AMM
Advanced Methods for Manufacturing

DEC 2 – 3, 2020

Integrating Dissolvable Supports, Topology Optimization, and Microstructure Design to Drastically Reduce Costs in Developing and Post-Processing Nuclear Plant Components Produced by Laser-based Powder Bed Additive Manufacturing

Award Number: DE-NE0008813

Award Dates: 10/2018 to 9/2021

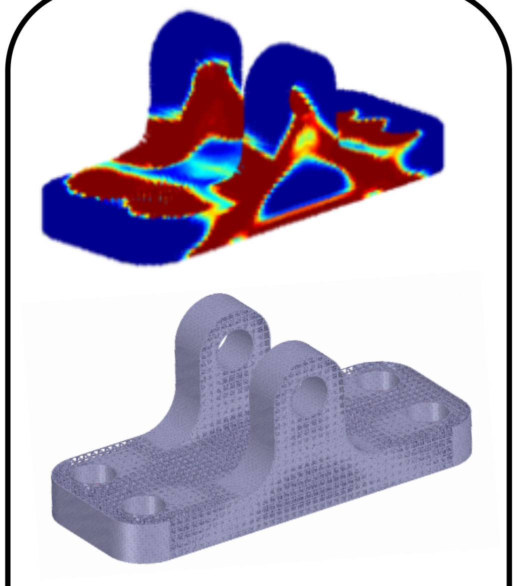
PI: Albert To (University of Pittsburgh)

Team Members: Wei Xiong (University of Pittsburgh), Owen Hildreth (Colorado School of Mines), Curtiss-Wright EMD, and Kennametal

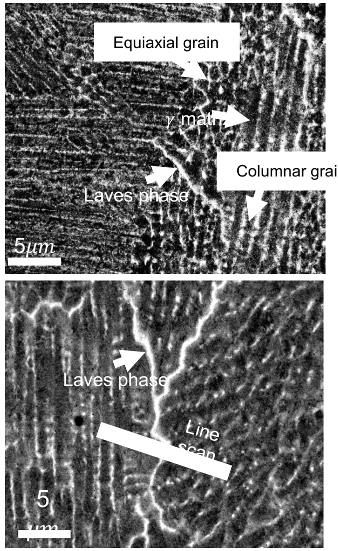
Project Goal

- This project aims to develop and establish an innovative approach to drastically reduce development and post-processing costs associated with laser powder bed additive manufacturing (AM) of complex nuclear reactor components with internal cavities and overhangs. The approach will integrate dissolvable supports, topology optimization, microstructure design to achieve the above goal.

Dissolvable Support



Support Optimization



Microstructure Design

Project Objectives

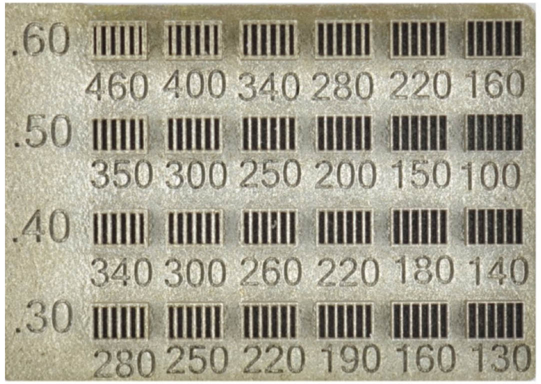
- Develop and validate recipes to dissolve support structures and reduce surface roughness using the self-terminating dissolution process (Lead: Dr. Owen Hildreth, CSM)
- Develop an automated support structure design tool capable of maximizing the support dissolution rate and minimizing residual stress and distortion of AM parts (Lead: Dr. Albert To, PITT)
- Design AM processing with post-heat treatment to optimize hierarchical structure of AM parts by applying the ICME (Integrated Computational Materials Engineering) modeling (Lead: Dr. Wei Xiong, PITT)
- Design surface heat treatment recipes for enhanced mechanical property (Lead: Dr. Wei Xiong, PITT)
- Demonstrate that the integrated technology is capable of removing internal support structures, not assessable by post-machining, for two complex nuclear reactor components in less than 24 hours (All)

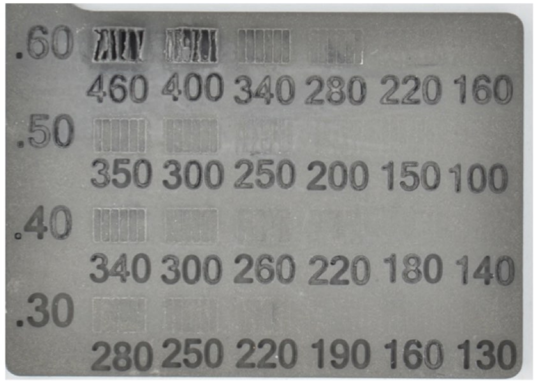
Technical Progress/Accomplishments

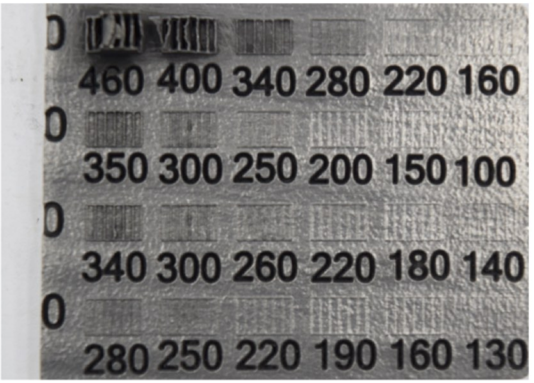
- It is possible to select a bias that preferentially gives a low surface roughness but still etch relatively thick supports, on the order of 250 µm to 300 µm thick.
- A wall thickness of 300-400 microns with the support parameters was determined to be the maximum to successfully dissolve.
- Based on dissolvability test results, a new support design with thin wall structure at the solid/support interface and lattice in the remaining part has been designed.
- The inherent strain models for 316L and 17-4PH were validated experimentally via XRD stress measurements.
- Inherent strains have been integrated with lattice structure optimization to minimize residual stress of solid components.
- Homogenization heat treatment of 17-4 PH has been carried out at 1050 ^{o}C with different times from 4 h to 10 hours based on the CALPHAD-based simulation and traditional post heat treatment, to dissolve the pre-existing Cu-cluster and strong texture induced by printing.
- The strengthening mechanism of precipitation in homogenization was elucidated via the fractography. The decarburization distance and time at different temperatures for SS316L was determined using DICTRA modeling.

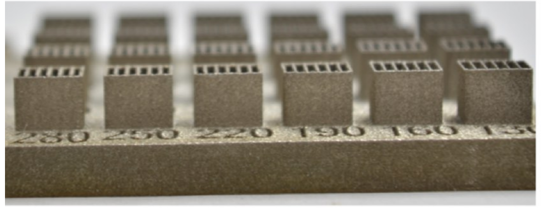
Support Thickness Study: Material Removed

As Printed | 400mVSHE | 550mVSHE

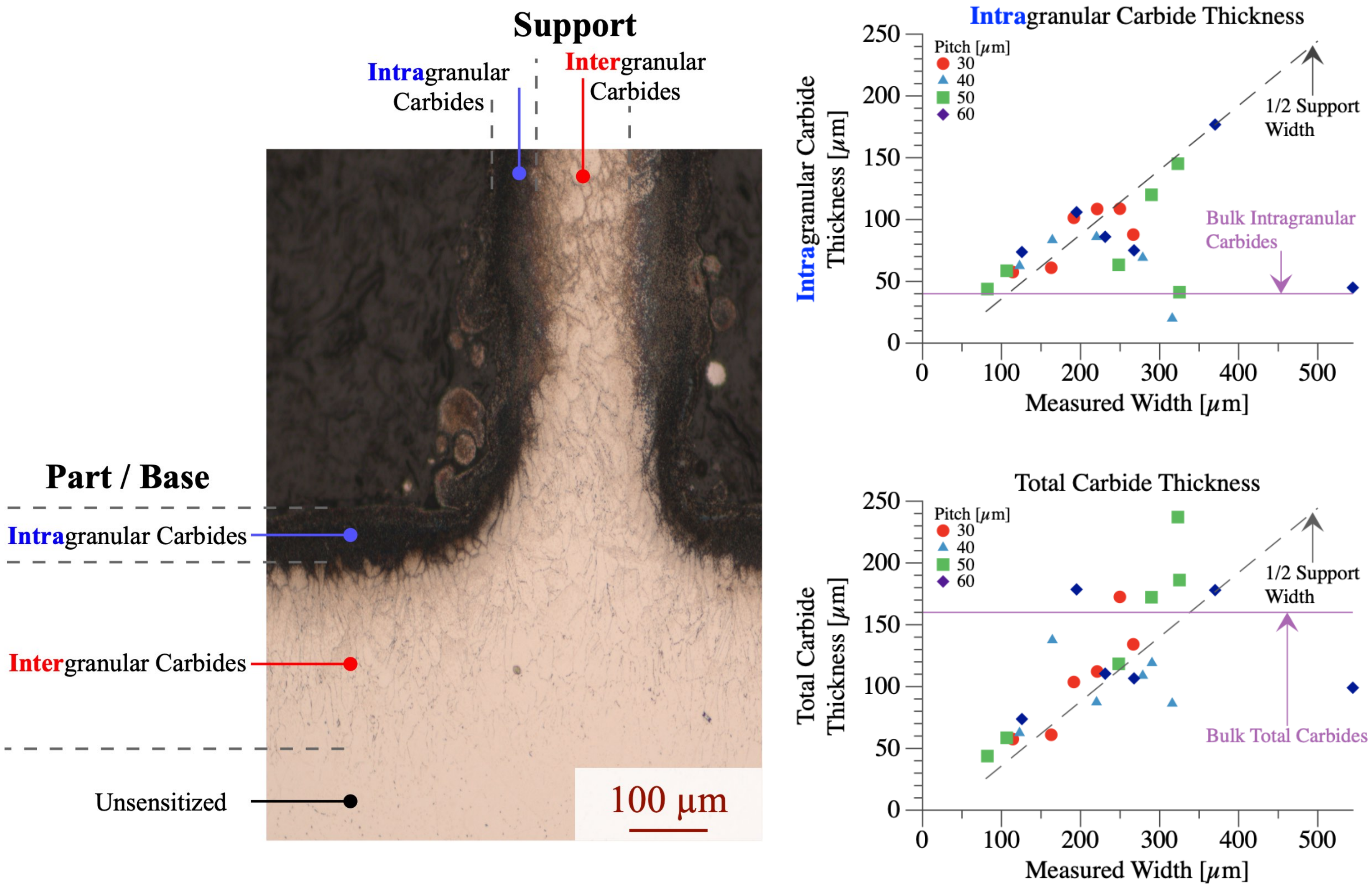




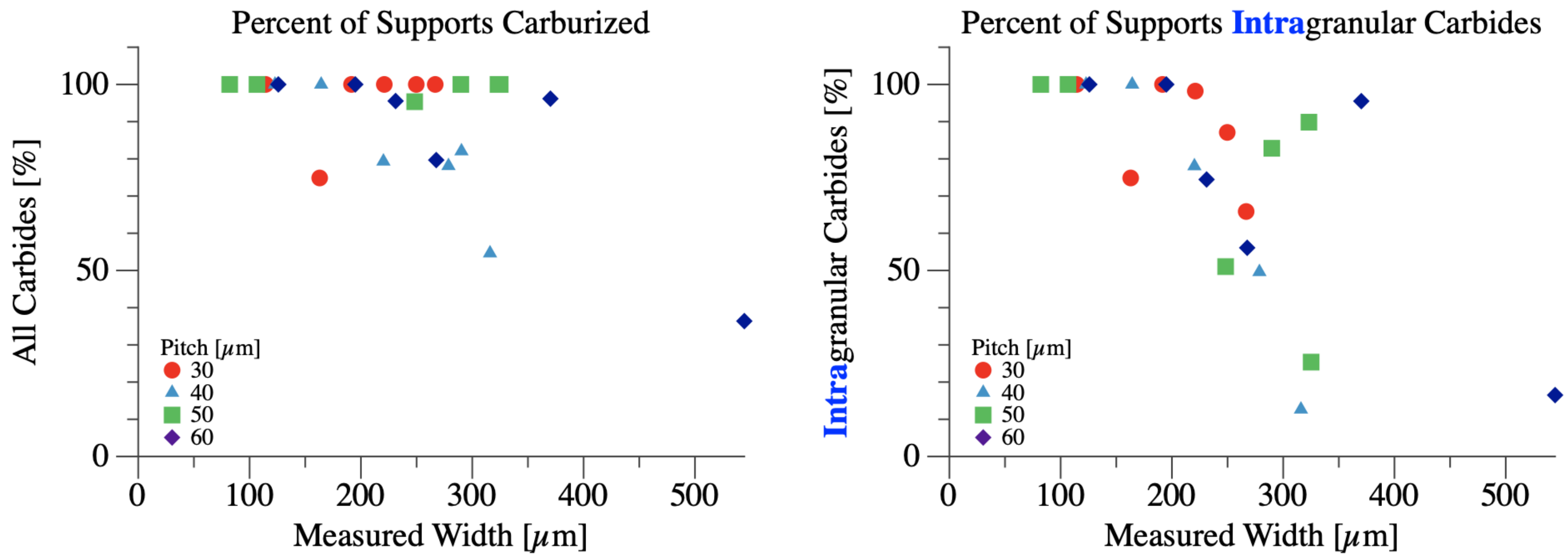




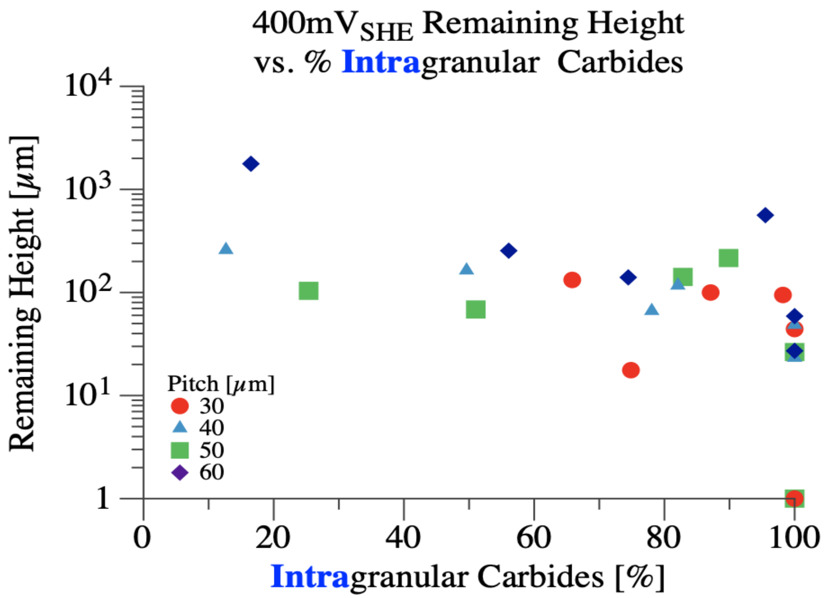
Support Thickness Study: Carbides

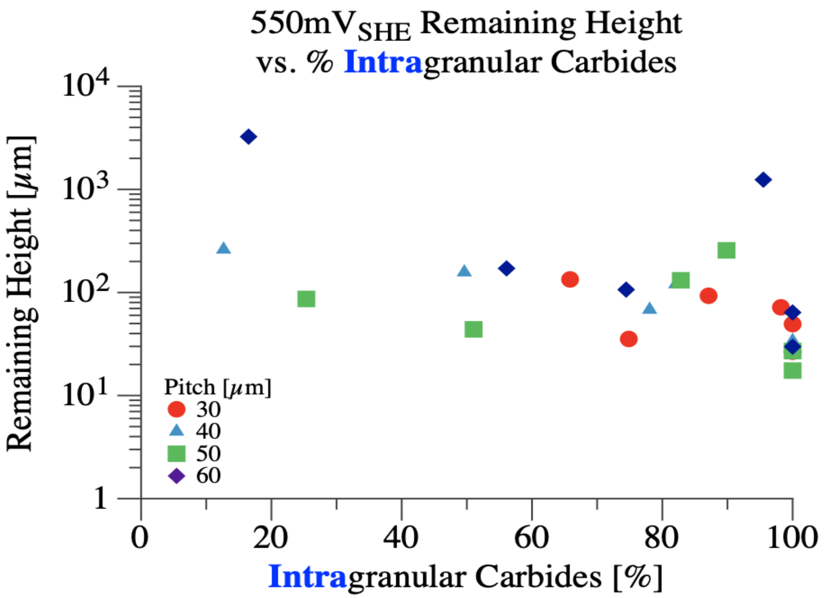


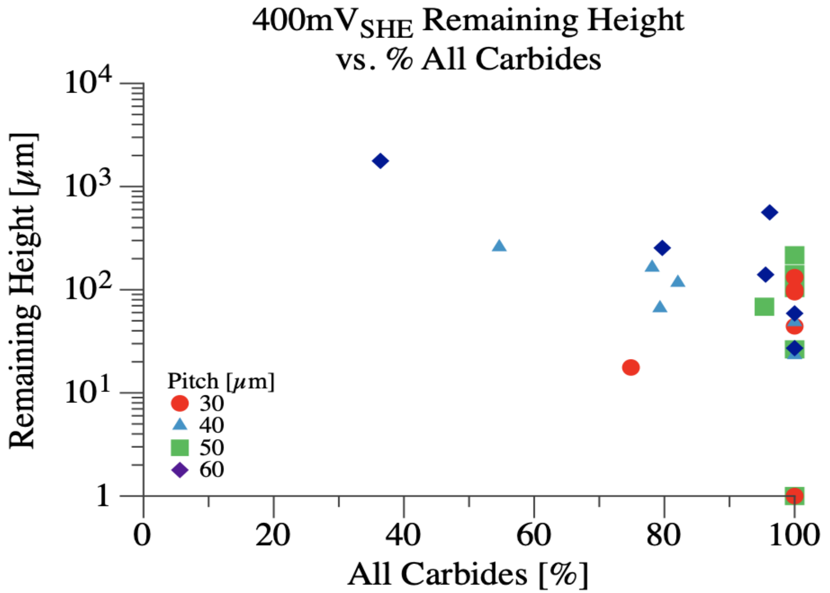
Support Thickness Study: Carbide Formation

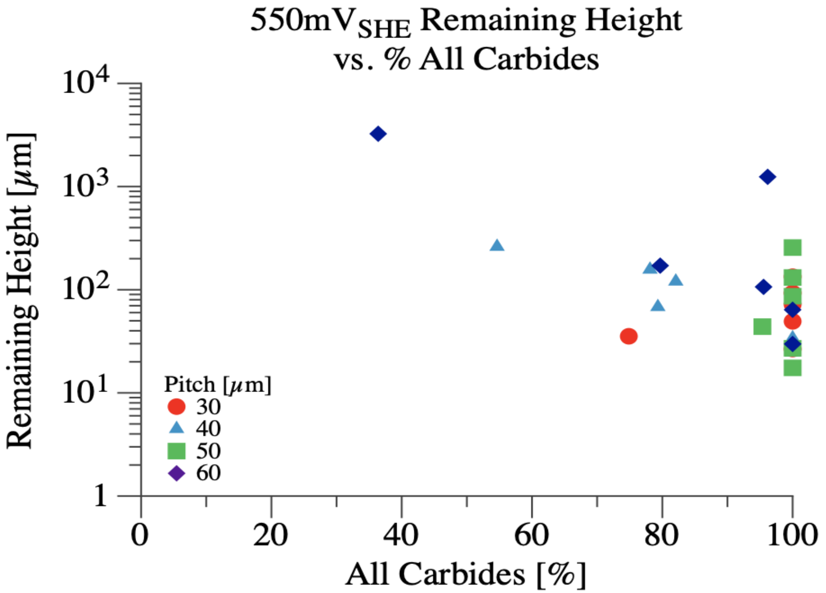


Support Thickness Study: Material Removed

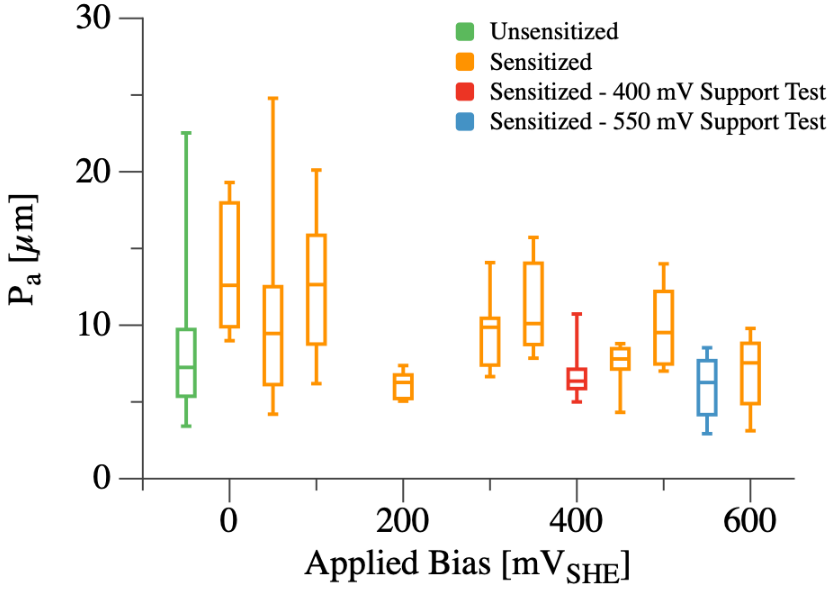


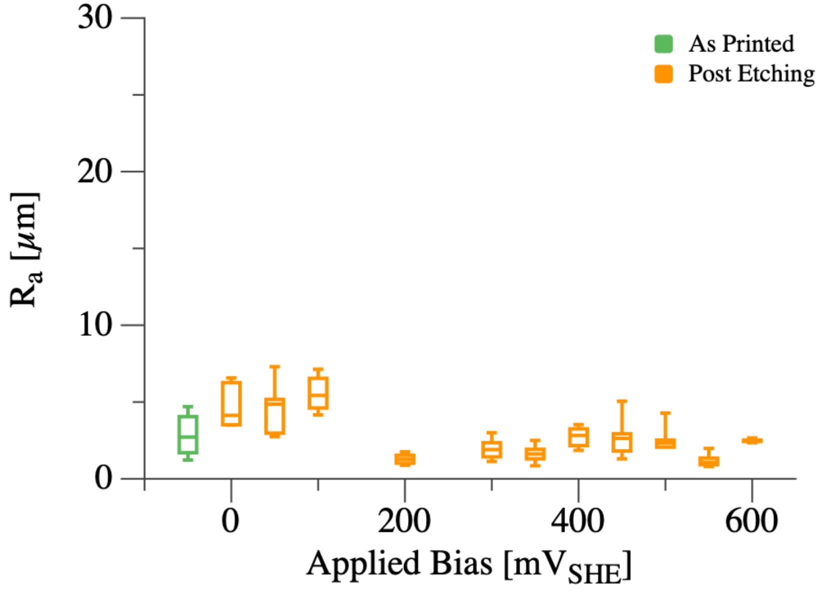






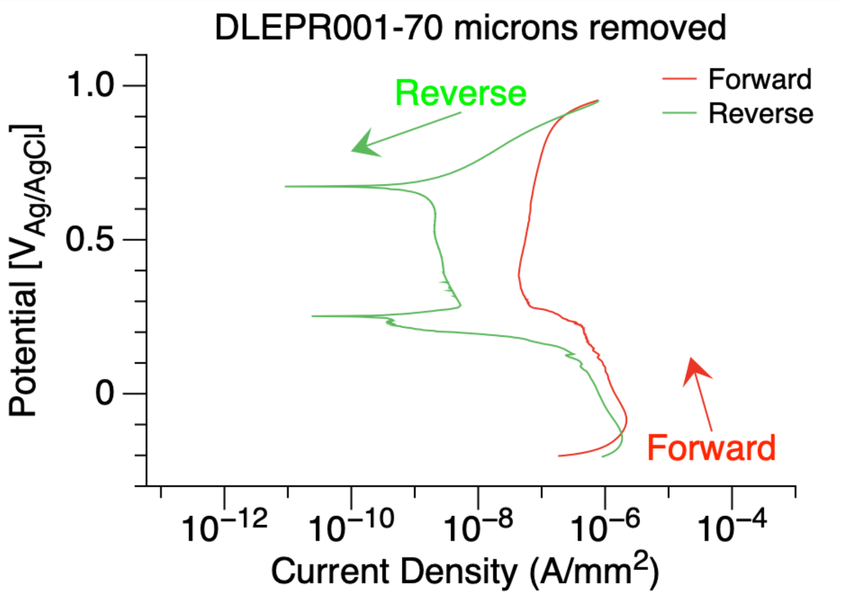
Updated Roughness Graphs

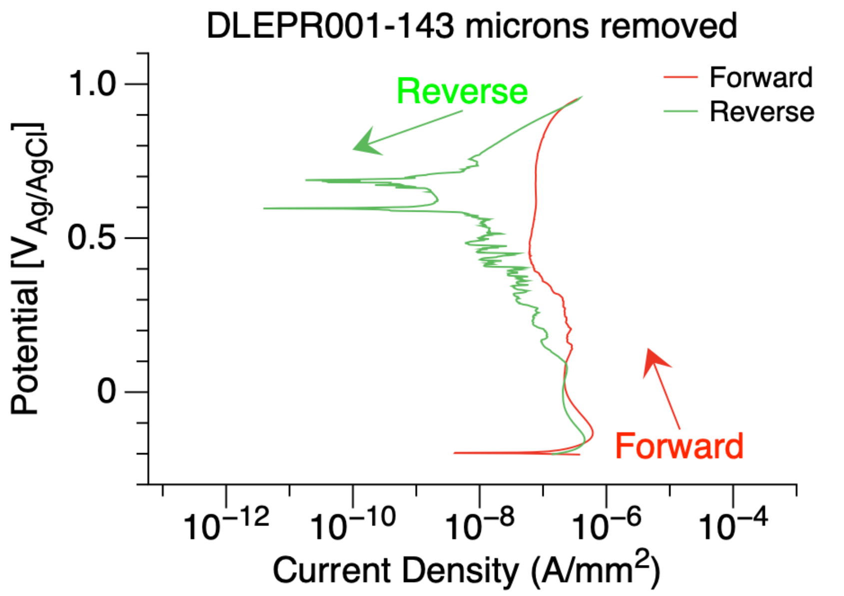


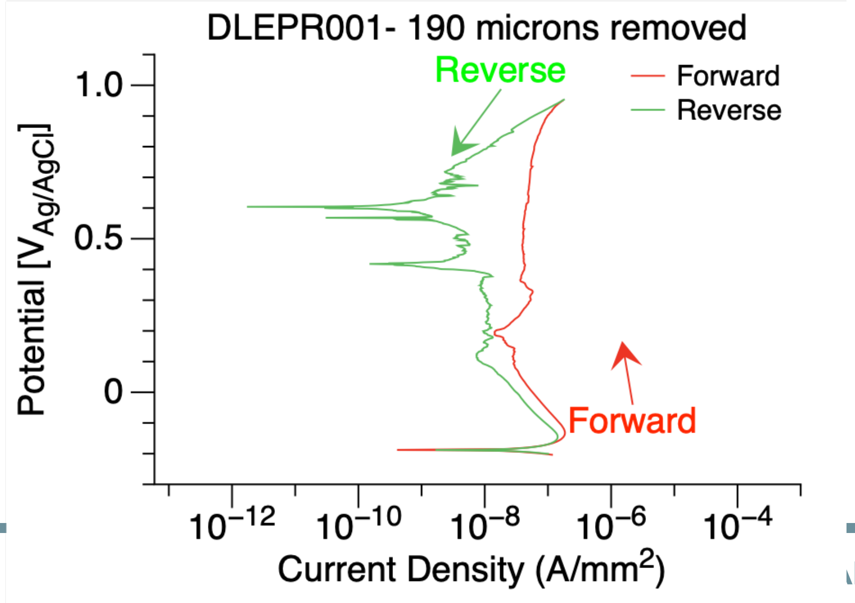


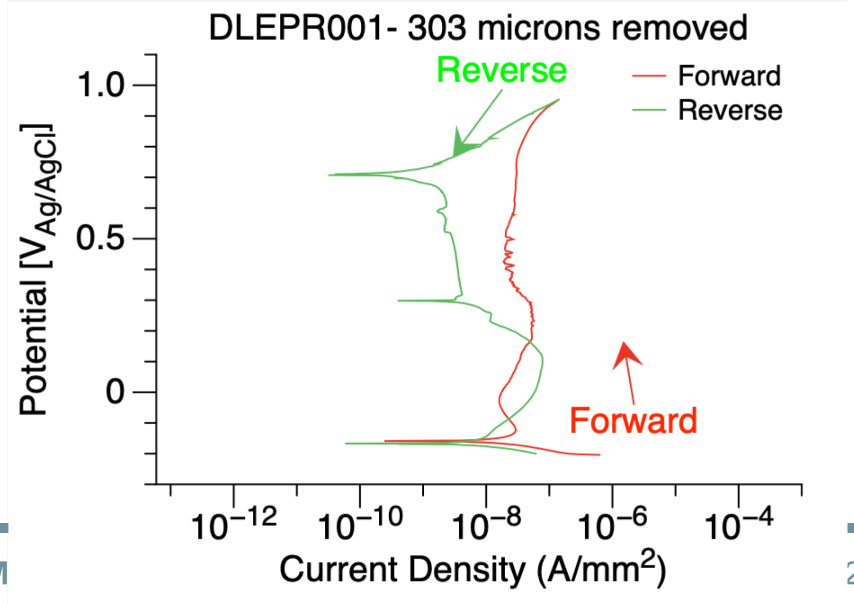
Advanced Methods for Manufacturing

DLEPR: Non-Heat Treated

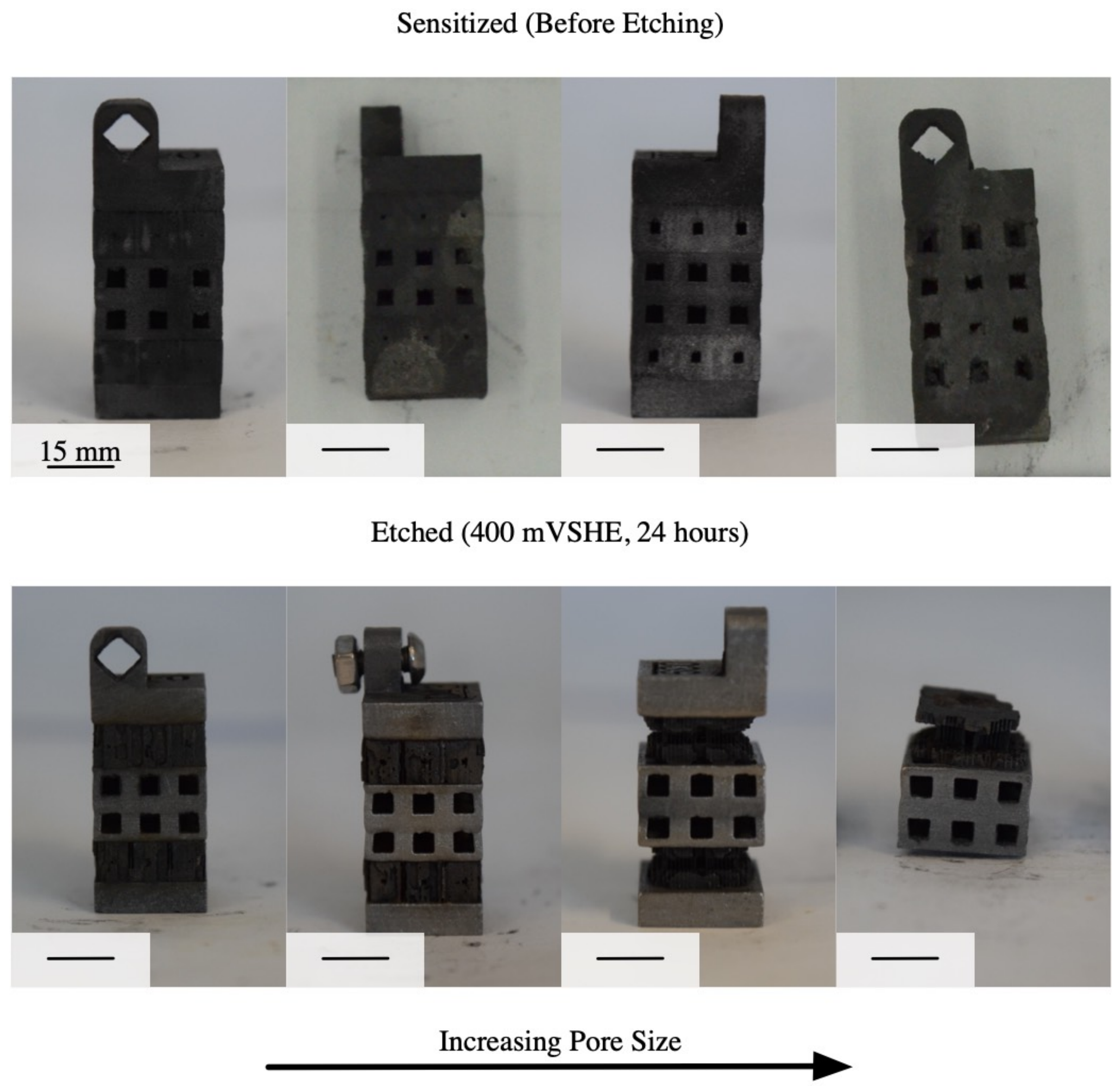






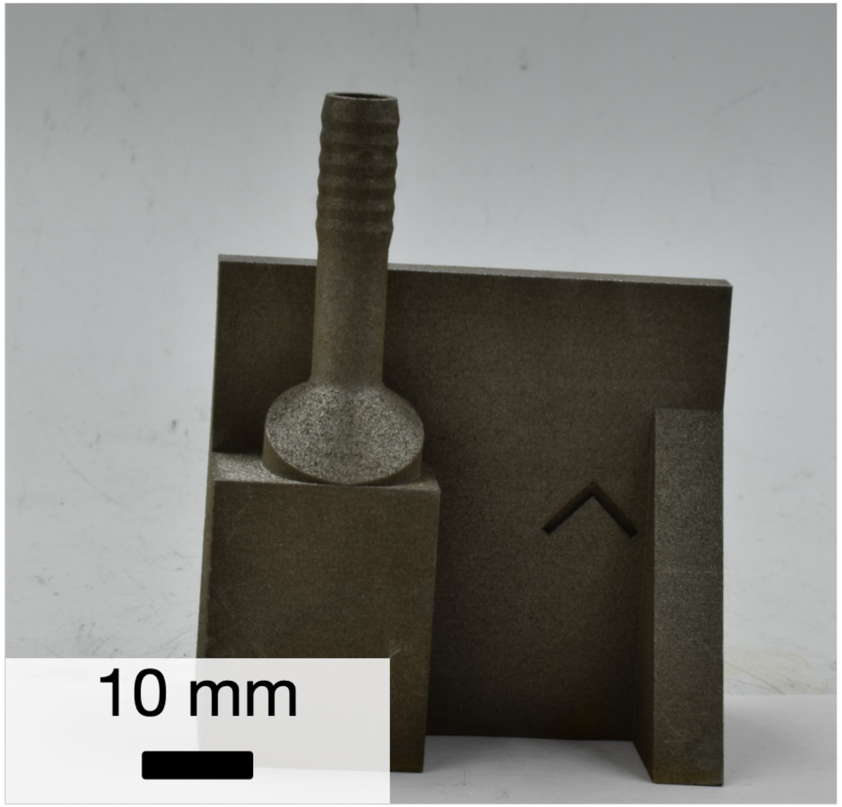


Boxes

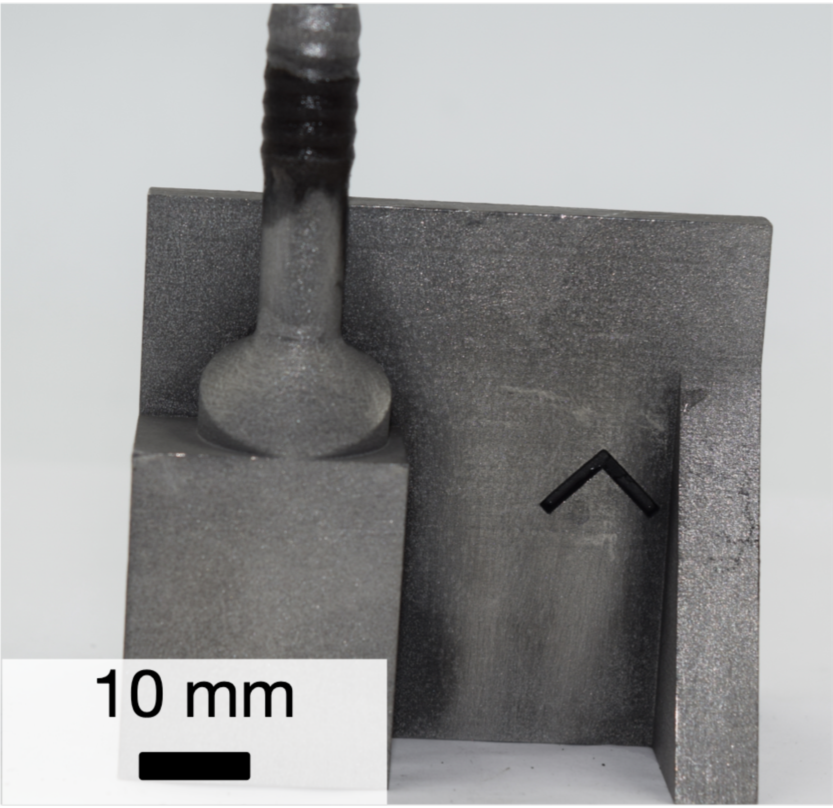

Sensitized (Before Etching)
15 mm
Etched (400 mVSHE, 24 hours)
Increasing Pore Size

Miniature Pressure Vessel

As Printed

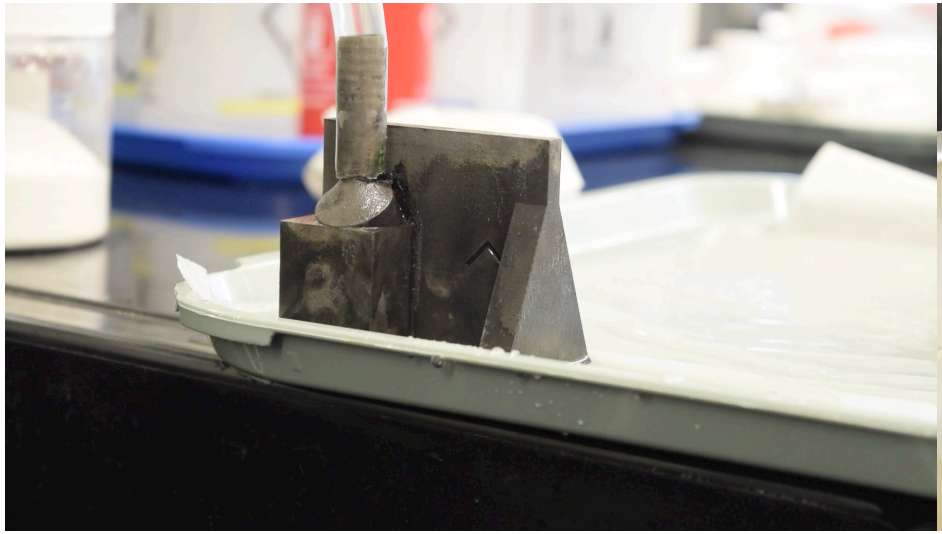


Etched 550mVSHE, 60 hours

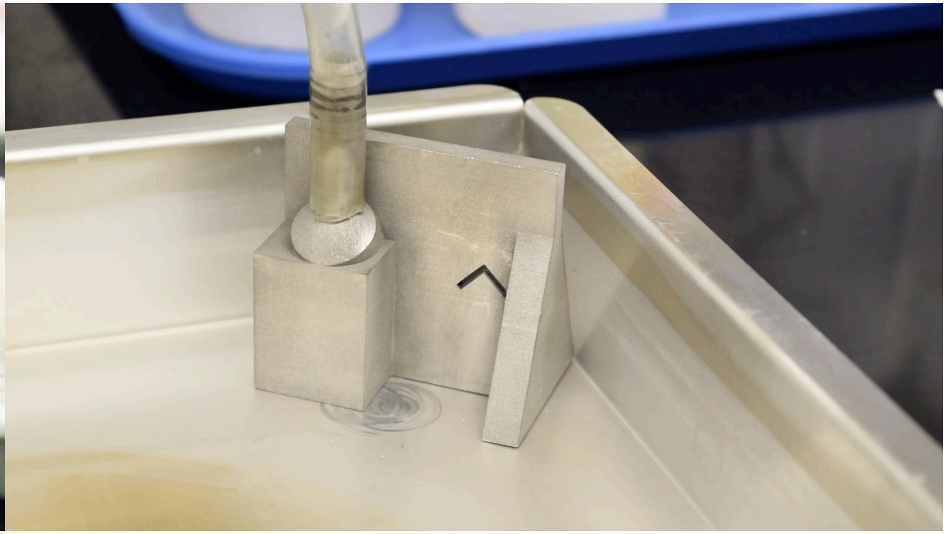


Miniature Pressure Vessel

Sensitized

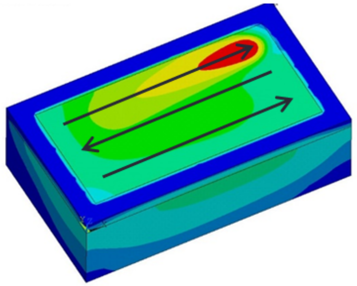


Etched 550mVSHE, 65 hours

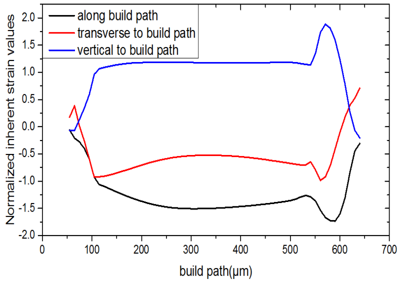


Modified Inherent Strain Method

Detailed model
- meso-scale (~0.1mm)
- sequentially coupled thermo-mechanical analysis

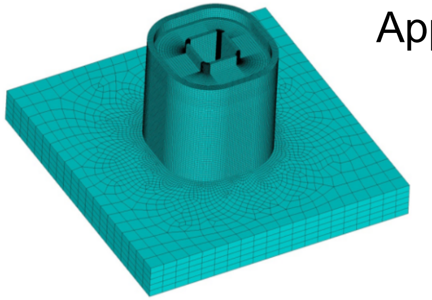


Extract inherent strains (element by element)

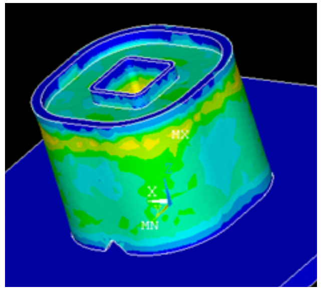


$$\varepsilon^{In} = \varepsilon_{t_i}^{Plastic} + \varepsilon_{t_i}^{Elastic} - \varepsilon_{t_s}^{Elastic}$$

Inherent strain model
- macro-scale (~100mm)
- static-mechanical analysis



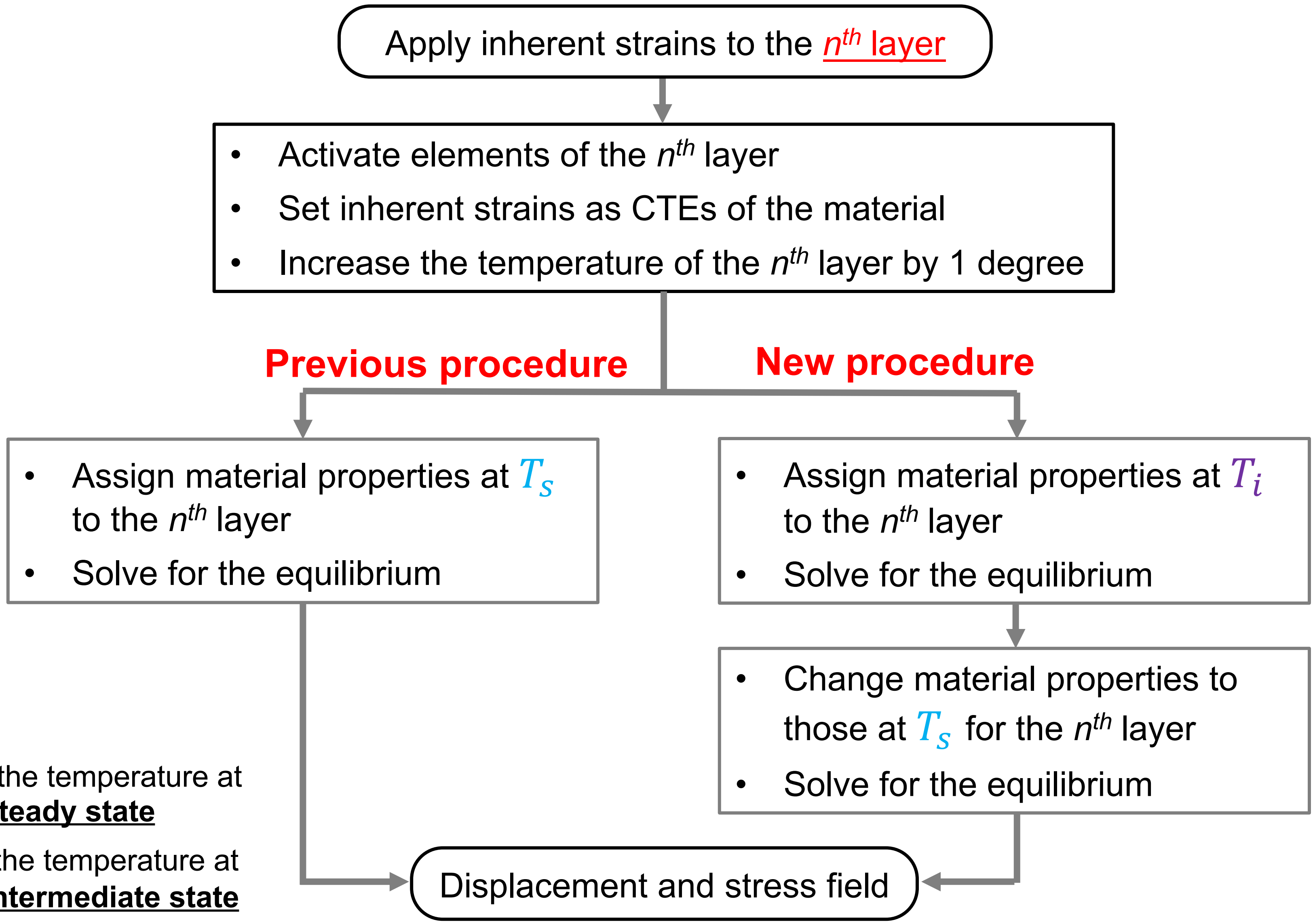
Apply inherent strains (layer-by-layer)



Q. Chen, A. C. To, et al., "An inherent strain based multiscale modeling framework for simulating part-scale residual deformation for direct metal laser sintering," *Additive Manufacturing*, vol. 28, 406-418, 2019.

X. Liang, A. C. To, et al., "Modified inherent strain method for fast prediction of residual deformation in direct metal laser sintered components," *Computational Mechanics*, vol. 64, 1719-1733, 2019.

Implementation Procedure

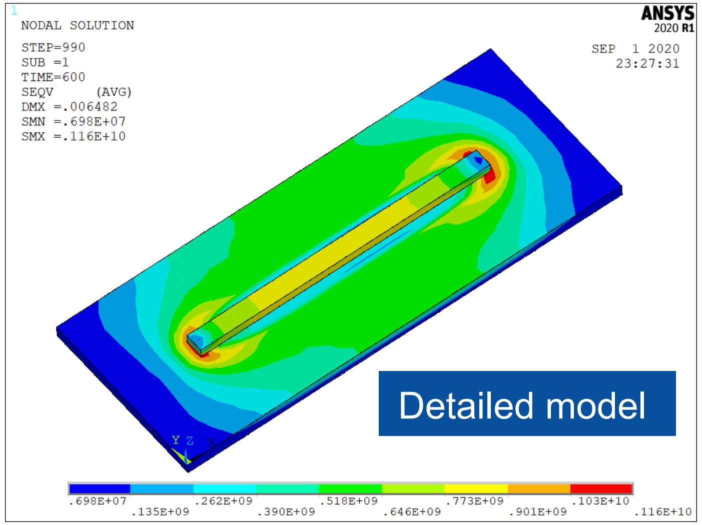


* T_s is the temperature at the **steady state**

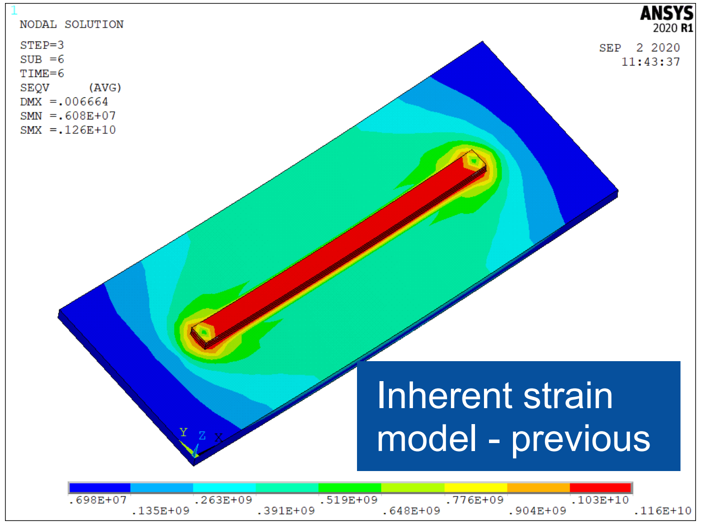
* T_i is the temperature at the **intermediate state**

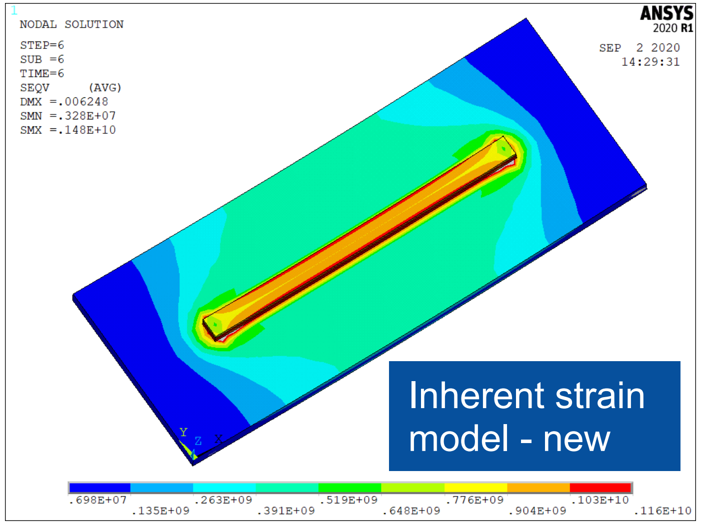
Residual Stress Comparison

A three-layer single track deposit with 17-4PH



Mises stress (Pa) after deposition





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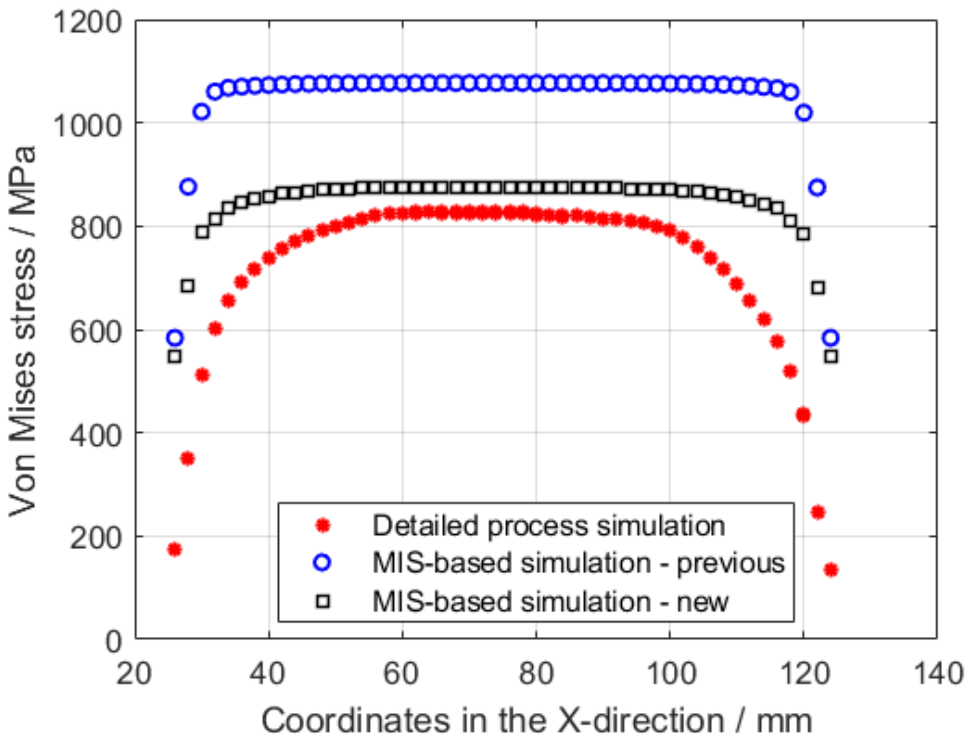
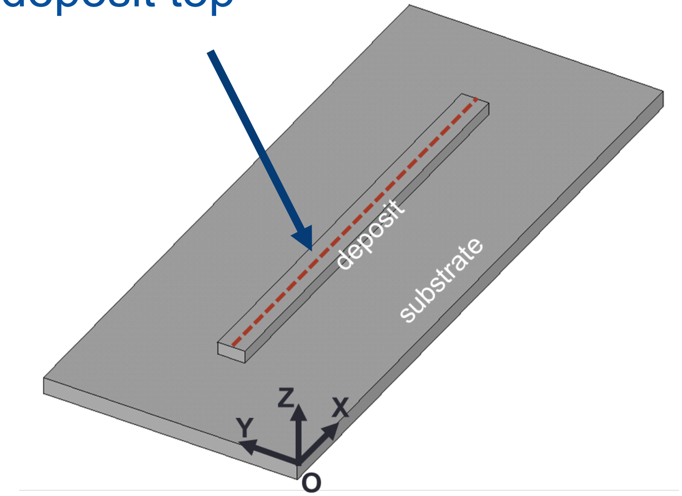


von Mises stress (MPa) along the centerline on the deposit top

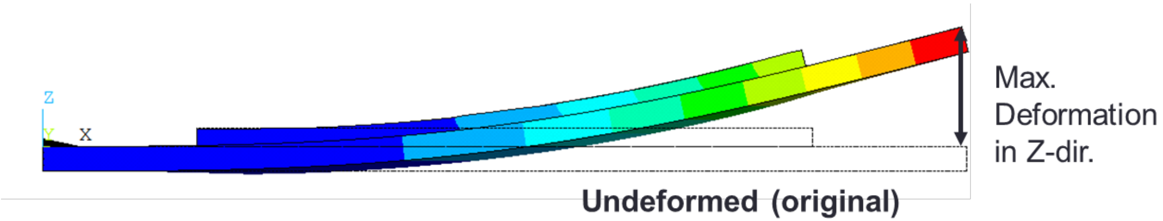


W. Dong, X. Liang, Q. Chen, S. Hinnebusch, Z. Zhou, and A. C. To, "A new procedure of implementing modified inherent strain model for improving prediction accuracy of both residual stress and deformation in laser powder bed fusion parts" in preparation.

Residual Distortion Comparison

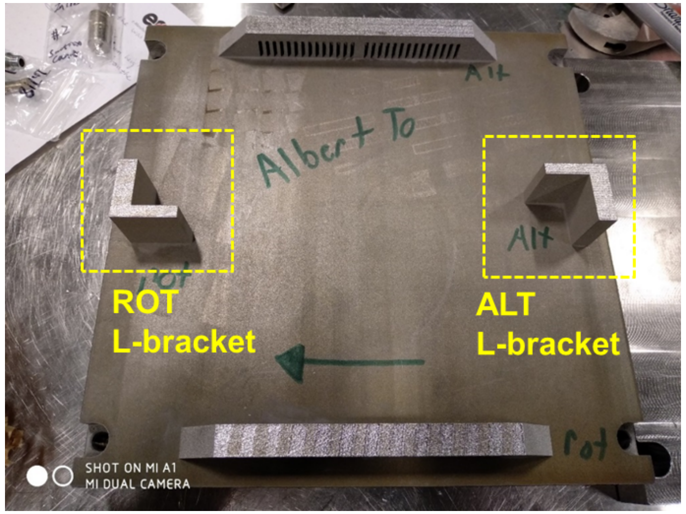
A three-layer single track deposit with 17-4PH

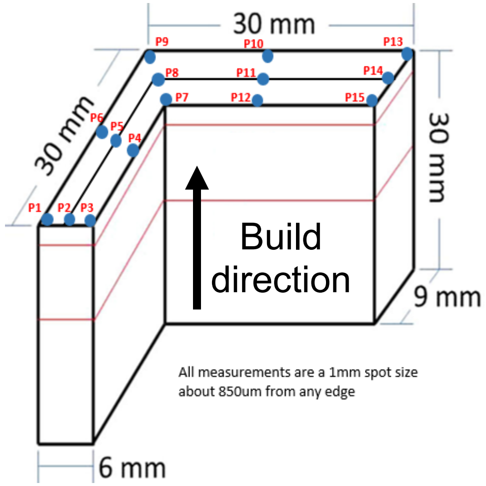
	Max. deformation in Z-dir.	Error
Detailed model	6.477 mm	-
Inherent strain model – previous procedure	6.660 mm	2.8%
Inherent strain model – new procedure	6.244 mm	3.6%

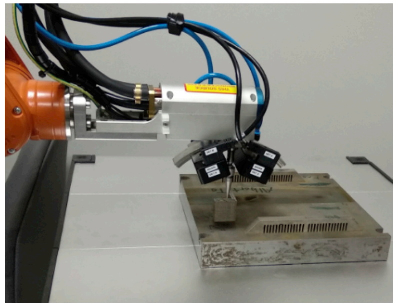


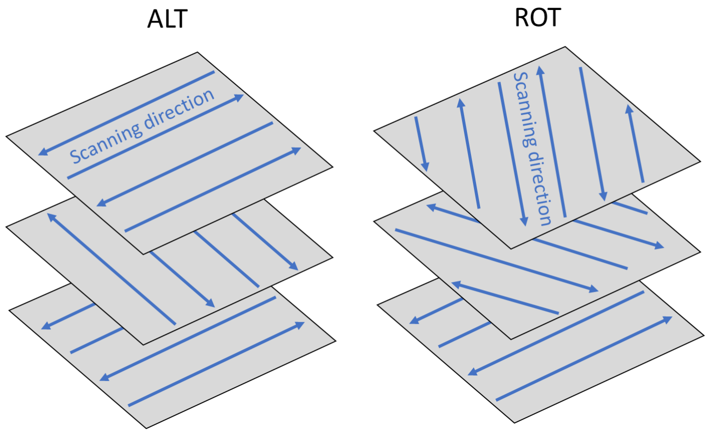
Experimental Validation

L-brackets with 17-4PH and 316L









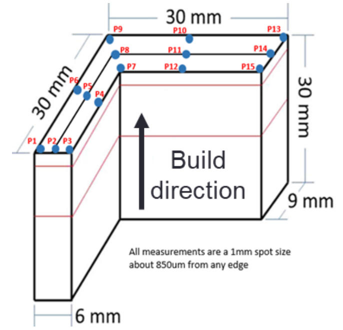
- 15 points (P1 - P15) are selected for X-ray diffraction (XRD) residual stress measurement

- ROT - angle difference between adjacent layers is 66.7°
- ALT - angle difference between adjacent layers is 90°

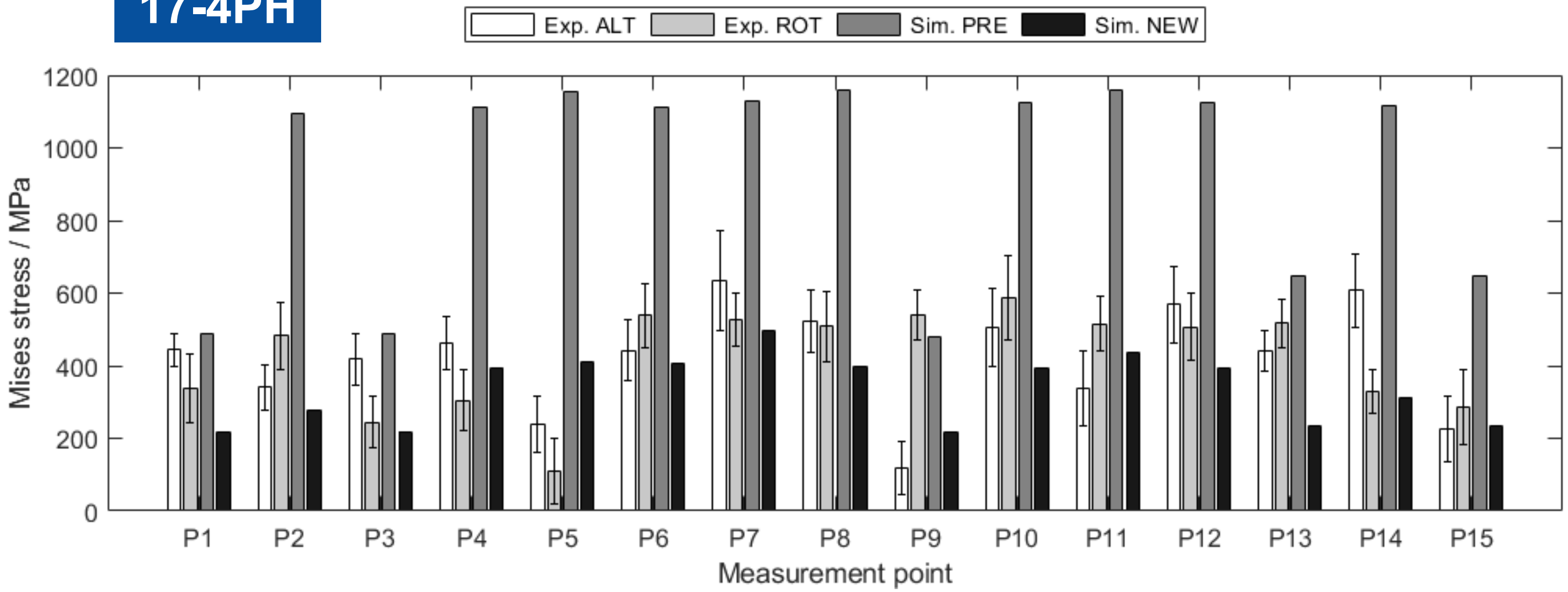
Experimental Validation

L-brackets with 17-4PH and 316L

- **Exp. ALT** - XRD measurement for samples with ALT scanning path
- **Exp. ROT** - XRD measurement for samples with ROT scanning path
- **Sim. PRE** - inherent strain model w/ previous procedure
- **Sim. NEW** - inherent strain model w/ new procedure



17-4PH



Experimental Validation

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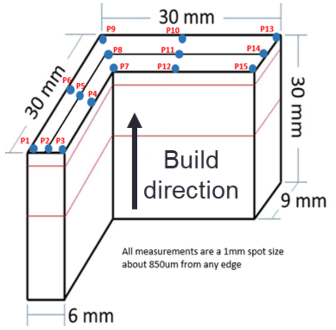
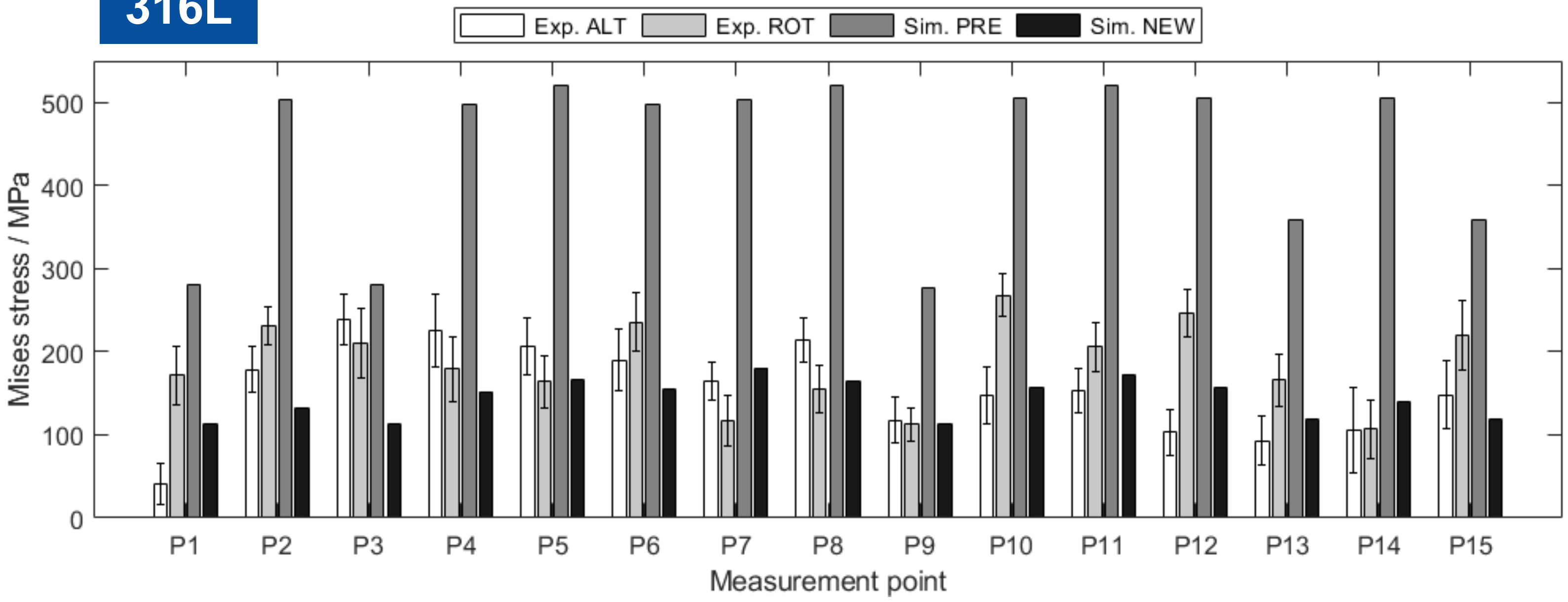
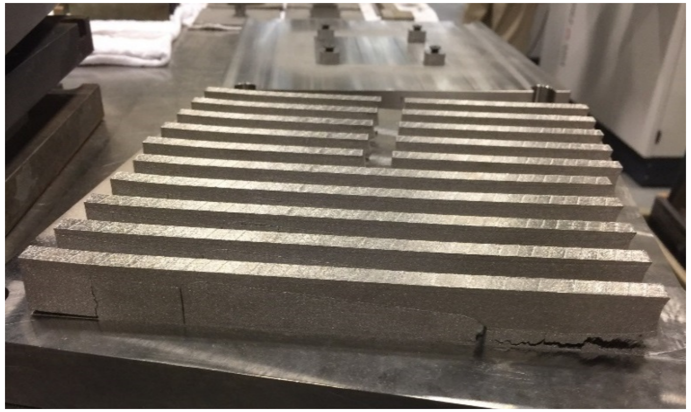


316L

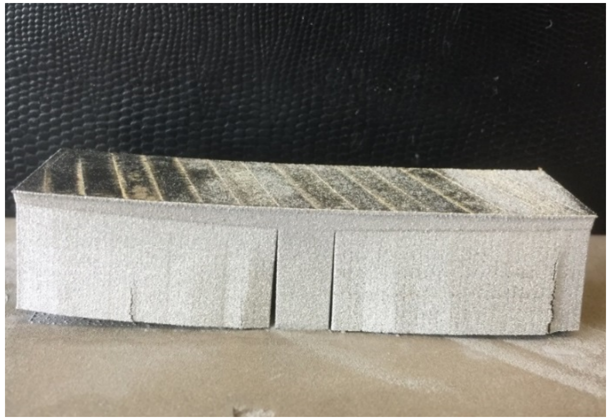


Support Structure Optimization

- Objective: Reduce build failure and deformation after cutting



Build failure



Deformation

- Design of support structures driven by topology optimization
- Optimization problem based on the minimization of the p-norm stress (maximal stress if P is high)

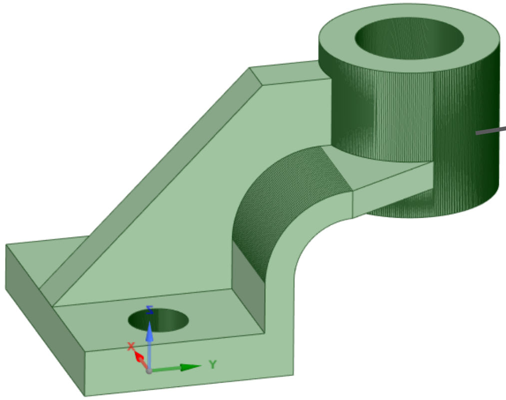
$$\min_{\rho_e} \sigma^{PN} \quad s.t \begin{cases} KU = F^{in} \\ C = C(\rho) \\ \rho_{min} \le \rho_e \le \rho_{max} \end{cases}$$

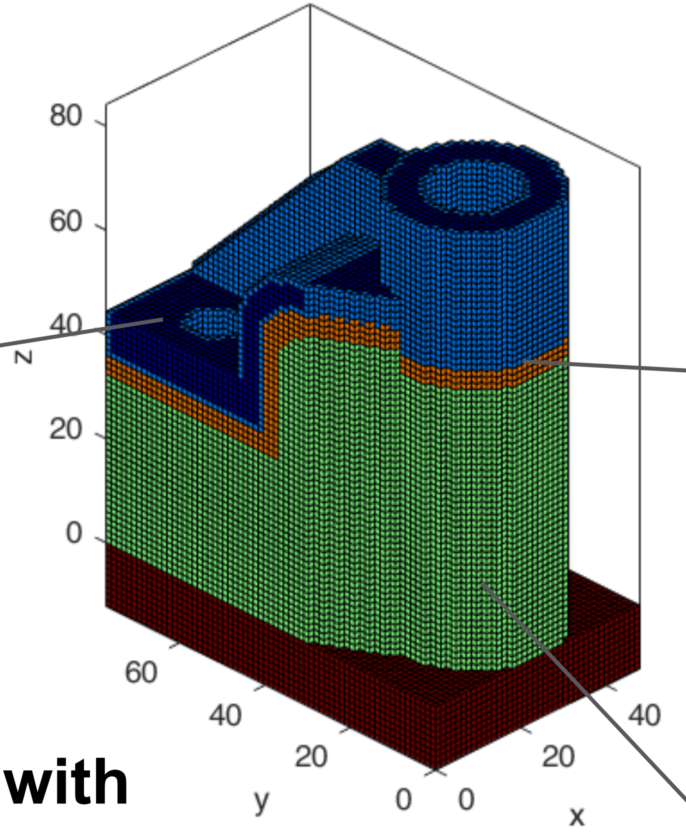
$$\sigma^{PN} = \left(\sum_{e=1}^{N} (\sigma_e)^P \right)^{\frac{1}{P}}$$

$$\sigma_e = \sqrt{(\bar{\boldsymbol{\varepsilon}} - \boldsymbol{\varepsilon}^{in})^T \mathbb{R} (\bar{\boldsymbol{\varepsilon}} - \boldsymbol{\varepsilon}^{in})}$$

Dissolvable Support Bracket Optimization Setup

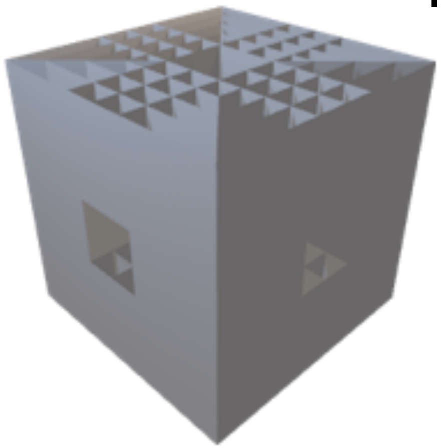
Bracket

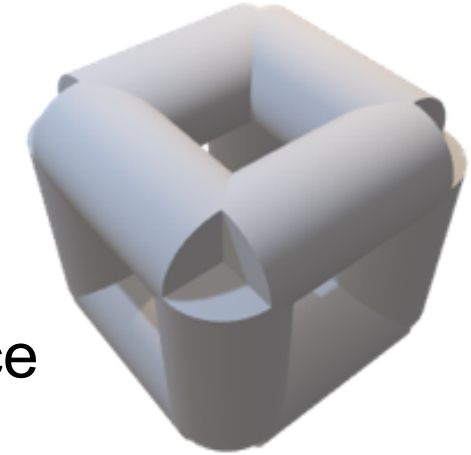




Bracket with support structure

Thin-walled cell for dissolvable support





Cubic lattice

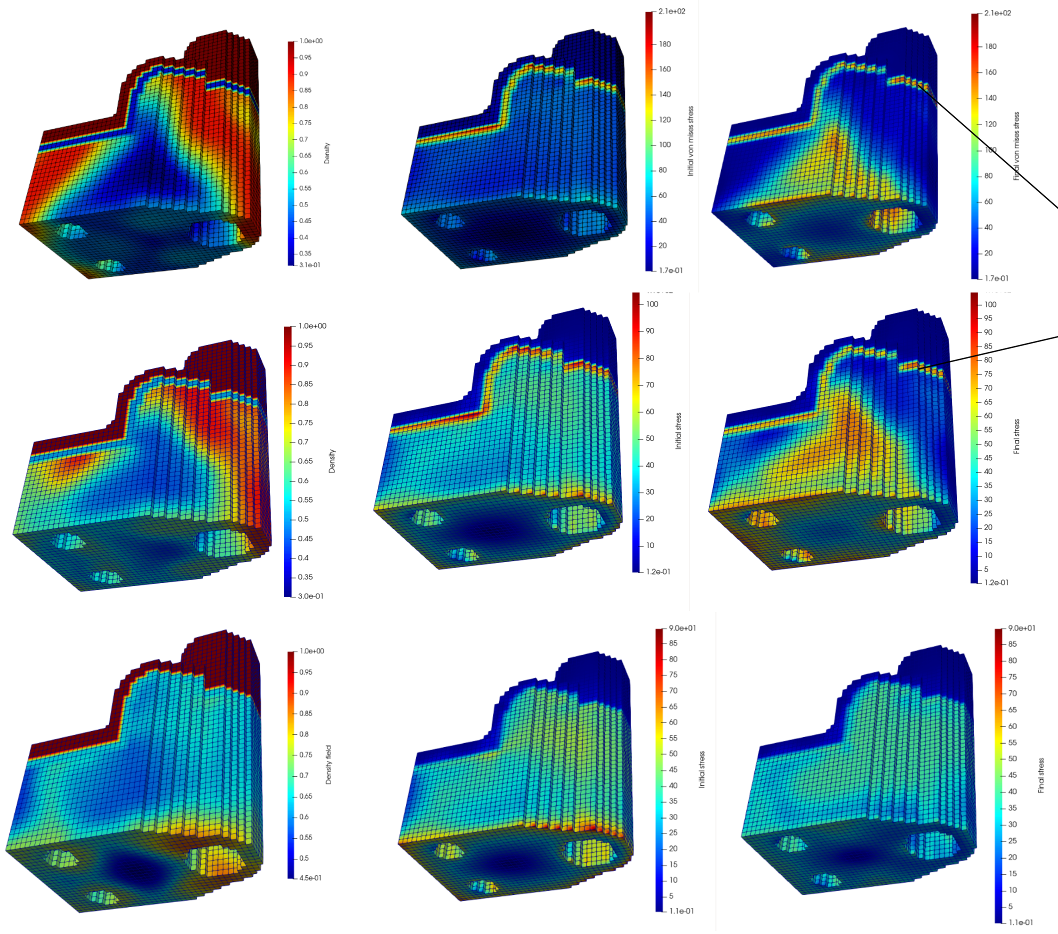
Support Optimization Results

Density | Initial stress | Optimized stress

Dissolvable support density = 0.4

Dissolvable support density = 0.5

Dissolvable support density = 0.64



Residual stress still high at the interface

Maximum residual stress reduced by > 50%

Post-heat treatment design for 17-4 PH steels

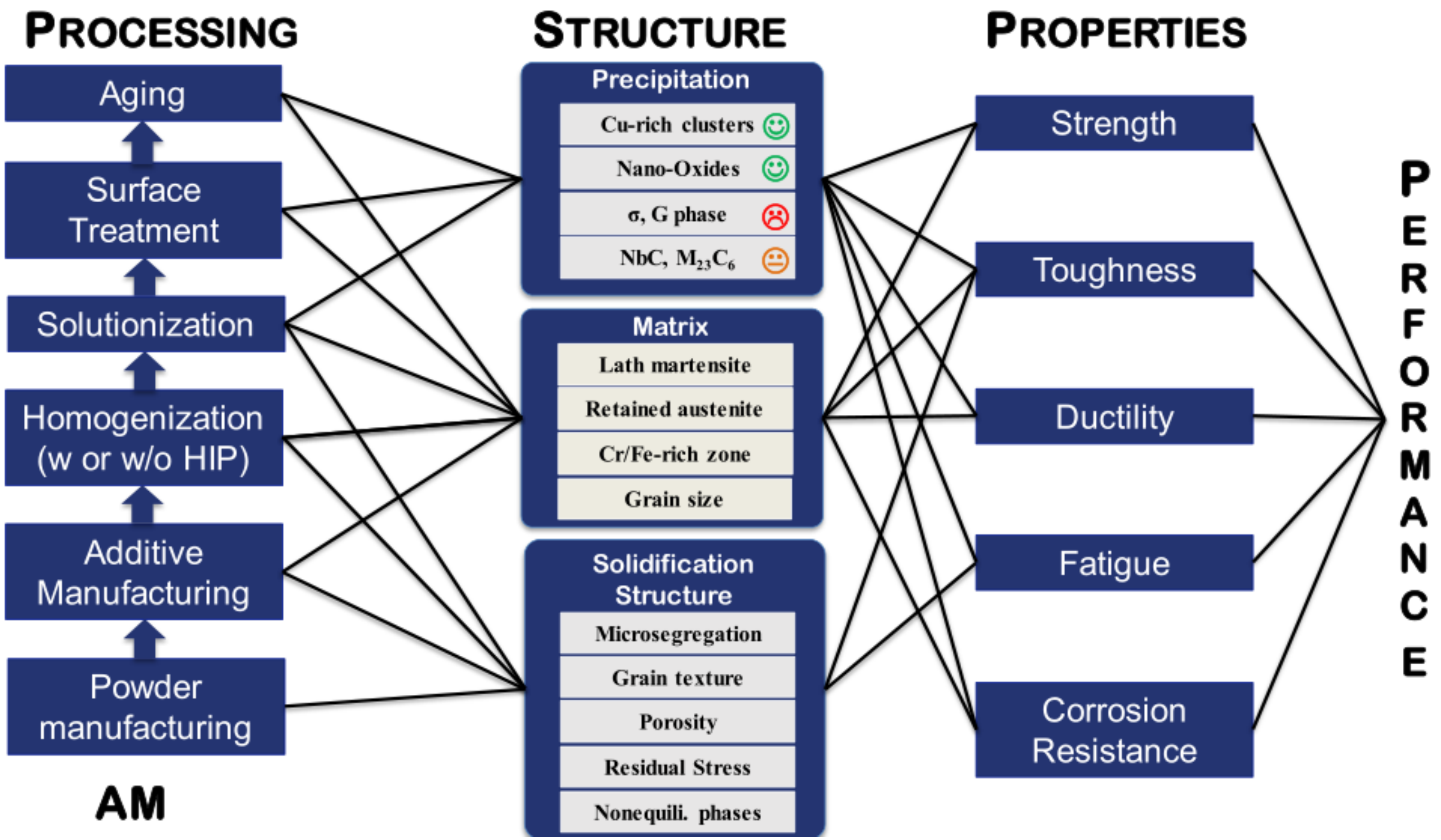
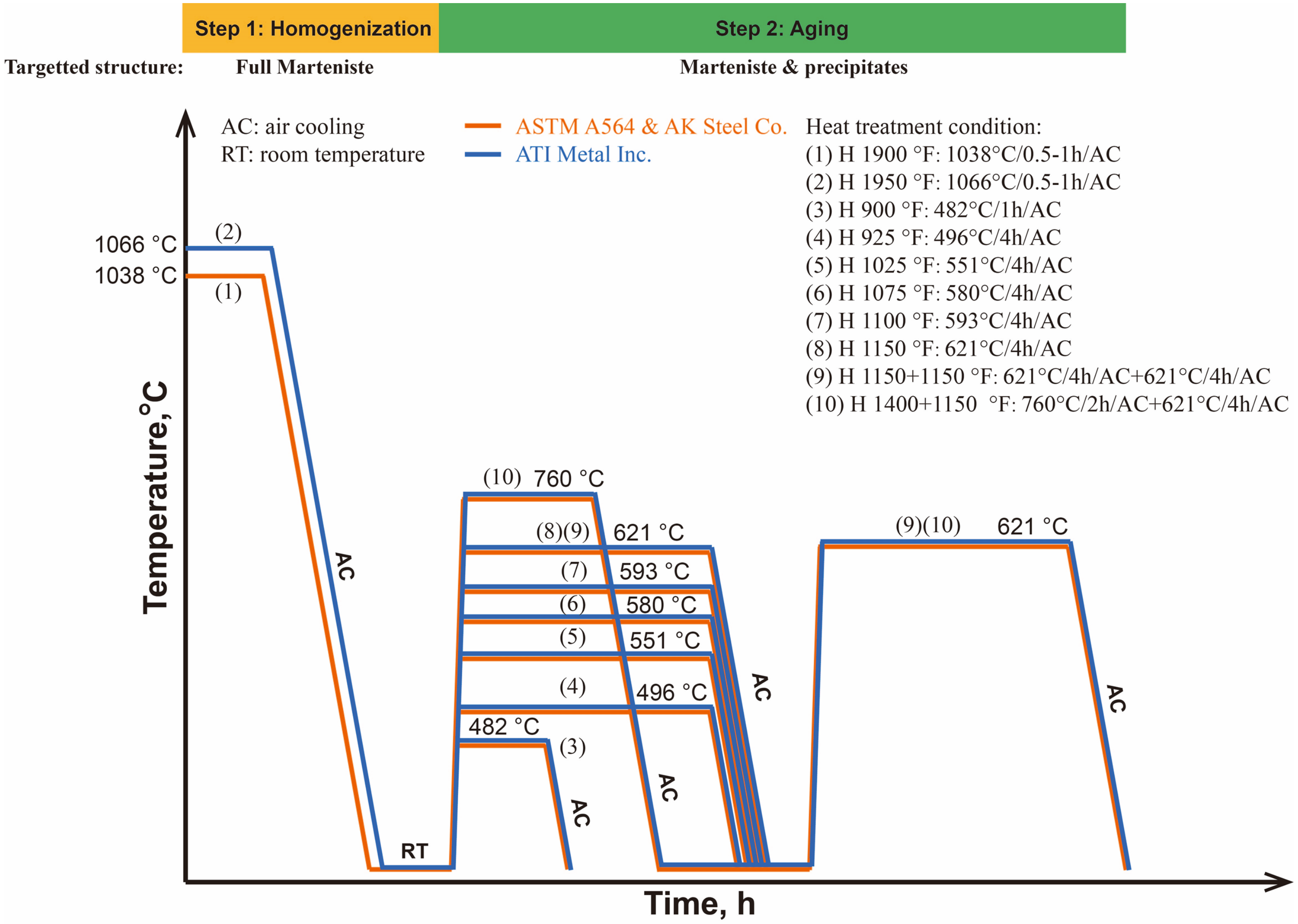


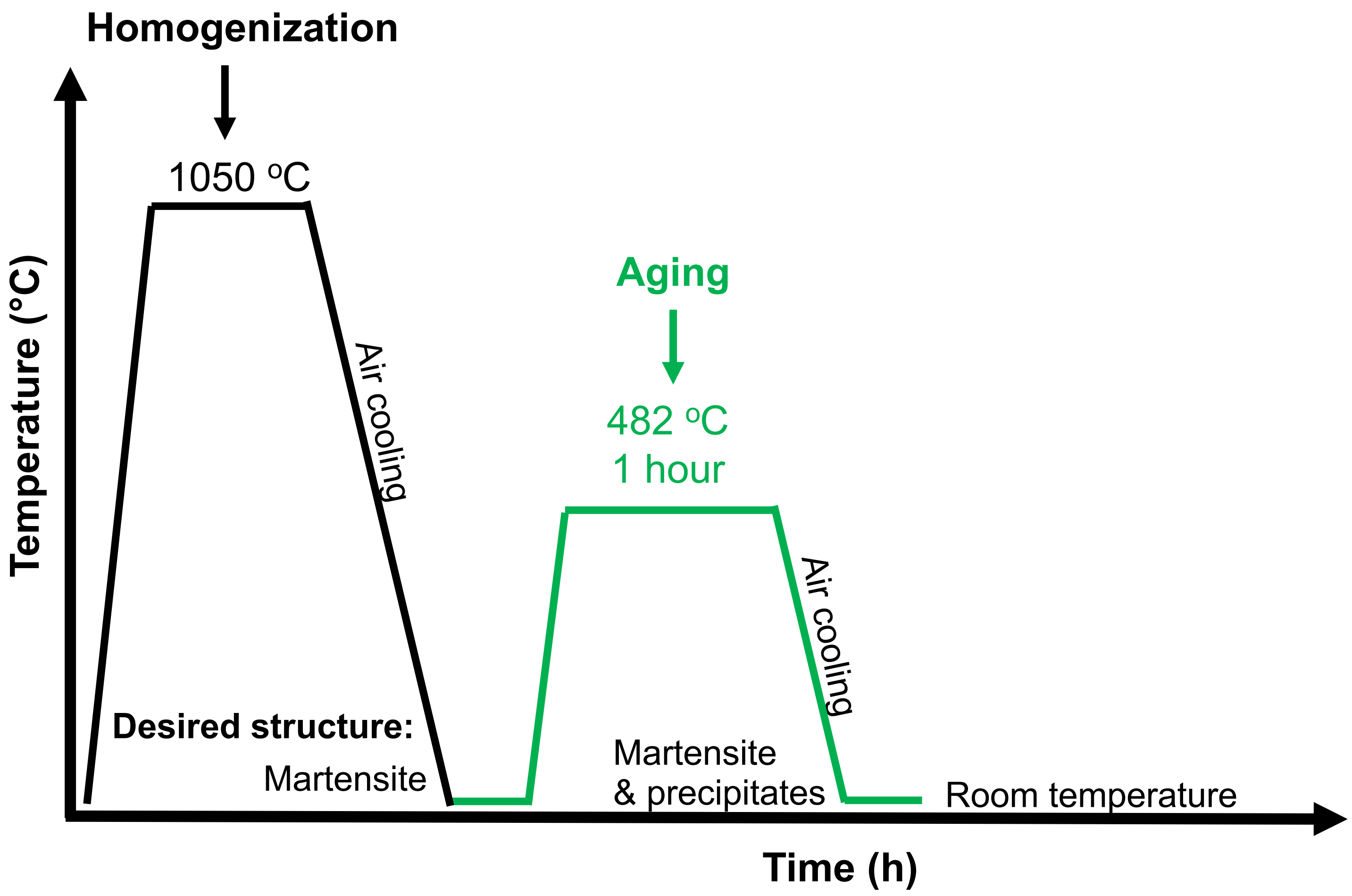
Table 1. The nominal composition of 17-4PH stainless steel powder (wt%).

Material	Cr	Ni	Cu	Nb	Mn	Si	C	O	N	Fe
17-4PH	15.84	4.55	3.87	0.37	0.32	0.36	0.019	0.05	0.01	Bal.

Conventional post-heat treatment for 17-4 PH steels



Investigation on Process-Microstructure-Property in this work for 17-4 PH steels

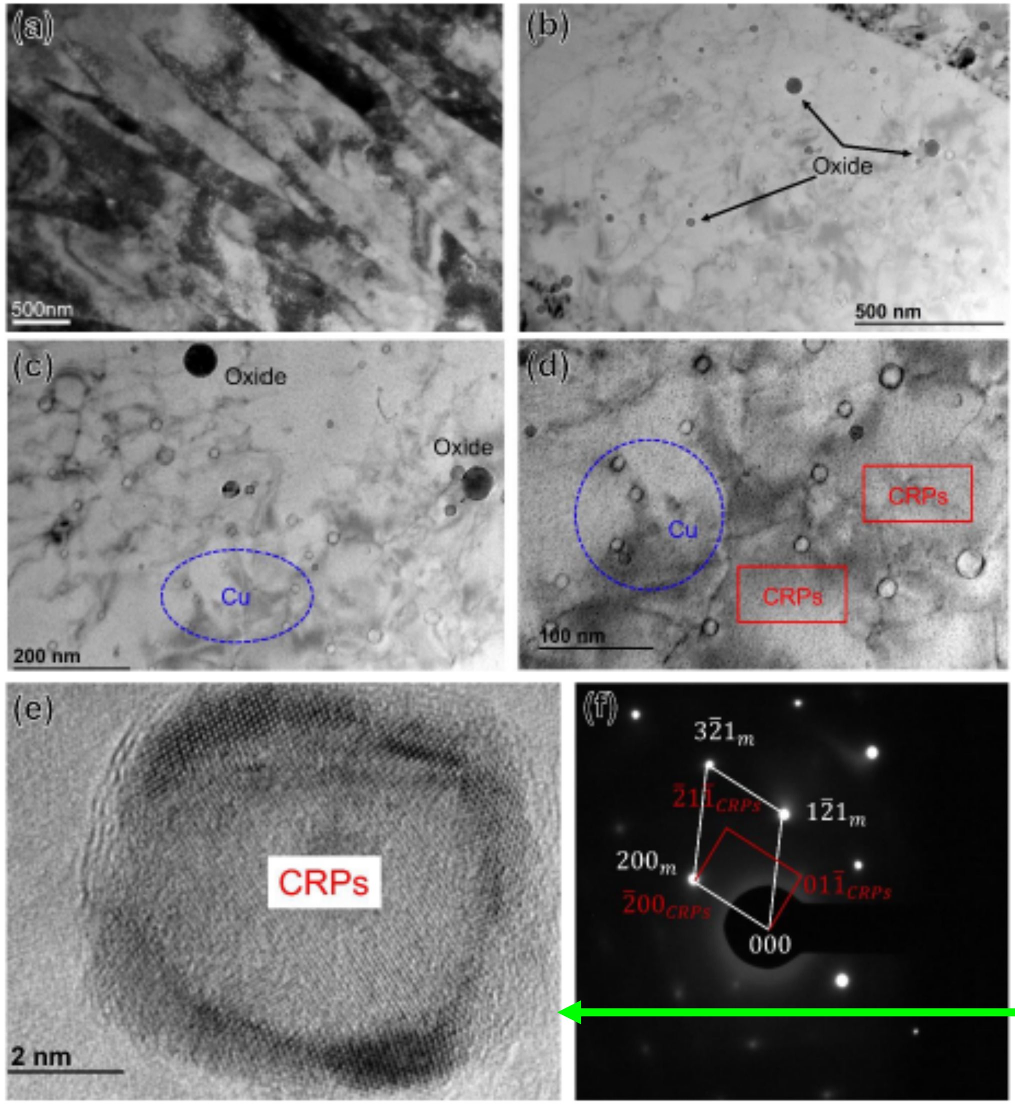


Homogenization:
Temperature: 1050°C
Time: 0 h, 0.5 h, 1 h, 2 h, 3 h and 4 h.

Aging:
Temperature: 482°C (900F)
Time: 1 h

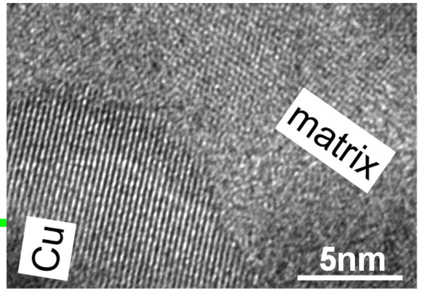
Sample notations:
AB: As-built
AB+H0.5A900: Homogenization at 1050°C for 0.5 hrs and aging at 482°C for 1 hr
AB+H1A900: Homogenization at 1050°C for 1 hr and aging at 482°C for 1 hr
AB+H4A900: Homogenization at 1050°C for 4 hrs and aging at 482°C for 1 hr
AB+H8A900: Homogenization at 1050°C for 8 hrs and aging at 482°C for 1 hr

Microstructure characterization

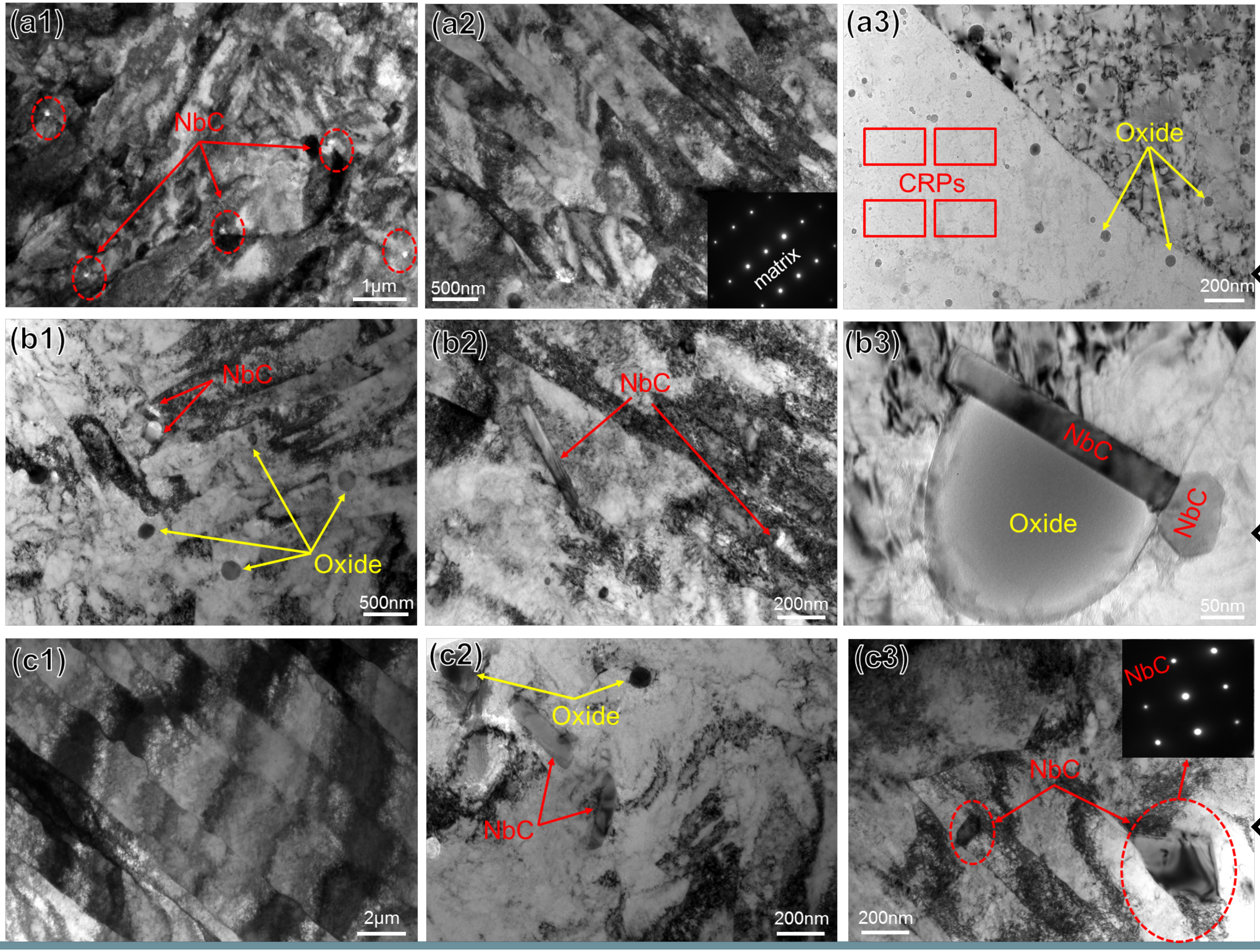


*TEM analysis on alloy "**AB+H0.5A900**":*
Homogenized at 1050C for 30 min + aging at 482C(900F) for 1 hour
(a) morphology of lath martensite,
(b) precipitates at low magnification,
(c) precipitates at high magnification,
(d) magnified zone of (c),
(e) high resolution morphology of CRP particle
(f) the corresponding selected area electron diffraction pattern of (e).

CRP: copper-rich precipitation



Impact of homogenization on precipitation during aging



TEM images of AM 17-4PH alloys

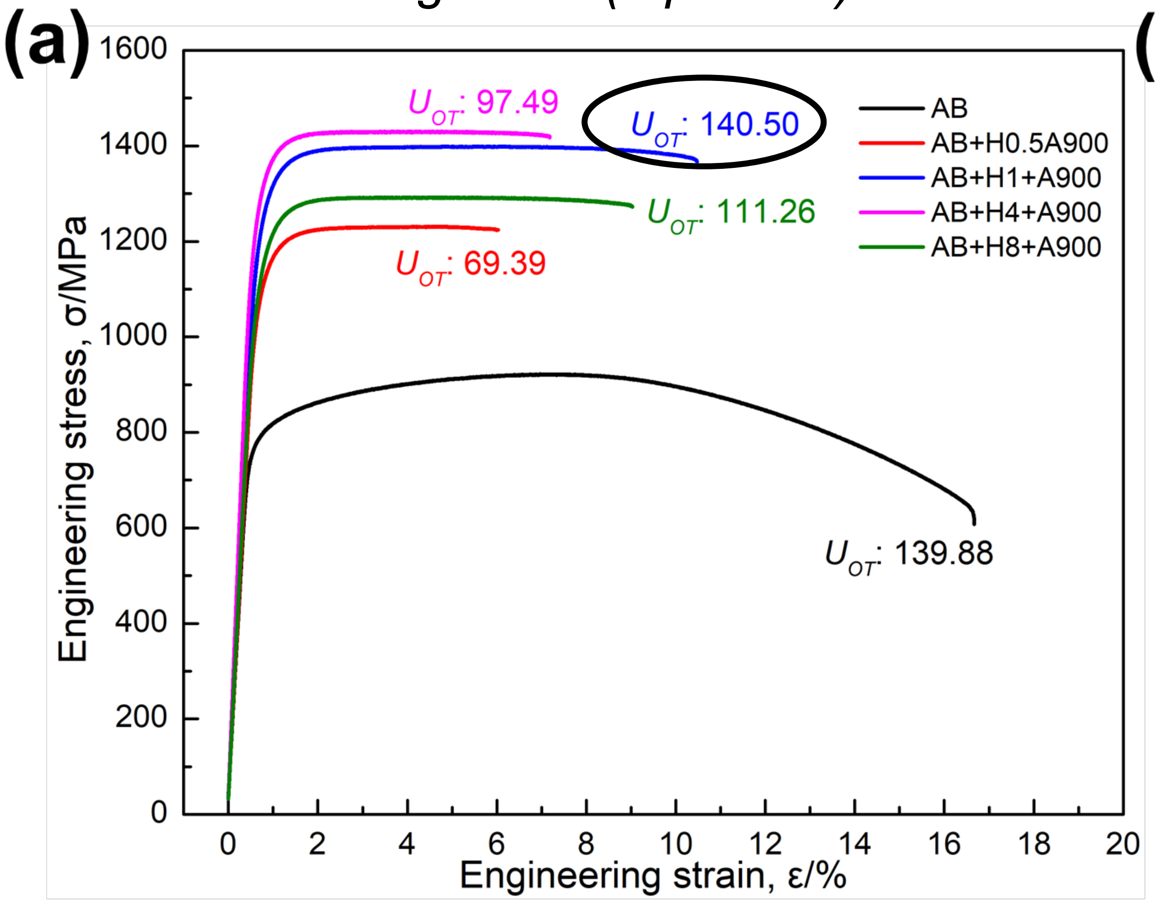
(a) AB+H1A900,
*Homogenized at 1050C for **1 hr** + aged at 482C(900F) for 1 hour*

(b) AB+H4A900
*Homogenized at 1050C for **4 hr** + aged at 482C(900F) for 1 hour*

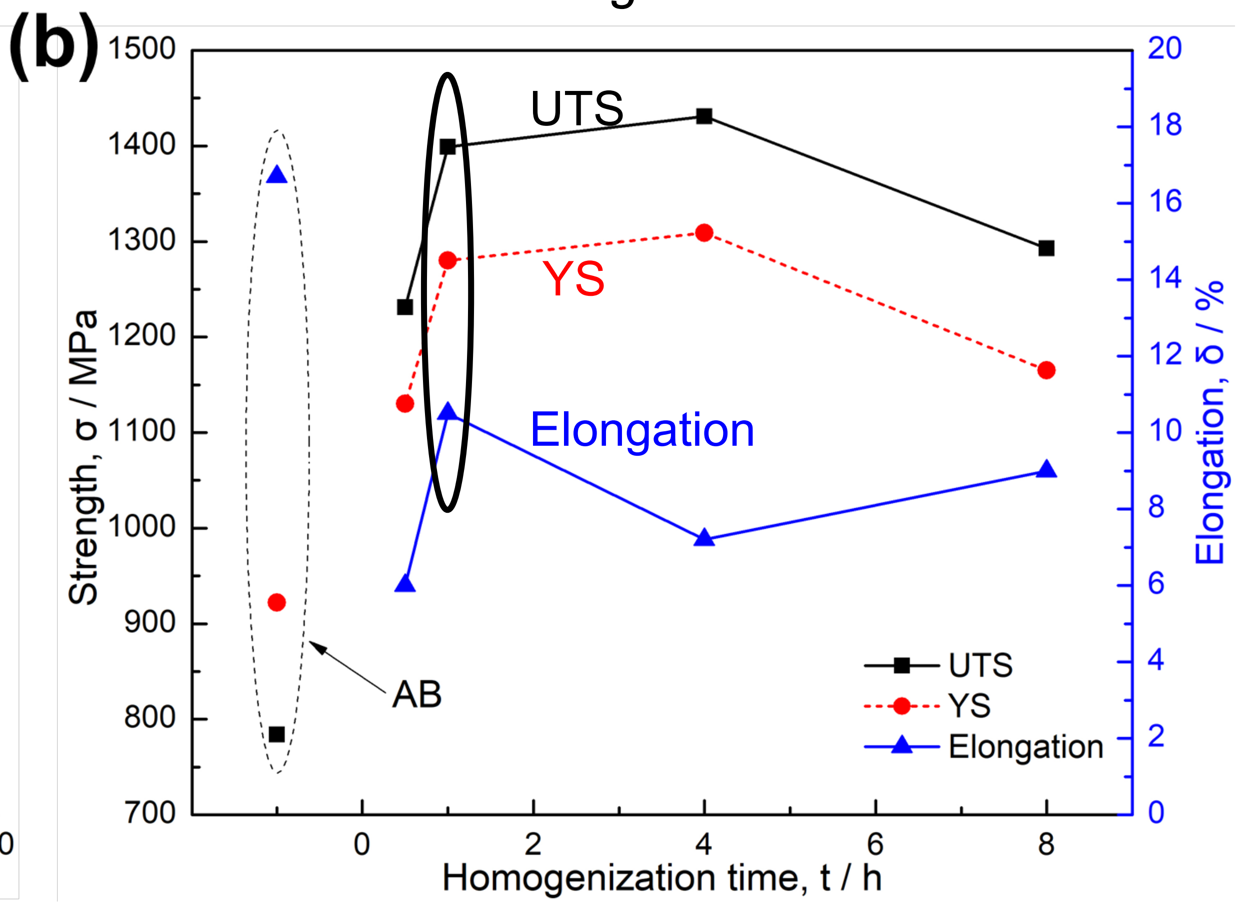
(c) AB+H8A900
*Homogenized at 1050C for **8 hr** + aged at 482C(900F) for 1 hour*

Tensile properties before and after post-heat treatment

Engineering strain-stress curves with static toughness (Mpa·m$^{1/2}$) values



Evolution of tensile properties versus homogenization time



Design simulation to integrate surface finishing and post-heat treatment

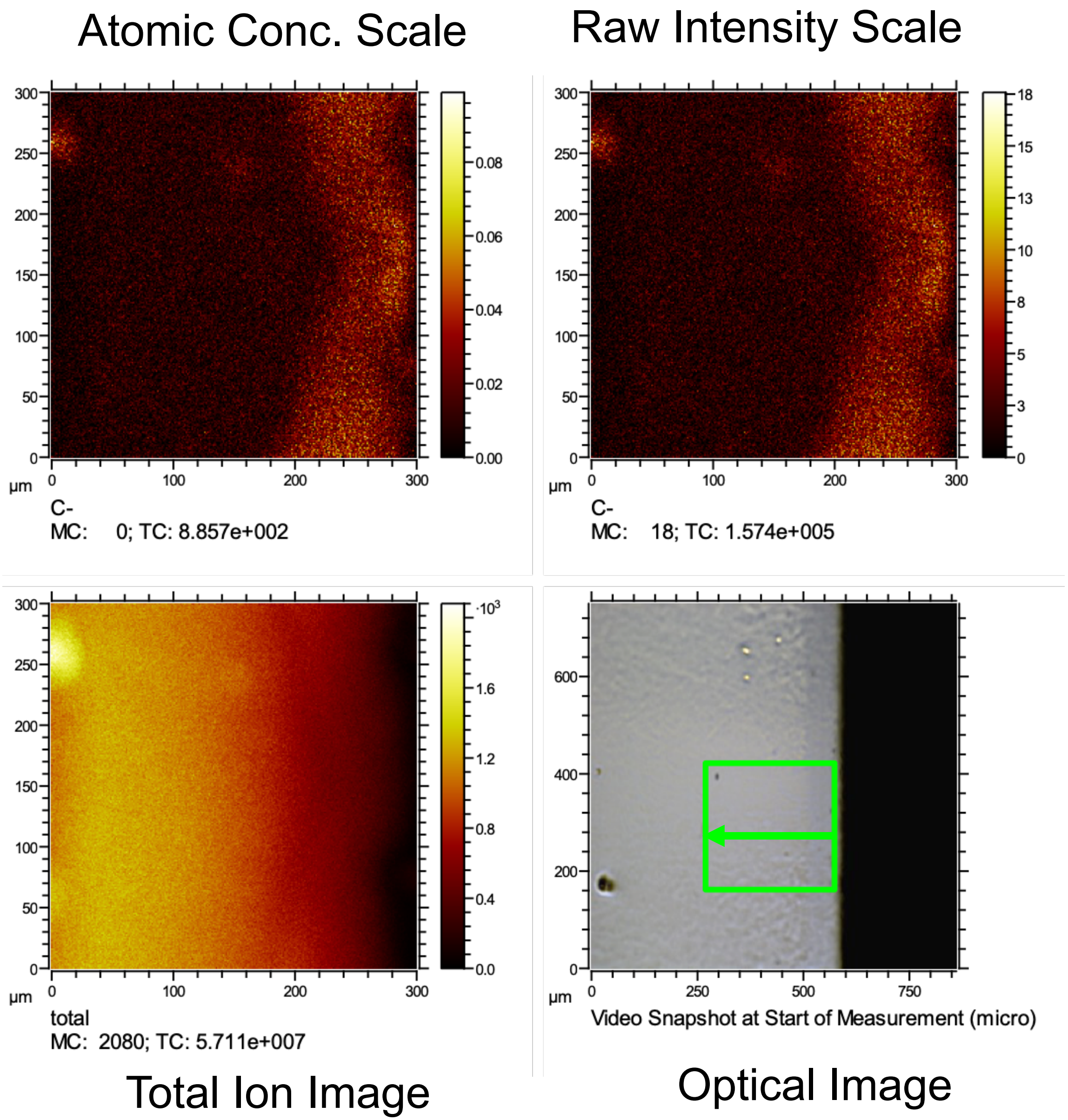
Temperature profile for surface treatment of SS316L

Cycle	Temperature (^oC)	Ramp rate (^oC/hour)	Dwell time (hour)
1	50	5	3
2	90	5	1
3	185	5	1
4	250	5	2
5	800	5	6

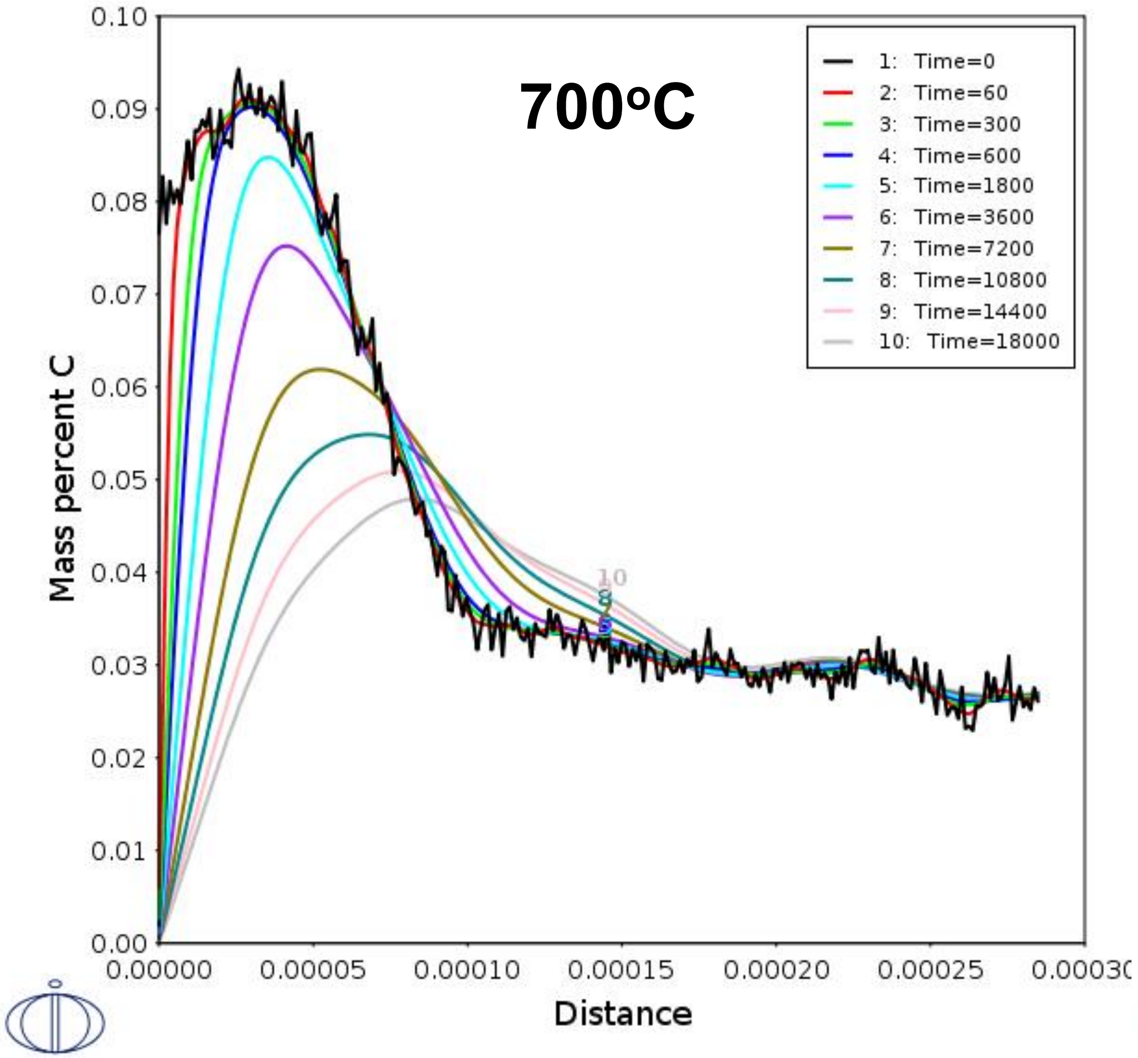
Cooling: furnace cooling for each cycle.

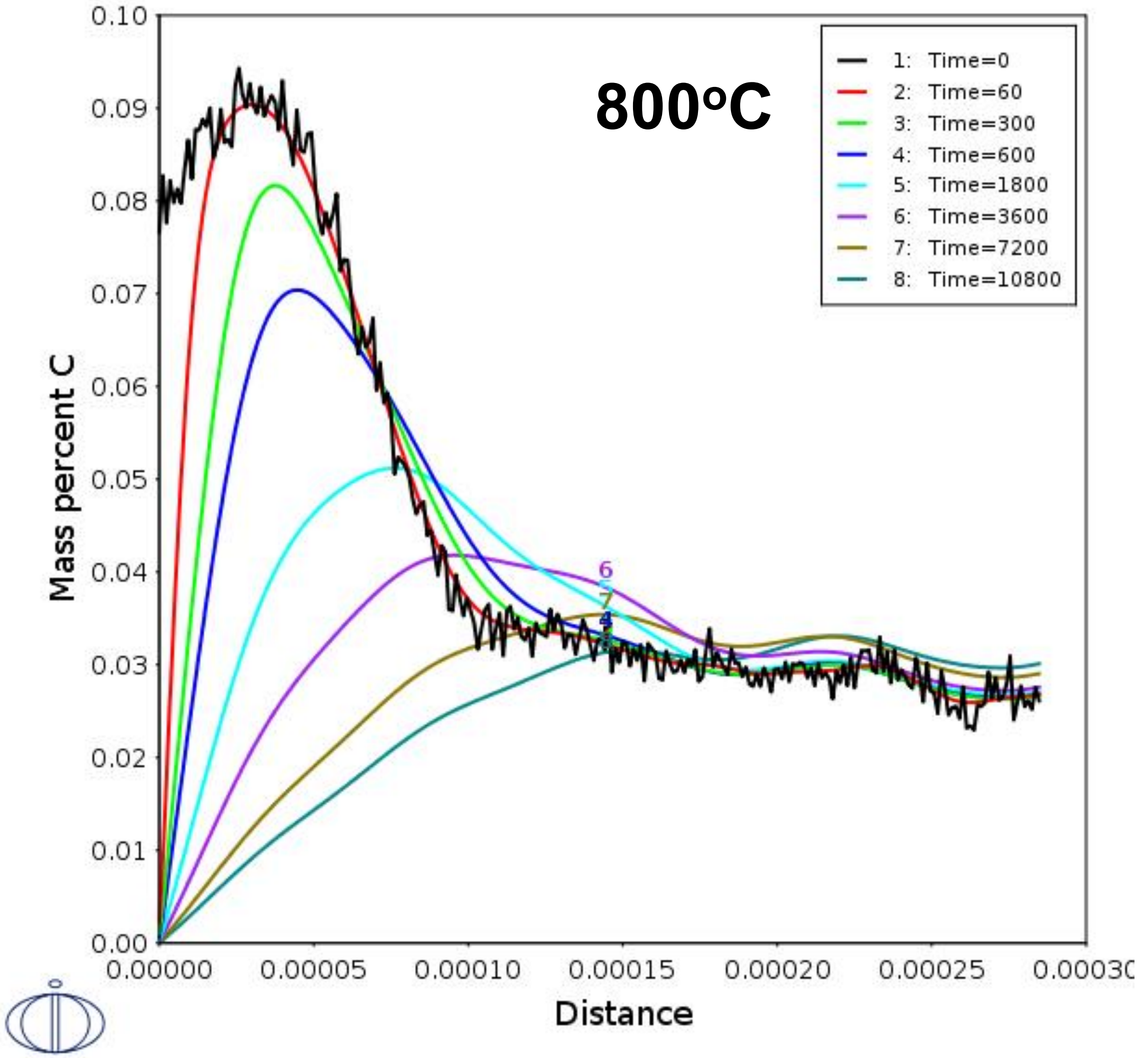
Carbon profile from SIMS
(Secondary-ion mass spectrometry)

- The concentration of carbon from the surface to the bulk was determined using secondary ion mass spectroscopy (SIMS) after surface treatment (Co-PI Owen Hildreth's lab at Colorado School of Mines)
  - The green box on the optical image marks the scan region
- Assuming 0.09 at.% carbon in the sample, the ion intensity was converted to a relative concentration

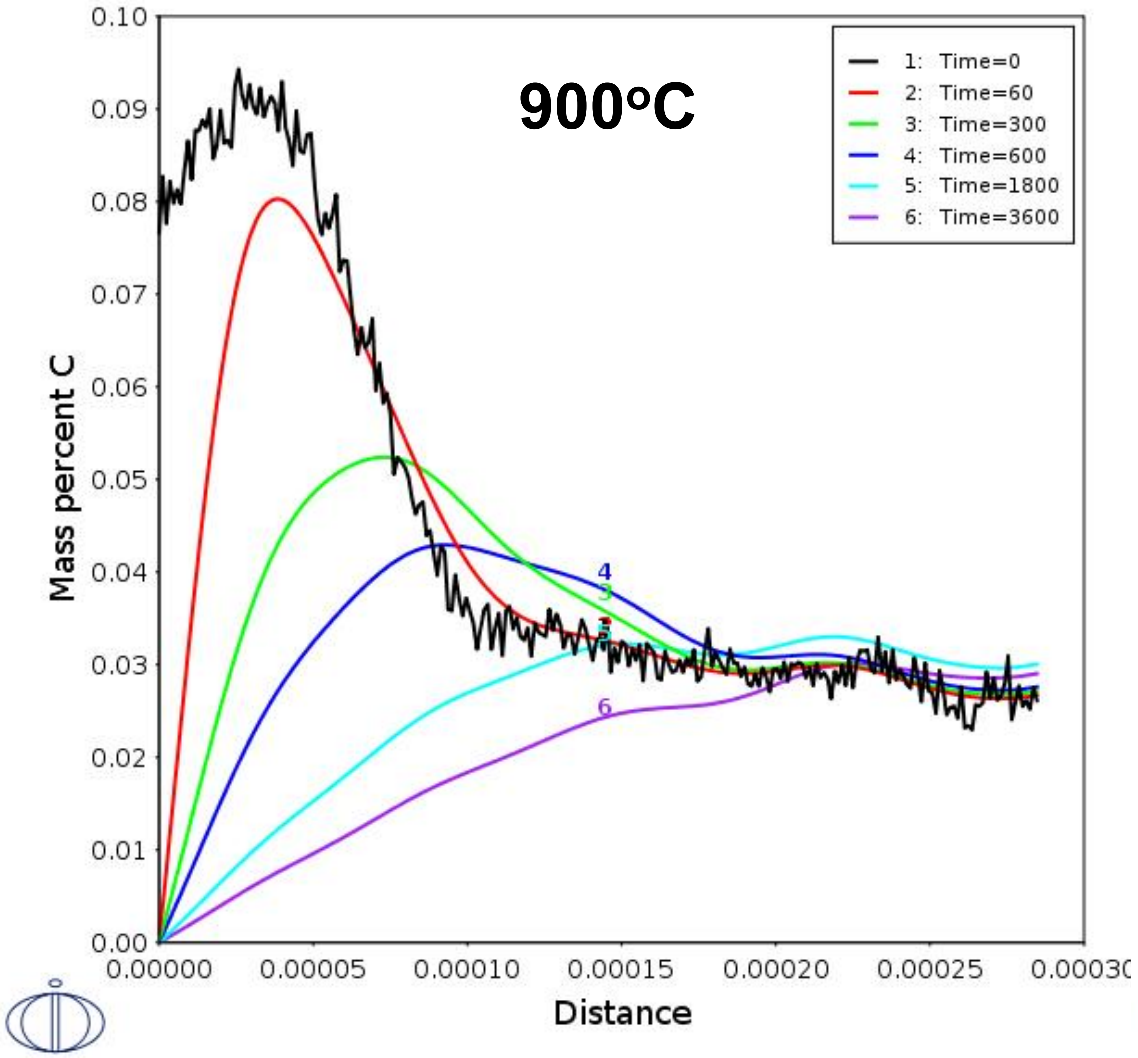


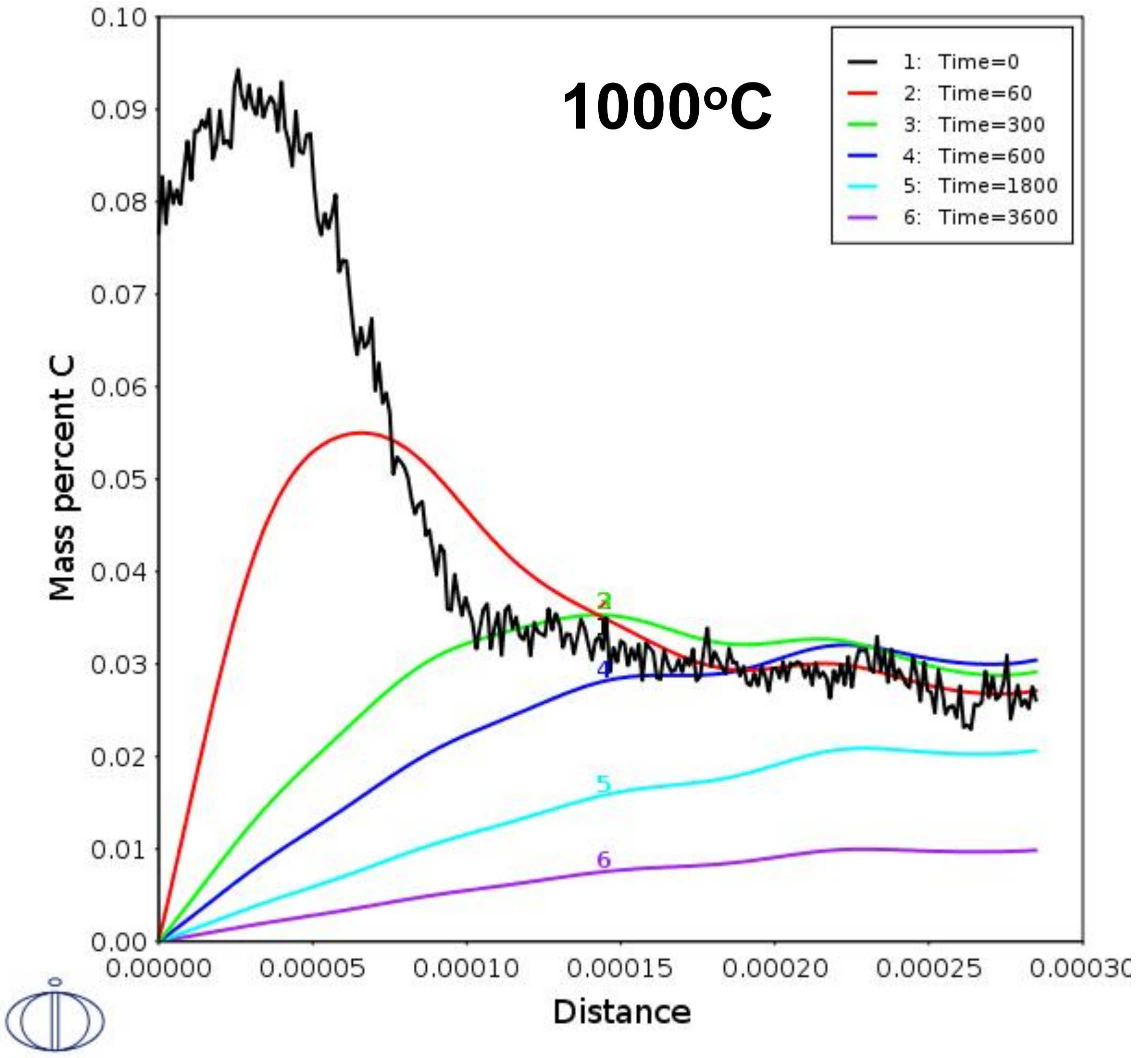
Composition profile as a function of distance (316L)



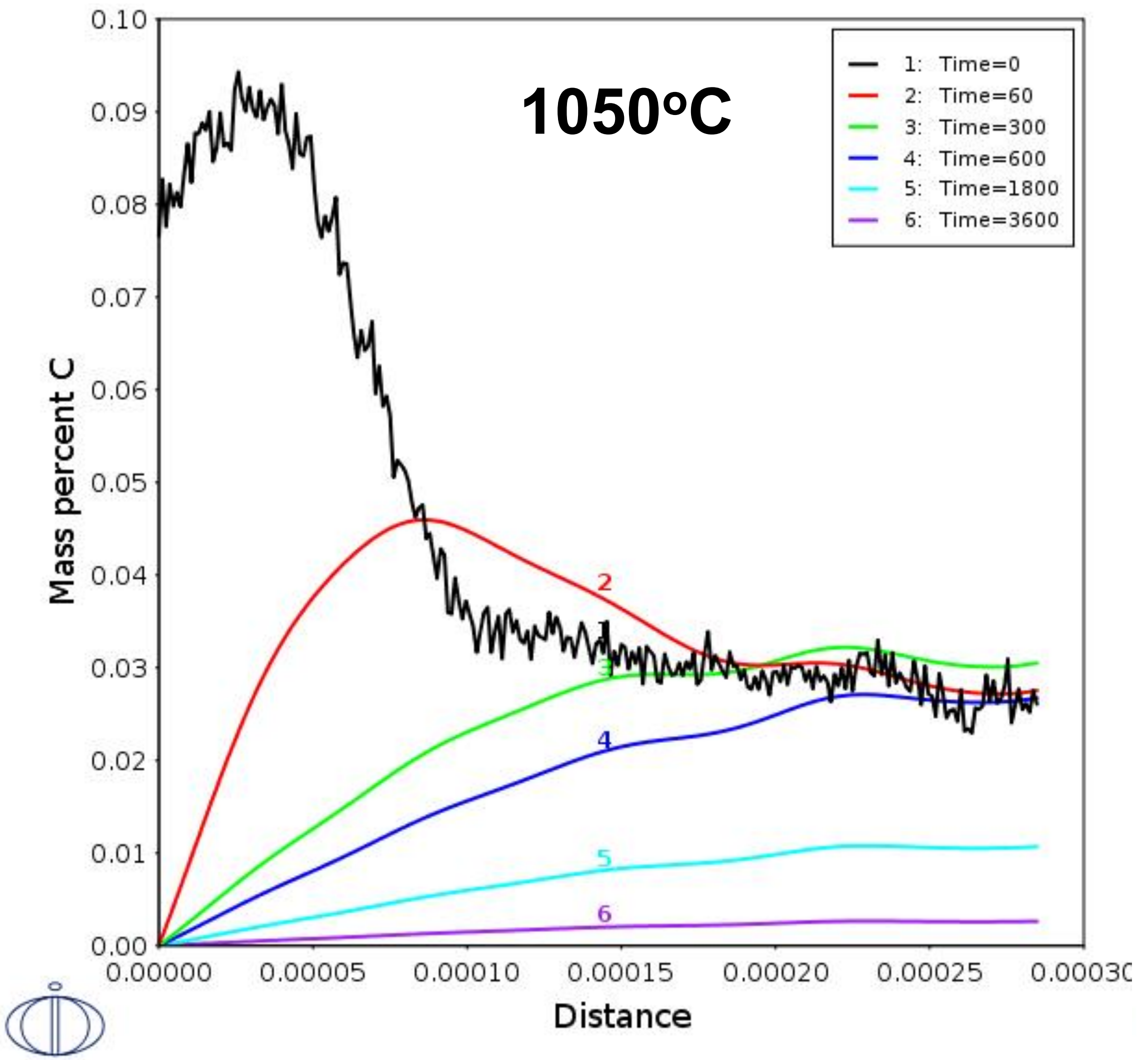


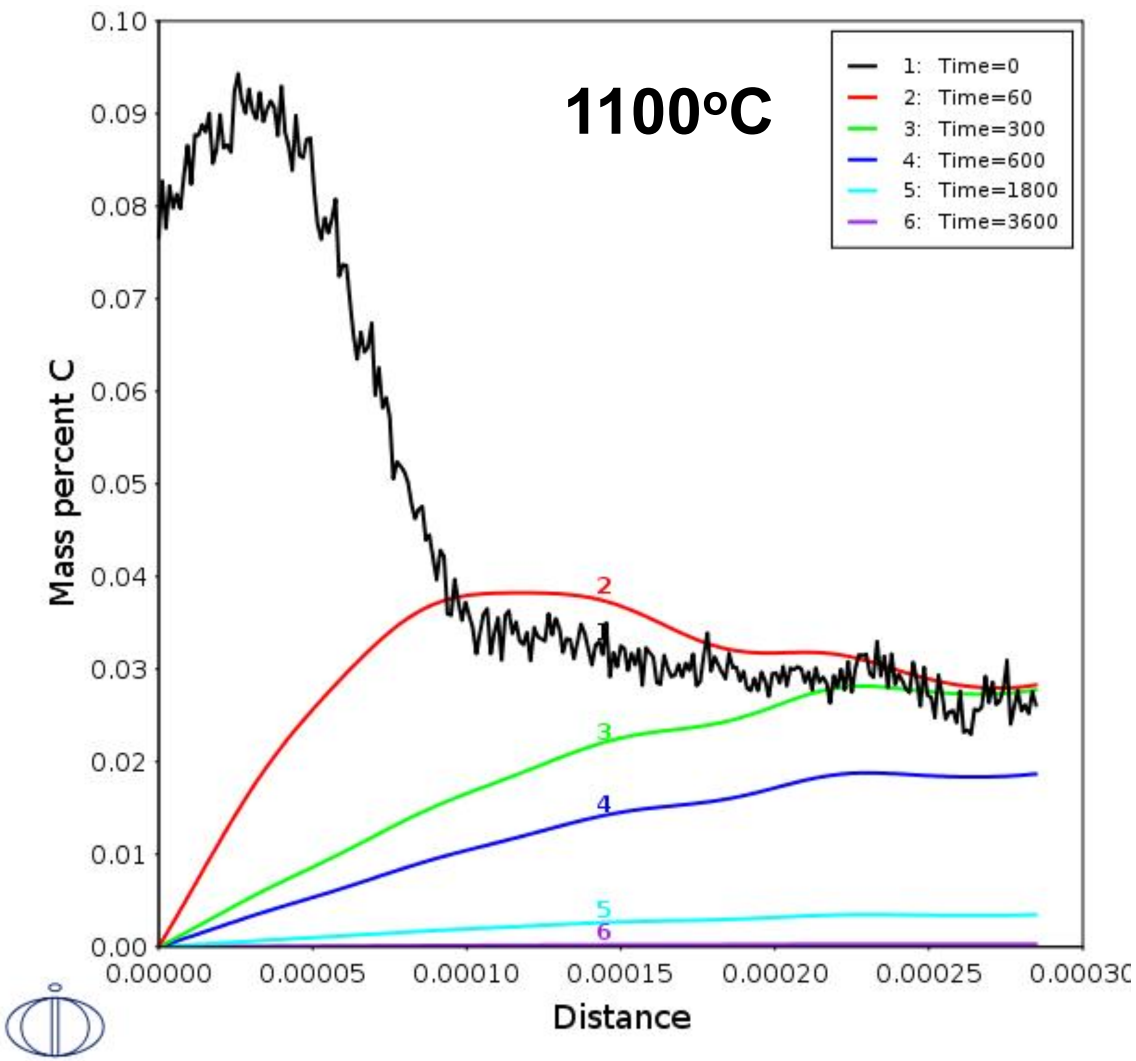
Composition profile as a function of distance (316L)



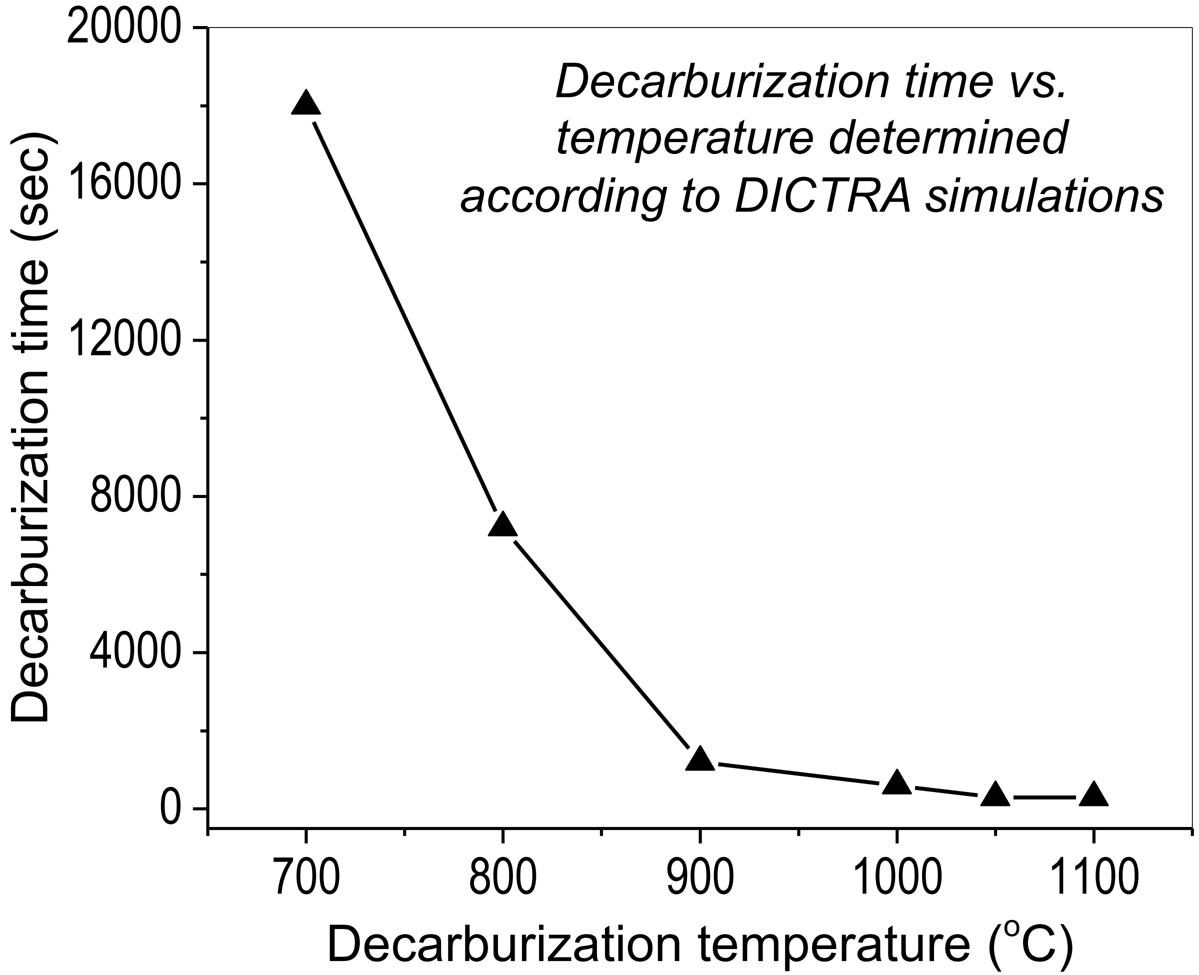


Composition profile as a function of distance (316L)





Decarburization temperature *vs.* time for SS316L

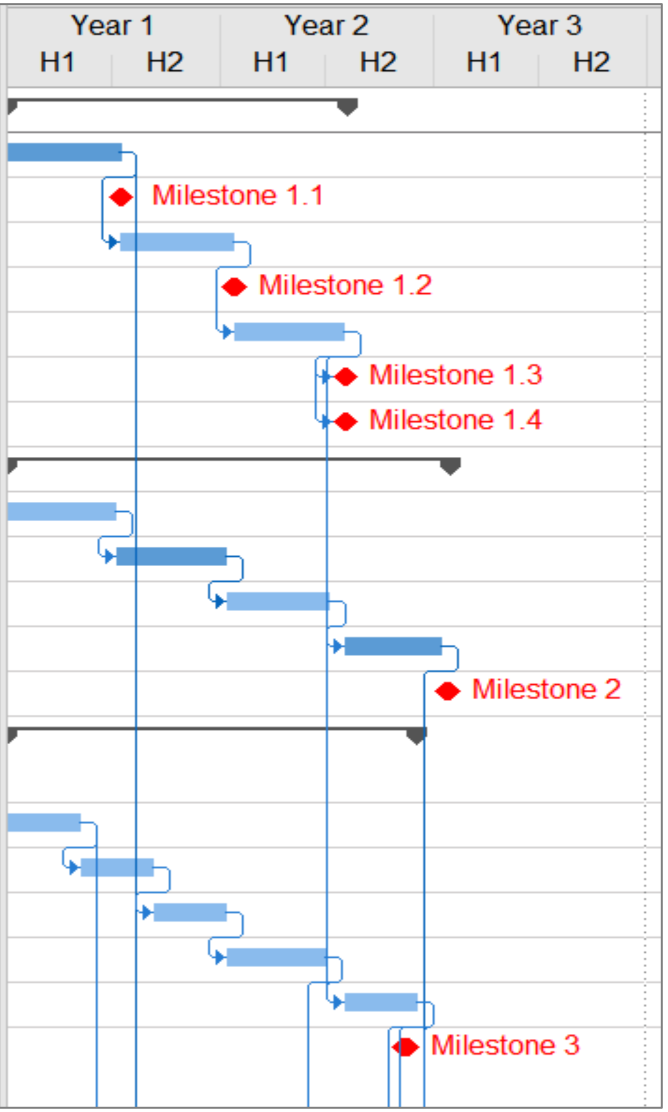


Project Impacts

- Journal Publications
  - R. Hoffman, S. Hinnebusch, S. Raiker, A. C. To, O. J. Hildreth, “Support Thickness, Pitch, and Applied Bias Effects on the Carbide Formation, Surface Roughness, and Material Removal of Additively Manufactured 316L Stainless Steel,” JOM, in press.
- Conference Presentations
  - W. Xiong, "CALPHAD-based ICME Design for Additive Manufacturing: Successes and Challenges”, Symposium: Additive Manufacturing: ICME Gap Analysis, TMS 2020, San Diego, CA, February 23-27, 2020.
  - S. Hinnebusch, K. Glunt, R. Hoffman, O. J. Hildreth, A. C. To “Additive Manufacturing Laser Powder Bed Fusion Optimization for Dissolvable Supports with SS 316L”, Symposium: Additive Manufacturing: Mechanical Behavior of Lattice Structures Produced via AM, MS&T20, Virtual event, November 2-6, 2020.
  - K. Glunt, S. Hinnebusch, W. Dong, X. Liang, F. Dugast, O. J. Hildreth, A. C. To "Design Optimization for Residual Stress in Complex Low-density Support Regions“, Symposium: Additive Manufacturing Modeling Simulation: AM Materials, Processes, and Mechanics, MS&T20, Virtual event, November 2-6, 2020.

Milestones and Deliverables for FY-20

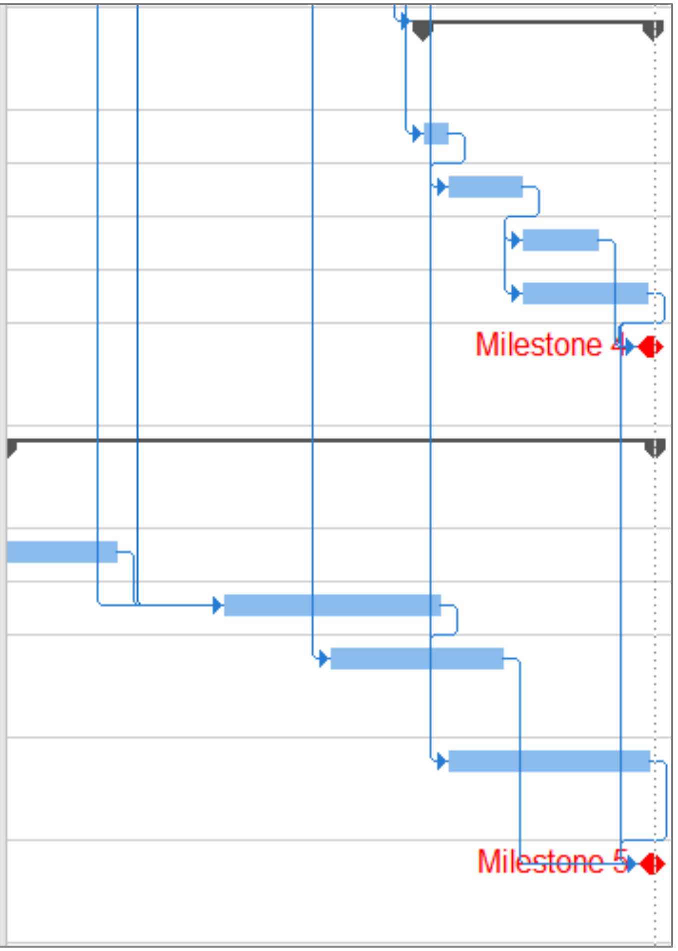
W	Task Name
1	Develop and validate recipes to dissolve support structures
1.1	Collect DLERP curves across the sensitization depth
M1.1	Identified bias and electrolyte for uniform etching
1.2	Measure microstructure and surface roughness of etched samples
M1.2	Identified bias and electrolyte for uniform etching w/ carbide removal
1.3	Optimize sensitization and dissolution process
M1.3	Surface roughness (Ra) less than 2 µm
M1.4	Composition matches bulk composition
2	Develop an automated support structure design tool
2.1	Enhance support optimization algorithm for complex parts
2.2	Develop detailed process model for 316L and 17-4PH
2.3	Develop inherent strain model for 316L and 17-4PH
2.4	Integrate inherent strain model with support optimization
M2	Demonstration of support optimization for a complex part
3	Design laser processing with post-heat treatment to optimize hierarchical structure of AM parts
3.1	Laser processing for steels and Stellite 6 for optimized microstructure
3.2	Study MC carbides for grain refinement
3.3	Study structure-property relationship for strengthening effects
3.4	Study phase stability and transformation of inclusions such as oxides
3.5	Study microstructure with disolvable support
M3	Demonstration of optimized AM processing with post-heat treatment for comparable/exceeded mechanical properties of wrought alloys



- Milestones 1.1-1.4 have been achieved
- Milestones 2 and 3 are delayed by 5 months

Milestones and Deliverables for FY-21

4	Surface heat treatment for 316L, 17-4PH steels and Stellite 6 for enhanced mechanical properties
4.1	CALPHAD simulation of carburizing processes for steels and Stellite
4.2	Carburization of 316L and 17-4PH steel and Stellite 6
4.3	Microstructure analysis and mechanical tests of carburized alloys
4.4	Optimize carburization with surface finishing for enhanced properties
M4	Demonstration enhanced mechanical properties with improved surface roughness after surface modification
5	Demonstrate that the integrated technology is capable of removing internal support structures, not assessable by post-machining
5.1	Design three nuclear components
5.2	Print and evaluate dissolvable supports for components
5.3	Design and perform mechanical tests on components after post-processing
5.4	Print and evaluate performance of components with optimized supports
M5	Demonstration of enhanced mechanical properties with improved surface roughness after surface modification



Issues and Concerns

- Both the additive manufacturing and material characterization labs were shut down between March to June (4 months) due to COVID-19
- During the shutdown, focus was shifted to modeling and simulation as well as analysis of data already acquired

Possible Areas/Industries/Programs (and Readiness) for Adoption

- Self-Terminating Etching Process has been licensed to InnovAMMP for commercialization
- InnovAMMP has $150,000 in contracts for this first quarter
- Contracts include companies designing and manufacturing AM parts for nuclear applications
- Materials:
  - Stainless steel
  - Inconel
  - Cobalt super alloys
  - Titanium alloys
  - Copper alloys

innovAMPP

Contact Information and Questions

Dr. Albert To (albertto@pitt.edu), William Kepler Whiteford Professor

Department of Mechanical Engineering and Materials Science

University of Pittsburgh

Dr. Owen Hildreth (ohildreth@mines.edu), Assistant Professor

Department of Mechanical Engineering

Colorado School of Mines

Dr. Wei Xiong (weixiong@pitt.edu), Assistant Professor

Department of Mechanical Engineering and Materials Science

University of Pittsburgh