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Exploring the Stress Corrosion Cracking Susceptibility of Additively Manufactured 316L in Boiling Magnesium Chloride

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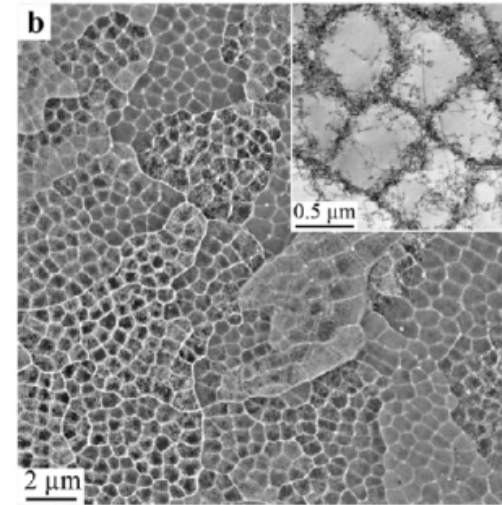
Additively Manufactured Metals

Additively manufactured (AM) metals are different from wrought:

- Microstructure
 - Sub-grain structure
 - Melt pool boundaries
- Processing defects
 - Lack of fusion pores
 - Surface roughness
- Residual stresses

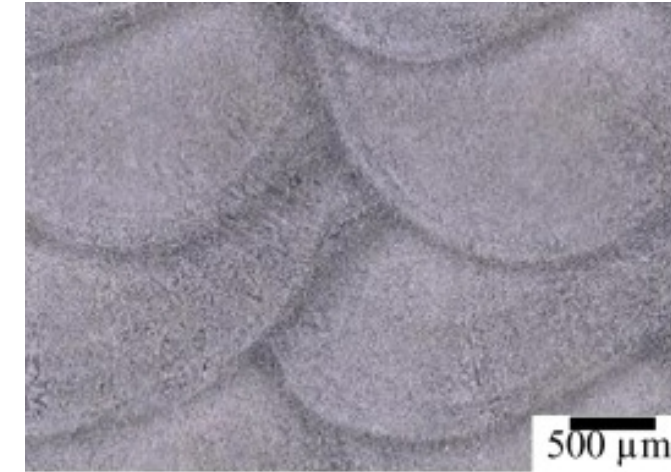
This work focuses on the relationship between residual stress and stress corrosion cracking in AM 316L.

Sub-Grain Structure



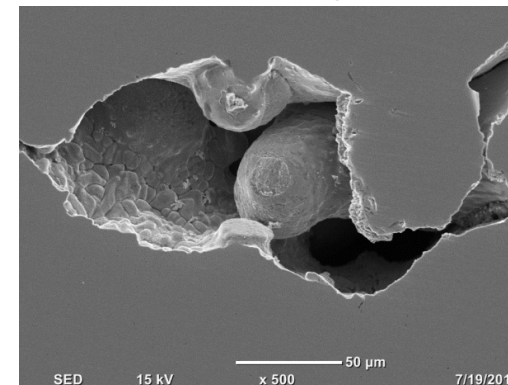
Voisin, 2021

Melt Pool Boundaries



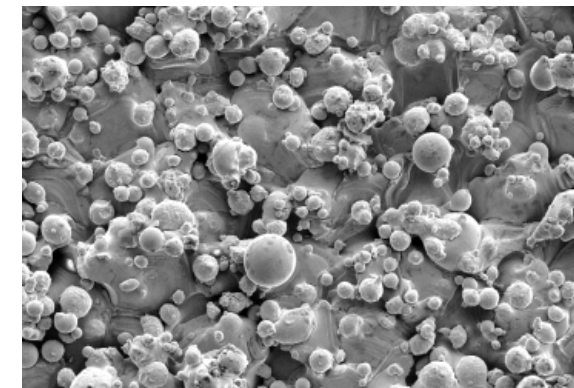
Melia, 2019

Porosity



Melia, 2019

Surface Roughness

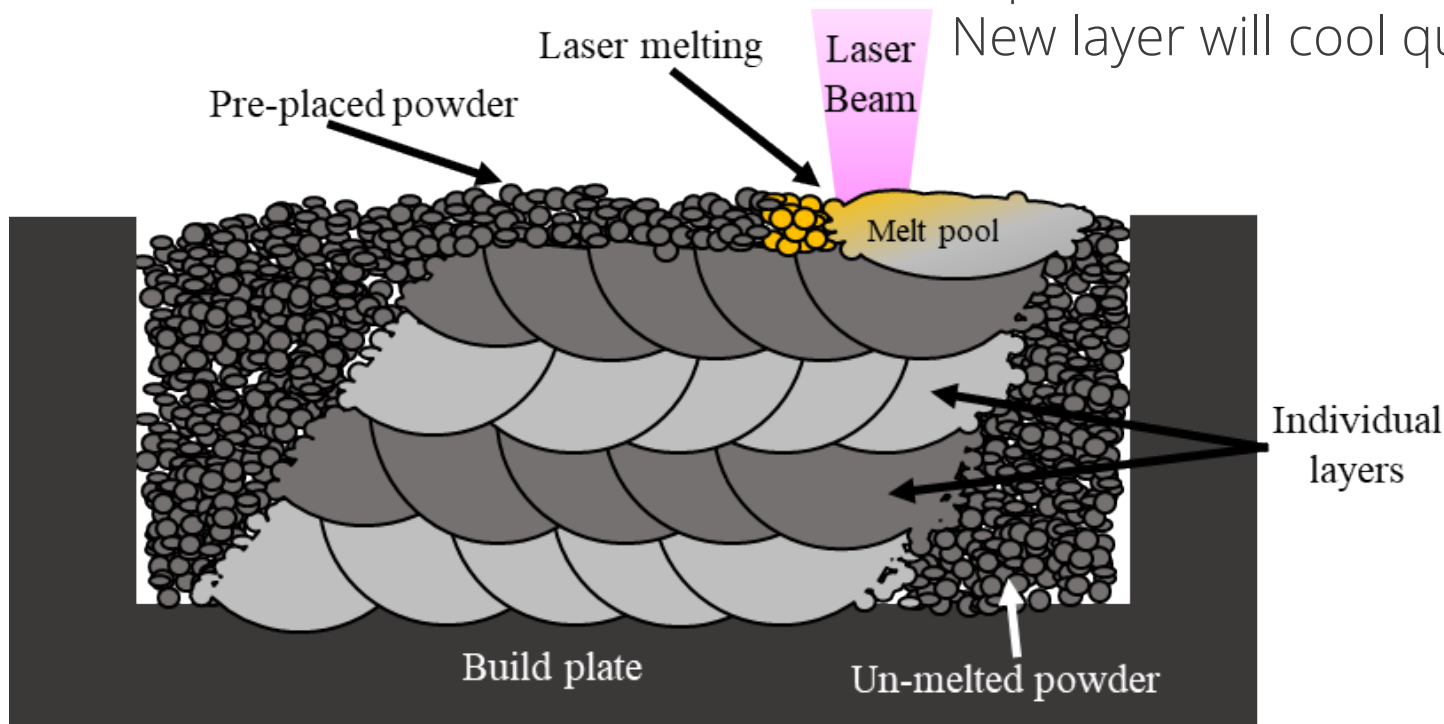


Melia, 2020

Laser Powder Bed Fusion & Residual Stress

Residual stress is a by-product inherent to the AM process and can contribute to stress corrosion cracking.

Heat from melting a new layer will heat layer underneath
Expansion of lower layer is restricted (part beneath has cooled)
New layer will cool quickly, contract faster than layers beneath





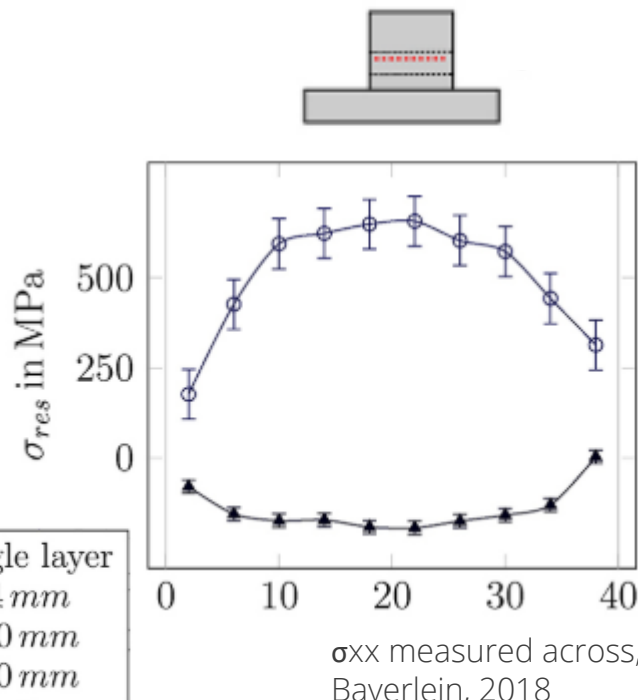
Laser Powder Bed Fusion & Residual Stress

From Strantza et al.

- Each layer of can have fluctuations in residual stress
- Residual stress varies on scale of part

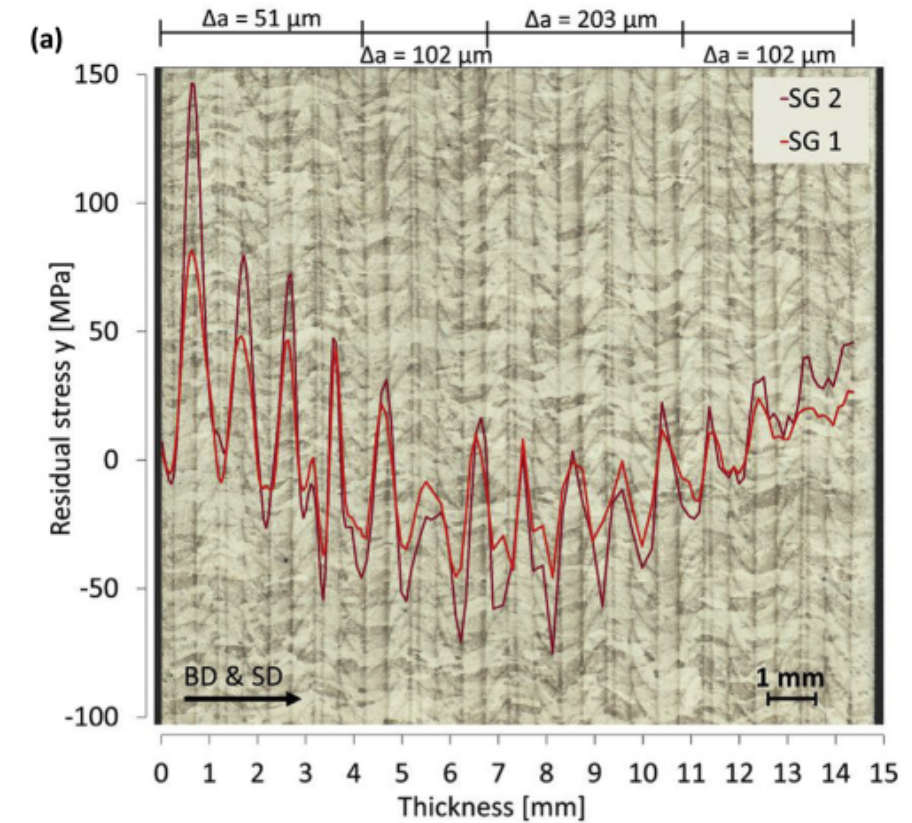
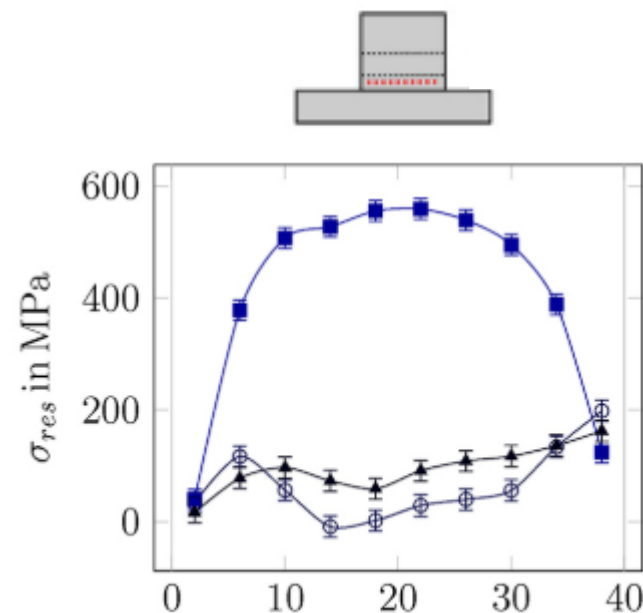
From Bayerlein et al.

- Cutting samples at different heights results in different residual stresses



σ_{xx} measured across, horizontal; (cut 18 mm, 2 mm)

Bayerlein, 2018



Strantza, 2019



Varying Residual Stress

Utilize multiple methods to vary residual stress:

- Cut samples at different heights from build plate → preserve microstructure
 - 4, 6, 8, 10, 15 mm
- Heat treatment → change microstructure/mechanical properties
 - 600, 800, 1200 °C; 1 hour

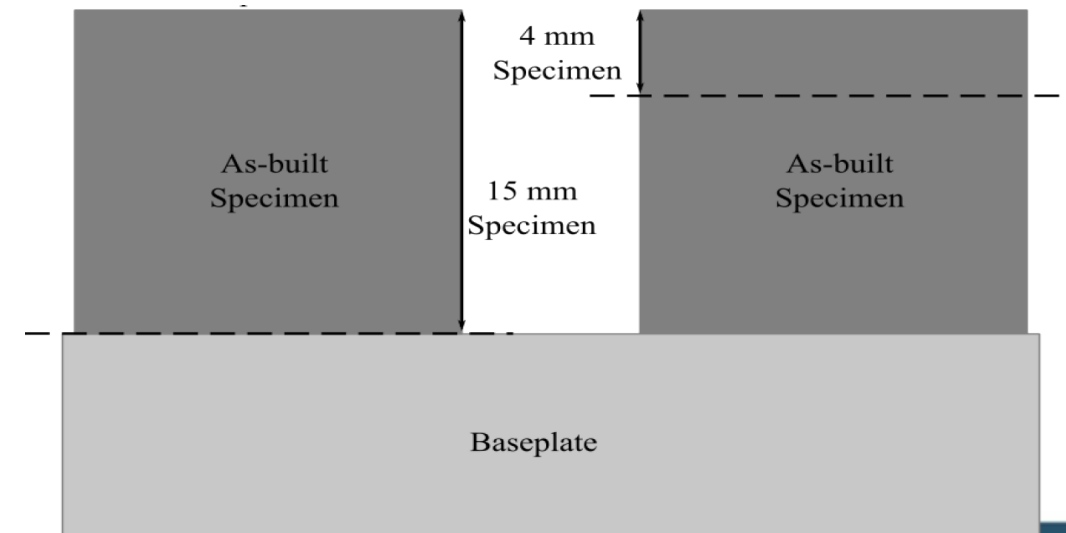
316L Stainless Steel Laser Powder Bed Fusion
15 x 15 x 15 mm blocks

Increasing residual stress



Large residual stress
on top surface after cut

Small residual stress
on top surface after cut





Finite Element Analysis

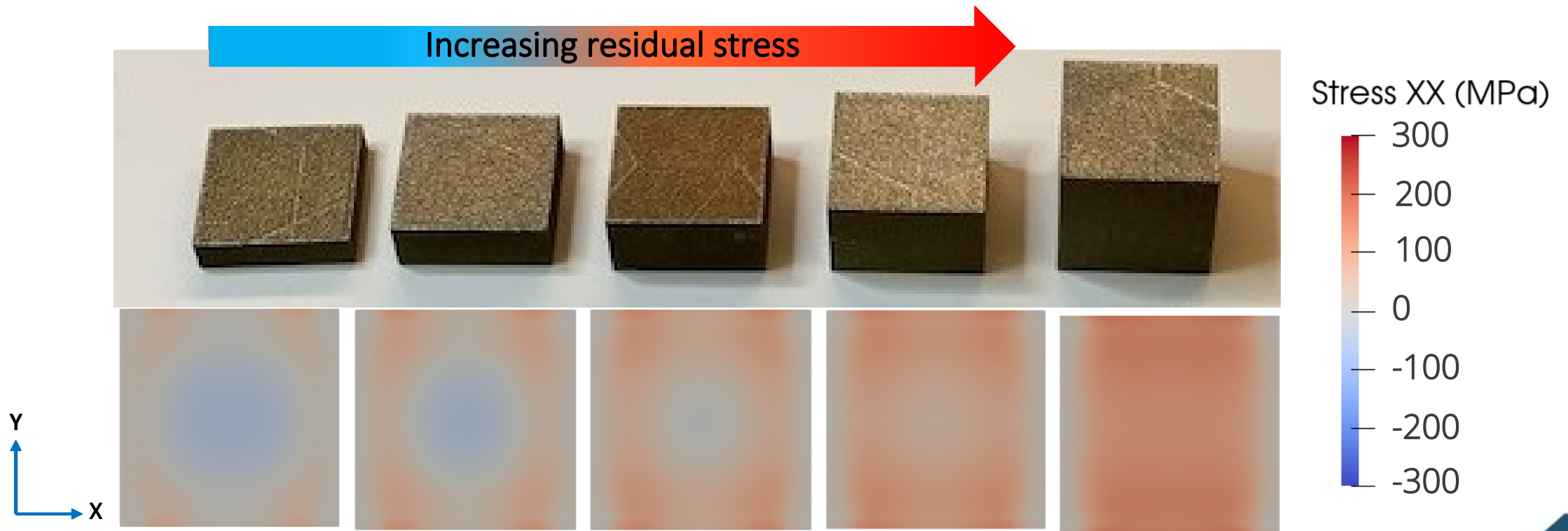
Simulation using Sierra Mechanics Module

Shorter Samples (4, 6, 8 mm)

- Tensile stresses around the perimeter
- Compressive stresses near center of the surface

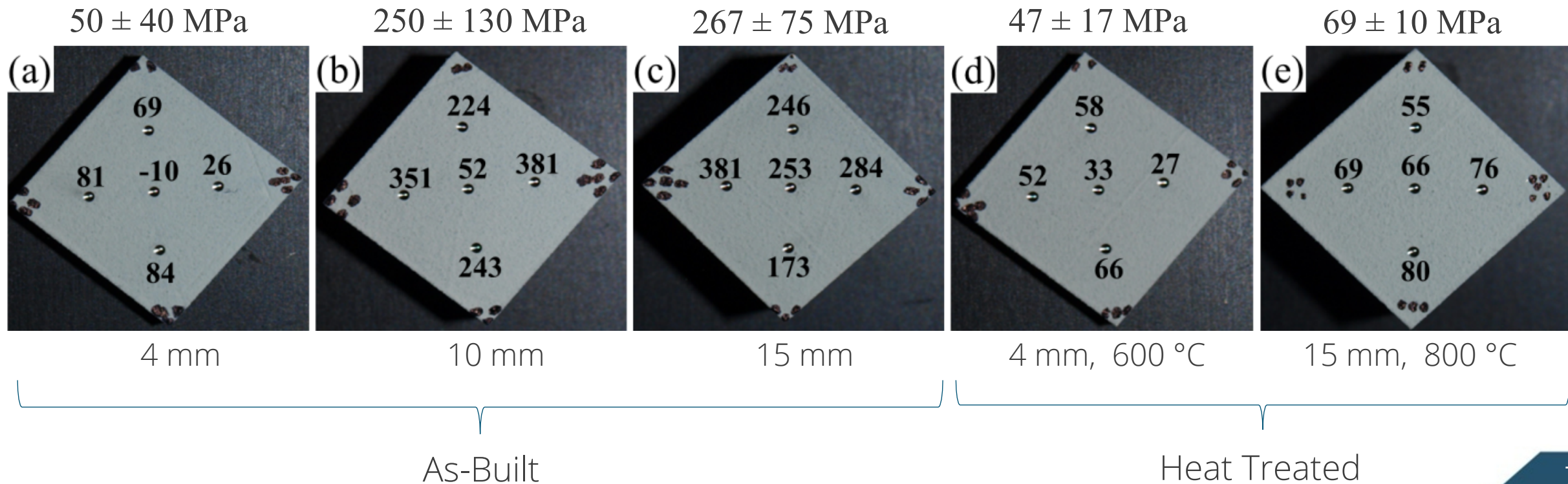
Taller samples (10, 15 mm)

- More even stress distribution
- Tensile stresses



Hole Drilling

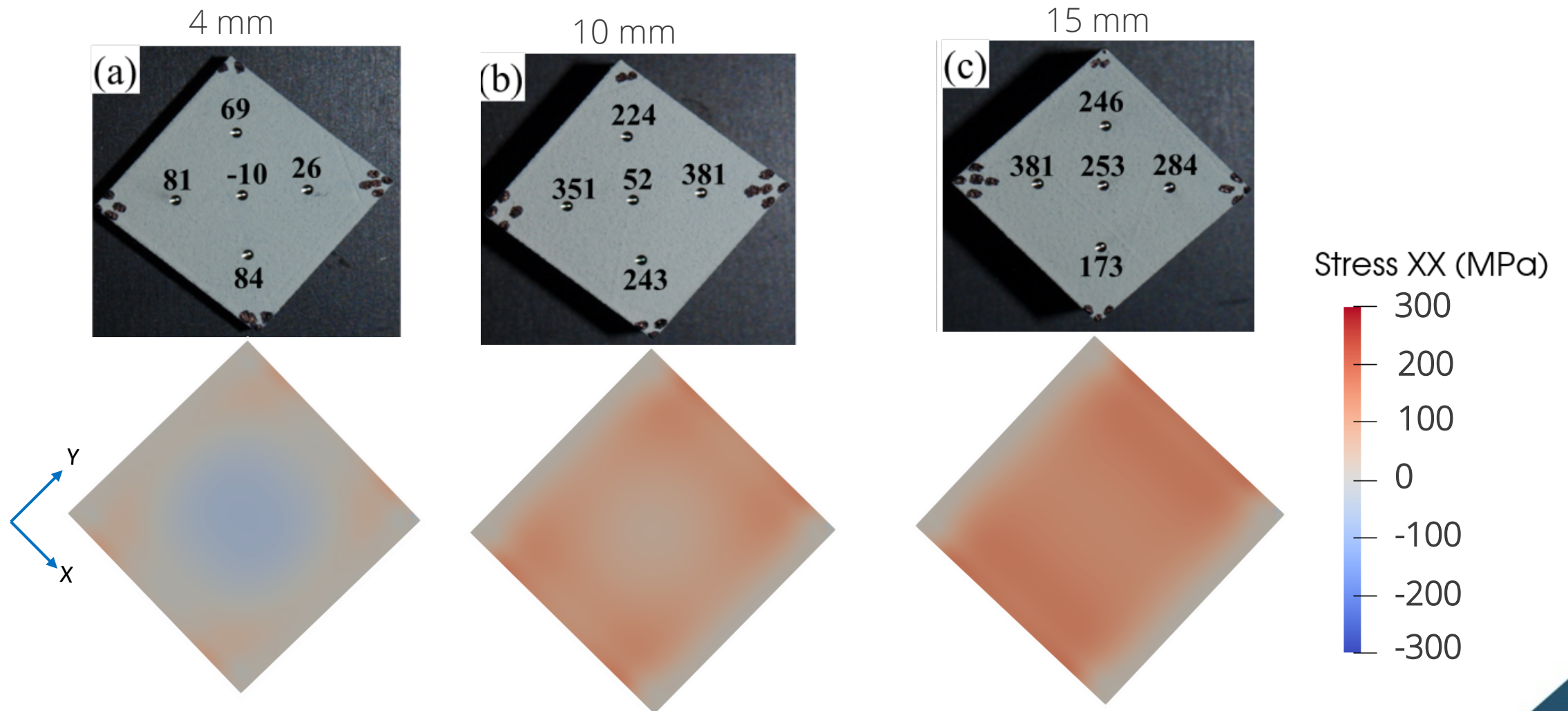
- Stresstech Electronic Speckle Pattern Interferometry system
- 0.8 mm diameter drill
- 0.025 mm increments to depth of 0.6 mm, 5 spots per sample
- Imaged with digital camera
- PrismS software analyzed relaxation





Simulation vs Experimental

The values from simulation align well with those from the hole-drilling in as-built samples.



Boiling MgCl_2 Experiments

Sides (except for top surface), coated in epoxy
3M Scotchkote liquid epoxy 323

ASTM G36-94

Full immersion in saturated, boiling MgCl_2
155 °C

- Samples were checked after 24 hrs
- If no cracks were visible, resubmerged and checked every 72 hours, up to 350 hours of exposure

Condenser

Vessel

Heating jacket

Thermometer

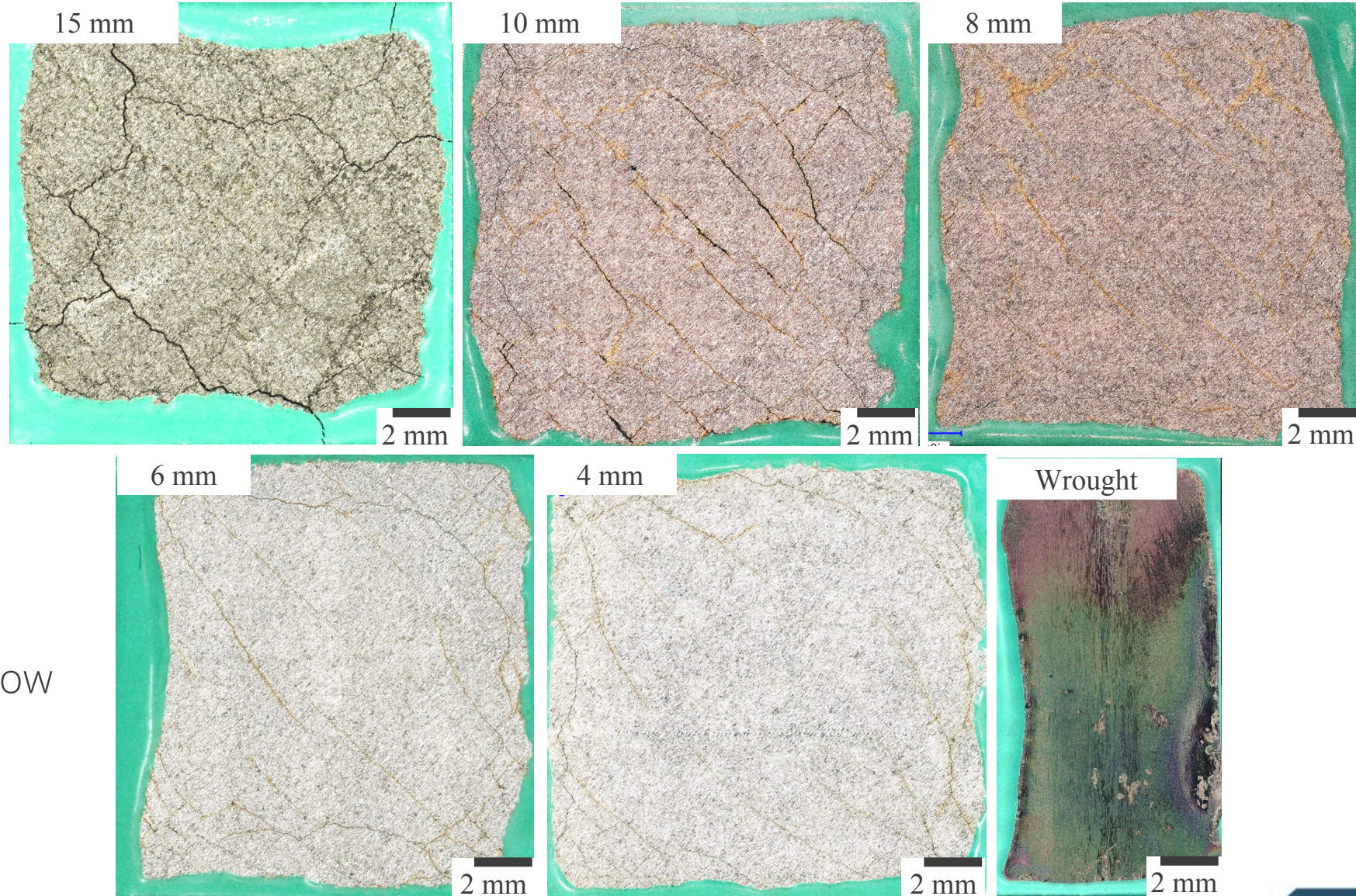
Thermocouple control





As-Built Samples

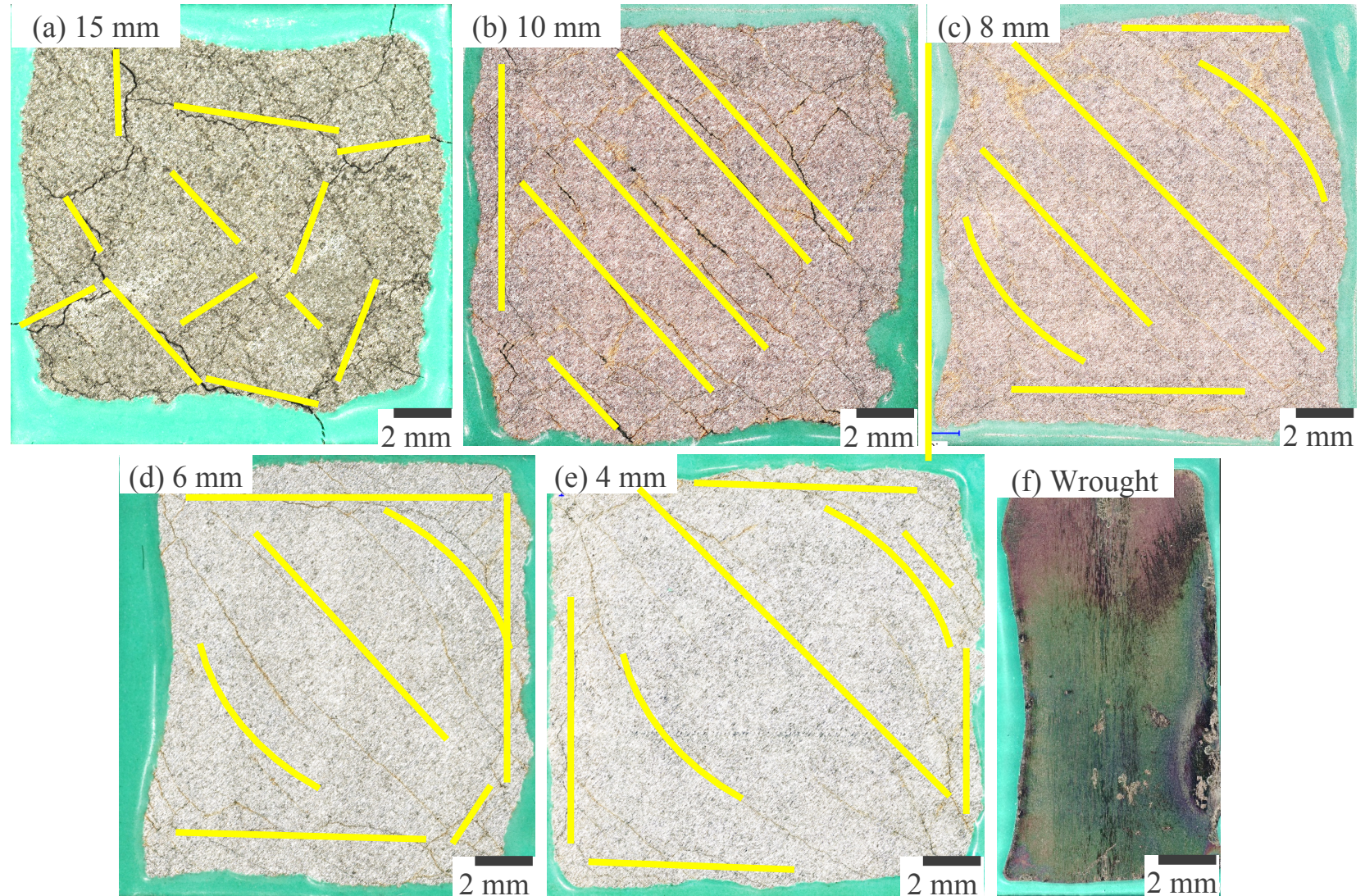
- All as-built showed cracking within 24 hours
- Shorter samples show oriented cracking
- Tallest sample more distributed cracking
- 15 & 10 mm tall samples show more pronounced cracks





As-Built Samples

- All as-built showed cracking within 24 hours
- Shorter samples show oriented cracking
- Tallest sample more distributed cracking
- 15 & 10 mm tall samples show more pronounced cracks

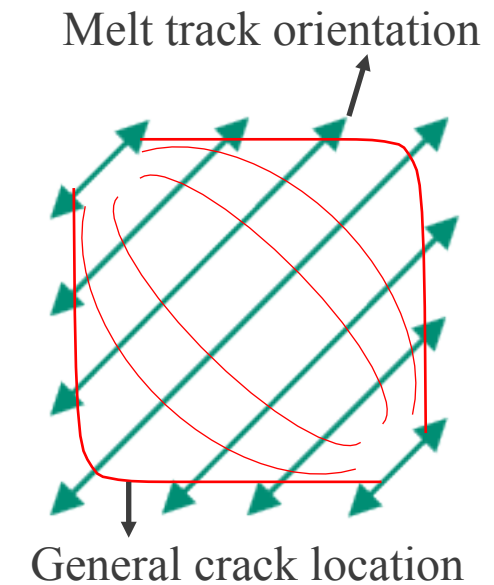
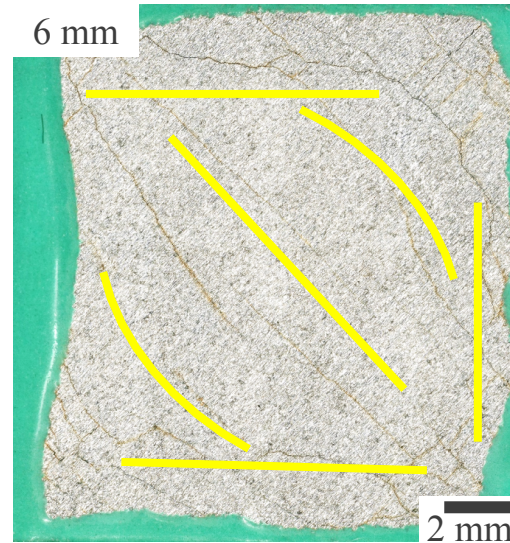
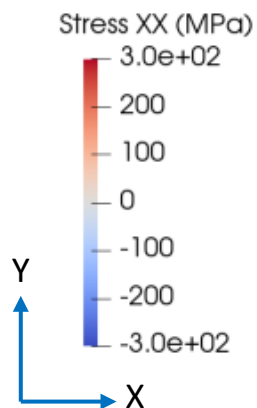




As-Built Sample Crack Location

Cracks in shorter samples run perpendicular to the melt tracks near the center of the sample

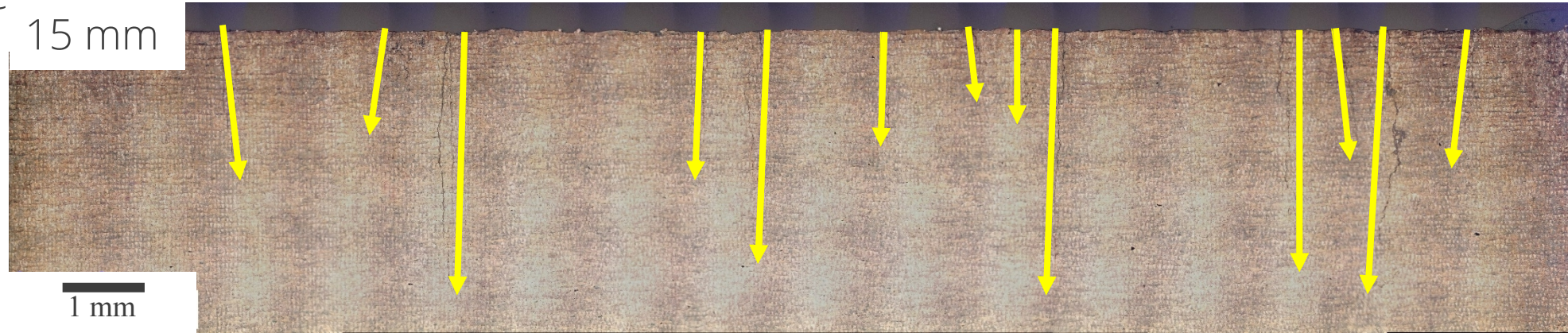
Cracks around circumference of the surface, align well with high stress areas identified in simulation





As-Built Sample Crack Depth

Cross-sectioned on the diagonal
Imaged with Keyence



Shorter Samples

- Deeper cracks near sample edge (higher stress regions)
- Cracks in the center probably too small to optically image

Taller Samples

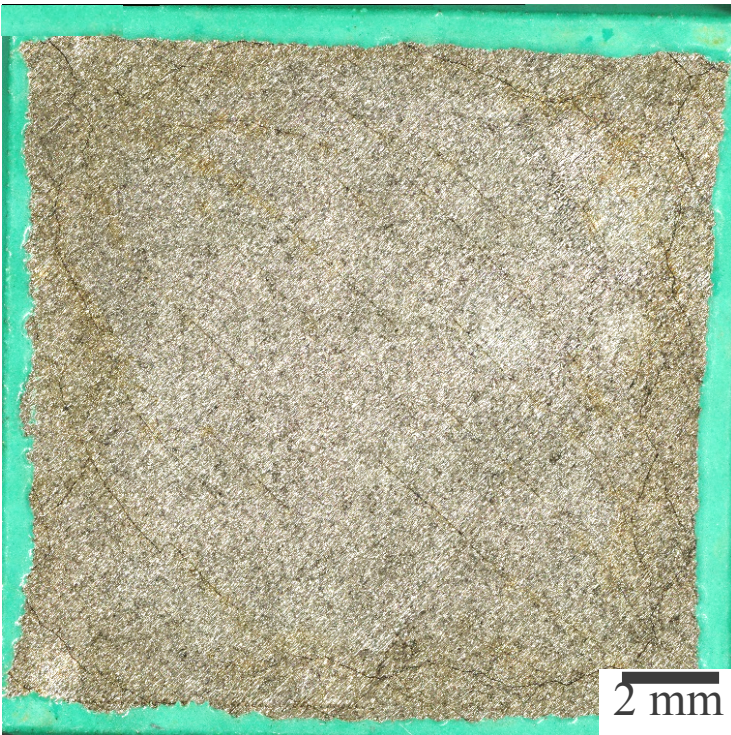
- Deepest cracks distributed across the cross-section



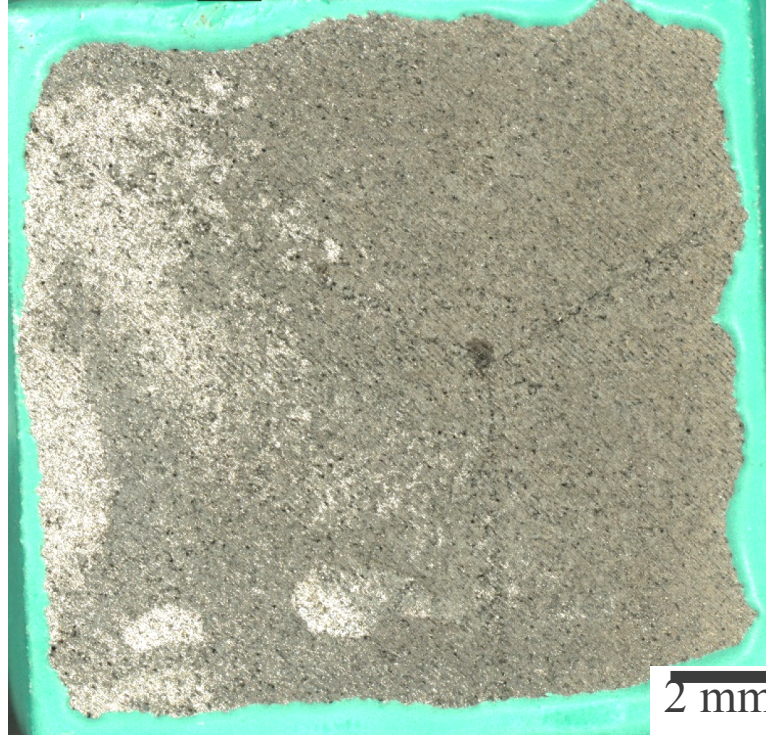
Heat Treated Samples

- No visible cracks for heat treatments of 800 °C or higher
- 800 °C has relieved all the residual stresses

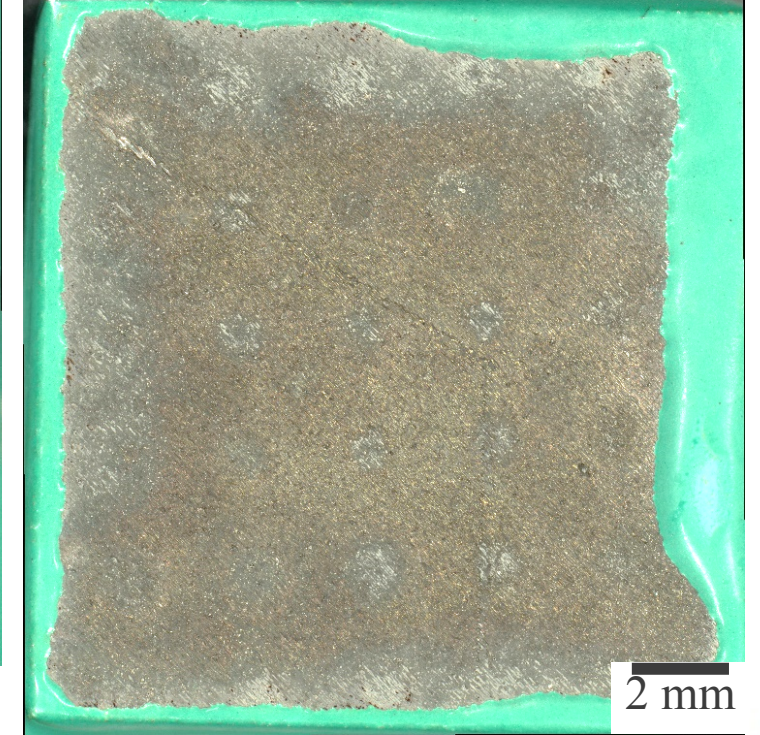
600 °C
 $\sigma_{xx} = +100 \text{ MPa}$



800 °C
 $\sigma_{xx} = 0 \text{ MPa}$



1200 °C
 $\sigma_{xx} = 0 \text{ MPa}$



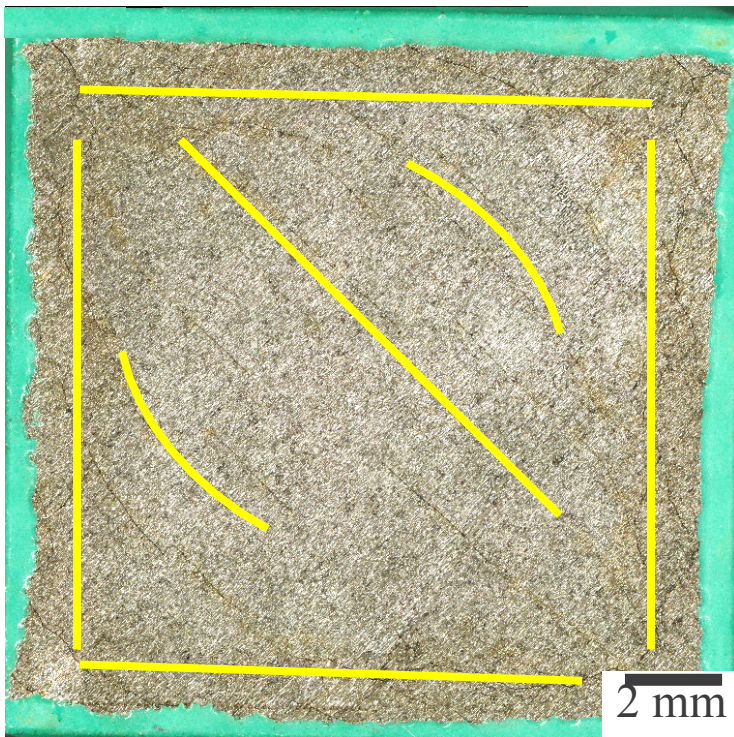


Heat Treated Samples

- No visible cracks for heat treatments of 800 °C or higher
- 800 °C 15 mm has more residual stress than 4 mm 600 °C but no visible cracks

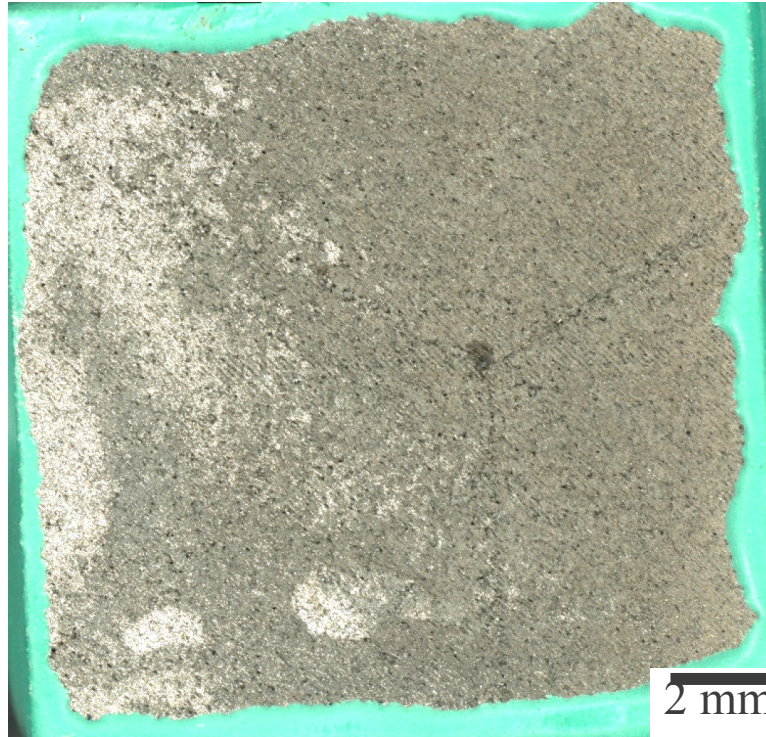
4 mm, 600 °C

$\sigma_{xx} = +50$ MPa



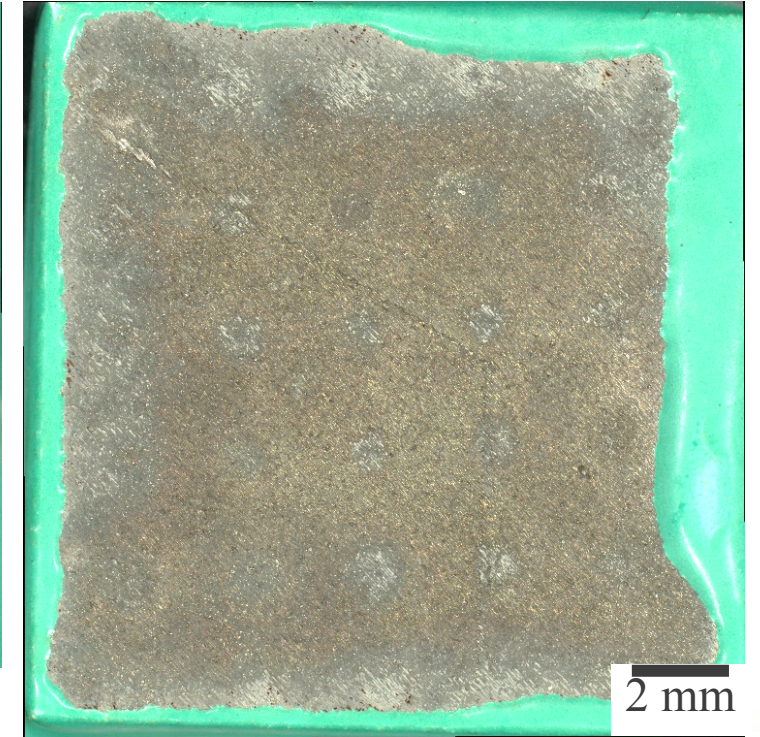
15 mm, 800 °C

$\sigma_{xx} = +70$ MPa



15 mm, 1200 °C

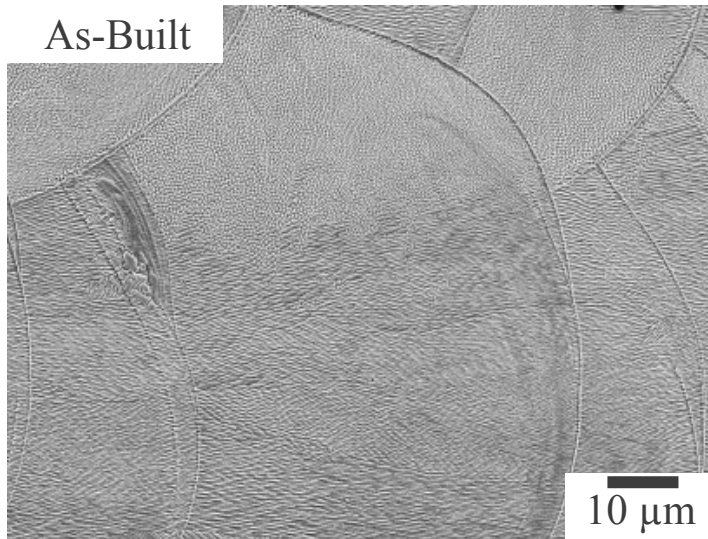
$\sigma_{xx} \sim 0$ MPa



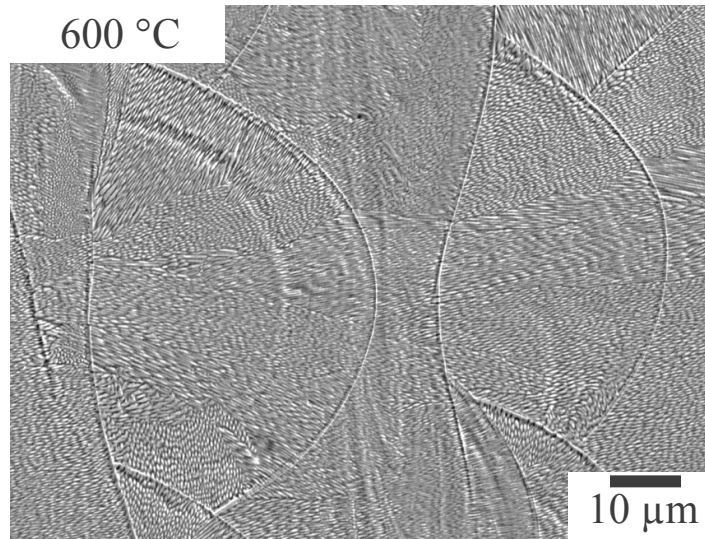


Heat Treated Etched Microstructure

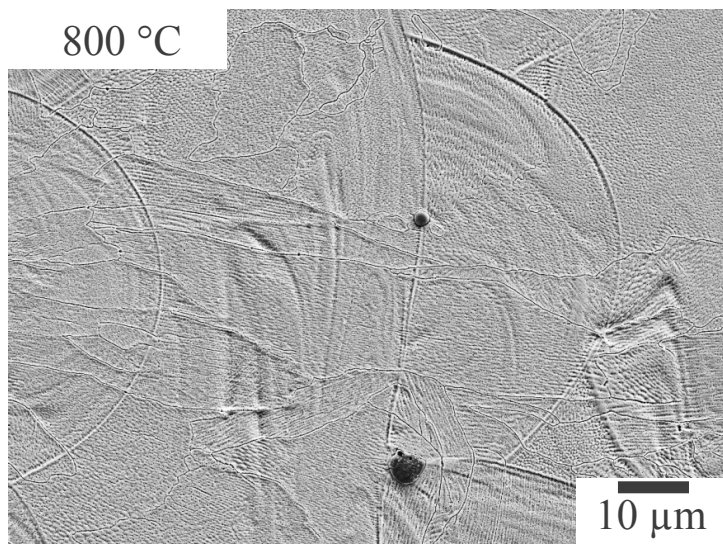
As-Built



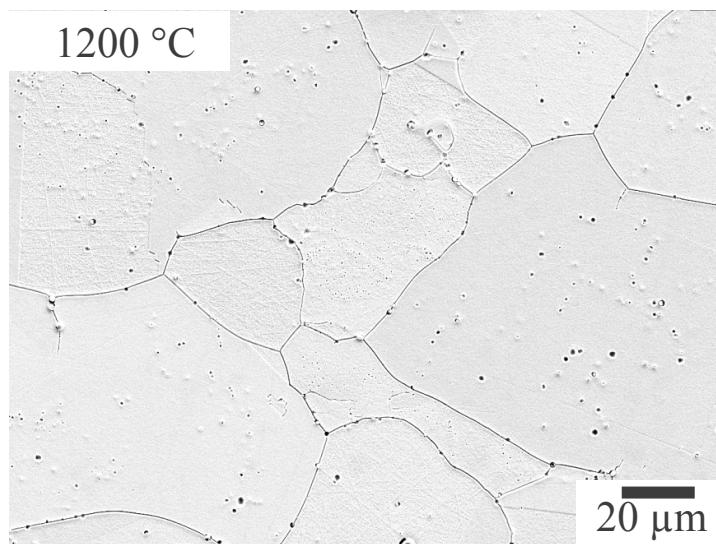
600 °C



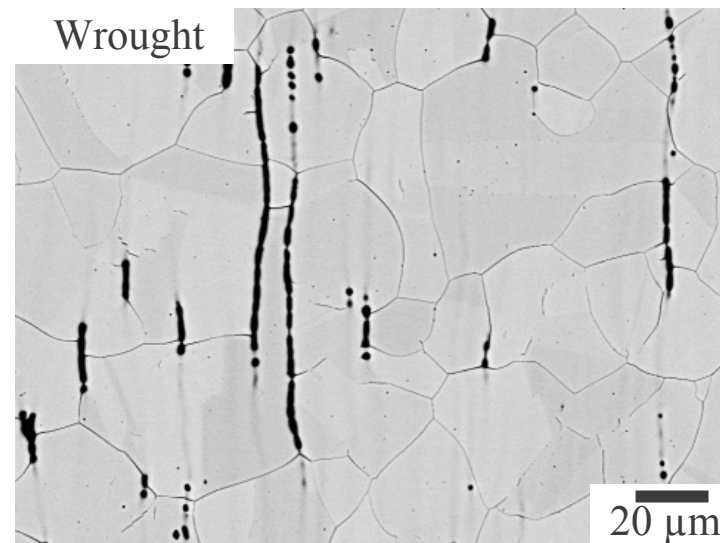
800 °C



1200 °C



Wrought



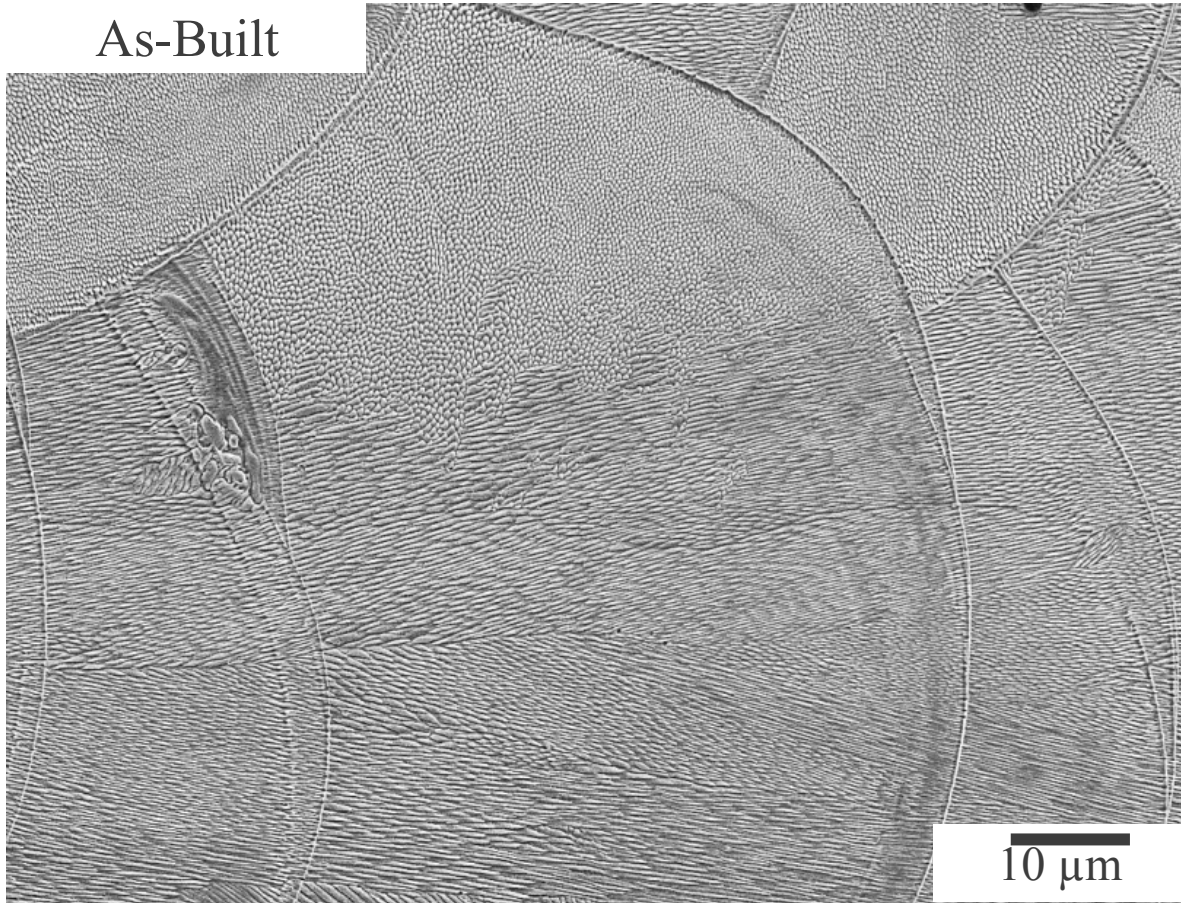


Heat Treated Etched Microstructure

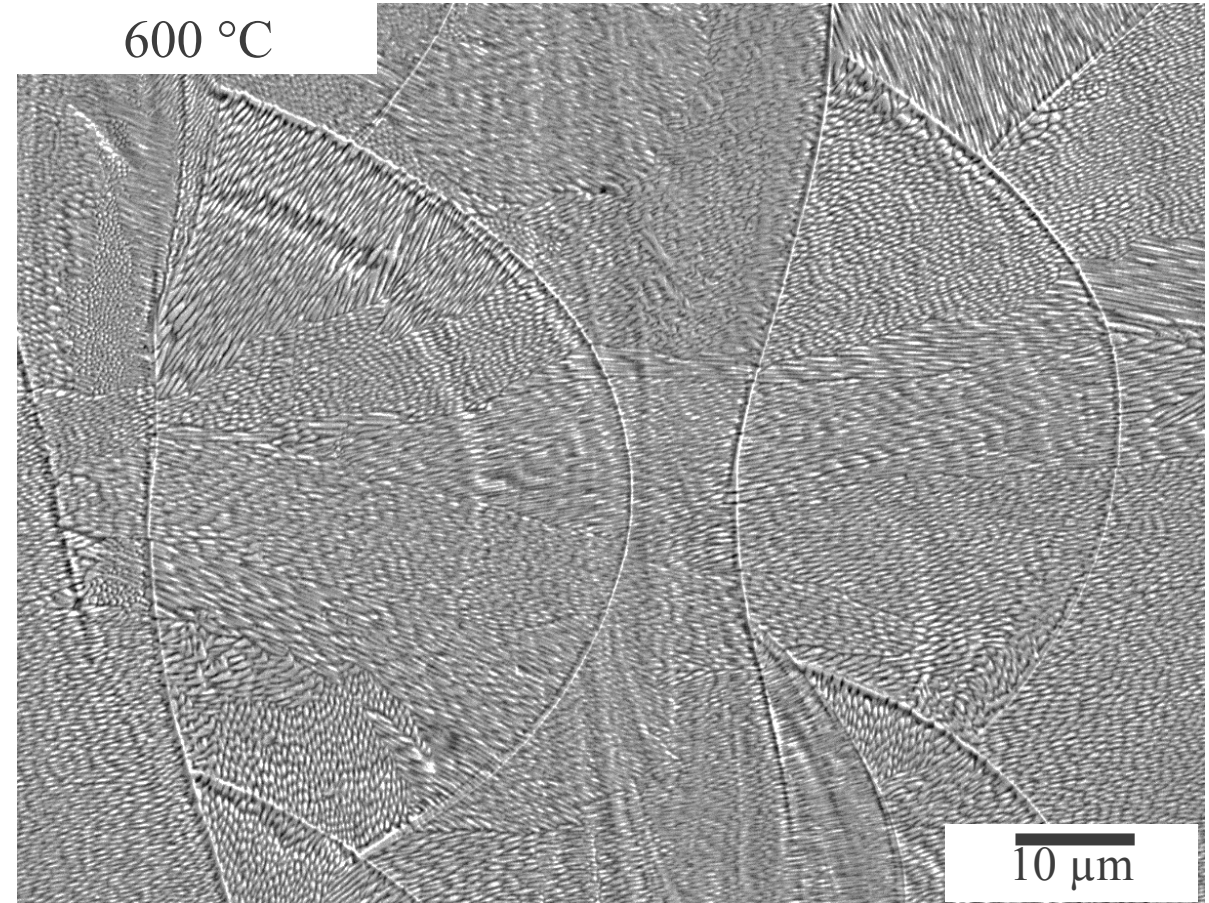
As-built & 600 °C look similar

- preferential attack at cell boundaries
- some melt pool boundaries

As-Built



600 °C



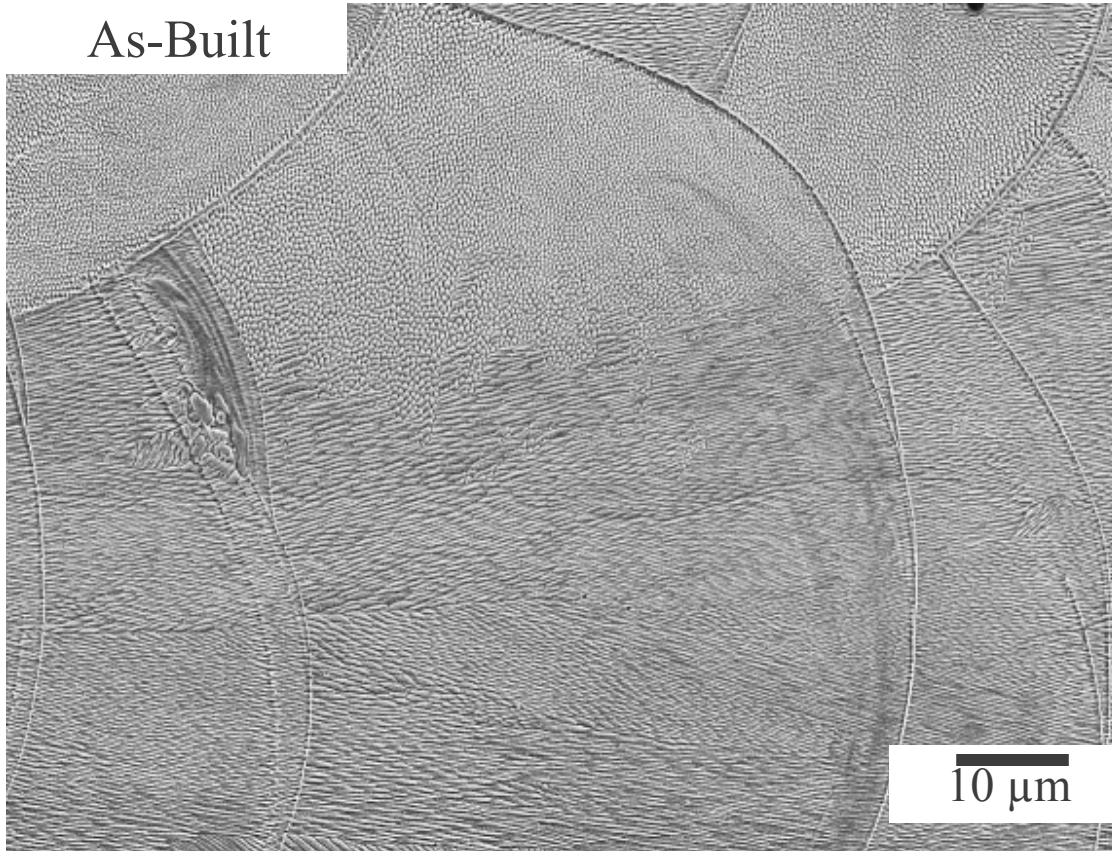


Heat Treated Etched Microstructure

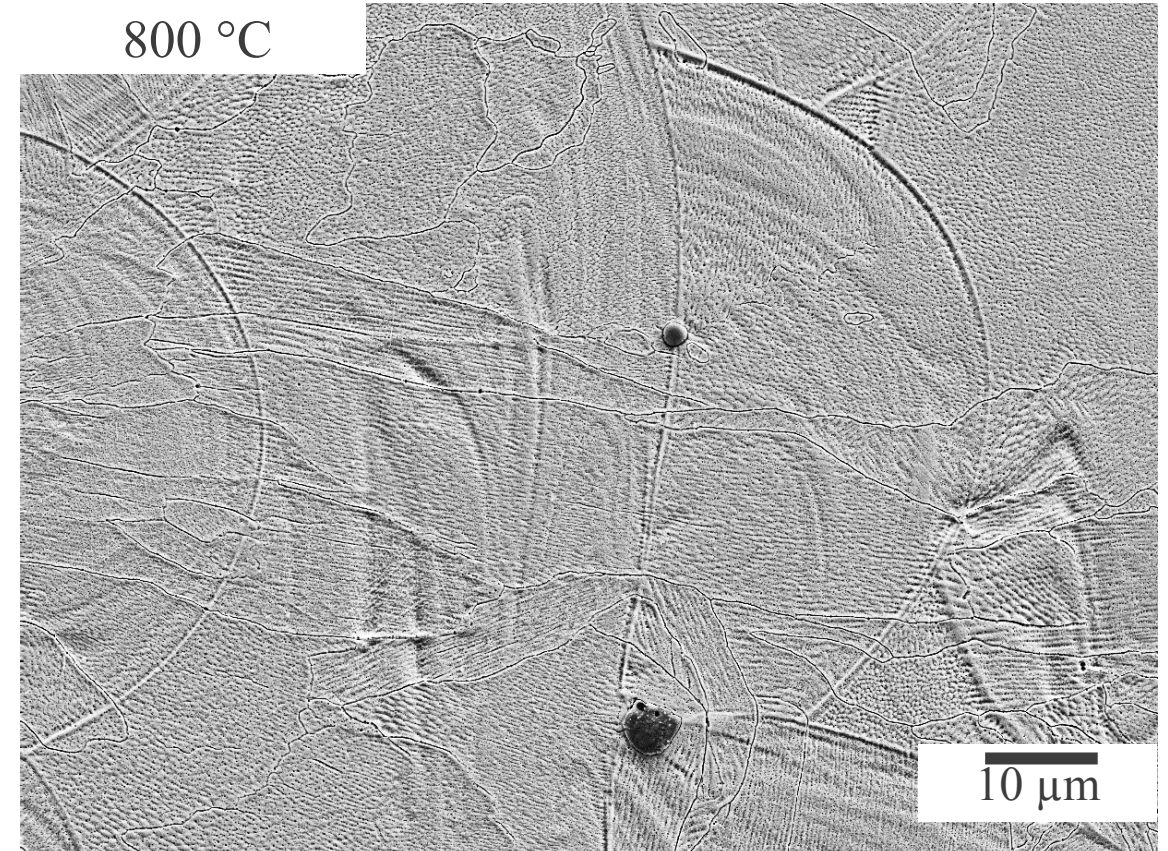
800 °C

- attack at cell boundaries & melt pool boundaries
- more grain boundaries visible (indicates recovery)

As-Built



800 °C

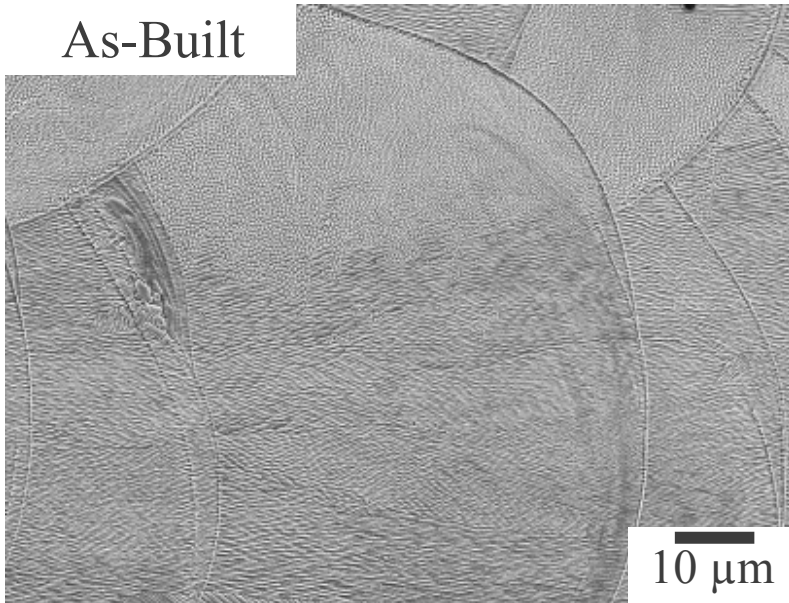


Heat Treated Etched Microstructure

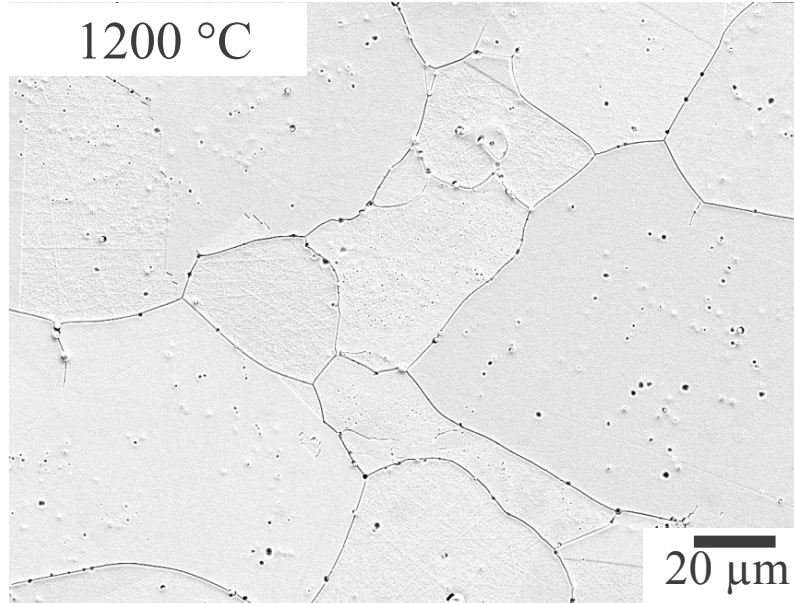
1200 °C

- No visible cell boundaries or melt pool boundaries
- Lots of grain boundaries visible-similar to wrought

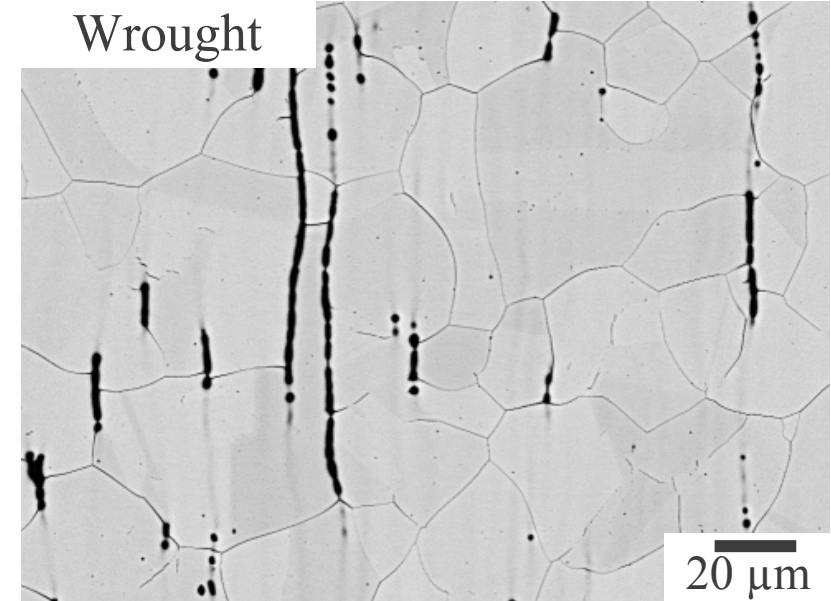
As-Built



1200 °C



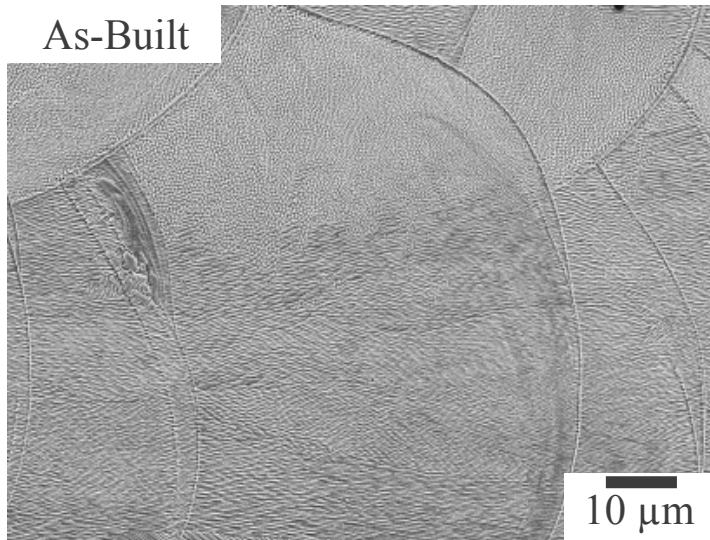
Wrought



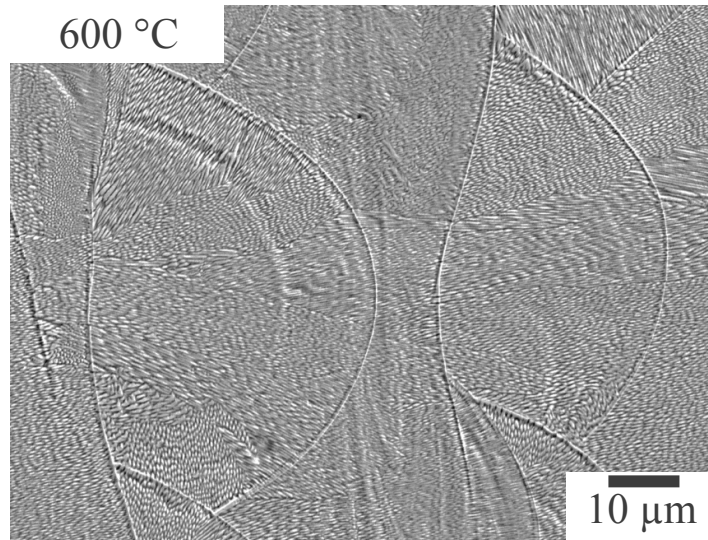


Heat Treated Etched Microstructure

As-Built

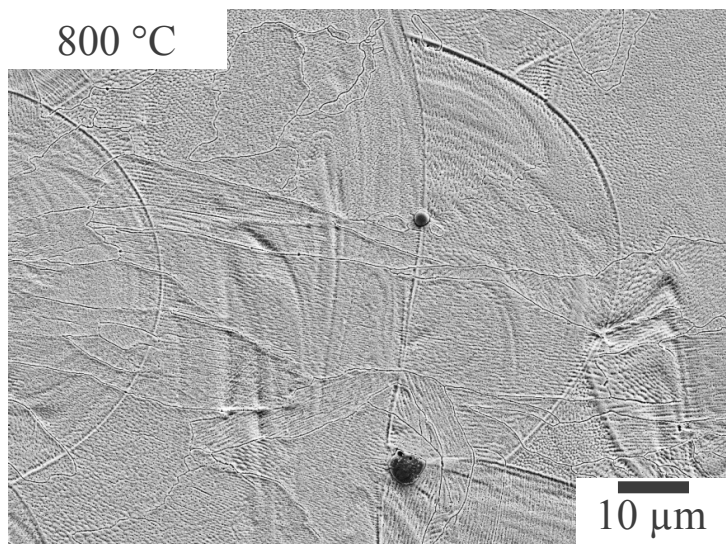


600 °C

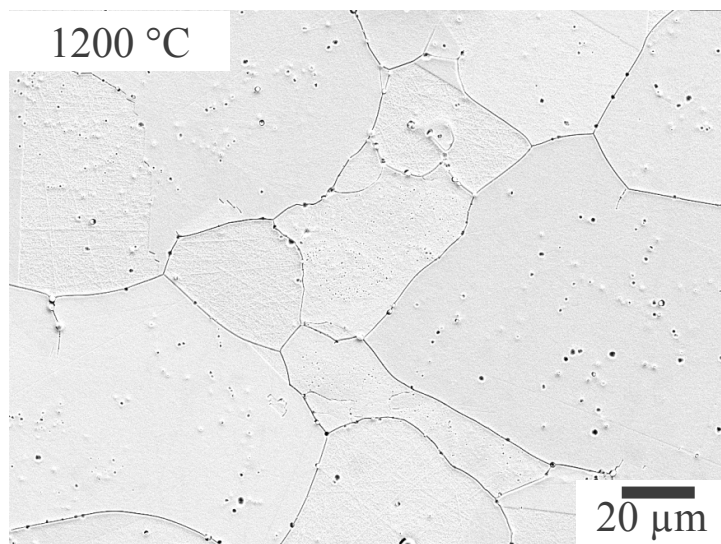


800 °C has no cracking
but isn't completely recrystallized

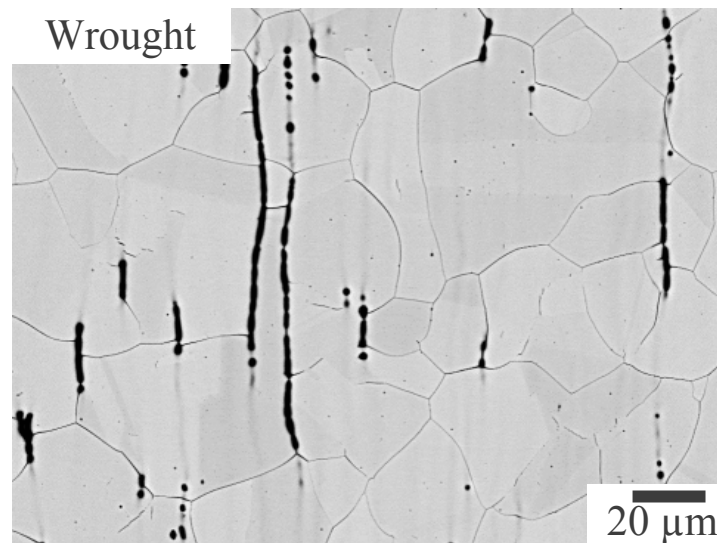
800 °C



1200 °C



Wrought

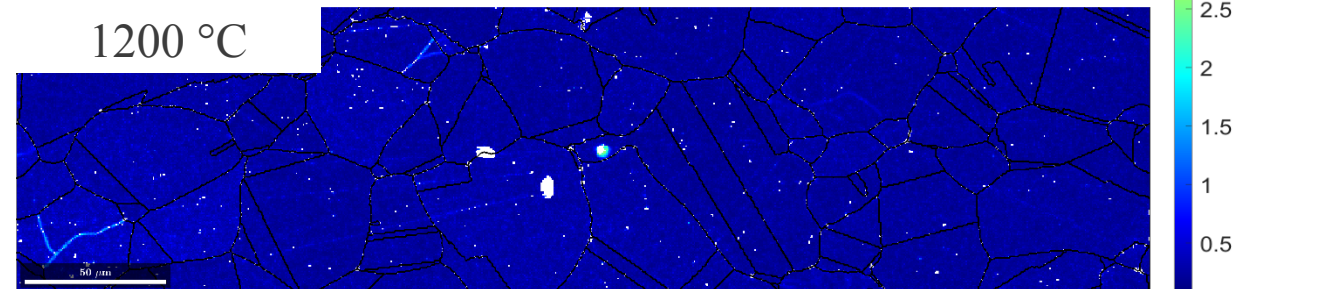
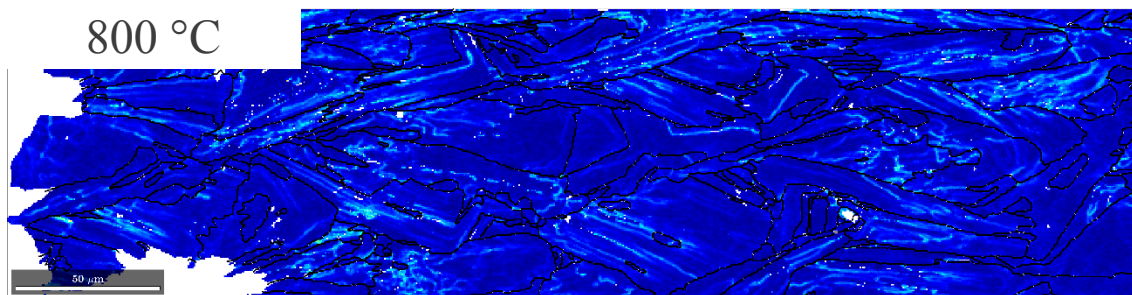
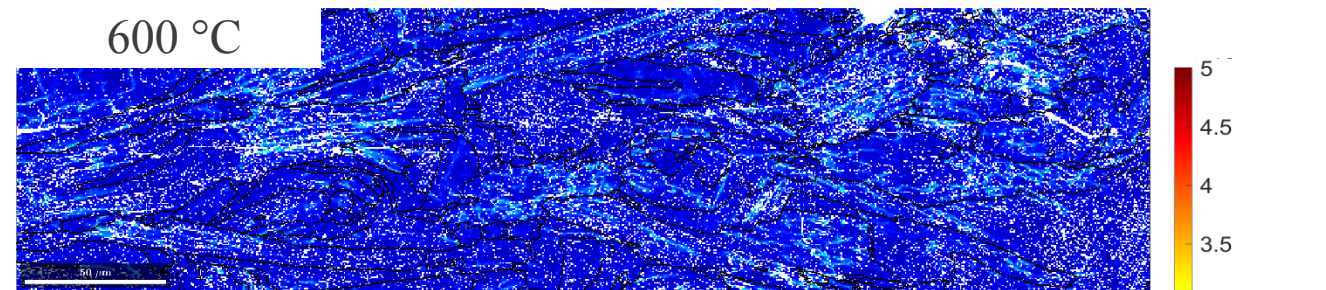
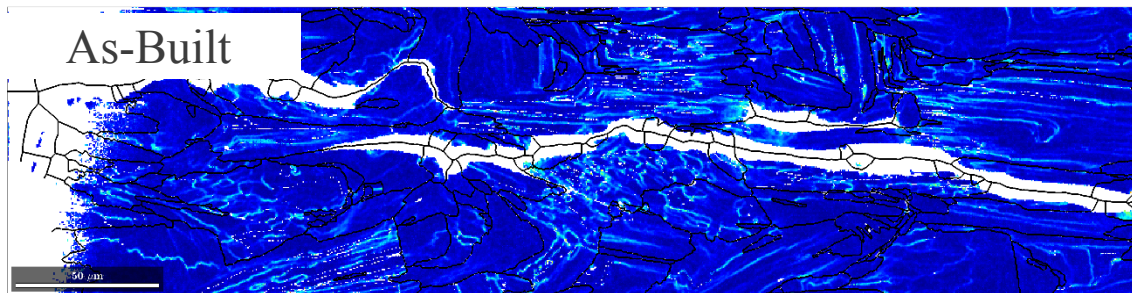




Geometrically Necessary Dislocation (GND) Density

600 °C minimal change in GND compared to as-built
800 °C lower GND density (not recrystallization)
1200 °C density similar to fully annealed wrought

$\times 10^{14}$ [dislocations/m²]

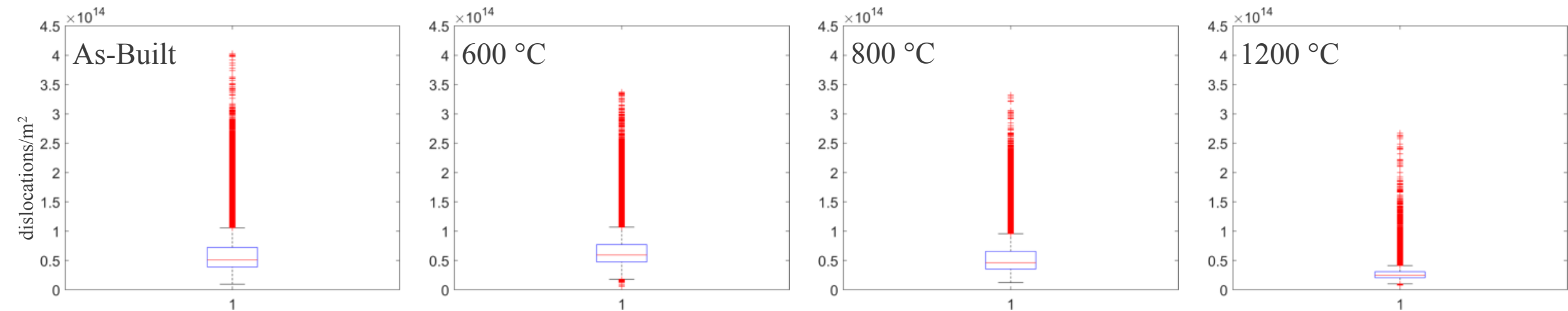




Geometrically Necessary Dislocation (GND) Density

Possible impacts of large local dislocation density at cell boundaries

- high localized stress
- cause H-trapping



Conclusions

- Reducing sample thickness & heat-treatments are effective ways of controlling residual stress
- All as-built showed SCC susceptibility regardless of thickness
- Distribution of stress affects distribution of cracking
- Residual stresses from melt tracks can be enough to affect SCC
- 800 °C & 1200 °C for 1 hour eliminated SCC susceptibility. EBSD, GND analysis suggest a reduction in dislocation density (not just reducing tensile stress) reduces SCC susceptibility





Future Work

Develop heat treatment specific to AM materials

- Control residual stress
- Address sensitization, segregation at melt pool boundaries

Further examine effects of residual stress

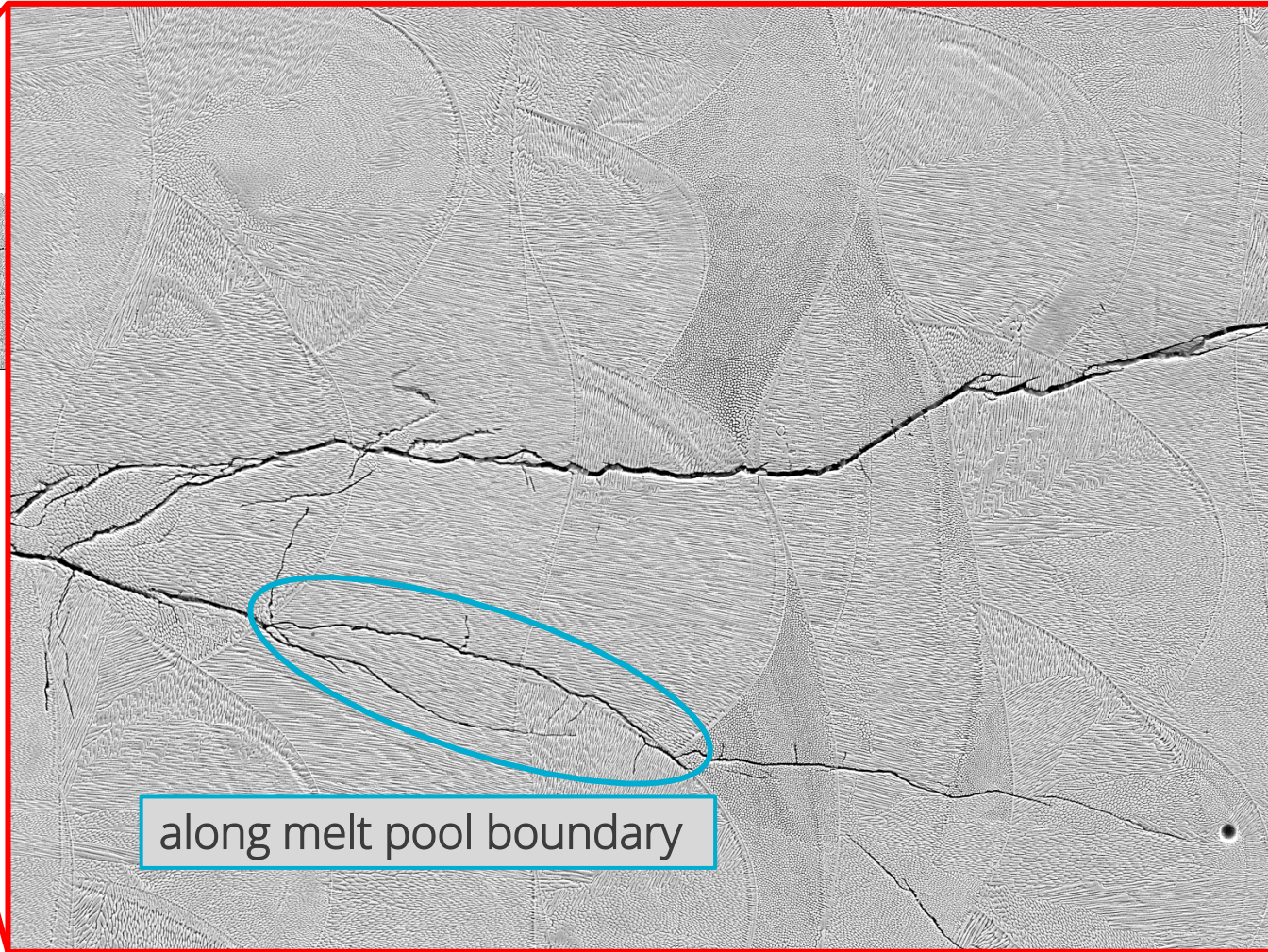
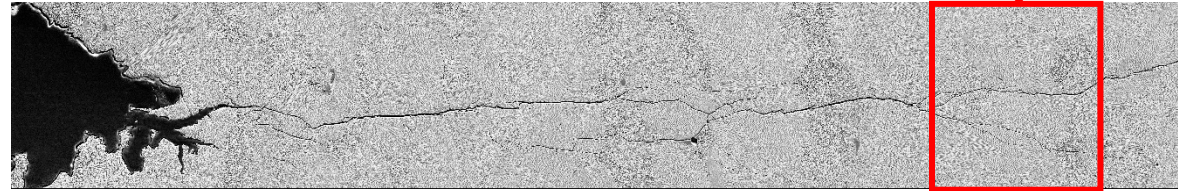
- Residual stress in build direction



Thank you!



15 mm – 600 C



along melt pool boundary



Standard way to remove residual stress is heat treatment.
AM metals react differently to heat treatment than wrought.
Heat treatment can decrease beneficial mechanical properties (yield strength) that would otherwise be a benefit stemming from the as-printed AM microstructure.

