

The Effect of Solidification Pathway on Grain Boundary Fractality

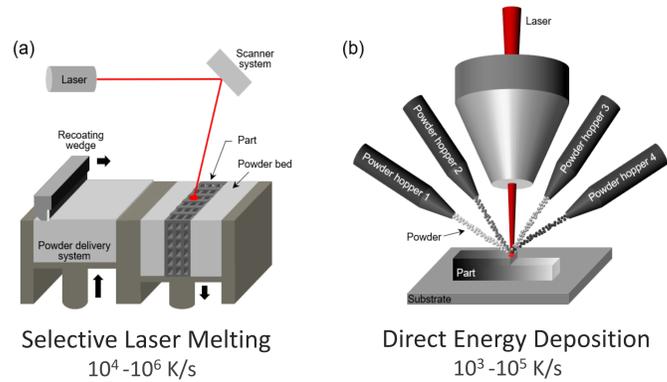
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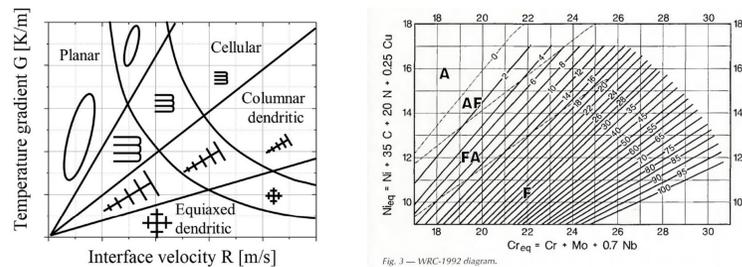
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The intense heat source used in additive manufacturing (AM) causes rapid cooling



We can engineer microstructures in AM by taking advantage of its fast cooling

Solidification behavior determines microstructure → Exploit access to metastable phases via rapid cooling to design microstructure



Two grades of stainless steel (304L and 316L) with different phase metastability were printed at fixed process parameters (P=200W, v=6 mm/s, h=2 mm) as a proof of concept

Element	304L	316L
Cr	18-20	16-18
Ni	8-10	10-14
Cr_{eq}/Ni_{eq}	1.52	1.39

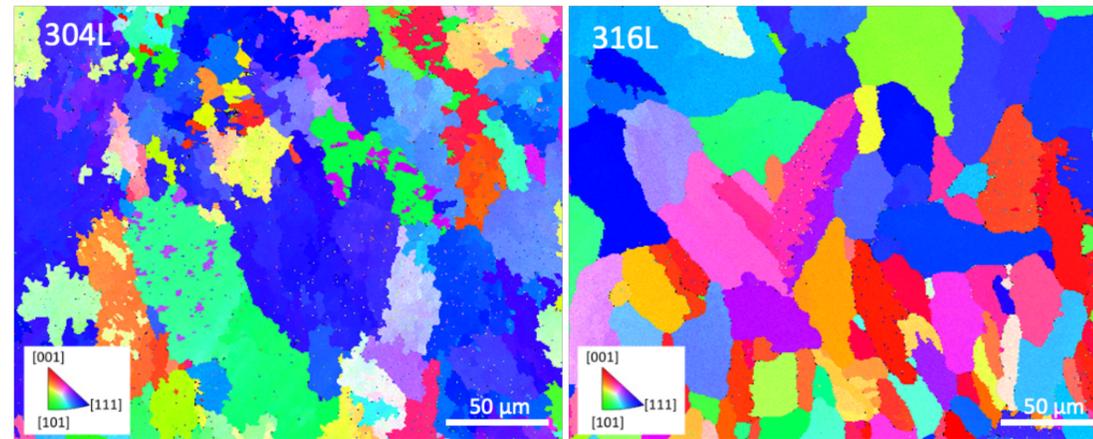
304L and 316L have different ratios of austenite-stabilizing and ferrite-stabilizing elements that influence solidification pathway

References

J Kim, A Wakai, A Moridi. *J. Mater Res*, (2020)
 Froend, M. *et al. Mat. Sci Eng.* (2020)
 Basinski, Z. *et al. Pro Roy Soc of Lon.* (1955)
 Lehto, P. *Ultramicroscopy*, (2021)
 Khanbareh, H. *et al. J. Mater Sci*, (2012)



304L and 316L have vastly different microstructures with three key distinctions



1. Grain size distributions

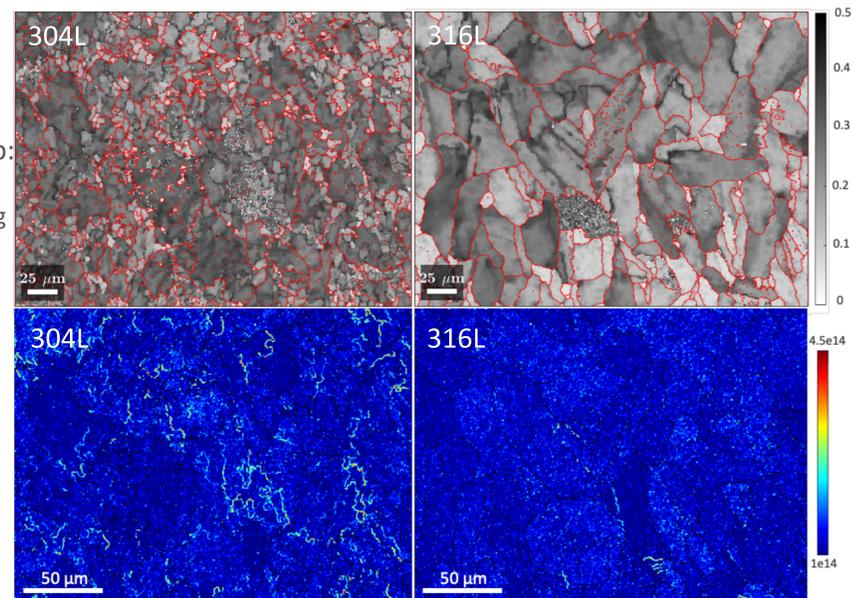
Wider range of grain size in 304L compared to 316L

2. Misorientation

There is noticeably more dislocation accumulation in 304L than in 316L

Misorientation map: Shows dislocation density (thus forming deformation cells)

Geometrically Necessary Dislocation Density

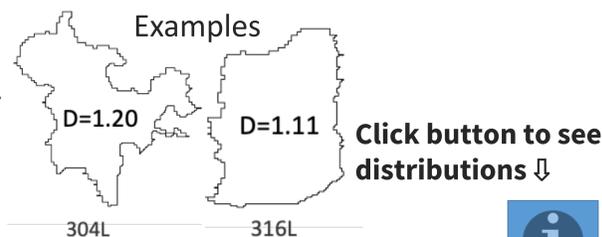


3. Rough vs smooth grain boundaries: roughness via Fractal Dimension

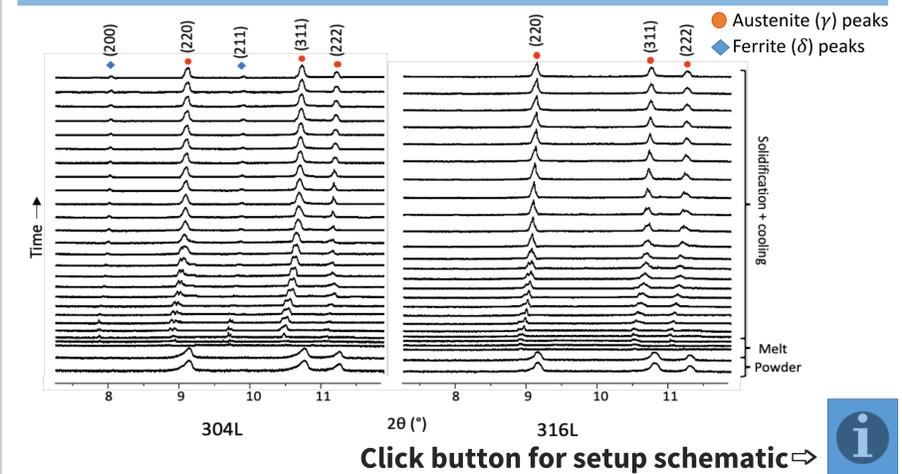
To quantitatively compare the roughness of grain boundaries, we applied a geometrical concept to index the irregularity of a geometry into a 'fractal dimension' (D)

The outlines of forty grains from each sample were indexed for their fractal dimensions

Grains of 304L have higher fractal dimensions



Operando time-resolved X-ray Diffraction at Cornell High Energy Synchrotron Source (CHESS) illustrates different solidification pathways



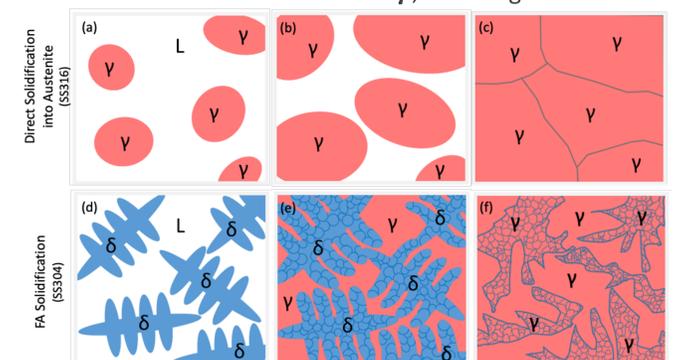
Mechanism behind fractal grains

Ferrite-to-austenite (FA):

Direct solidification into austenite:

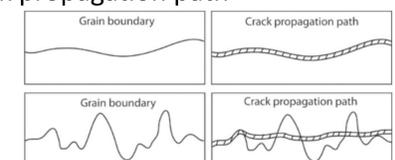
- (a) Liquid → austenite (γ)
- (b) Austenite cells grow
- (c) Complete solidification

- (e) Liquid → ferrite (δ)
- (f) Ferritic dendrites deform in fluid flow of AM (lower yield strength in ferrite above 600 °C); nucleation of γ
- (g) Solid-state transformation of δ into γ, retaining deformation



Designing fractal grain boundaries

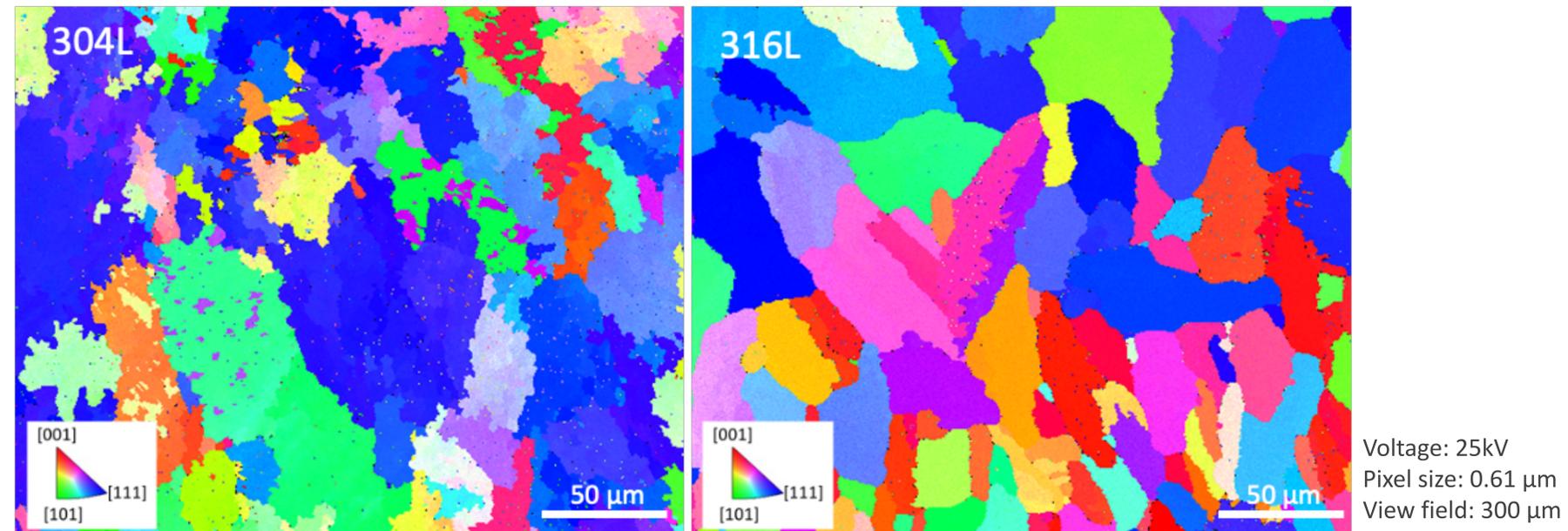
- Fractal grain boundaries improve mechanical properties by:
 - Increased friction between grains (prevents grain rotation)
 - Elongated crack propagation path



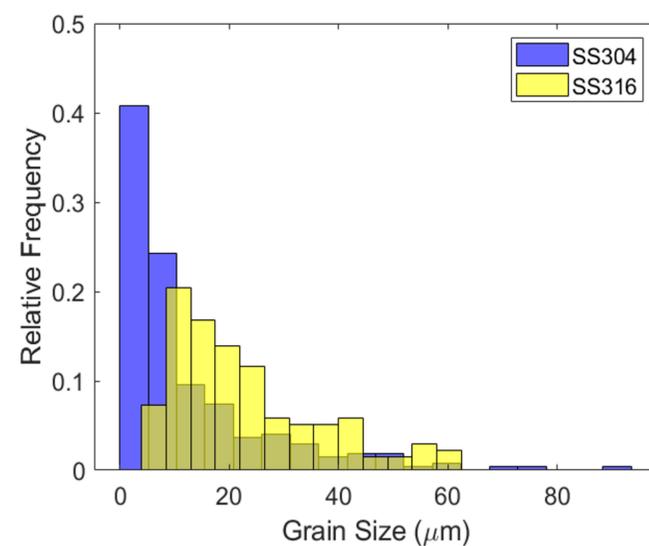
Changing chemical composition to tune solidification pathway can be a new grain boundary design criteria quantifiable by fractal dimensions



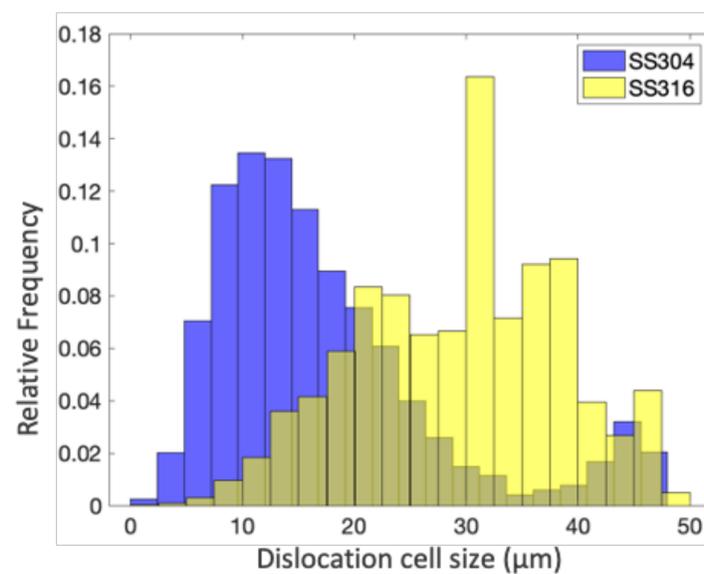
Microstructural differences



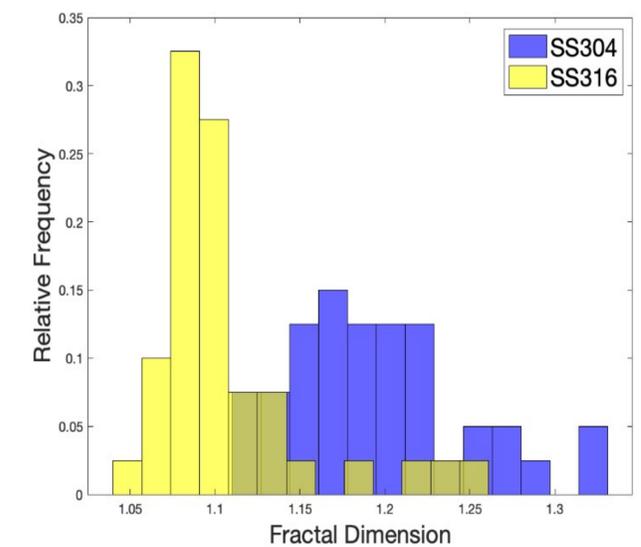
1. Grain size distribution



2. Misorientation



3. Fractal dimension



304L has a wider range encompassing many small grains ($\sim 4 \mu\text{m}$) embedded within larger grains

Median

SS304: $6.7 \pm 14.1 \mu\text{m}$

SS316: $19.1 \pm 13.6 \mu\text{m}$

Bimodal distribution of dislocation cells in 304L points to the existence of grains with many small dislocation cells and those without dislocation cells (where cell size = grain size)

Median

SS304: $10.67 \pm 7.77 \mu\text{m}$

SS316: $25.24 \pm 9.98 \mu\text{m}$

Higher fractal dimensions of grains in 304L reflects the higher roughness of grain boundaries in 304L

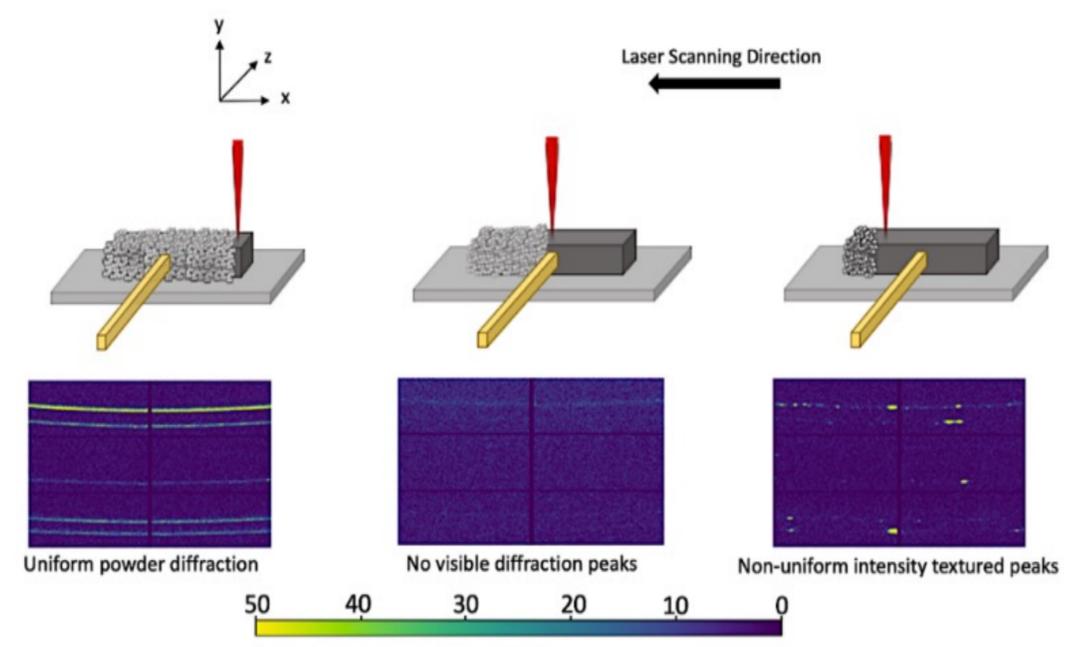
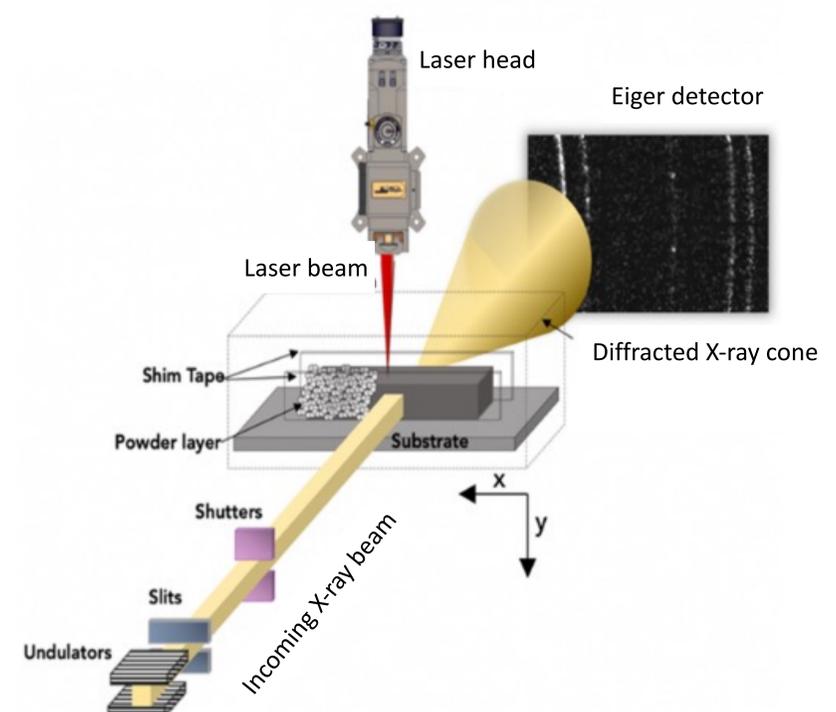
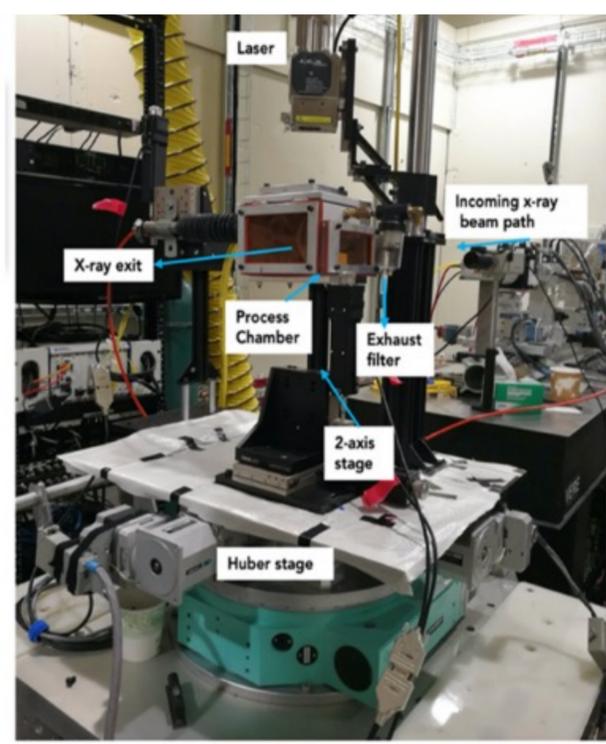
Median

SS304: 1.20 ± 0.05

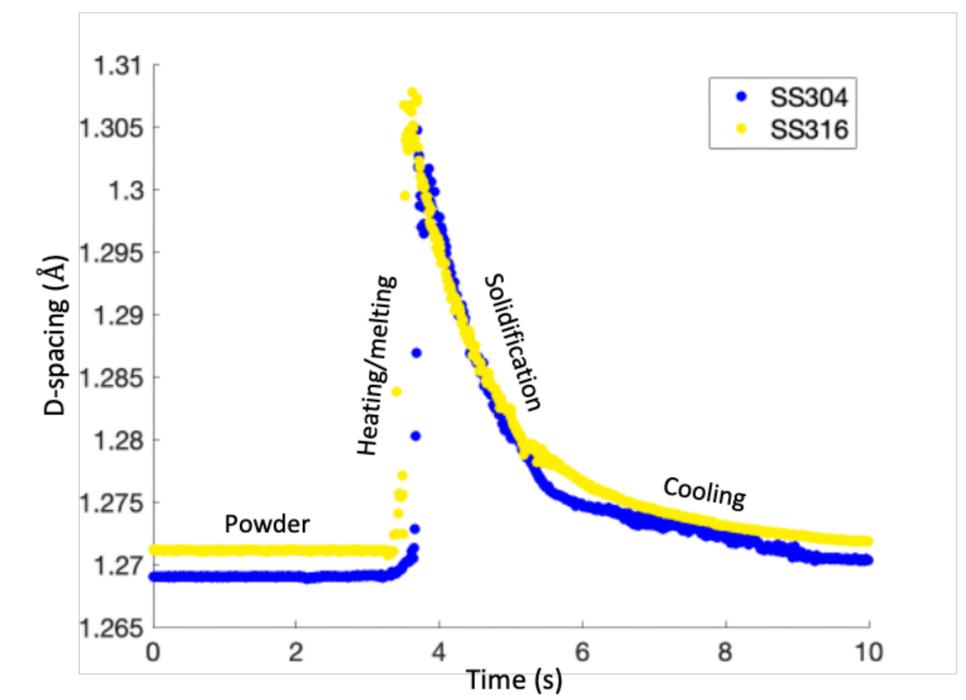
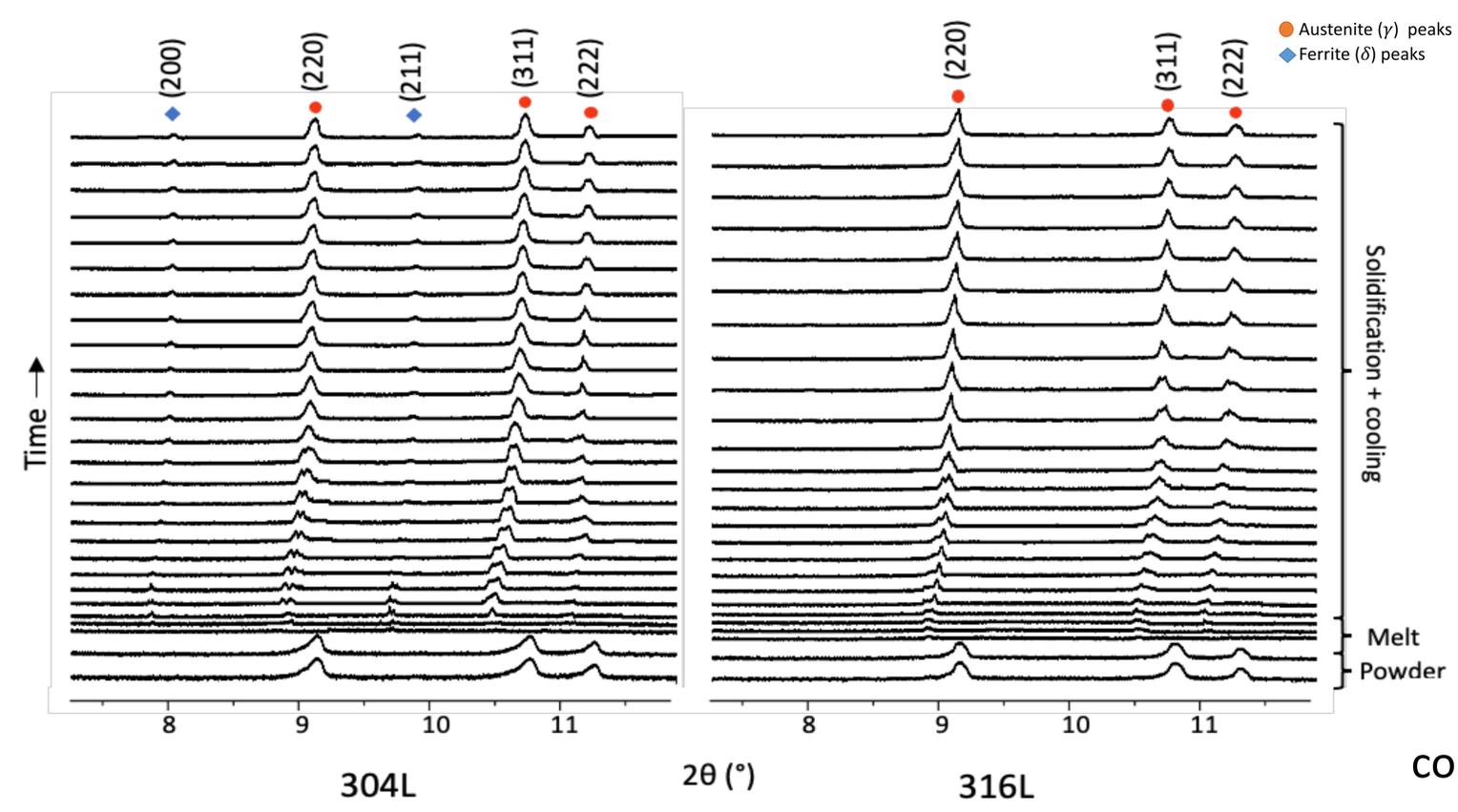
SS316: 1.10 ± 0.04



CHES setup



The incoming X-ray beam probes the sample as it undergoes the melting and solidification process of AM.



The d-spacing of the (220) peak in 304L and 316L shows comparable thermal history when processed at fixed parameters.