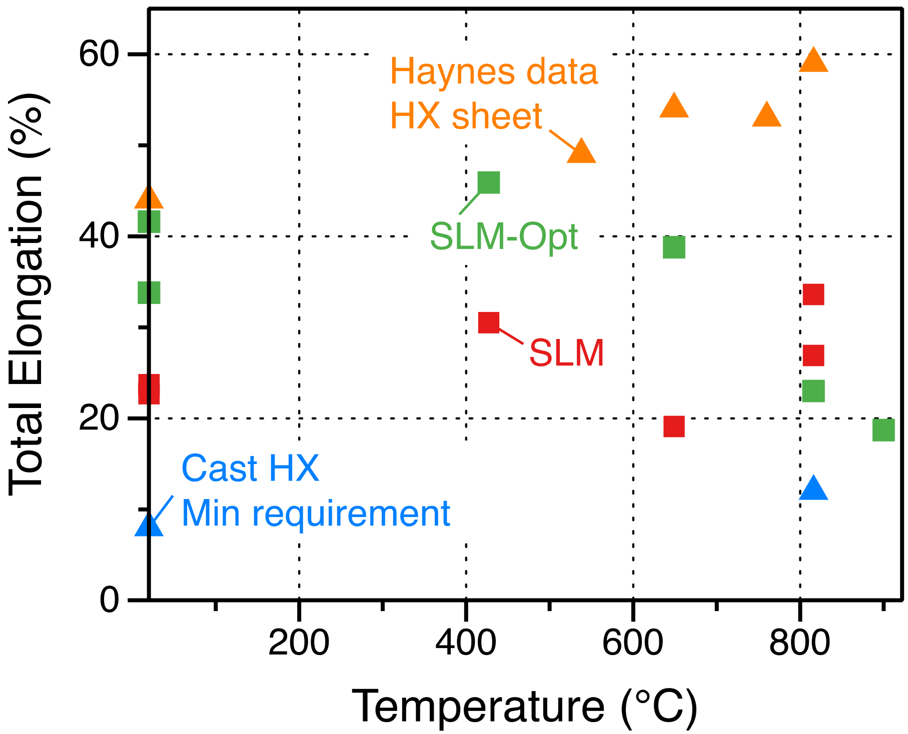


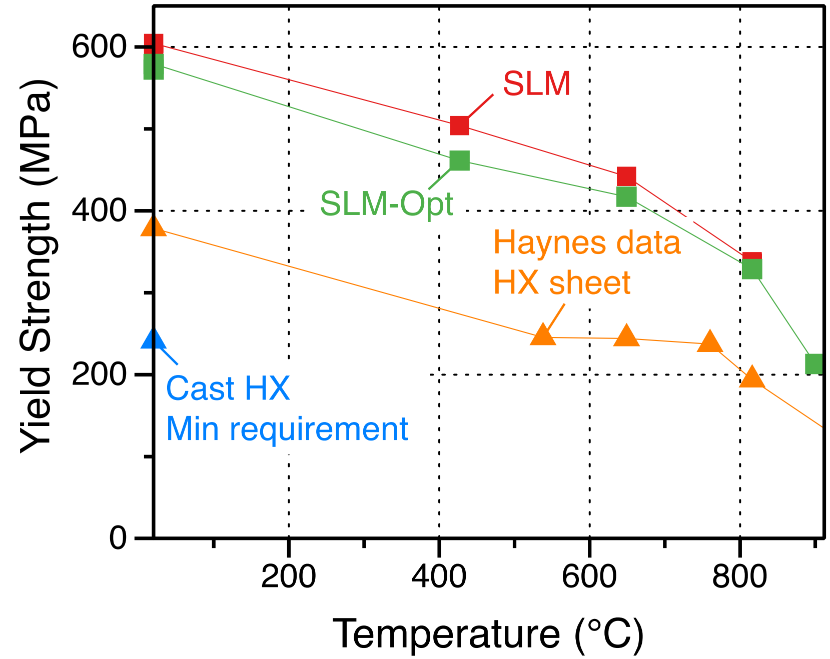
Meet cast HX AMS requirement

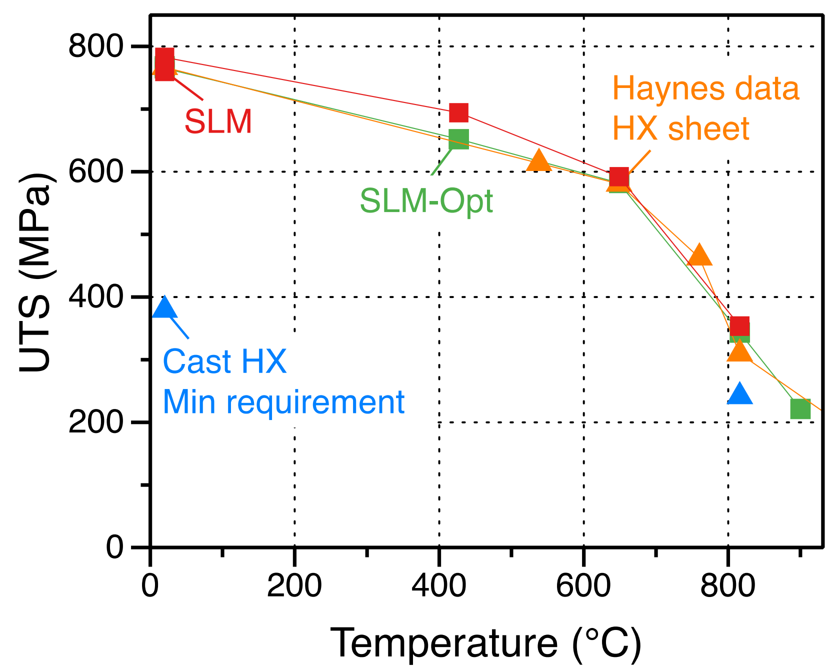




SLM HX Exhibits High Strength but Moderate Ductility

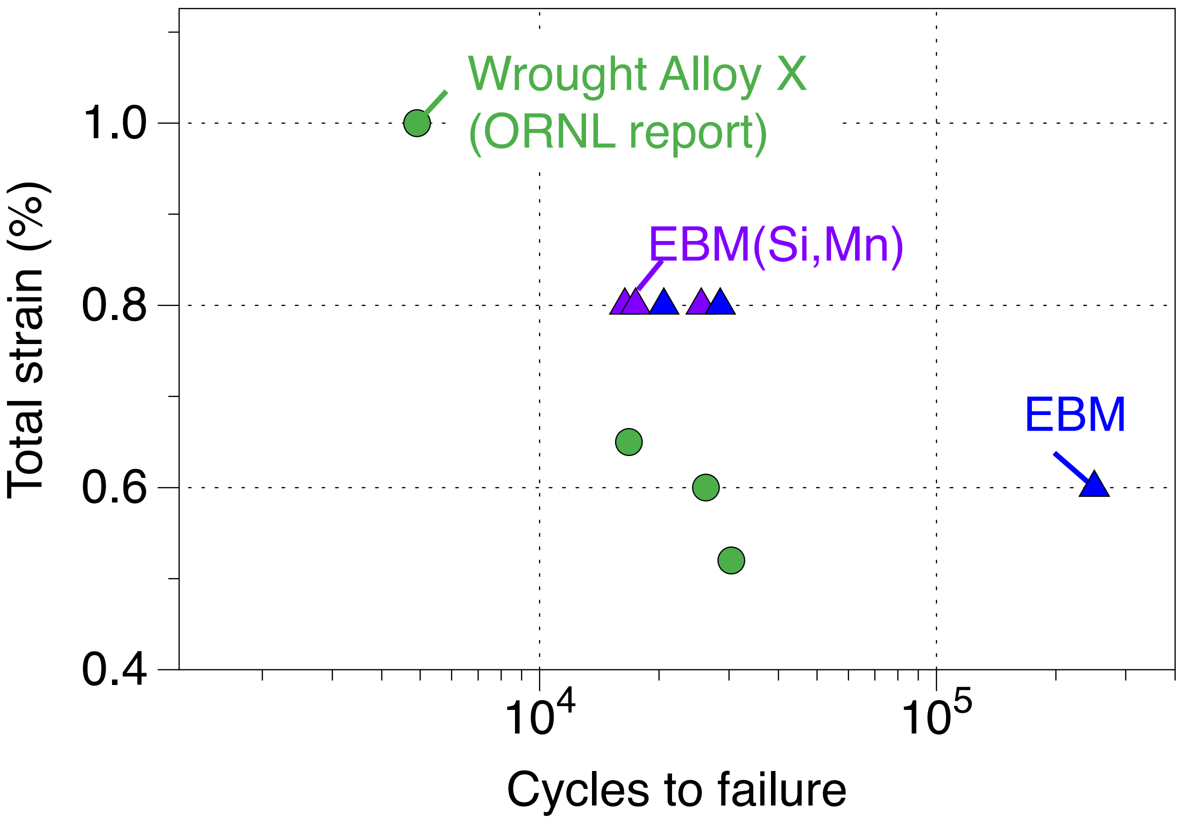






- High YS due to high residual stress
- Higher ductility for SLM-Opt

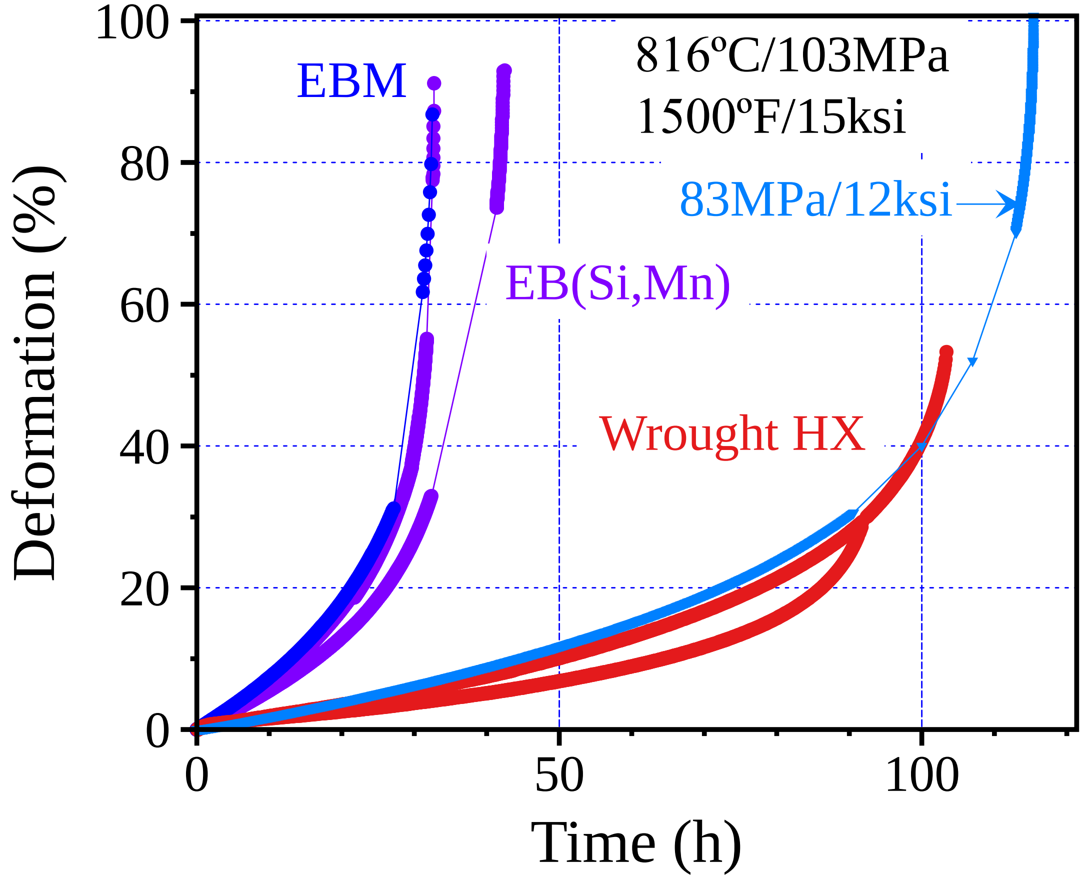
EBM & EBM(Si,Mn): Good Low-Cycle Fatigue Properties at 800ºF/425ºC



- Fully-reversed LCF
- Consistent with excellent HX EBM alloys ductility
- Similar results at other temperatures

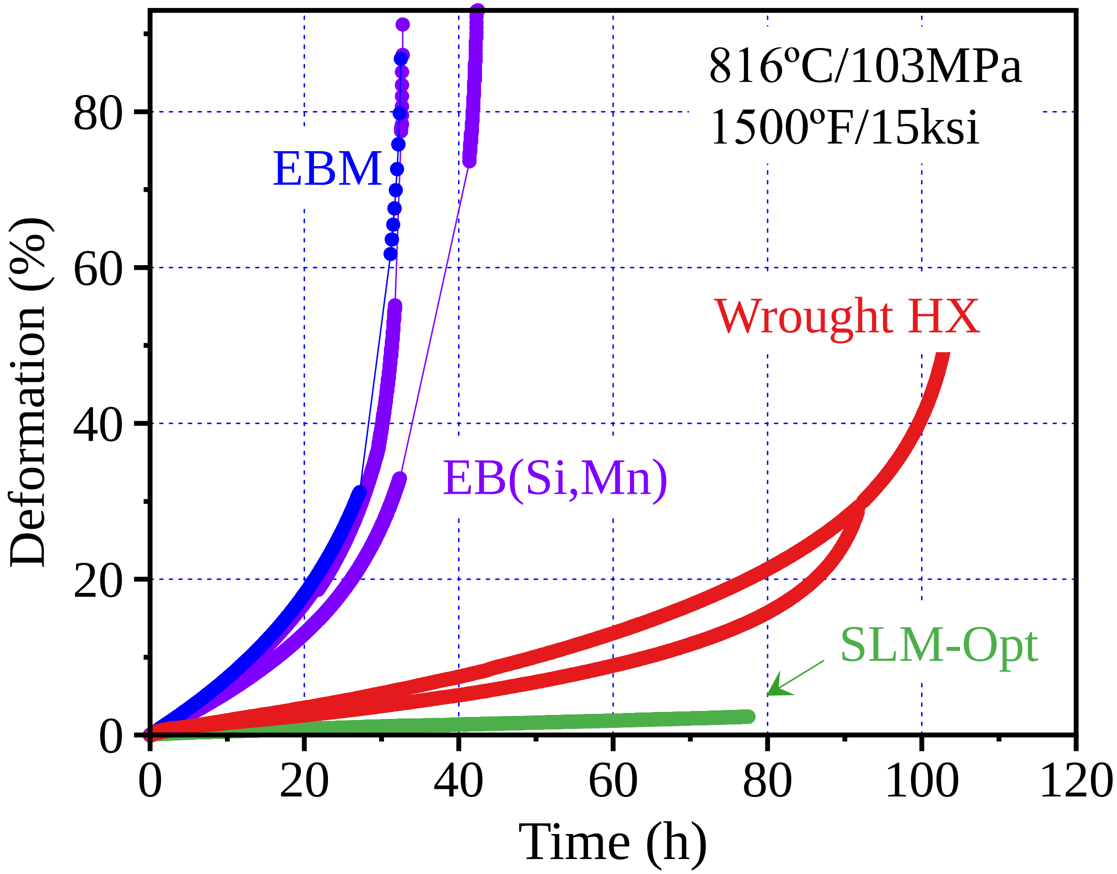
SLM testing coming soon

EBM&EBM(Si,Mn): Lower Creep Strength & Higher Ductility at 816ºC Similar Results for the Two Alloys

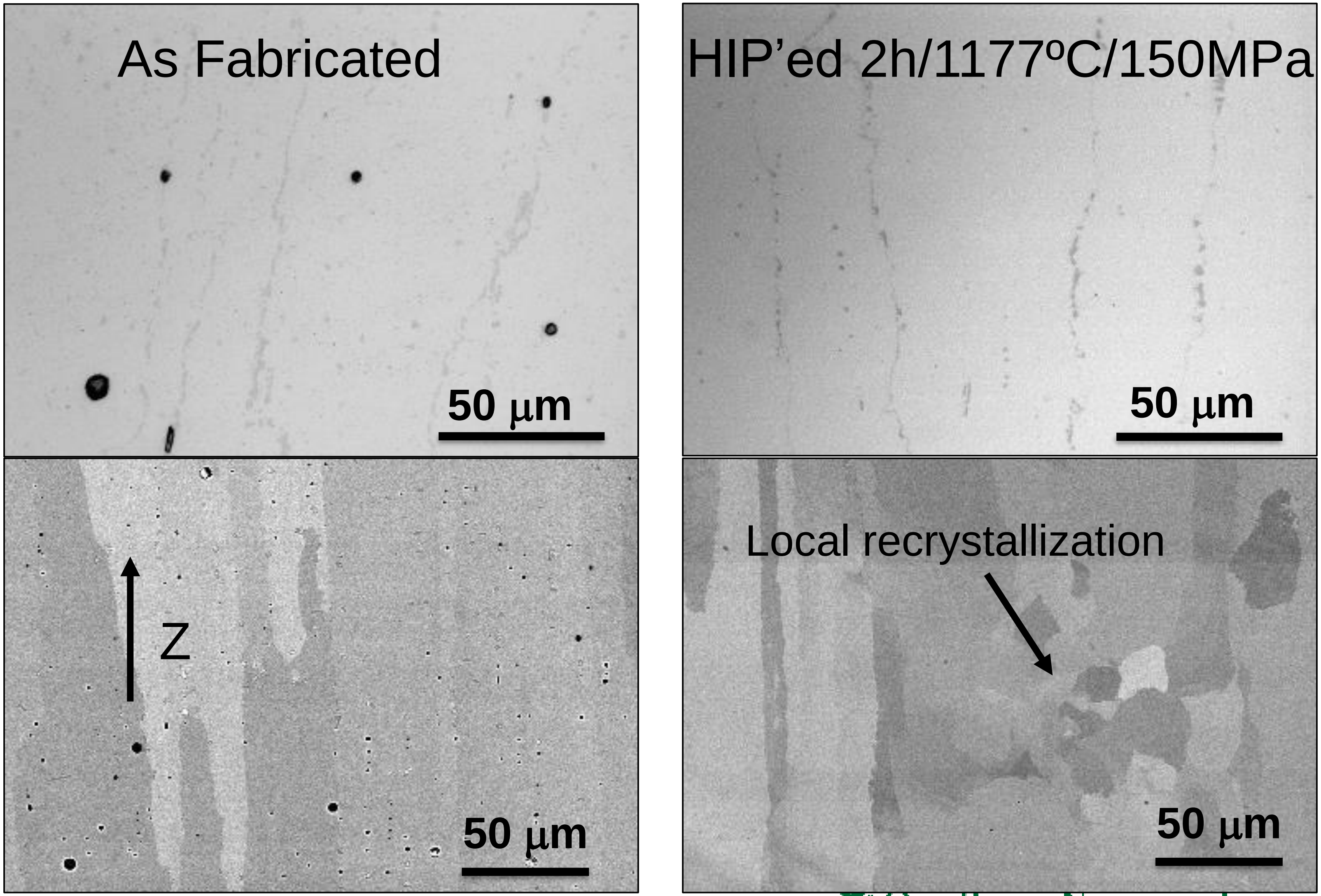


Need to compare with cast HX data

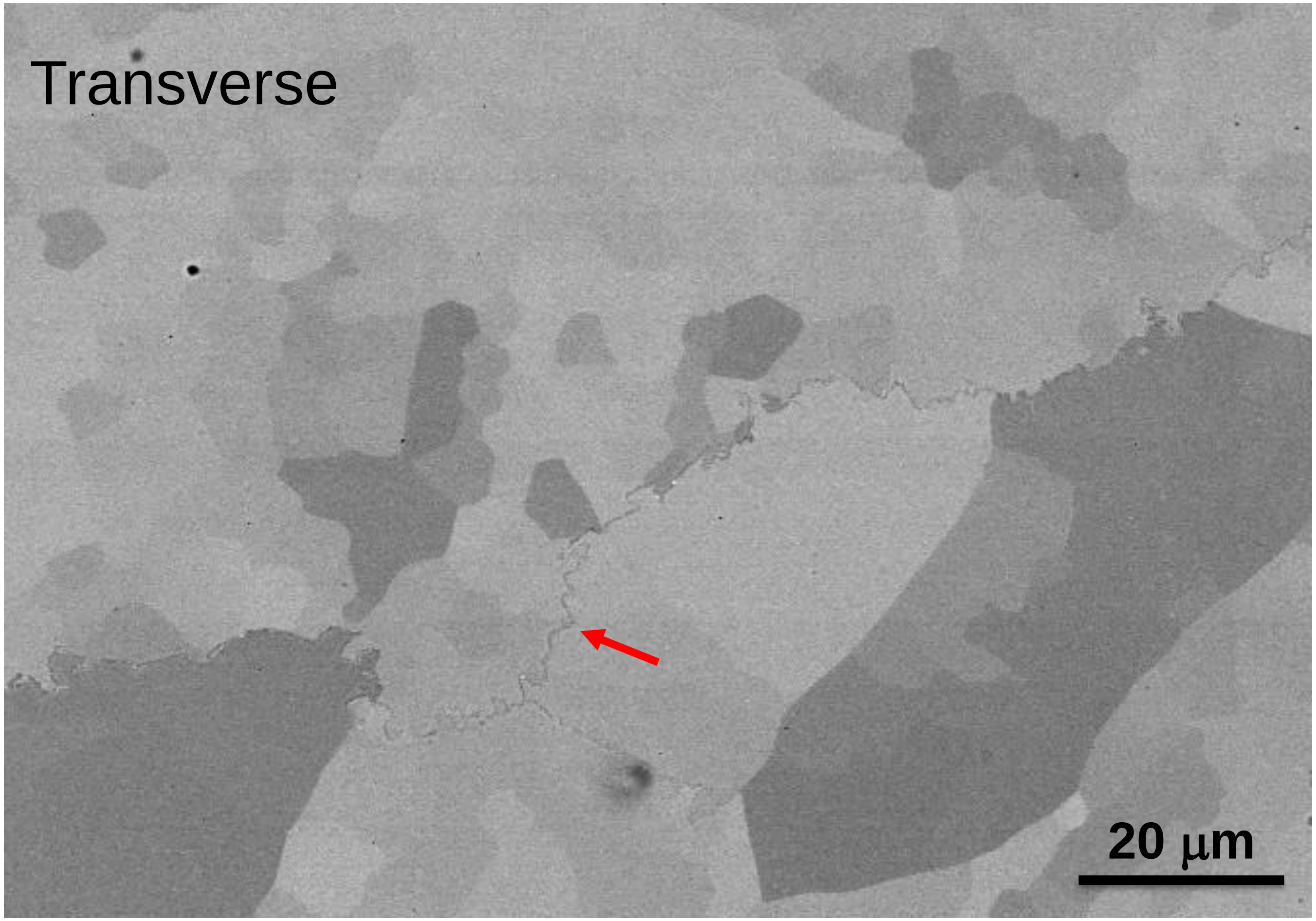
SLM: Similar Creep Strength but Limited Ductility at 816ºC



EBM: Fully Dense Material after HIP'ing at 1177ºC/2h/150MPa, "Fast Cooling"



HIP'ed EBM: Increasing Number of Small Grains + Precipitates Along Few Grain Boundaries



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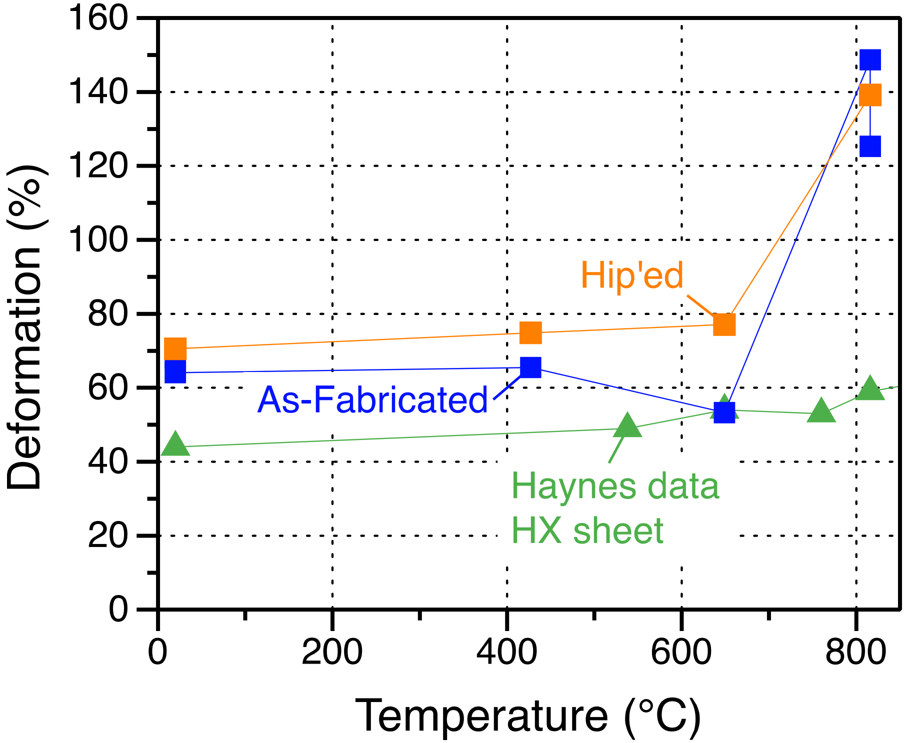
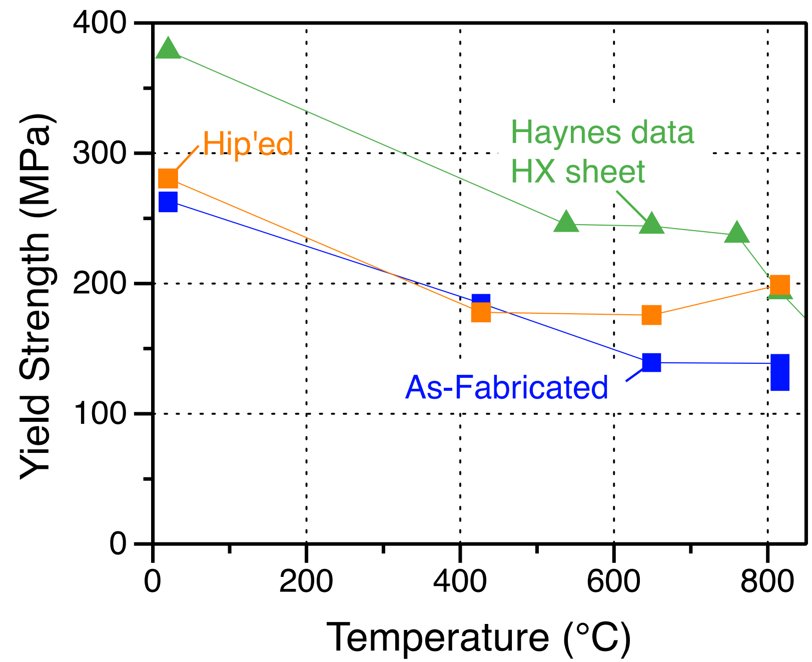
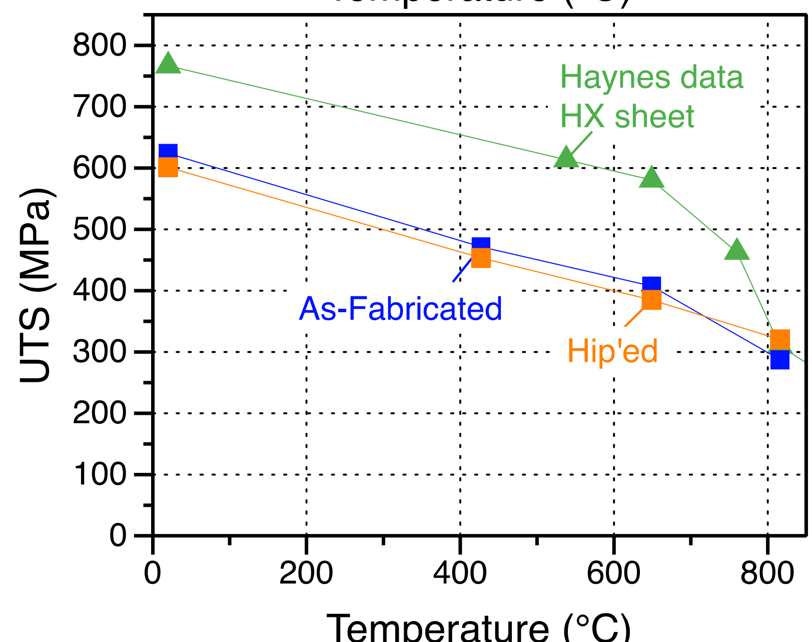
EBM: Similar Strength & Ductility After HIP'ing 2h/1177°C/150MPa







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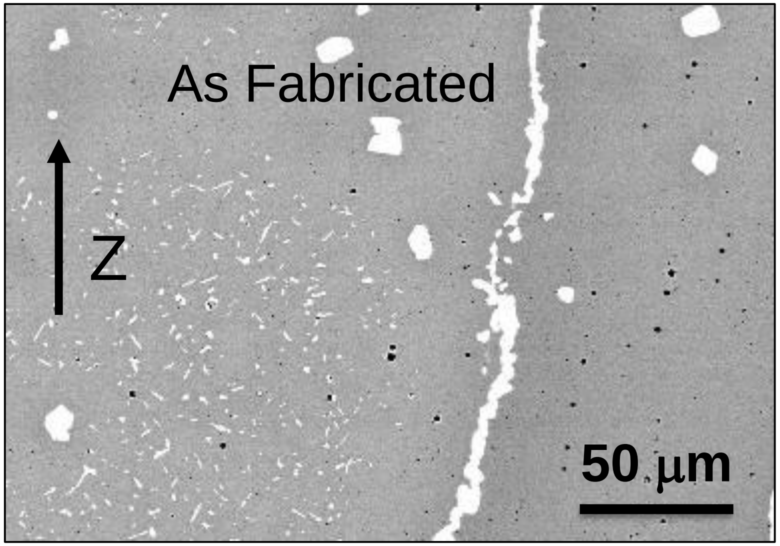
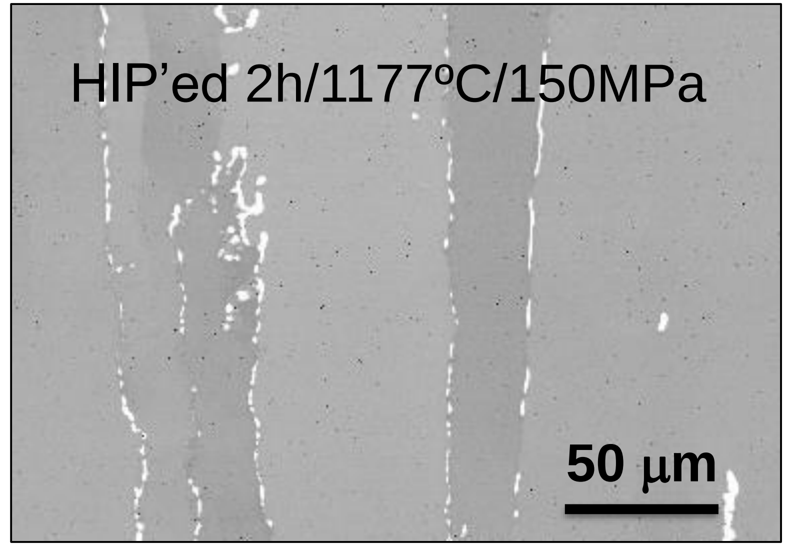
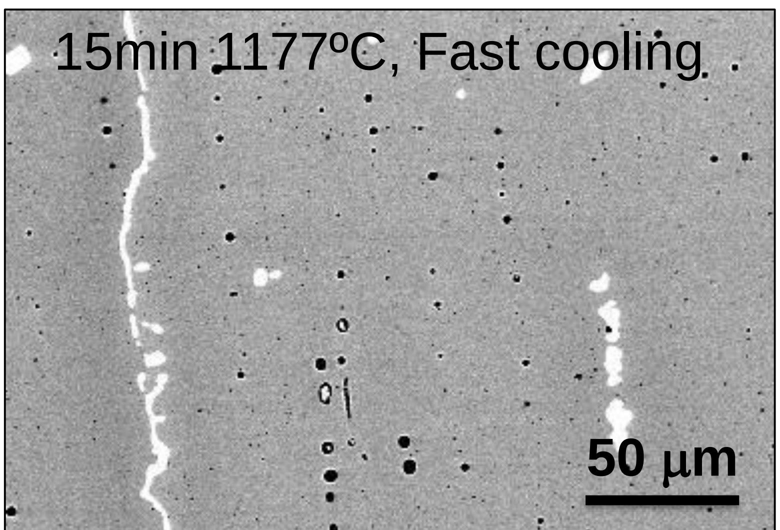
EBM(Si,Mn): Fully Dense Material after HIP'ing at 1177ºC/2h/150MPa

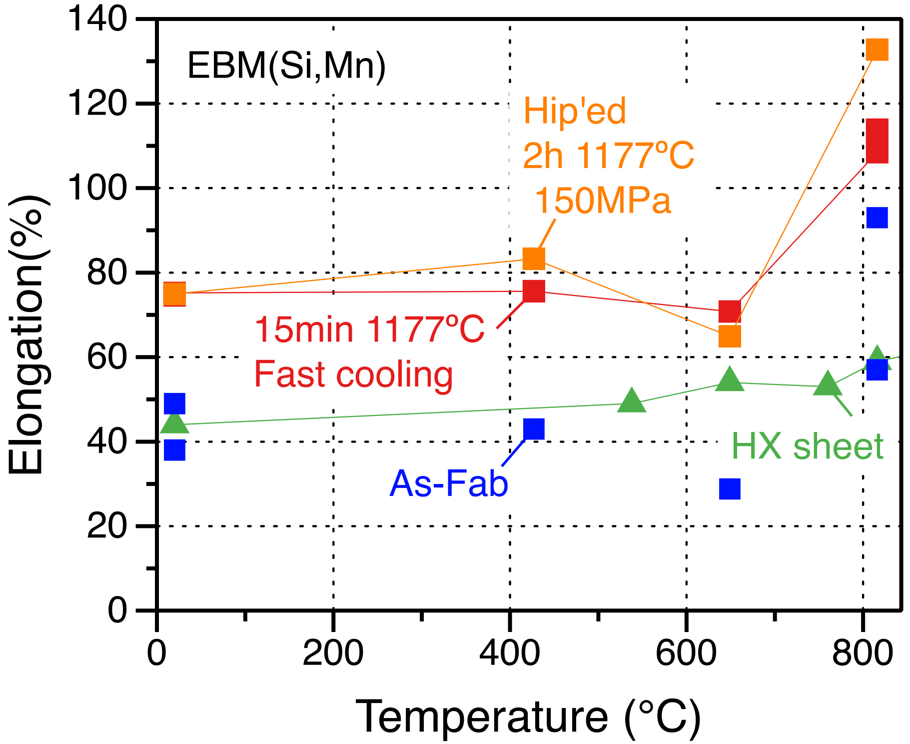


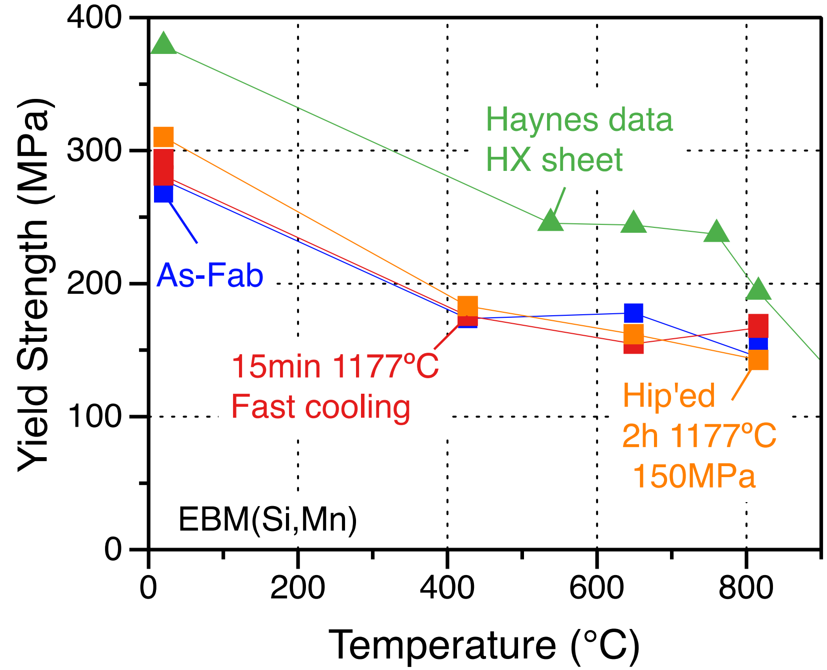


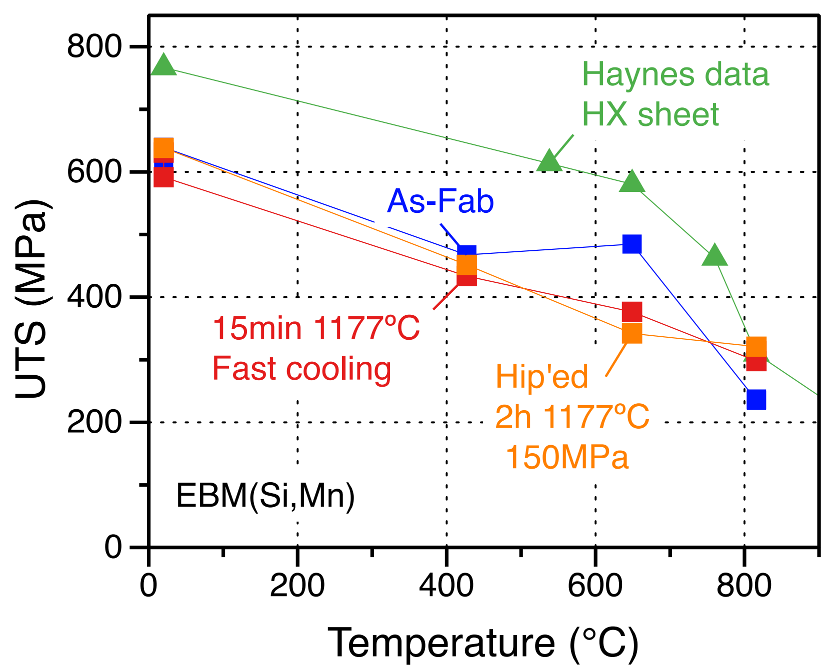


Main microstructure differences between as fab, annealed and HIP'ed HX are voids and precipitates in the grains

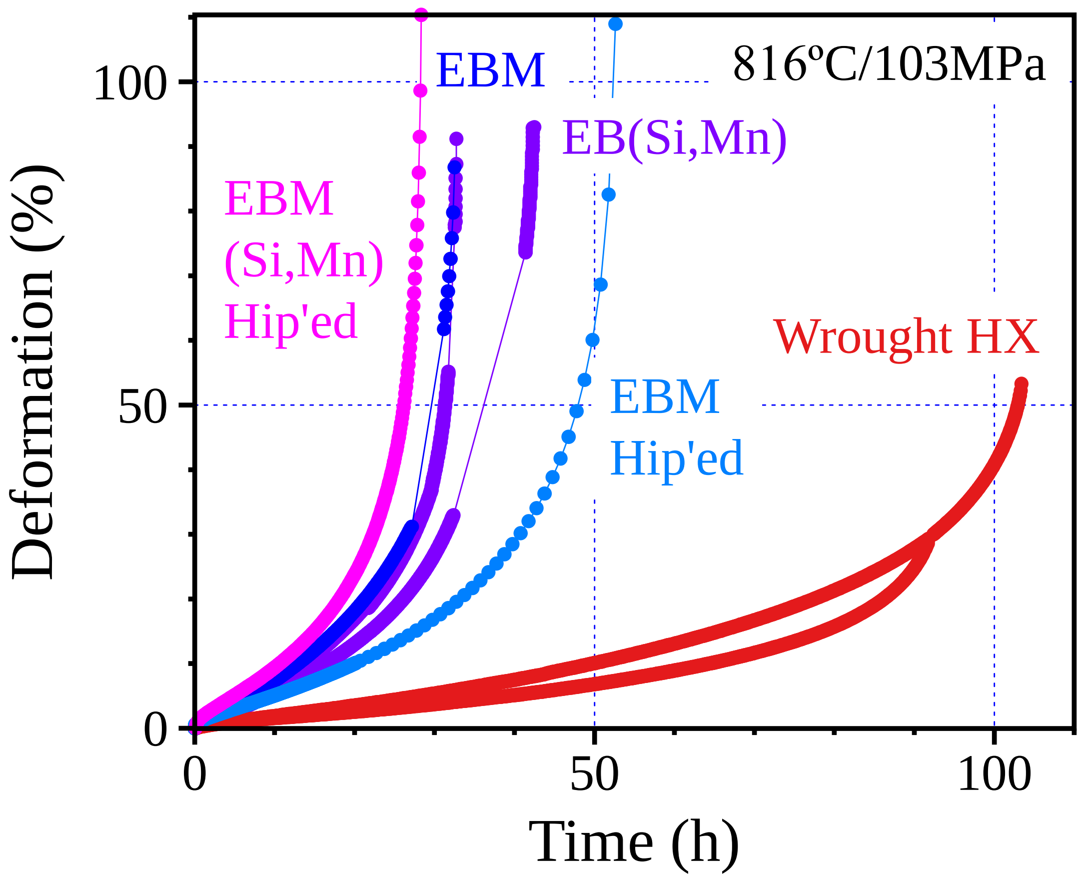
EBM(Si,Mn): Similar Strength & Better Ductility After HIP'ing or annealing at 1177ºC





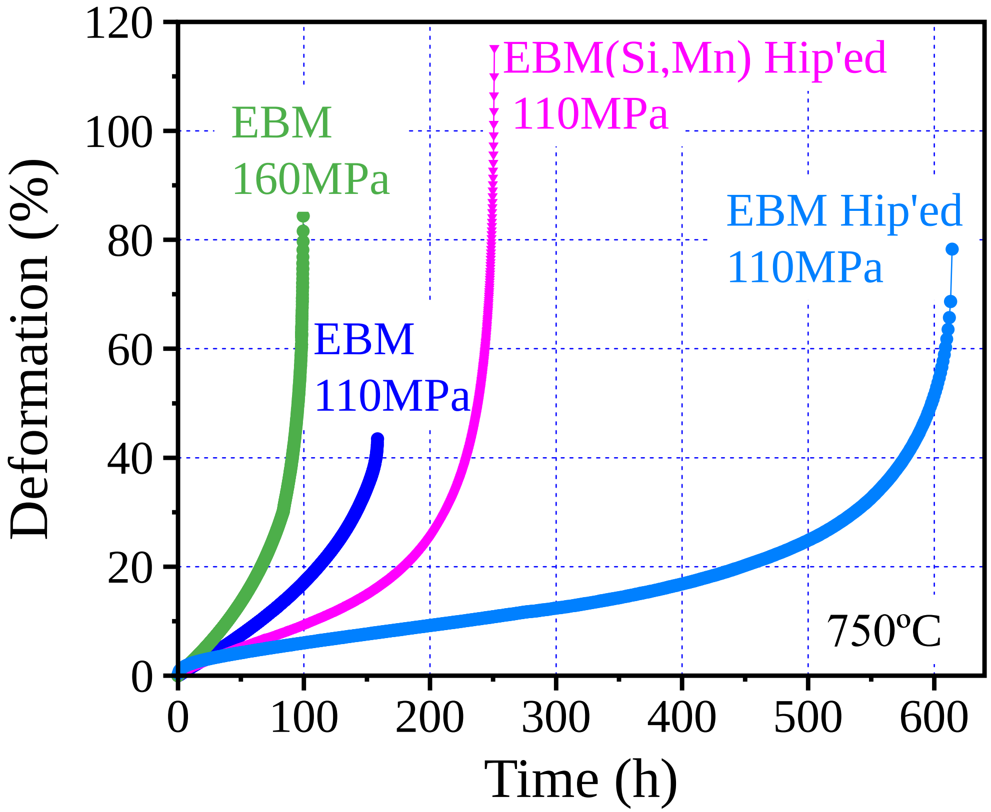


EBM(Si,Mn): No Clear Effect of Hip'ing on the Creep Properties at 816ºC Improvement for EBM?



EBM,750ºC: Better Creep Results For Short Term Tests?

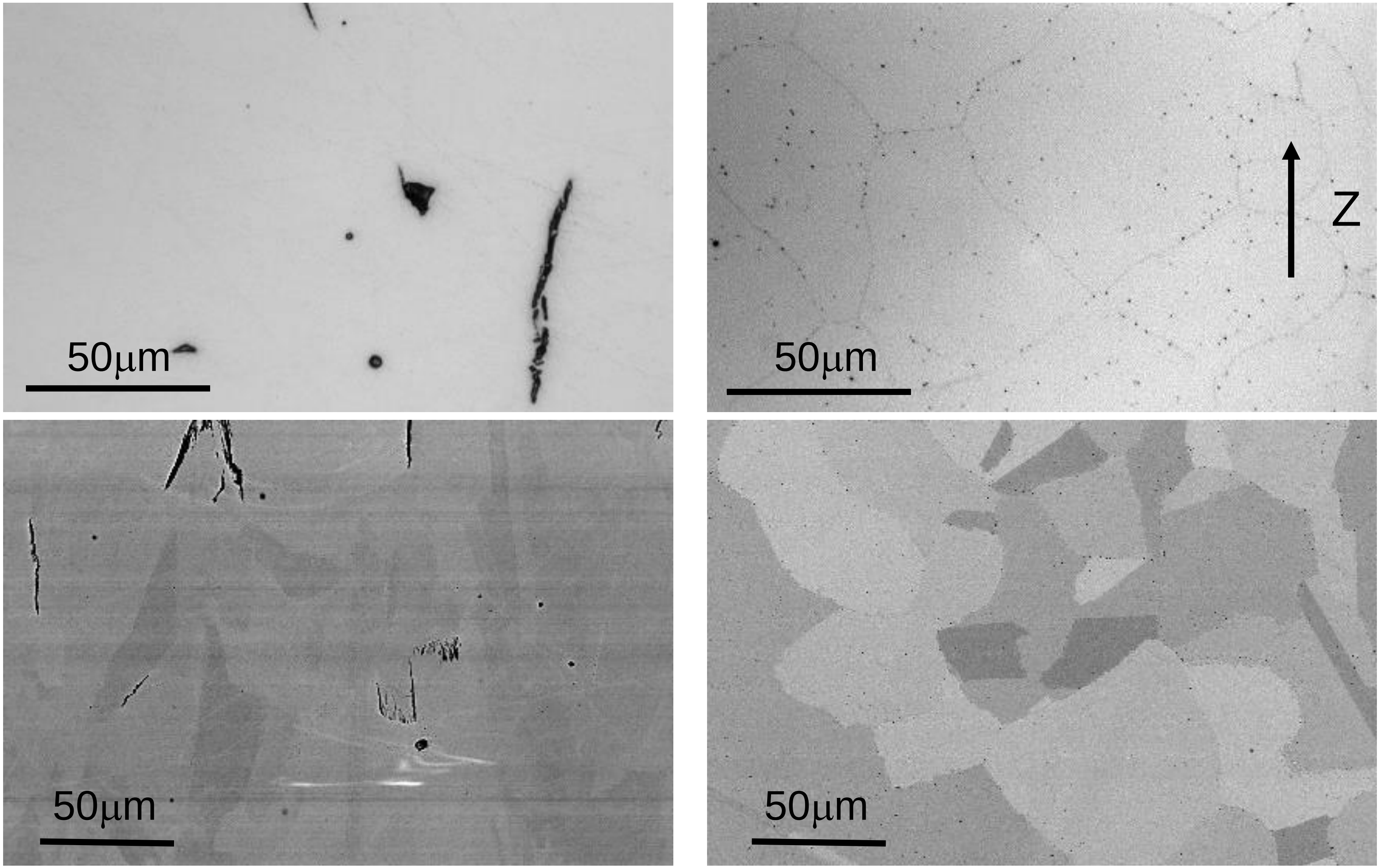
Significant improvement after HIP'ing for EBM alloy



Wrought expected lifetime:

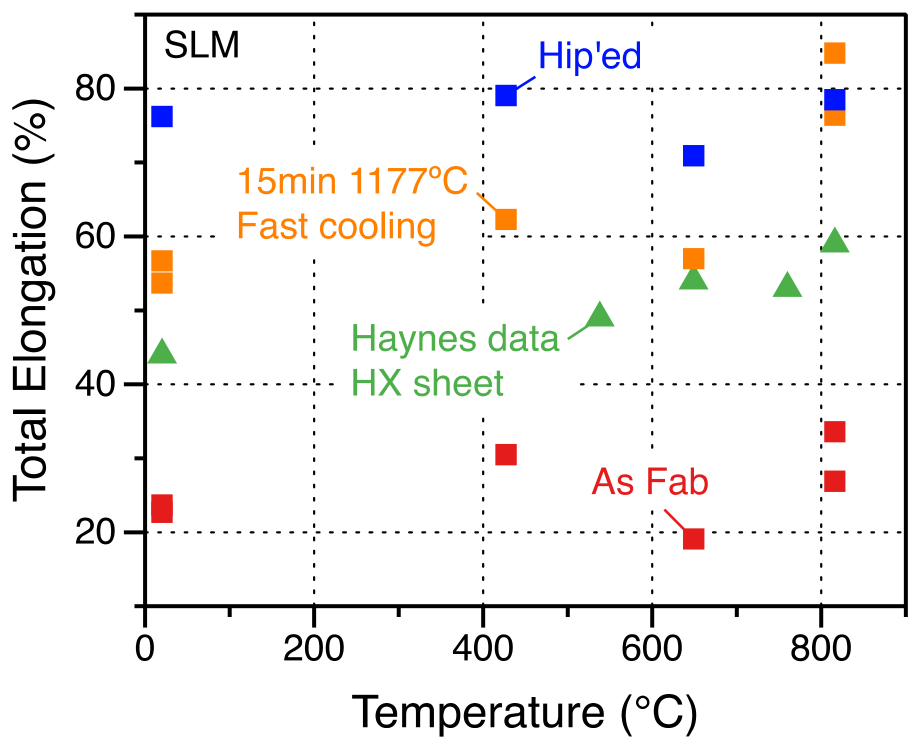
- 160MPa: 100h
- 110MPa: 1000h

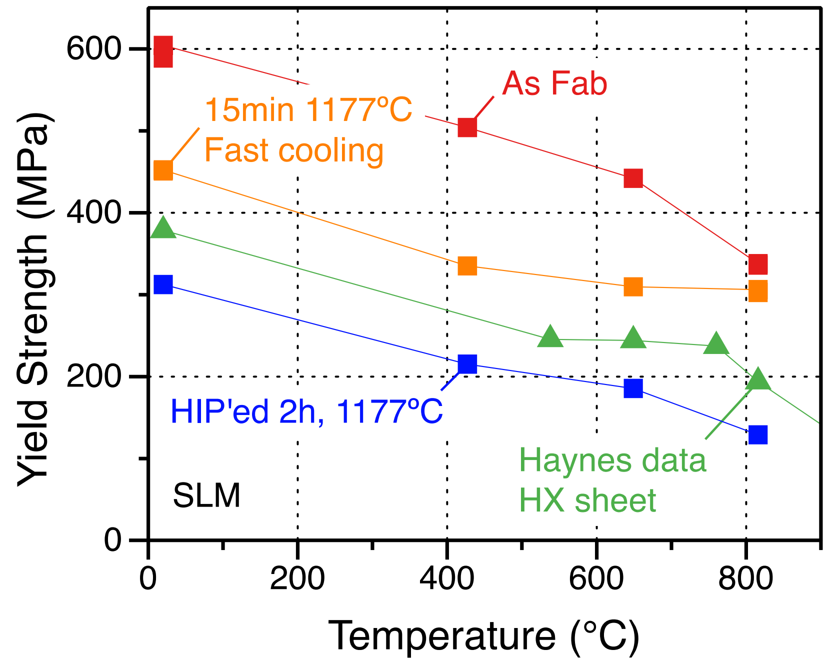
SLM: Fully Dense Material After HIP'ing at 1177ºC/2h/150MPa+Recrystallization

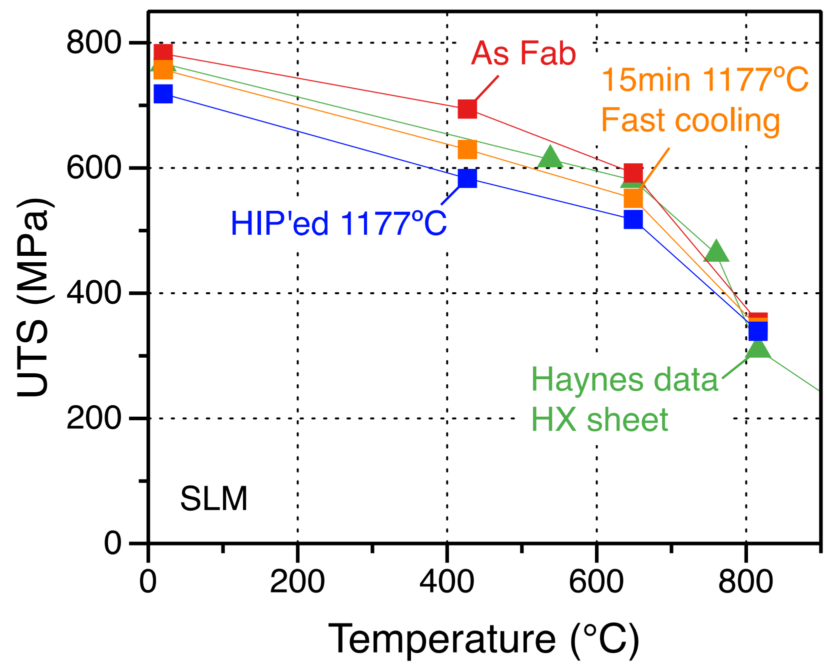


SLM: Increase of Ductility & Decrease of YS after 15min 1177°C or HIP'ing 2h/1177°C/150MPa

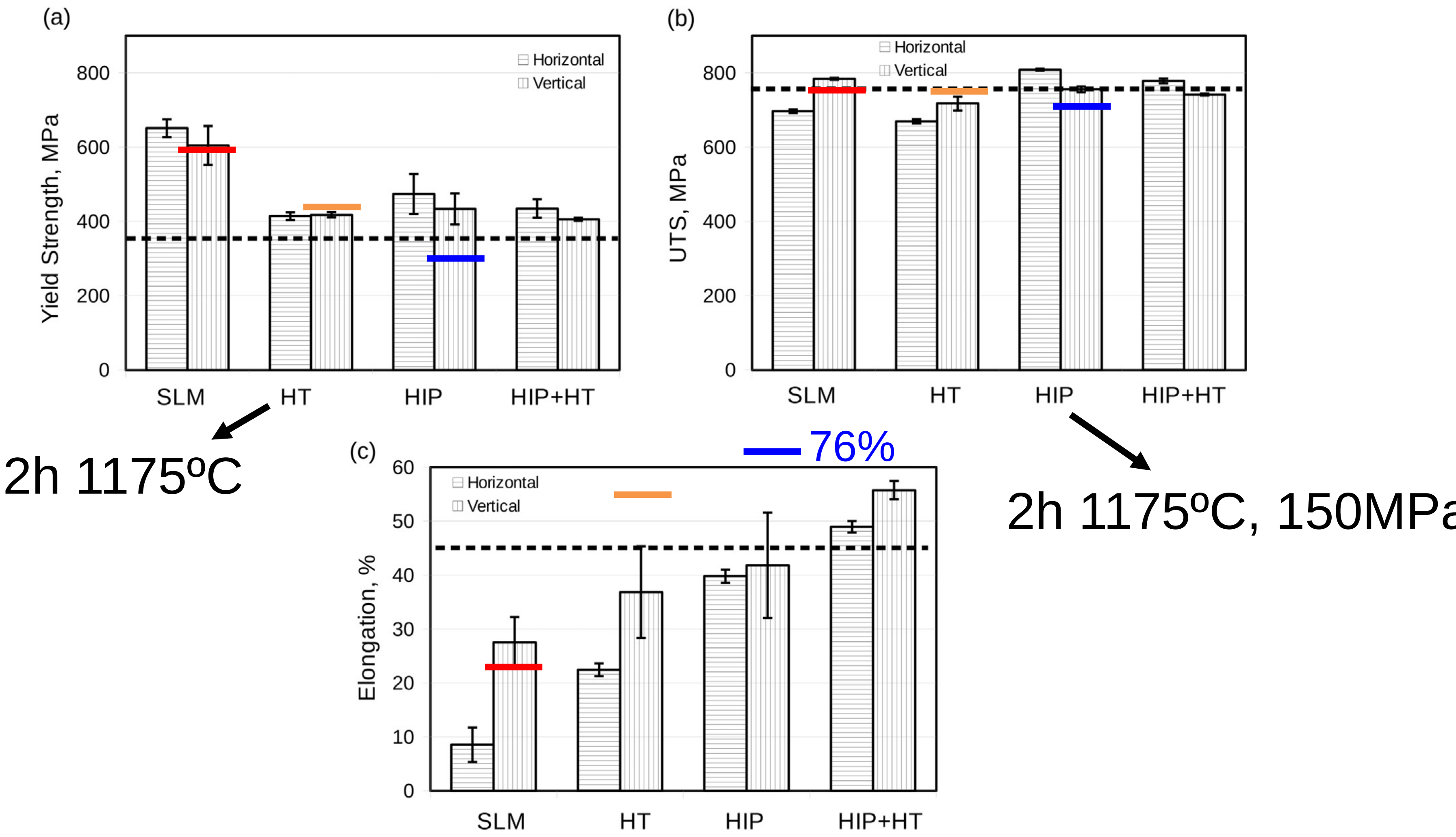
Release of Residual Stress + Microstructure evolution



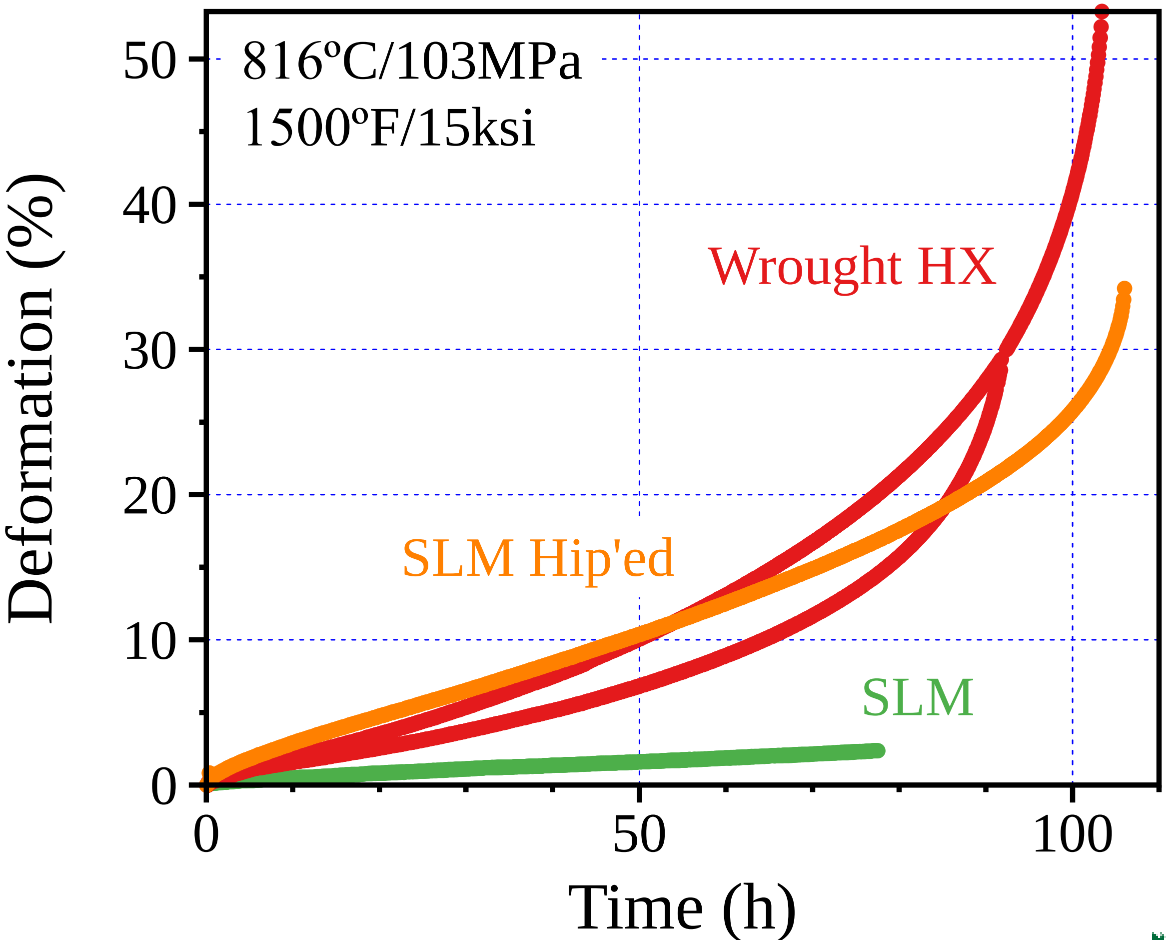




SLM: Result Consistent with Literature Data at Room Temperature

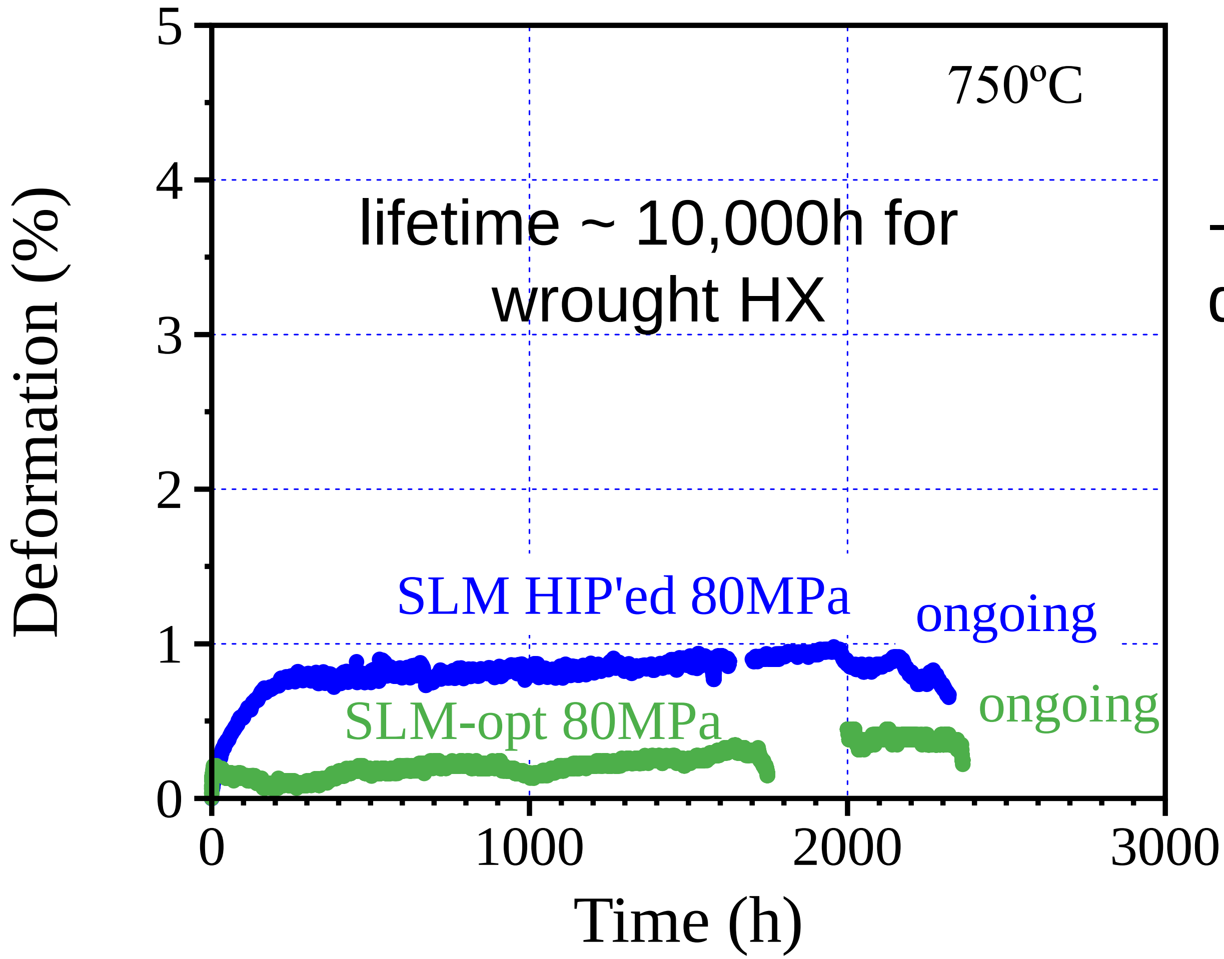


SLM: Significant Improvement of Creep Deformation at 816ºC After Hip'ing Consistent with Tensile Data



Similar properties as wrought HX for HIP'ed SLM HX

SLM: Long Term Creep Tests Have Been Running for 2,300h at 750ºC

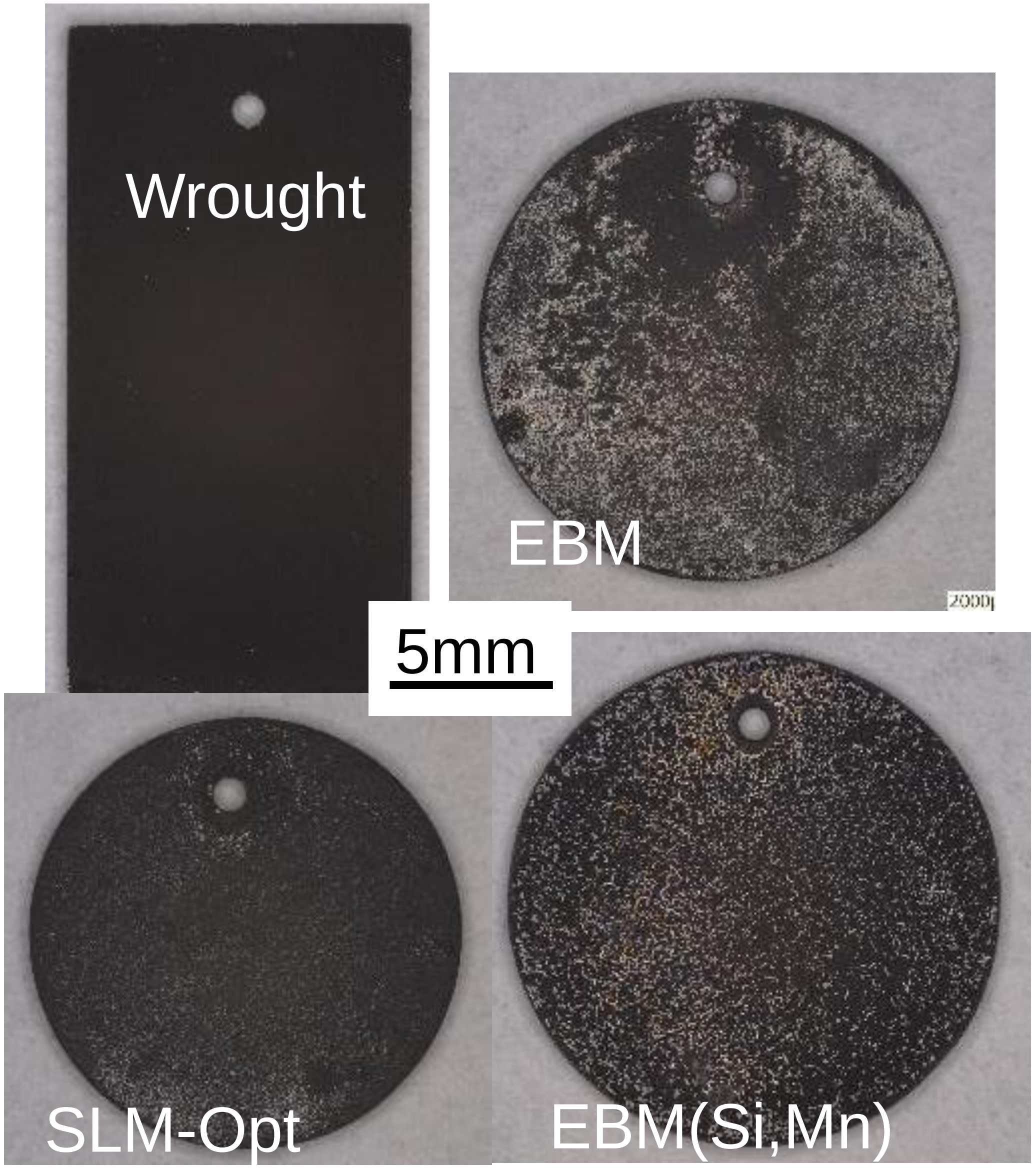


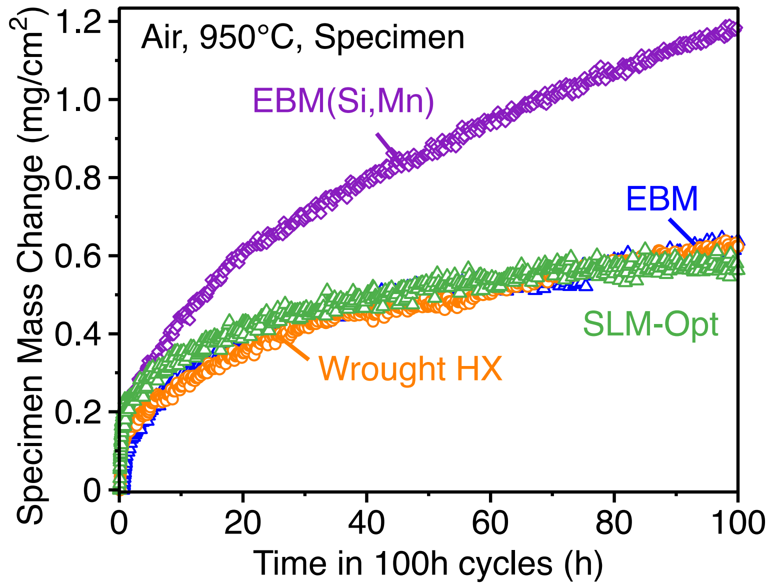
- Higher deformation during primary stage for HIP'ed HX
- 110MPa tests coming soon

Need Oxidation/Corrosion Studies on AM Ni-based Materials

- Different microstructure & surface finish = various oxidation behaviors?
- Topology optimization using AM will lead to thinner components and lower oxidation lifetime
- **Optimization of the microstructure at the surface for better oxidation/corrosion behavior?**

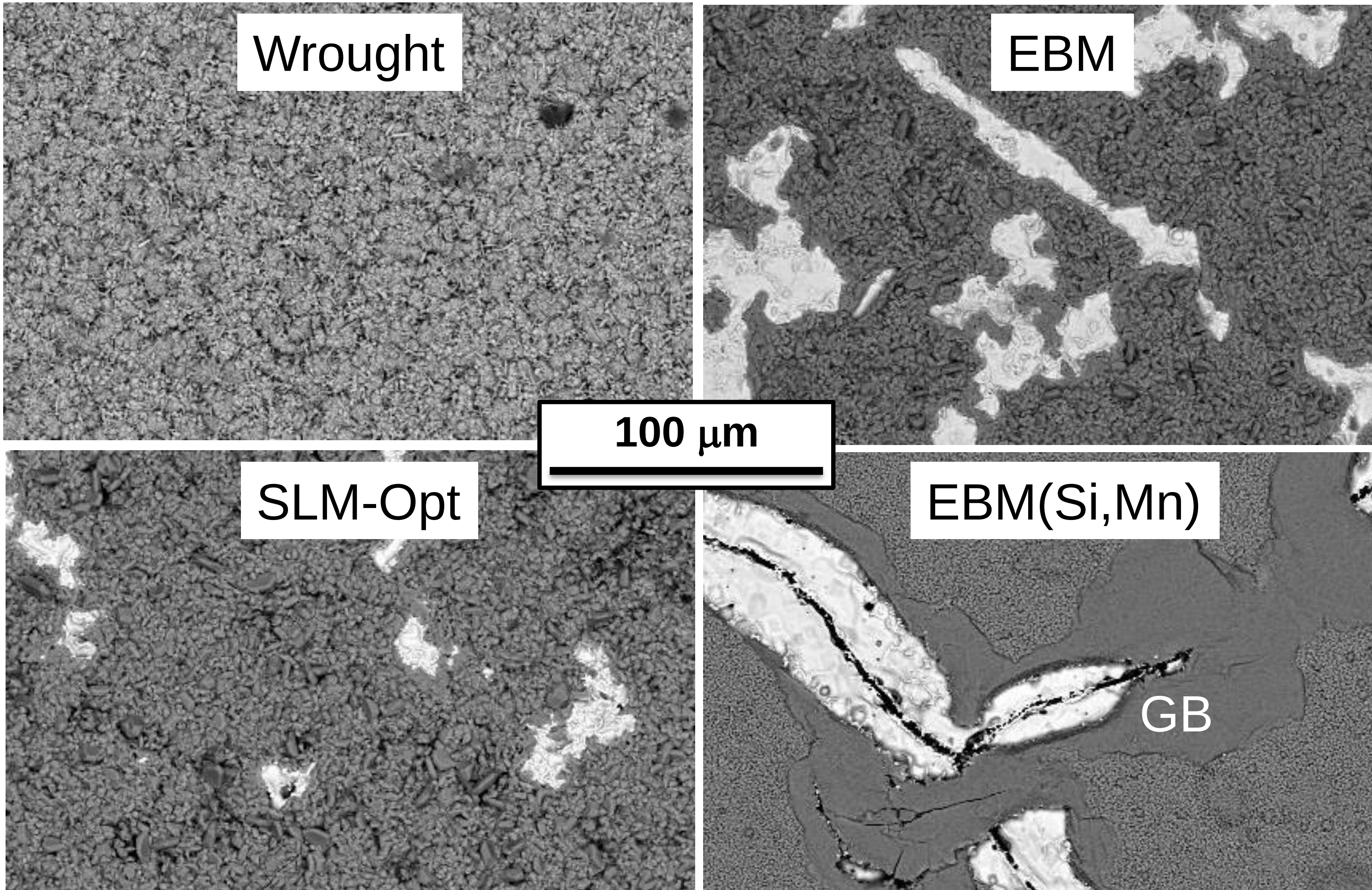
100h TGA Tests at 950ºC in Air to Assess AM HX Oxidation Resistance



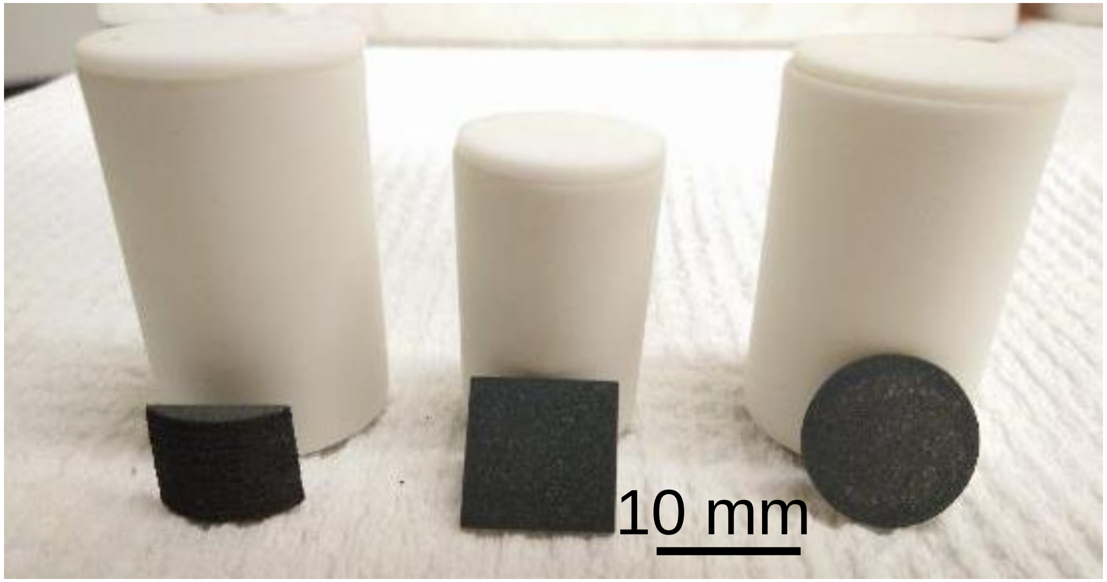


- Higher oxidation rate for EBM(Si,Mn)
- Spallation for EB alloys
- Moderate spallation for SLM-Opt

100h TGA at 950ºC : Spallation Initiates at Grain Boundaries for EBM(Si,Mn)

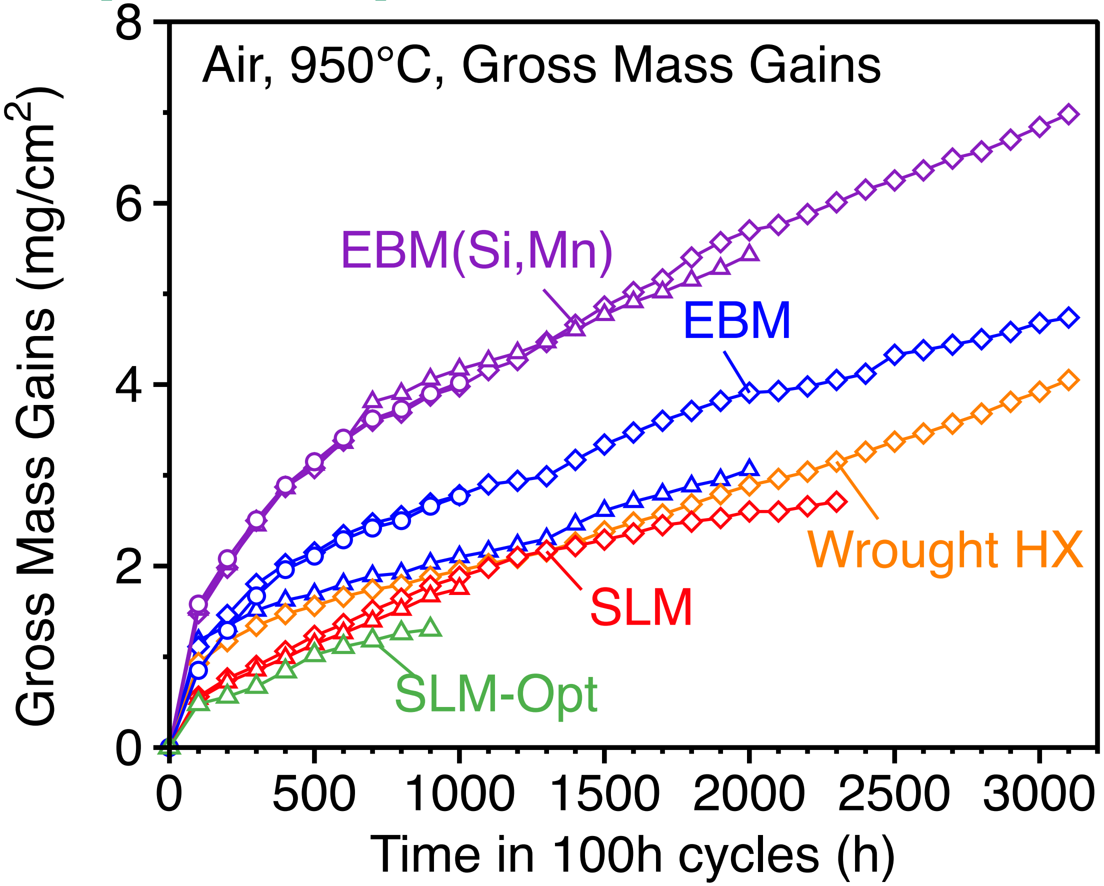


Cyclic Oxidation Testing of HX in Box Furnace at 950ºC, 100h Cycles



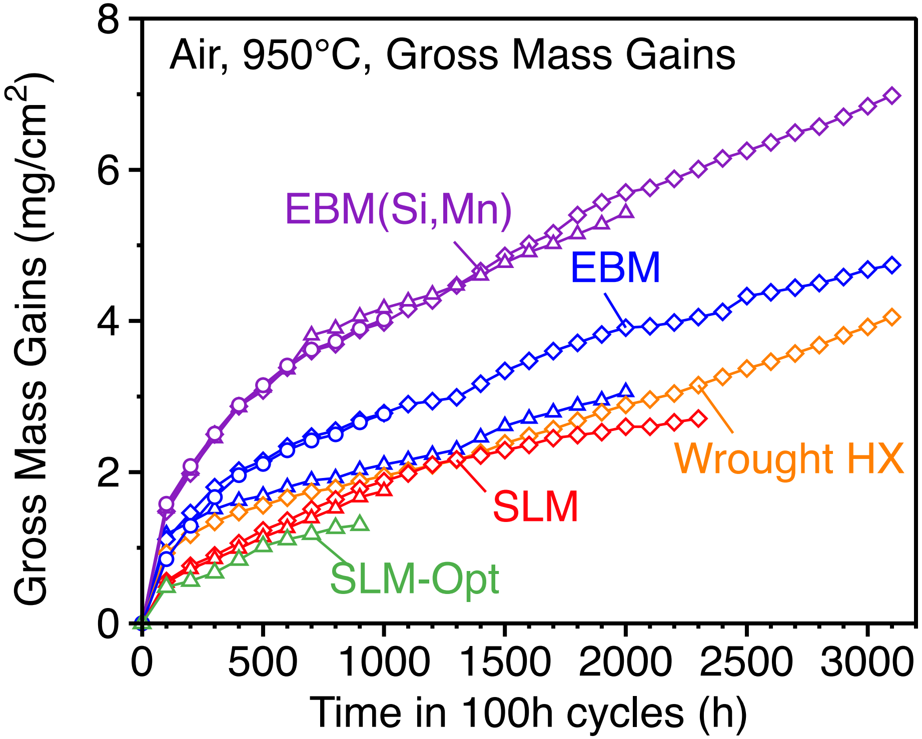
- 950ºC for fast assessment of long-term oxidation behavior
- As Fabricated AM coupons. **Polished** & not polished
- Crucible to generate both specimen and gross mass gains
- Gross mass gains = oxygen pickup ~ Metal (Cr) loss
- Surface imaging using Keyence 3D microscope

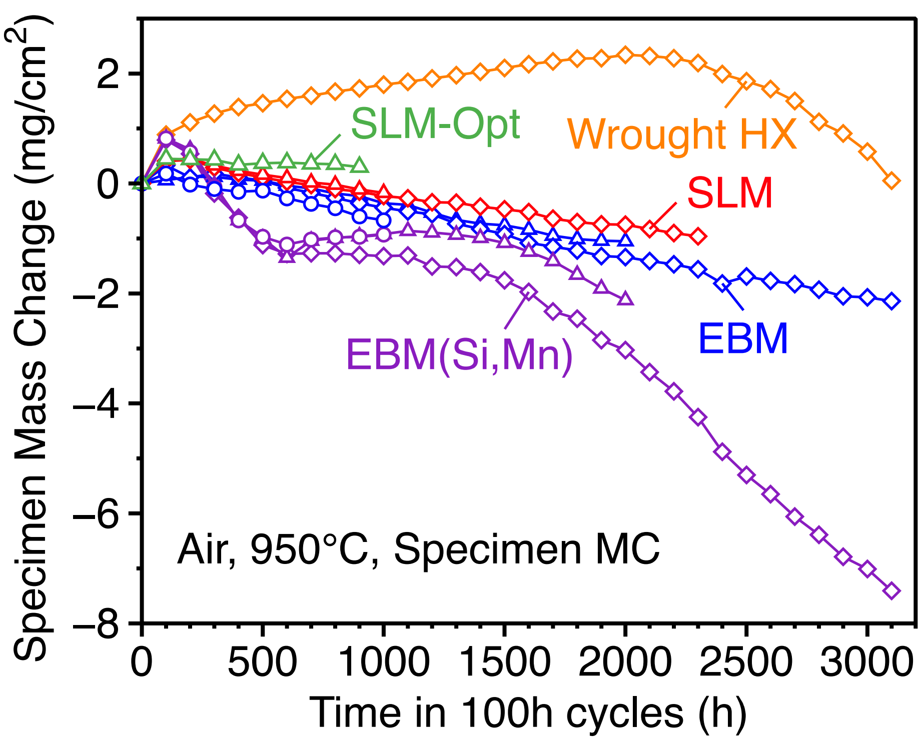
Faster Initial Oxidation Rate for EBM(Si,Mn) and 2 EBM specimens



- Similar Gross MG for Wrought & one EBM specimen
- Lower GMG for SLM and SLM-Opt specimen

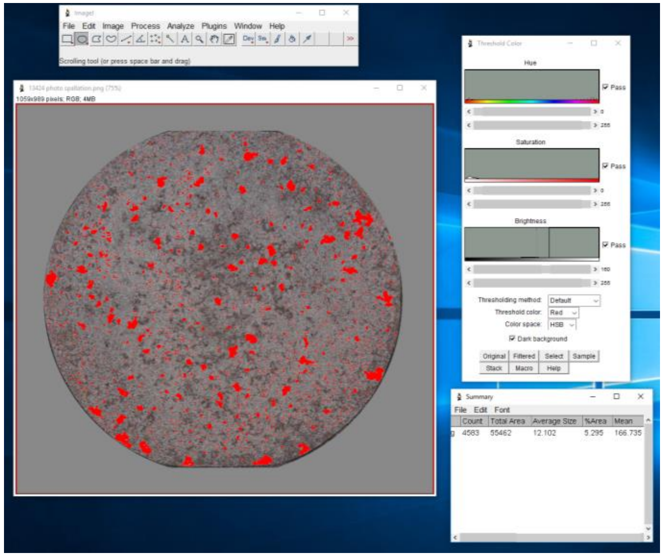
High Spallation for EBM(Si,Mn). Moderate Spallation for SLM & EBM. Increasing Spallation for Wrought HX





- Change of spallation rate with time
- Evaporation likely negligible compare to spallation

Decrease of Spalled Area Fraction With Time for AM Specimens. Increase for Wrought HX



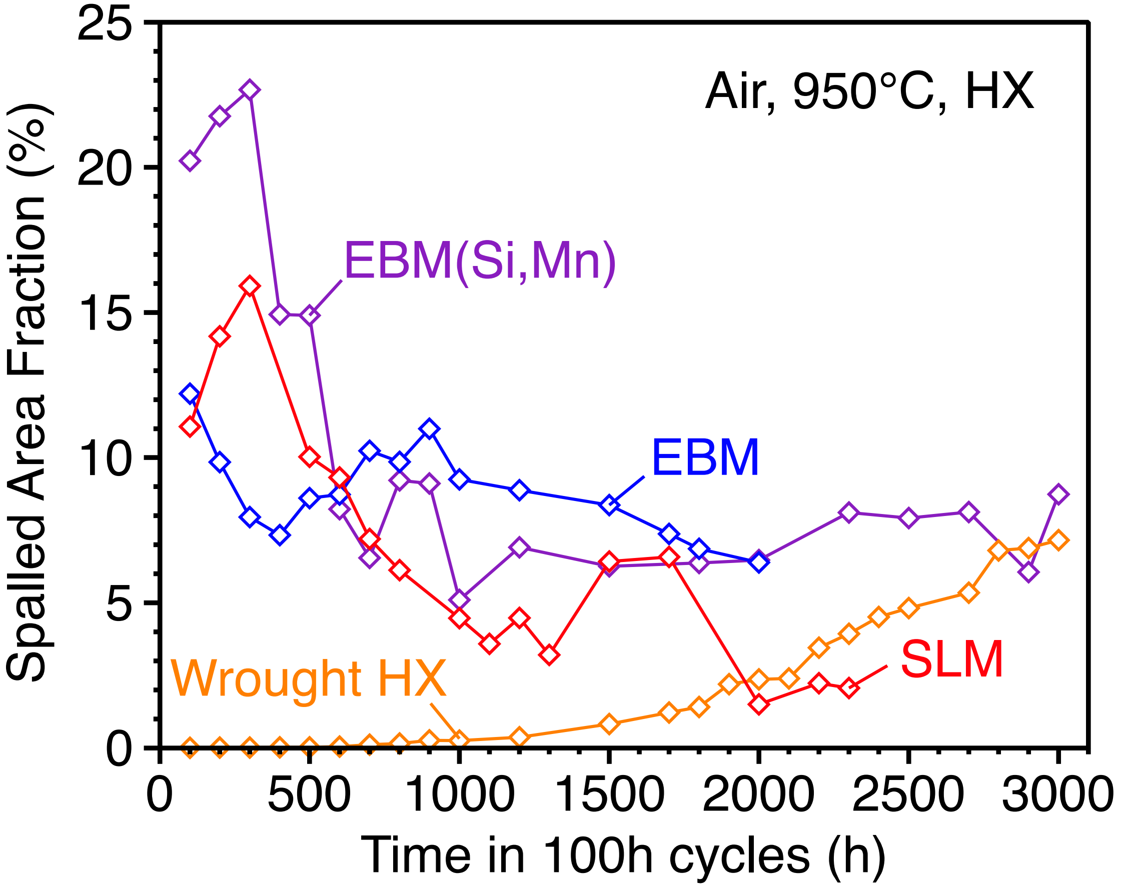
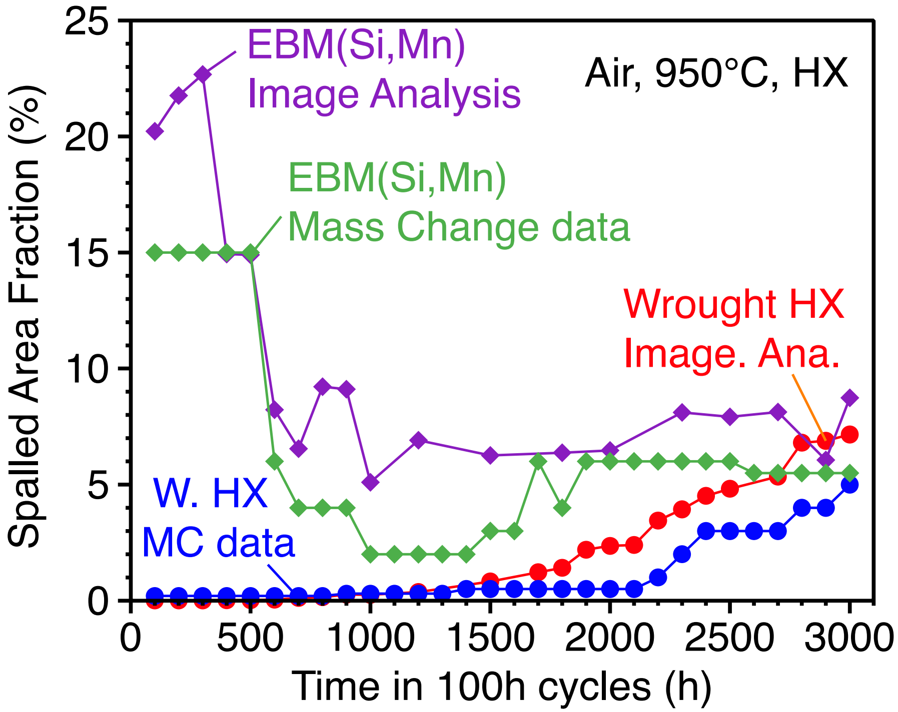


Image J Analysis
Chistina Cox UT
Austin Ngo UT
Caroline Widomski, Esirem (France)

Decrease of Spalled Area Fraction With Time for AM Specimens. Increase for Wrought HX



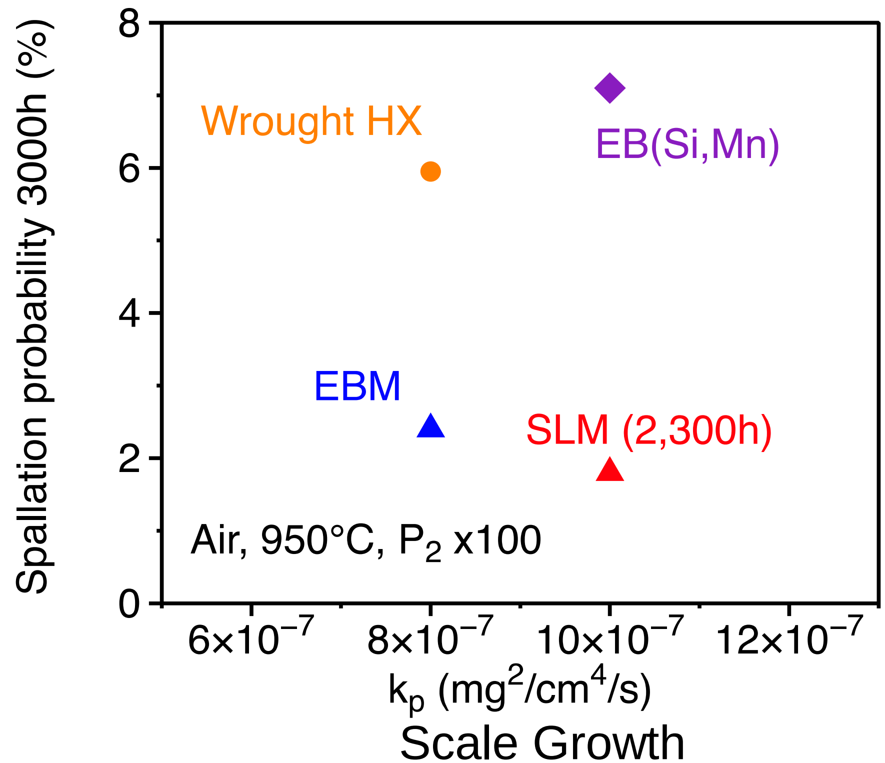
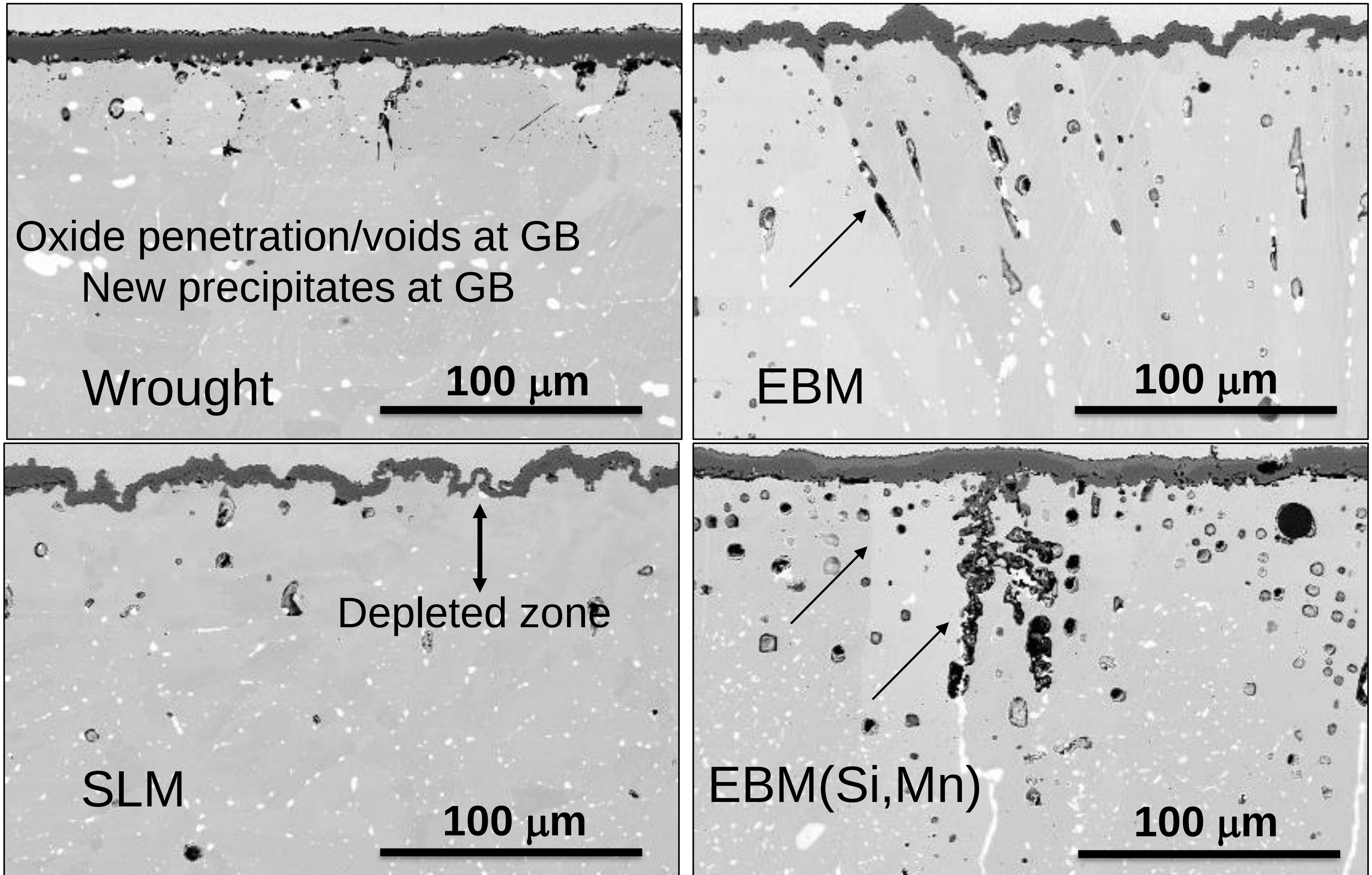


Image Analysis & Mass Change Data/Modeling Yield Similar Spallation Fraction

10x100h Cycles: Voids formation in the Grain and at GB for EBM HX



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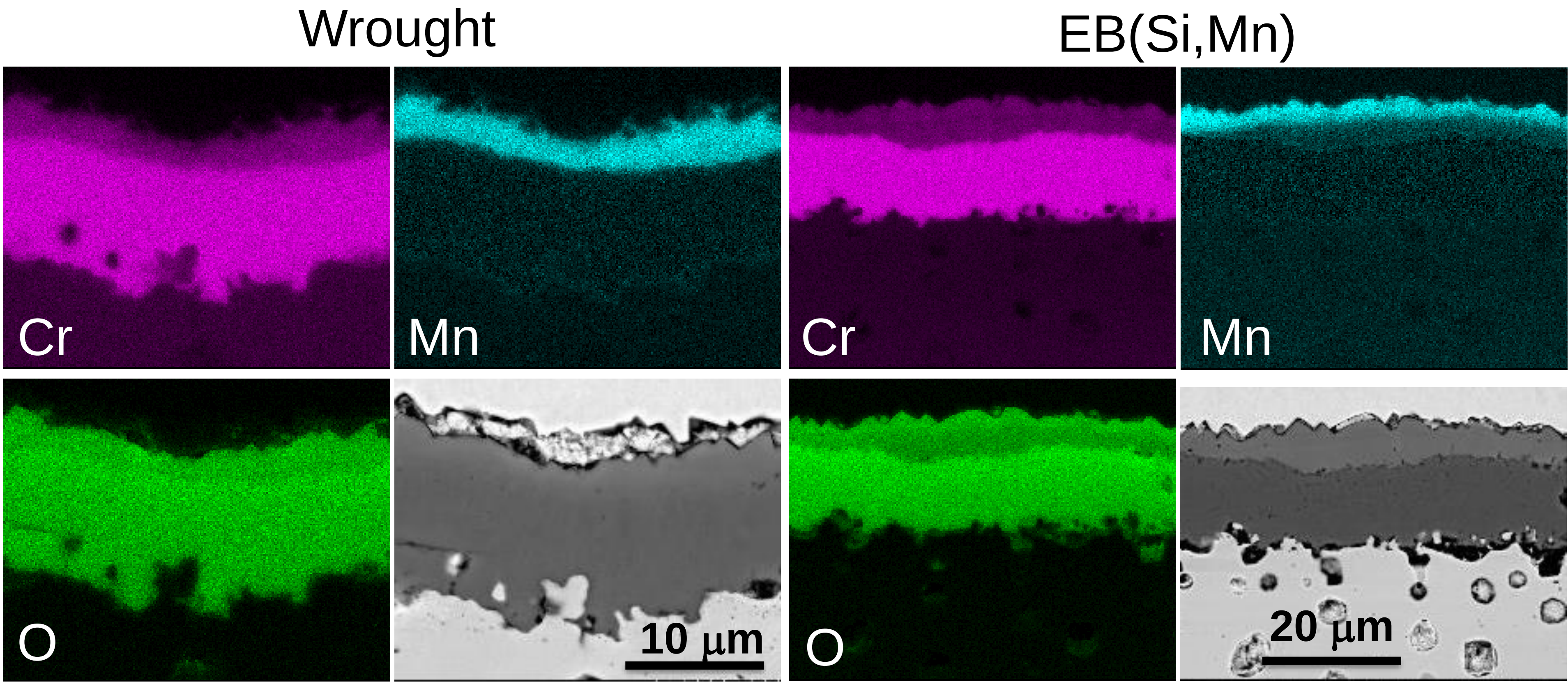
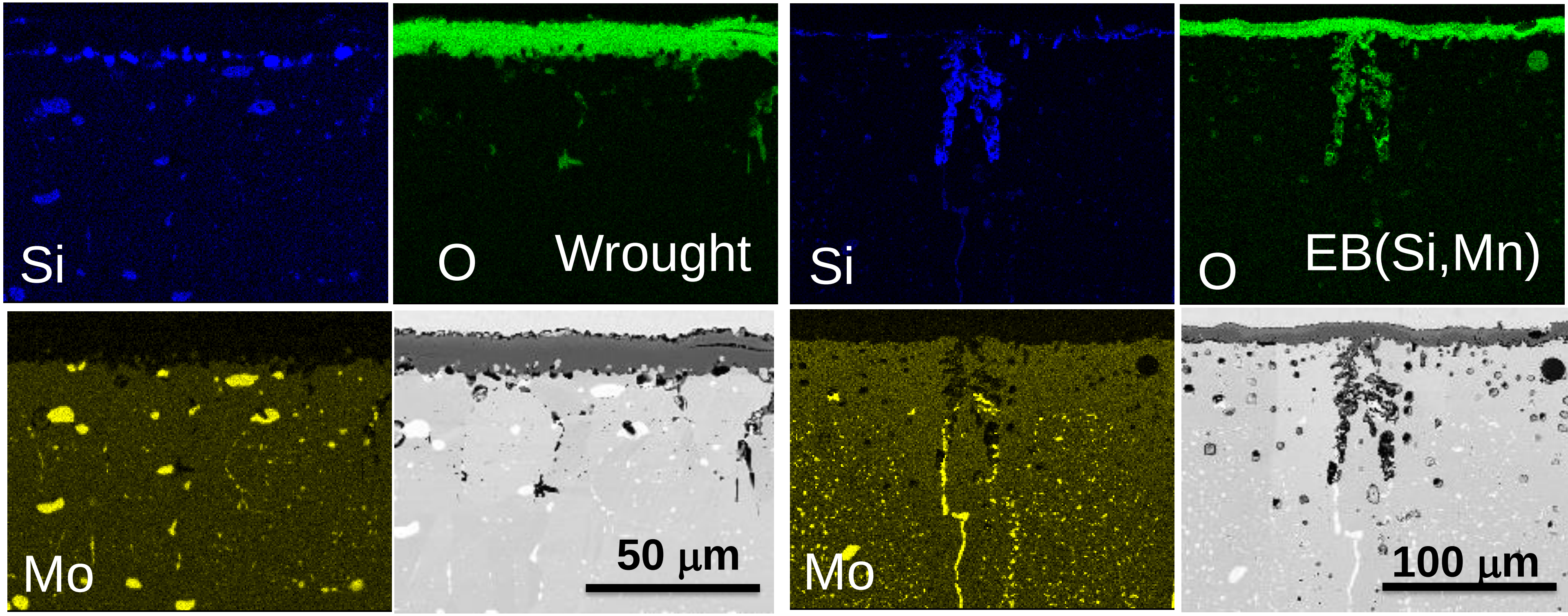
1000h, 950°C, Wrought & EBM(Si,Mn): (Cr,Mn) and Cr-Rich Layers

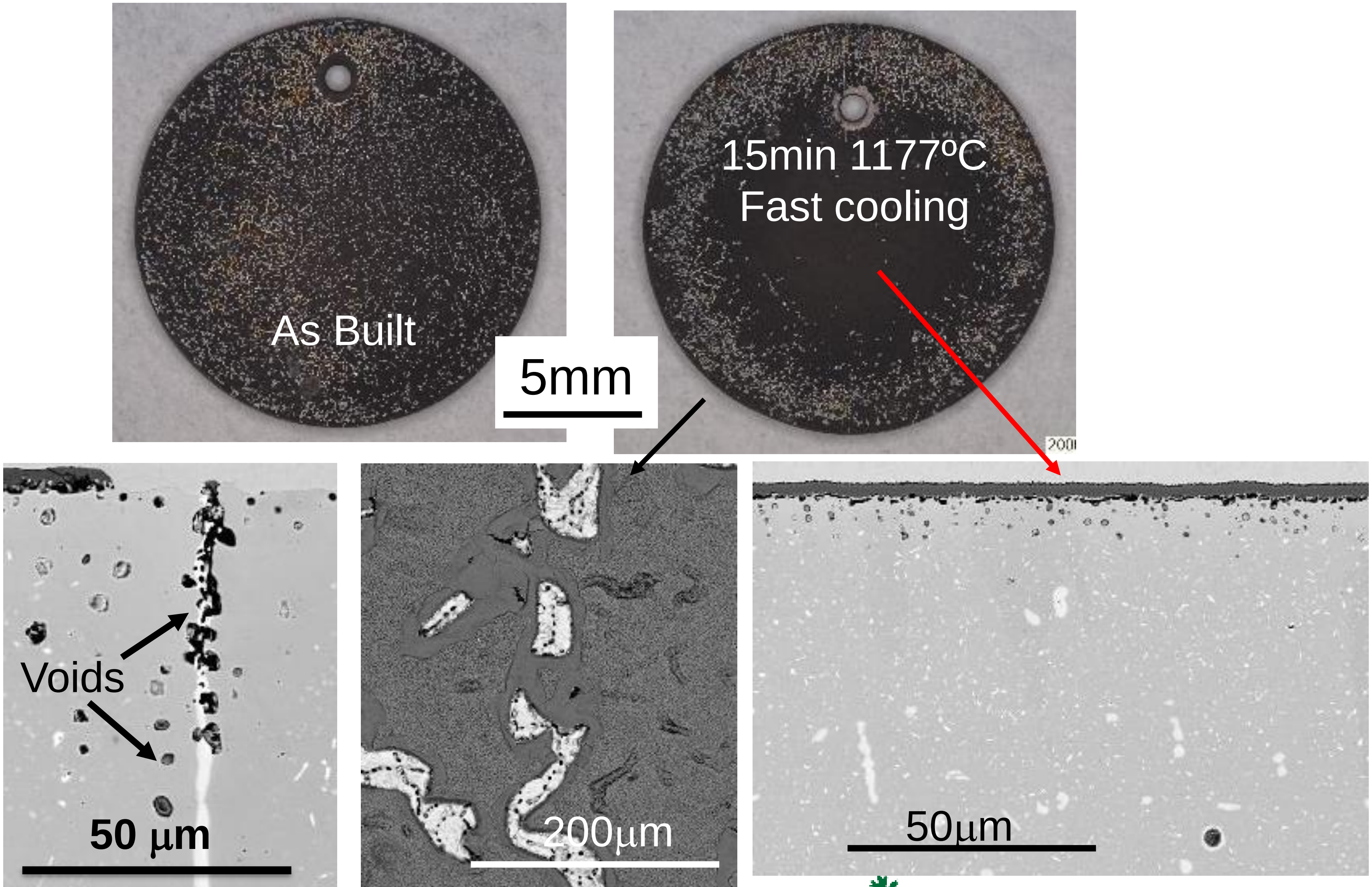


1000h, 950ºC, Wrought and EBM(Si,Mn): Si segregation at Interface + Oxide Penetration along GB



Cr-rich oxide + numerous voids at GB
Mo depleted zone for EBM(Si,Mn)

Locally Less spallation after 15min 1177ºC Fast Cooling for EB(Si,Mn)



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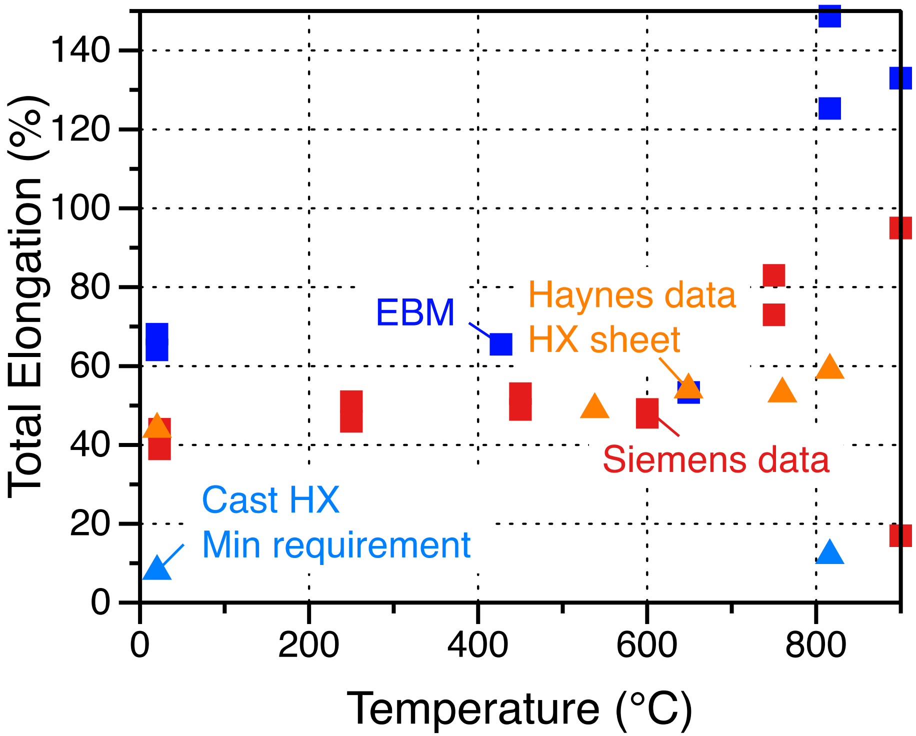
Conclusion-EBM

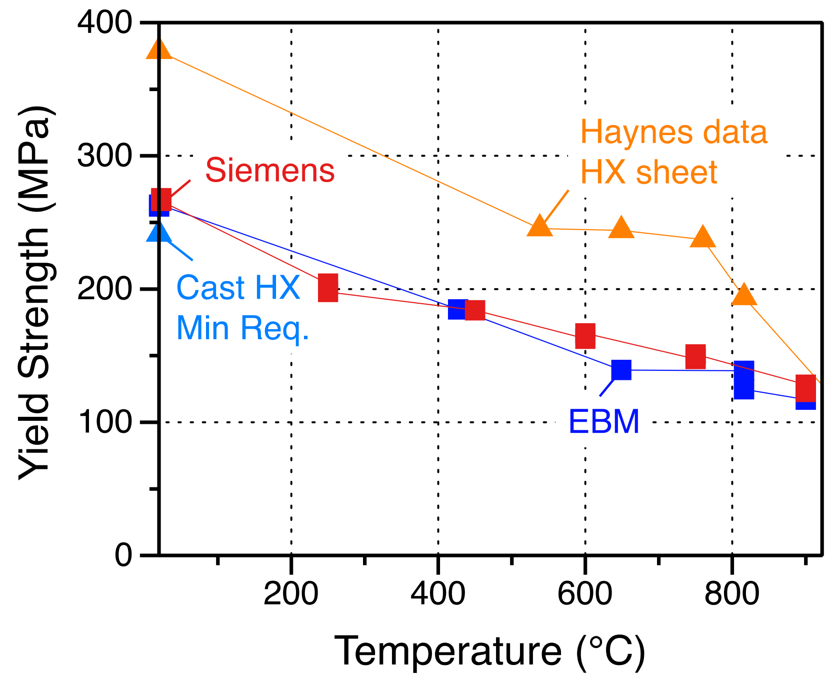
- EBM HX shows good ductility and tensile strength superior to **the cast HX requirement**
- Good fatigue properties for the EBM HX but lower creep strength. Sufficient for some applications using cast HX?
- EBM & EBM(Si,Mn)HX microstructure could certainly be further optimized to increase the alloy creep performance
- Hip'ing resulted in a significant improvement of HX EBM creep properties. No effect on EBM(Si,Mn).
- Significant effect of Si & Mn content on microstructure and oxidation.
- Great opportunity (already happening) to develop new AM HX alloy

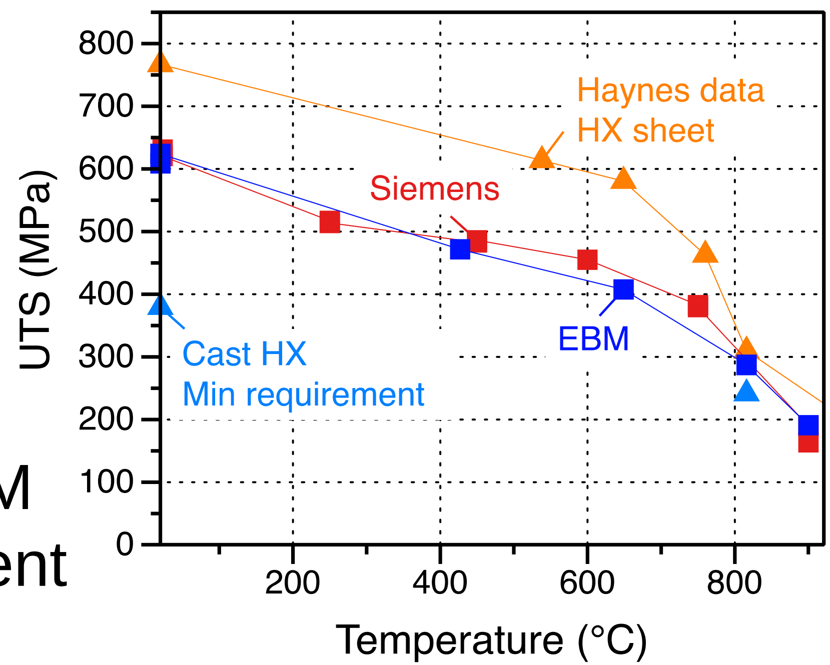
Conclusion-SLM

- Hot tearing led to crack formation as observed by others. Need to optimize fabrication parameters or alloy composition
- SLM HX exhibited good tensile strength and acceptable ductility. Good creep strength but limited ductility and high residual stress
- HT or Hip’ing increased the SLM HX ductility & reduced YS. Creep ductility was significantly improved by Hip’ing. Similar properties as wrought HX
- Increased spallation was observed for SLM HX alloy after 100h at 950ºC but lower spallation rate after 3,000h.
- Will start soon to generate data perpendicular the build direction

EBM HX Alloys Exhibit Good Ductility But Lower Strength







- Higher ductility for ORNL EBM
- Meet cast HX AMS requirement