



HITACHI

DOE Nuclear Energy Enabling Technologies (NEET)

Environmental Cracking and Irradiation Resistant Stainless Steel by Additive Manufacturing

Xiaoyuan Lou, GE Global Research
Frederick List, Oak Ridge National Laboratory
Gary Was, University of Michigan
Myles Connor, GE-Hitachi Nuclear Energy

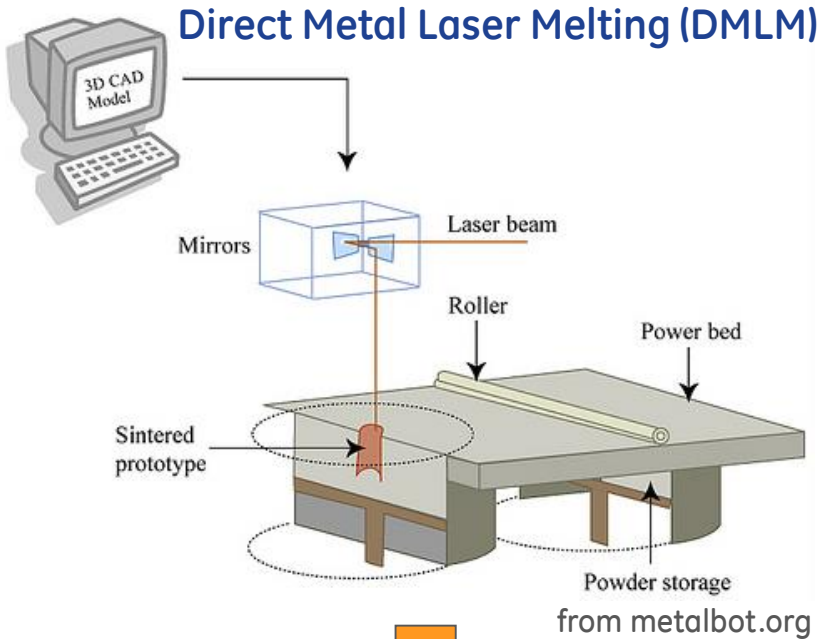
2016 DOE AMM Workshop, Germantown, MD
Oct. 17-18, 2016



Program Overview



Additive Manufacturing (3D Metal Printing)



Post Processing (HIP, Heat Treat, Surface Finishing, Machining, etc.)



Advantages:

1. Fast delivery
2. No tooling/fixturing investments
3. Ultimate manufacturing flexibility
4. Complex geometry capability
5. Material inventory simplification
6. Collapse supply chain
7. Alloy chemistry control

Disadvantages:

1. Expensive raw material/process
2. Early in additive development cycle
- knowledge gaps exist
3. Advanced inspection req'd

Goals of this Program

1. Support GE's goal of commercializing additive manufacturing in its nuclear business
2. Support fundamental understandings of AM material and advanced AM material development for nuclear applications



Addressing Technical Gaps in AM Materials

Evaluating nuclear specified properties:

In addition to common mechanical properties, the program will evaluate the following properties for AM 316L stainless steel under various post heat treatments:

- Stress corrosion cracking (SCC)
- Corrosion fatigue (CF)
- Irradiation resistance

Lowering the overall component life cost:

Understanding and utilizing the non-equilibrium microstructure by laser process to improve the nuclear specified material properties

- Complex geometry capability
- Eliminating post treatment cost from HIP
- Improving material properties and reduce asset management costs.

Developing nuclear specification for AM materials

- Understanding process variability in terms of nuclear properties
- Contributing to the development of nuclear specification for AM



Detailed Tasks of the Program

Task 1: Evaluating commercial AM stainless steel (GEGR, ORNL, UM)

- Four different manufacturers (machine, powder, process variabilities)
- Roles of laser and heat treatment on microstructure and surface
- Stress corrosion cracking (SCC) growth rate
- Corrosion fatigue (CF) cracking growth rate
- Irradiation and irradiation assisted stress corrosion cracking (IASCC)
- Effects of as-fabricated surface on SCC crack initiation

Task 2: Optimizing commercial AM stainless steel (GEGR, GEH)

- Laser process and heat treatment optimization
- Hot isostatic pressing (HIP) vs. Non-HIP
- Stainless steel chemistry optimization
- Process optimization for surface properties (roughness and microstructure)


Completed


On-going


Not started



Detailed Tasks of the Program

Task 3: Advanced AM stainless steel for SCC and radiation (GEGR, ORNL, UM)

- Grain boundary engineering
- Nano precipitate strengthened stainless steel by additive manufacturing
- Chemistry adjustment (effects of high Cr or high Ni)
- SCC, IASCC, mechanical properties

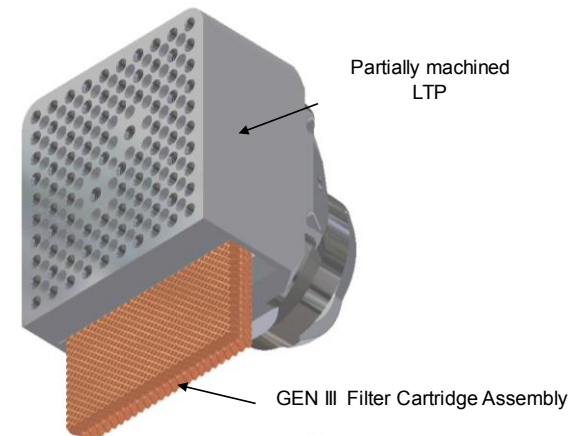
Task 4: Component demonstration and nuclear specification (GEGR, GEH, ORNL)

- Complex geometry component fabrication using optimized process
- Component evaluation (material and performance)
- Post inspection technique (micro-CT)
- Cost evaluation
- Contributions to nuclear specification


Completed


On-going


Not started



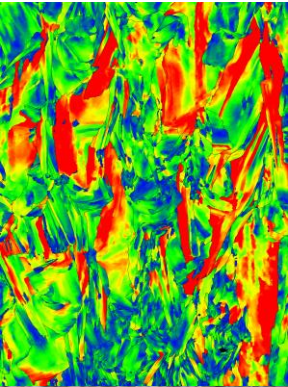
Material Microstructure and Properties



AM Stainless Steel Microstructure

Stress Relief

HIP+Solution Annealing



material build-up direction



7.0kV 8.3mm x100 PDBSE(CP) 10/21/2014

x100 PDBSE(CP) 10/21/2014

500um

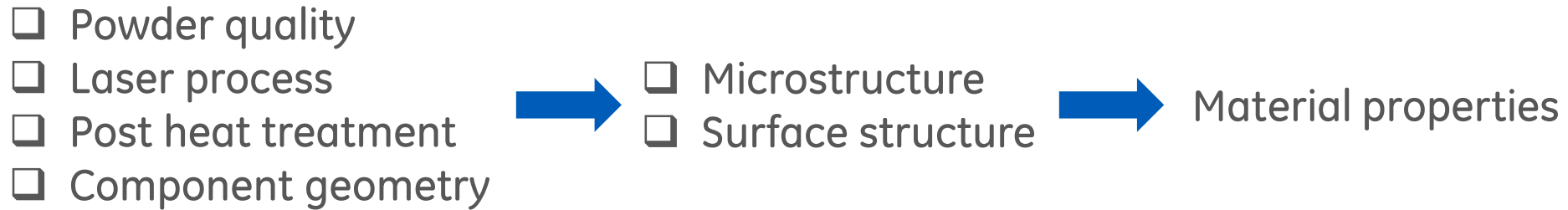
Non-equilibrium Solidification Structure

Fully Recrystallized Structure



Effects of Process Variabilities on Microstructure

Complex influence from the process:

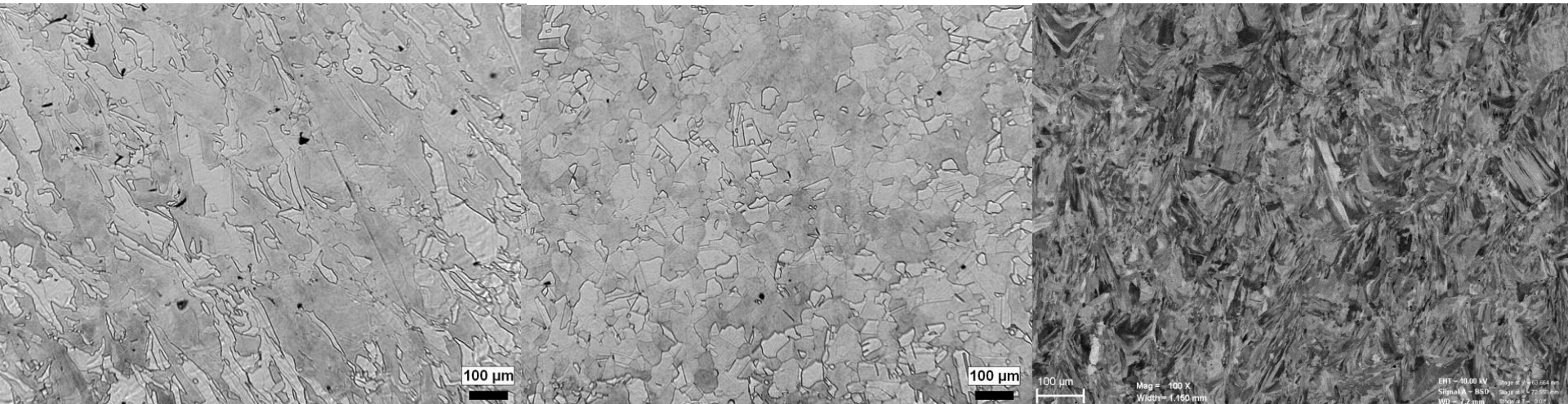


Under the same heat treatment (1950F for 1hr)

Vendor 1

Vendor 2

Vendor 3

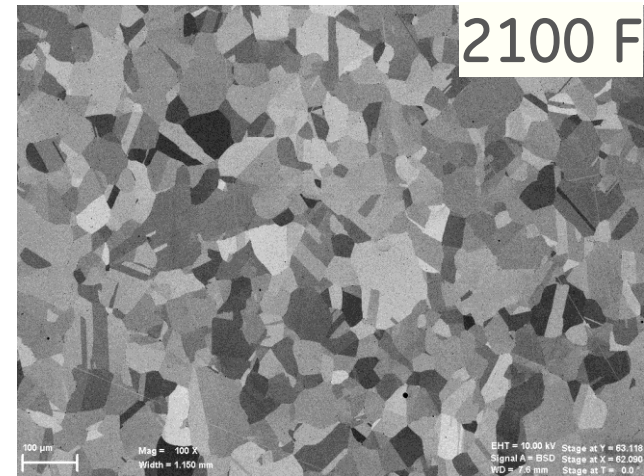
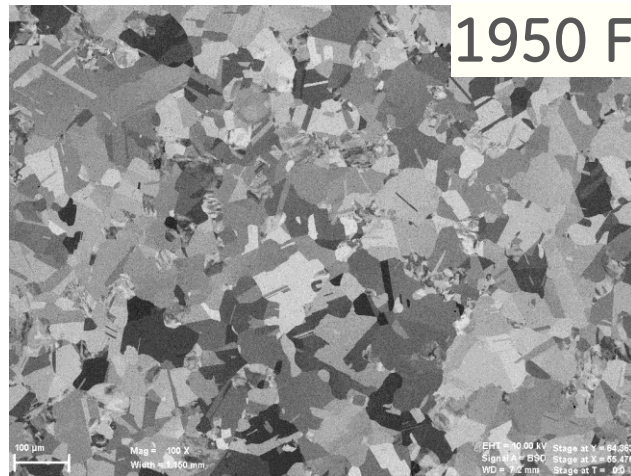
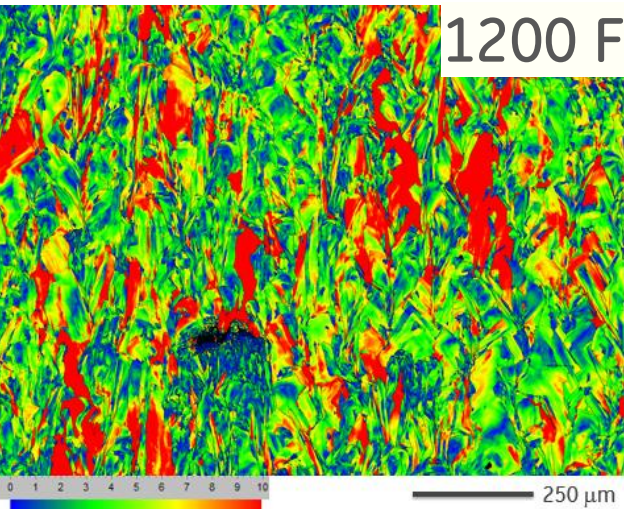


Microstructure can be very different if applying the same acceptable heat treatment on materials from different vendors

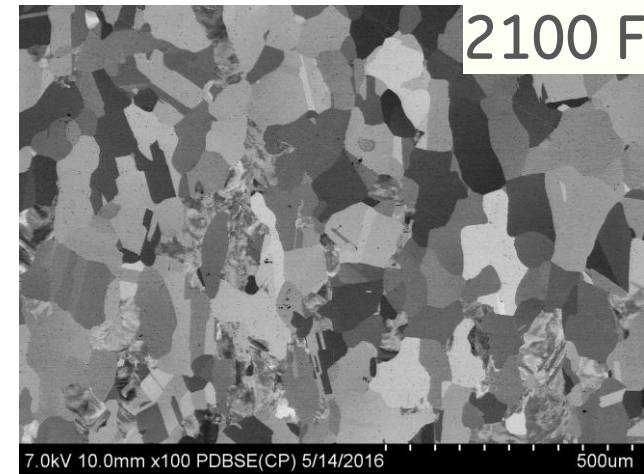
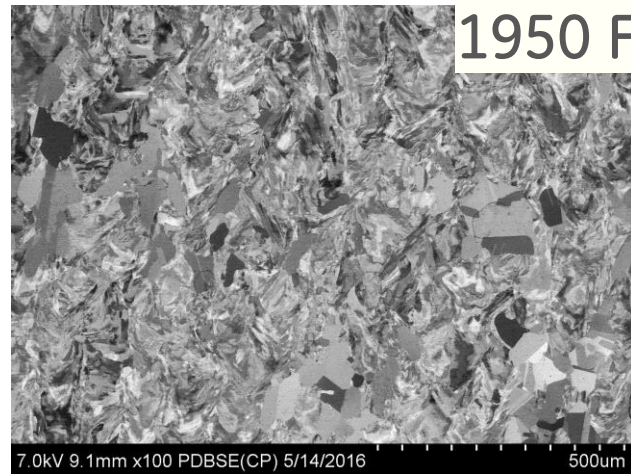
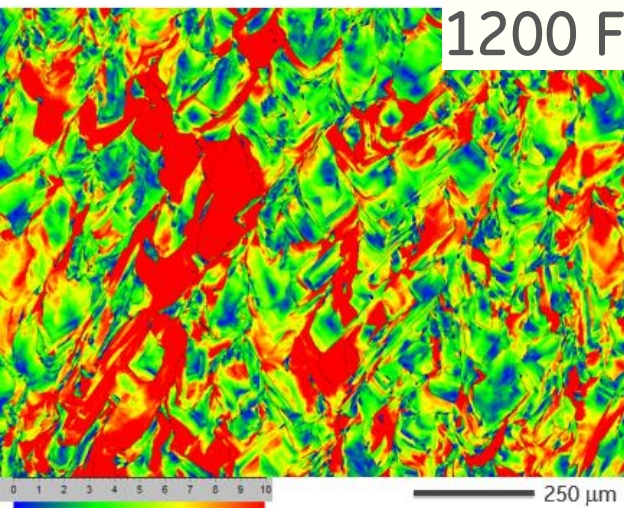


Effects of Laser Process on Heat Treated Structure

Laser Process #1



Laser Process #2



Effects of Process Variabilities on Porosity

	Porosity		Pore Size	
	Mean (%)	Std. Dev.(%)	Mean (μm)	Std. Dev. (μm)
Vendor #1	0.30	0.18	5.0	6.0
Vendor #2	0.08	0.03	4.2	4.2
Vendor #3	0.31	0.53	16.0	14.5

Vendor #1

Vendor #2

Vendor #3

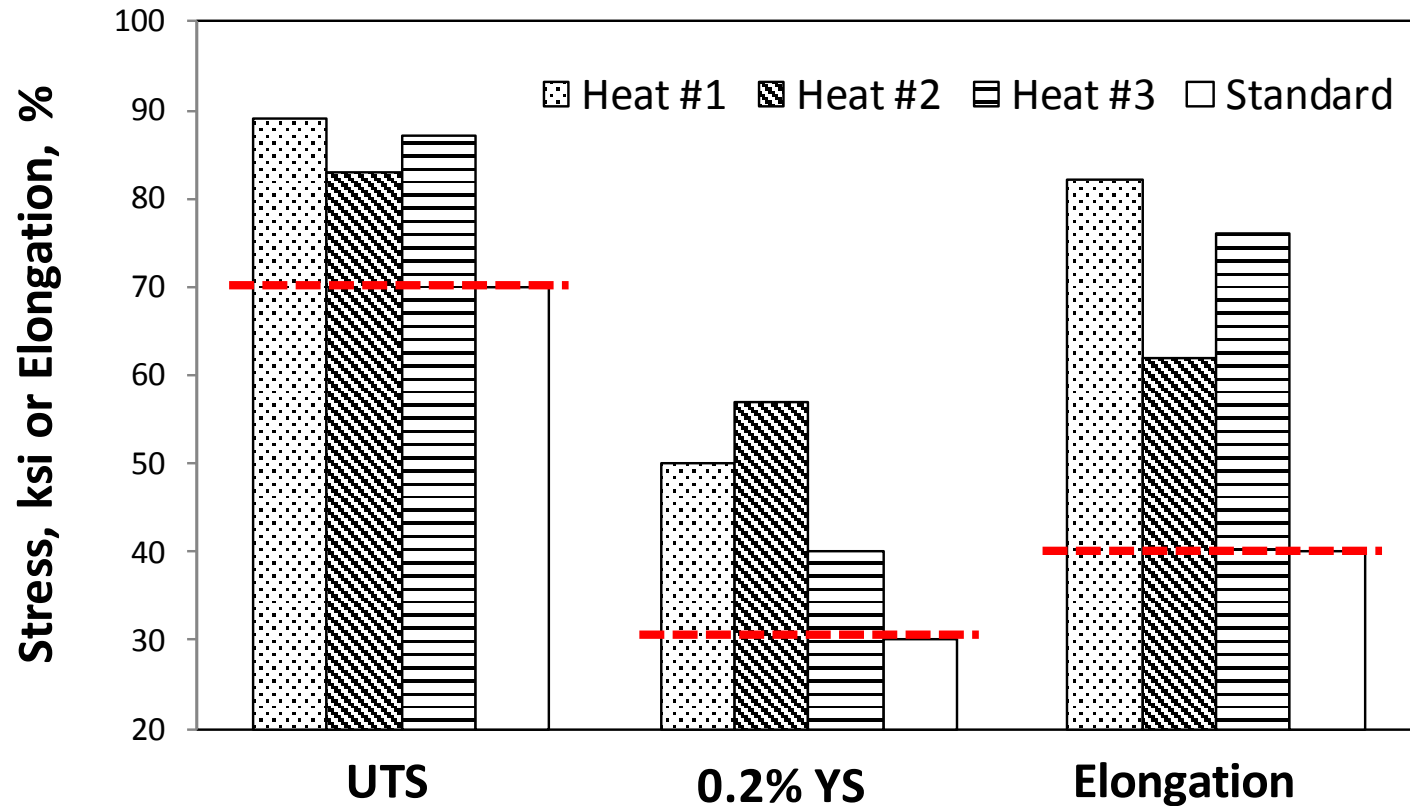


- Porosity also varies from vendor to vendor.
- Most materials have acceptable density.
- If process is optimized, porosity can be very low right after the laser process.



Effects of Process Variabilities on Tensile Properties

Solution Annealed



Mechanical properties (YS, UTS, elongation) of annealed AM stainless steel generally are beyond the spec. for wrought materials.

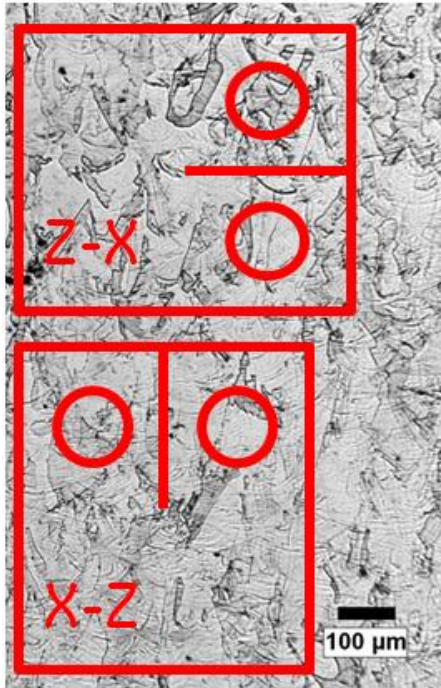


Corrosion Fatigue and Stress Corrosion Cracking



Compact Tension Specimen Orientations

Importance of orientation effect in DMLM processed material due to the directional solidification nature of the process



20% Thickness Reduction along Z



20% Thickness Reduction along X

BWR testing condition:

- 288 °C water
- 2 ppm O₂ or 63 ppb H₂
- K=22, 27.5, 33 MPa√m

5000-8000 testing hours per sample



Corrosion Fatigue Crack Growth Rate

Material	K, ksi/m	R=0.2, 0.5Hz (mm/s)	R=0.4, 0.5Hz (mm/s)	R=0.6, 0.5Hz (mm/s)	R=0.6, 0.1Hz (mm/s)	R=0.6, 0.01Hz (mm/s)
Material #1, Stress Relief, 23% CW along Z, Z-X	25	2.60E-05	2.40E-05	1.70E-05	8.00E-06	1.80E-06
Material #1, HIP+SA, 20%CW along Z, Z-X	25	2.60E-05	2.40E-05	1.60E-05	6.80E-06	1.20E-06
Material #1, Stress Relief, No CW, X-Z	25	3.60E-05	3.60E-05	2.10E-05	8.70E-06	1.90E-06
Material #1, Stress Relief, 20% CW along X, X-Z	25	3.90E-05	3.60E-05	2.20E-05	9.60E-06	1.90E-06
Material #1, Stress Relief, No CW, Z-X	25	2.50E-05	2.20E-05	1.20E-05	6.00E-06	1.20E-06
Material #1, HIP+SA, No CW, X-Z	25	1.90E-05	1.80E-05	1.30E-05	6.00E-06	1.40E-06
Material #1, 1750F, No CW, X-Z	25	2.24E-05	2.54E-05	1.65E-05	6.61E-06	1.29E-06
Material #2, stress relief, No CW, X-Z	25	3.18E-05	3.50E-05	1.82E-05	7.57E-06	1.71E-06
Material #3, 1950F, 20% CW along X, X-Z	25	3.80E-05	4.00E-05	2.35E-05	9.60E-06	1.84E-06
Material #3, 2100F, 20%CW along X, X-Z	25	2.82E-05	2.77E-05	1.73E-05	7.37E-06	1.39E-06

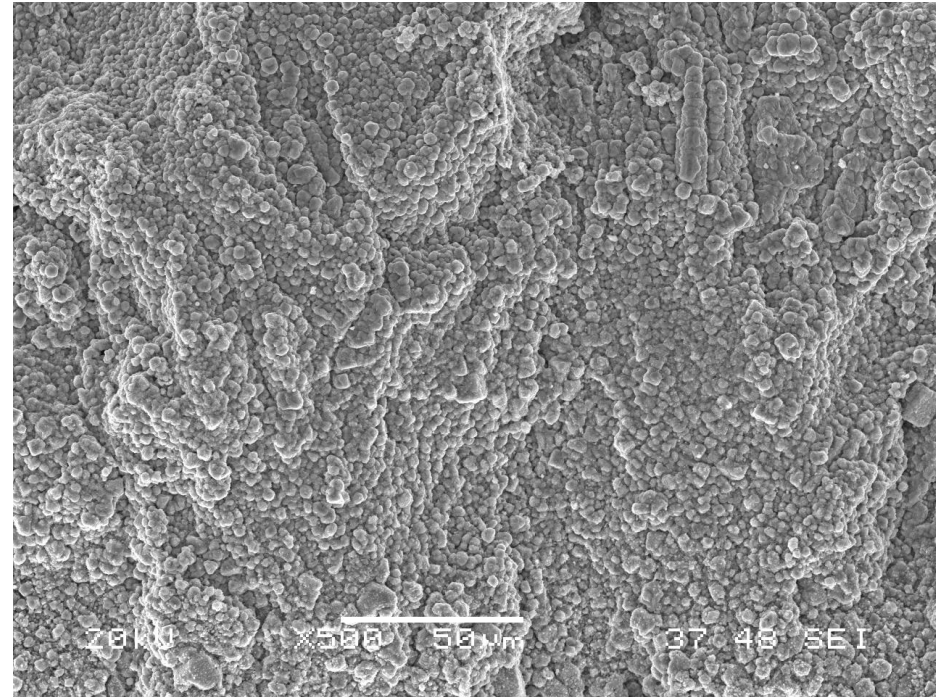
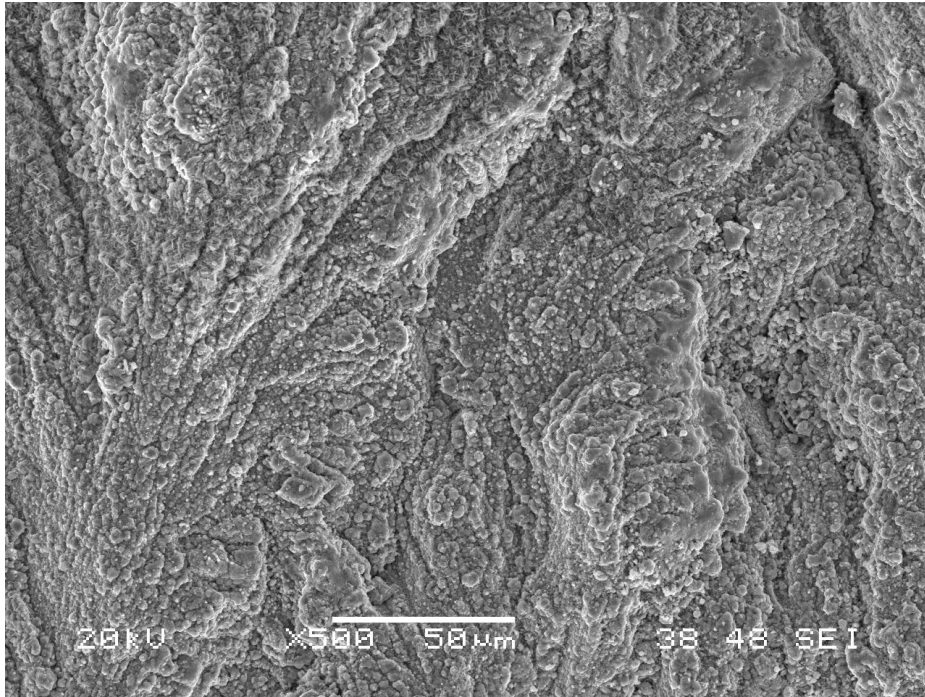
Tests have been done based on different material heats, heat treatment conditions, HIP vs. Non HIP, orientations, and different ways of cold work. No significant difference in corrosion fatigue crack growth was found for all the tested specimens.



Crack Morphology of Corrosion Fatigue Crack

Stress Relief

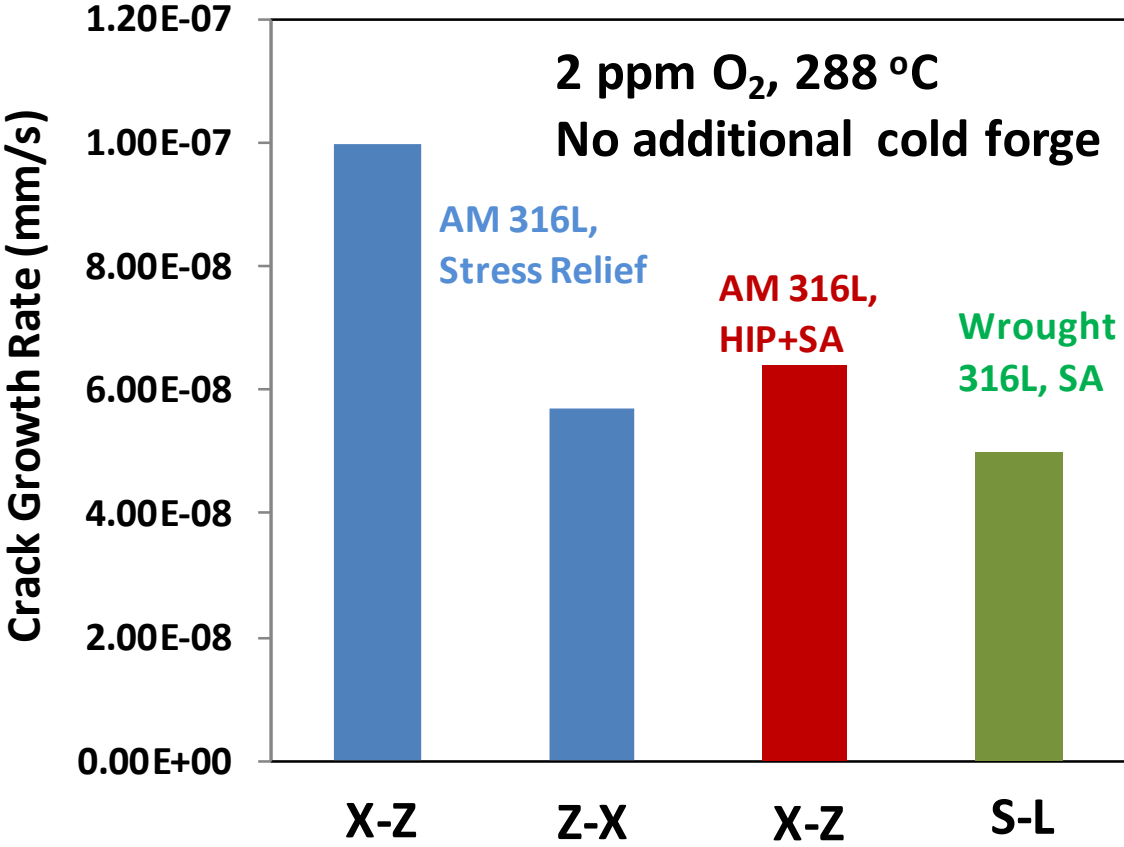
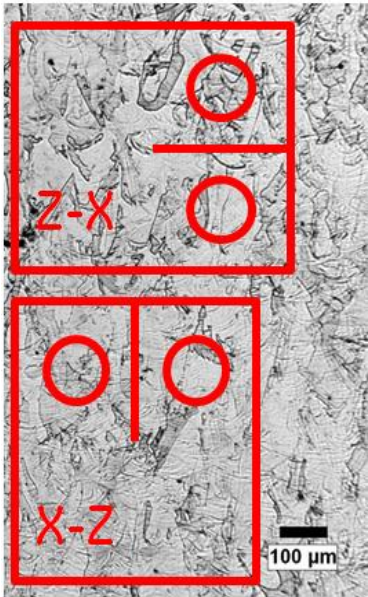
HIP + Annealing



All tested specimens show similar transgranular cracking mode



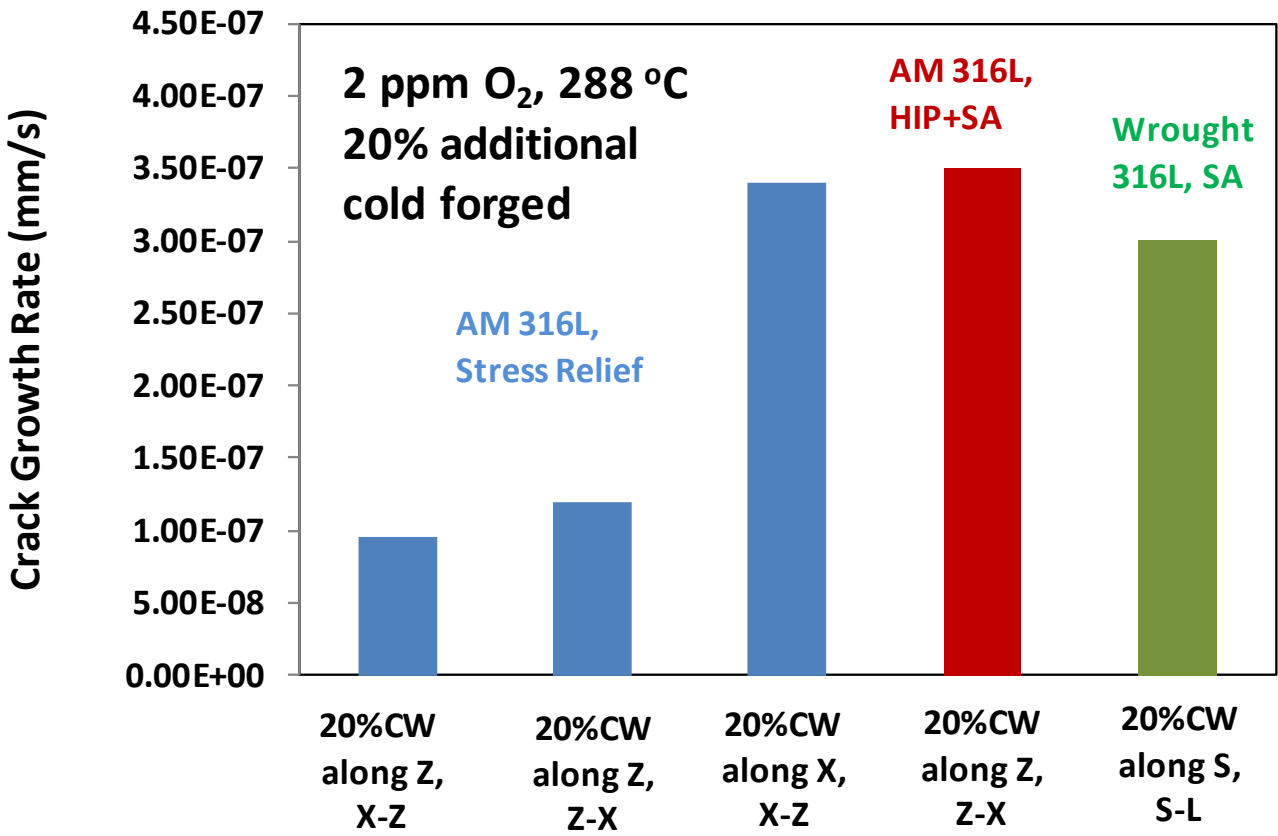
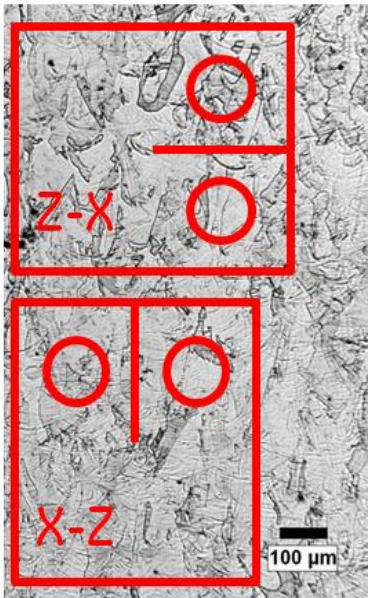
Stress Corrosion Crack Growth Rate with No CW (stress relief vs. HIP+SA vs. Wrought)



- ❑ Stress relieved sample shows anisotropy in SCC crack growth rate.
- ❑ Crack growth rate of stress relieved sample is higher than wrought along Z orientation.



Stress Corrosion Crack Growth Rate with 20% CW (stress relief vs. HIP+SA vs. Wrought)



- ❑ SCC growth rate on stress relieved sample is sensitive to cold forge orientation.
- ❑ In some orientations, the AM stainless steel under cold forge can show slower crack growth rate, which is due to the microstructure.

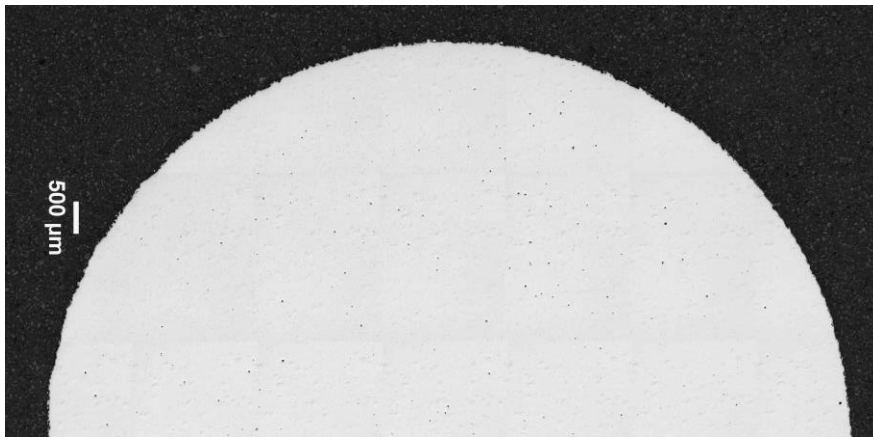


Stress Corrosion Cracking: HIP vs. Non-HIP vs. Wrought

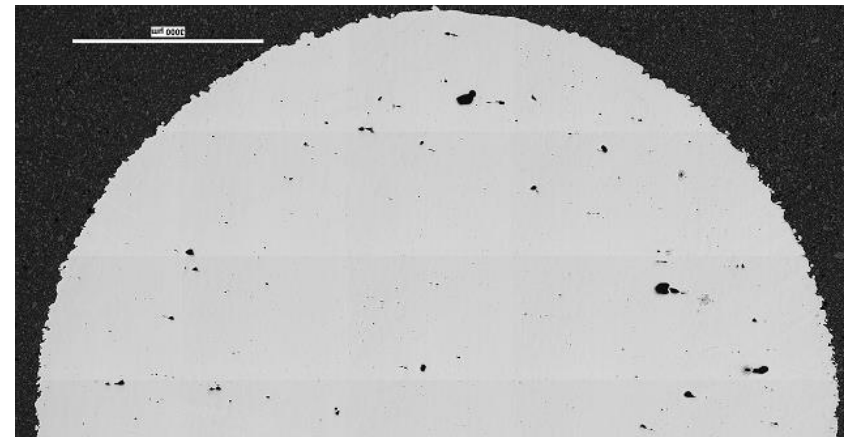
20% Cold Forged,
fully recrystallized
grain structure

Tested Materials	CGR (mm/s)
AM Heat #1 with HIP+SA	3.5×10^{-7}
AM Heat #2 with SA only	4.5×10^{-7}
Wrought with SA	$2 \sim 4 \times 10^{-7}$

AM Heat #1 with HIP+SA



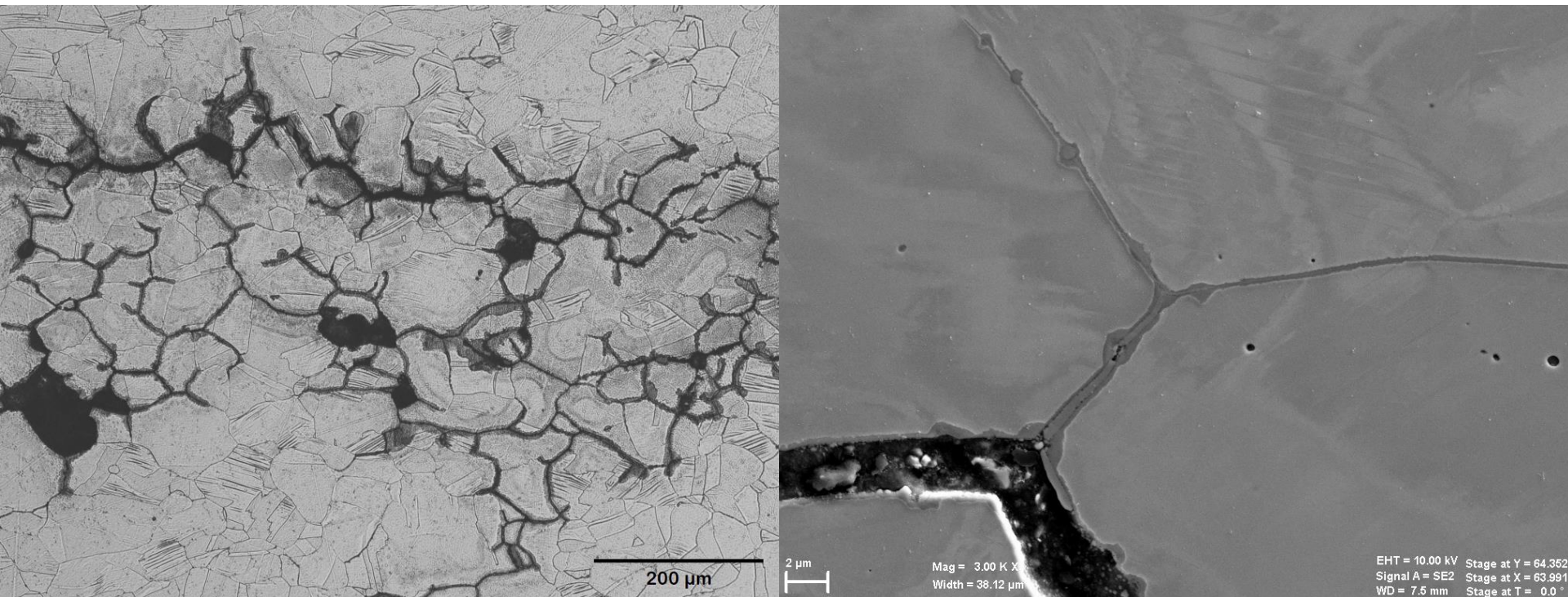
AM Heat #2 with SA (Non-HIP)



- ❑ Without HIP, the material shows acceptable SCC growth rate, even with the poor starting porosity in the material.
- ❑ More long term tests with various conditions are currently conducted to confirm this conclusion.



Fracture Morphology of HIP'ed AM 316L



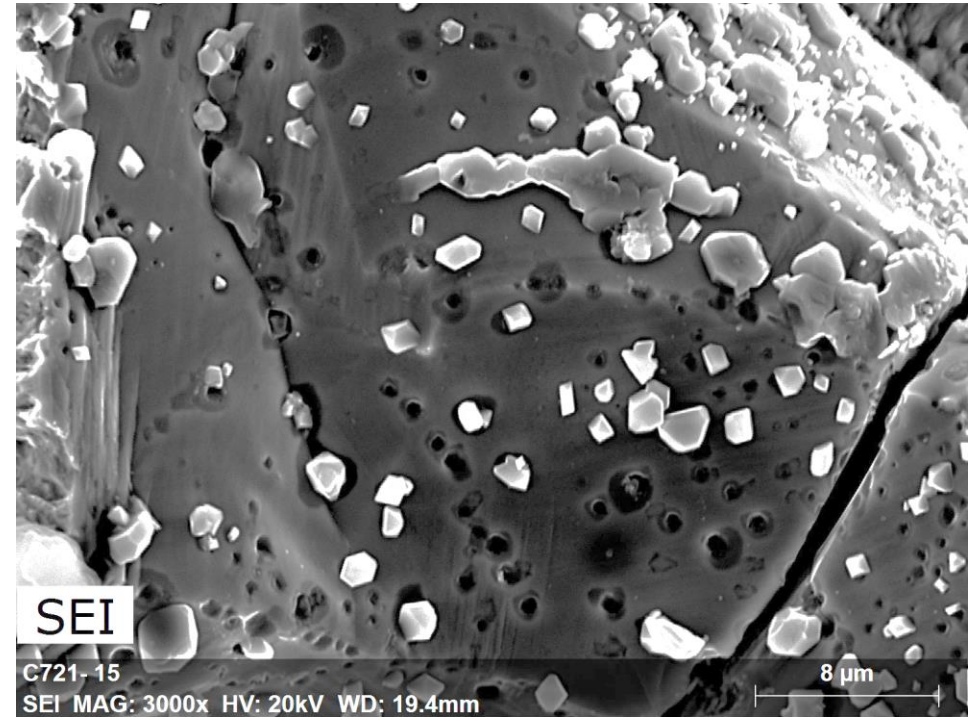
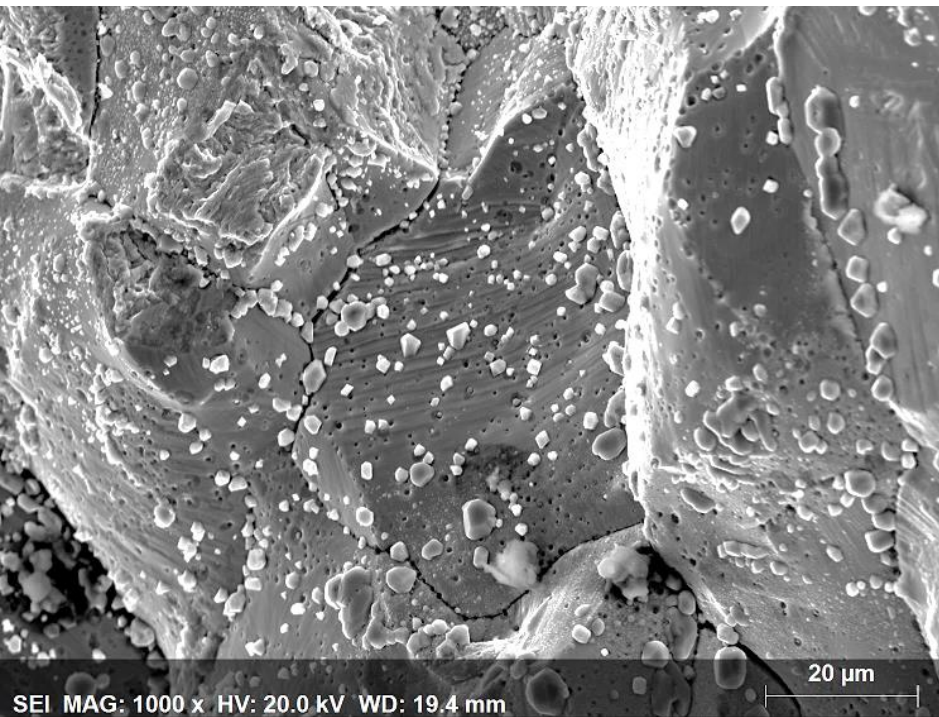
HIP'ed DMLM 316L shows heavily branched intergranular crack morphology

Large precipitates was observed along grain boundary



Si and Mn rich oxide precipitation along grain boundary

High Mag Fracture Surface Image



- ❑ Si & Mn rich oxide precipitates dissolve and re-precipitate in high temperature water from the grain boundary.

Si and Mn are suggested being removed from nuclear AM stainless steel chemistry

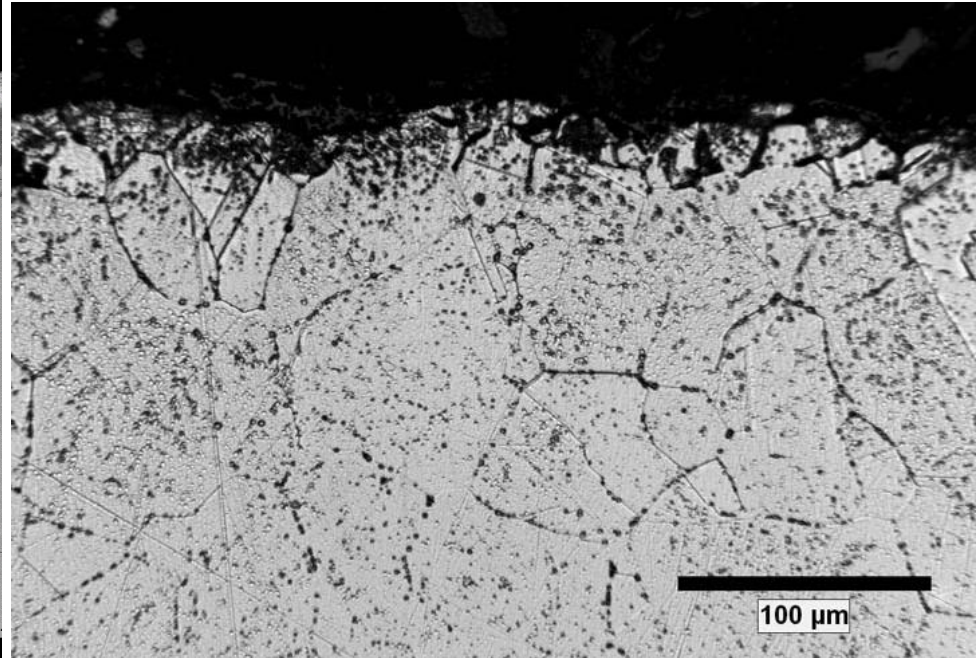
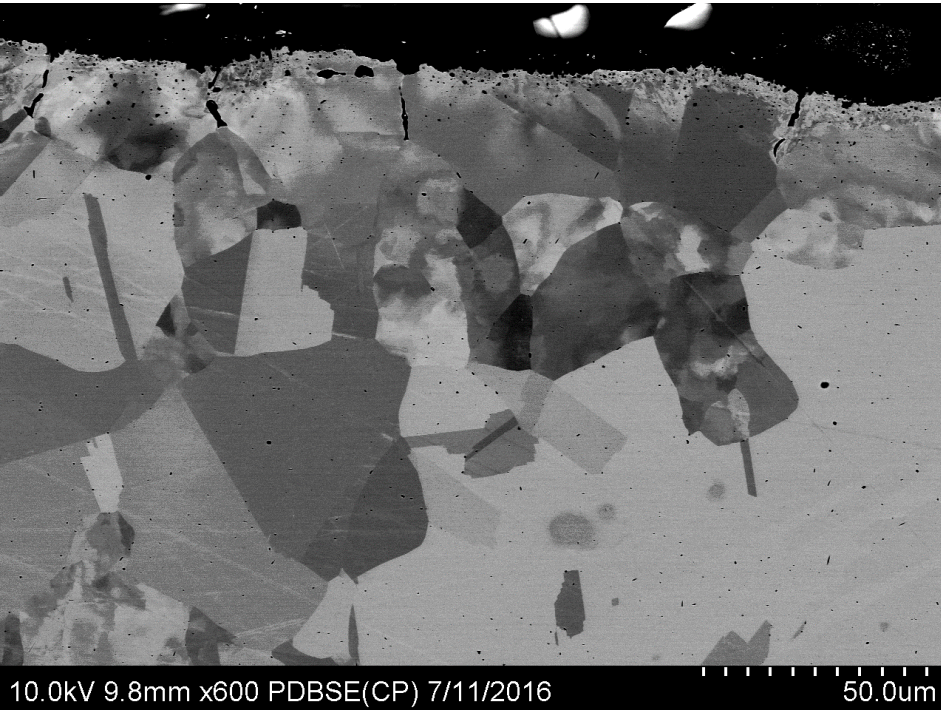


Surface Properties by AM



Surface Microstructure

Surface property (roughness and microstructure) is very critical for component performance and crack initiation.

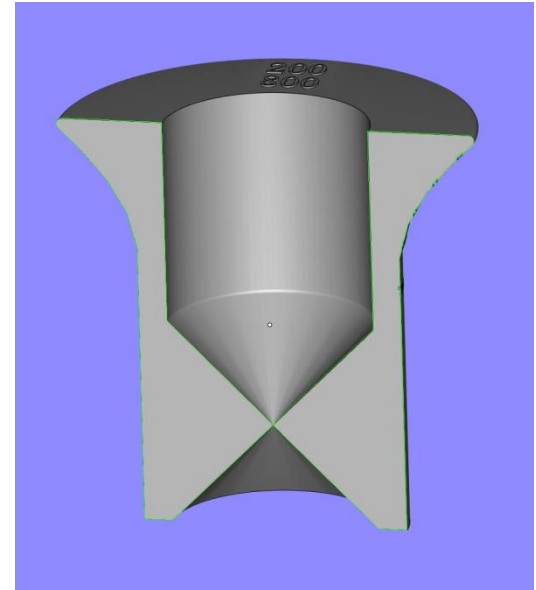


- ❑ More residual strain on the surface compared to the bulk
- ❑ Existing surface crack after process and heat treatment
- ❑ Higher density of precipitates on the surface compared to the bulk



Process Optimization for Surface Improvement and Crack Initiation Evaluation

- ❑ Parameter study is currently conducted to understand how process affects surface properties (both roughness and microstructure).
- ❑ Laser fabricated tensile samples will be used for SCC and IASCC initiation study.



SCC Initiation Sample



IASCC Initiation Sample



Summary



Material Evaluation

- There are business benefits by adopting DMLM as an advanced manufacturing method in nuclear industry for both reactor service and new component design.
- It is convinced that stainless steel by DMLM process shows at least similar mechanical properties, corrosion fatigue and stress corrosion cracking growth rate as its wrought counterpart.
- Stress relieved material is not recommended.
- Precipitation and surface microstructure are issues for nuclear applications.
- HIP may not be needed for nuclear applications. More evaluation is on-going.



Material Development

- An optimized AM process and heat treatment for component fabrication
- Removing Si and Mn from AM stainless steel chemistry
- Non-HIP'ed AM stainless steel for nuclear application
- Surface microstructure optimization for component build



Acknowledgment and Disclaimer

This program is supported by US Department of Energy and cost shared by General Electric Company under Award No. DE-NE0008428.

Disclaimer: This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.



