

Microstructure and Properties of Hastelloy X Fabricated by Additive Manufacturing

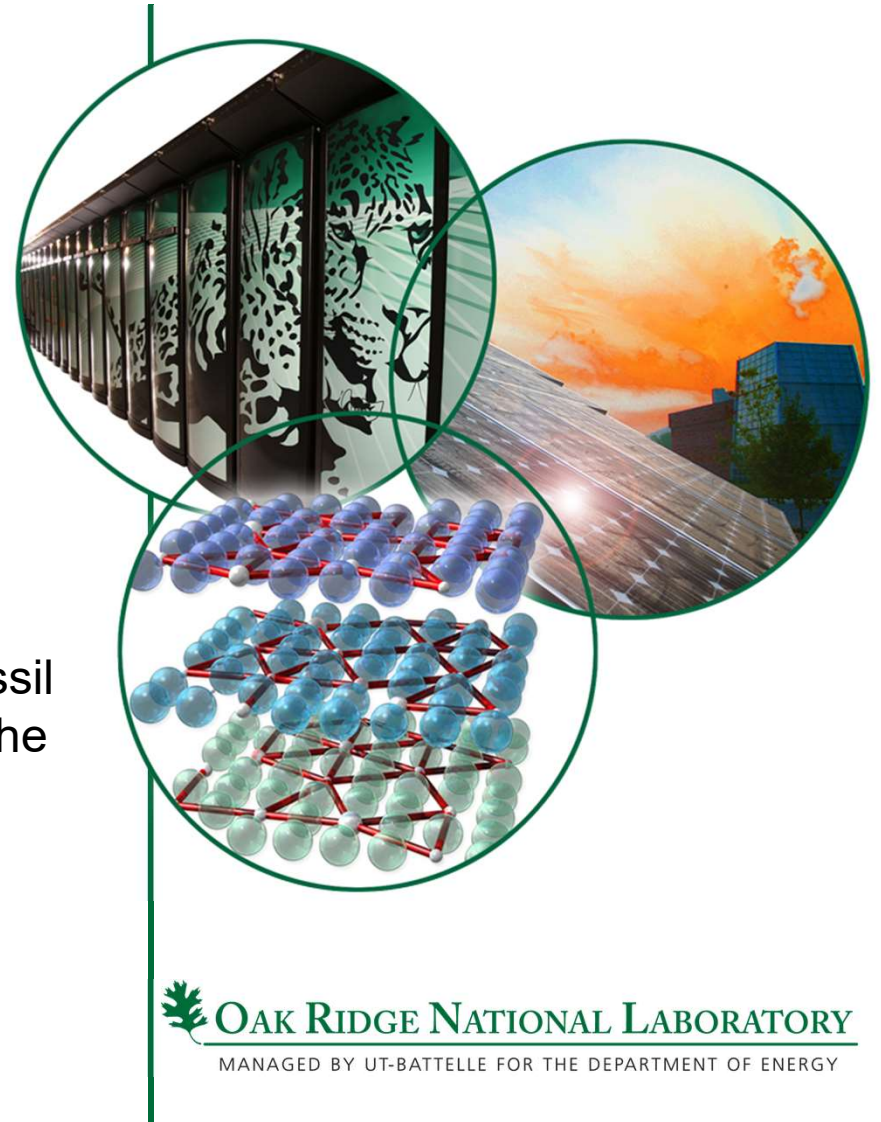
Sebastien Dryepondt, Mike Kirka
& Frederick A. List

Oak Ridge National Laboratory

Crosscutting Research Review Meeting

ORNL project FEAA119

This research was sponsored by the U.S. Department of Energy's (DOE), Office of Fossil Energy, Crosscutting Research Program & the DOE Advanced Manufacturing Office



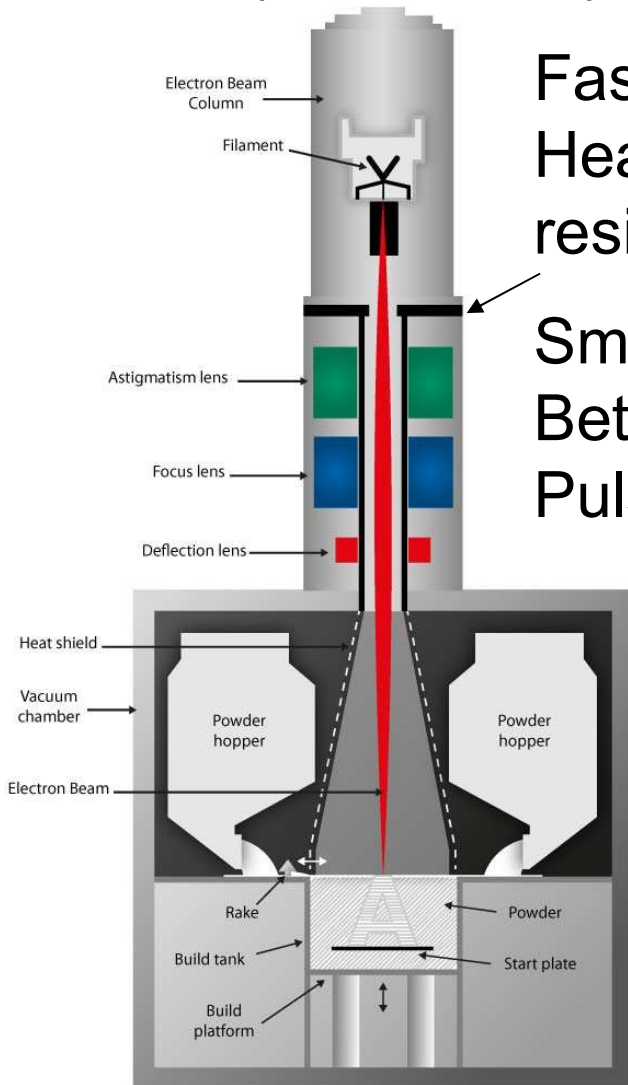
Project Objectives

- Optimize additive manufacturing (AM) fabrication processes for solution strengthened Hastelloy X (HX, Ni-22Cr-19Fe-9Mo) gas turbine components (Fuel injector, Combustor)
- Generate data (Tensile, Fatigue, **Creep, Oxidation**) relevant for FE applications for Hastelloy alloy
- Properties requirement depends on component and application (prototyping, production and repair)
- Compare three AM techniques, electron beam melting (EBM) and selective laser melting (SLM) and laser metal deposition (LMD)
- 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)

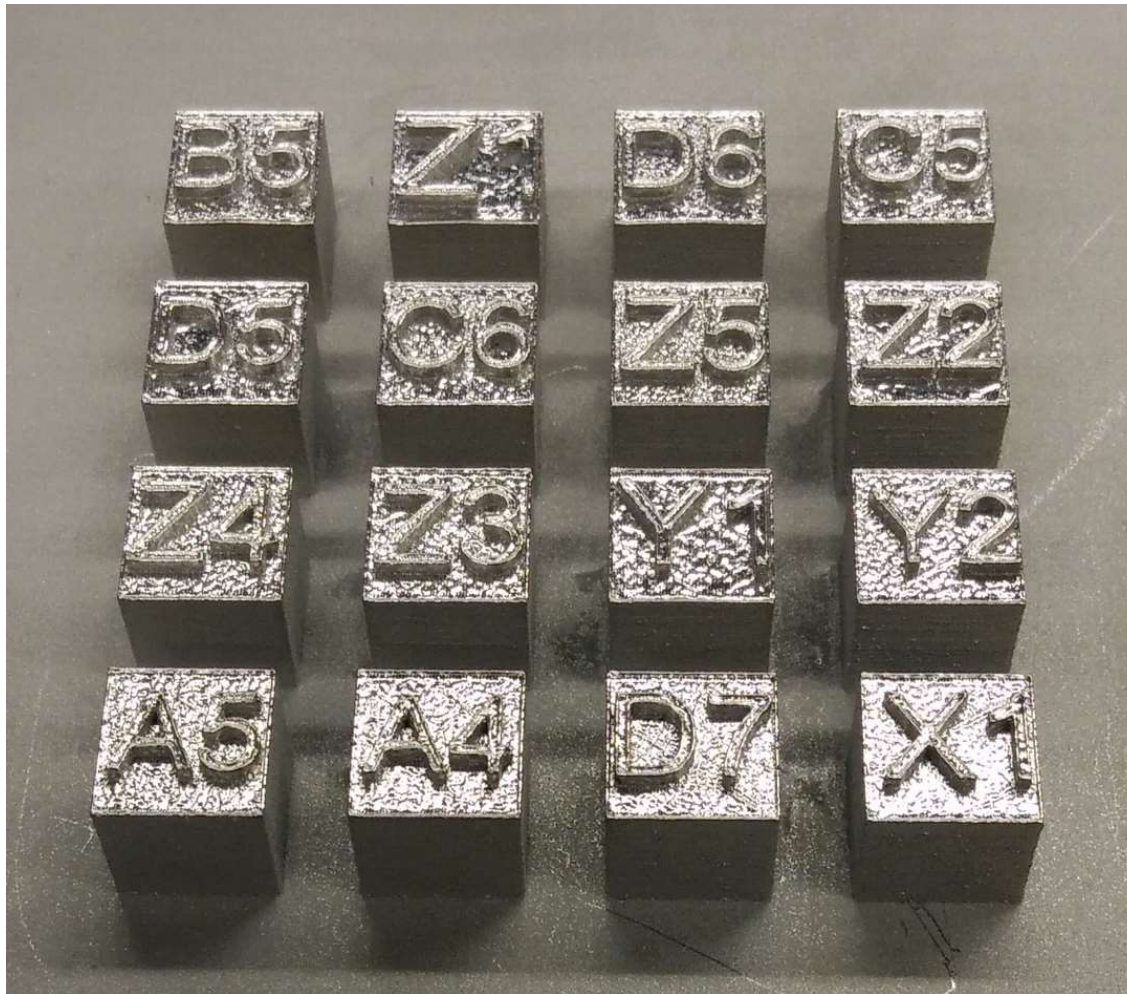
Laser (Renishaw AM250)



Faster
Heated bed = lower residual stress
Smaller beam size
Better resolution
Pulsed laser beam

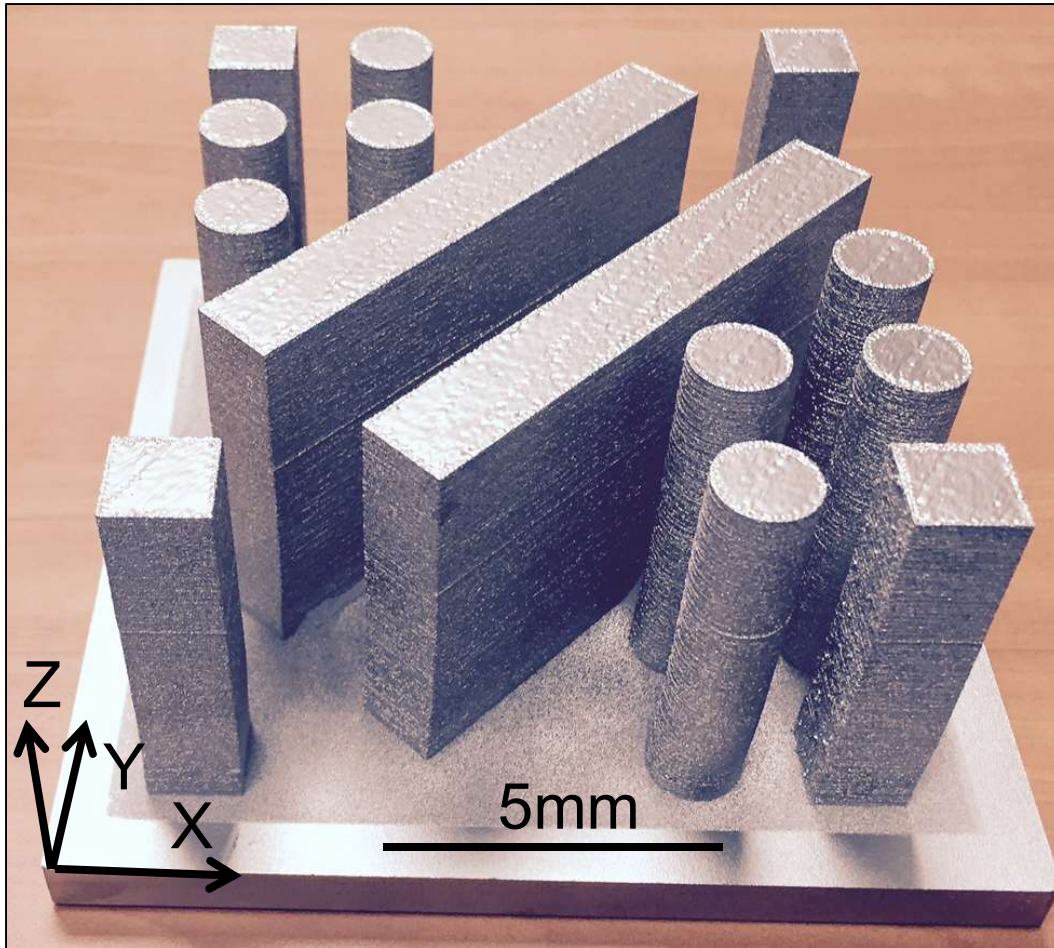


Small SLM HX Cube for Parameter Optimization Based on Microstructure



- Spot time
- Spacing
- Energy
- Optimization based on previous experience with Ni-based alloys
- typical duration ~2 weeks

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



- Study the effect of property anisotropy

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

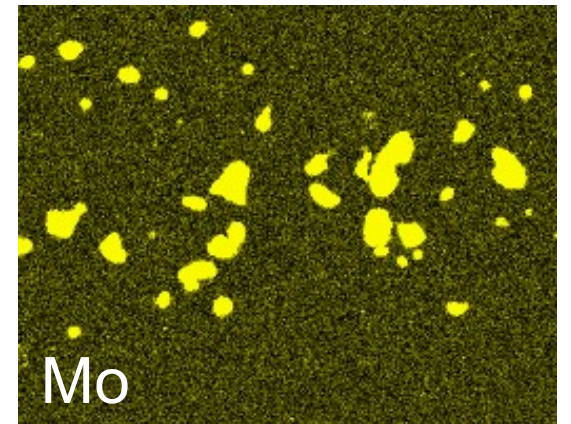
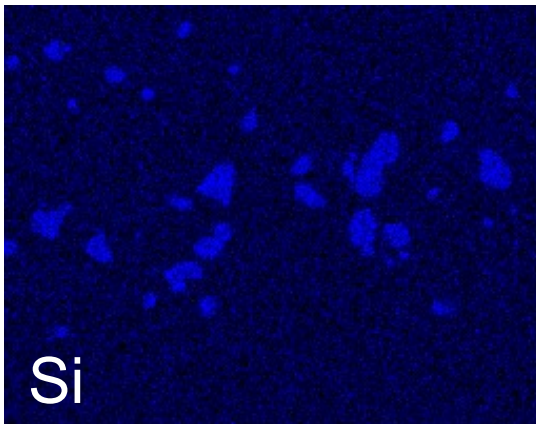
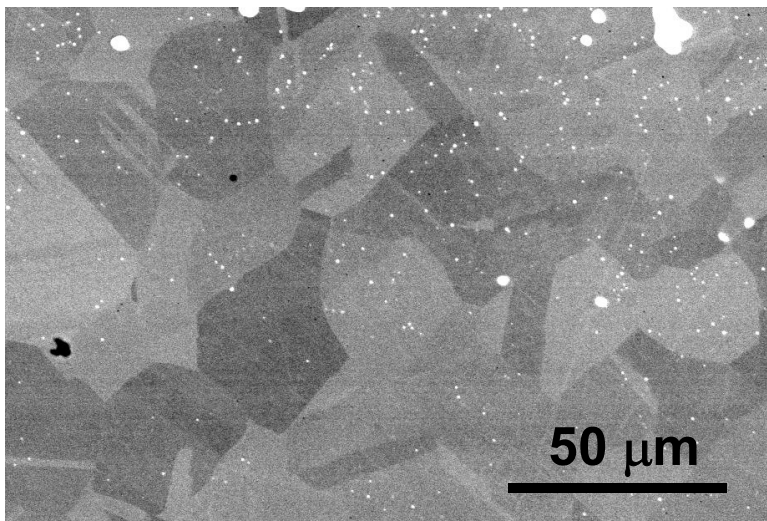
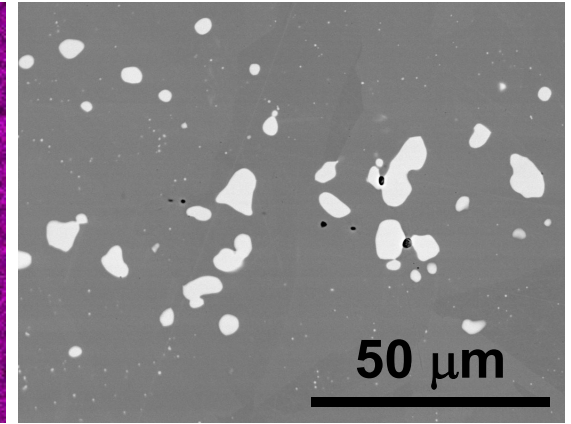
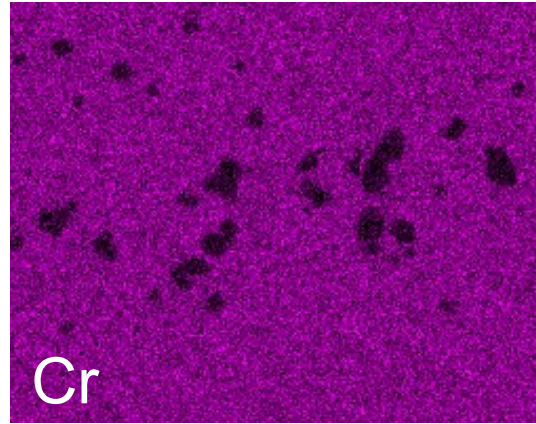
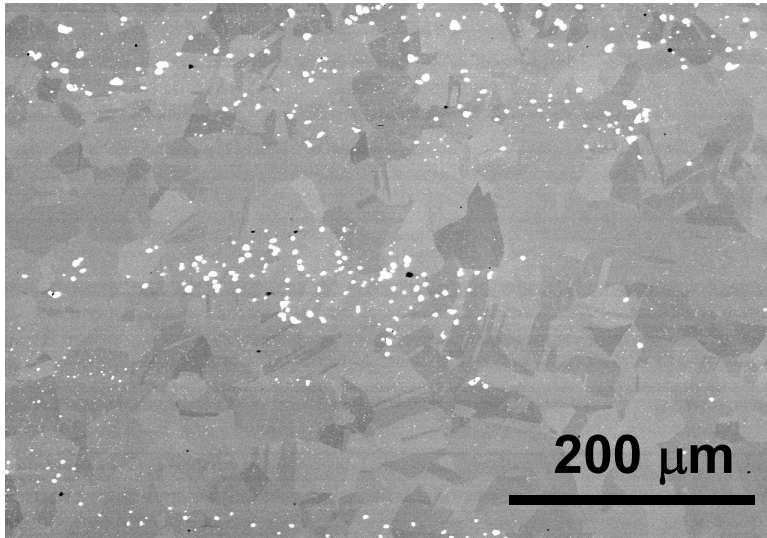
Hastelloy X (Ni-22Cr-19Fe-9Mo)

	Ni	Cr	Fe	Mo	Co	Mn	Si	W	C
EBM Alloy	Bal.	21.38	18.55	9.05	1.55	0.01	0.05	0.64	0.078
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

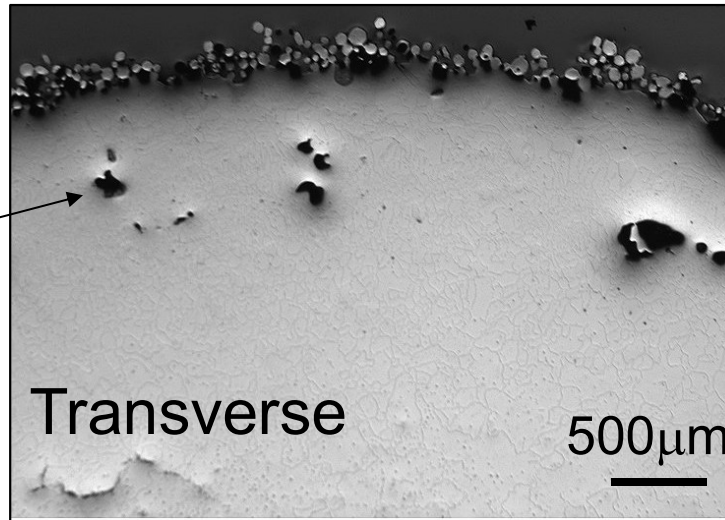
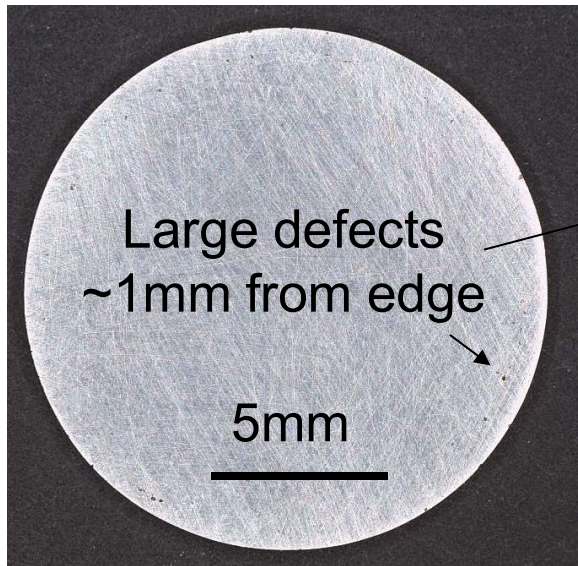
- Alloy composition consistent with EBM powder composition
- High concentration of Mn and Si in EBM(Si,Mn) and wrought HX. Specification: Mn and Si <1%

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

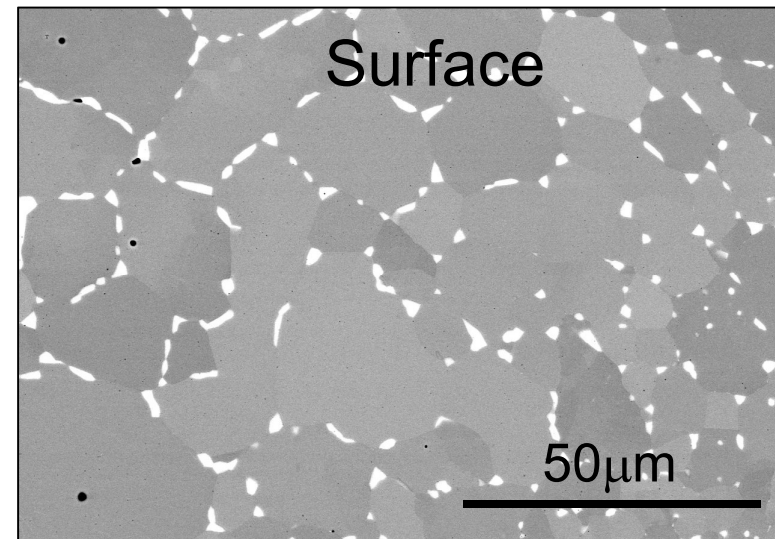
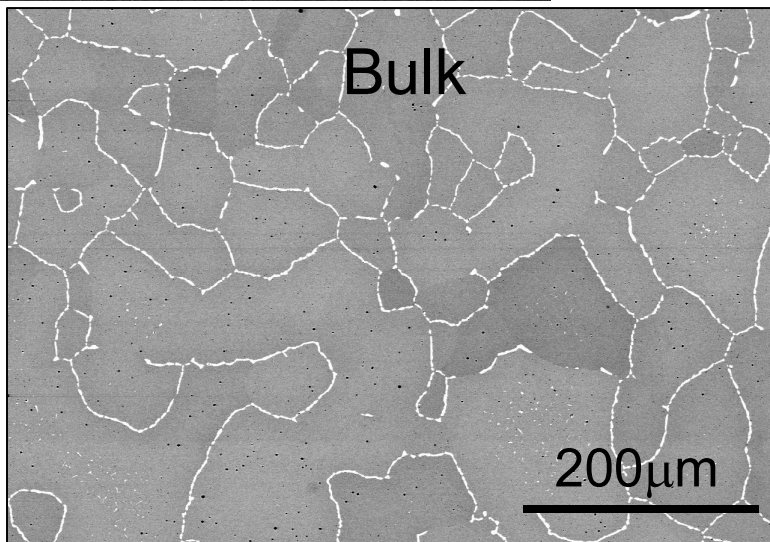
~50 μ m equiaxed grains



EBM: Larger Grains at the Center vs Surface



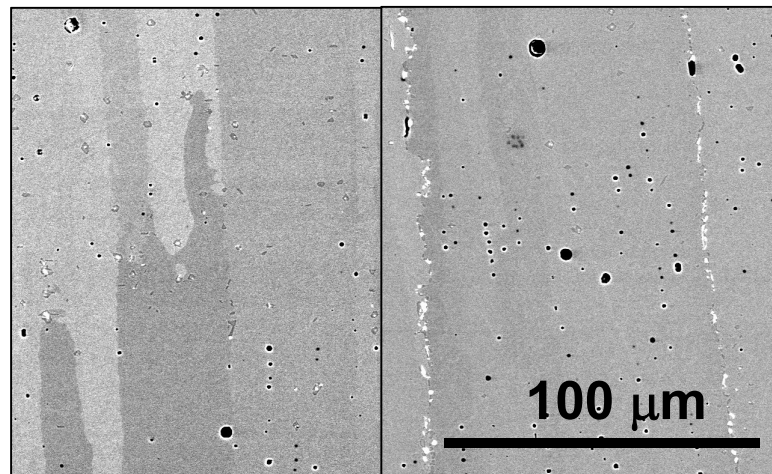
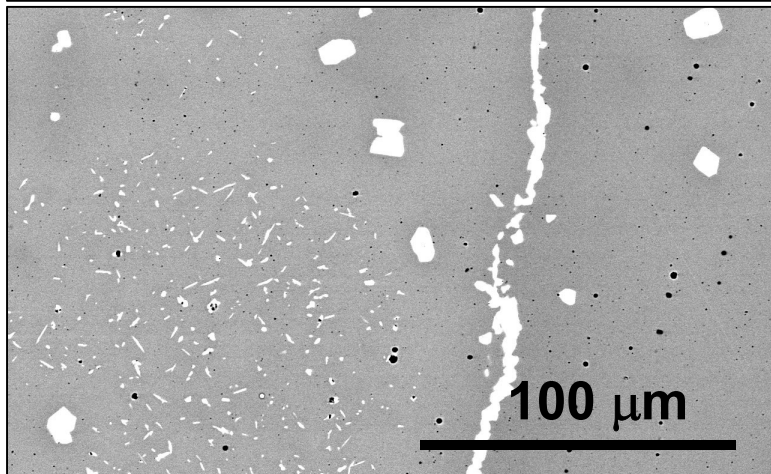
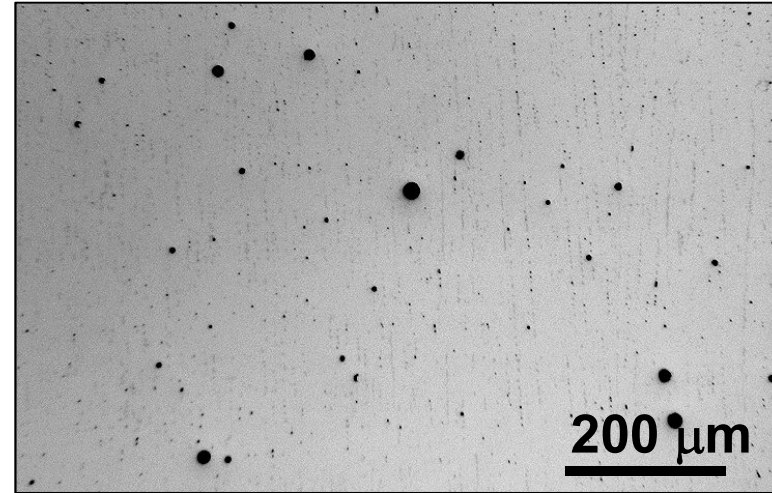
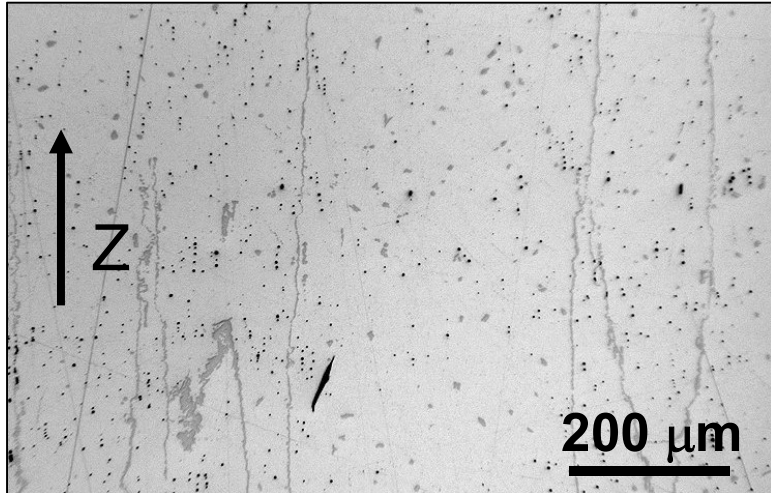
Fabrication
of contour
first



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

EBM(Si,Mn) = Larger grains

EBM = Larger voids

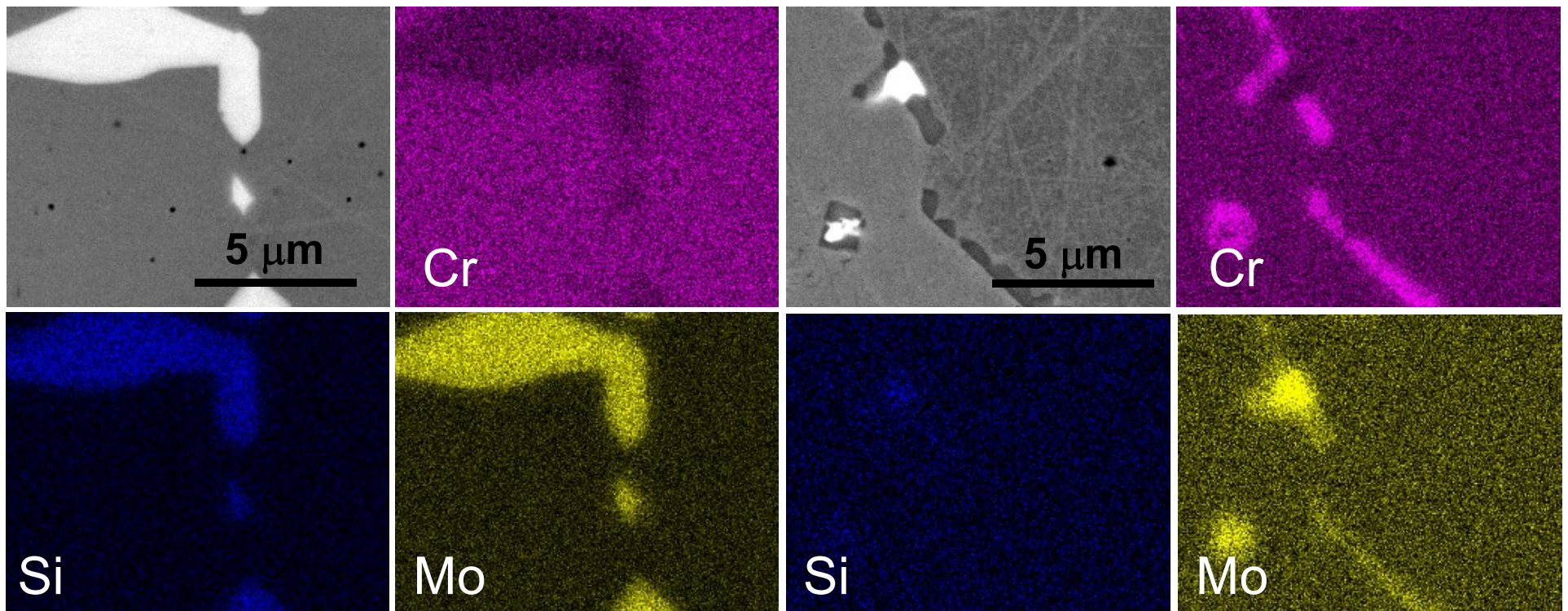


EBM(Si,Mn): Many (Mo,Si)-rich Carbides

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

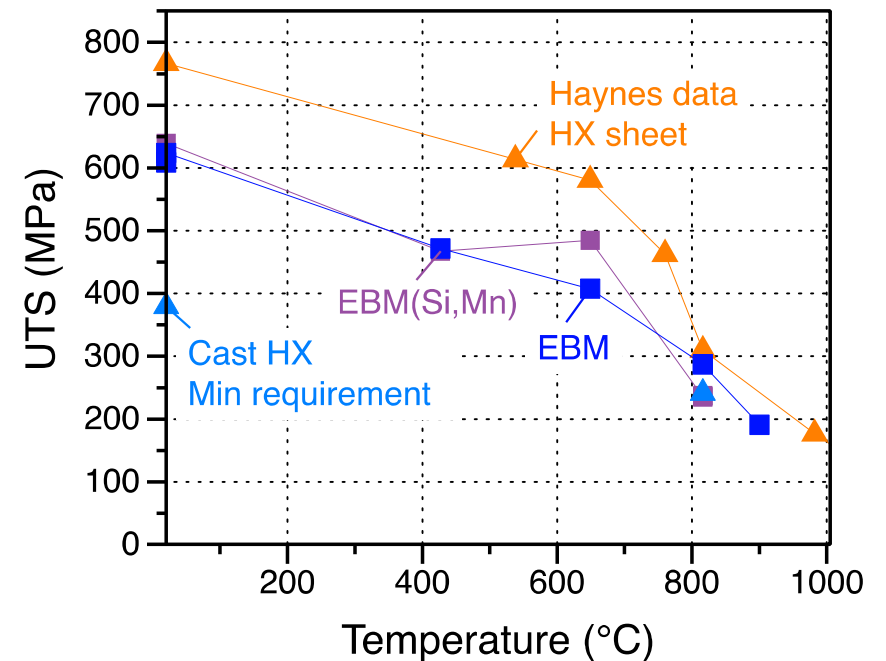
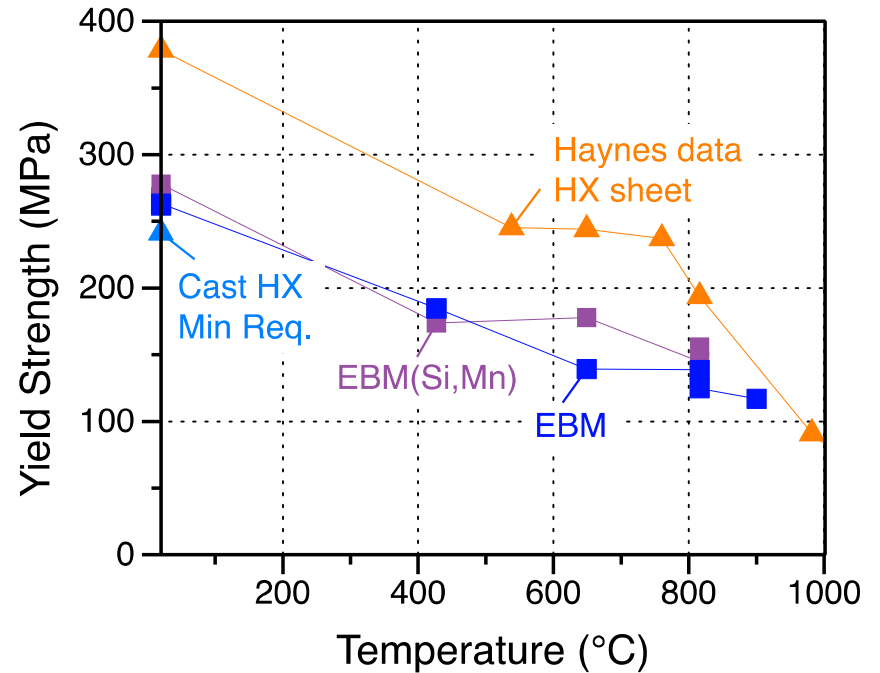
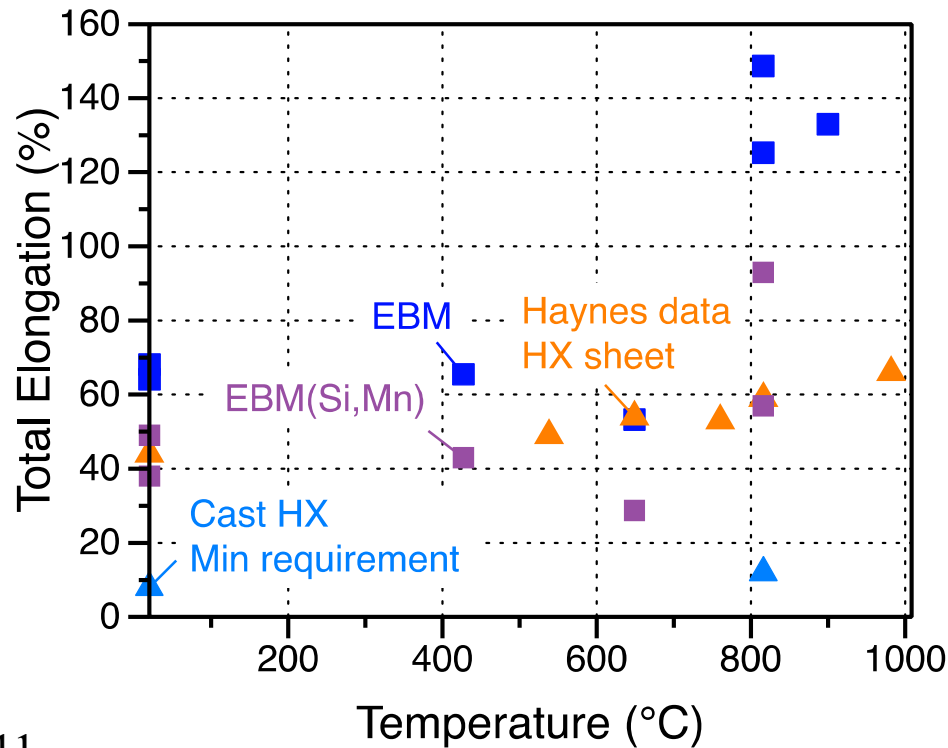
EBM(Si,Mn)

EBM

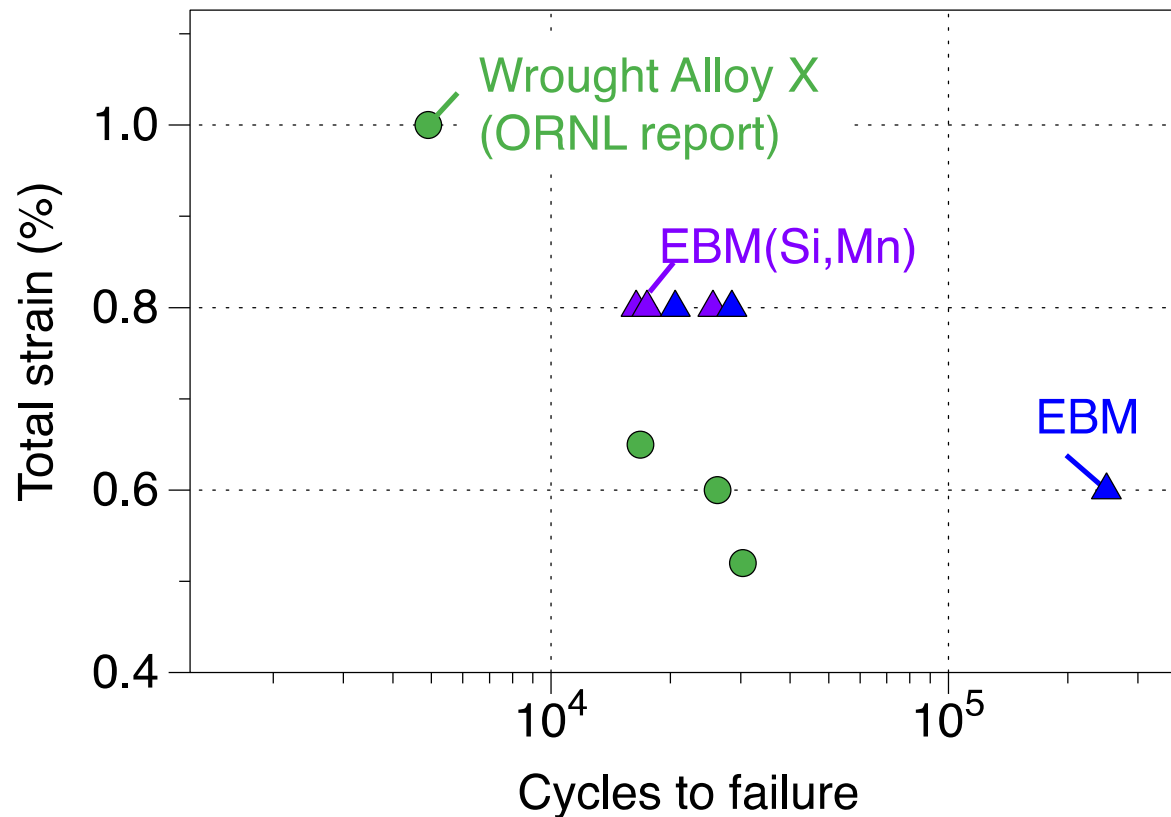


Both EBM & EBM(Si,Mn) HX Alloys Exhibit Good Ductility But Lower Strength < 800°C

Meet cast HX AMS requirement



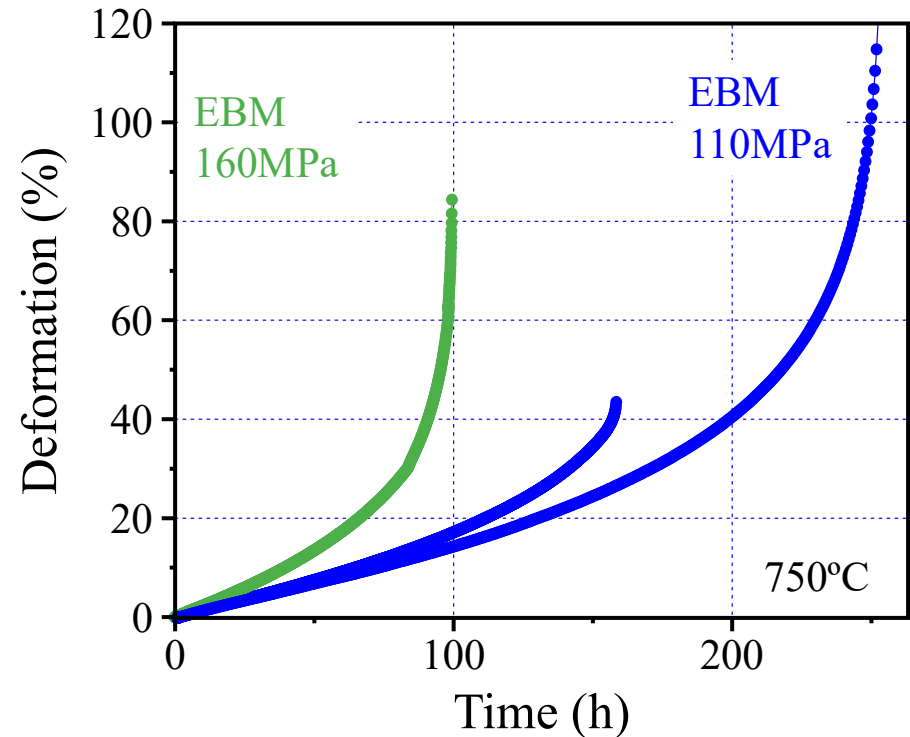
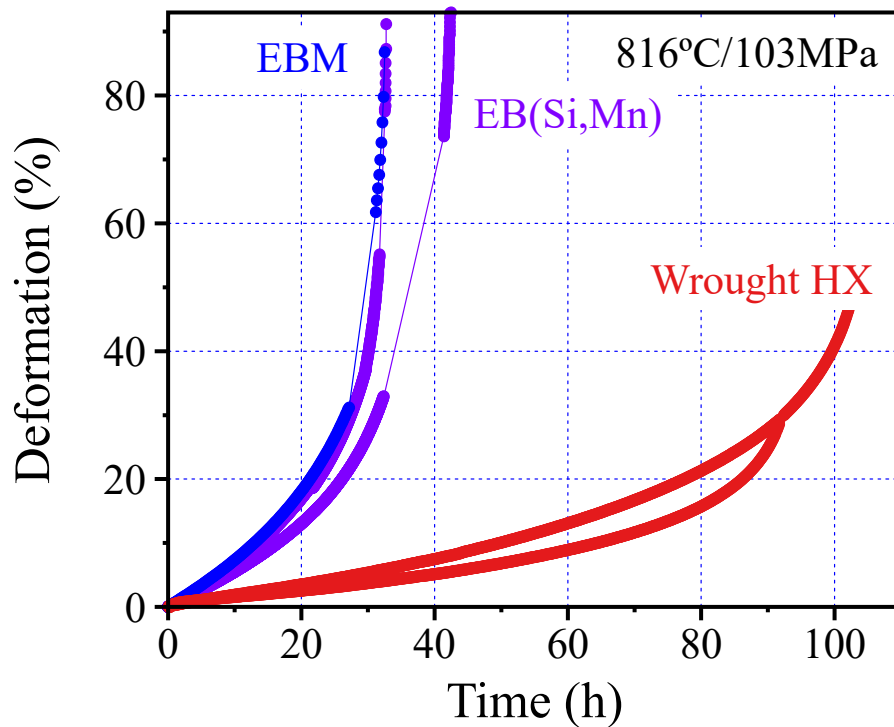
EBM & EBM(Si,Mn): Good Low-Cycle Fatigue Properties at 800°F/425°C



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

SLM testing coming soon

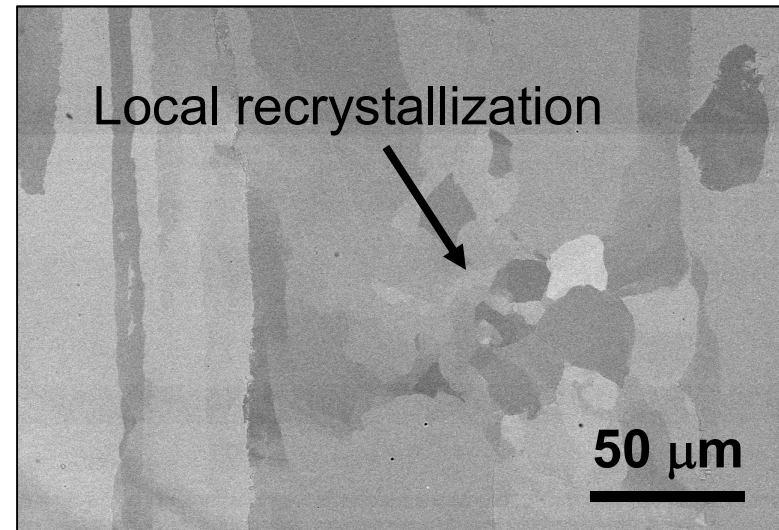
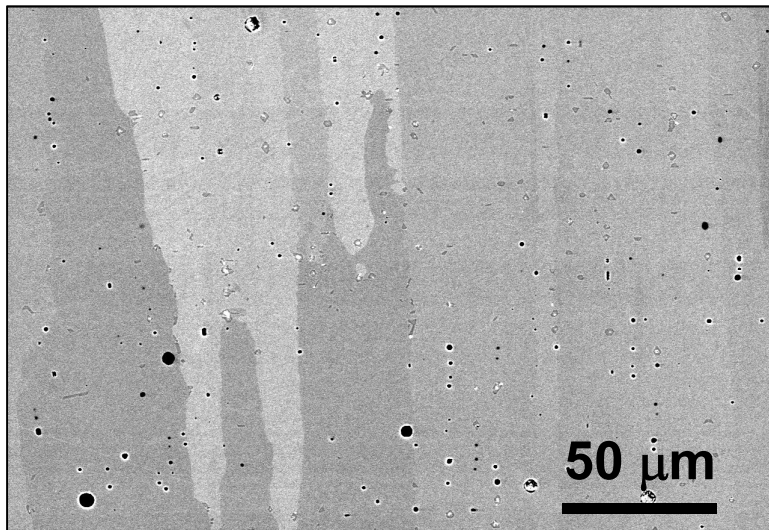
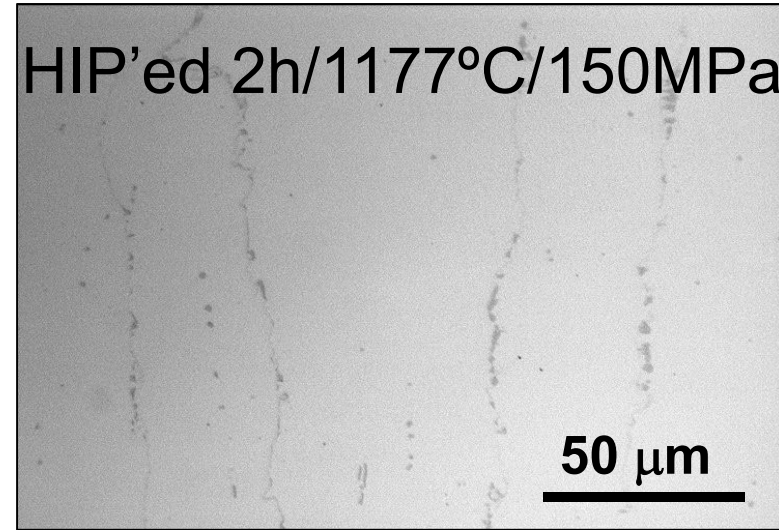
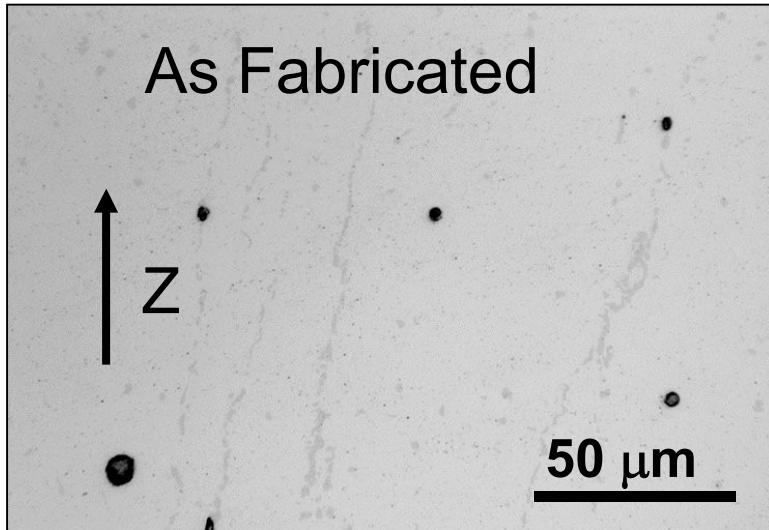
EBM & EBM(Si,Mn) = Lower Creep Strength at 750 and 816°C but High Ductility



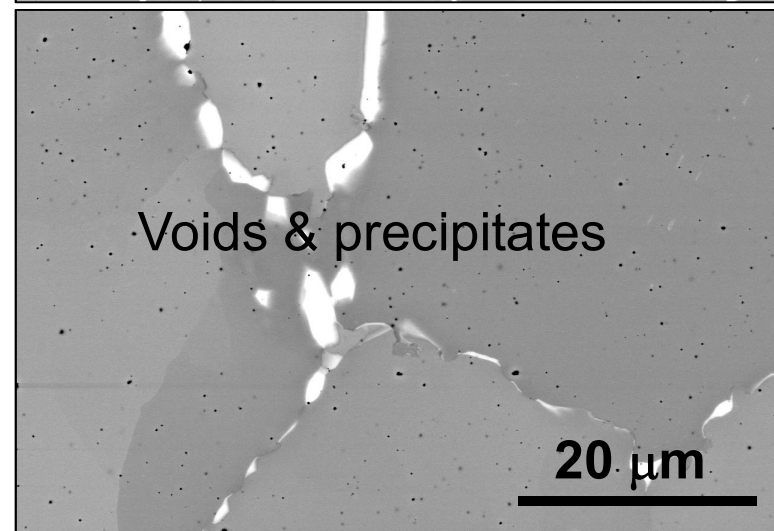
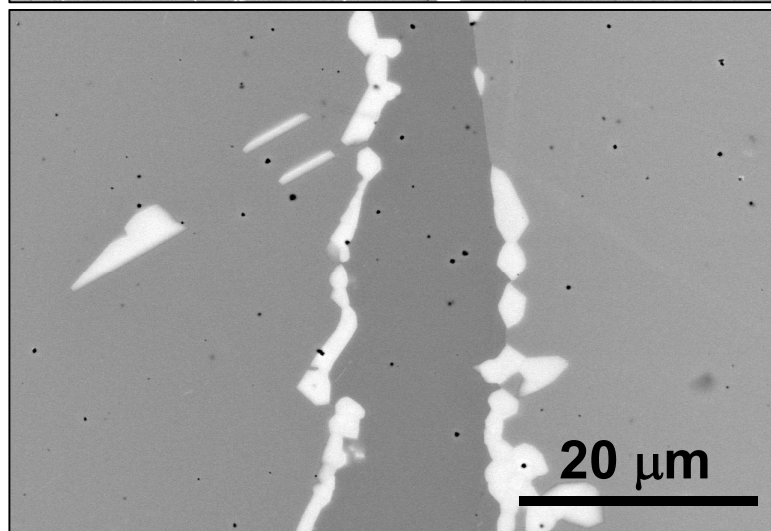
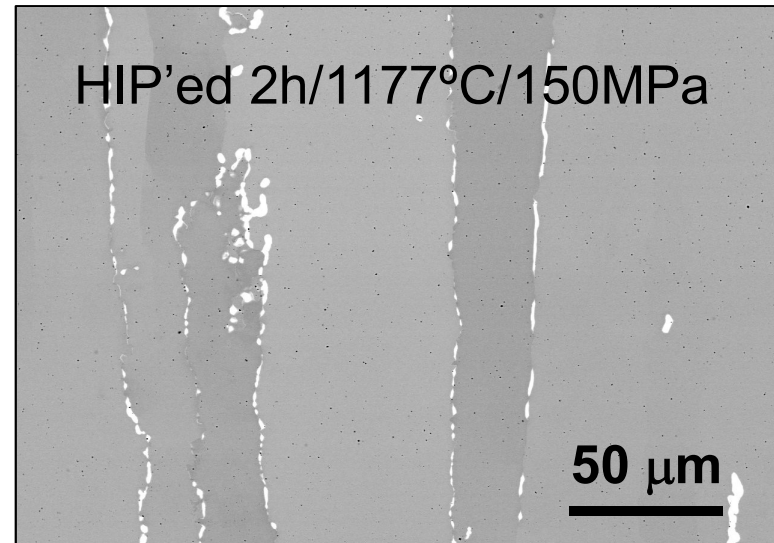
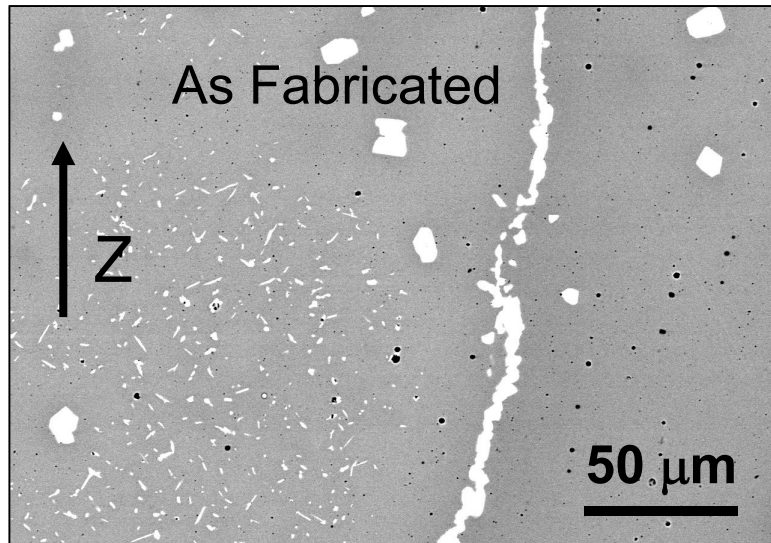
Wrought expected lifetime:

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

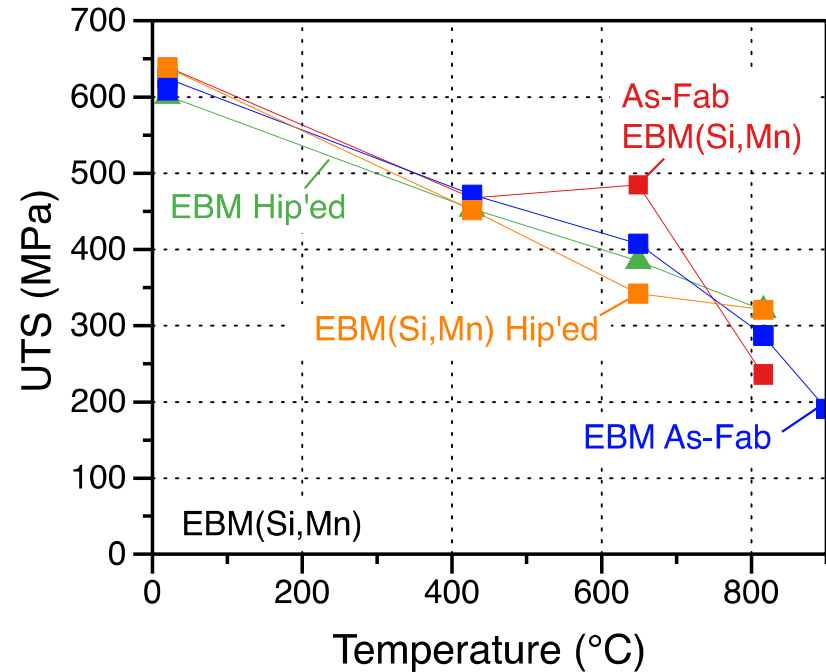
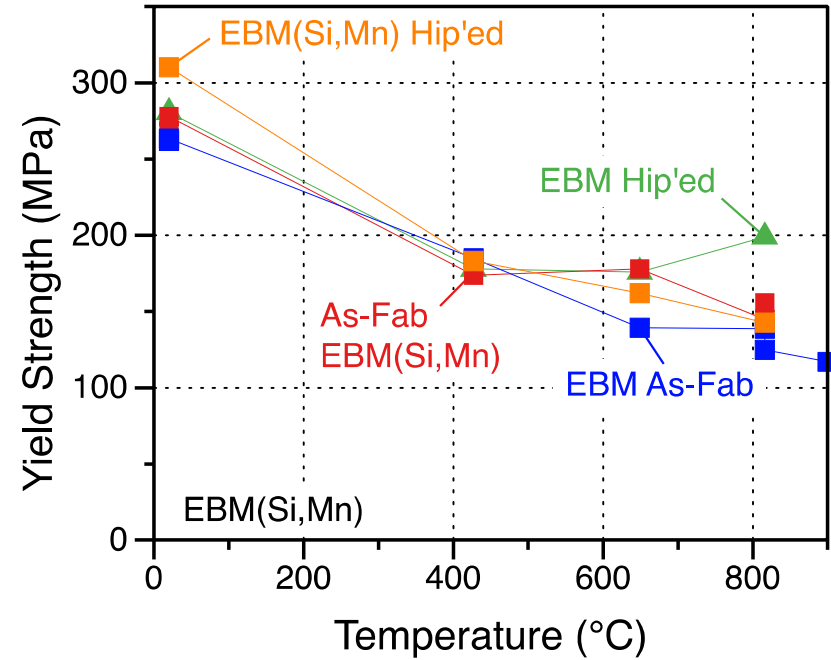
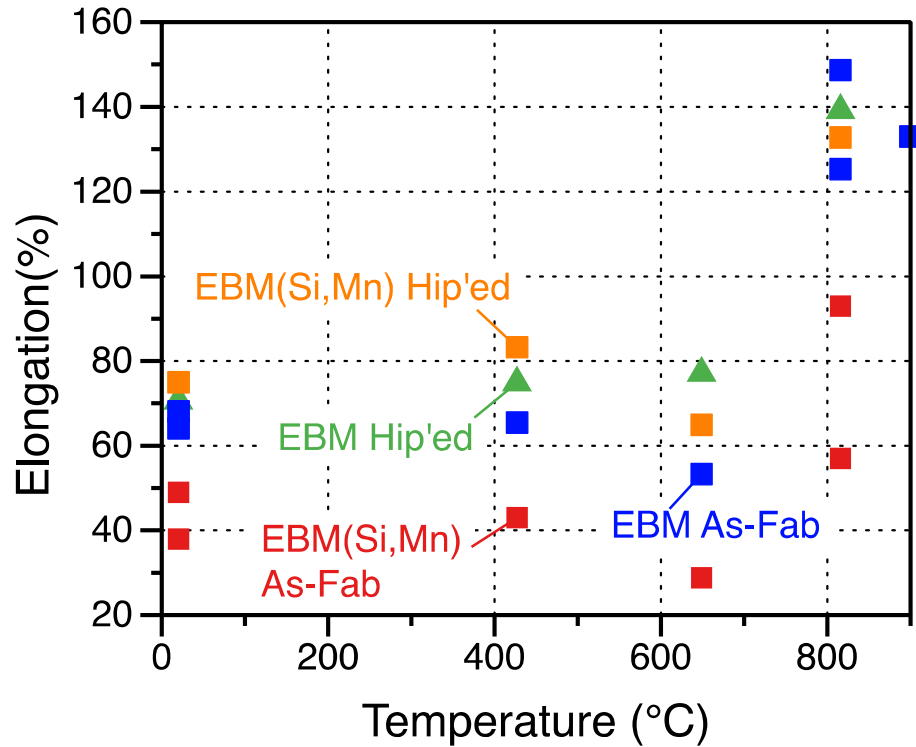
EBM: Fully Dense Material after HIP'ing at 1177°C/2h/150MPa, "Fast Cooling"



EBM(Si,Mn): Micro voids still present after HIP'ing + Precipitates at GB only

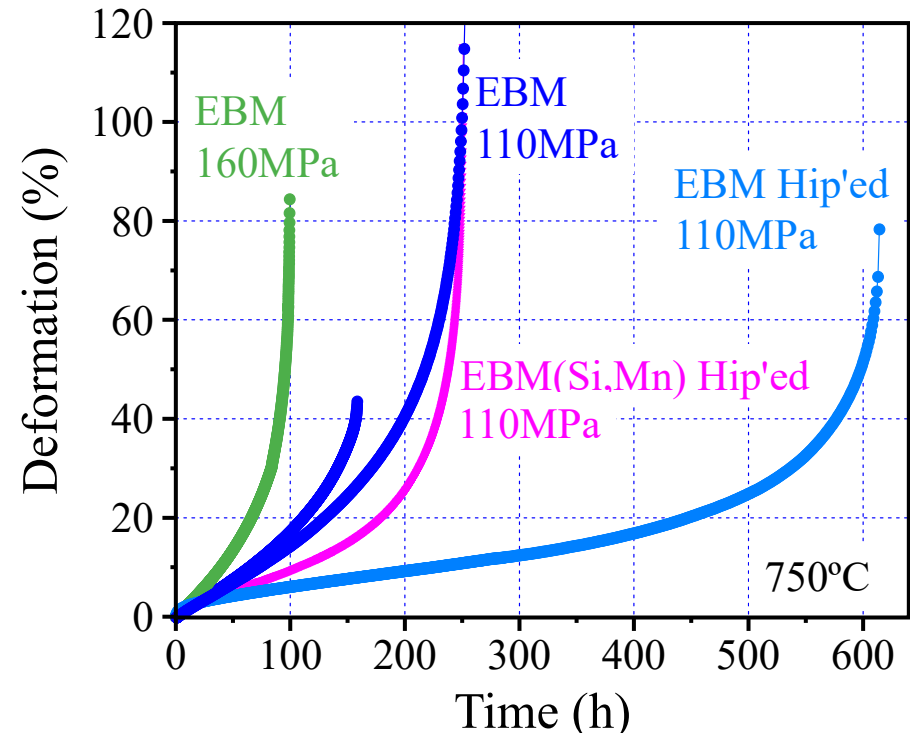
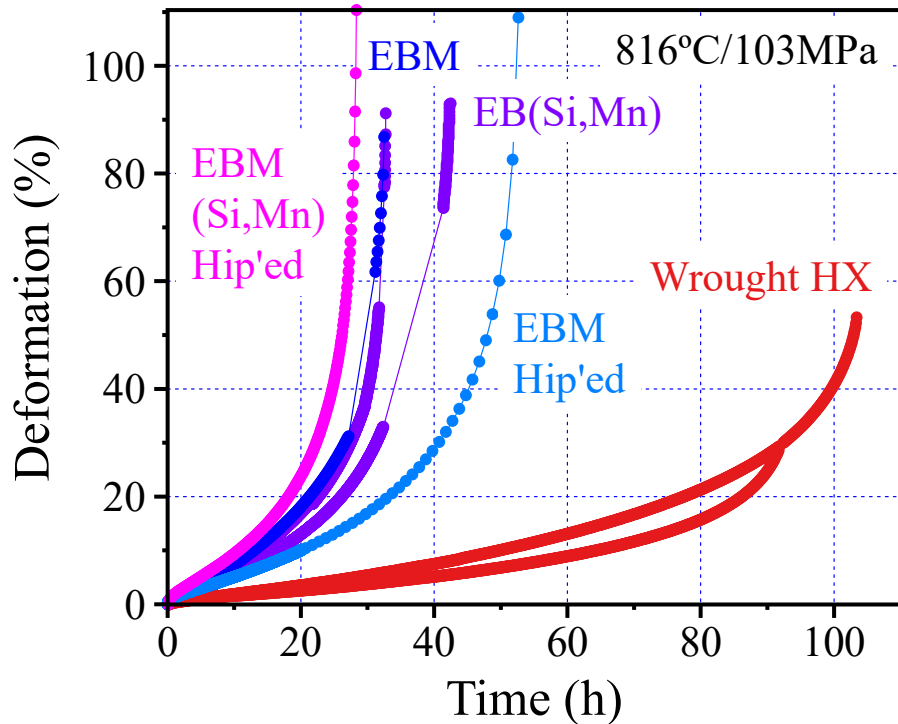


EBM: Similar Strength & Ductility After HIP'ing 2h/1177°C/150MPa



EBM & EBM(Si,Mn) = Lower Creep Strength but high ductility

Significant improvement after HIP'ing for EBM alloy at 750°C

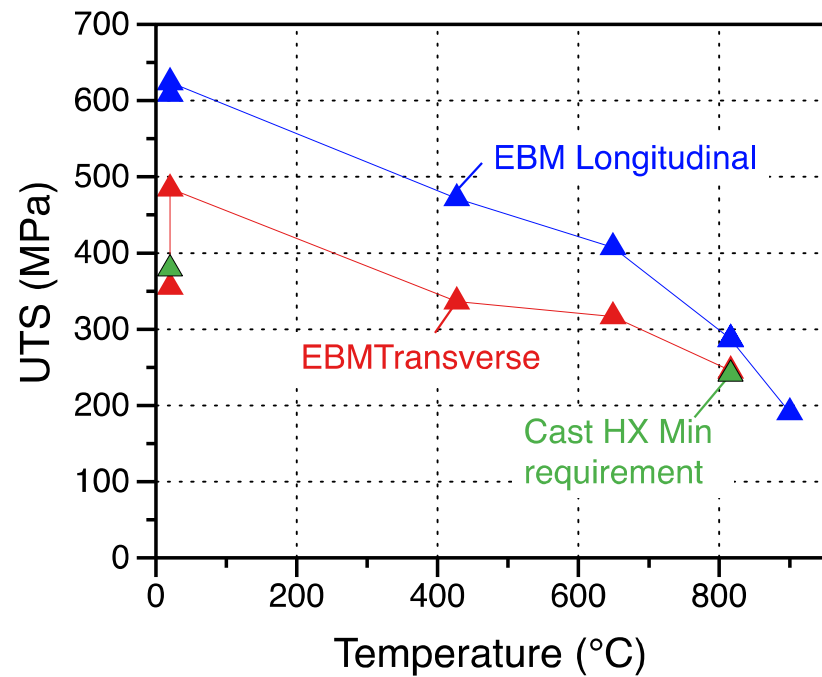
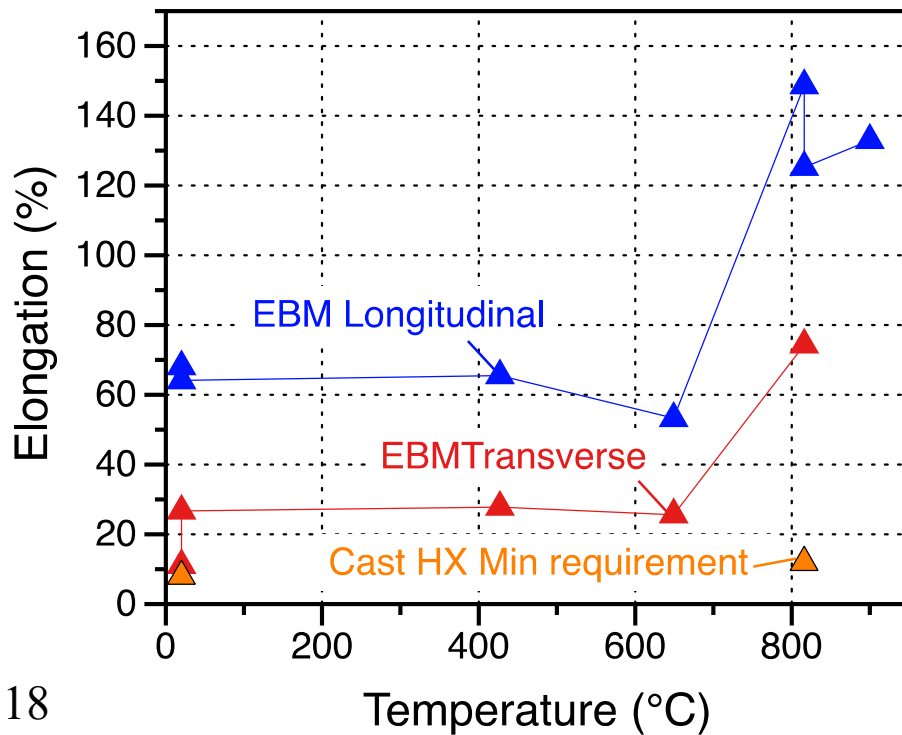
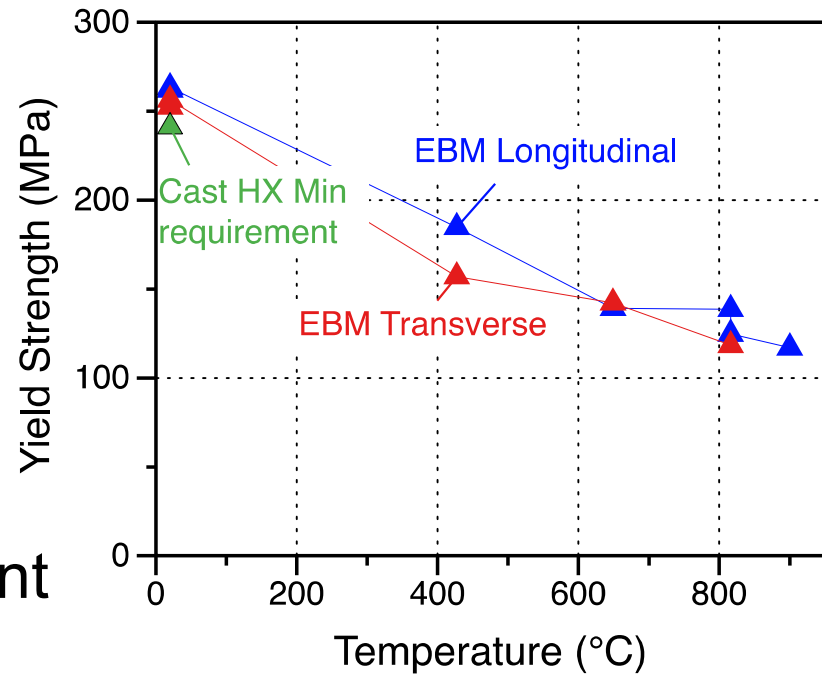


Wrought expected lifetime:

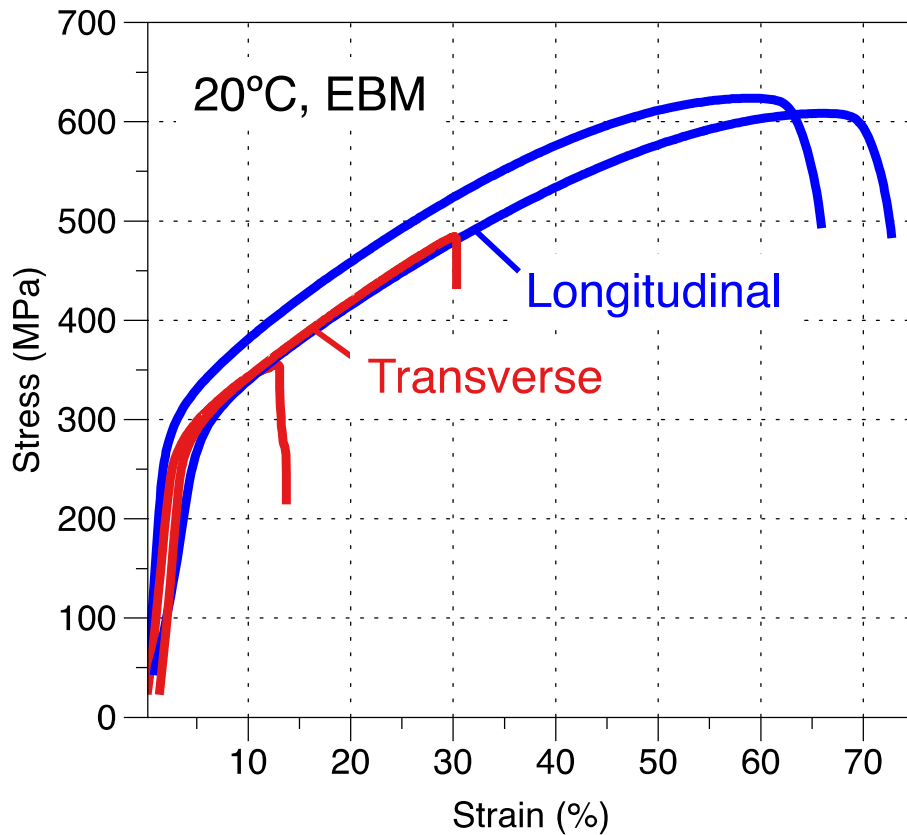
- 160MPa: 100h
- 110MPa: 1000h

EBM HX Alloys Decrease of UTS & Elongation in the Transverse Direction

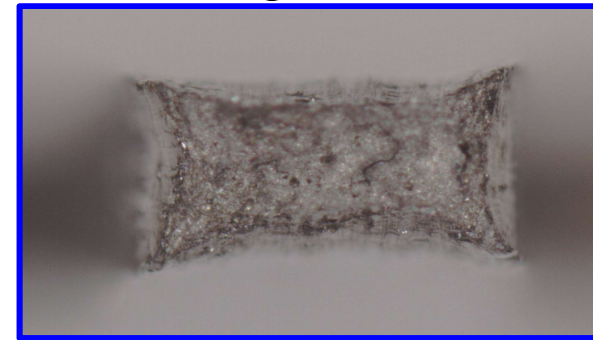
Close to cast HX AMS requirement



EBM HX Alloys Decrease of UTS and Elongation due to Microstructure Anisotropy

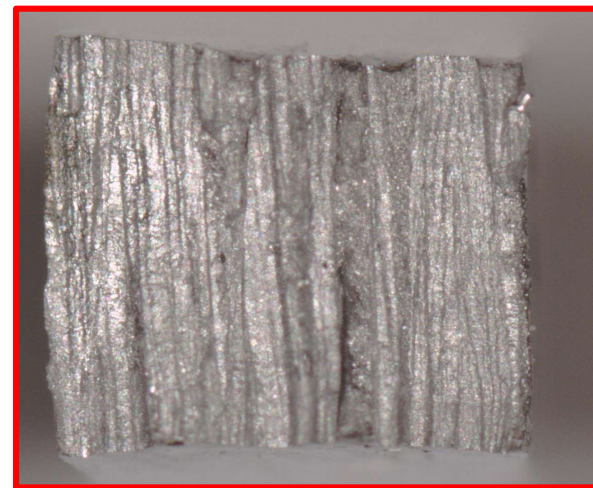


Longitudinal



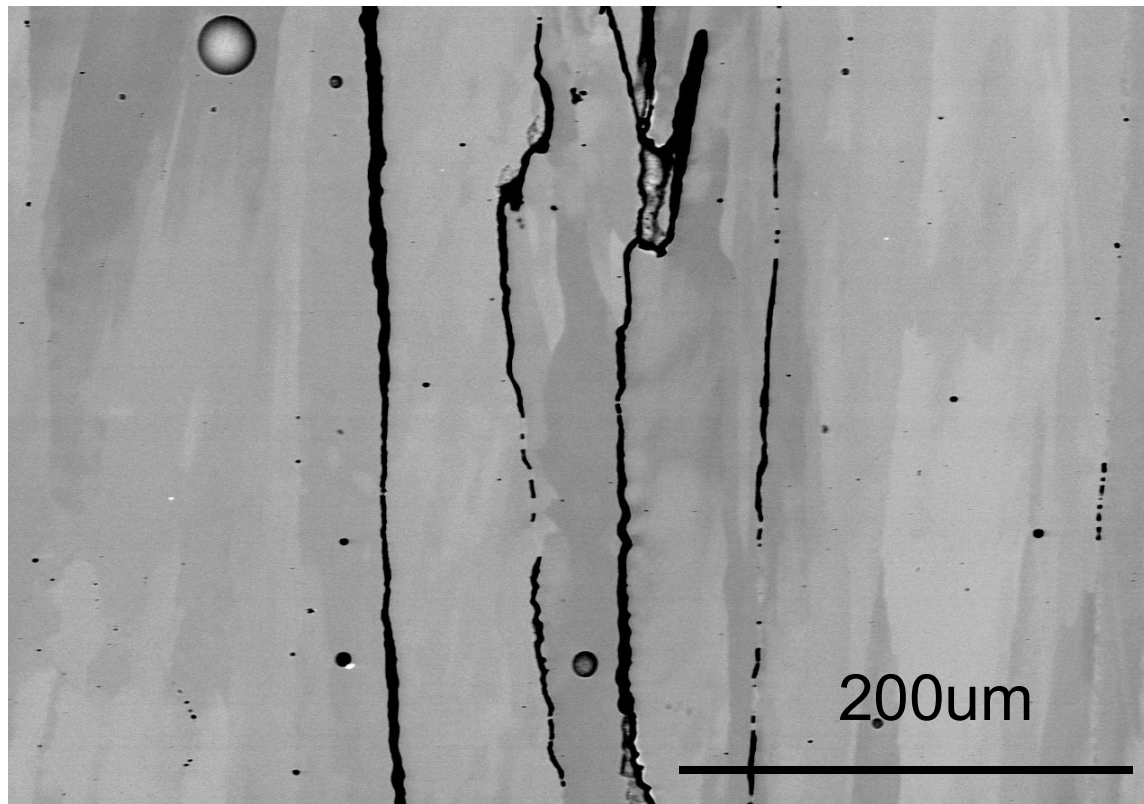
1mm

Transverse

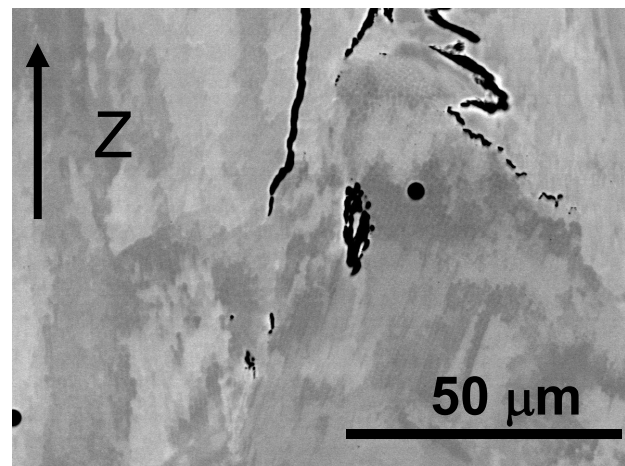
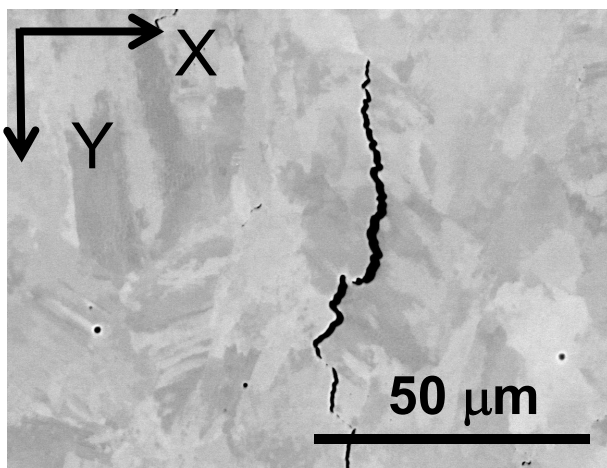
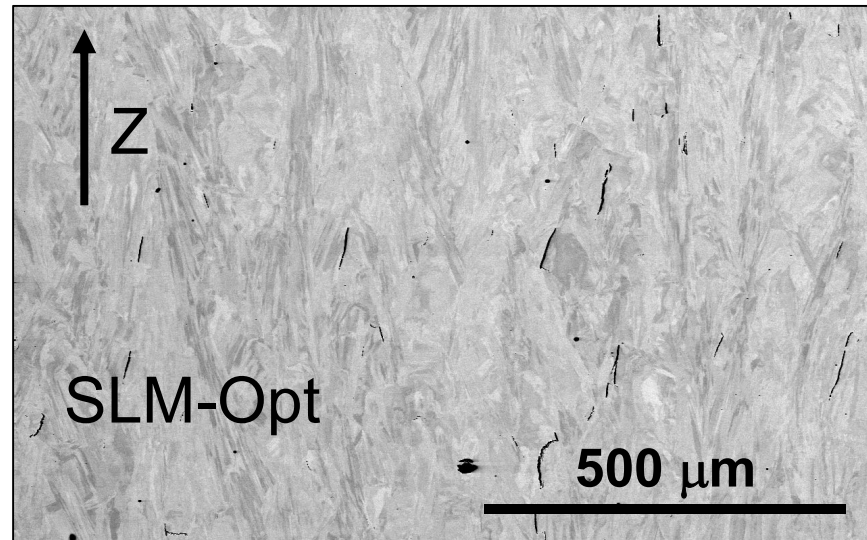


EBM HX Alloys: Need to Develop Build Parameters for a Given Composition?

Hot tearing observed for very low Si and Mn HX alloy
Opposite to what has been reported for SLM HX

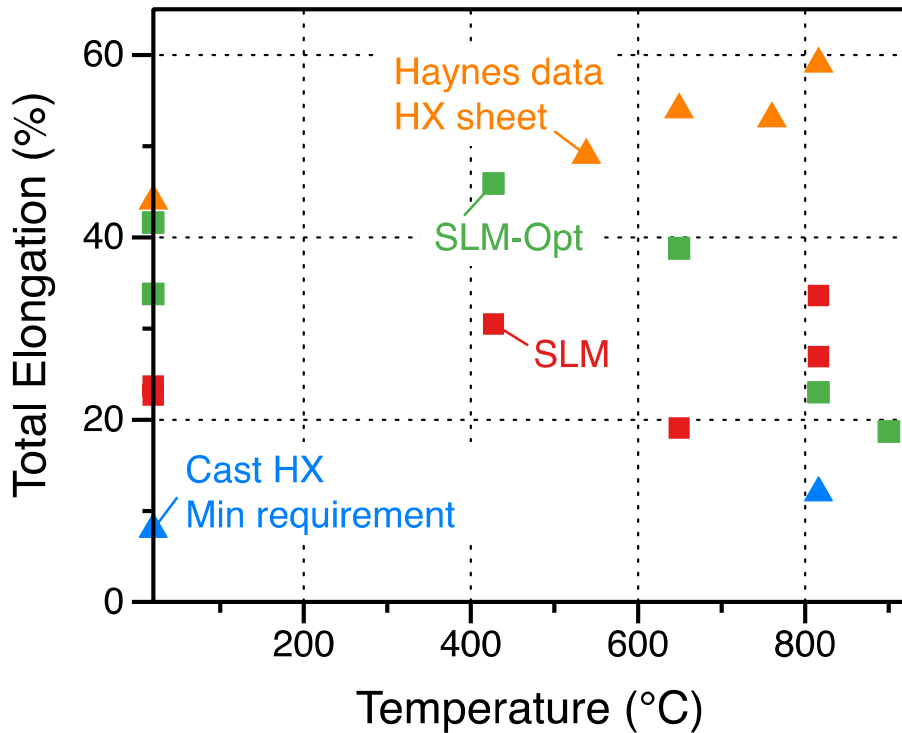


SLM-Opt: Small Grains, No Precipitate Hot Tearing Cracks

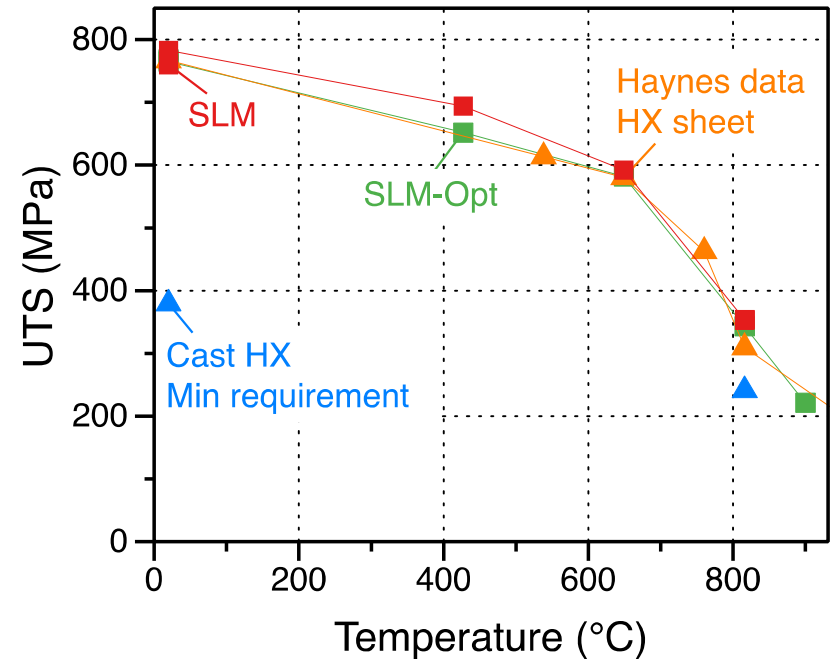
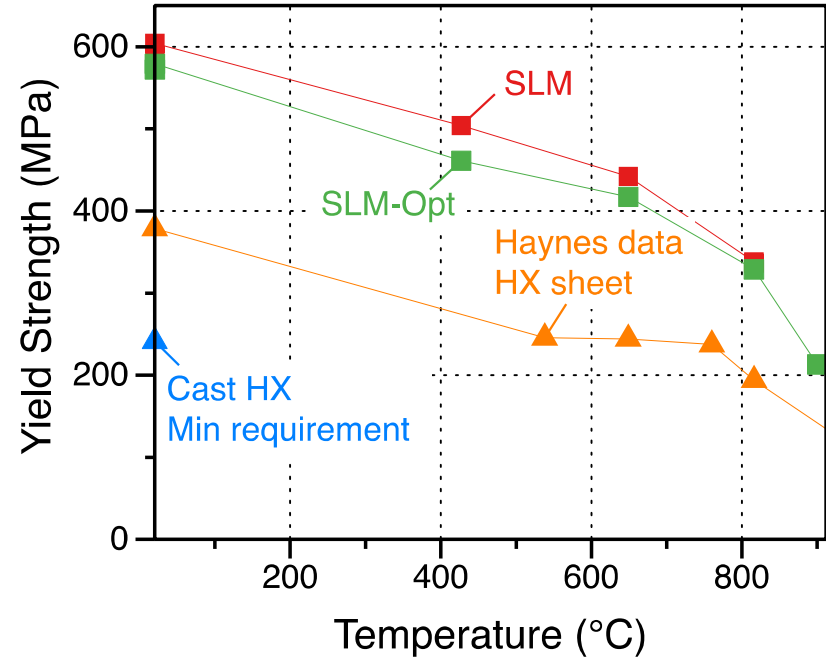


Typical of SLM microstructure
Could further optimize the SLM parameters

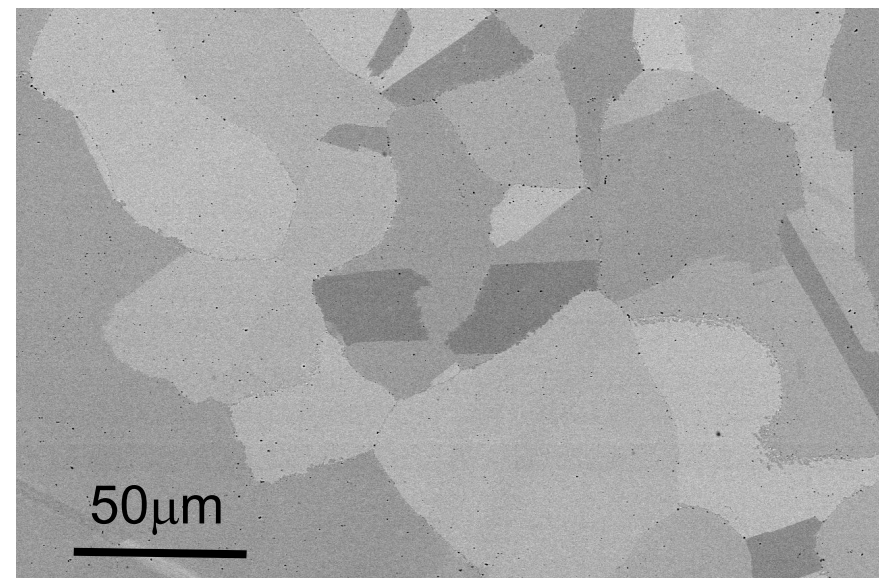
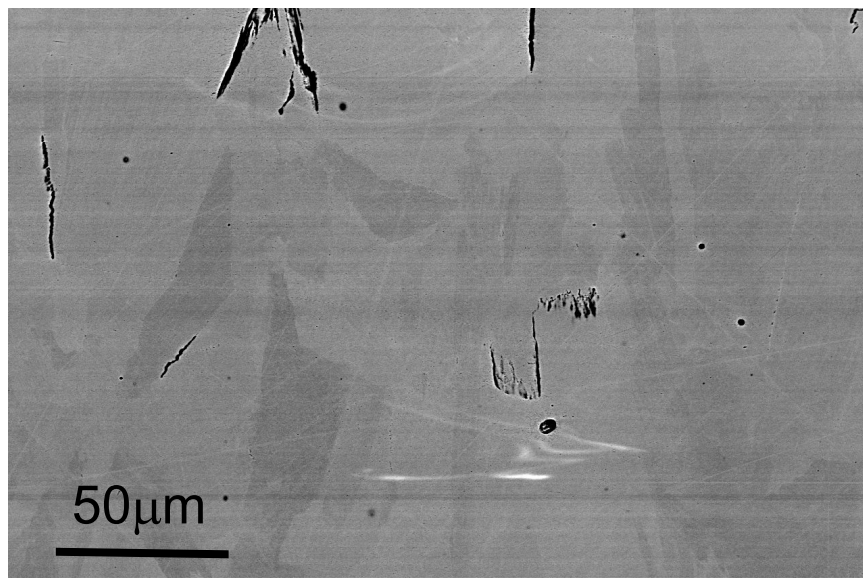
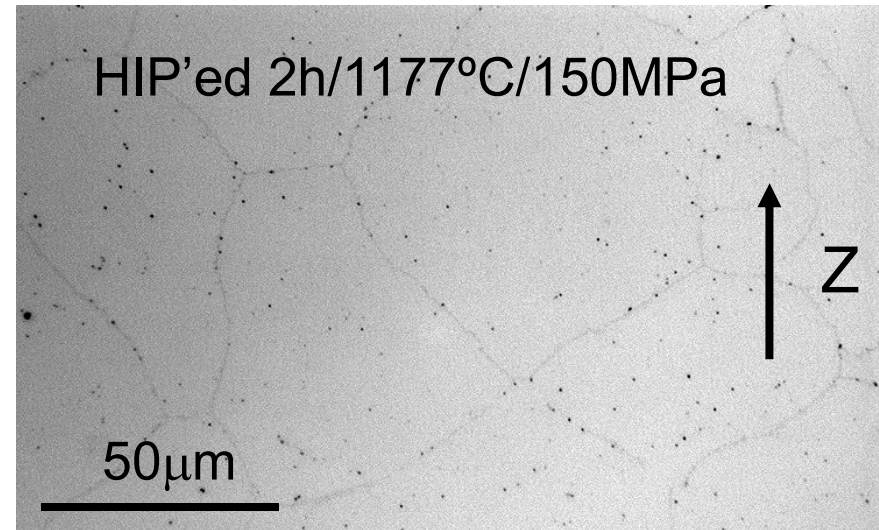
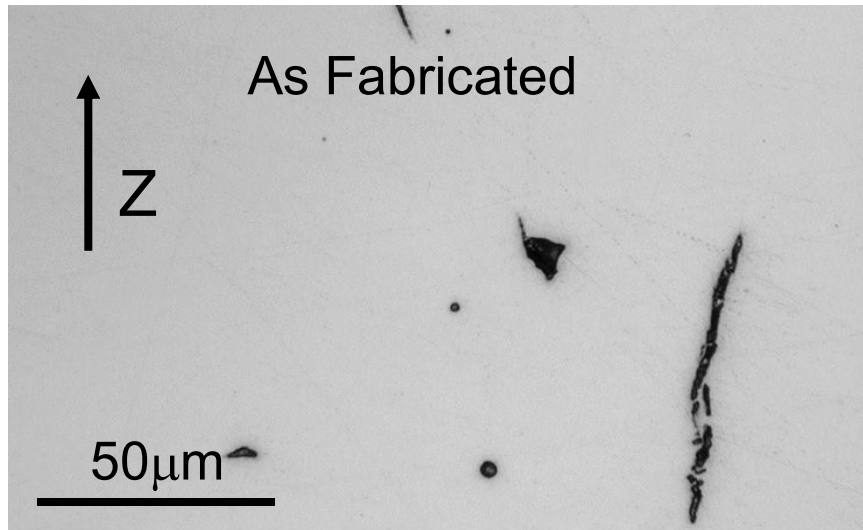
SLM HX Exhibits High Strength but Moderate Ductility



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

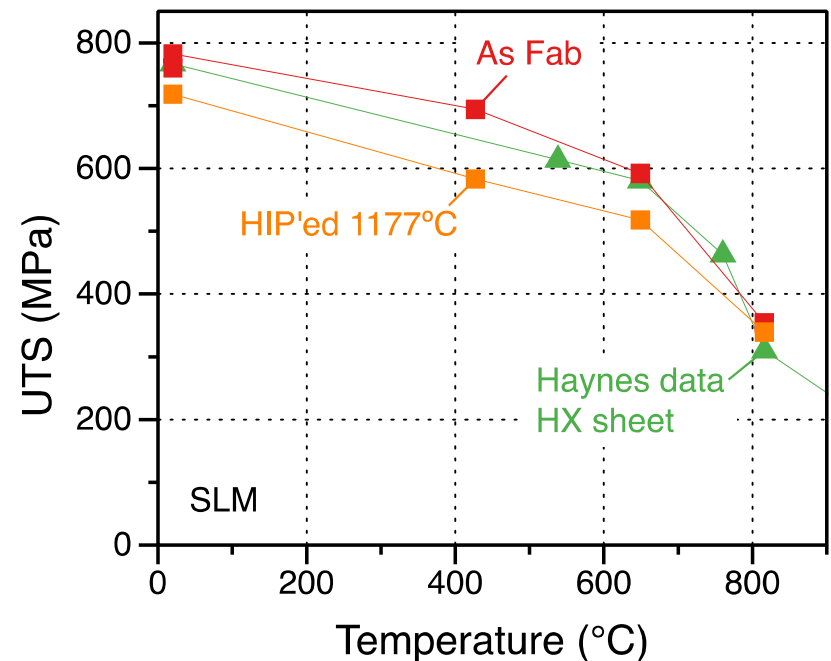
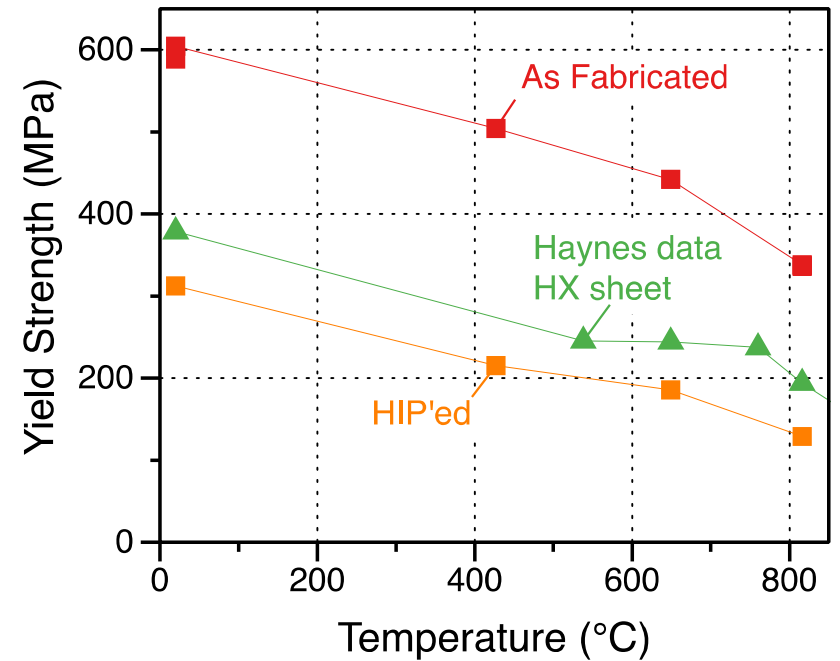
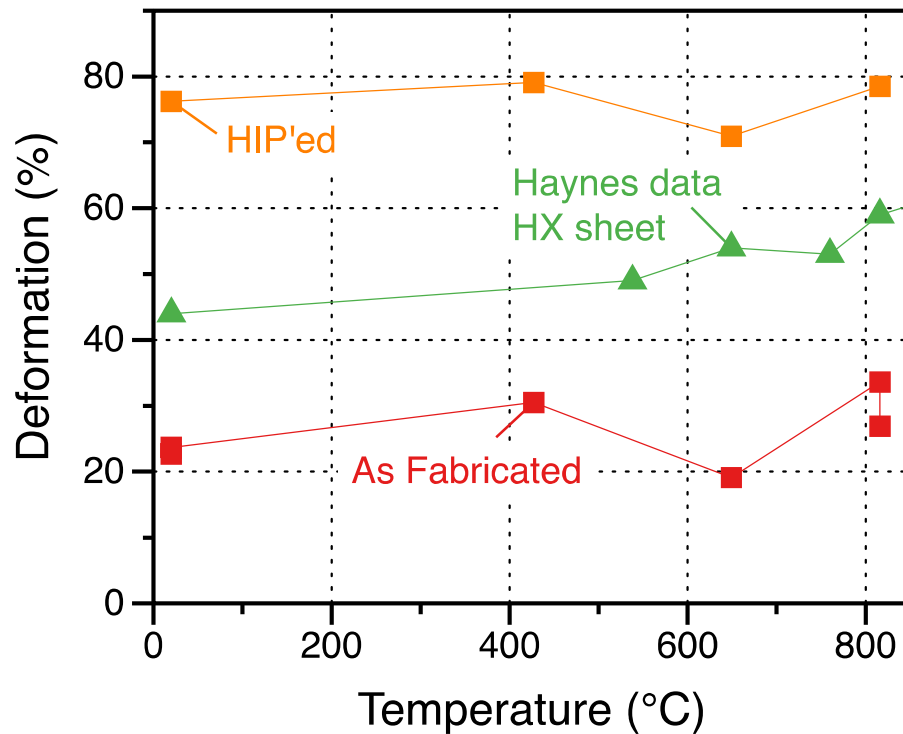


SLM: Fully Dense Material After HIP'ing at 1177°C/2h/150MPa+Recrystallization

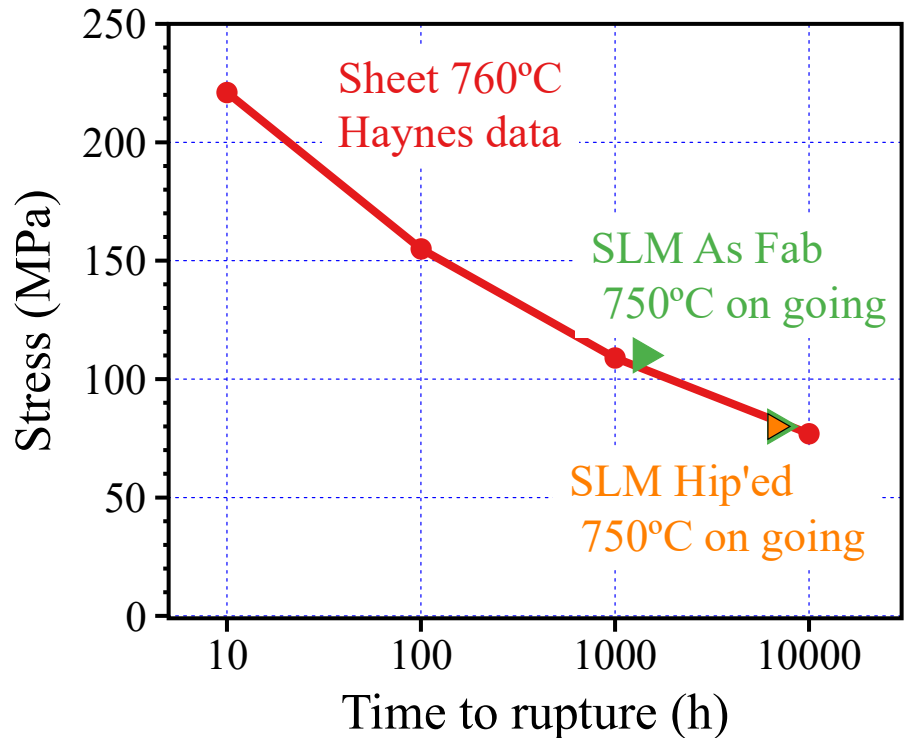
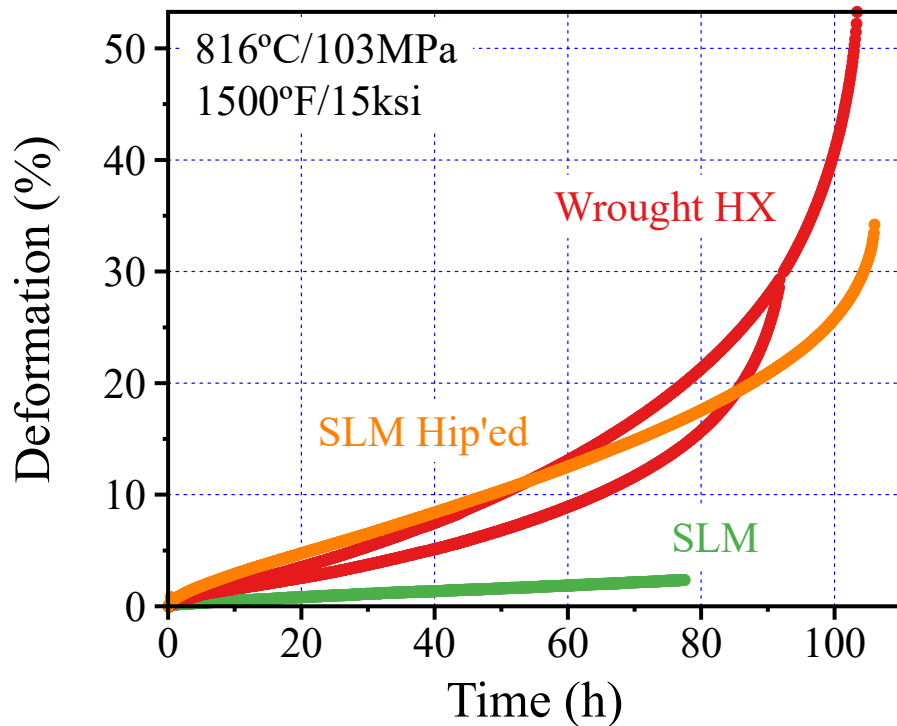


SLM: Increase of Ductility and Decrease of YS after HIP'ing 2h/1177°C/150MPa

Release of Residual Stress + Microstructure evolution



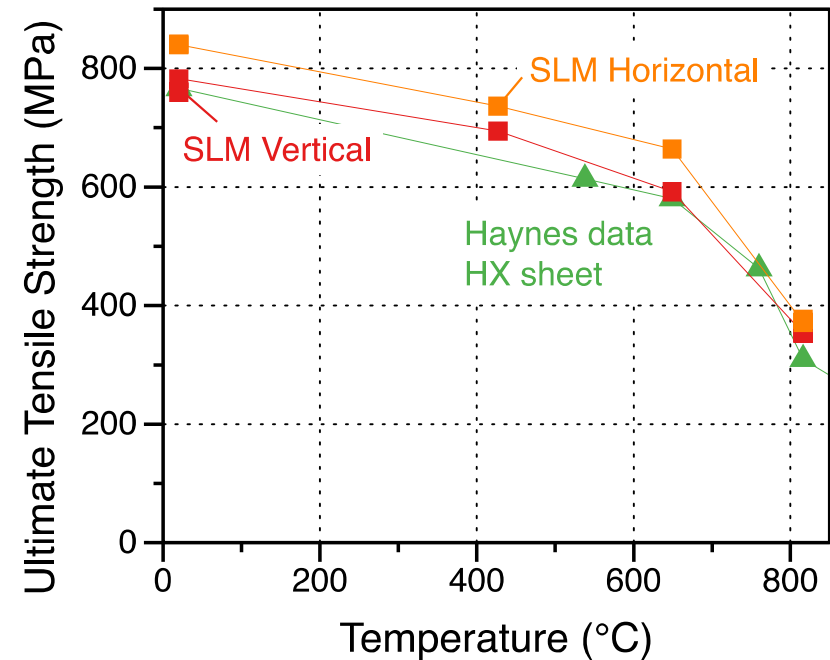
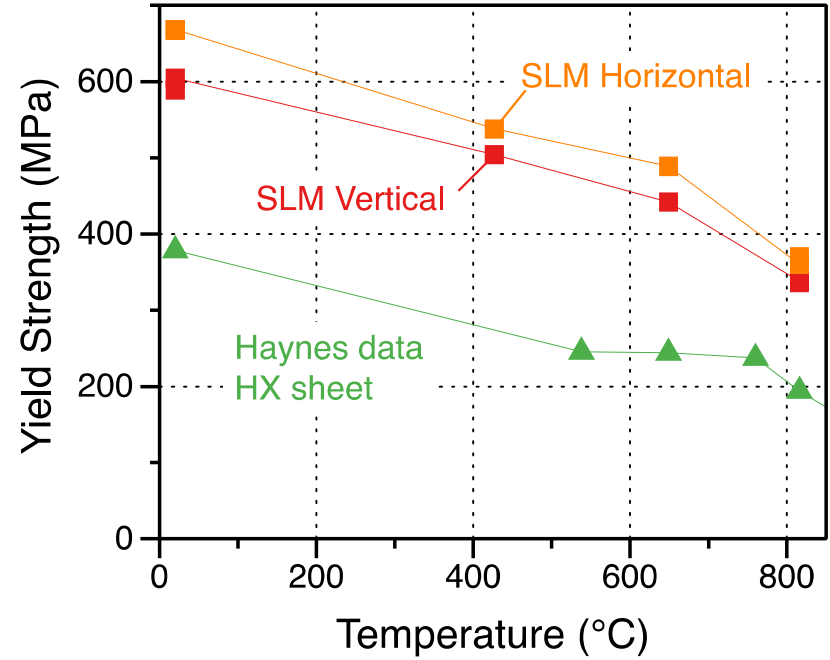
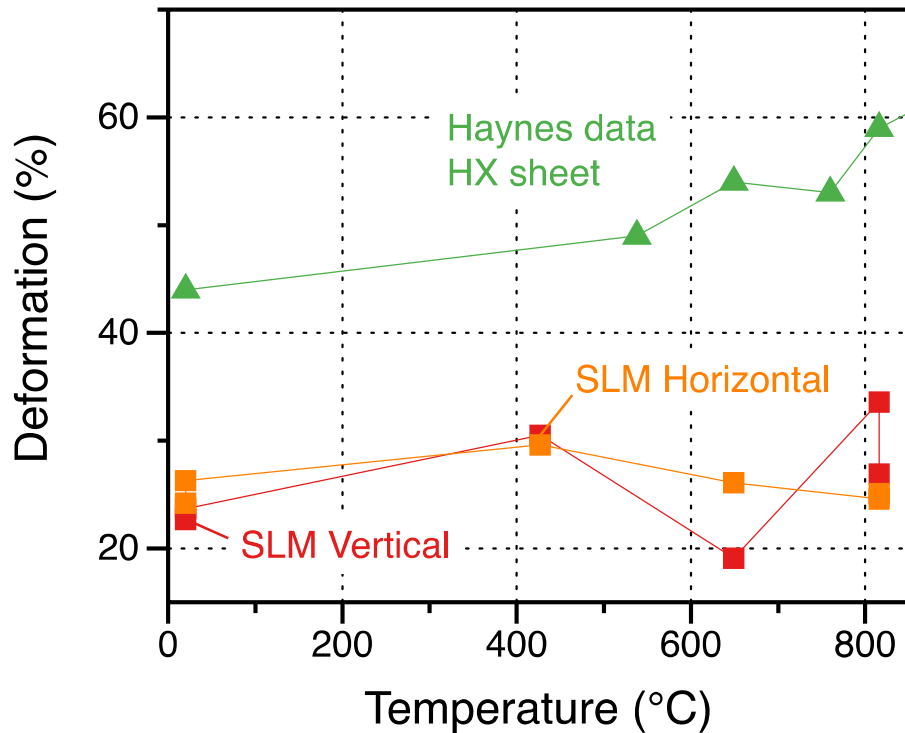
SLM: Very Good Creep Lifetime Ductility improvement after Hip'ing



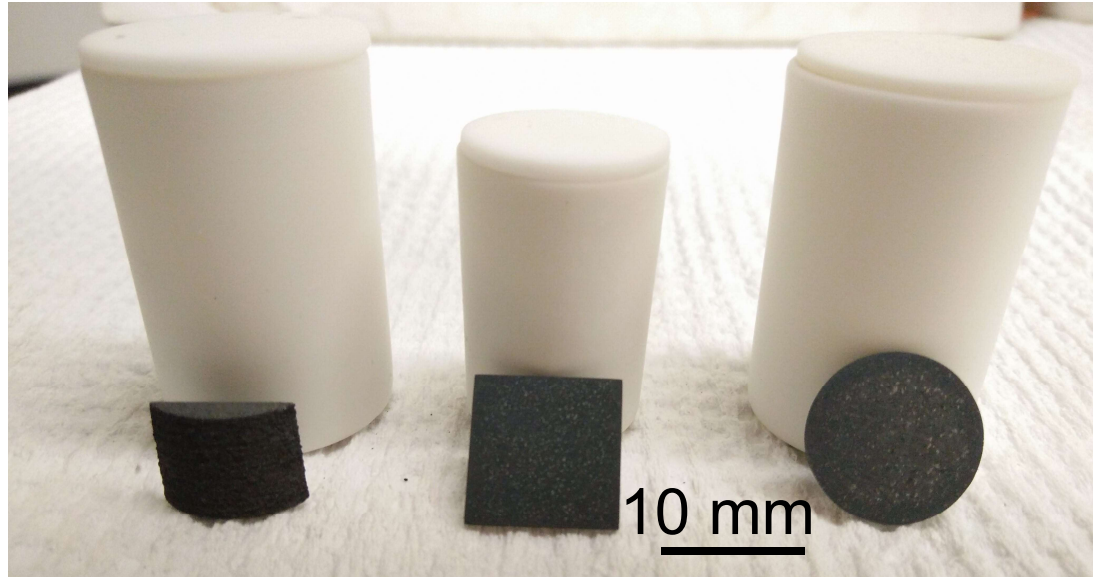
2 tests reached ~7,000h

SLM HX Alloys

Similar properties along and perpendicular the build direction

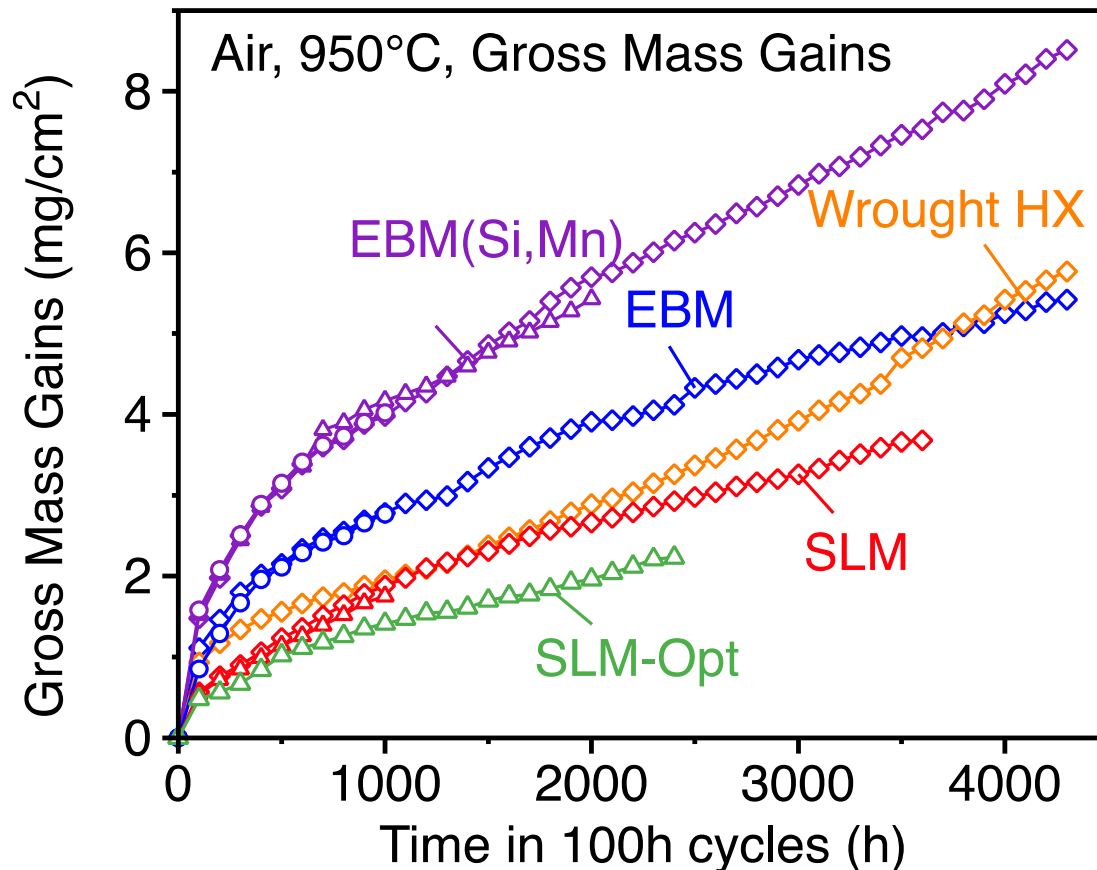


Cyclic Oxidation Testing of HX in Box Furnace at 950°C, 100h Cycles

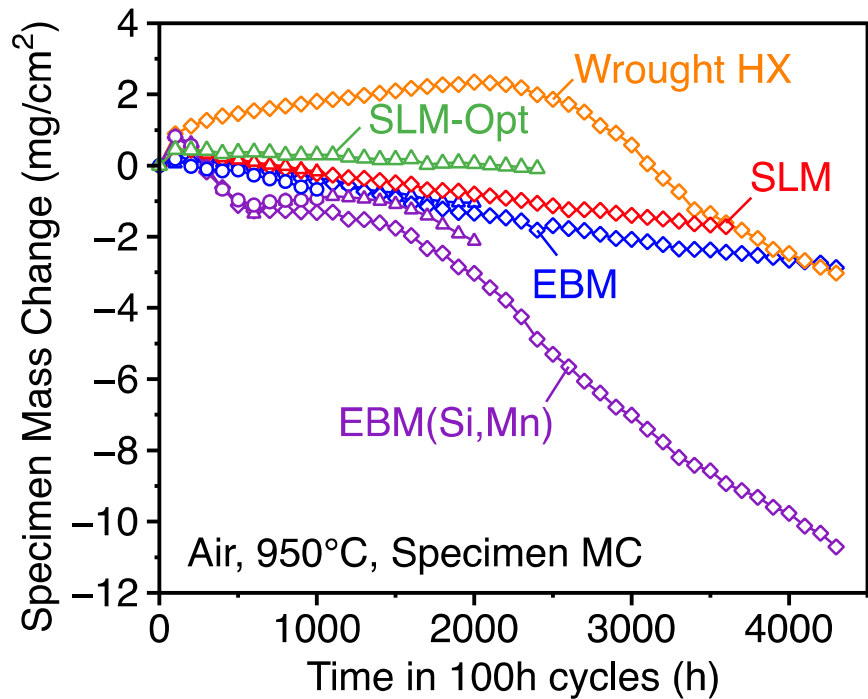
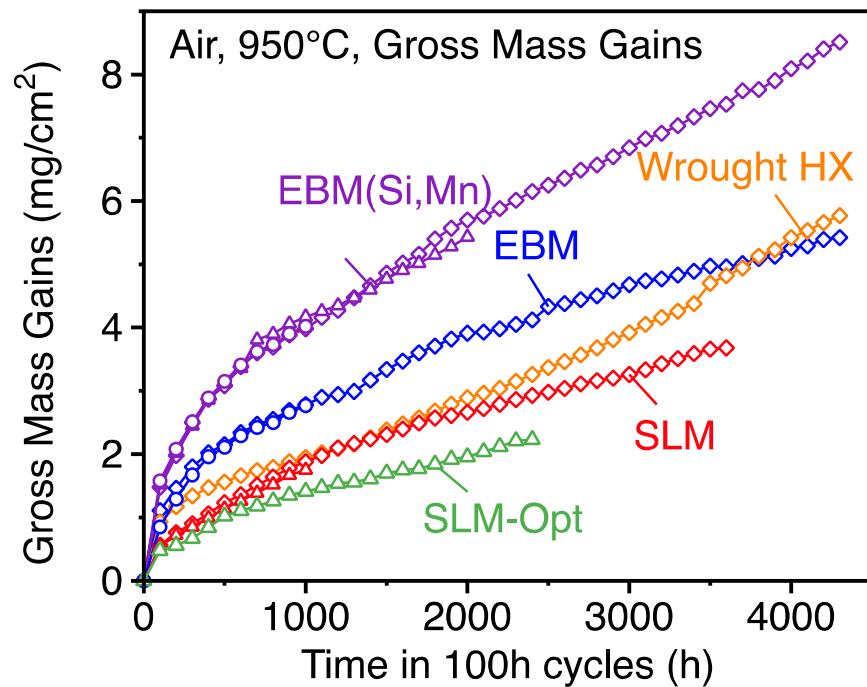


- 950°C for fast assessment of long-term oxidation behavior
- As Fabricated AM coupons. **Polished** & not polished
- Crucible to generate both specimen and gross mass gains
- Gross mass gains = oxygen pickup ~ Metal (Cr) loss
- Surface imaging using Keyence 3D microscope

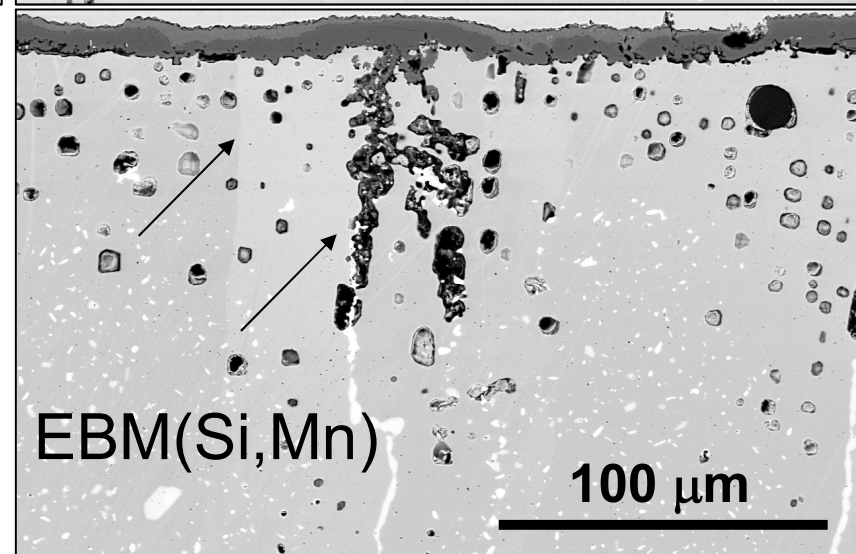
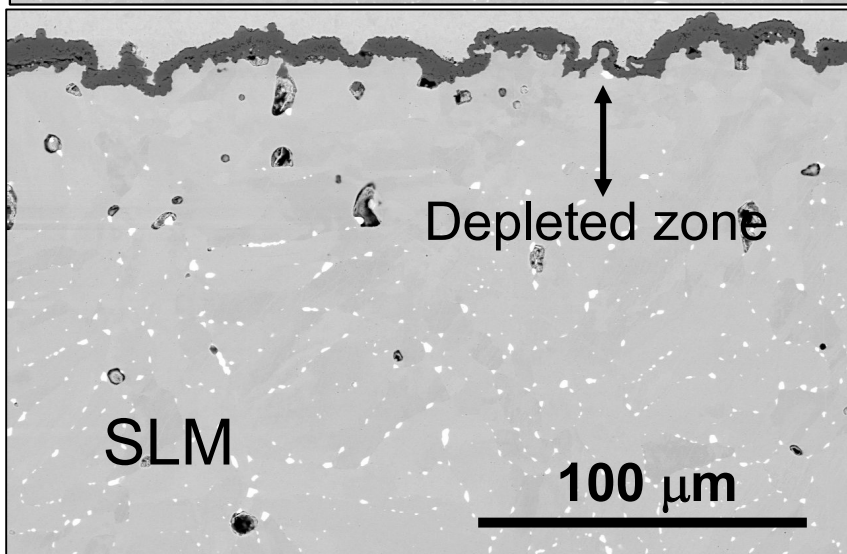
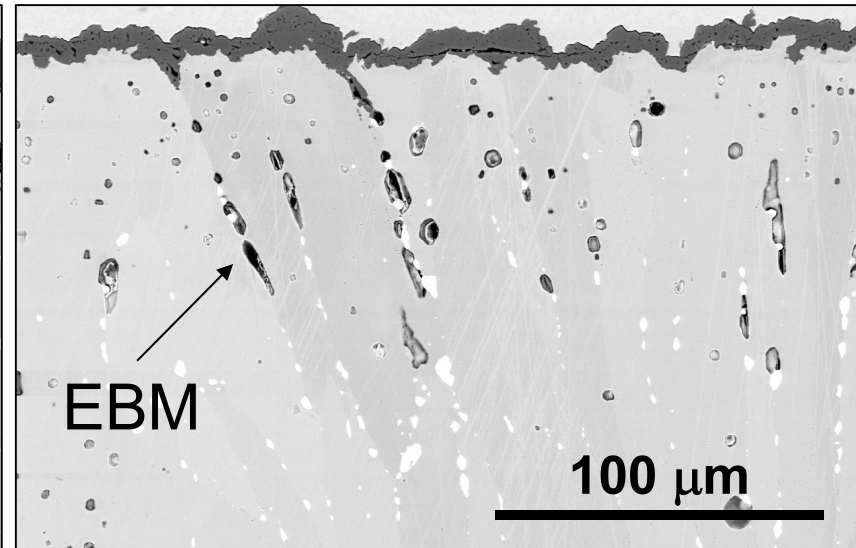
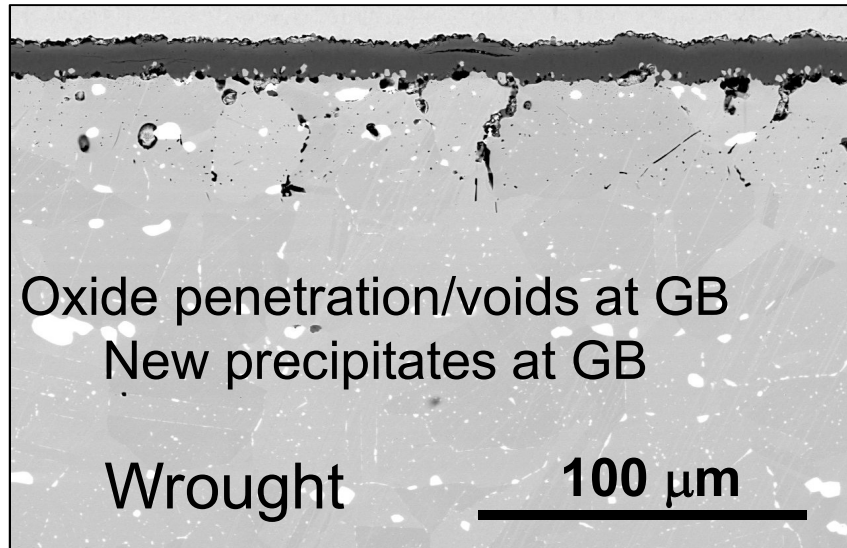
Faster Oxidation Rate for EBM(Si,Mn) Similar Rate for EBM and Wrought HX Lower GMG for SLM specimens



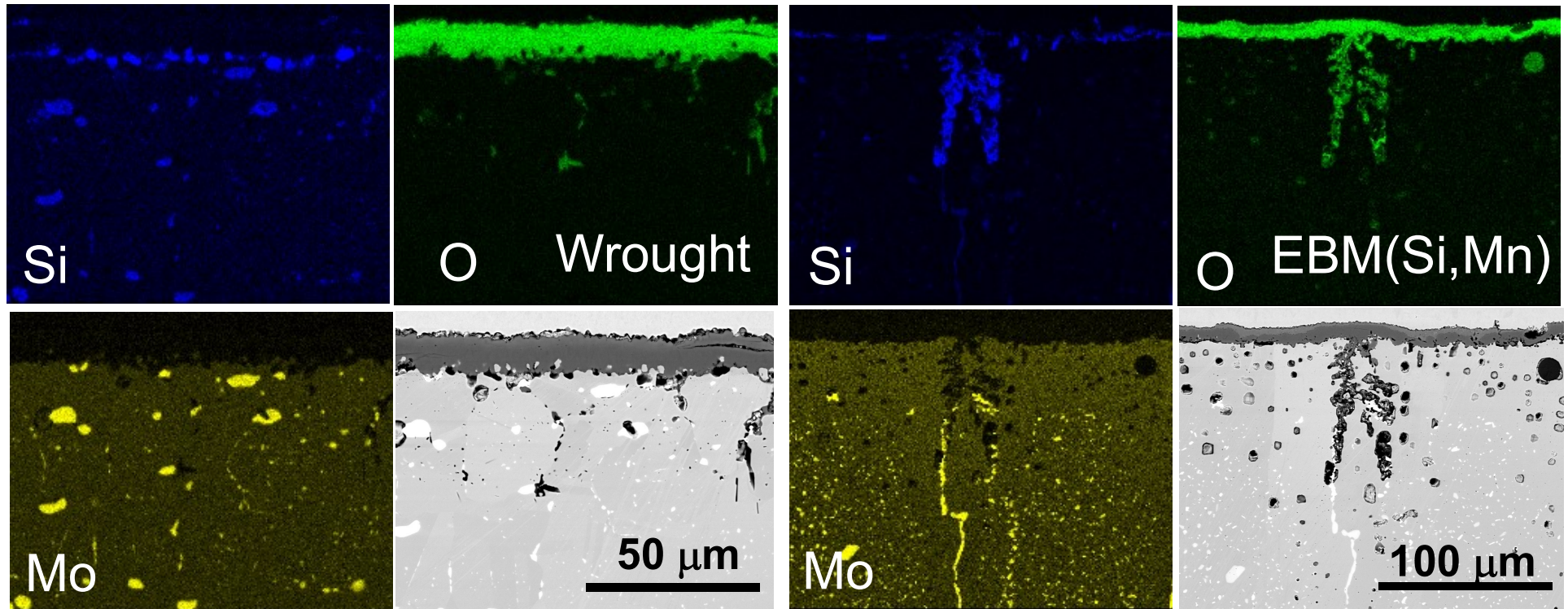
High Spallation for EBM(Si,Mn). Moderate Spallation for SLM & EBM. Increasing Spallation for Wrought HX



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



1000h, 950°C, Wrought and EBM(Si,Mn): Si segregation at Interface + Voids formation related to Mo evaporation

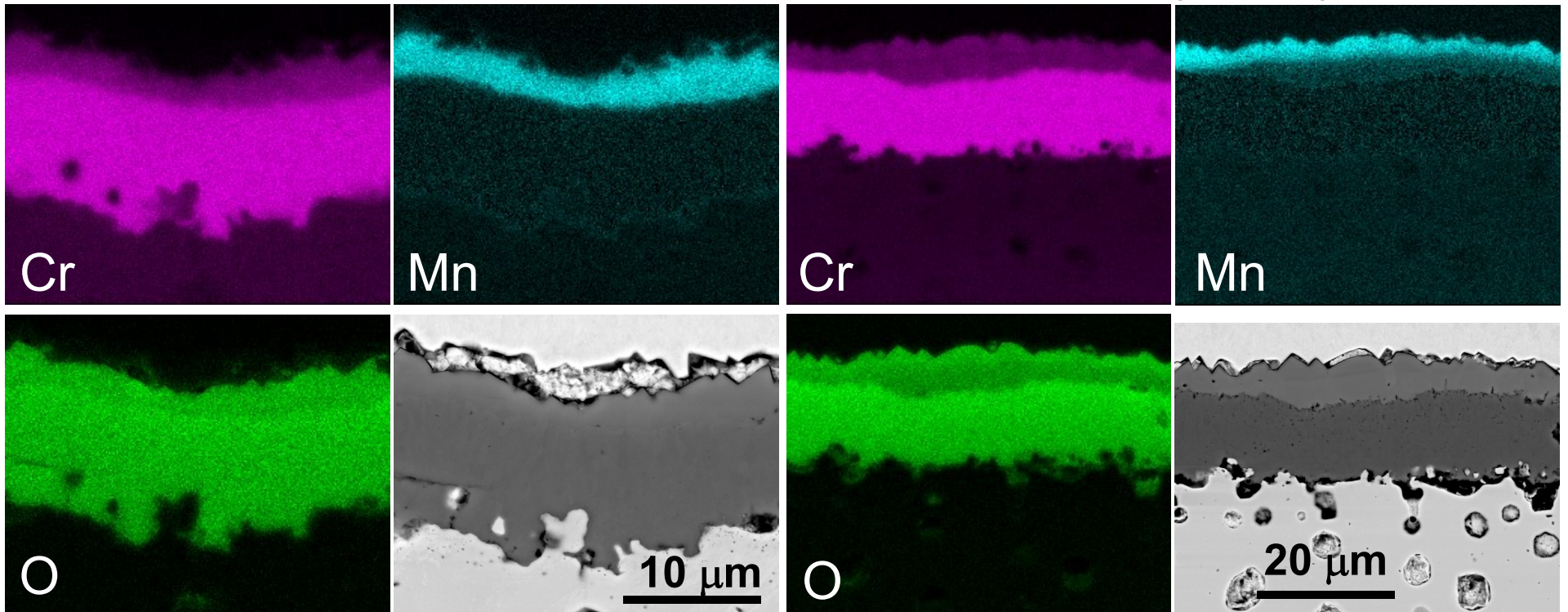


Cr-rich oxide + numerous voids at GB
Mo depleted zone for EBM(Si,Mn)

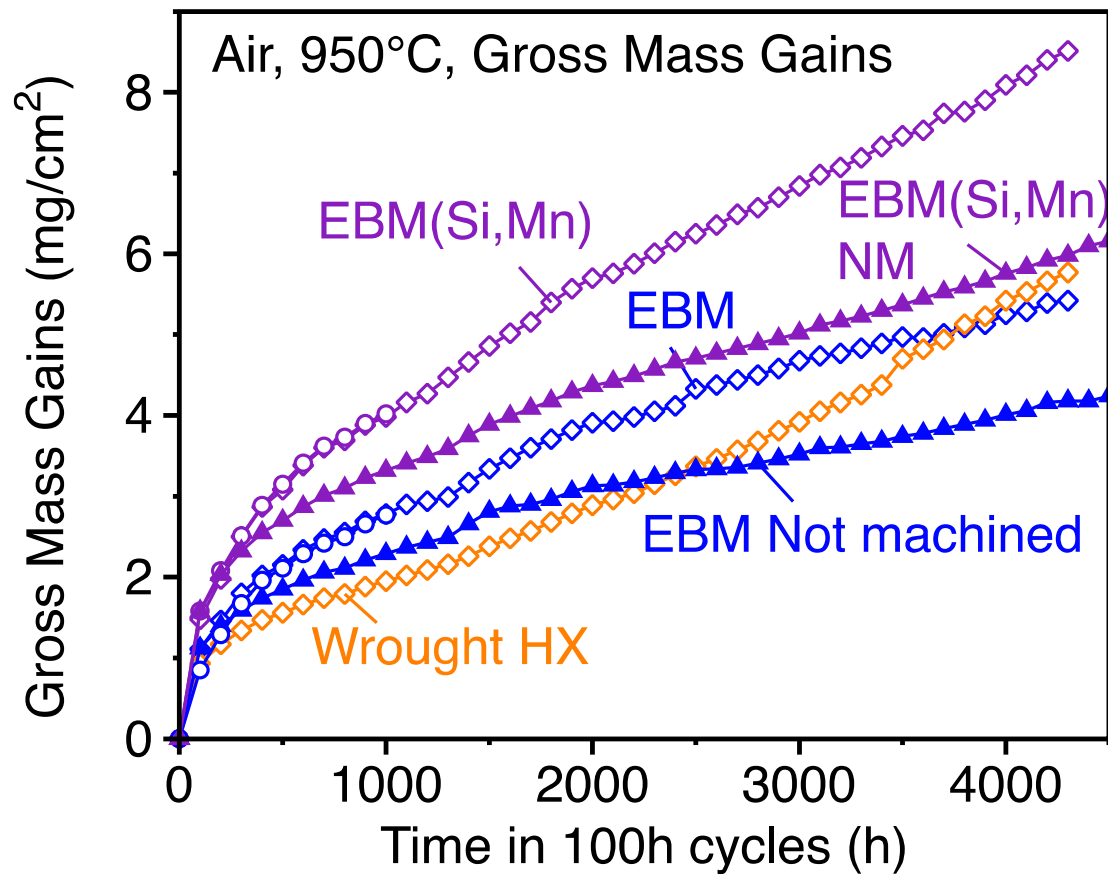
1000h, 950°C, Wrought & EBM(Si,Mn): (Cr,Mn) and Cr-Rich Layers

Wrought

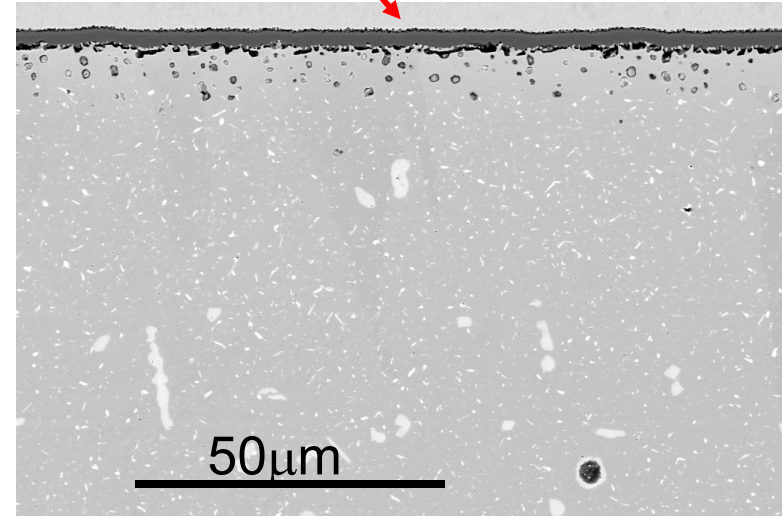
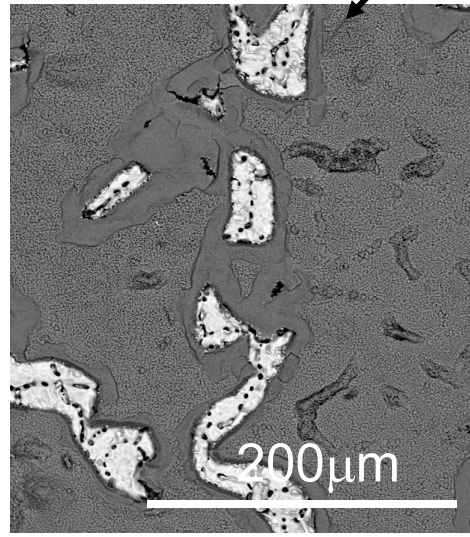
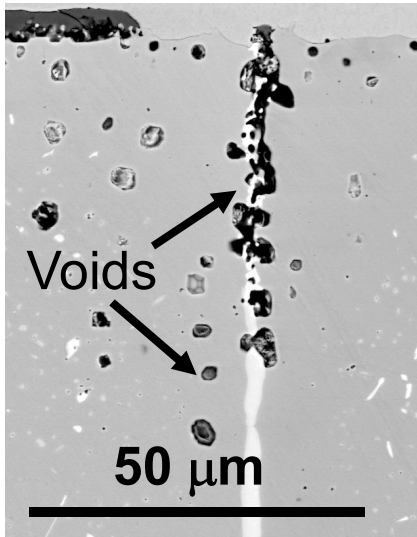
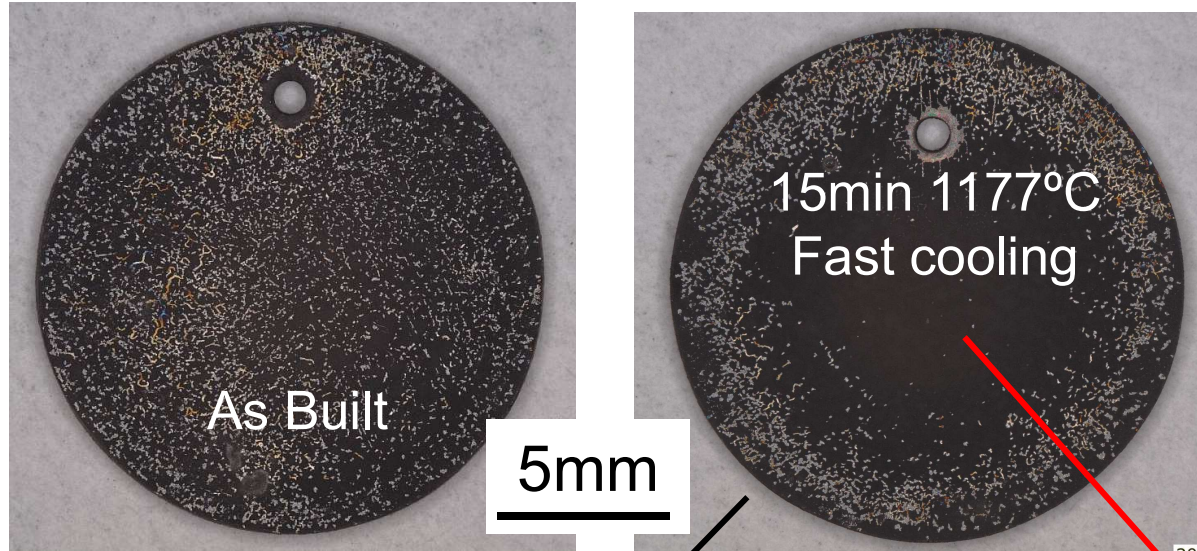
EBM(Si,Mn)



Better Oxidation Behavior for the EBM Specimen not Fully Machined



Locally Less spallation after 15min 1177°C Fast Cooling for EBM(Si,Mn)



Conclusion

- EBM HX shows good ductility and tensile strength superior to the cast HX requirement. Anisotropy might be an issue
- Good fatigue properties for the EBM HX but lower creep strength. Sufficient for some applications using cast HX?
- Hot tearing led to crack formation in SLM as observed by others. Could further optimize fabrication parameters and/or alloy composition
- SLM HX exhibited good tensile strength and acceptable ductility. Good creep strength but limited ductility. Great ductility improved after Hip'ing
- Great potential for improvement: chemistry, deposition parameters , post treatment...for properties superior to wrought HX?